FLUAZIFOP-P-BUTYL (283)

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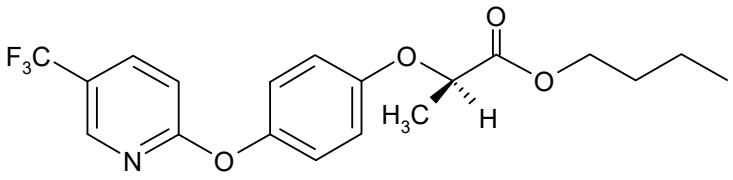
EXPLANATION

Fluazifop-P-butyl was scheduled for residue evaluation as a new compound by the 2015 JMPR at the 46th session of the CCPR (2014). Because the dossier was considered incomplete at the start of the 2015 JMPR, the evaluation was postponed to the 2016 JMPR. Fluazifop-P-butyl is used for the post-emergence control of grass (graminaceous) weeds in a wide range of broad- leaved crops. Fluazifop-P-butyl is quickly absorbed across leaf surfaces. Its hydrolysis product, fluazifop-P acid (or fluazifop-P), then distributes thouroughout the plant thourough both xylem and phloem transport and accumulates in the meristem tissue of the growing points of both shoots and roots. Speed of herbicidal action increases with weed vigour.

The Meeting received information from the manufacturer on identity, metabolism, storage stability, residue analysis, use pattern, residues resulting from supervised trials on various crops, fate of residue during processing, and livestock feeding studies.

IDENTITY

ISO common name:	fluazifop-P-butyl
Chemical name	
IUPAC:	butyl (R)-2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy]propionate
CAS:	(+)-butyl 2-[4-[[5-(trifluoromethyl)-2-pyridyl]oxy]phenoxy]propanoate
CAS Registry No:	79241-46-6
CIPAC No:	467.205
Synonyms and trade names:	aryloxyphenoxypropionate;
	PP005 (Zeneca, ICI)
	R15487 (Syngenta)
	ICIA 0005 (Zeneca, ICI)
	SL-118 (Ishihara Sangyo)
Structural formula:	Structure was verified by UV-VIS (acetonitrile/water, 50:50), IR (thin film between KBr plates), ^{1}H-NMR, ^{13}C-NMR and EI-GC-MS [Wollerton and Walter, 1999, PP5/0013, RJ2856B]
	
Molecular formula:	$C_{19}H_{20}F_3NO_4$
Molecular mass:	383.4 g/mol

Fluazifop-P-butyl is the active purified (resolved) R-enantiomer of the fluazifop-butyl (RS) racemate and this enantiomer possesses the majority of the herbicidal activity. The enantiomeric purity of fluazifop-P-butyl is 96–99% R-enantiomer and 1–4% S-enantiomer [French and Leahey, 1987, PP5/0082, RJ0569B]. Fluazifop-butyl is a racemate with its R and S enantiomers present in a 50:50 w/w ratio. The biological activity of the racemate is due primarily to the R enantiomer which gives equal herbicidal activity at half the rate of fluazifop-butyl (RS) racemate. Since a formulation based on the racemate was marketed first and was replaced by a formulation based on the R-enantiomer, several of the available studies have been performed with the racemate.

Physical and chemical properties

Pure active ingredient (fluazifop-P-butyl)

Parameter	Result	References	Guidelines/method
Appearance:	purity 98.3% odorless, pale yellow, clear liquid at 25 ºC	[Das, 2006, PP5/1446, report 115847]	EPA OPPTS Guidelines 830.6302, 830.6303 and 830.6304; visual, organoleptic
	purity 93.7% w/w colourless liquid with no characteristic odour	[Wollerton and Walter, 1999, PP5/0013, RJ2856B]	EPA OPPTS Guidelines 830.6302, 830.6303 and 830.6304 visual, organoleptic
Vapour pressure:	purity 98.3% 1.2×10^{-4} Pa at 20 ºC (extrapolated) 2.3×10^{-4} Pa at 25 ºC (extrapolated)	[Geoffrey, 2006, PP5/1458, report L06-001140]	OECD 104 Gas saturation method at 85, 94, 109, 127, 146 ºC
	purity 93.7% 0.33×10^{-4} Pa at 20°C (extrapolated)	[Wollerton and Walter, 1999, PP5/0013, RJ2856B]	OECD 104 Gas saturation method at 30, 40, 60 ºC
Melting point:	purity 98.3% no melting point solidification point (glass transition) -46 ºC	[Geoffroy, 2006; PP5/1459, report L06-001139]	OECD 102; Differential Scanning Calorimetry
	purity 93.7%; no melting point; glass-like at -20 ºC	[Wollerton and Walter, 1999, PP5/0013, RJ2856B]	OECD 102 Capillary tube in an air bath surrounded by a cooling bath of acetone and solid carbon dioxide at -40 ºC Remark: this method is not listed as OECD method
Octanol/water partition coefficient (log Pow):	purity 98.3% log Pow >5.3 at 25 ºC (concentration in the aqueous phase was less than the LOQ of 0.02 mg/L)	[Weissenfeld, 2006, PP5/1482, report A65698]	OECD 107; shake flask method Remark: this method is suitable for a log Pow between -2 and +4.
	purity 93.7% log P_{ow} 4.5 at 20 °C	[Wollerton and Walter, 1999, PP5/0013, RJ2856B]	OECD 117 HPLC method
Water solubility:	purity 98.3% 0.93 mg/L in water at 25 ºC	[Weissenfeld, 2006, PP5/1480, report A65700]	OECD 105; flask method
	purity 93.7% 1.1 mg/L in ASTM Type II water at 20 °C 1.0 mg/L in pH 5 buffer (0.2 M disodiumhydrogenphosphate-0.1 M citric acid) at 20 ºC Fluazifop-P-butyl is an extremely weak base and its water solubility will not be affected at environmentally significant pH levels	[Wollerton and Walter, 1999, PP5/0013, RJ2856B]	OECD 105; flask method

Parameter	Result	References	Guidelines/method
Solubility in organic solvents:	purity 92.2% (TGAI) * In n-heptane miscible at concentrations ≥ 60.0% w/w, brown precipitate produced at concentrations ≤ 58.0% w/w; * Miscible at all proportions in xylene, 1,2-dichloroethane, ethyl acetate, methanol, acetone or n-octanol.	[Woolley and Mullee, 1999, PP5/0014, report 1292/004]	OECD 105; flask method; range 5–95% w/w test material in organic solvent Remark: This OECD method is for water solubility; not for organic solvents
Specific gravity:	purity 98.3% 1.218 g/cm^3 at 20 ºC	[Das, 2006, PP5/1447, report 115848]	OECD 109; oscillating density meter
	purity 93.7% 1.215 g/cm^3 at 20 °C	[Wollerton and Walter, 1999, PP5/0013, RJ2856B]	OECD 109; pycnometer method
Hydrolysis:	Stable at pH 5.0 (0.01 M sodium acetate buffer) DT_{50} = 78 days at pH 7.0 (0.01 M sodium phosphate buffer) DT_{50} = 29 hours at pH 9 (0.03 M sodium borate buffer) at 25 ± 1 °C. Degradation products see [a]	[McCarron and Heath, 1989, PP5/0821, RJ0779B]	EPA guideline 161-1 0.9 mg/L sterile buffered solutions of ^{14}C-phenyl- or ^{14}C-pyridyl labelled fluazifop-P-butyl (purity > 97%) were kept for 30 days in the dark (3 days for pH 9).
Photolysis:	DT_{50} = 6.0 days at pH 5 (0.01 M sodium acetate buffer) at 25 ± 1 °C Degradation products see [b]	[Jessop *et al*., 1991, PP5/0822, report RJ0992B] [Embury and Leahey, 1994, PP5/0824, report RJ1537B]	0.5 mg/L sterile buffered solution of ^{14}C-phenyl- or ^{14}C-pyridyl labelled fluazifop-P-butyl was irradiated with a Xenon arc lamp at a light intensity between 33.72-42.91 W/m^2 for 5 consecutive days. Irradiation equivalent to 8 days of Florida summer sunlight (latitude 25-35 ºN, 12 hours of light per day).
Dissociation constant:	purity 98.3% No pKa was found in the range of 1.0 to 12.0	[Martin, 2006, PP5/1449, report L06-001141]	OECD 112 spectrophotometric titration
	purity 93.7% The dissociation constant is estimated to be much less than 1 i.e. very weakly basic	[Wollerton and Walter, 1999, PP5/0013, RJ2856B]	Estimated based on its structure by using the Hammett equation

[a] Total recovered radioactivity is 90–105% TAR. At pH 5, parent was recovered at > 96% TAR). At pH 7 parent decreased to 69–77% after 30days. The main decomposition product was fluazifop acid (II, up to 24% TAR). At pH 9, parent decreased to 18–23% TAR after about 3 days. The main decomposition product was fluazifop acid (II) at 70–79% TAR.

[b] Total recovered radioactivity is 97–87% TAR on day 1 to 5. Fluazifop-P-butyl decreased from 85% TAR on day 1 to 43% TAR on day 5 for the phenyl label and 82% TAR on day 1 to 32% TAR on day 5 for the pyridyl label. Main degradation products were:Pyr-Ph ether (IV, up to 3.5% TAR), SYN546933 (up to 10.8% TAR), 4-pyrano[1,2,3-b]pyridine-6-carboxylic acid (up to 12.4% TAR) and $^{14}CO_2$ (12.8% for phenyl label, 2.1% TAR for pyridyl label). The remainder was a mixture of at least 8 ethyl acetate and/or water soluble compounds.

Fluazifop-P-butyl Technical Grade Active Ingredient (TGAI)

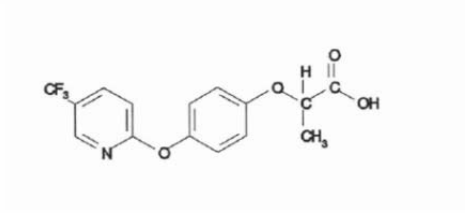
Parameter	Result	References	Guidelines
Appearance:	purity 92.2% (TGAI) dark brown, opaque liquid at 20.0 ± 0.5 ºC with a weakly aromatic odour, characteristic of toluene	[Woolley and Mullee, 1999, PP5/0014, report 1292/004]	EPA OPPTS Guidelines 830.6302, 830.6303 and 830.6304 visual, organoleptic
Density:	purity 92.2% (TGAI) 1.21 g/cm^3 at 20.0 ± 0.5 ºC	[Woolley and Mullee, 1999, PP5/0014, report 1292/004]	OECD 109; pycnometer
Melting range:	purity 92.2% (TGAI) Pour Point 17.0 ºC (256 °K)	[Woolley and Mullee, 1999, PP5/0014]	OECD 102 Test jar enclosed in a glass

	During cooling the test material became increasingly viscous and therefore the pour point was more appropriate than the freezing point determination.		jacket and kept in a bath of acetone and solid carbon dioxide at -35 ºC Remark: this method is not listed as OECD method
Stability:	purity 92.2% (TGAI) Boiling temperature 370 ºC (643K) at 101 kPa with possible slight decomposition.	[Woolley and Mullee, 1999, PP5/0014, report 1292/004]	OECD 103; differential scannig calorimetry
	This compound is not expected to be sensitive to metals or metal ions; No sensitivity to metals has been observed	[Woolley and Mullee, 1999, PP5/0014, report 1292/004]	OPPTS 830.6313
	purity 92.2% (TGAI) No significant physical or chemical degradation at 54 ± 2 ºC for 14 days. The test material is stable for at least 3 years and 11 months at ambient temperature	[Woolley and Mullee, 1999, PP5/0014, report 1292/004]	OPPTS 830.6317

Fluazifop-butyl (racemate)

Parameter	Result	References	Guidelines/method
Hydrolysis in sterile aqueous solution:	0.1 mg/L in the dark at room temperature Stable (DT 50 >120 days) at pH 4 (0.05 M potassium hydrogen phalate buffer) Stable (DT 50 >120 days) in distilled water (pH 6) Stable (DT 50 >120 days) at pH 7 (0.025 M potassium phosphate buffer) DT_{50} = 1.8 days at pH 9 (0.01 M sodium borate buffer) 0.1 mg/L in the dark at 40 °C Stable (DT_{50} >120 days) at pH 4 (0.05 M potassium hydrogen phalate buffer) DT_{50} = 35 days in distilled water (pH 6) DT_{50} = 17 days at pH 7 (0.025 M potassium phosphate buffer) DT_{50} = 0.2 days at pH 9 (0.01 M sodium borate buffer) Fluazifop acid (II) was the only decomposition product. No other compounds >5% TAR were present.	[Makin *et al*, 1980, PP9/0286, RJ0145B]	EPA guideline 161-1 0.1 mg/L sterile buffered solutions of ^{14}C-phenyl-labelled fluazifop-butyl (purity not indicated) were kept for 30 days in the dark
Photolysis in sterile aqueous solutions	Stable in distilled water under photolysis conditions Fluazifop-butyl (RS) decreased to 89% (phenyl label) or 77% (pyridinyl label) after 64 days. Degradation products were fluazifop acid (II, up to 2-3% TAR at day 64) and Pyr-Ph ether (IV, up to 2-4% TAR at day 64)	[MacNeil *et al*., 1981, PP9/0287, report RJ0176B]	0.1 mg/L ^{14}C-phenyl- or ^{14}C-pyridyl labelled fluazifop-butyl (RS) in 95:5 water/acetonitrile. The solution was exposed to sunlight from 5 June to 8 August, 1980 at latitude 51°23'N and longitude 0°47'W (Bracknell, Berkshire, UK) for a period of 64 days

Degradation product / metabolite (fluazifop-P-acid)

ISO common name:	fluazifop-P
Chemical name	
IUPAC:	(R)-2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy]propionic acid
CAS:	(R)-2-[4-[[5-(trifluoromethyl)-2-pyridinyl]oxy]phenoxy]propanoic acid
CAS Registry No:	83066-88-0
CIPAC No:	-
Synonyms and trade names: -	
Structural formula:	Structure was verified by UV-VIS, Fourier Transform IR (KBr disc); 1H NMR, 13C-NMR, EI-MS [Wollerton and Husband, 1992, R156172/0001, report RJ1263B; Sparrow, 2015, PP5_50598, report PC-15-037]
	
Molecular formula:	$C_{15}H_{12}F_3NO_4$
Molecular weight:	327.3

Parameter	Result	References	Guidelines/method
Appearance:	purity 100% pale yellow, clear glass-like liquid at 25 ºC	[Wollerton and Husband, 1992, R156172/0001, report RJ1263B; Sparrow, 2015, PP5_50598, report PC-15-037]	EPA OPPTS Guidelines 830.6302, 830.6303 visual
Vapour pressure:	purity 100% 7.9×10^{-10} Pa at 20 ºC (extrapolated)	[Wollerton and Husband, 1992, R156172/0001, report RJ1263B; Sparrow, 2015, PP5_50598, report PC-15-037]	OECD 104 Gas saturation method between 40-60 ºC
Melting point:	not applicable; fluazifop-P acid (II) is a glass like material purity 100% glass transition: +3.8 ºC	[Wollerton and Husband, 1992, R156172/0001, report RJ1263B; Sparrow, 2015, PP5_50598, report PC-15-037]	ASTM E 1356-91 Differential Scanning Calorimetry
Octanol/water partition coefficient (log P_{ow}):	true log Pow = +3.2 (calculated) apparent log Pow = +3.1 (measured at pH 2.57, at 20 °C) apparent log P_{ow} = -0.80 (calculated at pH 7.00 when no ion pairing is assumed) [a]	[Wollerton and Husband, 1992, R156172/0001, report RJ1263B; Sparrow, 2015, PP5_50598, report PC-15-037]	OECD 107; shake flask method Remark: this method is suitable for a log Pow between -2 and +4.
Water solubility:	purity 100% 780 mg/L in purified ASTM type II water at 20 °C	[Wollerton and Husband, 1992, R156172/0001, report	OECD 105; shake flask method

Parameter	Result	References	Guidelines/method
	8300 mg/L in pH 4.8 buffer (CONVOL) at 20 ºC Fluazifop-P acid (II) is significantly acidic and readily forms salts in alkaline solutions. The phosphate and borate buffer capacities (nominally 0.1 M) were exceeded and pHs dropped from pH 6.92 to 4.1 and pH 9.4 to 4.8 respectively. Hence no meaningful values are obtained in pH 7 and 9 buffered waters.	RJ1263B; Sparrow, 2015, PP5_50598, report PC-15-037]	
Hydrolysis:	Stable at pH 4.6 (0.01 M sodium citrate buffer) Stable at pH 7.2 (0.01 M TRIS – maleic acid buffer) Stable at pH 9.0 (0.01 M sodium borate buffer) at 25 ± 1 °C.	[Goodyear, 1995, PP5/0825, report 38/187-1015]	EPA Pesticide Assessment Guidelines Subdivision N para 161-1 (October 1982) 5 mg/L sterile buffered solutions of ^{14}C-phenyl- or ^{14}C-pyridyl labelled fluazifop-P acid (II) (purity > 98%) were kept for 31 days in the dark.
Photolysis:	DT_{50} = 17 days at pH 7 (0.02 M potassium dihydrogen phosphate buffer) at 25 ± 2 °C (single first order kinetics, applicable to Europe and North America at latitudes 30°, 40°, 50°) Degradation products see[b]	[Graham *et al*., 2013, R156172_10000, report 1983/106]	OECD 316 5 mg/L sterile buffered solution of ^{14}C-phenyl- or ^{14}C-pyridyl labelled fluazifop-P acid (II) irradiated for 35 or 30 consecutive days, respectively, with a Xenon lamp. The light intensity was 20.93 W/m^2 (300-400 nm), which is equivalent to 0.82 day of UK/US summer sunlight.
Dissociation constant:	purity 100% 2.98 in purified water at 20 °C This value was obtained in a 2.3×10^{-3} M fluazifop-P acid (II) solution in water containing 13.8% acetonitrile to improve solubility at the initial starting pH. The effect of acetonitrile addition was corrected by determining the pH difference with a model compound 2-(4-chlorophenoxy)propionic acid which was sufficiently soluble in water at all pHs.	[Wollerton and Husband, 1992, R156172/0001, report RJ1263B; Sparrow, 2015, PP5_50598, report PC-15-037]	OECD 112 potentiometric method

[a] The partition coefficient is defined as the ratio of the equilibrium concentrations of a dissolved, neutral substance in a two phase system consisting of two largely immiscible solvents. Fluazifop-P acid (II) was dissolved in octanol (0.01 M) and then partitioned with buffered water in 3 different octanol/water ratios: 1:1, 1:4, 2:3. Fluazifop-P acid (II) has an acidic pKa. Therefore during octanol/water partitioning dissociation will occur in the aqueous phas. The analytical method employed did not differentiate between the neutral and the dissociated species. Hence the value of the partition coefficient is an apparent result. The apparent low Pow was measured in water buffered at pH 2.57 (using citric acid and trisodium citrate), just below the pKa. From knowledge of the pKa and the apparent (measured) log Pow, the true log Pow can be calculated as well as the theoretical apparent low Pow at pH 7.00.

[b] Total recovered radioactivity is 97–103% TAR on day 0, 3, 5, 8, 15/18, 21/28, 30/35 days. Fluazifop-P acid (II) decreased from 78% TAR on day 3 to 21% TAR on day 35 for the phenyl label and 85% TAR on day 3 to 45% TAR on day 30 for the pyridyl label. Main degradation products were: Pyr-Ph ether (IV, CGA181847, up to 31.4% TAR), SYN546933 (up to 13% TAR pyridyl label only), R201189 (up to 13% TAR) and $^{14}CO_2$ (15% TAR for phenyl label, 3.8% TAR for pyridyl label). A number of minor degradation products (<5% TAR) and thouree unknown degradates (> 5% TAR, <10% TAR, phenyl label only) were observed.

Formulations

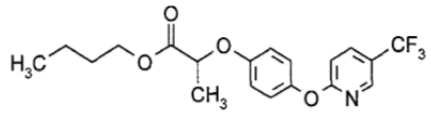
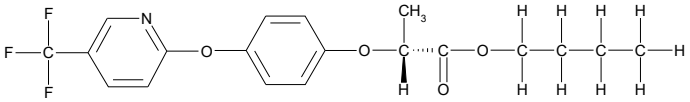
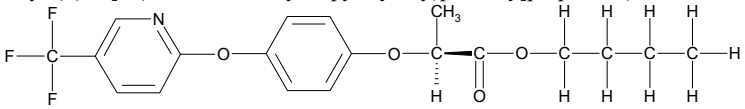
FAO specifications for fluazifop-P-butyl have been published by JMPS 2000 for: technical material (FAO specification 467.205/TC (2000)), emulsifiable concentrate (FAO specification 467.205/EC (2000)), and oil-in-water emulsions (FAO specification 467.205/EW (2000)).

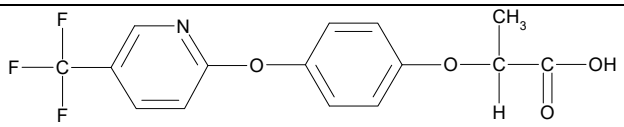
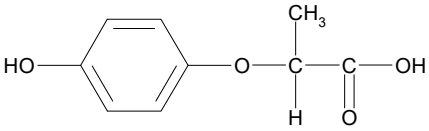
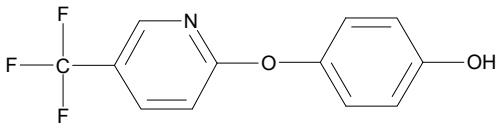
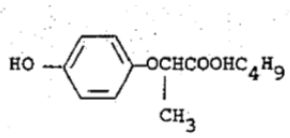
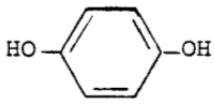
Formulations authorised for use: Brazil (Fusilade® 250 g ai/L EW, Fusiflex® 125 g ai/L SC), Belgium, France, Germany, the Netherlands, United Kingdom (FusiladeMax 125 g ai/L EC) and USA (Fusilade DX, 250 g ai/L EC).

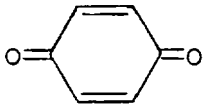
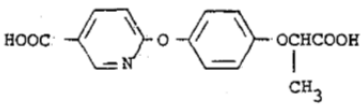
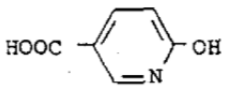
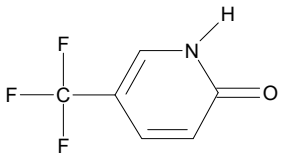
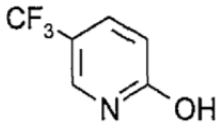
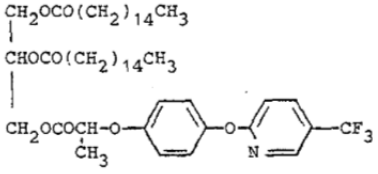
Reference compounds

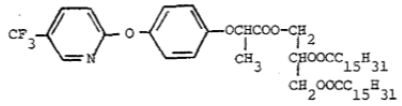
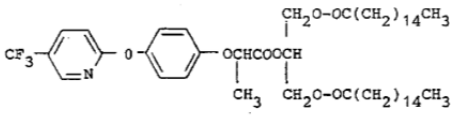
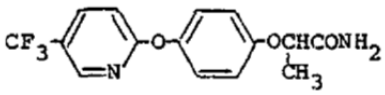
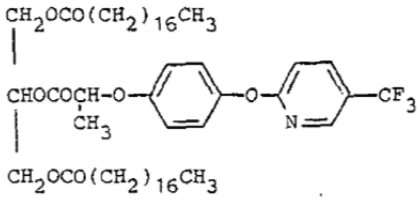
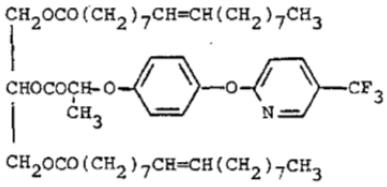
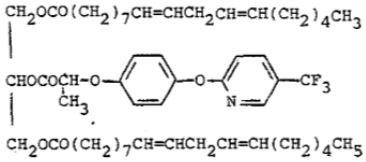
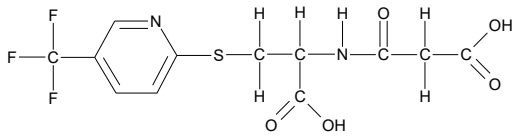
Table 1 lists the reference compounds used in the various study reports. It is indicated which of these reference compounds were identified in soil degradation studies, hydrolysis and photolysis studies, plants, rotational crops and livestock.

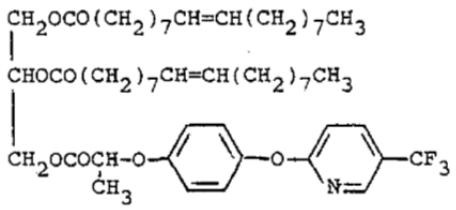
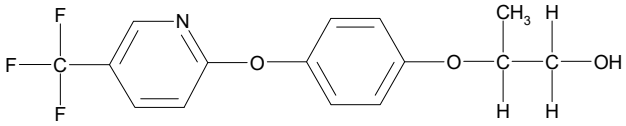
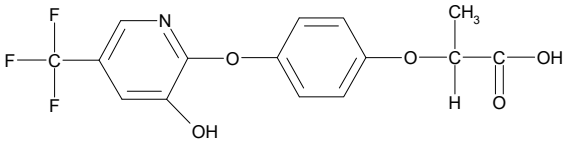
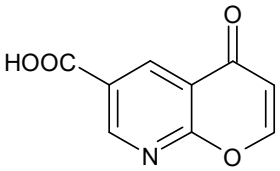
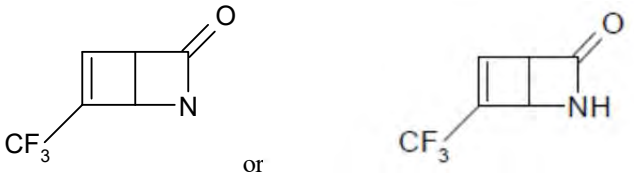
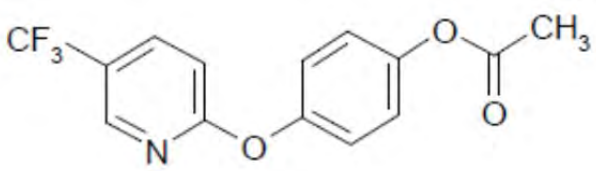
Table 1 List of reference compounds used in various study reports

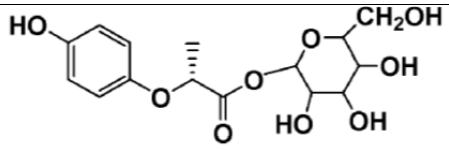
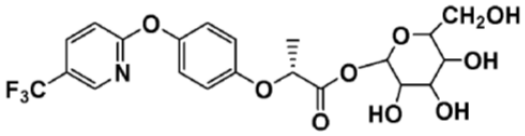
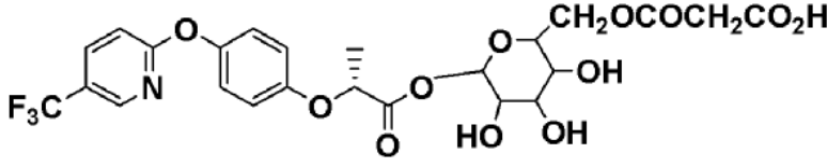
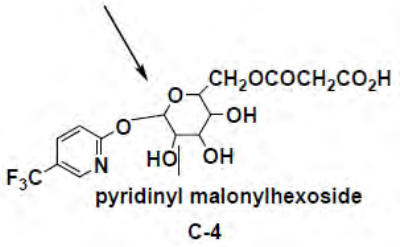
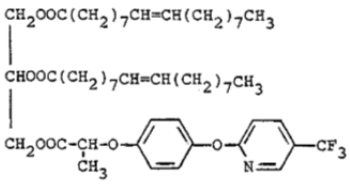
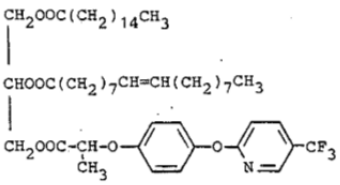
Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in Needs to be checked
1 (I)	Fluazifop-butyl (racemate) R117009  Fluazifop-P-butyl (i.e. R enantiomer), Fluazifop-butyl, R enantiomer butyl-(R)-2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy]propionate) butyl-(R)-2-[4-[[5-(trifluoromethyl)-2-pyridyl]oxy]phenoxy]propanoate PP005/01 R154875 (R enantiomer) 1689W-001 CAS no 79241-46-6  Fluazifop-butyl, S enantiomer butyl-(S)-2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy]propionate) 	aerobic soil degradation; soil photolysis; cucumber; lettuce; celery; cotton forage; cotton seeds; soya forage; soya seeds carrot roots
2 (II) MW 327.3	Fluazifop acid (racemate) (RS)-2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy]propionic acid 2-[4-[[5-(trifluoromethyl)-2-pyridyl]oxy]phenoxy]propanoic acid R115625 CASno 69335-91-7; Fluazifop-P acid (i.e. R enantiomer) Fluazifop-P PP005/02 R156172 1690W-001	hydrolysis in water; aerobic soil degradation; soil photolysis; cucumber; lettuce; celery; endive; alfalfa forage; cotton forage; cotton seeds rape seeds;

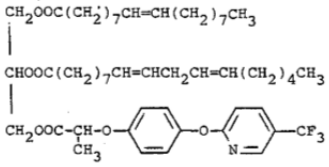
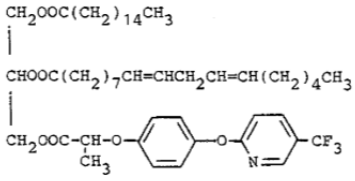
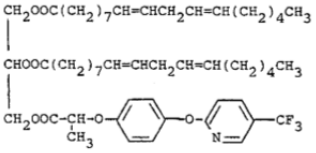
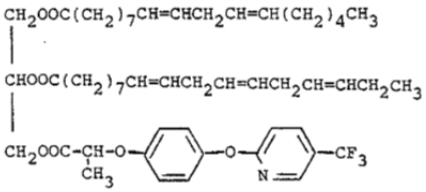
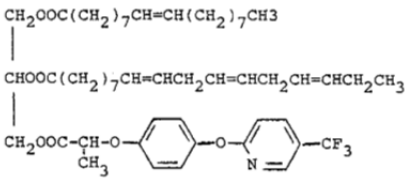
Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in Needs to be checked
		soya forage; soya seeds; carrot roots; carrot foliage potato tubers; sugarbeet roots; milk, eggs, livestock tissues
3 (III) MW = 182.17	despyridinyl acid (RS)-2-(4-hydroxyphenoxy)propionic acid PP009/03 R118106 CGA214111 1690W-002 CAS nr 94050-90-5 	cucumber; lettuce; celery; endive cotton forage; cotton seeds; soya seeds; carrot roots; carrot foliage potato tubers; sugarbeet roots
4 (IV) MW 255.20	Pyr-Ph ether IUPAC: 4-(5-trifluoromethylpyridin-2-yloxy)phenol CAS: 4-[[5-trifluoromethyl-2-pyridinyl]oxy]-phenol Other: 2-(4-hydroxyphenoxy)-5-trifluoromethyl pyridine Codes: PP009/04; R150397; CGA181847; CSAA169875, 1690W-003 CAS number: 69045-85-8 $C_{12}H_8F_3NO_2$ SMILES code: n1cc(C(F)(F)F)ccc1Oc2ccc(O)cc2 	photolysis in water; aerobic soil degradation; photolysis on soil; endive; cotton forage; soya forage; sugarbeet roots; cow liver; cow kidney; goat kidney; goat milk; egg white
5 (V)	(RS)-butyl-2-(4-hydroxyphenoxy)propanoate; butyl-2-(4-hydroxyphenoxy)propionate 	
6 (VI)	quinol 	
7 (VII)	benzoquinone	aerobic soil degradation

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in Needs to be checked
		
8 (VIII)	(RS)-6-[4-(1-carboxyethoxy)phenoxy]nicotinic acid 2-[4-(5-carboxy-2-pyridyloxy)phenoxy]propionic acid 	
9 (IX)	6-hydroxynicotinic acid; 2-hydroxypyridine-5-carboxylic acid CGA 319251 1690W-005 	
10 (X) MW 163.10	CF_3-pyridone pyridinol 5-trifluoromethyl-2-pyridone 5-(trifluoromethyl)-2-(1H)pyridinone 5-trifluoromethyl-pyridin-2-ol 5-trifluoromethyl-pyrid-2-one PP009/10 R154719 CGA142110 1690W-004 CAS no 33252-63-0  or 	aerobic soil degradation; photolysis on soil; celery; endive cotton forage; soya forage; soya seeds; carrot roots; carrot foliage sugarbeet roots; rotational crops
15 (XV)	mixture of X-glycerol, 1,2-dipalmitate 2-[(RS)-2-(4-(5-trifluoromethyl-2-pyridyloxy)phenoxypropionate)] and X-glycerol, 1,3-dipalmitate 2-[(RS)-2-(4-(5-trifluoromethyl-2-pyridyloxy)phenoxy) propionate)] (X-glycerol means that a glycerol backbone is chemically linked via ester bonds to two lipids and fluazifop acid (II), forming a triglyceride moiety) 	
16 (XVI)	mixture of 1,2 and 1,3-dipalmityl triglyceride esters of fluazifop (1,2-dipalmyltyl is shown in the figure below)	

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in Needs to be checked
	 (1,3-dipalmytyl is shown in the figure below) 	
24 (XXIV)	(RS) 2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy]propionamide 	
25 (XXV)	X-glycerol 1,3-distearate 2-[(RS)-2-(4-(5-trifluoromethyl-2-pyridyloxy)) phenoxypropionate (for the meaning of X-glycerol, see compound 15 (XV)) 	
26 (XXVI)	X-glycerol 1,3-dioleate 2-[(RS)-2-(4-(5-trifluoromethyl-2-pyridyloxy)phenoxy)propionate (for the meaning of X-glycerol, see compound 15 (XV)) 	
27 (XXVII)	lipophilic fluazifop conjugate X-glycerol 1,3 dilinoleate 2-[(RS)-2-(4-(5-trifluoromethyl-2-pyridyloxy)phenoxy)propionate] (for the meaning of X-glycerol, see compound 15 (XV)) 	
28 (XXVIII)	N-[1-carboxy-2-5-trifluoromethyl-2-pyridylthio)ethyl] malonamic acid 	celery

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in Needs to be checked
30 (XXX)	X-glycerol 1,2 dioletate 2-[(RS)-2-(4-(5-trifluoromethyl-2-pryidyloxy)phenoxy)propionate] (for the meaning of X-glycerol, see compound 15 (XV)) 	
34 (XXXIV)	Fluazifop alcohol (RS)-2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy] propanol 	lettuce; celery; carrot roots
40 (XL) MW 343.3	Hydroxyfluazifop acid 2-[4-(3-hydroxy-5-trifluoromethyl-2-phenoxy)pyridyloxy] propionic acid 	celery; potato tubers
	4-pyrano[1,2,3-b]pyridine-6-carboxylic acid 	photolysis in water
SYN546933	cis-2-amino-3-trifluoromethylcyclobut-3-ene carboxylic acid lactam (photo-rearrangement product of CF3-pyridone) 	photolysis in water
R201189	IUPAC: acetic acid 4-(5-trifluoromethyl-pyridin-2-yloxy)-phenyl ester 	photolysis in water
E1, E4=C1	despyridinyl acid hexoside (despyridinyl acid conjugate)	endive carrot roots carrot foliage

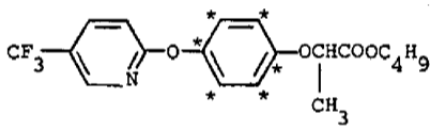
Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in Needs to be checked
		
E5, E7=C3	fluazifop acid hexoside (fluazifop acid conjugate) 	endive carrot roots carrot foliage
E6=C2	fluazifop acid malonylhexoside (fluazifop acid conjugate) 	endive carrot roots carrot foliage
C4	pyridinyl malonylhexoside of CF3-pyridone  pyridinyl malonylhexoside C-4	carrot roots carrot foliage
a	glyceride ester of fluazifop acid fatty acid conjugate of fluazifop acid 	soya bean seeds
b	glyceride ester of fluazifop acid fatty acid conjugate of fluazifop acid 	soya bean seeds
c	glyceride ester of fluazifop acid fatty acid conjugate of fluazifop acid	soya bean seeds

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in Needs to be checked
		
d	glyceride ester of fluazifop acid fatty acid conjugate of fluazifop acid 	soya bean seeds
e	glyceride ester of fluazifop acid fatty acid conjugate of fluazifop acid 	soya bean seeds
f	glyceride ester of fluazifop acid fatty acid conjugate of fluazifop acid 	soya bean seeds
g	glyceride ester of fluazifop acid fatty acid conjugate of fluazifop acid 	soya bean seeds

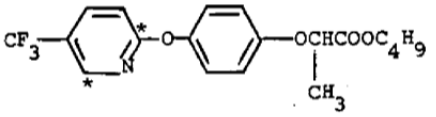
METABOLISM AND ENVIRONMENTAL FATE

The Meeting received information on the fate of fluazifop-P-butyl in plant commodities, rotational crops, livestock and soil. In some studies fluazifop-butyl (RS) was applied as a 50:50 mixture of the ^{14}C-phenyl- and ^{14}C-pyridyl-labelled fluazifop-butyl (RS), while in other studies only the R-

enantiomer or the S-enantiomer of ^{14}C-phenyl or ^{14}C-pyridyl labelled fluazifop-butyl was used. The position of the radiolabel in the compound used in the metabolism and environmental fate studies is indicated in Figure 1.



^{14}C-phenyl-labelled fluazifop-P-butyl



^{14}C-pyridyl-labelled fluazifop-P-butyl

Figure 1 Position of [^{14}C]- radiolabel in fluazifop-P-butyl; * denotes the position of the radiolabel

Plant metabolism

The Meeting received plant metabolism studies for fluazifop-P-butyl after soil directed or foliar applications on fruits and fruiting vegetables (grapes, cucumbers), leafy vegetables (lettuce, celery, endive), cereals (maize forage), pulses and oilseeds (alfalfa forage, cotton forage and seeds, oilseed rape seeds, soya bean forage and seeds) and root and tuber vegetables (carrot roots and foliage, potato tubers, sugarbeet roots). Fluazifop-butyl was applied as the ^{14}C-phenyl- and/or ^{14}C-pyridyl-labelled fluazifop-butyl as R-enantiomer, S-enantiomer or RS racemate. Additional studies with non-labelled samples from supervised trials were provided to investigate the nature of residues.

The majority of the provided metabolism studies were difficult to interpret, since the extracted or hydrolysed residues partitioned between an organic and aqueous phase and only the residues that partitioned into the organic phase were analysed. The aqueous phases were generally subjected to hydrolysis and then partitioned again between an organic and an aqueous phase. Since the analytical method section indicates that the water phase should be acidified to pH < 1, it appears that the pH of the water phase is essential for quantitative partitioning of fluazifop acid (II). CF3-pyridone (X) might be underestimated because of partial partitioning into the aqueous phase as is shown in the celery metabolism study. In some metabolism studies water phases were acidified to pH 2 or 3 before partitioning with diethyl ether. This implies that fluazifop acid (II), despyridinyl acid (III), fluazifop alcohol (XXXIV) and possibly Pyr-Ph ether (IV) and hydroxyfluazifop acid (XL) are partionally partitioned into the water phase and are possibly underestimated in these metabolism studies. Additional information on the partition characteristics of these metabolites at various pH values is desirable.

Interpretation of the metabolism studies is complicated further, because various hydrolysis conditions were used, of which some resulted in the degradation of fluazifop acid (II) into CF3-pyridone (X) and despyridinyl acid (III), the same compounds that were found as plant metabolites. Since the hydrolysis conditions affected the composition of the detected residues, the Meeting received several additional studies to show stability of fluazifop acid (II) or CF3-pyridone (X) under the hydrolysis conditions applied. Stability of despyridinyl acid (III), Pyr-Ph ether (IV) and hydroxyfluazifop acid (XL) under these conditions has not been investigated, but is desirable. Also

information on the cleavage efficiency of these metabolites from their conjugates is desirable under the hydrolysis conditions used.

For each metabolism study below it is indicated whether the study has acceptable hydrolysis conditions.

Stability of fluazifop-butyl and its metabolites under various hydrolysis conditions

Studies were submitted to investigate the behaviour and stability of fluazifop-butyl, fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV) and/or CF3-pyridone (X) under various hydrolysis conditions.

Study 1

The behaviour of ^{14}C fluazifop-butyl (RS) under base or acid hydrolysis conditions has been investigated [Evans and Cavell, 1980, PP9/0285, report RJ0121B]. ^{14}C-phenyl labelled fluazifop-butyl (RS) was refluxed for 14 hours in aqueous 0.001 M NaOH (pH 10.6) or 0.001 M HCl (pH 2.9) solution. The top of the condenser was connected to dreshel bottles to collect volatiles. Radioactivity in the hydrolysis solution and extracts were analysed by LSC. One and two dimensional TLC with reference standards for parent, fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), V were used to identify degradation products. Quantitative data are not indicated in the report.

The hydrolysis studies with 0.001 M HCl (pH 2.9) were complicated by the steam volatility of fluazifop-butyl at this pH. About 40–73% of the applied radioactivity was found in the condenser after acid hydrolysis where it was much less susceptible to hydrolysis. Fluazifop acid (II) was characterised as the major hydrolysis product in the solution remaining in the flask and despyridinyl acid (III) and Pyr-Ph ether (IV) were characterised as a very minor hydrolysis products.

The 0.001 M NaOH (pH 11) hydrolysis solutions were extracted twice with diethyl ether (0.8-2.5% TAR in ether A). The remaining aqueous phase was acidified to 0.1 M HCl and extracted twice to result in a diethyl ether phase (72–84% TAR in ether B) and a water phase (0.6–13% TAR). On acidification of the hydrolysis solution little or no loss of radioactivity occurred, proving that $^{14}CO2$ was not a significant hydrolysis product. The major component in diethyl ether phase A and nearly all the radioactivity in the diethylether phase B was identified as fluazifop acid (II). Fluazifop acid (II) could be partitioned into diethylether after acidification of the aqueous phase. Identity of this compound was confirmed by EI-MS. Despyridinyl acid (III) was characterised as a very minor hydrolysis product in ether phase B. The aqueous phase (12.6% TAR) contained compounds that chouromatographed at or near the origin of the TLC plate and their identity is unknown. About 1–2% of the initial radioactivity accumulated in the sodium hydroxide trap and this was probably $^{14}CO_2$.

Reviewer's conclusion: This study with fluazifop-butyl indicates that reflux at pH 2.9 or lower leads to losses of total fluazifop due to the volatility of fluazifop-butyl. The study further indicates that reflux at pH 11 or higher leads to degradation of total fluazifop (13% aqueous soluble unknown compounds). The study further indicates that total fluazifop quantitatively partitioned into diethyl ether after acidification of the aqueous phase.

Studies 2-7

Six studies were submitted where the stability of fluazifop acid (II) and CF3-pyridone (X) was investigated under a range of hydrolysis conditions in the absence or presence of crop matrix [Atreya *et al*, 1981, 462775, report PP009B030; Cavell *et al*., 1981, PP9/0200, report RJ0171B; Goddard *et al*., 1981, PP9/0203, report RJ0196B; MacNeil *et al*., 1981, PP9/0045, report RJ0211B]. [MacNeil and Cavell, 1984, PP9/0202, report RJ0342B] and [MacNeil *et al*., 1981, PP9/0201, report RJ0213B] described hydrolysis experiments already reported in report [RJ0211B].

Aqueous cotton leaf extracts were prepared by macerating cotton leaves from a young plant with acetonitrile/water (1:1, v/v). The extract was partitioned between diethylether and water. The remaining water fraction was rotary evaporated at 30 °C to remove residual diethyl ether. This water fraction was used for the hydrolysis experiments.

Aqueous cotton or soya seed extracts were prepared by extracting soya seeds or delinted cotton seeds with hexane and diethyl ether. The remaining solids were soaked in water for 2 hours, followed by two extractions with acetonitrile/water (1:1, v/v). The acetonitrile was removed by rotatory evaporation and the remaining water phase was adjusted to 0.1 M HCl and then extracted with diethyl ether. The remaining water fraction was rotary evaporated at 30 °C to remove residual diethyl ether. This water water fraction was used for the hydrolysis experiments.

The aqueous crop extracts were adjusted to the desired hydrolysis solvents (as indicated in Tables 2 and 3). ^{14}C-phenyl labelled fluazifop acid (II) or ^{14}C-CF3-pyridone (X) was added to these solutions or to solutions without crop extract and refluxed for the desired time period (as indicated in Table 2 and Table 3). The hydrolysates were then adjusted to pH 1 with HCl and then partitioned with diethyl ether. The diethyl ether fractions were analysed by TLC. Results are shown in Tables 2 and 3.

In case of hydrolysis with 0.5 M NaOH in methanol, water was added to the hydrolysate and the methanol was removed by rotary evaporation. The aqueous solution was first partitioned with diethyl ether. The aqueous phase was then acidified with HCl to pH1 and partitioned with diethyl ether. The diethyl ether phases were combined and analysed by TLC. Results are shown in Table 2.

Under the hydrolysis conditions used in report RJ0196B, all radioactivity derived from the ^{14}C-phenyl labelled fluazifop acid (II) was diethylether soluble.

Reviewer's conclusion:

- The presence of crop extractives did not affect the hydrolysis behaviour of fluazifop acid (II) or CF3-pyridone (X). Fluazifop acid (II) and CF3-pyridone (X) can be quantitatively partitioned into diethylether if the water phase is acidified.
- Fluazifop acid (II) was not significantly cleaved under the following conditions: 3 hours reflux in 0.1 M NaOH, 1 hour reflux in 0.5 M NaOH in methanol, 1 hour reflux in 1 M NaOH (see also GRM044.01 validation), 3 hours reflux in 0.1 M HCl or 6 hours at 60 ºC in 1–6 M HCl. Fluazifop acid (II) is cleaved at the pyridine-phenyl ether link resulting in despyridinyl acid (III, phenyl label) and CF3-pyridone (X, pyridyl label), when refluxed for 1 hour in 1–6 M HCl.
- CF3-pyridone (X) is stable during acid hydrolysis (1 hour reflux in 6 M HCl), but degrades to compound 9 when refluxed in basic conditions (reflux in 0.1 M NaOH). Compound 9 does not partition into diethyl ether from an acidic aqueous solution.

Analytical methods on plant commodities used 6 M HCl for 1 hour at 60 ºC or 1 hour reflux in 0.2 M NaOH in methanol (PP009B152, PPRAM 122) or 1 hour reflux in 1 M NaOH (GRM044.01A) to determine total fluazifop. Under these conditions the released fluazifop acid (II) remains stable.

Analytical methods on plant commodities used 6 M HCl for 1 hour at 60 °C or 1 hour reflux in 1 M HCl to determine total CF3-pyridone (X) or 1 hour reflux in 6 M HCl to determine total despyridinyl acid (III) (see analytical method section). Under these conditions the released CF3-pyridone (X) remains stable. Stability of despyridinyl acid (III) under these conditions has not been verified.

Many of the metabolism studies used hydrolysis conditions that affected the composition of the detected residues. For some of these hydrolysis conditions, stability of total fluazifop or its degradates has not been verified. Additional stability studies are desirable for fluazifop acid (II), CF3-pyridone (X), despyridinyl acid (III), Pyr-Ph ether (IV) and hydroxyfluazifop acid (XL) for:

- 2 M HCl for 2 hours at 60 ºC plus 6 M HCl for 2 hours at 80 ºC (celery metabolism study)
- 6 M HCl for 6 hours at 80 ºC (celery metabolism study)
- β-glucosidase treatment overnight at 37 ºC (endive metabolism study)
- 24% KOH treatment overnight at room temperature (endive, carrot metabolism study)

- 6 M HCl at room temperature for 24 hours (endive, carrot metabolism; confined rotation)
- 6 M HCl for 2 hours at 90 ºC (alfalfa forage metabolism study)
- 0.2 M NaOH for 3 hours reflux (soya metabolism study)
- cellulose and pectinase hydrolysis for 73 hours at pH 4.5 at 37 ºC (confined rotation).
- papain hydrolysis for 69 hours at pH 7 at 37 ºC (confined rotation).
- saponification with 2.5 M NaOH in methanol (goat and hen metabolism; temperature and duration not indicated in the study report)
- 0.5 M HCl, 1 hour reflux (hen metabolism)

Table 2 Stability of fluazifop acid (II) under various hydrolysis conditions

Label and Matrix	Hydrolysis conditions	Fluazi fop acid average recovery [a]	Com pound 3 %TRR	CF3-pyri done (X) %TRR	Code No Report No
^{14}C-fluazifop in soya bean extract	0.1 M HCl, 1 hour reflux	93%	na	na	PP9/0045; RJ0211B; and PP9/0202; RJ0342B
^{14}C-phenyl fluazifop acid (II) in cotton leaf extract	0.1 M HCl, 3 hours reflux	95%	3%	nr	PP9/0203; RJ0196B
non labelled fluazifop acid (II) in solvent	1 M HCl, 3 hours 60 ºC	94%	na	na	462775; PP009B030
^{14}C-fluazifop in soya bean extract	1 M HCl, 6 hours 60 ºC	84%	na	na	PP9/0045; RJ0211B; and PP9/0202; RJ0342B
non labelled fluazifop acid (II) in solvent	1 M HCl, 1 hour reflux	48%	na	na	462775; PP009B030
^{14}C-phenyl fluazifop acid (II) in solvent	1 M HCl, 1 hour reflux	57%	34%	nr	PP9/0203; RJ0196B
^{14}C-phenyl fluazifop acid (II) in solvent	1 M HCl, 1 hour reflux	54% [b]	41% [b]	na	PP9/0200; RJ0171B
^{14}C-phenyl fluazifop acid (II) in soya bean extract	1 M HCl, 1 hour reflux	55% [b]	36% [b]	na	PP9/0200; RJ0171B
^{14}C-phenyl fluazifop acid (II) in solvent	1 M HCl, 3 hours reflux	32%	59%	nr	PP9/0203; RJ0196B
^{14}C-phenyl fluazifop^{14} acid (II) in solvent	1 M HCl, 3 hours reflux	28% [b]	60% [b]	na	PP9/0200; RJ0171B
^{14}C-phenyl fluazifop acid (II) in soya bean extract	1 M HCl, 3 hours reflux	13% [b]	77% [b]	na	PP9/0200; RJ0171B
^{14}C-**pyridyl** fluazifop acid (II) in solvent	1 M HCl, 1 hour reflux	50%	nr	46%	PP9/0203; RJ0196B
^{14}C-**pyridyl** fluazifop acid (II) in solvent	1 M HCl, 3 hours reflux	12%	nr	84%	PP9/0203; RJ0196B
^{14}C-phenyl fluazifop acid (II) in solvent	2 M HCl, 1 hour reflux	47% [b]	47% [b]	na	PP9/0200; RJ0171B
non labelled fluazifop acid (II) in solvent	6 M HCl, 3 hours 60 ºC	92%	na	na	462775; PP009B030
^{14}C-fluazifop acid (II) in soya bean extract	6 M HCl, 6 hours 60 ºC	90%	na	na	PP9/0045; RJ0211B; and PP9/0202; RJ0342B
^{14}C-phenyl fluazifop acid (II) in solvent	6 M HCl, 1 hour reflux	63%	26%	nr	PP9/0203; RJ0196B
^{14}C-phenyl fluazifop acid (II) in solvent	6 M HCl, 3 hours reflux	27%	60%	nr	PP9/0203; RJ0196B
^{14}C-phenyl fluazifop acid (II) in cotton seed extract	6 M HCl, 3 hours reflux	42%	54%	nr	PP9/0203; RJ0196B

Label and Matrix	Hydrolysis conditions	Fluazi fop acid average recovery [a]	Com pound 3 %TRR	CF3-pyri done (X) %TRR	Code No Report No
^{14}C-fluazifop acid (II) in soya bean extract	6 M HCl, 3 hours reflux	27%	na	na	PP9/0045; RJ0211B; and PP9/0202; RJ0342B
^{14}C-phenyl fluazifop acid (II) in solvent	0.1 M NaOH, 1 hour reflux	93%	4%	nr	PP9/0203; RJ0196B
^{14}C-phenyl fluazifop acid (II) in solvent	0.1 M NaOH, 1 hour reflux	85% [b]	3% [b]	na	PP9/0200; RJ0171B
^{14}C-phenyl fluazifop acid (II) in solvent	0.1 M NaOH, 3 hours reflux	92%	3%	nr	PP9/0203; RJ0196B
^{14}C-phenyl fluazifop acid (II) in cotton leaf extract	0.1 M NaOH, 3 hours reflux	96%	2%	nr	PP9/0203; RJ0196B
^{14}C-phenyl fluazifop acid (II) in solvent	0.5 M NaOH in methanol, 1 hour reflux	88%	ND	na	PP9/0045; RJ0211B and PP9/0201; RJ0213B
^{14}C-phenyl fluazifop acid (II) in solvent	1 M NaOH, 1 hours reflux	81%	15%	nr	PP9/0203; RJ0196B and PP9/0200; RJ0171B
^{14}C-phenyl fluazifop acid (II) in soya bean extract	1 M NaOH, 1 hour reflux	72% [b]	15% [b]	na	PP9/0200; RJ0171B
^{14}C-phenyl fluazifop acid (II) in solvent	1 M NaOH, 2 hours reflux	65%	30%	nr	PP9/0203; RJ0196B and PP9/0200; RJ0171B
^{14}C-phenyl fluazifop acid (II) in soya bean extract	1 M NaOH, 2 hours reflux	52% [b]	42% [b]	na	PP9/0200; RJ0171B
^{14}C-phenyl fluazifop acid (II) in cotton seed extract	1 M NaOH, 2 hours reflux	65%	31%	nr	PP9/0203; RJ0196B

NR = not relevant (cannot be derived from this compound)

na = not analysed; ND = not detected

[a] recovery of fluazifop acid (II) calculated from %TRR fluazifop-butyl + -%TRR fluazifop acid (II)) in hydrolysate

[b] recovery of fluazifop acid (II) or despyridinyl acid (III) in percentage of ether soluble radioactivity. Based on the results from report [RJ0196B], it may be assumed that all radioactivity partitions into the diethyl ether fraction after acidification of the hydrolysate

Table 3 Stability of CF3-pyridone (X) under various hydrolysis conditions

Label and Matrix	Hydrolysis conditions	CF3-pyridone average recovery	Code No Report No
^{14}C-CF3-pyridone (X) in solvent	6 M HCl, 1 hour reflux	**93%**	PP9/0045; RJ0211B
^{14}C-CF3-pyridone (X) in cotton leaf extract	6 M HCl, 1 hour reflux	**93%**	PP9/0203; RJ0196B
non labelled CF3-pyridone (X) in solvent	0.1 M NaOH, 1 hour reflux	0%	PP9/0203; RJ0196B

Studies 8-9

Two studies were submitted to investigate R/S conversion of total fluazifop under hydrolysis conditions.

[Evans and Cavell, 1984, PP9/0043, report RJ0353B] used a sample from the metabolism study on lettuce treated with the ^{14}C labelled R- or S-enantiomer of fluazifop-butyl. They showed that

a fraction containing 54% TRR of the R-enantiomer (consisting of 50.6% TRR parent and 3.4% TRR unknowns) or a fraction containing 53.3% TRR of the S-enantiomer (consisting of 49.0% TRR parent, 0.2% TRR fluazifop acid (II), 0.6% TRR Pyr-Ph ether (IV) and 3.5% TRR unknowns) converted to 49.9% and 49.0% TRR fluazifop acid (II) using 1.0 M NaOH in 2-propanol for 4 hours at 21 ºC.

[Evans and Cavell, 1984, PP9/0048, report RJ0356B] used a sample of cotton forage from the metabolism study on cotton treated with ^{14}C labelled R- or S-enantiomer of fluazifop-butyl. They showed that a fraction containing 26% TRR of the R-enantiomer (consisting of 23.9% TRR parent, 0.7% TRR fluazifop acid (II), 0.8% TRR Pyr-Ph ether (IV) and 1.1% TRR unknowns) or a fraction containing 28.6% TRR of the S-enantiomer (consisting of 23.2% TRR parent, 0.9% TRR fluazifop acid (II), 1.6% TRR Pyr-Ph ether (IV) and 2.9% TRR unknowns) converted to 23.5% and 24.6% TRR fluazifop acid (II) using 1.0 M NaOH in 2-propanol for 4 hours at 21 ºC.

Reviewer's conclusion:

No significant conversion of R- to S- or S- to R- enantiomers occurred during hydrolysis.

Fruits and fruiting vegetables

Metabolism study 1 (grapes)

In a non-GLP study, the metabolism of a mixture of ^{14}C-phenyl-and ^{14}C-pyridyl-fluazifop-P-butyl (R enantiomer) was studied on field grown grapes sprayed at the base of the vine [French and Leahey, 1987, PP5/0082, report RJ0569B]. The grape vine (variety French Colombard) was sprayed at an area of 1 m^2 around the base with a 50:50 mixture of EC formulated phenyl- and pyridyl-labelled fluazifop-P-butyl (R-enantiomer) in Visalia, CA, USA, in 1986. The application was intended to consist of two treatments, each at a targeted rate of 0.84 kg ai/ha. After the first application (5 June) at early bunch formation, analysis of the spray mix showed that in the first application approximately 20% less active ingredient had been applied. This shortfall was made up on day 42 (17 July) with a supplementary application. The final (third) application was made 71 days after the first application (growth stage not stated, 15 August). Analysis showed that for the third treatment 91% of the required rate of 0.84 kg ai/ha had been achieved. A total of (0.670+0.163+0.765=) 1.6 kg ai/ha had been applied, approximating 2 × 0.84 kg ai/ha. Immature grapes were harvested from the vine at days 21, 30, 45 and 60 after the first application and mature grapes were harvested on days 85 and 101 after the first treatment (14 and 30 days after the final treatment). Grape samples of 500 g were taken for analysis. Samples frozen immediately after harvest were stored at -15±5 °C for a maximum of 8 months.

Grapes were de-stalked and extracted by maceration with acetonitrile. Total radioactivity in the extracts and remaining solids was determined by (combustion) LSC. The total radioactivity residue (TRR) was then calculated as the sum of these results and expressed as fluazifop-butyl equivalents.

Only very low levels of radioactive residues were found in immature grapes harvested between days 21 and 60 after the first application (0.004–0.005 mg/kg eq) and in mature grapes harvested on days 14 and 30 after the last application (0.008 and 0.009 mg/kg eq, respectively). Composition of the residue was not further investigated, since residues were below the trigger value of 0.01 mg/kg eq.

Reviewer's conclusion: This study indicates that no residues (< 0.01 mg/kg; DAT 14–30) are to be expected in the fruits after application of fluazifop-butyl at the base of the vines.

Metabolism study 2 (cucumbers)

In a non-GLP study, the metabolism of ^{14}C-phenyl or ^{14}C-pyridyl-fluazifop-butyl (RS) was studied in field grown cucumbers following a single foliar application [Patel *et al*., 1983, PP9/0044, report RJ0299B]. Plants (variety Burplus Greene) were grown in the field in a sandy loam soil in Bracknell, Berkshire, UK, in 1982. Two fruiting cucumber plants were treated by foliar spray either with an EC formulation of ^{14}C-phenyl- or ^{14}C-pyridyl-labelled fluazifop-butyl (RS). Plants were treated at an

actual equivalent field rate of 1 × 0.50 or 0.52 kg ai/ha, respectively for the phenyl or pyridyl labelled compound. An adjuvant was added (0.1% Agral 90). A single mature cucumber fruit was harvested at DAT 1 (phenyl-label) and DAT 14 (phenyl label, pyridyl label). The DAT 1 cucumber was treated when the cucumber was 28 cm long, while the DAT 14 cucumbers were treated when the cucumbers were 5–10 cm long. The cucumbers were peeled (peel thickness 3 mm) and the peel and flesh were stored separately at -20 ºC. The storage period is not stated, but does not exceed 6 months (start and end of experiments).

Samples from flesh and peel were extracted separately with acetonitrile/water (1:1, v/v). Extracts and remaining solids were analysed by (combustion) LSC. From both flesh and peel, >97% of the radioactivity could be extracted. High residues in both peel and flesh were found as the plants were treated with fluazifop-butyl when fruits were present. Results are shown in Table 4.

Acetonitrile/water extracts were partitioned with hexane. The hexane fraction was analysed by TLC. The acetonitrile was evaporated from the aqueous phase and the aqueous phase was acidified to pH2 (0.02 M HCl). The acidic aqueous phase was partititioned with diethyl ether. Peel fractions of DAT 14 were characterised further. The diethyl ether phase of DAT 14 peels was evaporated to dryness and redissolved in 6 M HCl, while the aqueous phase of DAT 14 peels was acidified with 6 M HCl. Both fractions were heated for 2 hours at 80 ºC in 6 M HCl and then partitioned with diethyl ether. All organic phases were analysed by TLC using reference standards for fluazifop-butyl (I), fluazifop (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X) and compound 9.

Results are shown in Table 5. After treatment with the phenyl label, the radioactive residue in the peel on day 1 was characterised as parent compound fluazifop-butyl (30% TRR) and as fluazifop acid (II; 43% TRR). In the flesh, 77% of the radioactive residue was fluazifop acid (II). On day 14 after application of the phenyl- and pyridyl-label no parent compound was found in peel nor in flesh and the main metabolite was fluazifop (II) as free acid or as conjugate (total 46–76% TRR).

The principal component of the residue in the whole fruit was fluazifop acid (II) in free or conjugated form (69-72% TRR). Despyridinyl acid (III) was released after hydrolysis at 3.3% TRR (DAT 14). CF3-pyridone (X) and Pyr-Ph ether (IV) were not detected. The presence of fluazifop alcohol (XXXIV) or hydroxyfluazifop acid (XL) was not verified. Unidentified organosoluble fractions represented 16-17% (phenyl) or 24% (pyridyl) in the whole fruit. Individual compounds were < 7% TRR, except for one compound which represented 20% TRR (free and conjugated, DAT 14, pyridyl) in the peel. Water fractions contained a maximum of 3.9-6.4% TRR in the whole fruit.

Reviewer's conclusion: Fluazifop acid (II) is stable when hydrolysed in 6 M HCl for 2 hours at 80 ºC (see analytical method section). The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. The CF3-pyridone (X) counterpart and Pyr-Ph ether (IV) were not detected. The presence of fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified, but they may be present in the organic fractions. Hexane fractions that were not analysed may contain some low levels of fluazifop-butyl (maximum 0.7–5.4% TRR). Fractions that were not subjected to hydrolysis (hexane, diethyl ether, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 11–25% TRR in whole fruit.

Table 4 Radioactive residues in cucumbers expressed as fluazifop-butyl equivalents

Treatment	harvest time (days)	sample	fresh weight (g)	TRR (mg/kg eq)	Proportion of the residue
phenyl label fluazifop-butyl (RS)	1	flesh peel RAC [a]	231.0 73.2 304.2	0.19 4.62 1.26	11% 88% 100%
phenyl label fluazifop-butyl (RS)	14	flesh peel RAC [a]	89.7 46.4 136.1	4.93 4.90 4.92	66% 34% 100%
pyridyl label fluazifop-butyl (RS)	14	flesh peel RAC [a]	224.9 91.0 315.9	2.52 2.07 2.39	75% 25% 100%

[a] Calculated by the present reviewer based on mass fractions.

Table 5 Radioactive residues in cucumbers expressed as fluazifop-butyl equivalents.

Treatment	Phen (RS) DAT1 peel %TRR	Phen (RS) DAT1 flesh %TRR	Phen (RS) DAT1 RAC %TRR [d]	Phen (RS) DAT 14 peel %TRR	Phen (RS) DAT 14 flesh %TRR	Phen (RS) DAT14 RAC %TRR [d]	Pyr (RS) DAT14 peel %TRR	Pyr (RS) DAT14 flesh %TRR	Pyr (RS) DAT14 RAC %TRR [d]
Dose rate (kg ai/ha)	0.50	0.50	0.50	0.50	0.50	0.50	0.52	0.52	0.52
TRR (mg/kg eq)	4.62	0.19	1.26	4.90	4.93	4.92	2.07	2.52	2.39
Parent	30.5	ND	7.3	ND	ND	ND	ND	ND	ND
Fluazifop (II, free + conj) - fluazifop acid (free) - fluazifop org sol conj - fluazifop polar conj	43.2 - 43.2 - -	76.8 - 76.8 - -	68.7 - 68.7 - -	64.0 - 44.8 - 16.8 - 2.4	75.5 - 75.5 - -	71.6 - 65.0 - 5.7 -0.8	45.8 - 43.3 - - 2.5	77.8 - 76.2 - - 1.6	68.6 - 66.7 - - 1.9
Pyr-Ph ether (IV)	ND	ND	ND	ND	ND	ND	ND	ND	ND
despyridinyl acid (III) - polar conj	ND	ND	ND	9.6 - 9.6	ND -	3.3 - 3.3	NR	NR	NR
CF3-pyridone (X)	NR	NR	NR	NR	NR	NR	ND	ND	ND
Fluazifop alcohol (XXXIV)	no std	no std	no std	no std	no std	no std	no std	no std	no std
Hydroxy fluazifop acid (XL)	no std	no std	no std	no std	no std	no std	no std	no std	no std
unknown A (hydrolysed) - A organo sol conj - A polar conjugate	ND	ND	ND	2.8 - - 2.8	-	0.95 - - 0.95	20.4 - 15.4 - 5.0	- - 2.0	5.9 - 4.4 - 2.9
unknown B (hydrolysed) - polar conj	ND	ND	ND	ND	ND	ND	4.9 -4.9	3.4 -3.4	3.8 -3.8
unknowns in hexane - not hydrolysed [a]	5.4	5.4 na	5.4	0.6 na	1.1 na	0.93	0.7 na	0.7 na	0.7 na
unknowns in diethylether - not hydrolysed [a] - not hydrolysed [a] - after hydrolysis	16.9 - 13.9 - 3.0 -	10.5 - 6.1 - 4.4 -	12.0 - 8.0 - 4.1 -	17.1 - - -13.5 [or] - 3.6 [c]	13.3 - 13.3	14.6 - 8.8 - -4.6 -1.2	18.0 - - - 14.7 [or] - 3.3 [c]	12.2 - 4.4 - 7.0 - 0.8 [c]	13.9 - 3.1 - 5.0 - 4.8 - 0.95
unknowns in aq fraction - not hydrolysed [a] - after hydrolysis	2.7 - 2.7 - -	5.4 - 5.4 - -	4.8 - 4.8 -	2.5 - - 0.8 [b] - 1.7 [aq]	8.5 - 8.5 - -	6.4 - 5.6 - 0.3 - 0.6	7.7 - 1.8 [b] 5.9 [aq]	2.3 - - -2.3 [aq]	- 3.9 - - 0.52 - 3.3
PES; not hydrolysed [a]	0.8	1.9	1.6	3.4	1.6	2.2	2.5	1.6	1.9
Total	99.5	100	99.9	100	100	100	100	100	100
- Total identified	73.7	76.8	76.0	73.6	75.5	74.8	45.8	77.8	68.6
- Total not hydrolysed [a]	25.0	23.2	23.6	4.0	23.4	16.8	3.2	13.7	10.7
- Total in aq fractions	2.7	5.4	4.8	2.5	8.5	6.4	7.7	2.2	3.8

ND not detected (reference standard available)

NR not relevant, since despyridinyl acid (III) and CF3-pyridone (X) don't contain the ^{14}C label for the specified case

[a] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X) depending on the label

[b] Partitioned into the aqueous fraction after treatment of the organic phase with 6 M HCl

[c] Partitioned into the organic fraction after treatment of the aqueous phase with 6 M HCl

[or] Stayed in the organic phase after treatment of the organic phase with 6 M HCl

[aq] Stayed in the aqueous phase after treatment of the aqueous phase with 6 M HCl

na not analysed; the hexane fraction may contain fluazifop-butyl

[d] Calculated by the current reviewer, using the mass fractions indicated in the table above

Stem and leafy vegetables

Metabolism study 3 (lettuce)

In a non-GLP study, the metabolism of ^{14}C-phenyl-fluazifop-butyl as R- or S-enantiomer was studied in indoor pot grown lettuce following a topical foliar and stem application [Evans and Cavell, 1984, PP9/0043, report RJ0353B]. Six 2-week old lettuce seedlings (variety Webb) were grown in two pots filled with sandy loam (2 plants per pot) under glasshouse conditions. The plants were treated either with EC formulated ^{14}C-phenyl-labelled- fluazifop-P-butyl (enantiomer ratio R:S = 97.5%:2.5%) or EC formulated ^{14}C-phenyl-labelled-S-fluazifop-butyl (enantiomer ratio R:S = 2.6%:97.4%). The formulations were applied in aqueous 0.01% Agral 90 solution by syringe to foliage and stems of each of the two plants per pot. The amounts applied were calculated to represent an actual application rate of 0.45 kg ai/ha. Plants were harvested 27 days after application (growth stage not stated). Samples were stored at -20 °C until analysis for a maximum of 9 months.

Chopped lettuce samples were extracted subsequently with acetonitrile: water (80:20, v/v) (93% and 96% TRR, R;S) and water (1.2% and 1.2% TRR), until 2.8% and 6.1% TRR (R;S) remained as solids. Extracts and remaining solids were analysed by (combustion) LSC. At harvest 70% and 65% of the applied doses of respectively the R and S enantiomer of fluazifop-butyl were recovered in the lettuce plants. Results in terms of mg/kg eq are not reported.

The acetonitrile/water extract (93% and 96% TRR for R and S enantiomer) was partitioned twice with hexane (A: 41%; 49% TRR) and then between diethyl ether (C: 26%; 19% TRR) and water (B: 29%; 24% TRR). The diethyl ether extract C (26%; 19% TRR) was fractionated on a Bond Elut Column into a polar fraction (3C: 2.3%; 2.5% TRR) and an apolar fraction (1C+2C: 24%; 16% TRR).

Water phase B (29%; 24% TRR) was mixed with the Bond Elut polar fraction 3C (2.3%; 2.5% TRR). These polar metabolites were subjected to acidic hydrolysis (2*hour*6 M HCl at 60 °C) and partitioned with diethyl ether (G 25%; 20% TRR).

The Bond Elut apolar fraction 1C+2C (24%; 16% TRR) was partitioned with 0.05 M sodium bicarbonate solution to get an aqueous phase (10%; 12% TRR) and a diethyl ether phase (D: 13%; 4.0% TRR). The aqueous phase (10%; 12% TRR) was acidified (pH <1) and then partitioned with diethyl ether (E 9.4%; 12% TRR).

The diethylether phase D (13%; 4.0% TRR) was mixed with hexane extract A (41%; 49% TRR). These organo soluble compounds were subjected to alcoholic caustic hydrolysis (1.0 M NaOH in 2-propanol, 4 hours, 21 °C), neutralised by addition of 0.1 M HCl and partitioned with diethyl ether. The remaining aqueous phase was acidified (pH < 1) and then again partitioned with diethyl ether (F: 52%; 52% TRR).

The ratio of fluazifop enantiomers was determined by chiral HPLC (using Altex ultrasphere IP column). Organic fractions were analysed by TLC with 3 solvent systems. Reference standards used were fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV) and fluazifop alcohol (XXXIV). Fractions containing fluazifop acid (II, free or released from fluazifop-butyl or fluazifop conjugates) were also analysed by chiral HPLC.

The (R)/(S) ratio remained unchanged in fluazifop acid (II) during alkaline and acid hydrolysis, indicating that no epimerisation occurred in the plant or during sample extraction and hydrolysis. The nature of the residues and enantiomeric ratios of the individual components for lettuce plants treated with phenyl-labelled fluazifop-butyl (R and S-enantiomers) are summarized in Table 6 and Table 7. Results in terms of mg/kg eq are not reported.

Approximately 50% TRR remained intact as unmetabolized parent compound in/on the lettuce leaves. Free fluazifop acid (II) and its conjugates were formed as the primary metabolites by both the enantiomers (19% TRR after hydrolysis). Pyr-Ph ether (IV, free and conjugates) was detected in both the enantiomers at 0.4–1.7% TRR. Despyridinyl acid (III) was released after hydrolysis at 4.1–8.7% TRR. Fluazifop alcohol (XXXIV, 5.3% TRR) was formed only from the (S)-enantiomer (i.e. fluazifop alcohol (XXXIV) is not a plant metabolite of fluazifop-butyl in lettuce). Unidentified

organosoluble fractions represented 5.2-6.8%TRR. Water fractions were not analysed (maximum 9.6–11% TRR).

Reviewer's conclusion: Fluazifop acid (II) is stable in 6 M HCl for 2 hours at 60 ºC and 1 M NaOH in 2-propanol during 4 hours at 21 ºC (see hydrolytic stability section above). The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. CF3-pyridone (X) is not detected, because only phenyl labelled compounds were used in this study. The presence of hydroxyfluazifop acid (XL) was not verified but might be present in the organic fractions. Fractions that were not subjected to hydrolysis (diethyl ether, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III), fluazifop alcohol (XXXIV) or hydroxyfluazifop acid (XL) at a maximum of 5.2–7.4% TRR.

Table 6 Enantiomer ratio in selected fractions of lettuce leaf extracts

Fraction	^{14}C-phenyl R-enantiomer (97.5%) 1 × 0.45 kg ai/ha DAT 27		^{14}C-phenyl S enantiomer (97.4%) DAT 27 %TRR	
	% R-enantiomer	%S enantiomer	% R-enantiomer	%S enantiomer
2F = fluazifop-butyl	97.6	2.4	0.9	99.1
2E = free fluazifop acid (II)	95.4	4.6	0.9	99.1
2H = fluazifop acid (II) released from conjugates	97.1	2.9	1.4	98.6

Table 7 Nature of residues in lettuce leaves

Enantiomers	^{14}C-phenyl R-enantiomer DAT 27 %TRR	^{14}C-phenyl S enantiomer DAT 27 %TRR
Dose rate (kg ai/ha)	0.45	0.45
TRR (mg/kg eq)	not determined	not determined
Parent	51.6 (38.4+12.2+1.0)	49.0 (46.8+2.2+0.0)
Fluazifop acid (II, free + conj) - Fluazifop acid (free) - Fluazifop organo soluble conj - Fluazifop polar conjugates	19.1 - 8.2 (0.1+0.0+8.1) - - - 10.9 (11.0 minus 0.1)	19.0 - 12.8 (0.2+0.2+12.4) - - - 6.2 (6.4 minus 0.2)
Pyr-Ph ether (IV, free + conj) - Pyr-Ph ether (free) - Pyr-Ph ether (polar conj)	0.4 - 0.0 - 0.4	1.7 - 0.6 - 1.1
Despyridinyl acid (III, polar conj)	8.7	4.1
CF3-pyridone (X)	NR	NR
Fluazifop alcohol (XXXIV) polar conj	ND	5.3
Hydroxyfluazifop acid (XL)	no ref std	no ref std
unknowns in diethylether fraction - not hydrolysed [a] - after hydrolysis	7.5 - 0.3 - 2.5 [or] - 4.7 [c]	6.0 - 0.0 - 2.5 [or] - 3.5 [c]
unknowns in aqueous fraction - not hydrolysed [a] - after hydrolysis	10.6 - 2.1 (1.2+0.9) - 1.6 [b] - 6.9 [aq]	9.6 - 1.3 (1.2+0.1) - 1.8 [b] - 6.5 [aq]
PES; not hydrolysed [a]	2.8	6.1
Total	100.7	100.8
- Total identified	79.8	79.1
- Total not hydrolysed [a]	5.2	7.4

- or ND = not detected;

NR not relevant, since CF3-pyridone (X) doesn't contain the phenyl group

[a] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)

[b] Partitioned into the aqueous fraction after treatment of the organic phase with 1M NaOH in 2-propanol

[c] Partitioned into the organic fraction after treatment of the aqueous phase with 6 M HCl

[or] Stayed in the organic phase after treatment of the organic phase with 1M NaOH in 2-propanol

[aq] Stayed in the aqueous phase after treatment of the aqueous phase with 6 M HCl

Metabolism study 4 (celery)

In a non-GLP study, the metabolism of ^{14}C-phenyl- or ^{14}C-pyridyl-fluazifop-P-butyl (R-enantiomer) was studied in field grown celery following two broadcast applications [French *et al*, 1987, PP5/0081, report RJ0590B]. Two plots with 5 celery plants (74 days old) each were treated twice with either EC formulated ^{14}C-phenyl-labelled or ^{14}C-pyridyl-labelled fluazifop-P-butyl (R-enantiomer) as an even spray over both plots. The treatment was carried out in Belle Glade, FL, USA in 1986. The first treatment (at 8-9-leave stage, 20–23 cm tall; on 20 October) was performed at an actual dose rate of 0.45 or 0.42 kg ai/ha for the phenyl or pyridyl label, respectively. A second treatment was performed 15 days later (9–11 leave stage, 33–38 cm tall, on 4 November) at an actual dose rate of 0.18 and 0.36 kg ai/ha, respectively. Mature plants were harvested 30 days after the last application (4 December). Samples were brushed to remove adhering soil, and leaves and stems were separated. Samples were stored at -20 ± 5 °C until analysis for a maximum of 6 months.

A single celery plant from each of the plots was taken for analysis. Stem and leaves were extracted separately by maceration first with acetonitrile and then with acetonitrile: water (1:1). The radioactivity in the extracts and solids was determined by (combustion) LSC. The TRR was determined by the sum of these values. A total of 81% and 87% TRR could be extracted from the phenyl labelled celery stems and leaves, respectively.

The combined acetonitrile and acetonitrile/water extracts (81% TRR for the phenyl label) from the celery stems were partitioned with hexane (0.7% TRR). The remaining aqueous phase was acidified to pH 2 (0.02 M HCl) and partitioned with diethyl ether. The diethyl ether fraction (A: 34% TRR) was evaporated to dryness and subjected to hydrolysis with 6M HCl for 6 hours at 80 °C. The resulting hydrolysate was partitioned with diethyl ether (B: 34% TRR). The aqueous fraction (43% TRR) was subjected to hydrolysis in 2M in HCl for 2 hours at 60 °C. The resulting hydrolysate was partitioned with diethyl ether (C: 33% TRR).

Similar procedures were followed for the phenyl labelled celery leaf extracts and the pyridyl label stem and leaf extracts, except that all hydrolysis steps were performed with 2 M HCl for 2 hours at 60 ºC and/or 6 M HCl for 2 hours at 80 ºC.

The solid residue from the celery stems (phenyl label only) was subjected to hydrolysis with 2M HCl for 2 hours at 60 °C plus 6 M HCl for 2 hours at 80 ºC. The acid liquid phase (13% TRR for the phenyl label) was partitioned with diethyl ether (D: 10% TRR). Similar procedures were followed for the solids from celery leaves. Solids from pyridinyl labels were not investigated.

All diethyl ether fractions and the water soluble fraction from pyridyl labelled celery leaves were analysed by TLC and HPLC-UV against reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), CF3-pyridone (X), compound 28, 34 and 40. The nature of radioactive residues in leaves and stems are summarized in Table 8.

In celery leaves, 52–63% the total radioactive residue (TRR) was identified as the free fluazifop acid (II). Parent was detected at trace levels (< 3% TRR). CF3-pyridone (X, free) and compound 28 (free) were detected in leaves at 8.1% and 5.6% TRR, respectively. The study author postulates that compound 28 is a product derived from a glutathione conjugate of CF3-pyridone (X). Despyridinyl acid (III) was released after hydrolysis at 7.1% TRR. Hydroxyfluazifop acid (XL) was present at 0.7–1.6% TRR. Unidentified organosoluble fractions represented 27%TRR (phenyl) and 3.8% (pyridyl). Individual compounds in the analysed fractions were below 5% TRR. Water fractions were not analysed: maximum 5.6% TRR (phenyl) or 20.3% TRR (pyridyl).

In celery stems, 40–43% TRR was identified as fluazifop acid (II, free and conjugates) and 18% TRR as despyridinyl acid (III) conjugates. Parent was not detected. CF3-pyridone (X, free and conjugates) and hydroxyfluazifop acid (XL) were detected in stems at 2.7% and 1.2% TRR,

respectively. Despyridinyl acid (III) was released after hydrolysis at 18% TRR. Hydroxyfluazifop acid (XL) was present at 1.2–4.4% TRR. Unidentified organosoluble fractions represented 11% TRR (phenyl) and 8.7% (pyridyl). Individual compounds were < 5% TRR. Water fractions were not analysed: maximum 12% TRR (phenyl) and 35% (pyridyl).

Reviewer's conclusion: Fluazifop acid (II) is stable in 6 M HCl for 3 hours at 80 ºC (see analytical method section). It is therefore assumed that fluazifop acid (II) is also stable in 2 M HCl for 2 hours at 60 ºC plus 6 M HCl for 2 hours at 80 ºC. Since only 2.0% TRR is released as despyridinyl acid (III) during 6 hours in 6 M HCl at 80 ºC, it can be concluded that fluazifop acid (II) is also stable under those conditions. The presence of Pyr-Ph ether (IV) was not verified, but may be present in the organic fractions. The presence of conjugated despyridinyl acid (III) in celery leaves and stems (7.1% and 18% TRR) indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant.

The presence of free and conjugated CF3-pyridone (X) in leaves and stems (8.1% and 2.7% TRR) could represent uptake from soil and/or metabolism within the plant. Since total CF3-pyridone (X) and total despiridinyl acid are found at similar amounts in the leaves (7.1% III versus 8.1% X) this suggests that both compounds were derived from metabolism within the plant, rather than uptake from soil.

In celery leaves from the pyridyl label both the diethyl ether fractions and the acidified (0.02 M HCl) water phase was analysed (before hydrolysis) and it was shown that free CF3-pyridone (X) distributes over the diethyl ether and pH 2 acidified water phases (2.5% versus 5.1% TRR). However, since all water phases were hydrolysed with 2–6 M HCl, this will have no impact on the amount of total CF3-pyridone (X). Fractions that were not subjected to hydrolysis (diethyl ether, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 0.7–8.4% TRR.

Table 8 Nature of residues in celery leaves and stems

	Leaves	Leaves	Stems	Stems
	^{14}C-Phenyl Label R-enantiomer 0.45+0.18 kg ai/ha DALT = 30 0.31 mg/kg eq	^{14}C-Pyridyl Label R-enantiomer 0.42+0.36 kg ai/ha DALT = 30 0.64 mg/kg eq	^{14}C-Phenyl Label R-enantiomer 0.45+0.18 kg ai/ha DALT = 30 0.05 mg/kg eq	^{14}C-Pyridyl Label R-enantiomer 0.42+0.36 kg ai/ha DALT = 30 0.08 mg/kg eq
	%TRR	%TRR	%TRR	%TRR
Parent	2.4	ND	ND	ND
Fluazifop acid (II, free + conj) – fluazifop acid (free) - fluazifop organo soluble conj - fluazifop polar conj - fluazifop released from solids	51.6 - 4.1 - 36.4 - 6.9 - 4.2	62.7 -2.6 -55.7 -4.4 NA	43.2 - 11.2 - 16.3 - 7.5 - 8.2	39.4 -8.9 - 26.2 - 4.3 NA
Pyr-Ph ether (IV)	no ref std	no ref std	no ref std	no ref std
Despyridinyl acid (III, conj) - III, organosoluble conj - III, polar conjugates - III, released from solids	7.1 - 1.1 - 5.9 - 0.1	NR	18.5 - 2.0 - 16.2 - 0.3	NR
CF3-pyridone (X, free + conj) - CF3-pyridone (X) free (org+water) - CF3-pyridone (X) organo sol - CF3-pyridone (X) polar conj - compound 28 (free in water)	NR	13.7 -2.5+5.1 -0.5 - -5.6	NR	2.7 -1.6+NA -0.3 - 0.8 - NA
Total fluazifop alcohol (XXXIV) - free - polar conjugates	0.3 - 0.3	ND	1.0 - 1.0 -	ND
Total hydroxyfluazifop acid (XL) - free - organosoluble conjugates - polar conjugates	1.6 - - 1.6	0.7 - - -0.7	4.4 - 0.3 - - 4.1	1.2 - 1.0 0.2
unknowns in organic fractions	26.6	3.8	12.1	8.7

	Leaves	Leaves	Stems	Stems
	^{14}C-Phenyl Label R-enantiomer 0.45+0.18 kg ai/ha DALT = 30 0.31 mg/kg eq	^{14}C-Pyridyl Label R-enantiomer 0.42+0.36 kg ai/ha DALT = 30 0.64 mg/kg eq	^{14}C-Phenyl Label R-enantiomer 0.45+0.18 kg ai/ha DALT = 30 0.05 mg/kg eq	^{14}C-Pyridyl Label R-enantiomer 0.42+0.36 kg ai/ha DALT = 30 0.08 mg/kg eq
	%TRR	%TRR	%TRR	%TRR
- not analysed, not hydrolysed [a] - after hydrolysis	- 18.5 [or] [d] 2.8 [c] 0.6 [solids] 1.1 [solids] 3.6 [c, aq and solids]	- 3.8 [or]	- 0.7 [hexane] - 5.0 [or] - 5.1 [c] - 0.6 [solids] - 0.7 [solids]	- -5.4 [or] -3.3 [c]
unknowns in aqueous fraction - after hydrolysis	5.6 - 1.6 [solids] - 4.0 [c, aq and solids]	20.3 -7.1 -13.2	12.2 - 0.8 [b] - 8.8 [aq] - 1.0 [solids] - 1.6 [solids]	34.6 - 2.8 [b] - 31.8 [aq]
PES - not hydrolysed [a] - after hydrolysis	 - 4.8	 5.6 -	 - 6.3	 8.4 -
Total	100	106.8	97.7	95
- Total identified	63.0	77.1	67.1	43.3
- Total – not hydrolysed [a]	-	5.6	0.7	8.4

TRR = total radioactive residue expressed as fluazifop-butyl equivalents.

ND = not detected.

NR = not relevant for this label, since the compound does not contain the phenyl or pyridyl group

[a] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridon (X)

[b] Partitioned into the aqueous fraction after treatment of the organic phase with 2 or 6 M HCl

[c] Partitioned into the organic fraction after treatment of the aqueous phase with 2 or 6 M HCl

[d] Contains 5.8% TRR (at least 2 compounds), 9.2% TRR polar material, 3.6% TRR background radioactivity

[or] Stayed in the organic phase after treatment of the organic phase with 2 or 6 M HCl

[aq] Stayed in the aqueous phase after treatment of the aqueous phase with 2 or 6 M HCl

[solids] released from solids after hydrolysis with 2 or 6 M HCl.

[hexane] hexane fraction (not analysed) may contain fluazifop-butyl

[e] Study author postulates that compound 28 is derived from a glutathione conjugate of CF3-pyridone (X)

Metabolism study 5 (endive)

In a GLP study, the metabolism of ^{14}C-phenyl- or ^{14}C-pyridyl-fluazifop-P-butyl (R-enantiomer) was studied in field grown endive following two broadcast applications [Quistad, 2008, PP005_50034, report 1690W]. Endive (variety Green Curled Ruffec) was planted in four plots (1× and 4× rate, 2 radiolabels) with a loamy sand, located in Madera, CA, USA in 2007. Each plot was treated twice with either EC formulated ^{14}C-phenyl-labelled or ^{14}C-pyridyl-labelled fluazifop-P-butyl by spraying uniformly over the top of foliage and soil. The first treatment was on 13 September (24 days after planting) and the second treatment was 21 days later (4 October). The target rate was two times 0.42 kg ai/ha (1× rate) or two times 1.7 kg ai/ha (4× rate) for each radiolabel. The actual application rates were 96–100% of targets. Half of the endive plants were harvested 20 days after the first application (immature, BBCH 43, 3 October) and the remaining plants were harvested 28 days after the second application (mature, 1 November). Samples were stored frozen for 1–64 days until primary extraction and analysis.

Only the 1× rate samples were analysed further. Homogenised endive was combusted to determine total radioactive residues (TRR). Endive was extracted by maceration twice with acetonitrile and then twice with acetonitrile: water (1:1). Further extractions used 30 min shaking in acetonitrile:0.5 M HCl, 30 min shaking in 6 M HCl to extract cellulose and overnight shaking in 24% KOH (4.3 M) to extract hemicellulose. The combined HCl and KOH extracts were partitioned

between ethylacetate and water. The radioactivity in the extracts and solids was determined by (combustion) LSC. Results are summarized in Table 9.

The combined acetonitrile and acetonitrile/water extracts and the ethyl acetate extracts were analysed by HPLC-UV using reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X) and compound 9. These assignments were investigated further by TLC analysis and peaks isolated by HPLC from the 4× rate samples. HPLC-UV analysis allowed assignment of structures to fluazifop acid (II), CF3-pyridone (X) and Pyr-Ph ether (IV), while 9 other components were purified for identification/characterisation by hydrolysis (6 M HCl overnight at room temperature), enzymatic cleavage (β-glucosidase, conditions not stated) and analysis by HPLC-MS. Results are shown in Table 10.

- E-1 could be hydrolysed with 6 M HCl or β-glucosidase to give despyridinyl acid (III).
- E-2 could not be hydrolysed by 6 M HCl. HPLC-MS analysis gave MH+ ions at 437.5, 481.5 or 525.5 that did not match with any proposed metabolite.
- E-3 was not characterised further
- E-4 could be hydrolysed with 6 M HCl to give 60% despiridinyl acid (III) in mature endive or 52% Pyr-Ph ether (IV) + 28% despyridinyl acid (III) in immature endive. HPLC-MS analysis gave a MH+ ion at 344.5, consistent with a hexoside conjugate of despyridinyl acid (III) in mature endive.
- E-5 could be hydrolysed with 6 M HCl to give 30% fluazifop acid (II). HPLC-MS analysis gave a MH+ ion at 490.5, consistent with a hexose conjugate of fluazifop acid (II).
- E-6 could by hydrolysed with 6 M HCl to give 91% fluazifop acid (II). HPLC-MS analysis gave a MH+ ion at 576.2, consistent with a malonylhexoside conjugate of fluazifop acid (II).
- E-7 could be hydrolysed with 6 M HCl to give 86% fluazifop acid (II). HPLC-MS analysis gave a MH+ ion at 490.5, consistent with a hexose conjugate of fluazifop acid (II).
- E-9 could not be hydrolysed by 6 M HCl. HPLC-MS gave a MH+ ion at 393.6 that is not consistent with any proposed metabolite
- E-10 and E-11 were the most polar metabolites. They could not be hydrolysed by 6 M HCl overnight at room temperature, but could be hydrolysed after 3 hours at 60 ºC, characteristic for pyridinyl N-sugar conjugates. The hydrolysis products did not match with CF3-pyridone (X) or compound 9 on TLC. E-10 did not have an UV response for analysis by HPLC and it did not produce significant ionisation in HPLC-MS with electron spray ionisation. E-11 eluted at the solvent front with a C18 column but could be retained using an NH2 column. The HPLC-MS spectrum indicates a possible parent ion with MW 540. A loss of two glucose units is observed with electron spray ionisation.

Fluazifop-butyl (I) was not detected in any endive extracts. The major metabolite is fluazifop acid (II, 36–49% TRR) in its free or conjugated form (E-5, E-6, E-7). CF3-pyridone (X, free, 11–14% TRR) was a major metabolite from the pyridyl label. Despyridinyl acid (III, 25%TRR in immature; 40% TRR in mature) was a major metabolite as a hexoside (E-1, E-4) from the phenyl label. Pyr-Ph ether (IV) was abundant (11–25% TRR) as a conjugate as a portion of fraction E-4 in immature endive. The most polar fractions (3.2–6.7% E-2, 13% E-9, 8.3% E-10 and 8.0% E-11) were derived from the pyridinyl radiolabel only but could not be identified as or hydrolysed to any known metabolite. Unidentified organosoluble fractions (including fractions E-2, E-9, E-10, E-11) represented 8.1% TRR (phenyl, immature), 13% (pyridyl, immature), 2.8–5.2% (phenyl, pyridyl, mature). Unidentified compounds in the water fractions represented 1.2–3.4% TRR. Individual unidentified compounds were <9% TRR, except fraction E-9 (MW 393) which represented 13% TRR (mature endive only, pyridyl only).

Reviewer's conclusion:

Whole extracts were analysed and fractions were isolated for identification with and without hydrolysis. Fluazifop acid (II) is stable in 6 M HCl for 6 hours at 60 ºC or 3 hours at 80 ºC (see hydrolytic stability section and analytical method section) and therefore fluazifop acid (II) is assumed to remain stable during hydrolysis overnight in 6 M HCl at room temperature. Stability during overnight extractions with 24% KOH has not been investigated, but since the KOH fractions contain less than 10% TRR this is considered to have no impact on the study results. The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. The CF3-pyridone (X) counterpart is detected at much lower levels (in its free form only). It is not clear why no CF3-pyridone (X) has been released by hydrolysis.

The presence of fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified, but they may be present in the unknown organic fractions. Fractions that were not subjected to hydrolysis (acetonitrile/water extract fraction) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 1.3–11.8% TRR.

Table 9 Extraction efficiency for ^{14}C treated endives

	Immature endive 1× 0.42 kg ai/ha, DAT 20		Mature endive 2 × 0.42 kg ai/ha, DAT 28	
	^{14}C-Phenyl Label R-enantiomer 0.65 mg/kg Fb eq	^{14}C-Pyridyl Label R-enantiomer 0.88 mg/kg Fb eq	^{14}C-Phenyl Label R-enantiomer 1.4 mg/kg Fb eq	^{14}C-Pyridyl Label R-enantiomer 1.8 mg/kg Fb eq
	%TRR	%TRR	%TRR	%TRR
Acetonitrile Acetonitrile/water	87.8%	90.5%	91.4%	91.4%
Acetonitrile:0.5M HCl (1:1); 6 M HCl; 24% KOH	8.8%	8.2%	6.4%	7.4%
Post Extraction Solids	3.4%	1.3%	2.2%	1.2%

Table 10 Nature of residues in endive leaves treated with fluazifop-butyl

	Immature endive				Mature endive			
	^{14}C-Phenyl Label R-enantiomer 1× 0.42 kg ai/ha DAT 20 0.65 mg/kg eq		^{14}C-Pyridyl Label R-enantiomer 1× 0.42 kg ai/ha DAT 20 0.88 mg/kg eq		^{14}C-Phenyl Label R-enantiomer 2 × 0.42 kg ai/ha; DAT 28 1.4 mg/kg eq		^{14}C-Pyridyl Label R-enantiomer 2 × 0.42 kg ai/ha DAT 28 1.8 mg/kg eq	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Parent	ND	-	ND	-	ND	-	ND	-
Fluazifop acid (II, free + conj) - fluazifop acid (free) [c] - E5, hexose [c] - E6, malonylhexoside [c] - E7, hexose [c]	47.6 -4.5 -14.0 -7.7 -21.4	- 0.029 0.091 0.050 0.14	36.5 -3.9 -10.6 -7.3 -14.7	- 0.035 0.093 0.064 0.12	49.4 -5.3 -14.7 -8.8 -20.6	- 0.075 0.21 0.13 0.29	43.3 -4.7 -13.8 -8.4 -16.4	- 0.084 0.25 0.15 0.29
Pyr-Ph ether (IV free+conj) - E8, Pyr-Ph ether (free) [c] - E4, conjugate	11.2 -1.7 -9.5	- 0.011 0.062	24.9 -2.1 -22.8	 0.018 0.200	0.5 -0.5 -	 0.007 -	- - [a]	- - [a]
Despyridinyl acid (III conjugates) - E1, hexose - E4, hexose	25.4 -20.3 -5.1	 0.13 0.033	NR	-	40.4 -35.2 -5.2	 0.51 0.074	NR	-
CF3-pyridone (X, free)	NR	-	13.6	0.12	NR	-	10.9	0.19
Fluazifop alcohol (XXXIV)	no std		no std		no std		no std	
Hydroxyfluazifop acid (XL)	no std		no std		no std		no std	
Unknown E2 - after hydrolysis	NR	-	6.7	0.059	NR	-	2.5 0.7	0.045 0.013
Unknown E3 – not hydrolysed [d]	NR	-	ND	-	NR	-	4.3	0.076
Unknown E4 – after hydrolysis	3.7	0.024	ND	-	2.9	0.042	ND	-
Unknown E9 – after hydrolysis	ND	-	ND	-	ND	-	7.4 5.6	0.13 0.099
Unknown E10 – after hydrolysis	ND	-	ND	-	ND	-	8.3	0.15

	Immature endive				Mature endive			
	^{14}C-Phenyl Label R-enantiomer 1× 0.42 kg ai/ha DAT 20 0.65 mg/kg eq		^{14}C-Pyridyl Label R-enantiomer 1× 0.42 kg ai/ha DAT 20 0.88 mg/kg eq		^{14}C-Phenyl Label R-enantiomer 2 × 0.42 kg ai/ha; DAT 28 1.4 mg/kg eq		^{14}C-Pyridyl Label R-enantiomer 2 × 0.42 kg ai/ha DAT 28 1.8 mg/kg eq	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
pyridinyl N-sugar conjugate								
Unknown E11 – after hydrolysis pyridinyl N-sugar conjugate	ND	-	ND	-	ND	-	8.0	0.14
Unknowns in acetonitrile/water; not hydrolysed [d]	3.0	0.020	11.8 [b]	0.10	1.3	0.018	2.2	0.040
Unknowns in EtOAc phase; after hydrolysis	1.4	0.009	0.9	0.008	1.0	0.014	0.6	0.011
Unknowns in water phase; after hydrolysis	4.3	0.028	4.3	0.038	2.3	0.033	3.1	0.055
PES; after hydrolysis	3.4	0.022	1.3	0.011	2.2	0.031	1.2	0.022
Total	100		100		100		98.6	
- Total identified	84.2		75.0		90.3		54.2	
- Total not hydrolysed [d]	3.0		11.8		1.3		6.5	

TRR = total radioactive residue expressed as fluazifop-butyl equivalents.

ND = not detected.

NR = not relevant for this label, since the compound does not contain the phenyl or pyridyl group

[a] included in CF3-pyridone (X, free) figures

[b] maximum 0.045 mg/kg eq or 5.1% TRR,

[c] includes residues released from solids by HCl/KOH

[d] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

Cereals, pulses and oilseeds

Metabolism study 6 (alfalfa forage)

In a non-GLP study, the metabolism of ^{14}C-phenyl- or ^{14}C-pyridyl-fluazifop-butyl (RS) was studied in outdoor pot grown alfalfa forage following a foliar application [Bell *et al*, 1984, PP9/0049, report RJ0340B]. Alfalfa plants (lucerne, variety Euver) were grown in pots in a netted area exposed to natural weathering conditions, in Bracknell, Berkshire, UK, in 1982. Alfalfa plants were grown until just flowering and were then cut to 8 cm. Eight days later the regrowth was sprayed with an EC formulation of ^{14}C- phenyl-labelled fluazifop-butyl (RS) or ^{14}C- pyridyl-labelled fluazifop-butyl (RS) at an actual rate of 0.49 kg ai/ha. An adjuvant was added (0.1% Agral 90). When plants were just flowering they were cut to 8 cm above soil level (DAT = 20) after which they were regrown to the flowering stage and cut again to 8 cm above soil level (DAT =87). The whole crop (typically 0.25 kg) was cut into 25 cm lengths. Samples were stored at -20 ºC. The storage period was not stated but does not exceed 11 months (start and end of the experimental period).

Samples were extracted with acetonitrile followed by thouree extractions with acetonitrile/water (1:1, v/v). Extracts and remaining solids were analysed by (combustion) LSC. Most of the radioactivity could be extracted with acetonitrile/water: 97% TRR (phenyl, DAT 20), 87% TRR (phenyl, DAT 87), 94% TRR (pyridyl, DAT 20). At the first harvest interval (day 20), radioactive residue was present at concentrations of 3.2 mg/kg eq for the phenyl label and 2.5 mg/kg eq for the pyridyl label. At the second harvest (day 87) only plants treated with phenyl label were examined and the residue had decreased to 0.13 mg/kg eq. Results are shown in Table 11.

Acetonitrile and acetonitrile/water extracts were combined (87–97% TRR) and the acetonitrile was evaporated from the extract. The remaining aqueous phase was acidified to pH 3 and partititioned with diethyl ether. The diethyl ether phase was evaporated to dryness and redissolved in 6 M HCl, while the aqueous phase of was acidified with 6 M HCl. Both fractions were heated for 2 hours at 58-63 ºC in 6 M HCl and then partitioned with diethyl ether. It proved necessary to re-

hydrolyse the aqueous phase. This was done with 6 M HCl at 80–89 ºC for 2 hours. This was followed by diethylether partition. All organic phases were analysed by TLC using reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III) and Pyr-Ph ether (IV).

After 20 days, the major compound of the phenyl-and pyridyl labelled precursor was fluazifop acid (II, free and conjugated) at 70–72% TRR. After 87 days, fluazifop acid (II, free and conjugated) had decreased to 37% in the phenyl-label group. Parent, despyridinyl acid (III) and Pyr-Ph ether (IV) were not detected. The presence of CF3-pyridone (X) and hydroxyfluazifop acid (XL) was not verified. Unidentified organosoluble fractions represented 16-20%TRR (phenyl) and 6% (pyridyl). Individual compounds were < 6% TRR in the organosoluble fractions. Water fractions were not analysed: maximum 2–11% TRR (phenyl) and 5% (pyridyl).

Reviewer's conclusion:

Fluazifop acid (II) is stable in 6 M HCl for 3 hours at 80 ºC (see hydrolytic stability section above). It is therefore assumed that fluazifop acid (II) is also stable in 6 M HCl for 2 hours at 80-89 ºC. It is not clear why no despiridinyl acid (III) was detected. The presence of CF3-pyridone (X), fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified (no reference standard), but they may be present in the organic fractions (individual compounds < 6% TRR). Fractions that were not subjected to hydrolysis (PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) or hydroxyfluazifop acid (XL) at a maximum of 6–13% TRR.

Table 11 Radioactive residues in alfalfa forage expressed as fluazifop-butyl equivalents.

Treatment	Phenyl (RS) 0.49 kg ai/ha DAT 20	Phenyl (RS) 0.49 kg ai/h DAT 87	Pyridyl (RS) 0.49 kg ai/ha DAT 20
TRR (mg/kg eq)	3.2	0.13	2.5
fluazifop-butyl	ND	ND	ND
Total fluazifop acid (II) (free + conjugates) - fluazifop acid (free) - fluazifop organo soluble conj - fluazifop polar conj	70 -13 -22 -35	37 -7 -17 -13	70 -10 -19 -41
Pyr-Ph ether (IV)	ND	ND	ND
Despyridinyl acid (III)	ND	ND	NR
CF3-pyridone (X)	NR	NR	no ref std
Fluazifop alcohol (XXXIV)	no ref std	no ref std	no ref std
Hydroxyfluazifop acid (XL)	no ref std	no ref std	no ref std
unknowns in organic fractions - after hydrolysis	20 [e] - 8 - 8 [c] - 4 [c]	16 [e] -4 -12 [c]	6 -3 -3 [c]
unknowns in aqueous fraction - after hydrolysis	2 - 2 [b] -	11 - 5 [b] - 6	5 - 1 [b] - 4
PES - not hydrolysed [a] - after hydrolysis	6 - 6 --	29 - 13 - 2 [d] - 14 [d]	13 - 6 - 7 [d]
Total	98	93	94
Total identified	70	37	70
Total - not hydrolysed [a]	6	13	6

TRR = total radioactive residue expressed as fluazifop-butyl equivalents.

NR = not relevant for this label, since the compound does not contain the phenyl or pyridyl group

[a] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

[b] Partitioned into the aqueous fraction after treatment of the organic phase with 6 M HCl

[c] Partitioned into the organic fraction after treatment of the aqueous phase with 6 M HCl

[d] Solids appearing after treatment of the organic or aqueous phase with 6 M HCl

[e] each compound <6% TRR
[or] Stayed in the organic phase after treatment of the organic phase with 6 M HCl
[aq] Stayed in the aqueous phase after treatment of the aqueous phase with 6 M HCl

Metabolism study 7 (cotton forage)

In a non-GLP study, the metabolism of ^{14}C-phenyl-fluazifop-butyl as R- or S-enantiomer was studied in indoor pot grown cotton plants following a topical application [Evans and Cavell, 1984, PP9/0048, report RJ0356B]. Six 40 day old cotton plants (variety Delta Pine) were grown in a glasshouse in pots filled with sandy loam (2 plants per pot). The plants were treated either with EC formulated ^{14}C-phenyl-labelled-fluazifop-P-butyl (enantiomer ratio R:S = 97.5%:2.5%) or ^{14}C-phenyl-labelled-S-fluazifop-butyl (enantiomer ratio R:S = 2.6%:97.4%). The formulations were applied in aqueous 0.01% Agral 90 by syringe to leaves and stems of each of the two plants per pot. The amounts applied were calculated to represent an actual application rate of about 0.45 kg ai/ha. Cotton forage was harvested 27 days after application (growth stage not stated). Samples were stored at -20 °C until analysis for a maximum of 9 months.

Chopped cotton forage samples were extracted subsequently with acetonitrile: water (80:20, v/v) and water. Extracts and remaining solids were analysed by (combustion) LSC. At harvest 71% and 81% of the applied doses of respectively the R and S enantiomer of fluazifop-butyl were recovered in the cotton plants. Results in terms of mg/kg eq are not reported.

The acetonitrile/water extract (89% and 95% TRR for R and S enantiomer) was partitioned twice with hexane (A: 22%; 23% TRR) and then between diethyl ether (C: 32%; 55% TRR) and water (B: 35%; 17% TRR). The diethyl ether extract C (32%; 55% TRR) was fractionated on a Bond Elut Column into a polar fraction (3C: 5.2%; 12% TRR) and an apolar fraction (1C+2C: 26%; 44% TRR).

Water phase B (35%; 17% TRR) was mixed with the Bond Elut polar fraction 3C (5.2%; 12% TRR). These polar metabolites were subjected to acidic hydrolysis (2*hour*6 M HCl at 60 °C) and partitioned with diethyl ether (G 29%; 24% TRR).

The Bond Elut apolar fraction 1C+2C (26%; 44% TRR) was partitioned with 0.05 M sodium bicarbonate solution to get an aqueous phase (22%; 38% TRR) and a diethyl ether phase (D: 4.0%; 5.5% TRR). The aqueous phase (22%; 38% TRR) was acidified and then partitioned with diethyl ether (E 22%; 38% TRR).

The diethylether phase D (4.0%; 5.5% TRR) was mixed with hexane extract A (22%; 23% TRR). These organo soluble compounds were subjected to alcoholic caustic hydrolysis (1.0 M NaOH in 2-propanol, 4 hours, 21 °C), neutralised by addition of 0.1 M HCl (pH1) and partitioned with diethyl ether.

The remaining aqueous phase was acidified and then again partitioned with diethyl ether. The combined diethyl ether fractions were partitioned with 0.1 M sodium bicarbonate solution. The aqueous phase was acidified and then partitioned with diethyl ether (F: 25%; 26% TRR).

The ratio of fluazifop acid (II) enantiomers was determined by chiral HPLC (using Altex ultrasphere IP column). Organic fractions were analysed by TLC with 2 solvent systems. Reference standards used were fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV) and fluazifop alcohol (XXXIV).

The (R)/(S) ratio remained unchanged in fluazifop acid (II) during alkaline and acid hydrolysis, suggesting that no epimerisation occurred in the plant or during sample extraction and hydrolysis. The nature of the residues and enantiomeric ratios of the individual components for cotton forage treated with phenyl-labelled fluazifop-butyl (R and S-enantiomers) are summarized in Table 12 and Table 13. Results in terms of mg/kg eq are not reported.

Approximately 24% TRR remained intact as unmetabolized parent compound in/on the cotton forage. Free fluazifop acid (II) and its conjugates were formed as the primary metabolites by both the enantiomers (37–56% TRR after hydrolysis). Pyr-Ph ether (IV, free and conjugate) was found 2.5–

2.7% TRR by both the enantiomers. Despyridinyl acid (III) was released after hydrolysis at 1.5–7.3% TRR. Unidentified organosoluble fractions represented 5.7–7.4%TRR. Water fractions were not analysed: maximum 8.6-14% TRR.

Reviewer's conclusion:

Fluazifop acid (II) is stable in 6 M HCl for 2 hours at 60 ºC and 1 M NaOH in 2-propanol during 4 hours at 21 ºC (see hydrolytic stability section above). The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. CF3-pyridone (X) is not detected, because only phenyl labelled compounds were used in this study. Fluazifop alcohol (XXXIV) was not detected. The presence of hydroxyfluazifop acid (XL) was not verified, but may be present in the organic fractions (maximum 5.7-7.4% TRR). Fractions that were not subjected to hydrolysis (organic, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III) at a maximum of 6-11% TRR.

Table 12 Enantiomer ratio in selected fractions of cotton leaf extracts

Fraction	^{14}C-phenyl R-enantiomer 1 × 0.45 kg ai/ha DAT 27		^{14}C-phenyl S enantiomer DAT 27 %TRR	
	% R-enantiomer	%S enantiomer	% R-enantiomer	%S enantiomer
2F = fluazifop-butyl	97.9	2.1	1.1	98.9
2E = free fluazifop acid (II)	96.1	3.9	0.3	99.7
2H = fluazifop acid (II) released from conjugates	96.0	4.0	0.6	99.4

Table 13 Nature of residues in cotton forage

Enantiomers	^{14}C-phenyl R-enantiomer 0.45 kg ai/ha DAT=27 %TRR	^{14}C-phenyl S enantiomer 0.45 kg ai/ha DAT=27 %TRR
TRR (mg/kg eq)	not determined	not determined
Parent	23.9	23.2
Total fluazifop acid (II, free + conjugates) - fluazifop acid free - fluazifop conjugates	37.5 - 22.7 - 14.8	55.9 - 37.9 - 18.1
Pyr-Ph ether (IV, free + conjugates) - Pyr-Ph ether (free) - Pyr-Ph ether (conjugates)	2.7 - 0.8 - 1.9	2.5 - 1.6 - 0.9
Despyridinyl acid (III, conjugates)	7.3	1.5
CF3-pyridone (X)	NR	NR
Fluazifop alcohol (XXXIV)	ND	ND
Hydroxyfluazifop acid (XL)	no ref std	no ref std
unknowns in organic fractions - not analysed; not hydrolysed [a] - after hydrolysis	7.4 - 0.6 -1.6 [or] -5.2 [c]	5.7 - 1.4 -1.4 [or] -2.9 [c]
unknowns in aqueous fraction -not hydrolysed [a] - after hydrolysis	14.2 -1.7 -1.4 [b] - 11.1 [aq]	8.6 -1.0 -2.6 [b] -5.0 [aq]
PES; not hydrolysed [a]	8.9	3.7
Total	101.9	101.1
Total identified	71.4	88.8
Total – not hydrolysed [a]	11.2	6.1

NR = not relevant for this label, since the compound does not contain the phenyl or pyridyl group

[a] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)

[b] Partitioned into the aqueous fraction after treatment of the organic phase with 1 M NaOH in 2-propanol

[c] Partitioned into the organic fraction after treatment of the aqueous phase with 6 M HCl

[or] Stayed in the organic phase after treatment of the organic phase with 1 M NaOH in 2-propanol

[aq] Stayed in the aqueous phase after treatment of the aqueous phase with 6M HCl

Metabolism study 8 (cotton forage and seeds)

In a non-GLP study, the metabolism of ^{14}C-phenyl- or ^{14}C-pyridyl-fluazifop-butyl (RS) was studied in outdoor pot grown cotton plants following a topical application [Goddard *et al*., 1981, PP9/0203, report RJ0196B]. The cotton plants (variety not indicated) were grown in pots with sandy soil outdoors in Goldsboro, NC, USA in 1979. Immature cotton plants were treated with a topical foliar or soil application of EC formulated ^{14}C- phenyl and ^{14}C- pyridyl-labelled fluazifop-butyl. Plants were treated at the 6 leaf stage in late May or early June. An adjuvant was added (0.01% Agral 90). Formulations were applied to 2–6 leaves/plant and to the growing tips as 1 ul spots or to the soil around the plants as 10 ul spots. The applications were equivalent to 0.05–0.06 kg ai/ha (A, D, E, 2 treated leaves, phenyl and pyridyl), 0.33 kg ai/ha (B, C, 6 treated leaves, phenyl only) and 0.47 kg ai/ha (F, G, soil treatment, phenyl only). During the first month after treatment, protection from rainfall was afforded by a polythene shelter. Plant A and D were harvested as immature plants at DAT=24 and 20, respectively. Plants B, C, E, F, G were harvested at maturity in October at DAT =130. Storage conditions were not indicated.

Cotton seeds from mature plants B, C, E, F and G were delinted, homogenised and analysed by combustion LSC. The total radioactive residue in the seeds is shown in Table 14. TRR in forage was not determined. Seeds and forage from soil applications were not characterised.

Treated leaves from immature plants A and D (phenyl and pyridyl, respectively) were washed by dipping in acetonitrile (3%, 5% TRR) and then extracted with acetonitrile/water (1:1, v/v) (84%, 73% TRR). The combined acetonitrile and acetonitrile/water extract (87%; 78% TRR) was evaporated to remove the acetonitrile, acidified to pH 1 and then partitioned between diethyl ether (23%, 34% TRR) and water (61%, 39% TRR). One aliquot of the water fraction for plant A and D was adjusted to 0.1 M NaOH, refluxed for 3 hours, adjusted to pH 1 with HCl and then partitioned between diethyl ether (35%, 12% TRR) and water (21%, 27% TRR). The other aliquot of the water fraction of plant A (phenyl label) was adjusted to 0.1 M HCl, refluxed for 3 hours and then partitioned between diethyl ether (45% TRR) and water (16% TRR). The other aliquot of the water fraction of plant D (pyridyl label) was adjusted to 1 M HCl, refluxed for 1 hour and then partitioned with diethyl ether (15% TRR). The water phase was adjusted to 6 M HCl, refluxed for 2 hours, adjusted to 1 M HCl and then partitioned between diethyl ether (13% TRR) and water (11% TRR).

Homogenised seeds from foliar treatments (B,C, E) were subsequently extracted with hexane (15%, 18% TRR) and diethyl ether (1%; 0% TRR). The remaining solids were soaked in water for 2 hours, followed by two extractions with acetonitrile/water (1:1, v/v) (46%; 47% TRR) and then with methanol (2%; 0% TRR). One aliquot of the remaining solids from seeds B and C (37% TRR) was treated with 0.1 M NaOH (6 hours, 90 ºC) and then partititoned between diethyl ether and water. Another aliquot of the remaining solids from seeds B and C (37% TRR) was treated with 1 M HCl (6 hours, 90 ºC) and then partitioned between diethyl ether and water.

The hexane and diethyl ether extracts from seeds B and C (phenyl label) were combined (16% TRR), evaporated to dryness and than partitioned between hexane (8% TRR) and acetonitrile (8% TRR). The hexane phase was cleaned up on a Florisil column. The clean hexane phase and the acetonitrile phase were evaporated to dryness and then refluxed in 0.1 M NaOH for 1 hour. The hydrolysates from each phase were partitioned between diethylether and water. The water phases were adjusted to pH 1 and then again partitioned between diethylether and water.

The acetonitrile/water extract from seeds B and C (phenyl label) was evaporated to remove the acetonitrile, adjusted to pH1 and then partitioned between diethyl ether and water. One aliquot of the water phase was adjusted to 1 M NaOH, refluxed for 2 hours, adjusted to pH1 and then partitioned between diethyl ether and water. The other aliquot of the water phase was adjusted to 6 M HCl, refluxed for 3 hours and then partitioned between diethyl ether and water.

The hexane extracts from seeds E (pyridyl label) was cleaned up on a Florisil column. The clean hexane phase was evaporated to dryness and then refluxed in 0.5 M NaOH for 1 hour. The hydrolysates were partitioned between diethylether and water. The water phases were adjusted to pH 1 and then again partitioned between diethylether and water.

The acetonitrile/water extract from seeds E (pyridyl label) was evaporated to remove the acetonitrile, adjusted to pH1 and then partitioned between diethyl ether and water. The water phase was adjusted to 6 M HCl, refluxed for 2 hours, adjusted to 1 M HCl and then partitioned between diethyl ether and water.

Leaf and seed extracts and remaining solids were analysed by (combustion) LSC. Diethyl ether fractions were analysed by TLC using reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), CF3-pyridone (X) and compound 9. Results are shown in Table 14.

Cotton forage (DAT 20–24) contained low levels of parent (2–6% TRR). The major compounds were despyridinyl acid (III, 21–32% TRR, conjugated) and fluazifop acid (II, 13–24% TRR, free or conjugated). Despyridinyl acid (III) and CF3-pyridone (X) were released after hydrolysis at 21–32% TRR (phenyl label) and 5% TRR (pyridyl label, acid hydrolysis only), respectively. The presence of Pyr-Ph ether (IV) and hydroxyfluazifop acid (XL) was not verified (no reference standards). Unidentified organosoluble fractions contained 21–25% TRR (phenyl) or 18–28% TRR (pyridyl). Individual organo-soluble compounds did not account for more than 4% TRR. Water fractions contained 16–21% TRR (phenyl) or 11–27% TRR (pyridyl).

Parent was detected at low levels (1% TRR) in cotton seeds from phenyl labelled foliar applications. The major metabolites were despyridinyl acid (III, 23–27% TRR; conjugated; phenyl label only) and fluazifop acid (10–14% TRR, free or conjugated). Despyridinyl acid (III) was released after hydrolysis at 23–27% TRR in seeds. The presence of Pyr-Ph ether (IV) and hydroxyfluazifop acid (XL) was not verified (no reference standards). Unidentified organosoluble fractions contained 21–22% TRR (phenyl) or 40% TRR (pyridyl). Individual organo-soluble compounds did not account for more than 4% TRR. Water fractions contained 16–19% TRR (phenyl) or 11% TRR (pyridyl).

Reviewer's conclusion:

This study is not acceptable. Fluazifop acid (II) degrades under the reflux conditions used, accounting for the low levels of fluazifop acid (II) and the high levels of despyridinyl acid (III). CF3-pyridone (X) degrades under alkaline hydrolysis conditions.

Table 14 Nature of residues in cotton forage and seeds

Plant	A – phenyl (RS)	D – pyridyl (RS)	B, C – phenyl (RS)	E- pyridyl (RS)	F, G- phenyl (RS)
Treatment	topical leaf treatment 0.05 kg ai/ha	topical leaf treatment 0.05 kg ai/ha	topical leaf treatment 0.33 kg ai/ha	topical leaf treatment 0.06 kg ai/ha	topical soil treatment 0.47 kg ai/ha
Harvest	DAT 24 forage	DAT 20 forage	DAT 130 seeds	DAT 130 seeds	DAT 130 seed
TRR (mg/kg eq)	-	-	0.09	0.008	0.02
	%TRR	%TRR	%TRR	-	-
Parent	2	6	1	-	-
Total fluazifop acid (II, free + conj) - Fluazifop acid (free) - Fluazifop (conjugated)	13; - 13; -	24N; 22H; - 22; - 2N; -H	10N; 10H; - 2; - 8N; 8H	14 - - 14N	-
Pyr-Ph ether (IV)	no ref std	no ref std	no ref std	no ref std	no ref std-
Despyridinyl acid (III, conjugates) *	21N; 32H	NR	23N; 27H	NR	-
CF3-pyridone (X, conjugates) [a] *	NR	-N; 5H	NR	-	NR
Compound U1 (conjugated)		3N; -H	-	-	-
Compound U2 (conjugated)		-N; 6H	-	-	-
Acetonitrile wash; not hydrolysed	3	5		-	-
unknowns in diethyl ether fraction - not hydrolysed [b] - after hydrolysis [c]	22N; 21H - 8 - 14N; 13H	13N; 23H - 6 - 7N; 17H	21N; 22H - 5 - 16N; 17H	40H - 15 - 25H	-
unknowns in aqueous fraction					

Plant	A – phenyl (RS)	D – pyridyl (RS)	B, C – phenyl (RS)	E- pyridyl (RS)	F, G- phenyl (RS)
Treatment	topical leaf treatment 0.05 kg ai/ha	topical leaf treatment 0.05 kg ai/ha	topical leaf treatment 0.33 kg ai/ha	topical leaf treatment 0.06 kg ai/ha	topical soil treatment 0.47 kg ai/ha
Harvest	DAT 24 forage	DAT 20 forage	DAT 130 seeds	DAT 130 seeds	DAT 130 seed
TRR (mg/kg eq)	-	-	0.09	0.008	0.02
- after hydrolysis [aq]	21N; 16H	27N; 11H	19N; 16H	11H	
PES – - not hydrolysed [b] - hydrolysed	 13 -	 23 -	 - 24N; 25H	 36 -	 - -
Total	95; 100	101; 101	98; 101	101	-
Total identified	36N; 47H	30N; 33H	34N; 38H	14H	-
Total – not hydrolysed	24	34	5	51	-

NR = not relevant for this label, since the compound does not contain the phenyl or pyridyl group

[a] when refluxed in 0.1 M NaOH, CF3-pyridone (X) converts to compound 9 which partitions into the aqueous phase (not analysed)

[b] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

[c] Partitioned into the organic fraction after treatment of the aqueous phase with 0.1 M NaOH or 0.1-6 M HCl

[aq] Stayed in the aqueous phase after treatment of the aqueous phase with 0.1 M NaOH or 0.1-6 M HCl

N after base hydrolysis

H after acid hydrolysis

* Despyridinyl acid (III) and CF3-pyridone (X) probably represent degradation of fluazifop acid (II) or fluazifop conjugates as the result of the hydrolysis conditions used

Metabolism study 9 (oilseed rape seeds)

In a non-GLP study, the metabolism of ^{14}C-pyridyl-fluazifop-butyl (RS) was studied in outdoor pot grown oilseed rape plants following a topical application [Day *et al*., 1981, PP009/0047, report RJ0187B]. Immature oilseed rape plants (variety Maris Haplona) were grown in pots (4 plants/pot) with sandy loam soil. Part of the EC formulation was applied by syringe to the foliage (the first 3 true leaves, the growing tip and the stem; 0.44 mg ai) and the other part to the soil of the same pot (2.93 mg ai). An adjuvant was added (0.01% Agral 90). The actual application rate was equivalent to 0.840 kg ai/ha. The oilseed rape plants were grown to maturity in the field under a polythene tent during the period July-October 1980 at Bracknell, Berkshire, UK. Pods were harvested 10-13 weeks after application when dry, brown and papery. Pods were stored at -20 ºC. Storage period was not stated, but does not exceed 5 months (start and end of the study conduct).

Seeds were separated from the pods and homogenised seeds were subsequently extracted with hexane (fraction A, 17% TRR) and diethyl ether (fraction B, 44% TRR). The remaining solids from the primary extraction were soaked in water overnight, followed by two extractions with acetonitrile/water (1:1, v/v) (fraction C, 32% TRR). Extracts and remaining solids (fraction D, 7.2% TRR) were analysed by (combustion) LSC.

Hexane fraction A (17% TRR) was rotatory evaporated until the oil remained. The oil was redissolved in hexane and partitioned with acetonitrile. The acetonitrile fraction was washed with hexane (fraction A1, 4% TRR). The hexane wash was combined with the hexane fraction and cleaned up by Florisil column chouromatography. Residues eluted in the 50% ether in hexane fraction (fraction A3, 12% TRR).

Diethyl ether fraction B (44% TRR) was rotary evaporated until the oil remained. The oil was redissolved in diethyl ether and partitioned with 1% NaHCO3 pH 8–9. The aqueous phase was acidified to pH 1 with HCl and residues were partitioned into diethyl ether (fraction B2, 33% TRR). The primary diethyl ether fraction (fraction B1, 11% TRR) was cleaned-up with Florisil column chouromatography and residues eluted with two fractions: 25% ether in hexane and 50% ether in hexane. The 25% ether in hexane fraction was rotary evaporated to the oil, redissolved in 15% ether in

hexane, cleaned-up with Florisil column chouromatography and residues now eluted in the 50% ether in hexane fraction. The two 50% ether in hexane fractions were combined (fraction B4, 11% TRR).

Aliquots of fraction A3 and fraction B4 were combined (24% TRR) and hydrolysed with 0.1 M KOH for 2 hours under reflux conditions. The residues were partitioned between diethyl ether (fraction D1, 3% TRR) and the aqueous phase. The aqueous phase was acidified to pH 1 with HCl and residues partitioned into diethyl ether (fraction D2, 21% TRR).

Acetonitrile/water fraction C (31% TRR) was partitioned between diethyl ether (fraction C1; 23% TRR) and an aqueous phase. The aqueous phase was hydrolysed with 0.1 M KOH for 2 hours under reflux conditions and partitioned with diethylether. The residues partitioned between diethyl ether (fraction C3, 4% TRR) and water (4% TRR).

Solid fraction D (8% TRR) was hydrolysed with 0.1 M KOH for 30 min under reflux conditions and then acidified to pH 1 with HCl and partitioned with diethyl ether. However, the majority of the residues remained in the solids.

Selected organic fractions were analysed by 1D- and 2D-TLC using reference standards for fluazifop-butyl (I), fluazifop acid (II), Pyr-Ph ether (IV), CF3-pyridone (X) and compounds 8 and 9. Results are shown in Table 15.

The total radioactive residue was 0.65 mg/kg, of which 93% could be extracted using solvent extraction. Parent was not detected. The major compound was the fluazifop acid (II, 69% TRR) as free acid (54% TRR), non-polar conjugated fluazifop (13% TRR) or polar conjugated fluazifop (2.0% TRR). Unidentified organosoluble fractions contained 22% TRR (pyridyl). Individual organo-soluble compounds were <6% TRR. Water fractions contained a maximum of 1% TRR (pyridyl).

Reviewer's conclusion:

This study is considered of limited value because of the hydrolysis conditions used for CF3-pyridone (X). Fluazifop acid (II) is stable in 0.1 M NaOH for 3 hours reflux (see hydrolytic stability section above) and therefore it is assumed that fluazifop acid (II) is also stable in 0.1 M KOH for 2 hours reflux. The presence of CF3-pyridone (X) conjugates remains unnoticed, because CF3-pyridone (X) degrades under alkaline hydrolysis conditions to compound 9 (see hydrolytic stability section). Despyridinyl acid (III) cannot be detected with the pyridyl label. The presence of fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified.

Table 15 Nature of residues in oilseed rape seeds

Enantiomers	Seeds Pyridyl label (RS) 1 × 0.84 kg ai/ha DAT 70-91
TRR	0.65 mg/kg eq
Parent	-
Total fluazifop acid (II, free + conjugates) - fluazifop acid - fluazifop lipophilic conjugates - fluazifop polar conjugates	69 -54 -13 -2.0
Pyr-Ph ether (IV)	ND
Despyridinyl acid (III)	NR
CF3-pyridone (X) *	ND (degraded)
Fluazifop alcohol (XXXIV)	no ref std
Hydroxyfluazifop acid (XL)	no ref std
unknowns in organic fraction [a] not hydrolysed after hydrolysis	22 - 2.0 (A1) - 4.0 (C1) - 11 (D2) - 3.0 (D1) - 2.0 (C3)
unknowns in water fraction after hydrolysis	4.0 (C4)

Solids (PES); after hydrolysis	6.2
Total	101.2
Total identified	69
Total – not hydrolysed	6.0

ND = not detected

[a] No metabolite accounted for more than 6% TRR

* CF3-pyridone (X) degrades under the hydrolysis conditions used

Metabolism study 10 (soya bean plant and maize plant)

The metabolism of ^{14}C-phenyl-fluazifop-butyl (RS) was investigated in immature maize and soya bean plants in a greenhouse [Hignett *et al.*, 1979, PP9/0199, report RJ0085C]. The radioactive formulation was applied by syringe to the leaves of nine immature soya bean plants (*Glycine max*, variety Amsoy) growing in soil (sand) and two plants in nutrient solution (sphagnum peat). The radioactive formulation was also applied to the leaves of five immature maize plants (*Zea mays*, variety 3369) growing in soil (sand) as well as two plants in nutrient solution (sphagnum peat). The treatment rate (10 microgram per leaf) was assumed to be similar to 0.75 kg ai/ha. Metabolism in maize was only briefly studied. It is not described in the report for how long and at what temperature the samples have been stored.

Phytotoxic effects on the maize plants were observed after 5 days, and after 13 days the plants were virtually dead. No phytotoxic effects were observed in soya bean plants.

Soya and maize plants growing in soil were harvested for freeze drying and autoradiography at 1 and 14 DAT. Whole plant autoradiograms showed that at DAT 1 very little translocation of radioactivity had occurred from the treated leaf (maize and soya). At DAT 14 the radiocarbon had spread thouroughout the soya bean plants including the roots and the new growth. In maize, uptake and translocation was slower.

Soya bean plants in soil were harvested at 1, 15, 28 and 50 DAT. Soya in nutrient solution was harvested at 6 and 29 DAT and maize in nutrient solution at 6 and 13 DAT. Whole plants were harvested, except at 50 DAT when the roots were discarded. Treated leaves were rinsed with acetonitrile and then sequentially extracted with acetonitrile and water. Roots and the remainder of the plant were separately extracted with acetonitrile and then water. Radioactive contents of washes, extracts and remaining solids were analysed by (combustion) LSC.

Total radioactive residues were not reported. Distribution of radioactivity is shown in table 16. In treated leaves from soya bean plants grown in soil radioactivity decreased from 77% TAR at DAT 1 to 44% TAR at DAT 50, while radioactivity increased in the rest of the plant (14% TAR at DAT 1; 30% TAR at DAT 50). Similar behaviour is found for soya bean plants grown in nutrient solution. In maize plants radioactivity was mainly recovered in the treated leaves.

Washes and extracts were further examined by TLC (see table 17) using fluazifop-butyl and fluazifop acid (II) as reference standards. The halflife of fluazifop-butyl in soya bean plants was less than 1 day. Contrary, the halflife of fluazifop-butyl in maize was about 6 days. Initially the major metabolite in soya bean plants was fluazifop acid (II). The proportion of baseline material increased during the experiment and was the dominant extractable component at DAT 50. The acetonitrile-extractable baseline radioactivity was subsequently shown to consist of at least 4 components when chouromatographed by TLC. The same components were not seen in a corresponding aqueous extract. Attempts were made to generate fluazifop acid (II) from the conjugates in the baseline material.

Treated leaf extracts (acetonitrile and water combined) from DAT 6 (nutrient solution) were acidified to pH3 and partitioned between a diethyl ether-fraction (14% TER (total extracted residue), not further analysed) and a water fraction (86% TER). The water fraction was partitioned between a butanol-fraction (80% TER) and water-fraction (6% TER). The butanol-fraction (80% TER) is assumed to contain conjugated material only, and was divided into two aliquots.

- Aliquot A was hydrolysed with 0.001 M HCl (1 hour reflux), followed by partitioning in a diethyl ether-fraction (10% TER) and water-fraction (70% TER). The water-fraction was hydrolysed again, now with 0.1 M HCl (1 hour reflux). Finally, partitioning took place between a diethyl ether-fraction (67% TER) and water-fraction (3% TER).
- Aliquot B was hydrolysed with 0.001 M NaOH (1 hour reflux) with final pH adjustment to pH3, and subsequently partitioned between diethyl ether (7% TER) and water (73% TER). The water-fraction was then hydrolysed again with 0.1 M NaOH (1 hour reflux) with final pH adjustment to pH1, followed by partitioning into diethyl ether (61% TER) and water (4% TER).

The two final diethyl ether-fractions were analysed by TLC, which demonstrated that complete hydrolysis to the fluazifop acid (II) was achieved.

The DAT 15 treated leaf and "rest of plant" acetonitrile extracts, the DAT 28 treated leaf water extract, the DAT 50 "rest of plant" water extract were hydrolysed with 0.1 M NaOH (1 hour reflux), after which the pH was adjusted to 1 or 2. Then partitioning took place into an diethyl ether-fraction (96%, not reported, 88%, 66% TER, respectively, of the original extract of DAT 15 leaf, DAT 15 rest of plant, DAT28 leaf and DAT50 rest of plant) and a water-fraction. The diethyl ether-fraction was examined by TLC, which showed that fluazifop acid (II) was generated in each of these extracts. However, no quantification took place.

To conclude, during time the ratio of conjugated to free acid increased, hydrolysis of the baseline material became less facile and less complete, and the amount of radioactivity associated with the post extracted solids increased. The increasing proportion of non-extracted ^{14}C may be the consequence of incorporation into the plant's normal metabolic system.

Note. The %TER values were recalculated to %TRR by the present reviewer, by assuming that the radioactive distribution in DAT 6 leaf extracts is identical to the combined extracts for the whole plant (i.e. 54% TAR in acetonitrile, 44% TAR in water, 7% TAR in solids = 104% TAR = 100% TRR). The %TER values were recalculated by assuming that total extracted residue (TER) is 54+44/104=94% TRR and remaing solids are 7/104=6.7% TRR. A 67% TER value is than recalculated as 0.67×94=63% TRR. Using this conversion, the following distribution is found for the DAT 6 leaf extract:

14% TER = 13% TRR in first diethyl ether fraction, assumed to be free fluazifop acid (II)

6.0% TER = 5.6% TRR in the water fraction after partitioning with butanol (not hydrolysed)

61–67% TER = 57–63% TRR conjugated fluazifop acid (II)

7.0–10% TER = 6.6–9.4% TRR in diethyl ether fraction (after first hydrolysis)

3.0–4.0% TER = 2.8–3.8% TRR in the water fraction (after second hydrolysis)

6.7% TRR = remaining solids (not hydrolysed)

Reviewer's conclusion:

Fluazifop acid (II) is stable after 1 hour reflux in 0.1 M NaOH or 0.1 M HCl (see hydrolytic stability section above). CF3-pyridone (X) cannot be detected with the phenyl label. The presence of Pyr-Ph ether (IV), despyridinyl acid (III), fluazifop alcohol (XXXIV) or hydroxyfluazifop acid (XL) was not verified, but may be present in the organic fractions (6.6–9.4% TRR, day 6 leaf). Fractions that were not subjected to hydrolysis (water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III) or hydroxyfluazifop acid (XL) at a maximum of 12.3% TRR (day 6 leaf).

Table 16 Distribution of radioactivity in soya and maize plants after topical treatment with ^{14}C-phenyl-fluazifop-butyl (RS)

Plant	DAT	Treated leaf wash %TAR	Treated leaf [a] %TAR	Rest of plant [a] %TAR	Rootwash and nutrient solution %TAR	Total recovered (%TAR)
Soya forage; grown in soil 1 × 0.75 kg ai/ha TRR not reported	0	104	-	-	-	104
	1	9	68	14	-	91
	2	1	83	9	-	93
	15	0	47	36	-	83
	28	0	38	44	-	82
	50	0	44	30	-	74
soya forage, grown in nutrient 1 × 0.75 kg ai/ha TRR not reported	6	<1	73	30	<1	104
	29	<1	93 [b]	[b]	3	96
maize forage, grown in nutrient 1 × 0.75 kg ai/ha; TRR not reported	6	30	29	5	<1	65
	13	3	71	9	3	86

[a] Radioactivity in extracts and remaining solids

[b] Radioactivity in leaves plus rest of shoots and roots

Table 17 Distribution of radioactivity of soya bean plant extracts treated with ^{14}C-phenyl-fluazifop-butyl (RS) [b] [c]

DAT	Acetonitrile extract (% TAR)	Water extract (% TAR)	Solids (% TAR)	Total recovered (% TAR)	Characterisation of acetonitrile/water extracts by TLC (%TAR)
Immature soya bean plants grown in pots in sand (greenhouse), topical leaf and stem treatment 1 × 0.75 kg ai/ha, TRR ns					
1	82	- [a]	9	91	15% parent 40% fluazifop acid (II, free) 20% baseline
2	47	42	4	93	1% parent 50% fluazifop acid (II, free) 30% baseline
15	55	22	3	80	0% parent 30% fluazifop acid (II, free) 50% baseline [d]
28	49	20	13	82	0% parent 20% fluazifop acid (II, free) 50% baseline [d]
50	16	36	24	74	0% parent 10% fluazifop acid (II, free) 40% baseline [d]
Immature soya bean plants grown in nutrient solution (greenhouse), topical leaf and stem treatment 1 × 0.75 kg ai/ha, TRR ns					
6	54	44	7	104	0% parent 20% fluazifop acid (II, free) 75% baseline [d]
29	60	16	17	93	0% parent 15% fluazifop acid (II, free) 60% baseline

[a] No aqueous extraction was included.

[b] The proportions of ^{14}C-compounds were obtained from peak height ratios, hence there is an uncertainty of ±10% in these values, and the total extractable radioactivity figures do not correlate exactly with the proportions found in the extracts.

[c] TRRs are not mentioned in the report.

[d] Baseline was further investigated (see text).

Metabolism study 11 (soya bean plant and maize plant)

Translocation and metabolism of ^{14}C-phenyl labelled fluazifop-butyl (RS) was studied in soya and maize plants following injection into the stem [Hignett and Cavell, 1979, PP9/0197, RJ0101B]. Maize (*Zea mays*, variety 3369) and soya (*Glycine max*, variety Amsoy) were grown to a height of 10-15 cm in sphagnum peat under greenhouse conditions. Plants were injected approximately 5 cm above soil with a syringe. Plant roots and shoots were analysed 1, 7, 14 and 28 days after injection. The equivalent dose rate as kg ai/ha was not indicated in the report. The injected stem of soya bean plants (from soil level to a height of 10 cm) was analysed separately.

Autoradiography of freeze dried plants showed that radioactivity was present in both the roots and topshoots of both plants (i.e. xylem and phloem mobile). Translocation was less rapid in maize possibly because of the phytotoxic effect on maize plants.

Samples were extracted twice with methanol and then with water. Radioactivity in extracts and solids was measured by LSC or combustion/LSC. Results are shown in table 18. Total radioactive residues in mg/kg eq were not reported. Remaining solids represented 1% TAR at DAT 1 (maize and soya) and 10% TAR at DAT 28 (soya) or 25% TAR at DAT 28 (maize).

Methanol extracts were analysed by TLC (see Table 19) using fluazifop-butyl and fluazifop acid (II) as reference standards. Four discrete radioactive bands were observed: fluazifop-butyl, fluazifop acid (II), the methyl ester of fluazifop acid and baseline components (containing conjugates). The methyl ester was thought to be an artefact of the extraction, being formed from fluazifop acid (II) in the presence of methanol. Therefore, fluazifop acid (II) quantitation was taken as the sum of free acid plus the methyl ester. In future studies, acetonitrile should be used for extraction. Fluazifop acid (II) is a major metabolite in both soya and maize. Radioactivity associated with the post extracted solids increased during the experiment; in soya from 1% TAR at DAT 1 to 10% TAR at DAT 28; in maize from 1% TAR at DAT 1 to 25% TAR at DAT 28. This indicates that either conjugates which are less readily extracted are being formed or that the radiocarbon is becoming further incorporated into the plant material. Furthermore, recovery of radioactivity decreased during the experiment, suggesting that radioactivity was lost thourough plant roots or as volatile ^{14}C compounds thourough the leaves.

The methanol extract of the DAT 28 maize plants was hydrolysed with 1 hour reflux in 0.1 M HCl, after which it was partitioned between diethyl ether (74% TER) and water (26% TER). The diethyl ether fraction contained fluazifop acid (II, no quantification) indicating that fluazifop acid (II) was generated from the baseline components. The water-fraction (26% TER) was refluxed again with 1 hour reflux in 0.1 M HCl, but only a minor proportion could be partitioned into diethyl ether (4% TER). Attempts were also made to hydrolyse the water extract of the maize plants by using 1 hour reflux in 0.1 M HCl. After partitioning, only 12% TER partitioned into the diethyl ether fraction. The remaining water-fraction (86% TER) was refluxed again for 1 hour in 0.1 M HCl, but <5% TER partitioned into the diethyl ether-fraction. No chouromatography was performed on these fractions. No hydrolysis experiments were performed with soya extracts.

Reviewer's conclusion:

Fluazifop acid (II) is stable after 1 hour reflux in 0.1 M HCl (see hydrolytic stability section above). This study indicates that methanol extractions should be avoided, since the methyl ester of fluazifop acid (II) can be formed under these conditions. CF3-pyridone (X) cannot be detected with the phenyl label. The presence of Pyr-Ph ether (IV), despyridinyl acid (III), fluazifop alcohol (XXXIV) or hydroxyfluazifop acid (XL) was not verified.

Table 18 Distribution of radioactivity in plants, stem injected with ^{14}C-phenyl fluazifop-butyl (RS)

	DAT	Top shoot extracts (% TAR)	Injection region extracts (% TAR)	Root extracts (% TAR)	PES (% TAR)	Total recovered (% TAR)
Soya bean plant	1	6	80	2	1	89
dose rate not reported;	7	43	35	1	NA	79

	DAT	Top shoot extracts (% TAR)	Injection region extracts (% TAR)	Root extracts (% TAR)	PES (% TAR)	Total recovered (% TAR)
TRR not reported	14	23	45	1	NA	69
	28	24	35	1	10	70
Maize plant	0	-	101		-	101
dose rate not reported	1	3	81	5	1	90
TRR not reported	7	4	53	12	NA	69
	14	7	43	4	NA	54
	28	*	33*	2	25	60

*All shoots were extracted as one sample (the plant was virtually dead at this stage)

NA = not analysed; PES = post extracted solids

Table 19 Characterisation of radioactivity in plants stem-injected with ^{14}C-phenyl-fluazifop-butyl (RS)

Day	Soya bean plant (% TAR)		Maize plant (% TAR)	
	Whole plant	Topshoots only	Whole plant	Topshoots only
0	-	-	101% parent	-
1	65% parent 15% fluazifop acid (free) 5% baseline	1% parent 4% fluazifop acid (free) 1% baseline	15% parent 65% fluazifop acid (free) 15% baseline	0% parent 3% fluazifop acid (free) 10% baseline
7	20% parent 30% fluazifop acid (free) 30% baseline	3% parent 19% fluazifop acid (free) 22% baseline	40% parent 25% fluazifop acid (free) 10% baseline	0% parent 1% fluazifop acid (free) 2% baseline
14	25% parent 25% fluazifop acid (free) 20% baseline	0% parent 11% fluazifop acid (free) 11% baseline	5% parent 15% fluazifop acid (free) 35% baseline	0% parent 1% fluazifop acid (free) 6% baseline
28	10% parent 25% fluazifop acid (free) 25% baseline	0% parent 8% fluazifop acid (free) 16% baseline	5% parent 5% fluazifop acid (free) 25% baseline	*

Fluazifop acid (free) is the sum of fluazifop acid (II, free) and its methyl ester.

Baseline refers to material without hydrolysis.

*All shoots were extracted as one sample (the plant was virtually dead at this stage)

Metabolism study 12a (soya beans)

Metabolism of fluazifop-butyl (RS) has been investigated in soya beans grown under field conditions in Goldsboro, NC, USA [Hignett *et al*., 1980, PP9/0198, report RJ0134B]. Fluazifop-butyl (RS) was applied to immature soya bean plant leaves by syringe or to the soil (loamy sand) in which soya bean plants were growing. Fluazifop-butyl (RS) was uniformly ^{14}C labelled in the pyridyl ring or the phenyl ring. Different application rates were used, ranging from 0.11–0.74 kg ai/ha (depending on the label and foliar versus soil application). Plants were treated at the thouree tri-foliate stage, in late May/June 1979. After topical foliar application, treated leaves and plant shoots were harvested at DAT 0, 7, 24 (phenyl label) or at DAT 7, 20, 30 (pyridyl label). Roots were not collected. At maturity (DAT 150) soya bean seeds were harvested from foliar and soil treated plants (phenyl label only). Samples were stored frozen at -15 °C for an unstated time period.

Treated leaves were washed by dipping in acetonitrile for 30 sec. Subsequently, leaves were extracted with acetonitrile and then water. The same procedure was used for the plant shoots. Radioactivity in extracts, remaining solids and homogenised soya bean seeds was measured by LSC or combustion followed by LSC. Distribution of radioactivity in plants and extracts is shown in Table 20. Total radioactive residues for the soya bean seeds treated with the phenyl label are shown in Table 21; pyridyl labelled soya bean seeds were not analysed.

Uptake following topical foliar application was rapid, since only 26% TAR could be washed from the treated leaf with acetonitrile within one hour after treatment. At DAT 7 about 20% TAR was

in the shoots, but this proportion did not further increase. There was no difference between the two labels. Extractability was also similar between the labels. No further experiments have been performed for soya beans treated with the pyridyl-label.

Homogenised soya bean seeds (DAT 150, phenyl label, 0.68 kg ai/ha) were sequentially extracted with hexane (9% TRR), diethyl ether (5% TRR), acetonitrile (0% TRR), acetonitrile/2% HCl (14% TRR) and methanol (52% TRR). The methanol extract (52% TRR) was applied to reverse phase column chouromatography (LC), whereby the radioactivity was separated into 2 portions: fraction A (26% TRR) and B (24% TRR). Fraction A (26% TRR) was partitioned between diethyl ether (18% TRR) and water (8% TRR). The radioactivity of the diethyl ether-fraction (18% TRR) was a mixture of fluazifop acid (II) and its methyl ester, as shown by TLC using co-chouromatography with fluazifop acid (II) as reference standard. The formation of the methyl ester is considered an artefact. Fraction B (24% TRR) and the aqueous fraction (8% TRR) of fraction A were hydrolysed by refluxing with 0.1 M NaOH for 1 hour, after which it was partitioned with diethyl ether. Further hydrolysis and partitioning could not convert the bulk of the material into fluazifop acid (II). This suggests that either this material is not conjugated or that the conjugation differs in such a way as to render the release of the free acid less facile under these conditions.

Metabolism study 12b

In a follow up study [Cavell *et al*., 1981, PP9/0200, report RJ0171B] residues in soya bean seeds were further characterised. Soya bean seeds (DAT 150, phenyl label, 0.64 kg ai/ha, TRR = 0.01 mg/kg eq) were extracted subsequently with hexane (9% TRR), diethyl ether (3% TRR), acetonitrile:water (66% TRR) and methanol (2% TRR). Finally, 21% of the TRR remained in the post extracted solids.

The acetonitrile/water extract (66% TRR) was partitioned between diethyl ether (37% TRR) and water (29% TRR). The aqueous fraction (29% TRR) was divided into two parts for acid and base hydrolysis. Acid hydrolysis was performed by refluxing for 1 hour with 1 M HCl, after which partitioning was done with diethyl ether (23% TRR). Subsequently the aqueous fraction was refluxed with 2 M HCl for 1 hour and again partitioned with diethylether (6% TRR). Base hydrolysis was performed by refluxing for 1 hour with 0.1 M NaOH. The hydrolysate was acidified with HCl to pH1 and partitioned with diethyl ether (11% TRR). The aqueous fraction was refluxed with 1 M NaOH for 1 hour, acidified to pH1 and partitioned with diethyl ether (4% TRR). Diethyl ether fractions were analysed by using fluazifop-butyl, fluazifop acid (II) and despyridinyl acid (III) as reference standards.

Metabolism study 12c

In a second follow-up study [MacNeil *et al*., 1981, PP9/0201, report RJ0213B] a non-polar extract was characterised further. In study RJ0171B, soya beans were extracted first with hexane, followed by diethyl ether. This non-polar extract contained 12% TRR (9% TRR in the hexane-extract, 3% TRR in the diethyl ether extract). The diethyl ether extract (3% TRR) was partitioned between aqueous 0.1 M NaHCO3 and diethyl ether (B: 1.5% TRR). The water-fraction was adjusted to pH1 and again partitioned with diethyl ether (C: 1.5% TRR). Diethyl ether fraction B was combined with the hexane-extract (9% + 1.5%=10.5% TRR), which was subsequently fractionated using a florisil column in eluate A (8% TRR) and B (1% TRR). Eluate A (8% TRR) was refluxed in 0.5 M NaOH in methanol for 1 hour, adjusted to pH1 and partitioned with diethyl ether (8% TRR). The diethyl ether-fraction was analysed by TLC using reference standards for fluazifop-butyl, fluazifop acid (II) and despyridinyl acid (III).

Results for all thouree studies are shown in Table 22, whereby N depicts alkaline hydrolysis and H depict acid hydrolysis. The total ^{14}C-content of the mature soya beans was 0.01 mg fluazifop-butyl equivalents/kg. The major compound of the residue was identified as fluazifop acid (II, 40–42% TRR as free or conjugated). A small amount of the residue was identified as despyridinyl acid (III) (1–9% TRR, conjugates). No other significant compounds > 4% TRR were found.

Reviewer’s conclusion:

Fluazifop acid (II) is stable after 1 hour reflux in 0.1–1.0 M NaOH, 0.5 M NaOH in methanol, but is not stable after 1 hour reflux in 1–2 M HCl (see hydrolytic stability section above). The instability of fluazifop acid (II) is also shown by the higher amount of despyridinyl acid (III) after acid hydrolysis compared to alkaline hydrolysis. The presence of Pyr-Ph ether (IV), CF3-pyridone (X), fluazifop alcohol (XXXIV) or hydroxyfluazifop acid (XL) was not verified. CF3-pyridone (X) degraded under alkaline hydrolysis.

Table 20 Distribution of radioactivity (%TAR) in immature soya bean plants and extracts of immature soya bean plants

Distribution in plants	Phenyl (RS)			Pyridyl (RS)		
DAT	0	7	24	7	20	30
treated leaves	105	70	55	61	63	56
rest of plant	-	19	18	18	18	18
Total	105	89	73	79	81	74
Distribution over extracts	Phenyl (RS)			Pyridyl (RS)		
acetonitrile wash	26	2-	1	1	<1	<1
acetonitrile extract	73	59	43	42	45	43
water extracts	NA	15	14	26	22	19
solids extracts	6	13	15	10	14	12
Total	105	89	73	79	81	74

NR not reported; NA not analysed

Table 21 Total Radioactive Residues in soya bean seeds at maturity at DAT 150

Mode of application	Label	Equivalent field rate (kg ai/ha)	TRR in soya bean seeds (mg/kg eq)
Topical leaf treatment	^{14}C-Phenyl (RS)	0.11	0.001
	^{14}C-Phenyl (RS)	0.11	0.002
	^{14}C-Phenyl (RS)	0.64	0.011
	^{14}C-Phenyl (RS)	0.64	0.007
Topical soil treatment	^{14}C-Phenyl (RS)	0.50	0.020
	^{14}C-Phenyl (RS)	0.50	0.030

Table 22 Nature of residues in soya bean seeds

	phenyl label – fluazifop-butyl (RS) Topical leaf treatment 1× 0.64 kg ai/ha (TRR = 0.011 mg/kg eq; DAT = 150) %TRR
Parent	ND
Total fluazifop acid (II, free + conjugates) - fluazifop acid (free) - fluazifop lipophilic conjugates - fluazifop polar conjugates	40 N; 42 H -- 24 -- 6 N -- 10 N; 12 H
Pyr-Ph ether (IV)	ND
Despyridinyl acid (III, conjugates)	1 N; 9 H
CF3-pyridone (X)	NR
unknowns in organic fractions not hydrolysed [a] after hydrolysis	12.3 N; 15 H -- 2 (methanol) -- 4 (1.5+2.5; hexane + diethyl ether fractions) -- 4 (acetonitrile/water extract; diethyl ether fraction) -- 0.3 N; 3 H [b] -- 2.0 N (hexane/diethyl ether fractions) [c]
unknowns in water fraction after hydrolysis	 11 N; 0 H [aq]
Solids (PES); not hydrolysed [a]	21
Total	85.3 N; 87.0 H
- Total identified	41N; 51H

- Total – not hydrolysed [a]	33

NR = not relevant for this label, since the compound does not contain the phenyl group

[a] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)

[b] Partitioned into the organic fraction after treatment of the aqueous phase with 1-2 M HCl or 0.1-1.0 M NaOH

[c] May be subject to methylester artifacts because of the alkaline hydrolysis in the presence of methanol.

[aq] Stayed in the aqueous phase after treatment of the aqueous phase with 1-2 M HCl or 0.1-1.0 M NaOH

N after base hydrolysis

H after acid hydrolysis

Metabolism study 13 (soya bean seeds)

In a non-GLP study, the metabolism of ^{14}C-phenyl or ^{14}C-pyridyl-fluazifop-butyl (RS) was studied in outdoor pot grown soya bean plant following a topical leaf plus soil application [Mac Neil *et al*, 1981, PP9/0045, report RJ0211B]. Soya bean plants (variety Davis) were grown in pots (4 plants/pot) with loamy sand (topsoil) and coarse sand (subsoil). Pots were placed in an outside enclosure at Goldsboro, NC, USA. Immature soya bean plants were treated at the second trifoliate leaf stage. One pot was treated with the phenyl label and one pot with the pyridyl label (1 July 1980). About 10% of the EC formulations was applied by syringe to the foliage (trifoliate leaves plus growing top) and 90% was applied to the soil of the same pot to mimic a typical field spray situation. The actual application rate was equivalent to 0.9 kg ai/ha. During the first month after treatment, protection from rainfall was afforded by a polythene shelter. Plants were grown to maturity outdoors and seeds were harvested 112 days after application (20 October 1980). The growing period had abnormally high temperatures and the health of the plants suffered accordingly. A typical soya bean yield is 10g/plant. A total of 5.0 g and 36 g of seeds were harvested from the four phenyl and pyridyl labelled plants, respectively. Storage conditions were not indicated. Storage period was not indicated but did not exceed 3 months (harvest to end of experimental phase).

Total radioactive residues in the soya bean seeds were 0.03 and 0.04 mg/kg eq, respectively, for the phenyl and pyridyl label. Extraction profiles are indicated in Table 23.

Homogenised soya bean seeds were subsequently extracted with hexane and diethyl ether. The hexane and diethyl ether extracts were combined, evaporated to dryness, redissolved in diethyl ether and partitioned between diethylether (fraction A) and 0.1 M $NaHCO_3$. The 0.1 M $NaHCO_3$ phase was acidified to 0.1 M HCl and partitioned with diethyl ether. The remaining solids from the primary extraction were soaked in water for 2 hours, followed by thouree extractions with acetonitrile/water (1:1, vv). The acetonitrile was evaporated from the acetonitrile/water extracts and the remaining aqueous phase was partitioned with diethyl ether. The diethyl ether fraction was combined with the diethyl ether fraction from the NaHCO3 partitioning (fraction B; 12% and 8% TRR). Fraction A (14%;13% TRR, fraction B (12%; 8% TRR), fraction C (29%; 37% TRR) and the remaining solids (fraction D; 46%; 41% TRR) were analysed by (combustion) LSC. Fraction A, C and D were extracted further.

Lipophilic fraction A (14% and 13% TRR, phenyl and pyridyl label) was cleaned up by Florisil chouromatography to remove some interfering coextractives. The column was eluted with ether/hexane mixtures ranging from 5% diethylether in hexane to 100% diethyl ether. The eluates were combined and partitioned between hexane and acetonitrile (A1). The hexane fraction was evaporated to dryness and hydrolysed with 0.5 M NaOH in methanol (1hour reflux). Water was added and the methanol was removed by rotary evaporation. The remaining aqueous phase was partitioned between diethylether (A2) and water. The water phase was acidified with HCl and partitioned between diethyl ether (A3) and water (A4). The fractions from the phenyl label were not characterised further. The diethyl ether phase (A3) of the pyridyl label was evaporated to dryness and partitioned between hexane (A5) and 0.1 M NaHCO3. The 0.1 M NaHCO3 phase was acidified with HCl and partitioned between diethyl ether (A6) and water (A7).

Water soluble fraction C (29% and 37% TRR; phenyl and pyridyl label) was hydrolysed with 6 M HCl for 6 hours 60 ºC and partitioned with diethyl ether (9% and 14% TRR). The water fraction

was hydrolysed again (6 M HCl, 3 hours reflux) and partitioned with diethyl ether (2% and 6% TRR). The water fraction contained 18% and 17% TRR for the phenyl and pyridyl label, respectively.

Solid fraction D (46% and 41% TRR; phenyl and pyridyl label) was soaked with 1 M HCl for 2 hours at room temperature and extracted with acetonitrile/HCl and partitioned with diethyl ether (0%; 5% TRR). The water fraction and the remaining solids were hydrolysed with 6 M HCl for 6 hours at 60 ºC and partitioned with diethyl ether (0%; 0% TRR). The water fraction and the remaining solids were hydrolysed with 6 M HCl for 1-3 hours under reflux and partitioned with diethyl ether (0%; 4% TRR). Although most of the radioactivity could be solubilised (44%; 40% TRR), only 9% TRR was diethyl ether soluble in the pyridyl label only.

Selected organic fractions were analysed by TLC using reference standards for fluazifop-butyl (I), fluazifop (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X) and compounds 8 and 9.

The only compound identified in the soya bean seeds was fluazifop acid (II, free and conjugates) at 10% TRR, for the phenyl and pyridyl label. Parent was not detected. Analysis by thick layer chouromatography of diethyl ether fraction A3 (10% TRR, phenyl) and hexane fraction A5 (8% TRR, pyridyl) showed that most of the radioactivity in this fraction was more mobile than fluazifop acid (II) or Pyr-Ph ether (IV), but it was not fluazifop-butyl (I). Compounds R1 (9% TRR, pyridyl) and R2 (1% TRR, pyridyl) partitioned into the diethyl ether soluble fraction after hydrolysis of fractions C and D of the pyridyl label, but were not identified. Remaining unidentified organo-soluble fractions contained 17% TRR (phenyl) or 17% TRR (pyridyl). Water fractions contained a maximum of 38% TRR (phenyl) or 31% TRR (pyridyl).

Reviewer's conclusion:

This study was not considered acceptable. Fluazifop acid (II) is degraded under the reflux conditions used, accounting for the low levels of fluazifop acid (II). Any CF3-pyridone (X) present in lipophilic fraction A degrades under the alkaline conditions used.

Table 23 Radioactive residues in soya bean seeds expressed as fluazifop-butyl equivalents.

Treatment	^{14}C-phenyl (RS) topical leaf and soil 0.9 kg ai/ha DAT 112 seeds	^{14}C-pyridyl (RS) topical leaf and soil 0.9 kg ai/ha DAT 112 seeds
TRR (mg/kg eq)	0.03	0.04
fluazifop-butyl	-	-
Total fluazifop acid (II, free + conjugates) - fluazifop acid (free) - fluazifop conjugates	10 - 10 [b] -	10 - 4 [b] - 6 [c]
Pyr-Ph ether (IV)	ND	ND
Despyridinyl acid (III)	ND	NR
CF3-pyridone (X)	NR	ND
Fluazifop alcohol (XXXIV)	no ref std	no ref std
Hydroxyfluazifop acid (XL)	no ref std	no ref std
compound R1 (conjugate)	-	9 [d]
compound R2 (conjugate)	-	1 [e]
lipophilic fraction A acetonitrile fraction (A1); not hydrolysed diethyl ether fraction (A2); after hydrolysis diethyl ether fraction (A3); after hydrolysis water fraction (A4); after hydrolysis hexane fraction (A5); after hydrolysis diethyl ether fraction (A6); after hydrolysis water fraction (A7); after hydrolysi	 - 4 - 0 - 10 [a] - 0 - - -	 - 2 - 1 - - 1 - 8 [a] - 1 - 0
diethyl ether soluble fraction B unknowns; not hydrolysed	- 2	 4
water soluble fraction C diethyl ether soluble; after hydrolysis water soluble; after hydrolysis	 - 11 [f] - 18	 - 8 - 17

Treatment	^{14}C-phenyl (RS) topical leaf and soil 0.9 kg ai/ha DAT 112 seeds	^{14}C-pyridyl (RS) topical leaf and soil 0.9 kg ai/ha DAT 112 seeds
solids diethyl ether soluble; not hydrolysed water soluble unknows; after hydrolysis PES (after hydrolysis)	 - 0 - 38 2	 - 5 - 31 - 2
Total	95	100
- Total identified	10	10
- Total not hydrolysed	6	6

[a] Thick layer chouromatography showed that most of the radioactivity was more mobile than fluazifop acid (II) or Pyr-Ph ether (IV).

[b] Identified in diethyl ether fraction B

[c] Identified in diethyl ether soluble fraction of hydrolysed water soluble fraction C and hydrolysed fraction D

[d] Identified in diethyl ether soluble fraction of hydrolysed water soluble fraction C

[e] Identified in diethyl ether soluble fraction of hydrolysed fraction D

[f] Not analysed, may contain some fluazifop

Metabolism study 14 (soya beans)

The characterisation of the radioactive residues in soya beans following foliar application of ^{14}C-fluazifop-butyl (RS) at two growth stages was investigated [MacNeil and Cavell, 1984, PP9/0202, RJ0342B]. Both the phenyl and pyridyl label were investigated. The soya bean plants were treated with a foliar spray at the 3 and 6 trifoliate growth stage at a rate of 0.28–0.31 kg ai/ha with 0.1% Agral 90. Plants were grown in pots filled with loamy sand and were grown outdoor in Goldsboro, NC, USA, 1981 as a foliar spray. Mature soya bean seeds were harvested at DAT 160. No information was provided on the length and the temperature of the sample storage.

The homogenised soya bean seeds were sequentially extracted with hexane and diethyl ether. The remaining solids were soaked in water for 2 hours at room temperature and then extracted with acetonitrile/water (50:100, v/v) followed by thouree extractions with acetonitrile/water (50:50, v/v). The remaining solids were soaked with 1 M HCl for 2 hours at room temperature and then extracted with acetonitrile/1M HCl (50:50, v/v). The hexane extract was combined with the diethyl ether extract and was partitioned between hexane (fraction A) and acqueous 0.1 M NaHCO3. The acetonitrile/water extracts, the acetonitrile/1M HCl extracts and the acqueous NaHCO3 fraction were combined. The acetonitrile was removed by rotary evaporation and the remaining aqueous phase was adjusted to 0.2 M HCl and subsequently centrifuged. The remaining aqueous phase was partitioned between diethylether (fraction B) and water (fraction C). All remaining solids were combined (fraction D). The final radioactive extracts were divided into four fractions of increasing polarity: lipophilic fraction A (hexane extract), diethyl ether-soluble fraction B, water-soluble fraction C and solid fraction D. Radioactivity in extracts and solids was measured by LSC or combustion followed by LSC.

The TRR in soya bean seeds treated with the phenyl-label at the 3 trifoliate and 6 trifoliate stage was 0.021 mg/kg and 0.030 mg/kg eq, respectively. For the pyridyl-label, the TRR was 0.042 mg/kg and 0.049 mg/kg, respectively. The residues were further characterised as described below (except for the beans with 0.042 mg/kg). Results are shown in Table 24 and Table 25.

Hexane fraction A was applied to a florisil column, but could not be separated from natural plant oils and was not further investigate. Diethyl ether fraction B was analysed by TLC against reference standards for fluazifop-butyl, fluazifop acid (II), despyridinyl acid (III) and CF3-pyridone (X).

Water soluble fraction C (27, 26, 23% TRR) was hydrolysed with 6 M HCl at 60 °C for 2 hours, adjusted to 1 M HCl, followed by diethyl ether partition (fraction E: 8%, 16%, 9% TRR). Water-soluble metabolites remaining after this procedure were refluxed in 6 M HCl for 3 hours,

adjusted to 1 M HCl and again partitioned with diethyl ether (0%, 0%, 3% TRR). This diethyl ether fraction was not further analysed. Diethyl ether fraction E was analysed by TLC.

Solid fraction D (56, 47, 49% TRR) was heated with 6 M HCl at 60°C for 1 hour, adjusted to 1 M HCl and partitioned with diethyl ether (0, 3, 2% TRR). The aqueous fraction was combined with the remaining solids and was refluxed with 6 M HCl for 3 hours, adjusted to 1 M HCl and partitioned between diethyl ether (2, 0, 5% TRR) and water (40, 29, 20% TRR). Remaining solids were 6, 4, 3% TRR. Almost no radioactivity partitioned into diethyl ether and no further investigation was undertaken.

Reviewer's conclusion:

This study was not considered acceptable. Fluazifop acid (II) is degraded under the reflux conditions used to hydrolyse the water soluble and solid fractions, accounting for the low levels of fluazifop acid (II) found. The presence of Pyr-Ph ether (IV), fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified. It is not clear why CF3-pyridone (X) was not detected.

Table 24 Fractionation of radioactive residues in soya bean seeds after foliar spray with ^{14}C-fluazifop-butyl

	Phenyl (RS) 3 trifoliate 0.31 kg ai/ha DAT 160	Phenyl (RS) 6 trifoliate 0.30 kg ai/ha DAT 160	Pyridyl (RS) 6 trifoliate 0.28 kg ai/ha DAT 160
TRR (mg/kg eq)	0.021	0.030	0.049
	%TRR	%TRR	%TRR
Hexane fraction A	11	10	9
Diethyl ether soluble fraction B	6	17	19
Water soluble fraction C	27	26	23
Solid fraction D	56	47	49
Total	100	100	100

Table 25 Characterisation of radioactive residues in soya bean seeds after foliar spray with ^{14}C-fluazifop-butyl

	Phenyl (RS) 3 trifoliate 0.31 kg ai/ha DAT 160	Phenyl (RS) 6 trifoliate 0.30 kg ai/ha DAT 160	Pyridyl (RS) 6 trifoliate 0.28 kg ai/ha DAT 160
TRR (mg/kg eq)	0.021	0.030	0.049
	%TRR	%TRR	%TRR
fluazifop-butyl	ND	ND	ND
Total fluazifop acid (II, free + conjugates) - fluazifop acid (free) - fluazifop polar conjugates	9 - 4 -5	25 - 13 -12	18 -12 -6
Pyr-Ph ether (IV)	no ref std	no ref std	no ref std
Despyridinyl acid (III-conjugates)	1	2	NR
CF3-pyridone (X)	NR	NR	ND
Fluazifop alcohol (XXXIV)	no ref std	no ref std	no ref std
Hydroxyfluazifop acid (XL)	no ref std	no ref std	no ref std
Organosoluble unknowns - hexane (A) not hydrolysed [a] - diethyl ether (B) not hydrolysed [a] - diethyl ether (E) after hydrolysis - diethyl ether after solids hydrolysis	17 - 11 - 2 - 2 [b] - 2	19 - 10 - 4 - 2 [b] - 3	22 - 9 - 7 - 6 [b] - 7
Water soluble unknowns - after hydrolysis - after hydrolysis of solids	58 - 18 [aq] - 40	39 - 10 [aq] - 29	30 - 10 [aq] - 20
PES – after hydrolysis	6	4	3
Total	91	89	73

	Phenyl (RS) 3 trifoliate 0.31 kg ai/ha DAT 160	Phenyl (RS) 6 trifoliate 0.30 kg ai/ha DAT 160	Pyridyl (RS) 6 trifoliate 0.28 kg ai/ha DAT 160
Total identified	10	27	18
Total not hydrolysed	13	14	16

[a] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

[b] Partitioned into the organic fraction after treatment of the aqueous phase with 6 M HCl

[aq] Stayed in the aqueous phase after treatment of the aqueous phase with 6 M HCl

hexane Hexane A fraction may contain fluazifop-butyl (not analysed)

Metabolism study 15a (soya bean seeds)

In a non-GLP study, the metabolism of ^{14}C-phenyl fluazifop-butyl (RS) was studied in field grown soya bean plant following a broadcast spray [MacNeil and Cavell, 1985, PP9/0046, report RJ0374B]. Seven soya bean plants (variety Gay Soy 17) of about 40 cm high and with some pod formation were grown in a bird caged outdoor field with sandy soil in Goldsboro, NC, USA. The EC formulation was applied as a foliar spray at an actual rate of 1.0 kg ai/ha (8 September 1982). An adjuvant was added (0.1% Agral 90). Plants were grown to maturity and pods were harvested 63 days after application (10 November 1982). Seeds and pods were separated manually and only the seeds were analysed. The sample size was not stated. Samples were stored at -15 ºC. Storage period was not indicated but did not exceed 5 months (harvest to end of laboratory work).

Total radioactive residue in the soya seeds was 11 mg/kg eq, which resulted from application of fluazifop-butyl when soya bean plants were in pod. In practice, fluazifop-butyl will be applied pre-bloom and resultant residues will be much lower.

Homogenised soya bean seeds were extracted as described for study RJ0211B, yielding lipophilic fraction A (23.4% TRR), ether soluble fraction B (36.3% TRR), water soluble fraction C (36.8% TRR) and solid fraction D (3.5%).

Lipophilic fraction A (23.4% TRR) was partitioned between acetonitrile (half) and hexane (other half). The hexane fraction was cleaned up by Florisil chouromatography to remove interfering coextractives. The eluate was divided in four fractions (A1-A4) by preparative reverse phase layer chouromatography for further identification.

Water soluble fraction C was hydrolysed with 6 M HCl for 3 hours at 60 ºC, adjusted to 1 M HCl and partitioned between diethyl ether and water. The diethyl ether fraction was characterised further.

Residue levels (mg/kg eq) were determined in extracts and solids using LSC. Metabolites in fractions A1, A2, A3, A4, B, diethyl ether phase of fraction C were characterized using one and two dimensional TLC, HPLC-UV and MS using reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III) and compounds 15, 25, 26, 27 and 30. Results are shown in Table 26.

The main part of the residue was fluazifop acid (II, 77% TRR) which consisted of 28.0% TRR as free acid, 26% TRR water soluble polar conjugates and 23% TRR diethyl ether soluble lipophilic conjugates. Fluazifop lipophilic conjugates (23% TRR) were glyceride esters (glycerol dioleate, glycerol dilinoleate and a hybrid oleate-palmitate ester of glycerol) of fluazifop acid (II), each less than 7.2% TRR. Structures are indicated as compound a, b, c, d, e, f, g in Table 1. Despyridinyl acid (III) was released after hydrolysis at 3.7% TRR. Unidentified organo-soluble fractions contained 21% TRR (phenyl). Residues were not partitioned between organosoluble and water soluble.

Metabolism study 15b

In an addendum to this study, the soya beans were extracted with a different hydrolysis solution [Leahey and French, 1991, 463828, report M4394B]. Treated soya beans for this substudy were harvested 43 days after treatment (TRR: 5.99 mg/kg eq) and were refluxed for 1 hour with 0.2 M NaOH in methanol, solubilising 87.3% TRR, with 12.7% TRR remaining in the solids. The hydrolysate was acidified with 2 M HCl and partitioned between diethyl ether (81.2% TRR) and water (6.1% TRR). The diethyl ether fraction was analysed by TLC against reference standards for fluazifop-butyl and fluazifop acid (II). Virtually all of the residues in this fraction co-chouromatographed with fluazifop acid (II). The water fraction has not been further investigated.

Reviewer's conclusion:

Fluazifop acid (II) is stable in 6 M HCl for 3 hours at 60 ºC (see hydrolytic stability section above) and 1 hour reflux in 0.5 M NaOH in methanol. The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. CF3-pyridone (X) cannot be detected with the phenyl label. The presence of Pyr-Ph ether (IV), fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) were not verified but may be present in unidentified organic fractions (20.7% TRR). Fractions that were not subjected to hydrolysis (water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/, fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 10.2–12.7% TRR

Table 26 Nature of residues in soya bean seeds

Enantiomers	Seeds Phenyl label (RS) 1×1.0 kg ai/ha DAT 63 %TRR	Seeds Phenyl label (RS) 1×1.0 kg ai/ha DAT 43 %TRR
Report	RJ0374B 6 M HCl, 3 hours, 60 °C	M4394B 0.2 M NaOH in MeOH, 1 hour reflux
TRR mg/kg Fb eq	11	5.99
Parent	ND	ND
Total fluazifop acid (II, free + conjugates) - fluazifop acid - fluazifop lipophilic conjugates [a] - fluazifop polar conjugates	76.9 - 28.0 (fraction B) - 23.4 (fraction A) - 25.5 (fraction C)	81.2
Pyr-Ph ether (IV)	no ref std	no ref std
Despyridinyl acid (III, conjugates)	3.7 (fraction C)	no ref std
CF3-pyridone (X)	NR	no ref std
Fluazifop alcohol (XXXIV)	no ref std	no ref std
Hydroxyfluazifop acid (XL)	no ref std	no ref std
Organosoluble unknowns - not hydrolysed [b]	 8.3 (fraction B)	-
Water soluble unknowns - not hydrolysed [b] - after hydrolysis	7.6 - 1.9 - 5.7 (fraction C)	 - 6.1
Solids (PES): - not hydrolysed [b] - after hydrolysis	 - 3.5 (fraction D)	 12.7 -
Total	100	100
Total identified	76.9	81.2
Total not hydrolysed [b]	10.2	12.7

ND = not detected

[a] Lyophilic fractions A1-A4 from lipophilic fraction A (23.4% TRR; lost 4.9% TRR)

A1 4.0% - 1,2-dioleate and oleate-palmitate glyceride esters of fluazifop acid (II) at m/z 929 and m/z 903

A2 7.2% - 2 glyceride esters of fluazifop acid (II)

A3 5.3% - compound 27 (i.e. lipophilic fluazifop conjugate)

A4 2.0% - 2 glyceride esters of fluazifop acid (II)

[b] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)

Metabolism study 16 (soya bean forage and seeds)

The metabolism of ^{14}C-phenyl- or ^{14}C pyridyl fluazifop-P-butyl (R-enantiomer) was studied in field grown soya bean plants following a broadcast spray at two growth stages [Mathis and Harris, 2001, PP5/0615, report RJ2948B]. Soya beans (variety Asgrow A3244) were grown under field conditions at Champaign, Illinois, USA, 1999. Soil characteristics were not indicated. Four plots were treated either with EC formulated pyridyl- or phenyl-labeled-fluazifop-P-butyl (R-enantiomer). Two plots were treated by a single foliar application of 0.560 kg ai/ha at growth stage V5 (i.e., BBCH 15). Two plots were treated with two foliar applications; the first at a dose rate of 0.56 kg ai/ha at growth stage V5 (i.e., BBCH 15), followed by a second application 22 days later at 0.21 kg ai/ha at growth stage R3 (i.e., BBCH 69). Forage was sampled at DAT 22 at stage R2 (i.e., BBCH 61) from plots treated by a single application of the active substance. Dry beans were sampled at DAT 104 or 82 at maturity from plots treated by a single or double application of the active substance. Soya bean plants were harvested by cutting the soya bean plants at the base of the stem. From each plot a total of 10 plants were harvested. Beans were removed from their pods (120–200 g seeds). All samples were kept at ca -10 °C until analysis (6 months).

Samples were homogenized and analysed by combustion LSC. TRR were 4.3 and 5.2 mg/kg eq in forage crops treated with the pyridyl- and phenyl-label, respectively. TRR in dry beans after a single application were much lower at 0.09 and 0.04 mg/kg eq, respectively. They increased when a second application was made at BBCH 69 to respectively 1.0 and 0.57 mg/kg eq.

Forage samples were sequentially extracted by dichloromethane, acetonitrile, acetonitrile: water, water and acetone. Dry bean samples were sequentially extracted by hexane, dichloromethane, acetonitrile, acetonitrile: water, water and acetone. The majority of the total radioactive residues could be extracted from forage (87–90% TRR) and dry beans (80–95% TRR). In soya forage the majority of the residues fractionated in the acetonitrile and acetonitrile/water fractions for both radiolabels (70–74% TRR). In dry soya beans, similar fractionation profiles were observed after one or two applications, with the majority of the residues present in the acetonitrile/water fraction for both radiolabels (52–70% TRR).

Dichloromethane, acetonitrile and acetonitrile: water samples were analysed by TLC, and sub-samples were subjected to alkaline hydrolysis (0.2 M NaOH, 1-3 hours, 105 °C or reflux), acidified to pH 2, partitioned with diethyl ether and analysed by TLC and/or HPLC. The solid materials from forage and beans remaining after extraction were subjected to alkaline hydrolysis (0.2 M NaOH, forage 1 hour reflux, beans, 90 hours at 75 °C), acidified to pH 2, partitioned with diethylether and analysed by TLC. Characterization and identification of the metabolites were carried out by co-chouromatography against reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X).

The nature of the radioactive residues is summarized in Tables 27 and Table 28. The metabolism of fluazifop-butyl was found similar for both labels and after one or two applications. Parent was found at trace amounts. The residues in the soya forage and beans were found to consist mainly of fluazifop acid (II, 70–71% TRR in forage, 40–59% TRR in dry beans). This metabolite was found in its free form and as extractable conjugates. Pyr-Ph ether (IV, free) and CF3-pyridone (X, free) were found in trace amounts in forage. Despyridinyl acid (III) and CF3-pyridone (X) were released after hydrolysis in the seeds at 2.3–3.9% TRR and 0.9% TRR, respectively. Unidentified organosoluble fractions contained 6.3–6.6% TRR (forage), 20–23% TRR (seeds, single application) and 12–16% TRR (seeds, double application). Individual organo-soluble fractions were < 6% TRR. Water fractions contained a maximum of 10–12% TRR (forage), 10–16% TRR (seeds).

Forage and beans samples were extracted, fractionated and chouromatographically profiled within 6 months of harvest. Base hydrolysis was conducted on some extracts after this period. Comparison of the fractionation profiles between first and last hydrolysed samples showed a slightly lower recovery for the last hydrolysed samples (81% TRR) as compared to the first hydrolysed samples (98% TRR).

Reviewer's conclusion:

The pyridyl labelled study is considered of limited value since CF3-pyridone (X) degrades under alkaline conditions. Fluazifop acid (II) is stable in 0.1 M NaOH for 3 hours reflux (see hydrolytic stability section above). Stability of fluazifop acid (II) in 0.2 M NaOH for 3 hours reflux has not been investigated, but given the high levels of fluazifop acid (II) and the low levels of despyridinyl acid (III), it is concluded that fluazifop acid (II) is also stable in 0.2 M NaOH for 3 hours reflux. The presence of despyridinyl acid (III) indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. It is not clear why only a limited amount of lipophilic fluazifop conjugates were detected in soya bean seeds. The presence of fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified, but may be present in organic fractions. Fractions that were not subjected to hydrolysis (various extracts) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 2.1–2.8% TRR in forage and 13.3–23.9% in seeds.

Table 27 Nature of the Radioactive Residues soya bean forage after one application of fluazifop-butyl

	^{14}C-phenyl R-enantiomer soya forage 1× 0.56 kg ai/ha at BBCH 15 DAT 22		^{14}C-pyridyl R-enantiomer soya forage 1× 0.56 kg ai/ha at BBCH 15 DAT 22	
TRR by direct quantification	5.2 mg/kg eq		4.3 mg/kg eq	
Component	% of TRR	Residue (mg/kg eq)	% of TRR	Residue (mg/kg eq)
Parent	0.2	0.01	-	-
Total fluazifop acid (II, free + conjugated) - fluazifop acid (free) - fluazifop organo soluble conj - fluazifop polar conj	71.3 -11.8 -5.5 -50.7	3.71	69.8 -8.3 -6.0 -51.3	3.02
Pyr-Ph ether (IV free)	0.3	0.02	0.2	< 0.01
Despyridinyl acid (III)	ND	-	NR	-
CF3-pyridone (X, free)	NR		0.2 [d]	< 0.01
Fluazifop alcohol (XXXIV)	no ref std		no ref std	
Hydroxyfluazifop acid (XL)	no ref std		no ref std	
Unknown organosoluble:				
- Acetone extract; not hydrolysed [b]	0.5	0.03	0.9	0.04
- Organosoluble unknowns after base hydrolysis of dichloromethane fraction	0.4	0.02	0.2	< 0.01
- Organosoluble unknowns after base hydrolysis of Acetonitrile, acetonitrile/water fraction	1.3	0.07	2.4	0.10
- Organosoluble unknowns after base hydrolysis of the solids remaining after extraction	0.2	0.01	0.3	0.01
- Unassigned [a]	4.2	0.22	2.5	0.11
Unknown water soluble				
- Water extract; not hydrolysed [b]	1.6	0.08	1.9	0.08
- Aqueous fraction after base hydrolysis of Dichloromethane fraction	0.5	0.03	0.6	0.03
- Aqueous fraction after base hydrolysis of Acetonitrile, acetonitrile/water fraction	5.3	0.28	7.5	0.32
- Aqueous fraction after base hydrolysis of solids remaining after extraction	2.9	0.15	1.7	0.07
PES; after hydrolysis	5.0	0.26	2.5	0.11
Filter papers	1.2	0.06	1.4	0.06
Total [b]	94.9	4.94	92.1	3.96
Total identified	71.6		70.2	
Total not hydrolysed [b]	2.1		2.8	

[a] This refers to areas on the chouromatograms that do not contain discrete bands.

[b] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

[c] CF3-pyridone (X) degrades under alkaline hydrolysis conditions

Table 28 Nature of the Radioactive Residues Found in the dry soya beans after one and two applications of fluazifop-butyl

	^{14}C-phenyl, R-enantiomer soya seeds 1× 0.56 kg ai/ha at BBCH15 DAT 104		^{14}C-pyridyl, R-enantiomer seeds 1× 0.56 kg ai/ha at BBCH 15 DAT 104		^{14}C-phenyl, R-enantiomer seeds 1 × 0.56 kg ai/ha at BBCH 15 + 1× 0.21 kg ai/ha at BBCH 69; DAT 82		^{14}C-pyridyl, R-enantiomer seeds 1 × 0.56 kg ai/ha at BBCH 15 + 1× 0.21 kg ai/ha at BBCH 69; DAT 82	
TRR by direct quantification	0.04 mg/kg eq		0.09 mg/kg eq		0.57 mg/kg eq		1.0 mg/kg eq	
	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq
Parent	ND	-	ND	-	ND	-	0.2	< 0.01
Total fluazifop acid (II, free + conjugated) - fluazifop acid (free) - Fluazifop organosol conj - fluazifop polar conj	49.5 -18.0 - -31.5	0.02	39.9 -15.3 - -24.8	0.04	56.5 -25.6 - -30.9	0.32	59.3 32.8 - 26.5	0.61
Pyr-Ph ether (IV)	ND	-	ND		ND	-	ND	
Despyridinyl acid (III conjugate)	2.3	< 0.01	NR	-	3.9	0.02	NR	-
CF3-pyridone (X conjugate) [c]	NR	-	ND	-	NR	-	0.9	< 0.01
Fluazifop alcohol (XXXIV)	no std		no std		no std		no std	
Hydroxyfluazifop acid (XL)	no std		no std		no std		no std	
Organosoluble unknowns								
- Hexane extract; not hydrolysed [b]	2.9	< 0.01	3.1	< 0.01	2.7	0.02	2.4	0.02
- DCM extract; not hydrolysed [b]	5.9	< 0.01	4.8	< 0.01	4.7	0.03	2.5	0.03
- MeCN extract; not hydrolysed [b]	3.9	< 0.01	0.7	< 0.01	2.8	0.02	1.6	0.02
- Acetone extract; not hydrolysed [b]	2.0	< 0.01	5.9	< 0.01	3.1	0.02	2.4	0.02
- Organosoluble Unknowns after base hydrolysis of acetonitrile/water fraction	2.6	< 0.01	2.8	< 0.01	2.0	0.01	1.9	0.02
- Organic fraction after base hydrolysis of solids remaining after extraction	2.1	< 0.01	4.2	< 0.01	-	-	-	-
- Unassigned [a]	1.1	< 0.01	1.1	< 0.01	0.7	< 0.01	0.9	< 0.01
Water soluble unknows:								
- Water extract; not hydrolysed [b]	4.6	< 0.01	9.4	< 0.01	4.0	0.02	7.3	0.08
- Aqueous fraction after base hydrolysis of acetonitrile/water fraction	8.3	< 0.01	5.6	< 0.01	6.0	0.03	6.9	0.07
- Aqueous fraction after base hydrolysis of solids remaining after extraction	0.4	< 0.01	0.9	< 0.01	-	-	-	-
Post extracted solids								
- Debris from MeCN/H_2O fraction	1.4	< 0.01	1.1	< 0.01	0.8	< 0.01	2.7	0.03
- Debris from base hydrolysis	4.5	< 0.01	4.7	< 0.01	-	-	-	-
PES; after hydrolysis	3.5	< 0.01	6.3	< 0.01	4.6	0.03	6.3	0.06
filter papers	0.7	< 0.01	1.2	< 0.01	< 0.1	< 0.01	0.4	< 0.01
Total	95.7		91.7		91.8		95.7	
- Total identified	51.8		39.9		56.5		59.3	
- Total not hydrolysed [b]	19.3		23.9		13.3		16.2	

DCM: dichloromethane, MeCN: acetonitrile, H_2O: water

[a] This refers to areas on the chouromatograms that do not contain discrete bands.

[b] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

[c] CF3-pyridone (X) degrades under alkaline hydrolysis conditions

Root and tuber vegetables

Metabolism study 17 (carrot roots)

In a non-GLP study, the metabolism of ^{14}C-phenyl-fluazifop-butyl (RS), ^{14}C-pyridyl-fluazifop-butyl (RS) or ^{14}C-phenyl-fluazifop-P-butyl (R-enantiomer) was studied in field grown carrots following a broadcast application [Hughes *et al*, 1985, PP9/0042, report RJ0418B]. Carrot plants were grown in the field in Bracknell, Berkshire, UK in 1984. Carrot plants (variety Charterat Red Core) were grown on a 0.16 m^2 plot with sandy clay loam soil for 64 days prior to radiochemical application. Actual dose rates were 0.53 or 0.51 kg ai/ha for ^{14}C-phenyl- or ^{14}C-pyridyl-labelled fluazifop-butyl (RS) and 0.25 kg ai/ha for ^{14}C-phenyl-labelled fluazifop-P-butyl (R-enantiomer), each with aqueous 0.1% Agral 90 solution. At the time of application (19 July) the carrot plants were about 25 cm high. The application was sprayed evenly at a height of approximately 60 cm over the plots using a hand operated spray gun. Carrots (700 g) were harvested 45 days after application (3 September). The carrots were washed with water to remove adhering soil and further washed by ultrasonication in water. Samples were stored frozen at -20 °C for a maximum of 8 months.

Roots were successively extracted by acetonitrile, twice by acetonitrile:water (50:50, v/v) and finally by water. The radioactivity levels (TRR) in combined extracts and in combusted solid fraction were quantified by LSC. The TRR was determined by the sum of these values. Results are shown in Table 29.

The acetonitrile and first acetonitrile/water extracts were combined (84–88% TRR), acidified to pH 4 and partitioned with diethyl ether. The aqueous fraction was further hydrolysed by 6 M HCl for 2 hours at 60 °C and partitioned with diethyl ether. Diethyl ether fractions were analysed by 1D- and 2D-TLC in parallel to reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X), fluazifop alcohol (XXXIV) and compounds 5, 8, 9, 16. Selected fractions were analysed by ^{19}F-NMR to confirm the identity of the compounds.

The nature of the residues in the treated carrots is summarized in Table 29. Metabolism of I and (RS) fluazifop-butyl was demonstrated to be essentially the same. Parent was not detected. Fluazifop acid (II, free and conjugated) was the major metabolite (44–63% TRR). CF3-pyridone (X, free) was found at trace levels and could be taken up from the soil. Despyridinyl acid (III) was released after hydrolysis at 4.8–6.4%. Fluazifop alcohol (free and conjugated) was found a levels of 10–13% TRR in carrots treated with ^{14}C-fluazifop-butyl only as it is formed stereospecifically from the (S)-enantiomer of fluazifop-butyl. Unidentified organo-soluble compounds U1, U2 and U3, each < 7% TRR, contain an intact diphenyl ether moiety and are only formed by the metabolism of the S-enantiomer of fluazifop-butyl and not the R-enantiomer. Water fractions contained 9.1–12% TRR.

Reviewer's conclusion:

Fluazifop acid (II) is stable in 6 M HCl for 2 hours at 60 ºC (see hydrolytic stability section above). The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. CF3-pyridone (X) was detected at trace levels. Pyr-Ph ether (IV) and fluazifop alcohol (XXXIV) were not detected. The presence of hydroxyfluazifop acid (XL) was not verified.

Table 29 Nature of residues in carrot roots

	^{14}C-Phenyl labelled R-enantiomer 1× 0.25 kg ai/ha; DAT 45 TRR = 0.15 mg/kg Fb eq		^{14}C-Phenyl labelled RS - racemate 1× 0.53 kg ai/ha; DAT 45 TRR = 0.18 mg/kg Fb eq		^{14}C-Pyridyl labelled RS-racemate 1× 0.51 kg ai/ha; DAT 45 TRR = 0.33 mg/kg Fb eq	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Parent	ND	-	ND	-	ND	-
Total fluazifop acid (II, free + conj) Fluazifop (free) Fluazifop polar conjugates	63.1 -38.6 -24.5	0.095	45.7 -28.9 -16.8	0.082	43.5 -25.6 -17.9	0.14
Pyr-Ph ether (IV)	ND	-	ND	-	ND	-
Despyridinyl acid (III, polar conj)	6.4	0.010	4.8	0.008	NR	-

	^{14}C-Phenyl labelled R-enantiomer 1× 0.25 kg ai/ha; DAT 45 TRR = 0.15 mg/kg Fb eq		^{14}C-Phenyl labelled RS - racemate 1× 0.53 kg ai/ha; DAT 45 TRR = 0.18 mg/kg Fb eq		^{14}C-Pyridyl labelled RS-racemate 1× 0.51 kg ai/ha; DAT 45 TRR = 0.33 mg/kg Fb eq	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
CF3-pyridone (X, free)	NR	-	NR	-	1.0 [a]	0.003
Fluazifop alcohol (free + conj) - fluazifop alcohol (free) - fluazifop alcohol polar conjugates	trace	trace	13.1 -11.3 -1.8	0.024	10.5 -7.7 -2.8	0.035
Hydroxyfluazifop acid (XL)	no ref std		no ref std		no ref std	
Compound U1 and U2 (not hydrolysed) [b]	ND	ND	4.1	0.007	2.4	0.008
Compound U3 (not hydrolysed) [b]	ND	ND	4.2	0.007	6.5	0.021
Compound U4 (after hydrolysis)	ND	-	ND	-	4.4 [a]	0.014
Water soluble unknows - not analysed; not hydrolysed [b] - not analysed; hydrolysed	12.4 -4.0 -8.4	0.02	9.1 - 2.9 - 6.2	0.016	11.7 - 4.2 - 7.5	0.038
PES: not hydrolysed [b]	8.2	0.012	12.2	0.022	12.3	0.041
Total	90.1		93.2		92.3	
Total identified	69.5		63.6		55.0	
Total not hydrolysed [b]	12.2		23.4		25.4	

NR = not relevant for this label, since the compound does not contain the phenyl or pyridyl group

[a] approximately quantified by 19F-NMR

[b] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

Metabolism study 18 (carrots)

In a GLP study, the metabolism of ^{14}C-phenyl- or ^{14}C-pyridyl-fluazifop-P-butyl (R-enantiomer) was studied in field grown carrots following two broadcast applications [Quistad, 2008, PP5_50002, report 1689W]. Carrots (variety Danvers Half Long 126) was planted in two plots (2 radiolabels) with a sandy loam, located in Madera, CA, USA in 2007. Each plot was treated twice with either EC formulated ^{14}C-phenyl-labelled or ^{14}C-pyridyl-labelled fluazifop-P-butyl by spraying uniformly over the top of foliage and soil. The first treatment was on 13 September (24 days after planting as seeds) and the second treatment was 21 days later (4 October). The target rate was 2 × 0.42 kg ai/ha for each radiolabel. The actual application rates were 98–100% of targets. Half of the carrot plants were harvested 20 days after the first application (immature, BBCH 43, 3 October) and the remaining plants were harvested 45 days after the second application (mature, 18 November). Samples were stored frozen for 8–37 days until primary extraction and analysis.

Homogenised carrot roots or foliage were combusted to determine total radioactive residues (TRR). Carrot roots or foliage were extracted by maceration twice with acetonitrile and then twice with acetonitrile: water (1:1). Further extractions used 30 min shaking in acetonitrile:0.5 M HCl, 30 minutes shaking in 6 M HCl to extract cellulose, overnight shaking in 24% KOH to extract hemicellulose. The combined HCl and KOH extracts were partitioned between ethylacetate (EtOAc) and water. Solids remaining from mature carrot foliage from the pyridinyl label was refluxed for 4 hours at 85 ºC with 1,4-dioxane/0.25 M HCl (4:1, v/v) to extract lignin. The radioactivity in the extracts and solids was determined by (combustion) LSC. Results are summarized in Table 30.

The combined acetonitrile and acetonitrile/water extracts and the ethyl acetate fractions (from the HCl and KOH extracts) were analysed by HPLC-UV using reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X) and compound 9. These assignments were investigated further by TLC analysis and peaks isolated by HPLC from the 4× rate samples. HPLC-UV analysis allowed assignment of structures to fluazifop acid (II) and CF3-pyridone (X), while four other components were purified for identification/characterisation and analysis by HPLC-UV and HPLC-MS. Results are shown in Tables 31 and 32.

- C1 = E-4 in the endive metabolism study. C1 could be hydrolysed with 6 M HCl overnight at room temperature to give 60% despiridinyl acid (III) in immature carrot roots. Based on similar HPLC-retention times, similar acid hydrolysis products and HPLC-MS analysis in the endive study, C1 is identified as a hexoside conjugate of despyridinyl acid (III).
- C2 = E-6 in the endive metabolism study. C2 could by hydrolysed with 6 M HCl overnight at room temperature to give 60% fluazifop acid (II) and 13% Pyr-Ph ether (IV) in immature carrot roots. Based on similar HPLC-retention times, similar acid hydrolysis products and HPLC-MS analysis in the endive study, C2 is identified as a fluazifop malonylhexoside conjugate.
- C3 = E-7 in the endive metabolism study. C3 could be hydrolysed with 6 M HCl overnight at room temperature to give 75% fluazifop acid (II) in immature carrot roots. Based on similar HPLC-retention times, similar acid hydrolysis products and HPLC-MS analysis in the endive study, C3 is identified as a fluazifop hexose conjugate (glucoside)
- C4 was only found from the pyridyl radiolabel. When the acetonitrile/water extract was partitioned with ethyl acetate, C4 partitioned into the aqueous phase and could be recovered on a silica SPE column. C4 could be hydrolysed with 6 M HCl overnight at room temperature to CF3-pyridone (X). HPLC-MS analysis gave a MH+ ion at 412.3, consistent with pyridinyl malonylhexoside.

Fluazifop-butyl (I) was detected in trace amounts (<0.01 mg/kg) in immature carrot roots only. The major metabolite in carrot foliage and roots is fluazifop acid (II) in its free or conjugated form (C-2, C-3). Additional metabolites of fluazifop-butyl resulted from cleavage at both ether linkages. CF3-pyridone (X, free) was a major metabolite from the pyridyl label for foliage and roots. CF3-pyridone (X) is conjugated as malonylhexoside (C-4) in mature foliage, but not detected in roots. Despyridinyl acid (III) was a major metabolite as a hexoside (C-1) for foliage and roots. No other metabolites represented >0.05 mg/kg or >10% TRR.

Reviewer's conclusion:

Whole extracts were analysed and fractions were isolated for identification with and without hydrolysis. Fluazifop acid (II) is stable in 6 M HCl for 6 hours at 60 ºC or 3 hours at 80 ºC (see hydrolytic stability section above) and therefore fluazifop acid (II) is assumed to remain stable during hydrolysis overnight in 6 M HCl at room temperature. Stability during overnight extractions with 24% KOH has not been investigated, but since significant amounts of fluazifop acid (II) were retained from the combined HCl/KOH fractions it is assumed that fluazifop acid (II) remains stable under these conditions. The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. The CF3-pyridone (X) counterpart is detected at higher levels and could represent additional uptake of CF3-pyridone (X) from soil. The presence of fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified, but they may be present in the organic fractions. Fractions that were not or partially subjected to hydrolysis (ACN/water, EtOAc, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X).

Table 30 Distribution of radioactivity in extracts of ^{14}C treated carrot plants

	Immature foliage R-enantiomer 1× 0.42 kg ai/ha DALT 20		Mature foliage R-enantiomer 2 × 0.42 kg ai/ha DALT 45		Immature roots R-enantiomer 1× 0.42 kg ai/ha DALT 20		Mature roots R-enantiomer 2 × 0.42 kg ai/ha DALT 45	
	^{14}C-Phenyl 0.86 mg/kg eq	^{14}C-Pyridyl 1.3 mg/kg eq	^{14}C-Phenyl 1.0 mg/kg eq	^{14}C-Pyridyl 1.5 mg/kg eq	^{14}C-Phenyl 0.38 mg/kg eq	^{14}C-Pyridyl 0.54 mg/kg eq	^{14}C-Phenyl 0.091 mg/kg eq	^{14}C-Pyridyl 0.13 mg/kg eq
	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Combined ACN and ACN/water	58.7%	74.1%	40.0%	56.4%	61.2%	64.3%	86.8%	88.0%

Combined ACN:0.5M HCl (1:1); 6 M HCl room temp; 24% KOH overnight	35.2%	23.7%	56.6%	32.0%	30.0%	28.1%	9.9%	9.8%
1 M HCl rinse	-	-	-	4.0%	-	-	-	-
1,4-dioxane/0.25 M HCl, 4 hours 85 °C	-	-	-	6.4%	-	-	-	
Solids	6.0%	2.2%	3.4%	1.3%	8.7%	7.5%	3.3%	2.3%

ACN = acetonitrile

Table 31 Nature of residues in carrot foliage

	Immature carrot foliage 1 × 0.42 kg ai/ha; DALT 20				Mature carrot foliage 2 × 0.42 kg ai/ha; DALT 45			
	^{14}C-Phenyl Label R-enantiomer 0.86 mg/kg eq		^{14}C-Pyridyl Label R-enantiomer 1.3 mg/kg eq		^{14}C-Phenyl Label R-enantiomer 1.0 mg/kg eq		^{14}C-Pyridyl Label R-enantiomer 1.5 mg/kg eq	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Parent	ND	-	ND	-	ND	-	ND	-
Total fluazifop acid (II, free + conj)	81.9		42.4		81.6		47.4	
- fluazifop acid (free)	-18.5	-0.16	-8.2	-0.11	-9.1	-0.091	-6.7	-0.10
- C2, malonylhexoside	-19.5	-0.17	-7.8	-0.10	-24.3	-0.24	-14.5	-0.22
- C3, hexose	-15.2	-0.13	-7.3	-0.097	[a]	[a]	[a]	[a]
- fluazifop acid, HCl/KOH released	-28.7	-0.25	-18.3	-0.24	-48.2	-0.48	-26.2	-0.40
- C2, HCl/KOH released	-	-	-0.8	-0.011	-	-	-	-
Pyr-Ph ether (IV)	ND		ND		ND		ND	
Despyridinyl acid (III conjugates)			NR	-			NR	-
- C1, hexose	1.7	0.015			5.9	0.059		
CF3-pyridone (X, free + conj)	NR	-	48.0		NR	-	31.1	
- CF3-pyridone free			-46.1	0.61			-17.0	0.26
- C4, pyridinyl malonyl hexose			-	-			-12.2	0.18
- CF3-pyridone, HCl/KOH released			-1.9	0.025			-1.9	0.028
Fluazifop alcohol (XXXIV)	no std		no std		no std		no std	
Hydroxyfluazifop acid (XL)	no std		no std		no std		no std	
Unknown organosoluble								
- ACN/water extract; not hydrolysed [c]	3.8	0.033	4.7 [b]	0.062	0.7	0.007	6.0 [b]	0.091
- EtOAc phase; partially hydrolysed [d]	3.7	0.032	0.7	0.009	4.9	0.049	1.7	0.026
Unknown water soluble								
- water phase; partially hydrolysed [d]	2.8	0.024	2.0	0.026	3.5	0.035	2.2	0.033
- 1 M HCl rinse; partially hydrolysed [d]	-	-	-	-	-	-	4.0	0.060
- 1,4-dioxane/0.25 M HCl extract; partially hydrolysed [d]	-	-	-	-	-	-	6.4	0.097
PES; partially hydrolysed [d]	6.0	0.052	2.2	0.029	3.4	0.035	1.3	0.019
Total	99.9	-	100.0	-	100.0	-	100.1	-
- Total identified	83.6		90.4		87.5		78.5	
- Total not or partially hydrolysed [c, d]	16.3		9.6		12.5		21.6	

TRR = total radioactive residue expressed as fluazifop-butyl equivalents.

ND = not detected.

NA = not analysed

NR = not relevant for this label, since the compound does not contain the phenyl or pyridyl group

[a] included in C2 figures

[b] maximum single residues 2.7% TRR or 0.041 mg/kg eq

[c] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone

[d] Has been hydrolysed with 0.25 M HCl, 6M HCl for 30 min; 24% KOH overnight or 1.4-dioxane with 0.25 M HCl 4 hours at 85 C; since this is a mixture of extracts, it may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X) after hydrolysis with 6 M HCl at 60 °C for 1-3 hours.

Table 32 Nature of residues in carrot roots treated with fluazifop-butyl

	Immature carrot roots 1 × 0.42 kg ai/ha; DALT 20				Mature carrot roots 2 × 0.42 kg ai/ha; DALT 45			
	^{14}C-Phenyl Label R-enantiomer 0.38 mg/kg eq		^{14}C-Pyridyl Label R-enantiomer 0.54 mg/kg eq		^{14}C-Phenyl Label R-enantiomer 0.091 mg/kg eq		^{14}C-Pyridyl Label R-enantiomer 0.13 mg/kg eq	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Parent	0.5	0.002	ND	-	ND	-	ND	-
Fluazifop acid (II, free + conj) - fluazifop acid (free) - C2, malonylhexoside - C3, hexose - fluazifop acid, HCl/KOH released	61.5 -19.5 -16.9 -6.1 -19.0	 0.074 0.064 0.023 0.072	48.6 -12.9 -17.3 [a] -18.4	 0.070 0.094 [a] 0.100	63.8 -35.2 -28.6 trace NA	 0.032 0.026 - -	58.7 -31.6 -27.1 trace NA	 0.042 0.036 - -
Pyr-Ph ether (IV)	ND		ND		ND		ND	
Despyridinyl acid (III conj) - C1, hexose	 12.9	 0.049	NR	-	 17.6	 0.016	NR	-
CF3-pyridone (X, free + conj) - CF3-pyridone free - C4, pyridinyl malonyl hexose - CF3-pyridone, HCl/KOH released	NR	-	37.0 -18.0 -16.1 -2.9	 0.098 0.088 0.016	NR	-	29.3 -15.0 -14.3 NA	 0.020 0.019 NA
Fluazifop alcohol (XXXIV)	no std		no std		no std		no std	
Hydroxyfluazifop acid (XL)	no std		no std		no std		no std	
Unknown organosoluble								
- ACN/water extract; not hydrolysed [b]	5.3	0.020	16.1	0.088	5.4	0.005 c	14.3	0.019 c
- EtOAc phase; partially hydrolysed [c]	3.4	0.013	2.6	0.014	5.5	0.005	6.0	0.008
Unknown water soluble								
- Water phase; partially hydrolysed [c]	7.7	0.029	4.2	0.023	4.4	0.004	3.8	0.005
PES; partially hydrolysed [c]	8.7	0.033	7.5	0.041	3.3	0.003	2.3	0.003
Total	100	-	99.9	-	100.0	-	100.1	-
Total identified	74.9		85.6		81.4		88.0	
Total not or partially hydrolysed [b, c]	25.1		14.4		18.6		12.0	

TRR total radioactive residue expressed as fluazifop-butyl equivalents.

ND not detected.

NA not analysed

NR not relevant for this label, since the compound does not contain the phenyl or pyridyl group

[a] included in C-2 figures

[b] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

[c] Hydrolysed with 0.25 M HCl or 6M HCl for 30 min. Since this is a mixture of extracts, it may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X) after hydrolysis with 6 M HCl at 60 °C for 1-3 hours.

Metabolism study 19 (potato tubers)

In a non-GLP study, the metabolism of ^{14}C-phenyl- or ^{14}C-pyridyl-fluazifop-butyl (RS) was studied in outdoor pot grown potatoes following a topical application to leaves plus soil [Snow *et al.*, 1983,

PP9/0041, report RJ0325B]. Potato plants (variety Desiree) were grown in pots (2 plants per pot) filled with a sandy loam soil and placed outside in a polythene enclosure at Blacknell, Berkshire, UK in 1980. Of each EC formulation half the amount was applied to the foliage (by syringe) when the foliage canopy measured approximately 45 cm and the other half to the soil of the same pot. An adjuvant was added (0.01% Agral 90). The actual application rates were equivalent to 0.86 or 0.84 kg ai/ha, respectively, for the phenyl and pyridyl label. Potato tubers were harvested 8 weeks after treatment. Samples were stored at -20 ºC. The storage period was not stated but is less than 32 months (start study to end laboratory operations).

Whole potato tubers were washed ultrasonically with acetonitrile and water to remove soil particles. Portions of 0.2 kg homogenised potato tubers were successively extracted by acetonitrile, acetonitrile:water (50:50, v/v) and finally by water. The radioactivity levels (TRR) in combined extracts and in combusted solid fraction were quantified by LSC.

The extracts were combined (89% and 87% TRR for phenyl and pyridyl label, respectively), concentrated and partitioned with hexane (fraction D; 0.2% and 0.3% TRR). The aqueous fraction was acidified to pH 3 with 1 M HCl and partitioned with diethyl ether (fraction F; 57% and 54% TRR). The aqueous phase (32% and 32% TRR) was evaporated to dryness and hydrolysed by 6 M HCl for 2 hours at 60 °C and partitioned between diethyl ether (fraction H; 27% and 12% TRR) and water (fraction I; 4.6% TRR and 21% TRR). Diethyl ether fractions were analysed by 1D- and 2D-TLC in parallel to reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X) and compounds 8, 9, and 24. Results are shown in Table 33.

The total radioactive residue was 0.37 mg/kg eq for the phenyl label and 0.29 mg/kg eq for the pyridyl label. Parent was not detected. In the ^{14}C- phenyl-labelled fluazifop-butyl treated group, the major residue consisted of 42% TRR fluazifop acid (II; 37% TRR as free acid, 4.2% TRR as water-soluble conjugated), 13% TRR hydroxyfluazifop acid (XL) and 18% TRR despyridinyl acid (III, 3.4% TRR as free and 15% as water-soluble conjugate). In the ^{14}C- pyridyl-labelled fluazifop-butyl treated group, the major residue consisted of 25% TRR fluazifop acid (II; 22% as free acid and 3.0% TRR as water-soluble conjugated), 15% TRR metabolite C and 15% despyridinyl acid (III) conjugates. Unidentified organosoluble fractions contained 11% TRR (phenyl), 26% TRR (pyridyl). Water fractions contained a maximum of 4.6% TRR (phenyl), 21% TRR (pyridyl).

Fractions containing fluazifop acid (II), despyridinyl acid (III) and metabolite C were isolated and cleaned-up by preparative layer chouromatography and HPLC. The identity of fluazifop acid (II) was confirmed by MS. The identity of despyridinyl acid (III) was confirmed GC-MS after derivatisation with n-butanol/acetyl chloride (to effect esterification) and by reaction with heptafluorobutyric anhydride (to derivatise the phenolic group). A portion of metabolite C fraction was methylated with diazomethane and the metabolite was identified by GC-MS as being hydroxyfluazifop acid (XL).

Reviewer’s conclusion:

Fluazifop acid (II) is stable in 6 M HCl for 2 hours at 60 ºC (see hydrolytic stability section above). The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. CF3-pyridone (X) and Pyr-Ph ether (IV) were not detected. Hexane fractions that were not analysed may contain some low levels of fluazifop-butyl (maximum 0.2–0.3% TRR). Fractions that were not subjected to hydrolysis (hexane, diethyl ether, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 14.8–30.0% TRR.

Table 33 Radioactive residues in potato tubers expressed as fluazifop-butyl equivalents.

Treatment	Phenyl (RS) 0.86 kg ai/ha DAT 56	Pyridyl (RS) 0.84 kg ai/ha DAT 56
TRR (mg/kg eq)	0.37	0.29
fluazifop-butyl	ND	ND
Total fluazifop acid (II, free + conjugates - fluazifop acid (free) - fluazifop polar conjugates	41.5 -37.3; [a] -4.2; [b]	24.7 -22.1; [a] -2.6; [b]
Pyr-Ph ether (IV)	ND	ND
despyridinyl acid (III, free + conjugates) - despyridinyl acid (free) - despyridinyl acid (III) polar conjugates	18.2 -3.4; [a] -14.8; [b]	NR
CF3-pyridone (X)	NR	ND
Fluazifop alcohol (XXXIV)	no ref std	no ref std
metabolite C = hydroxyfluazifop acid (XL, free)	13.1 [a]	15.4 [a]
Unknown organosoluble		
- hexane fraction D; not hydrolysed [d, e]	0.2	0.3
- diethyl ether fraction F; not hydrolysed [e]	3.2	16.5 [c]
- diethyl ether fraction H; after hydrolysis	7.9	9.3 [c]
Unknown water soluble		
- water fraction I; after hydrolysis	4.6	20.6
PES: not hydrolysed [e]	11.4	13.2
Total	100.1	100.0
- Total identified	72.8	40.1
- Total not hydrolysed [e]	14.8	30.0

NR not relevant for this label

[a] Identified in diethyl ether fraction F

[b] Identified in diethyl ether fraction H

[c] More than 5-6 components

[d] hexane fraction may contain some fluazifop-butyl (parent)

[e] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

Metabolism study 20a (sugarbeet roots)

In a non-GLP study, the metabolism of ^{14}C-fluazifop-butyl (RS) was investigated in pot grown sugar beet plants in a greenhouse [Cavell and Evans, 1981, PP9/0369, no report number]. Sugarbeet plants (variety Amono) at the 6-leaf stage were grown in 36 cm diameter pots (1 plant/pot) filled with sandy loam soil. EC formulated ^{14}C-phenyl- or ^{14}C-pyridyl-labelled fluazifop-butyl (RS) was at a nominal dose rate equivalent to 2.8–3.0 kg ai/ha. The formulation was applied evenly to sugar beet foliage (500 ul and 10 ul spots) using a syringe and 4000 ul was applied to the soil centring the plant. Adjuvant 0.01% Agral 90 was added. Sugar beet roots (sample size unknown) were harvested 90 days after application (87 days phenyl; 91 days pyridyl). Samples were kept at -18 ºC until analysis (period not stated).

Samples, extracts and remaining solids were analysed by (combustion) LSC. The roots contained 0.049-0.054 and 0.095 mg/kg Fb equivalents for the phenyl and pyridyl label, respectively.

The phenyl labelled roots were successively extracted by acetonitrile (28.4% TRR), methanol (7.5% TRR) and water (57.5% TRR). The acetonitrile and methanol extracts were combined (35.9% TRR) and analysed by TLC. The water extract was hydrolysed (no details given) and partitioned between diethyl ether (24% TRR) and water (33% TRR). In addition, the acetonitrile extract (28.4% TRR) was hydrolysed (no details given) and partitioned between diethylether (27% TRR) and water (1.7% TRR). The diethyl ether fractions were analysed by TLC.

The pyridyl labelled roots were extracted with aqueous acetonitrile (70% TRR) and the extract was evaporated to the aqueous remainder. The extract was partitioned between diethyl ether

(24% TRR) and water (46% TRR). The water phase was treated with acid hydrolysis (no details given) and partitioned between diethyl ether (37% TRR) and water (9.6% TRR). The solids (30% TRR) were re-extracted with hot HCl (18% TRR) and partitioned between diethyl ether (5.5% TRR) and water (13% TRR). The diethyl ether fractions were analysed by TLC.

Organo soluble fractions were analysed by TLC against reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV) and CF3-pyridone (X). The results are shown in Table 34.

Metabolism study 20b

In an addendum to this study [Evans *et al*, 1982, PP9/0193, report RJ0221B], the characterisation of residues was repeated with a different extraction scheme.

The phenyl labelled roots were washed with acetonitrile, homogenised and then extracted with acetonitrile/water (1:2, v/v, twice), water and finally acetonitrile until 17% TRR remained as solids. All extracts were combined (81% TRR) and the acetonitrile was removed by evaporation. The water phase was acidified with 2 M HCl and then centrifuged, whereby 9.8% TRR precipitated as solids. The remaining water phase (73% TRR) was partitioned between diethyl ether (20% TRR) and water (53% TRR). The solids were resuspended in water and partitioned between diethylether (5.0% TRR), water (1.1% TRR) and solids (3.7% TRR). Both diethyl ether phases (A; 25% TRR), both water phases (B; 54% TRR) and both solid fractions were combined (C; 21% TRR). The combined diethyl ether phase A was partitioned into 0.1 M NaHCO3, acidified with 2 M HCl and then again partitioned between diethyl ether (20.0% TRR) and water (5.0% TRR). The water phase was refluxed for an unstated time in 0.5 M methanolic NaOH, acidified and partitioned between diethyl ether (3.7% TRR) and water (0.6% TRR). The combined water phase B received an acid hydrolysis (6 M HCl, 1 hour, 60 ºC) or an alkaline hydrolysis (0.1 M NaOH, 1 hour, reflux), separately. The hydrolysates were acidified and then partitioned between diethyl ether (19%; 13% TRR for acid and base) and water (35%; 41% TRR for acid and base). The combined solid fractions C were hydrolysed with 0.1 M NaOH 1 hour reflux and than partitioned between diethyl ether (0.6% TRR), water (15% TRR) and solids (3.9% TRR). The water phase was adjusted to pH5 and then partitioned with diethylether (4.2% TRR) and water (11% TRR).

The pyridyl labelled roots were extracted with acetonitrile/water (1:2, v/v) until 30% TRR remained as solids. The extract (70% TRR) was evaporated to the aqueous remainder and partitioned between diethyl ether (10% TRR) and water (60% TRR). The water phase was adjusted to pH 3 and then partitioned between diethyl ether (14% TRR) and water (46% TRR). The water phase received an acid hydrolysis (0.1 M HCl, reflux, 2 ×1 hour, 6 M HCl, reflux 1 hour) or alkaline hydrolysis (0.1 M NaOH, reflux 2 hours, 1 M NaOH, reflux 2 × 1 hour). The hydrolysates were adjusted to 1.0 M HCl and were then partitioned between diethyl ether (37%TRR; 19.0% TRR for acid and base) and water (10%; 27% TRR for acid and base). The solids were refluxed for 1 hour in 1.0 M HCl and then partitioned between diethyl ether (5.5% TRR), water (13% TRR) and solids (12% TRR).

Diethyl ether fractions from both labels were analysed by TLC against reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III) and CF3-pyridone (X) and compound 9. The results are shown in Table 34.

The combined water phases from the phenyl label (54% TRR) were treated with 0.008 M HCl, heated for 30 min at 35 ºC to convert the sucrose present in the sugarbeet to glucose and fructose. Phenyl hydrazine HCl and sodium acetate were added and the mixture was heated for 90 min under reflux and then left for 16 hours at ambient temperature. During this process, phenylhydrazine reacted with glucose or fructose to form osazone. The crystalline osazone was collected and analysed by LSC.

Metabolism study 20c

In an addendum to study RJ0221B, more rigorous hydrolysis of the water-soluble fractions from the phenyl labelled residues was applied, aiming to further characterise the residues [Cavell and Evans, 1985, PP9/0194, report RJ0373B].

The water phase remaining after acid hydrolysis (34.7% TRR; phenyl), was further hydrolysed with 6.0 M HCl under reflux conditions for 1 hour. Subsequently acidity was reduced with 5.0 M NaOH, followed by partitioning with diethylether, resulting in an organosoluble fraction (9.1% TRR) and a water fraction (16.1% TRR). Characterisation of the organo-soluble fraction was not possible due to large amounts of hydrolysed plant coextractives. Furthermore, 9.5% TRR was occluded in a heavy black precipitate which formed after hydrolysis.

The water phase remaining after base hydrolysis (41.3% TRR; phenyl), was further hydrolysed with aqueous 1.0 M NaOH under reflux conditions for 1 hour. Subsequently the hydrolysate was acidified with 11 M HCl, followed by partitioning with diethylether. This resulted in a water fraction (27.6% TRR) and an organosoluble fraction (13.7% TRR) in which despyridinyl acid (III) was identified (10.9% TRR).

Results from the thouree studies are summarized in Table 34. Parent was not detected. The major metabolites were fluazifop acid (II, free and conjugated) at 25–26%; 9.9–12% TRR (phenyl; pyridyl label) and CF3-pyridone (X) at 27.2% TRR (pyridyl label only). Despyridinyl acid (III, 6.8–18.2% TRR, phenyl label only) and the major part of CF3-pyridone (X, 21% TRR) was released after hydrolysis. Pyr-Ph ether (IV, 1.0% TRR) was found with the phenyl label only. About 4% TRR (phenyl label) incorporation of radioactivity into sucrose was observed. Unidentified organosoluble fractions contained 12–23% TRR (phenyl), 22–29% TRR (pyridyl). Water fractions contained a maximum of 30–39% TRR (phenyl), 22–45% TRR (pyridyl). Because of the high sugar content of this fraction, it was not possible to investigate this residue further.

Reviewer's conclusion:

The pyridyl labelled study is not acceptable since fluazifop acid (II) is degraded under the reflux conditions used in the pyridyl label and CF3-pyridone (X) is degraded under alkaline conditions. Fluazifop acid (II) is stable in 6 M HCl for 2 hours at 60 ºC, 0.1 M NaOH at 1 hour reflux or 0.5 M NaOH in methanol at 1 hour reflux (see hydrolytic stability section above) as used for the phenyl labelled samples. The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. Fractions that were not subjected to hydrolysis (methanol, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 4.3–44.5% TRR.

Table 34 Characterisation of residues in sugarbeetroots treated with fluazifop-butyl

Metabolites	Phenyl (RS) 3.0 kg ai/ha DAT 90	Phenyl (RS) 2.8 kg ai/ha DAT 87	Phenyl (RS) 2.8 kg ai/ha DAT 87	Pyridyl (RS) 3.0 kg ai/ha DAT 90	Pyridyl (RS) 3.0 kg ai/ha DAT 91	Pyridyl (RS) 3.0 kg ai/ha DAT 91
TRR (mg/kg Fb eq)	0.054	0.049	0.049	0.095	0.095	0.095
Study	Cavell	RJ0221B acid	RJ0221B base	Cavell	RJ0221B acid	RJ0221B base
Parent	ND	ND	ND	ND	ND	ND
Total fluazifop acid (II, free + conj) - Fluazifop (free) - Fluazifop organosoluble conj - Fluazifop water soluble conj - Fluazifop solid conjugates	**25.5** - 16.0 - 4.7 - 4.8 -	**25.3** - 18.0 - 1.0 - 2.9 - 3.4	**25.0** - 18.0 - 1.0 - 2.6 - 3.4	**9.9** - 9.2 - trace - 0.7	**9.9** - 9.2 - - 0.7	**11.8** -9.2 - 1.9 0.7
Pyr-Ph ether (IV conjugates) - water soluble conjugates	1.0	no ref std	no ref std	ND	no ref std	no ref std
Despyridinyl acid (III, conjugates) - organosoluble conjugates - water soluble conjugates	**9.2** - 3.5 - 5.7	**6.8** - - 6.8	**18.2** - - 7.3 - 10.9 [d]	NR	NR	NR

Metabolites	Phenyl (RS) 3.0 kg ai/ha DAT 90	Phenyl (RS) 2.8 kg ai/ha DAT 87	Phenyl (RS) 2.8 kg ai/ha DAT 87	Pyridyl (RS) 3.0 kg ai/ha DAT 90	Pyridyl (RS) 3.0 kg ai/ha DAT 91	Pyridyl (RS) 3.0 kg ai/ha DAT 91
TRR (mg/kg Fb eq)	0.054	0.049	0.049	0.095	0.095	0.095
Study	Cavell	RJ0221B acid	RJ0221B base	Cavell	RJ0221B acid	RJ0221B base
CF3-pyridone (X, free + conj) - free - water soluble conjugates - solid conjugates	NR	NR	NR	27.2 - 6.3 - 18.2 - 2.7	27.2 - 6.3 - 18.2 - 2.7	27.2 - 6.3 - - 2.7
Fluazifop alcohol (XXXIV)	no ref std	no ref std	no ref std	no ref std	no ref std	no ref std
Hydroxyfluazifop acid (XL)	no ref std	no ref std	no ref std	no ref std	no ref std	no ref std
Organosoluble unknowns (total)	**22.4**	**22.8**	**12.4**	**29.2**	**29.2**	**22.5**
- methanol extract; not hydrolysed [a]	- 4.3	-	-	-	-	-
- diethyl ether phase (water) after hydrolysis	- 3.1 - 6.0 - 3.3	-	-	-	-	-
- diethyl ether phase (ACN/water) not hydrolysed [a]	-	- 0.2 - 0.6 - 2.0	- 0.2 - 0.6 - 2.0	- 5.7 - 2.9	- 1.4 - 7.2	- 1.4 - 7.2
- diethyl ether phase (ACN/water) after hydrolysis	- 1.4 - 4.3	- 2.7 - 2.6 [c] - 4.2 [c] - 9.1 [d]	- 2.7 - 2.7 [c] - 2.8 [d]	- 18.5	- 18.5 [c]	- 1.8 [c] - 10.0 [c]
- diehtyl ether phase (solids); after hydrolysis	-	- 0.6 - 0.8	- 0.6 - 0.8	- 1.1 - 1.0	- 2.1	- 2.1
Water soluble unknowns (total)	**35.3**	**30.2**	**39.0**	**22.2**	**22.2**	**45.2**
- water phase; not hydrolysed [a]	- 33.6	-	-	-	-	-
- water phase (ACN/water); after hydrolysis	- 1.7	- 0.6 [b] - 2.7 - 16.1 [d]	- 0.6 [b] - 27.6 [d]	-9.6	- 9.6	- 27.3 - 5.3
- water phase (solids); after hydrolysis	-	- 10.8	- 10.8	- 12.6	- 12.6	- 12.6
PES - not hydrolysed [a] - after hydrolysis	**6.6** - 6.6 -	**14.9** - 1.5 - 3.9 - 9.5 [d]	**5.4** - 1.5 - 3.9	**11.5** - - 11.5	**11.5** - - 11.5	**11.5** - - 11.5
Total (% TRR)	100.0	100.0	100.0	100.0	100.0	100.0
Total identified	35.7	31.1	43.2	37.1	37.1	39.0
Total not hydrolysed [a]	44.5	4.3	4.3	8.6	8.6	8.6

[a] Not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

[b] Partitioned into the aqueous phase after treatment of the organic fraction with 0.5 M NaOH in methanol

[c] Partitioned into the organic phase after treatment of the aqueous phase with acid or base

[d] Fractions derived from 34.7% TRR and 41.3% TRR water fractions (which remained after hydrolysis with 6 M HCl, 1 hour, 60 °C) using more stringent hydrolysis conditions as described in report RJ0373B [see text].

acid The water phase of the acetonitrile/water extract was treated with acid

base The water phase of the acetonitrile/water extract was treated with base

Metabolism study 21 (sugarbeet roots)

In a non-GLP study, the metabolism of ^{14}C-phenyl- or ^{14}C-pyridyl-fluazifop-butyl (RS) or ^{14}C-phenyl-fluazifop-P-butyl (R enantiomer) was studied in field grown sugarbeets following a broadcast application [Hughes *et al*, 1986, PP9/0040, report RJ0490B]. Sugar beet plants (variety Julia) were grown on a 0.16 m^2 plot with sandy clay loam soil for 64 days prior to radiochemical application. Plants were grown in the field in Bracknell, Berkshire, UK in 1984. Actual dose rates were 0.52 and 0.51 kg ai/ha for EC formulated ^{14}C-phenyl-labelled and ^{14}C-pyridyl-labelled fluazifop-butyl and 0.25 kg ai/ha ^{14}C-phenyl-labelled fluazifop-P-butyl. Application was in an aqueous 0.1% Agral 90

solution. At the time of application (19 July) the sugarbeet plants were about 25 cm high. The application was sprayed evenly at a height of approximately 60 cm over the plots using a hand operated spray gun. Sugar beet roots (0.7–2.4 kg) were harvested 90 days after application (17 October). The roots were washed with water to remove adhering soil. Samples were stored at -20 °C for a maximum of 6 months.

Roots were successively extracted by acetonitrile, acetonitrile:water (50:50, v/v) and finally by water. The radioactivity levels (TRR) in combined extracts and in combusted solid fraction were quantified by LSC. The TRR was determined by the sum of these values and expressed as fluazifop-butyl equivalents. Results are shown in Table 35.

The acetonitrile and acetonitrile/water extracts were combined (87–93% TRR), rotary evaporated to remove the acetonitrile, acidified to pH 3 and partitioned with diethyl ether. The diethyl ether phase was backwashed with water. The aqueous phase was passed thourough an XAD-4 resin column to remove any co-extractives. The column was eluted with water and then with acetonitrile. The acetonitrile eluate was evaporated down to a small volume, redissolved in acetonitrile/water (50:50, v/v), hydrolysed with 2 M HCl for 2 hours at 80 °C and partitioned with diethyl ether. All diethyl ether fractions were analysed by 1D- and 2D-TLC in parallel to reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X), fluazifop alcohol (XXXIV) and compounds 5, 8 and 9.

The nature of the residues in the treated sugar beets is summarized in Table 35. Parent was not detected. Fluazifop acid (II, free and conjugated) was the major metabolite and represented a maximum of 34–52% TRR (0.047–0.068 mg/kg eq). Despyridinyl acid (III, 15–17% TRR) was released after hydrolysis and could be derived from degradation of fluazifop (II) or its conjugates as the result of the hydrolysis conditions used. Unknown compound U1 was found at levels of 15–20% TRR (free plus conjugates) for the racemate for both fluazifop-butyl labels, while it was only a minor residue (5.5% TRR) for the R-enantiomer, suggesting that this metabolic route is less important for the R-enantiomer of fluazifop-butyl.

Phenyl labelled compound U1 is a polar metabolite, possibly an amino-acid conjugate, and was shown to yield fluazifop acid (II, 13%), despyridinyl acid (III, 49%), unknown U4 (17%) and water solubles (12%) under strong acid hydrolytic conditions (6 M HCl under reflux for 1 hour). Under the same hydrolysis conditions, pyridyl labelled compound U1 yielded fluazifop acid (II, 41%), CF3-pyridone (X, 16%), unknown U5 (14%) and water solubles (18%). These strong acid conditions are likely to cleave to central ether bond of fluazifop acid (II) and/or fluazifop-butyl. Attempts to cleave U1 under milder conditions (6 M HCl at 60 °C), where fluazifop acid (II) is known to be stable, yielded small amounts of fluazifop acid (II, 5.5%) and despyridinyl acid (III, 9.8%), but most of the radioactivity was still polar in nature. Other unidentified organosoluble fractions contained 2.3–4.4% TRR (phenyl), 17% TRR (pyridyl). Water fractions contained a maximum of 8.5–14% TRR (phenyl), 17% TRR (pyridyl).

Reviewer’s conclusion:

Fluazifop acid (II) is stable in 6 M HCl for 3 hours at 80 ºC (see hydrolytic stability section above) and therefore also in 2 M HCl for 3 hours at 80 ºC. The presence of despyridinyl acid (III) therefore indicates cleavage of the phenyl-pyridyl ether linkage as part of the metabolism within the plant. CF3-pyridone (X) was detected at much lower levels. Pyr-Ph ether (IV) and fluazifop alcohol (XXXIV) were not detected. The presence of hydroxyfluazifop acid (XL) was not verified. Fractions that were not subjected to hydrolysis (diethyl ether, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 16.6–18.1% TRR.

Table 35 Characterisation of residues in sugar beet roots treated with fluazifop-butyl

Metabolites	Phenyl (R enantiomer) 0.25 kg ai/ha, DAT 90 TRR = 0.09 mg/kg eq	Phenyl- (RS) racemate 0.52 kg ai/ha, DAT 90 TRR = 0.08 mg/kg eq	Pyridyl (RS) racemate 0.51 kg ai/ha, DAT 90 TRR = 0.20 mg/kg eq
Parent	ND	ND	ND
Total fluazifop acid (II, free + conj) -- Fluazifop acid (free) -- Fluazifop (conjugated)	52.1 -42.0 -10.1	40.4 -31.4 -9.0	33.8 -19.6 -14.2 (11.8 + 2.4)
Pyr-Ph ether (IV)	ND	ND	ND
Despyridinyl acid (III, conjugates)	17.1	15.2	NR
CF3-pyridone (X, free + conj) -- CF3-pyridone (free) -- CF3-pyridone (conjugates)	NR	NR	3.4 -1.0 -2.4
Fluazifop alcohol (XXXIV)	ND	ND	ND
Hydroxyfluazifop acid (XL)	no ref std	no ref std	no ref std
Unknown U1 (free+conj) -- Unknown U1 (free) -- Unknown U1 (conjugated)	5.5 -3.5 -2.0	15.5 -10.3 -5.2	20.5 -10.0 -10.5
Unknown U2 (conjugated)	--	--	4.4
Unknown U3 (conjugated)	--	--	1.6
Organo soluble unknowns - not hydrolysed [a] - after hydrolysis	2.3 - 1.1 - 1.2	4.4 - 2.2 - 2.2	10.7 - 4.1 - 6.6 (2.6+2.7+1.3)
Water soluble unknowns - not hydrolysed [a] - after hydrolysis	14 -3.4 -5.4 -5.2	8.5 -3.7 -1.8 -3.0	16.9 -1.4 -6.8 -8.7
PES; not hydrolysed [a]	7.8	8.9	5.8
Total (% TRR)	98.8	92.9	97.1
Total identified	69.2	55.6	37.2
Total not hydrolysed	17.7	16.6	18.1

NR = not relevant for this label, since the compound does not contain the phenyl or pyridyl group

[a] Not hydrolysed: may contain additional fluazifop acid (II), Pyr-Ph ether (IV) and/or despyridinyl acid (III)/CF3-pyridone (X)

Additional studies using non-labelled samples from supervised residue trials

The Meeting received additional non-labelled studies to investigate the nature of residues in various crop commodities.

Study 1

Samples from crops treated with fluazifop-butyl (RS) were analysed separately for fluazifop-butyl and free fluazifop acid (II) [Atreya *et al*., 1981, PP9/0384, report RJ0226B]. Fluazifop-butyl was determined by HPLC-UV method PPRAM 51. Free fluazifop acid (II) was determined by HPLC-UV method PPRAM 53. Validation for these methods is not available, therefore results can only be considered qualitative.

Several crop commodities were analysed. The study report summarizes data from study reports PP009 B001-B005, B007-B011, B013-B016, B018-B019, B021-B029, B033-B036, B038-B039, B043-B058, B060, B062-B063. Only those crop commodities with sampling points before 100 days were listed in the table below; longer sampling points did not show fluazifop-butyl (parent). Crop commodities where fluazifop-butyl (parent) was not detected were not listed:

- Potato tubers: < 0.01 mg/kg parent at 1× 1.0 kg ai/ha (in original report PP009B013, summary report RJ0226B indicates 0.1 kg ai/ha) with harvest at 33–105 DAT (Germany, 1980) or 1× 0.25–0.50–1.0 kg ai/ha with harvest at 85–95 DAT (Canada 1979) or 1 × 0.50–1.0–2.0 kg ai/ha with harvest at DAT118–141 (Netherlands, 1979, 1980)

- Dry soya bean seeds: < 0.01 mg/kg parent at 1 × 0.5–1.0 kg ai/ha, at 77–109 DAT (Canada 1980)
- Linseeds: < 0.01 mg/kg parent at 1 × 0.25–0.60 kg ai/ha, at 76–98 DAT (Canada 1979)
- Sunflowers: < 0.01 mg/kg parent at 1 × 0.50 kg ai/ha,at 130–137 DAT (Canada 1979)

Results for strawberries, sugarbeet roots, sugarbeet tops and oilseed rape seeds are summarized in Table 36.

Reviewer's conclusion:

Fluazifop-butyl (parent compound) is found at significant quantities at the day of application, but is found at low levels up to 8 days in fruits, up to 12 days in roots, up to 16 days in oilseed forage and up to 98 days in root forage.

Table 36 Fluazifop-butyl and free fluazifop acid (II) content in strawberries and sugarbeets

Crop	Trial information	DAT	Crop part	Fluazifop-butyl mg/kg	Free fluazifop acid (II) mg/kg
Strawberries	UK, 1980 1× 1.5 kg ai/ha	0 3 8	fruit fruit fruit	0.23 0.06 0.01	0.48 2.06 2.01
	UK, 1980 1× 2.0 kg ai/ha	52 56 71	fruit fruit fruit	< 0.01 < 0.01 < 0.01	0.06 0.03 < 0.02
Sugarbeets	UK, 1979, 1× 1.0 kg ai/ha	0 6 12 13 17 21	roots roots roots roots roots roots	0.12 0.02 0.01 < 0.01 < 0.01 < 0.01	0.57 0.58 0.12 0.12 0.09 0.04
Sugarbeets	UK, 1980, 1× 1.0 kg ai/ha	77 77	roots tops	< 0.01 0.03	< 0.02 0.02
	UK, 1980, 1× 2.0 kg ai/ha	77 77	roots tops	< 0.01 0.03	0.02 0.02
	UK, 1980, 1× 4.0 kg ai/ha	77 77	roots tops	< 0.01 < 0.01	0.03 0.16
	UK, 1980, 1× 1.0 kg ai/ha	96 96	roots tops	< 0.01 < 0.01	< 0.02 0.02
Sugarbeets	UK, 1980 1× 1.0 kg ai/ha	77 77	roots tops	< 0.01 < 0.01	0.03 0.14
	UK, 1980, 1× 2.0 kg ai/ha	98 98	roots tops	< 0.01 0.03	0.03 0.16
Sugarbeet	UK, 1980 1× 1.0 kg ai/ha	21 42 75 0 21 42 75	roots roots roots tops tops tops tops	< 0.01 < 0.01 < 0.01 34.0 0.08 0.02 < 0.01	0.62 < 0.02 < 0.02 5.40 0.40 < 0.05 < 0.05
	UK, 1980, 1× 1.0 kg ai/ha	20 42 81 0 20 42 81	roots roots roots tops tops tops tops	< 0.01 < 0.01 < 0.01 46 0.1 < 0.01 < 0.01	0.44 < 0.02 < 0.02 18 0.06 < 0.05 < 0.05
	UK, 1980, 1× 1.0 kg ai/ha	23 41 62 86 0 23 41 62	roots roots roots roots tops tops tops tops	< 0.01 < 0.01 < 0.01 < 0.01 39.0 < 0.01 < 0.01 < 0.01	0.12 0.04 0.04 < 0.02 7.4 0.29 0.10 0.11

Crop	Trial information	DAT	Crop part	Fluazifop-butyl mg/kg	Free fluazifop acid (II) mg/kg
		86	tops	< 0.01	0.06
Sugarbeets	Germany, 1980	19	roots	< 0.01	0.51
	1× 1.0 kg ai/ha	42	roots	< 0.01	0.11
		64	roots	< 0.01	0.07
		85	roots	< 0.01	0.03
		0	tops	65	8.2
		19	tops	< 0.01	1.20
		42	tops	< 0.01	0.28
		64	tops	< 0.01	0.15
		85	tops	< 0.01	0.14
	Germany, 1980	21	roots	< 0.01	0.36
	1× 1.0 kg ai/ha	43	roots	< 0.01	0.17
		62	roots	< 0.01	0.16
		85	roots	< 0.01	0.03
		0	tops	42	9.7
		21	tops	< 0.01	0.36
		43	tops	< 0.01	0.17
		62	tops	< 0.01	0.16
		85	tops	< 0.01	0.03
	Germany, 1980	21	roots	< 0.01	0.27
	1× 1.0 kg ai/ha	43	roots	< 0.01	0.09
		62	roots	< 0.01	0.02
		85	roots	< 0.01	< 0.02
		0	tops	41	12
		21	tops	< 0.01	0.60
		43	tops	< 0.01	0.08
		62	tops	< 0.01	0.07
		85	tops	< 0.01	< 0.02
Oilseed rape	Germany, 1979-1980	0	forage	9.0	17.0
	2 × 0.5 kg ai/ha	23	forage	< 0.01	9.2
		101	forage	< 0.01	6.8
		256	seeds	< 0.01	0.07
	Germany, 1979-1980	0	forage	5.6	16
	2 × 0.5 kg ai/ha	22	forage	< 0.01	8.5
		95	forage	< 0.01	8.3
		253	seeds	< 0.01	0.46
	Germany, 1979-1980	0	forage	14	16
	1× 1.0 kg ai/ha	18	forage	< 0.01	3.9
		91	forage	< 0.01	1.9
		276	seeds	< 0.01	0.06
	Germany, 1979-1980	0	forage	9.7	12
	1× 1.0 kg ai/ha	15	forage	< 0.01	5.0
		88	forage	< 0.01	1.9
		273	seeds	< 0.01	< 0.02
	Germany, 1979-1980	0	forage	3.6	8.9
	1× 1.0 kg ai/ha	14	forage	0.03	7.3
		101	forage	< 0.01	3.8
		271	seeds	< 0.01	0.12
	Germany, 1979-1980	0	forage	3.7	17.0
	1× 1.0 kg ai/ha	16	forage	0.06	8.1
		88	forage	< 0.01	5.8
		256	seeds	< 0.01	< 0.02
Oilseed rape	Canada, 1979	80	seed	< 0.01	5.3
	1 × 0.50 kg ai/ha	95	seed	< 0.01	0.21
	Canada, 1979	77	seed	< 0.01	6.2, 7.1
	1 × 0.50 kg ai/ha	92	seed	< 0.01	0.18, 0.28
Oilseed rape	Canada, 1980	81	seed	< 0.01	0.54
	1× 0.50 kg ai/ha	90	seed	< 0.01	0.09
	Canada, 1980	69	seed	< 0.01	1.7
	1× 1.0 kg ai/ha	70	seed	< 0.01	1.3
	Canada, 1980	65	seed	< 0.01	10.2
	1× 0.75kg ai/ha				
	Canada, 1980	61	seed	< 0.01	0.91

Crop	Trial information	DAT	Crop part	Fluazifop-butyl mg/kg	Free fluazifop acid (II) mg/kg
	1× 0.50 kg ai/ha	65 95 98	seed seed seed	< 0.01 < 0.01 < 0.01	5.5 0.38 0.50
	Canada, 1980 1 × 0.50 kg ai/ha	60 67 74 81 88	seed seed seed seed seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	9.80 3.50 1.10 0.48 0.25
	Canada, 1980 1× 0.50 kg ai/ha	83 93	seed seed	< 0.01 < 0.01	17 14
	Canada, 1980 1 × 0.50 kg ai/ha	83	seed	< 0.01	0.38

Study 2

Soya bean seed samples from supervised residue trials were analysed separately for free fluazifop acid (II) and total fluazifop [Atreya *et al*., 1981, 462775, report PP009B030]. Free fluazifop acid (II) was determined by HPLC-UV method PPRAM 53. Total fluazifop was determined by HPLC-UV method PPRAM 62. Control samples were < 0.02 mg/kg with both methods. Concurrent recoveries were 90% at 0.5 mg/kg. Results are shown in Table 37.

Reviewer's conclusion:

Significantly higher residues were found when residues were determined as total fluazifop (which includes fluazifop-butyl and fluazifop (II) conjugates) than when residues were determined as fluazifop acid (II) alone.

Table 37 Residues in soya bean seed samples from supervised residue trials

Sample ID	Total fluazifop mg/kg	Fluazifop acid (II, free) acid, mg/kg
5560	0.05	0.05
5561	0.21	0.13
5562	1.02	0.79
5557	0.38	0.29
4820	1.46	0.79
4819	0.86	0.55

Study 3

Samples from crops treated with fluazifop-butyl (RS) were analysed for R- and S-enantiomers of total fluazifop [Davy and Atreya, 1983, PP9/0192, report RJ0298B]. All crop samples came from 1982 supervised field trials with fluazifop-butyl (RS), except for kale samples which came from 1980 supervised field trials.

An analytical method was developed, based on HPLC using a mobile phase containing the chiral chelate L-prolyl-n-octylamide Ni (II). Good separation of R- and S-enantiomers can be achieved by manipulation of pH, ionic strength, metal ion and temperature. The most satisfactory separation of crop impurities from the fluazifop enantiomers as well as complete separation of the enantiomers was obtained with 2.5 mM L-prolyl-n-octylamide Ni (II) with acetonitrile/methanol/water solvent system (35:15:50, v/v/v), pH =7.8, column temperature 35 ºC. Addition of ammonium acetate 0.055 M was found to shorten the retention times. The pH was achieved by adding glacial acetic acid and then adjusting the pH by addition of ammonia solution. Ratio's of R/S enantiomers in a 10 mg/L fluazifop acid (II) standard solution were 1.21–1.25 (or 55–56% R-enantiomer).

Samples were extracted, hydrolysed and cleaned-up as for method PPRAM 62. Samples were extracted with acetonitrile/concentrated HCl (98:2) for wet crops or with acetonitrile after a pre-soak

in 1 M HCl for dry crops. The extract was hydrolysed with 6 M HCl, 1 hour, 60 ºC, to ensure that any fluazifop-butyl or any fluazifop conjugates were converted to fluazifop acid (II). After clean-up by liquid/liquid partition, coagulation and adsorption chouromatography, the final residue was dissolved in the chiral mobile phase solution for enantiomer separation. Results are shown in Table 38.

Reviewer's conclusion:

An increase in the proportion of the R-enantiomer of fluazifop acid (II) in the residues has been found, with a crop to crop variation in the rate and content of the conversion. The proportion of the R-enantiomer of fluazifop acid (II) remained approximately the same in carrot roots at 21 days after treatment (46–54%), but increased to 74–82% in apple at 35–49 days after treatment, 78% in head cabbage at 49 days after treatment, 62% in kale at 27–41 days after treatment, 69–77% in dry peas at 54 days after treatment and 76–84% in oilseed rape seeds.

Table 38 R- and S-enantiomer content of fluazifop acid (II) in various commodities

Crop	Trial information	Report [a]	DAT	Crop part	Total fluazifop mg/kg	R-enantiomer percentage
apple	Elgin, South Africa 1.5 kg ai/ha	PP009B177	42 49	fruit	0.6 0.8	74% 77%
apple	Elgin, South Africa 3.0 kg ai/ha	PP009B177	35 42 49	fruit	1.2 2.6 1.1	81% 82% 79%
head cabbage (whole)	Gamlingay, UK 1× 0.75 kg ai/ha	PP009B164	0 11 21 34 49	whole whole whole whole whole	8.1 6.9 5.2 3.3 3.0	51% 62% 70% 70% 78%
carrot	RS 8274 B1, Germany 1× 0.38 kg ai/ha 30 cm roots; 1.0-1.5 cm diameter	PP009B161	0 10 21	whole roots roots	5.1 0.21 0.14	46% 51% 54%
carrot	RS 8274 B2, Germany 1× 0.38 kg ai/ha 25 cm roots; 1.0 cm diameter	PP009B161	0 10 21	whole roots roots	4.0 0.32 0.08	46% 57% 55%
kale	RS 8054 III-1, Germany 1× 0.38 kg ai/ha 15 cm roots	PP009B088	0 7 13 20 27 34 41	whole whole whole whole whole leaves leaves	13.0 4.7 2.6 2.0 1.9 1.5 0.6	48% 51% 57% 57% 62% 62% 62%
dry peas	82/137, Netherlands 1× 0.38 kg ai/ha; 40 cm crop height	PP009B1B2	54	seed	1.4	69%
dry peas	82/137, Netherlands 1× 0.75 kg ai/ha 40 cm crop height	PP009B1B2	54	seed	3.3	77%
oilseed rape seed	CA/MA/HE/80/014/C; Canada 1× 0.5 kg ai/ha	PP009B018	60	seed	9.8	81%
oilseed rape seed	RS 8282 E; Germany 1× 0.75 kg ai/ha 20-30 cm crop height	ns	99	seed	8.7	76%
oilseed rape seed	RS 8282 B; Germany 1× 0.75 kg ai/ha 59 cm crop height	ns	105	seed	3.2	84%

[a] Original reports were not submitted

Study 4

Samples from crops treated with fluazifop-butyl (RS) were analysed separately for total fluazifop (II), total despyridinyl acid (III) and/or total CF3-pyridone (X). No data are available for Pyr-Ph ether (IV). Results are summarized in Table 39.

Reviewer's conclusion:

Analytical methods either used refluxed conditions to extract despyridinyl acid (III) and CF3-pyridone (X), whereby these metabolites could also be released from fluazifop acid (II) or they did not use hydrolysis conditions, whereby only the free metabolites were quantified. Therefore levels of despyridinyl acid (III) and CF3-pyridone (X) are either underestimated (no hydrolysis) or overestimated (reflux conditions). Only the trials conducted on celery are considered reliable, since extraction conditions will not degrade fluazifop acid (II) and are sufficient to release CF3-pyridone (X) from its conjugates.

Table 39 Total fluazifop (II, including fluazifop-butyl and fluazifop conjugates), despyridinyl acid (III), and CF3-pyridone (X) in crops treated with fluazifop-butyl

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
Dry harvested onion bulbs	Niland, CA, USA, 1981 2 ×1.1 kg ai/ha (racemate)	45	< 0.04	0.08	< 0.05	38CA81-038 II: [Koubek, 1984, 406215, report TMU1257/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Dry harvested onion bulbs	Brawley, CA, USA, 1982 2 ×1.1 kg ai/ha (racemate)	46	0.48	0.08 [cntrl=0.10]	0.06	38CA82-005 II: [Koubek, 1984, 406215, report TMU1257/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Dry harvested onion bulbs	Calipatria, CA, USA, 1982 2 ×1.1 kg ai/ha (racemate)	45	0.26	0.09	< 0.05	38CA82-007 II: [Koubek, 1984, 406215, report TMU1257/B];] III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Dry harvested onion bulbs	Ruskin, FL, USA, 1982 2 × 0.56 kg ai/ha (racemate)	39	0.05	< 0.05	< 0.05	53FL82-005 II: [Koubek, 1984, 406215, report TMU1257/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Dry harvested onion bulbs	AZ, USA, 1982 2 ×1.1 kg ai/ha (racemate)	46	no study available	0.05	< 0.05	42AZ82-001 III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Dry	Fort Collins,	45	0.06 (P)	-	< 0.05	37CO84-056

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
harvested onion bulbs	CO, USA, 1984 2 × 0.42 kg ai/ha (R-enantiomer)					II: [Francis, 1985, 434142, TMU1815B] X: [Morgan and Crook, 1986, PP5/0250, M4266B]
Dry harvested onion bulbs	Hastings, FL, USA, 1984 2 × 0.42 kg ai/ha (racemate; R-enantiomer)	44 44	0.06 (P) 0.11 (rac)	- -	< 0.05 < 0.05	75FL84-023 II: [Francis, 1985, 434142, TMU1815B] X: [Morgan and Crook, 1986, PP5/0250, M4266B]
Dry harvested onion bulbs	Donna, TX, USA, 1984 2 × 0.42 kg ai/ha (racemate)	39 39	0.04 (P) < 0.02 (rac)	- -	< 0.05 < 0.05	71TX83-037 II: [Francis, 1985, 434142, TMU1815B] X: [Morgan and Crook, 1986, PP5/0250, M4266B]
Dry harvested onion bulbs	Mission, TX, USA, 1984 2 × 0.42 kg ai/ha (racemate; R-enantiomer)	46 46 46	0.04 (P) 0.04 (P) 0.03 (rac)	- - -	< 0.05 < 0.05 < 0.05	71TX83-056 II: [Francis, 1985, 434142, TMU1815B] X: [Morgan and Crook, 1986, PP5/0250, M4266B]
Dry harvested onion bulbs	Visalia, CA, USA, 1984 2 × 0.42 kg ai/ha (racemate; R-enantiomer)	45 45	0.18 (P) 0.12 (rac)	- -	< 0.05 < 0.05	US2-83-S14 II: [Francis, 1985, 434142, TMU1815B] X: [Morgan and Crook, 1986, PP5/0250, M4266B]
Dry harvested onion bulbs	Visalia, CA, USA, 1984 2 × 0.42 kg ai/ha (racemate; R-enantiomer)	45 45	0.16 (P) 0.07 (rac)	- -	< 0.05 < 0.05	US2-83-S15 II: [Francis, 1985, 434142, TMU1815B] X: [Morgan and Crook, 1986, PP5/0250, M4266B]
Dry harvested onion bulbs	location ns; USA, 1986; 2 × 0.42 kg ai/ha (R-enantiomer)	45	0.02 (P)	-	< 0.01	NCA/87/204 IIandX: [Hayward, 1987, PP5/0251; M4545B]
Dry harvested onion bulbs	location ns; USA, 1986; 2 × 0.42 kg ai/ha (R-enantiomer)	45	0.13 (P)	-	< 0.01	NCA/87/224 IIandX: [Hayward, 1987, PP5/0251; M4545B]
Green onions	CA, USA, 1982 2 ×1.1 kg ai/ha (racemate)	14 21	No study available	0.10 0.08	0.18 0.23	38CA82-005; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Cucumbers	FL, USA, 1981; 2 × 0.28 kg ai/ha (racemate)	10	no study available	0.21	-	53FL81-057; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Cucumbers	FL, USA, 1981; 2 × 0.56 kg ai/ha (racemate)	10	no study available	0.41; 0.56	-	53FL81-057; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Cucumbers	FL, USA, 1981; 2 × 0.28 kg ai/ha (racemate)	11	no study available	0.29	-	53FL81-047; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
Cucumbers	FL, USA, 1981; 2 × 0.56. kg ai/ha (racemate)	11	no study available	0.39	-	53FL81-047; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Cucumbers	FL, USA, 1981; 2 × 0.50 kg ai/ha (racemate)	11	no study available	< 0.05	-	53FL81-046; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Cucumbers	FL, USA, 1981; 2 × 1.1 kg ai/ha (racemate)	11	no study available	< 0.05	-	53FL81-046; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Cucumbers	FL, USA, 1982; 1× 1.1 kg ai/ha (racemate)	15	no study available	0.55 0.62	- -	53FL82-046; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Lettuce	FL, USA, 1981; 2 × 0.56 kg ai/ha (racemate)	32	no study available	0.13	-	53FL81-050; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Lettuce	FL, USA, 1981, 2 × 0.56 kg ai/ha (racemate)	21 30	no study available	0.07 0.05	- -	53FL81-053 III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Lettuce	FL, USA, 1981, 2 × 0.56 kg ai/ha; (racemate)	18 32	no study available	0.16 0.14	- -	53FL81-052 III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Lettuce	FL, USA, 1981, 2 × 0.56 kg ai/ha; (racemate)	34	no study available	0.06	-	53FL81-054 III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Lettuce	FL, USA, 1982; 2 × 0.28 kg ai/ha (racemate)	21 21 30	no study available	0.10; 0.09; < 0.05	- - -	53FL82-004; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Lettuce	FL, USA, 1982; 2 × 0.56 kg ai/ha (racemate)	21 21 30	no study available	0.23; 0.32; < 0.05	- - -	53FL82-004; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Lettuce	FL, USA, 1982; 1× 1.1 kg ai/ha (racemate)	69	no study available	< 0.05	-	53FL82-004; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Lettuce	FL, USA, 1982; 2 × 0.56 kg ai/ha (racemate)	30	no study available	< 0.05	-	53FL82-028; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Lettuce	FL, USA, 1982, 2 × 0.56 kg ai/ha; (racemate)	13 18	no study available	0.17 0.15	- -	53FL82-009 III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
Dry soya seeds	USA, 1979; 1× 0.56 kg ai/ha (racemate)	96	0.39 (rac)	-	< 0.03 (free only)	HU5-79-05; II: [Atreya *et al*., 1981, PP9/0736, report PP009B036] X: [Atreya *et al*., 1981, PP9/0733, report PP009B061]
Dry soya seeds	LA, USA, 1980 2 × 0.28 kg ai/ha (racemate)	73 73	0.39 (rac); 0.45 (rac)	- -	< 0.04; < 0.05	36LA80-008; II: [Atreya *et al*., 1981, PP9/0736, report

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
					(free only)	PP009B036] X: [Atreya *et al*., 1981, PP9/0733, report PP009B061]
Dry soya seeds	MO, USA, 1980; 2 × 0.56 kg ai/ha (racemate)	105	< 0.05 (rac)	-	< 0.03 (free only)	48MO80-009; II: [Atreya *et al*., 1981, PP9/0736, report PP009B036] X: [Atreya *et al*., 1981, PP9/0733, report PP009B061]
Carrot roots	Zellwood, FL, USA, 1981-82 2 × 0.28 kg ai/ha (racemate)	19 36	0.35 (rac) 0.11 (rac)	< 0.05 -	- -	53FL81-043 II: [Koubek, 1982, 406305, report TMU0902/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
idem	idem	19	0.28 (rac)	< 0.05	-	53FL81-043 II, III, X [Atreya *et al*., 1984, PP9/0065, report PP009B300] (other hydrolysis conditions for III)
Carrot roots	Zellwood, FL, USA, 1981-82 2 × 0.56 kg ai/ha (racemate)	19 36	0.67 (rac) 0.19 (rac)	< 0.05; < 0.05;	< 0.05 < 0.05	53FL81-043 II: [Koubek, 1982, 406305, report TMU0902/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
idem	idem	19 36	0.71 (rac) 0.21 (rac)	< 0.05 < 0.05	< 0.05 < 0.05	53FL81-043 II, III, X [Atreya *et al*., 1984, PP9/0065, report PP009B300] (other hydrolysis conditions for III)
Carrot roots	Zellwood, FL, USA, 1981-82 3× 0.28 kg ai/ha (racemate)	19 36	0.20 (rac) 0.07 (rac)	< 0.05 -	- -	53FL81-043 II: [Koubek, 1982, 406305, report TMU0902/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]
idem	idem	19	0.16 (rac)	< 0.05	-	53FL81-043 II, III, X [Atreya *et al*., 1984, PP9/0065, report PP009B300] (other hydrolysis conditions for III)
Carrot roots	Zellwood, FL, USA, 1981 3× 0.56 kg ai/ha (racemate)	19 36	0.65 (rac) 0.22 (rac)	< 0.05 < 0.05	- < 0.05	53FL81-043 II: [Koubek, 1982, 406305, report TMU0902/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
						PP009B290]
idem	idem	19 36	0.59 (rac) 0.17	< 0.05 < 0.05	- < 0.05	53FL81-043 II, III, X [Atreya *et al*., 1984, PP9/0065, report PP009B300] (other hydrolysis conditions for III)
Carrot roots	Zellwood, FL, USA, 1982 2 × 0.56 kg ai/ha (racemate)	27 43 50	0.05 (rac) - -	- - < 0.05	- - < 0.05	53FL82-022 II: [Koubek, 1982, 406305, report TMU0902/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
idem	idem	50	< 0.05	< 0.05	< 0.05	53FL82-022 II, III, X [Atreya *et al*., 1984, PP9/0065, report PP009B300] (other hydrolysis conditions for III)
Carrot roots	Belle Glade, FL, USA, 1981-82 2 × 0.56 kg ai/ha (racemate)	29	0.16 (rac)	< 0.05	< 0.05	53FL82-003 II: [Koubek, 1982, 406305, report TMU0902/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
idem	idem	29	0.12 (rac)	< 0.05	< 0.05	53FL82-003 II, III, X [Atreya *et al*., 1984, PP9/0065, report PP009B300] (other hydrolysis conditions for III)
Carrot roots	Belle Glade, FL, USA, 1981-82 3× 0.56 kg ai/ha (racemate)	20	0.34 (rac)	< 0.05	< 0.05	53FL82-003 II: [Koubek, 1982, 406305, report TMU0902/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
idem	idem	20	0.35 (rac)	< 0.05	< 0.05	53FL82-003 II, III, X [Atreya *et al*., 1984, PP9/0065, report PP009B300] (other hydrolysis conditions for III)
Carrot roots	Belle Glade, FL, USA, 1982 2 × 0.56 kg ai/ha (racemate)	24 44	< 0.03 (rac) -	< 0.05 < 0.05	< 0.05 < 0.05	53FL82-029 II: [Koubek, 1982, 406305, report TMU0902/B]; III: [Atreya and Dick, 1984, PP9/0728, report PP009B272]

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
						X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
idem	idem	24 44	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	53FL82-029 II, III, X [Atreya *et al*., 1984, PP9/0065, report PP009B300] (other hydrolysis conditions for III)
Carrot roots	Visalia, CA, USA, 1983, 2 × 0.42 kg ai/ha (R-enantiomer)	45	no study available	-	< 0.05	US2-83-524 X: [Dick and Rounds., 1985, PP5/0238; M4041B]
Carrot roots	Mission, TX, USA, 1983, 2 × 0.42 kg ai/ha (R-enantiomer)	30 30 45 45	0.13 0.12 0.04 0.04	- - - -	- - < 0.05 -	71TX-83-055; II: [Francis 1985, 406311, report TMU1812B]; X: [Dick and Rounds., 1985, PP5/0238; M4041B];
Carrot roots	Santa Rosa, TX, USA, 1983, 2 × 0.42 kg ai/ha (R-enantiomer)	31 31 45 45	0.09 0.06 0.04; 0.05 [cntrl =0.07]	- - - -	- - < 0.05; < 0.05	71TX-83-044; II: [Francis 1985, 406311, report TMU1812B]; X: [Dick and Rounds., 1985, PP5/0238; M4041B];
Carrot roots	South Bay, FL, USA, 1984, 2 × 0.42 kg ai/ha (R-enantiomer)	29 44 44	0.08 - < 0.03	- - -	- < 0.05; < 0.05	75FL-84-004; II: [Francis 1985, 406311, report TMU1812B]; X: [Dick and Rounds., 1985, PP5/0238; M4041B];
Carrot roots	Zellwood, FL, USA, 1984, 2 × 0.42 kg ai/ha (R-enantiomer)	31 48 48	0.08 - < 0.03	- - -	- < 0.05; < 0.05	75FL-84-032; II: [Francis 1985, 406311, report TMU1812B]; X: [Dick and Rounds., 1985, PP5/0238; M4041B];
Sugar beet roots	Longmont, CO, USA, 1981 1×1.1 kg ai/ha (racemate)	33 132	0.34 0.02	- -	0.08 < 0.06	37CO81-049 II: [Koubek, 1983, 405726, report TMU1211/B] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Sugar beet roots	Chico, CA, USA, 1982 2 ×1.1 kg ai/ha (racemate)	60	0.37	-	0.09	41CA82-041 II: [Koubek, 1983, 405726, report TMU1211/B] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Sugar beet roots	Longmont, CO, USA, 1982 2 ×1.1 kg ai/ha (racemate)	59	0.39	-	0.07	37CO82-052 II: [Koubek, 1983, 405726, report TMU1211/B] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
Sugar beet roots	Ft Collins, CO, USA, 1982 2 × 0.84 kg ai/ha (racemate)	60	0.12	-	< 0.06	37CO82-031 II: [Koubek, 1983, 405726, report TMU1211/B] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Sugar beet roots	Aberdeen, ID, USA, 1982 2 ×1.1 kg ai/ha (racemate)	56	0.15	-	< 0.06	32ID82-008 II: [Koubek, 1983, 405726, report TMU1211/B] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Sugar beet roots	Ontario, OR, USA, 1982 2 ×1.1 kg ai/ha (racemate)	129	< 0.06	-	< 0.06	32OR82-071 II: [Koubek, 1983, 405726, report TMU1211/B] X: [Atreya and Upton, 1984, PP9/0731, report PP009B290]
Sugarbeet roots	Hillsboro, ND, USA, 1984, 2 × 0.42 kg ai/ha (R-enantiomer)	103	no study available	-	< 0.02	64ND-84-094 X: [Dick and Rounds., 1985, PP5/0238; report M4041B];
Sugarbeet roots	Scotts Bluff, NE, USA, 1984, 2 × 0.42 kg ai/ha (R-enantiomer)	86	no study available	-	< 0.02	52NB-84-073; X: [Dick and Rounds., 1985, PP5/0238; report M4041B];
Sugarbeet roots	Fort Collins, CO, USA, 1984, 2 × 0.42 kg ai/ha (R-enantiomer)	99	no study available	-	< 0.02	37CO-84-057; X: [Dick and Rounds., 1985, PP5/0238; report M4041B];
Celery	Mission, TX, USA, 1986 2 × 0.42 kg ai/ha (R-enantiomer)	30	0.66 [a]	-	< 0.04 [b]	60TX-907R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Celery	Belle Glade, FL, USA, 1986 2 × 0.42 kg ai/ha (R-enantiomer)	30	0.52 [a]	-	< 0.04 [b]	75FL86-930R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Celery	Belle Glade, FL, USA, 1986, 2 × 0.42 kg ai/ha (R-enantiomer)	30	0.28 [a]	-	< 0.04 [b]	75FL86-931R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Celery	Romney, WV, USA, 1986 2 × 0.42 kg ai/ha (R-enantiomer)	28	2.3 [a]	-	0.06 [b]	15WV86-909R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Celery	Waterloo, NY, USA, 1986, 2 × 0.42 kg ai/ha (R-enantiomer)	30	1.2 [a]	-	< 0.04 [b]	34NY86-912R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Celery	Westmorland, CA, USA, 1986 2 × 0.42 kg ai/ha (R-enantiomer)	30	2.7 [a]	-	0.08 [b]	38CA86-910R IIandX: [Watford and Francis, 1988, PP5/0323, report

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
						TMU3418/B]
Celery	Westmorland, CA, USA, 1986 2 × 0.42 kg ai/ha (R-enantiomer)	30	2.5 [a]	-	0.08 [b]	38CA86-910R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Celery	Salina, CA, USA, 1986 2 × 0.42 kg ai/ha (R-enantiomer)	30	0.96 [a]	-	< 0.04 [b]	45CA86-911R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Celery	Delmar, DE, USA, 1986 2 × 0.42 kg ai/ha (R-enantiomer)	30	1.2 [a]	-	0.08 [b]	44DE86-905R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Celery	Berlin, WI, USA, 1986 2 × 0.42 kg ai/ha (R-enantiomer)	28	0.20 [a]	-	< 0.04 [b]	52WI86-902R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Celery	Pyallup, WA, USA, 1986 2 × 0.42 kg ai/ha (R-enantiomer)	30	0.52 [a]	-	< 0.04 [b]	32WA86-926R IIandX: [Watford and Francis, 1988, PP5/0323, report TMU3418/B]
Cotton seeds	Centre Point, TX, USA, 1980 2 × 0.28 kg ai/ha (racemate)	110 110	0.03 0.02	< 0.05 < 0.05	< 0.04 < 0.07 (free only)	20TX80-007; II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035] III: [Ussary, 1981, 405793; report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042] X: [Atreya *et al*., 1981, PP9/0733, report PP009B061]
Cotton seeds	Tipton, CA, USA, 1980 2 × 0.56 kg ai/ha (racemate)	136 136	0.04 < 0.02	< 0.05 < 0.05	< 0.03; < 0.05 (free only)	41CA80-004; II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035] III: [Ussary, 1981, 405793; report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042] X: [Atreya *et al*., 1981, PP9/0733, report PP009B061]
Cotton seeds	Tulleson, AZ, USA, 1980 2 × 0.56 kg ai/ha, (racemate)	154	0.03	< 0.05	-	42AZ80-001 II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035]

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
						III: [Ussary, 1981, 405793; report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042]
Cotton seeds	Vicksburg, MS, USA, 1979 2 × 0.28 kg ai/ha, (racemate)	133	< 0.02	< 0.05	-	HU5-79-04 II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035] III: [Ussary, 1981, 405793; report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042]
Cotton seeds	Visalia, CA, USA, 1979, 1×1.1 kg ai/ha (racemate)	147	< 0.02	< 0.05	-	HU2-79-10 II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035] III: [Ussary, 1981, 405793; report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042]
Cotton seeds	Idalon, TX, USA, 1980 2 × 0.28 kg ai/ha (racemate)	103 103	< 0.02 0.02	< 0.05 < 0.05	-	33TX80-001 II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035] III: [Ussary, 1981, 405793; report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042]
Cotton seeds	Sunnyside, MS, USA, 1980 2 × 0.28 kg ai/ha (racemate)	83 83	< 0.02 0.02	< 0.05 < 0.05	-	29MS80-017 II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035] III: [Ussary, 1981, 405793; report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042]
Cotton seeds	Rosedale, CA, USA, 1980, 2 × 0.56 kg ai/ha (racemate)	179 179	< 0.02 < 0.02	< 0.05 < 0.05	-	41CA80-003 II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035] III: [Ussary, 1981, 405793; report

Commodity	Trial information	DAT	Total II fluazifop (mg/kg)	Total III despyridinyl acid (mg/kg)	Total X CF3-pyridone (mg/kg)	Trial; References
						TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042]
Cotton seeds	Goldsboro, NC, USA, 1980 1×1.1 kg ai/ha (racemate)	193	< 0.02	< 0.05	-	RU1-80-01 II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035] III: [Ussary, 1981, 405793; report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042]
Cotton seeds	Cochouran, GA, USA, 1980 2 × 0.28 kg ai/ha (racemate)	105 105	0.02 0.02	< 0.05 < 0.05	-	28GA80-001 II: [Ussary, 1981, 405792, report TMU0679/B; Atreya *et al*., 1981, PP9/0734; report PP009B035] III: [Ussary, 1981, 405793; report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042]
Sunflower seeds	Petersburg, Hale, TX, USA, 1989, 2 x 0.56 kg ai/ha (R-enantiomer)	66	0.37	-	0.01	13TX89-851 IIand X: [Alferness and Kleinschmidt, 1991, PP5/0233, report RR 91-010B]
Sunflower seeds	Mooreton, Richland, ND, USA, 1989 2 x 0.56 kg ai/ha (R-enantiomer)	99	< 0.01	-	< 0.01	33ND89-852 IIand X: [Alferness and Kleinschmidt, 1991, PP5/0233, report RR 91-010B]

[a] Results corrected by the average internal standard recovery

[b] Results corrected by the average external recovery value of Ref. X with each run.

TMU1257/B and PP009B272 and PP009B290 (dry and green onions): Total fluazifop was determined by **HPLC-UV method PPRAM 62** with a valid LOQ of 0.05 mg/kg. Concurrent recoveries were 98% at 0.05-1.0 mg/kg (fluazifop acid) or 91% (fluazifop-butyl). Control < 0.04 mg/kg. Total despyridinyl acid (III) was determined by **GC-MS method Ref III modification A**. Concurrent recoveries were not reported. Control samples were 0.10 mg/kg (dry onions). Total CF3-pyridone (X) was determined by **NMR method PPRAM 103**. Concurrent recoveries were not reported. Control samples < 0.05 mg/kg. Since reflux conditions are used to release despyridinyl acid (III) and CF3-pyridone (X), results are considered not reliable.

TMU1815B and M4266B (onions): Total fluazifop was determined by **HPLC-UV method PPRAM 62**. Individual concurrent recoveries for fluazifop acid were 81–108% at 0.03-0.8 mg/kg. Control samples < 0.03 mg/kg (n=8). Total CF3-pyridone (X) was determined by **NMR method PPRAM 103 modification C**. Residues were corrected for recoveries (80–93% at 0.2 mg/kg); uncorrected results were not available. Control samples < 0.05 mg/kg. Since reflux conditions are used to release CF3-pyridone (X), results are considered not reliable.

M4545B (onions) Total fluazifop was determined with **HPLC-UV method PPRAM 62/2**. Concurrent method recoveries or results in control samples were not recorded. Total CF3-pyridone (X) was determined by **NMR method PPRAM 103 modification B**. Concurrent method recoveries or results in control samples were not recorded. Since reflux conditions are used to release CF3-pyridone (X), results are considered not reliable

PP009B272 (cucumbers): Total despyridinyl acid (III) was determined by **GC-MS method Ref III modification A**. Concurrent recoveries were not reported. Control samples were < 0.05 mg/kg. Since reflux conditions are used to release despyridinyl acid (III), results are considered not reliable

PP009B272 (lettuce): Total despyridinyl acid (III) was determined by **GC-MS method Ref III modification A**. Concurrent recoveries were not reported. Control samples were < 0.05 mg/kg. Since reflux conditions are used to release despyridinyl acid (III), results are considered not reliable

PP009B036 and PP009B061 (soya): Total fluazifop was determined by **HPLC-UV method PPRAM 62**. Concurrent recovery (66–80% at 0.05-0.5 mg/kg). Control samples < 0.02 to < 0.05 mg/kg. CF3-pyridone (X) was analysed using **NMR method PP009B061.** This method only determines the free CF3-pyridone (X); conjugates are not analysed. Concurrent recovery was 80% at unstated levels. Control samples were not reported.

TMU0902B and PP009B272 and PP009B290 (carrots): Total fluazifop was determined by HPLC-UV method PPRAM 62/2, with minor modifications. Individual concurrent recoveries for fluazifop acid were 86–89% (0.08-0.5 mg/kg). Control samples were < 0.02 mg/kg. Total despyridinyl acid (III) was determined by **GC-MS method Ref III modification A**. Concurrent recoveries were not reported. Control samples were < 0.05 mg/kg. Total CF3-pyridone (X) was determined by **NMR method PPRAM 103**. Concurrent recoveries were not reported. Control samples < 0.05 mg/kg. Since reflux conditions are used to release despyridinyl acid (III) and CF3-pyridone (X), results are considered not reliable

PP009B300 (carrots) Total fluazifop and despyridinyl acid (III) were determined by **GC-MS or HPLC-UV method ref III modification B**. Concurrent recoveries were not reported. Control samples were < 0.05 mg/kg. Total CF3-pyridone (X) was determined by **NMR method PPRAM 103**. Concurrent recoveries were not reported. Control samples < 0.05 mg/kg. Total fluazifop and despyridinyl acid (III) are determined in the same sample by 1 hour 6 M HCl at 60 (acceptable hydrolysis conditions); CF3-pyridone (X) is released using reflux conditions, thereby overestimating the CF3-pyridone (X) residues.

TMU1812B and M4041B (carrots). Total fluazifop was determined by **HPLC-UV method PPRAM 62/2**, with minor modifications. Indivicual concurrent recoveries for fluazifop acid were 70–113% (0.04-0.6 mg/kg). Residues were corrected for recoveries; uncorrected results are not reported. Control samples were < 0.03 mg/kg, except for trial 71TX83-044 where residues were 0.07 mg/kg. Total CF3-pyridone (X) was determined by **NMR method PPRAM 103**. Residues were corrected for recoveries (100–129% at 0.2 and 0.5 mg/kg); uncorrected results are not reported. Control samples were < 0.02 mg/kg (carrots) or < 0.05 mg/kg (sugarbeet roots). Since reflux conditions are used to release CF3-pyridone (X), results are considered not reliable

TMU1211/B and PP009B290 (sugar beet roots): Total fluazifop was determined by **HPLC-UV method PPRAM 62** with a valid LOQ of 0.05 mg/kg. Average concurrent recoveries were 101% (fluazifop acid) or 97% (fluazifop-butyl). Control < 0.02 to < 0.06 mg/kg. Total CF3-pyridone (X) was determined by **NMR method PPRAM 103**. Concurrent recoveries were not reported. Control samples < 0.06 mg/kg. Since reflux conditions are used to release CF3-pyridone (X), results are considered not reliable

M4041B (sugarbeet roots). Total CF3-pyridone (X) was determined by **NMR method PPRAM 103**. Residues were corrected for recoveries (100–129% at 0.2 and 0.5 mg/kg); uncorrected results are not reported. Control samples were < 0.02 mg/kg (carrots) or < 0.05 mg/kg (sugarbeet roots). Since reflux conditions are used to release CF3-pyridone (X), results are considered not reliable

TMU3418B (celery): Total fluazifop and total CF3-pyridone (X) were determined by **NMR method TMU4318B**. Concurrent recoveries were 80–90% for fluazifop acid (0.05-0.5 mg/kg) and 58–62% for CF3-pyridone (X, 0.05-0.1 mg/kg). Control samples were < 0.04 mg/kg for each analyte. Since recovery for CF3-pyridone (X) was not satisfactory, CF3-pyridone (X) results were corrected for concurrent method recoveries; uncorrected results were not indicated.

TMU0679B and TMU0680B and PP009B061 (cotton): Total fluazifop was determined by HPLC-UV method PPRAM 53 modified with a hydrolysis step, i.e. PPRAM 62. Samples were corrected for average concurrent recovery (99% at 0.1 mg/kg); uncorrected results were not available. Control samples < 0.02 mg/kg. Total despyridinyl acid (III) was determined by **GC-MS method ref III.** Concurrent recoveries were not reported. Control samples were < 0.05 mg/kg. Since reflux conditions are used to release despyridinyl acid (III), results are considered not reliable CF3-pyridone (X) was analysed using **NMR method PP009B061**. This method only determines the free CF3-pyridone (X); conjugates are not analysed. Concurrent recovery was 80% at unstated levels. Control samples were not reported.

RR 91-010B (sunflower seeds). Total fluazifop was determined by modification A of **GC-MS method RR89-073B**. Average concurrent method recoveries were 76-101% (0.01–0.1 mg/kg) in sunflower seed and its processed commodities. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (<0.01 mg/kg). Total CF3-pyridone (X) was determined using method **GC-MS method R90-384B.** Average concurrent method recoveries were 71-93% (0.01–0.1 mg/kg) in sunflower seed and its processed commodities. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (<0.01 mg/kg). Since reflux conditions are used to release CF3-pyridone (X), results are considered not reliable

Overview metabolic pathway of fluazifop-P-butyl in plants

Metabolism of fluazifop-P-butyl in plants after foliar and/or soil application has been studied in five different representative crop groups –fruits and fruiting vegetables (grapes, cucumbers), leafy vegetables (lettuce, celery, endive), cereals (maize forage), pulses and oilseeds (alfalfa forage, cotton forage and seeds, oilseed rape seeds, soya bean forage and seeds) and root and tuber vegetables

(carrot roots, potato tubers, sugarbeet roots). An overview of the studies with acceptable hydrolysis conditons is given in Table 40.

Table 40 Overview of metabolism studies with acceptable hydrolysis conditions

							Parent (I) and metabolites as %TRR								
No	Crop	Treatment	Label		PHI	TRR mg/kg	I	II	I+ II	IV	III	X	34	40	NH
2	cucumber	1× 0.50 kg ai/ha foliar spray	Ph	RS	1	1.3	7.3	69	76	-	-	nr	na	na	24
2	cucumber	1× 0.50 kg ai/ha foliar spray	Ph	RS	14	4.9	-	72	72	-	3.3	nr	na	na	17
2	cucumber	1× 0.52 kg ai/ha foliar spray	Py	RS	14	2.4	-	69	69	-	nr	-	na	na	11
9	rape seeds	1 × 0.84 kg ai/ha; topical leaf and stem and soil	Py	RS	70-91	0.65	-	69	69	-	nr	D (-)	na	na	6
15	soya seeds	1×1.0 kg ai/ha; pods present broadcast	Ph	RS	63	11	-	77	77	na	3.7	nr	na	na	10
15	soya seeds	1×1.0 kg ai/ha; pods present broadcast	Ph	RS	43	6.0	-	81	81	na	na	nr	na	na	13
16	soya seeds	1 × 0.56 kg ai/ha; BBCH 15 broadcast	Ph	R	104	0.04	-	50	50	-	2.3	nr	na	na	19
16	soya seeds	1 × 0.56 kg ai/ha; BBCH 15 broadcast	Py	R	104	0.09	-	40	40	-	nr	D (-)	na	na	24
16	soya seeds	0.56+0.21 kg ai/ha; BBCH 69 broadcast	Ph	R	82	0.57	-	57	57	-	3.9	nr	na	na	13
16	soya seeds	0.56+0.21 kg ai/ha; BBCH 69 broadcast	Py	R	82	1.0	0.2	59	60	-	nr	D 0.9	na	na	16
17	carrot roots	1 × 0.25 kg ai/ha; broadcast	Ph	R	45	0.15	-	63	63	-	6.4	nr	-	na	12
17	carrot roots	1 × 0.53 kg ai/ha; broadcast	Ph	RS	45	0.18	-	46	46	-	4.8	nr	13	na	23
17	carrot roots	1 × 0.51 kg ai/ha; broadcast	Py	RS	45	0.33	-	44	44	-	nr	1.0	11	na	25
18	carrot roots	1 × 0.42 kg ai/ha; broadcast	Ph	R	20	0.38	0.5	62	62	-	13	nr	na	na	25
18	carrot roots	1 × 0.43 kg ai/ha broadcast	Py	R	20	0.54	-	49	49	-	nr	37	na	na	14
18	carrot roots	0.42+0.42 kg ai/ha; broadcast	Ph	R	45	0.091	-	64	64	-	18	nr	na	na	19
18	carrot roots	0.42+0.42 kg ai/ha; broadcast	Py	R	45	0.13	-	59	59	-	nr	29	na	na	12
19	potato tubers	1 × 0.86 kg ai/ha; topical leaf and soil	Ph	RS	56	0.37	-	42	42	-	18	nr	na	13	15
19	potato tubers	1 × 0.84 kg ai/ha; topical leaf and soil	Py	RS	56	0.29	-	25	25	-	nr	-	na	15	30
20	sugarbeet roots	1×2.8 kg ai/ha; topical leaf and soil	Ph	RS	87	0.049	-	25	25	-	18	nr	na	na	4
21	sugarbeet roots	1 × 0.25 kg ai/ha; broadcast	Ph	R	90	0.09	-	52	52	-	17	nr	-	na	18
21	sugarbeet roots	1 × 0.52 kg ai/ha;broadcast	Ph	RS	90	0.08	-	40	40	-	15	nr	-	na	17
21	sugarbeet roots	1 × 0.51 kg ai/ha; broadcast	Py	RS	90	0.20	-	34	34	-	NR	IC 3.4	-	na	18
4	celery stems	0.45+0.18 kg ai/ha; broadcast	Ph	R	30	0.05	-	43	43	na	18	nr	1.0	4.4	1
4	celery stems	0.42+0.36 kg ai/ha; broadcast	Py	R	30	0.08	-	39	39	na	nr	2.7	-	1.2	8

							Parent (I) and metabolites as %TRR								
No	Crop	Treatment	Label		PHI	TRR mg/kg	I	II	I+II	IV	III	X	34	40	NH
3	lettuce	1× 0.45 kg ai/ha; topical leaf and stem	Ph	R	27	NA	52	19	71	0.4	8.7	nr	-	na	5
3	lettuce	1× 0.45 kg ai/ha;topical leaf and stem	Ph	S	27	NA	49	19	68	1.7	4.1	nr	5.3	na	7
5	endive	1× 0.42 kg ai/ha; broadcast	Ph	R	20	0.65	-	48	48	11	25	nr	na	na	3
5	endive	1× 0.42 kg ai/ha; broadcast	Py	R	20	0.88	-	37	37	25	nr	14	na	na	12
5	endive	0.42+0.42 kg ai/ha; broadcast	Ph	R	28	1.4	-	49	49	0.5	40	nr	na	na	1
5	endive	0.42+0.42 kg ai/ha; broadcast	Py	R	28	1.8	-	43	43	-	nr	11	na	na	2
4	celery leaves	0.45+0.18 kg ai/ha; broadcast	Ph	R	30	0.31	2.4	52	54	na	7.1	nr	0.3	1.6	-
4	celery leaves	0.42+0.36 kg ai/ha; broadcast	Py	R	30	0.64	-	63	63	na	nr	14	-	0.7	6
11	maize forage	dose rate ns; topical stem injection	Ph	RS	1	NA	15	65 fr	80	na	na	nr	na	na	20
11	maize forage	dose rate ns; topical stem injection	Ph	RS	7	NA	40	25 fr	65	na	na	nr	na	na	35
6	alfalfa forage	1 × 0.49 kg ai/ha; foliar spray	Ph	RS	20	3.2	-	70	70	-	-	nr	na	na	6
6	alfalfa forage	1 × 0.49 kg ai/ha; foliar spray	Py	RS	20	2.5	-	70	70	-	nr	na	na	na	6
6	alfalfa forage	1 × 0.49 kg ai/ha; foliar spray	Ph	RS	87	0.13	-	37	37	-	-	nr	na	na	13
7	cotton forage	1 × 0.45 kg ai/ha;topical leaf and stem	Ph	R	27	NA	24	38	61	2.7	7.3	nr	-	na	11
7	cotton forage	1 × 0.45 kg ai/ha; topical leaf and stem	Ph	S	27	NA	23	56	79	2.5	1.5	nr	-	na	6
10	soya forage	1 × 0.75 kg ai/ha; topical leaf and stem	Ph	RS	1	NA	15	40 fr	55	na	na	nr	na	na	45
10	soya forage	1 × 0.75 kg ai/ha; topical leaf and stem	Ph	RS	2	NA	1.0	50 fr	51	na	na	nr	na	na	49
10	soya forage in nutrient	1 × 0.75 kg ai/ha; topical leaf and stem	Ph	RS	6	NA	-	76	76	na	na	nr	na	na	12
10	soya forage in nutrient	1 × 0.75 kg ai/ha; topical leaf and stem	Ph	RS	29	NA	-	15	15	na	na	nr	na	na	85
11	soya forage	dose rate ns; topical stem injection	Ph	RS	1	NA	65	15 fr	80	na	na	nr	na	na	20
16	soya forage	1 × 0.56 kg ai/ha; BBCH 15 broadcast	Ph	R	22	5.2	0.2	71	72	0.3	-	nr	na	na	2
16	soya forage	1 × 0.56 kg ai/ha; BBCH 15 broadcast	Py	R	22	4.3	-	70	70	0.2	nr	D 0.2	na	na	3
18	carrot foliage	1 × 0.42 kg ai/ha; broadcast	Ph	R	20	0.86	-	82	82	-	1.7	nr	na	na	16
18	carrot foliage	1 × 0.42 kg ai/ha; broadcast	Py	R	20	1.3	-	42	42	-	nr	48	na	na	10
18	carrot foliage	0.42+0.42 kg ai/ha; broadcast	Ph	R	45	1.0	-	82	82	-	5.9	nr	na	na	13
18	carrot foliage	0.42+0.42 kg ai/ha;broadcast	Py	R	45	1.5	-	47	47	-	nr	31	na	na	22

I = fluazifop-butyl; II = fluazifop acid; I+II = total fluazifop; IV = Pyr=Ph ether; III = despyridinyl acid; X = CF3-pyridone;

34 = fluazifop alcohol (XXXIV); 40 = hydroxyfluazifop acid (XL);

NR = not relevant, because the label does not generate this metabolite;

NA = not analysed, since the reference standard for this compound was not included

- = not detected (reference standard for this compound was included in the analysis)

IC = incomplete hydrolysis, since CF3-pyridone N-pyridinyl conjugates need at least 6 M HCl for 3 hours at 60 °C to be released completely

D = degraded, since CF3-pyridone (X) degrades under alkaline conditions

Free = no hydrolysis used and therefore only the free fluazifop acid (II) is analysed

Label: Ph = phenyl label; Py = pyridinyl; RS = racemate, R=R-enantiomer; S= S-enantiomer

NH = total not hydrolysed; may contain conjugates of fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III) or CF3-pyridone (X)

These studies show that metabolism is similar in all five crop categories, but the quantity of the different metabolites is different between the fruit or seed parts, the leafy parts and the root parts of the crop. Table 41 gives an overview of the ratios between metabolites and total fluazifop. Fluazifop-p-butyl is phytotoxic to cereals resulting in the death of the crop within 28 days after treatment.

Table 41 Ratios between metabolite and total fluazifop in various metabolism studies

Crop group	Pyr-Ph ether (IV)	Despyridinyl acid (III)	CF3-pyridone (X)	Hydroxy fluazifop acid (XL)
	ratio to total fluazifop	ratio to total fluazifop	ratio to total fluazifop	ratio to total fluazifop
Cucumber	ND	3.3/71.6=0.046		
Lettuce	1.7/68=0.025	8.7/70.7=0.12	0.12 [a]	
Celery leaves		7.1/54.0=0.13	13.7/62.7=0.22	1.6/54.0=0.03
Endive immature	24.9/36.5=0.68	25.4/47.6=0.53	13.6/36.5=0.37	
Endive mature	0.5/49.4=0.010	40.4/49.4=0.82	10.9/43.3=0.25	
cotton forage	2.7/61.4=0.044	7.3/61.4=0.12	0.12 [a]	
soya forage	0.3/71.5=0.0042	ND	0.2/69.8= 0.003	
carrot foliage	ND	1.7/81.9=0.021	48.0/42.4=1.13	
carrot foliage	ND	5.9/81.6=0.072	31.1/47.4=0.66	
	Median = 0.010	**Median = 0.12**	**Median = 0.24**	**Median = 0.03**
	Max = 0.044	**Max = 0.82**	**Max= 1.13**	**Max =0.03**
	Mature crops			
Celery stems		18.5/43.2=0.43	2.7/39.4=0.07	4.4/43.2=0.10
soya beans		3.7/76.9=0.05	0.05 [a]	
soya beans		2.3/49.5=0.05	0.05 [a]	
soya beans		3.9/56.5=0.07	0.07 [a]	
		Median =0.05	**Mean = 0.05**	
		Max = 0.07	**Max = 0.07**	
carrot roots		6.4/63.1=0.10	0.10 [a]	
carrot roots		4.8/45.7=0.11	1.0/43.5=0.02	
carrot roots		12.9/61.5=0.21	37.0/48.6=0.76	
carrot roots		17.6/63.8=0.28	29.3/58.7=0.50	
potato tubers		18.2/41.5=0.44	ND	15.4/24.7=0.62
sugarbeet roots		17.1/52.1=0.33	0.33 [a]	
sugarbeet roots		15.2/40.4=0.38	3.4/33.8=0.10	
		Median = 0.28	**Median =0.33**	**Median = 0.62**
		Max = 0.44	**Max = 0.76**	**Max = 0.62**

[a] CF3-pyridone (X) degraded or not investigated; assumed to be at least as high as despyridinyl acid (III), since CF3-pyridone (X) and despyridinyl acid (III) are formed at the same time

Studies (5 and 7) with radiolabelled R and S-enantiomers in lettuce and cotton forage showed that the (R)/(S) ratio remained unchanged in the parent fluazifop-butyl, fluazifop acid (II) and fluazifop conjugates suggesting that no epimerisation occurred in the plant within 27 days of treatment or during sample extraction and alkaline or acid hydrolysis. Contrary, analysis of samples from supervised field trials treated with fluazifop-butyl (RS) showed an increase in the proportion of

the fluazifop acid R-enantiomer with a crop to crop variation in the rate and content of conversion. The total fluazifop proportion of the R-enantiomer remained approximately the same in carrot roots at 21 days after treatment (46–54%), but increased to 74–82% in apple at 35–49 days after treatment, 78% in head cabbage at 49 days after treatment, 62% in kale at 27–41 days after treatment, 69–77% in dry peas at 54 days after treatment and 76–84% in oilseed rape seeds.

Fluazifop-P-butyl hydrolyses rapidly on contact with the plant. Fluazifop-butyl is found at significant quantities at the day of application in all crops and up to 8 days in fruits, up to 20 days in roots, at least 27-30 days in leaves of leafy vegetables and forage of cereals, pulses and oilseeds, up to 82 days in seeds and up to 98 days in root forage.

The proposed metabolic pathway of fluazifop-P-butyl in plants is shown in Figure 2. The primary metabolite found in all plants was fluazifop acid (II), formed by the hydrolysis of the parent ester bond and subsequent O-debutylation. Fluazifop acid (II) is mobile thouroughout the plant and at harvest is found as the free acid or as conjugate esters. Fluazifop acid (II) is less abundant as a free metabolite and is converted to water soluble and organo soluble conjugates. In studies (5 and 18) on endive, carrot roots and carrot two hexosides (E-5 and E-7 = C-3) and a malonylhexoside (E-6 = C-2) conjugate of fluazifop acid (II) were identified. In a study (15) on soya seeds 1,2-dioleate, oleate-palmitate and glyceride esters of fluazifop acid (II) were identified (A1, A2, A3=compound 27, A4).

Secondary breakdown products of fluazifop acid (II), containing a single phenyl or pyridyl ring, are resulting from cleavage at the pyridyl-phenyl ether linkage: CF3-pyridone (X) and despyridinyl acid (III). CF3-pyridone (X) is found in free and conjugated forms. In a study (18) on carrots a malonylhexoside (C-4) was identified. Despyridinyl acid (III) is only found in conjugated form. In studies (5and18) on endive and carrots a hexoside (E-1, E-4 = C-1) was identified.

Other miscellaneous metabolites derived from fluazifop acid (II) were Pyr-Ph ether (IV), fluazifop alcohol (XXXIV), hydroxyfluazifop acid (XL) and compound 28. Pyr-Ph ether (IV) is derived from cleavage of the aliphatic ether bond and can also be found as conjugate (e.g,. E-4 in endive). Compound 28 is most likely formed from glutathione-S-transferase catalysed cleavage of the central ether bond to form a gluthatione conjugate of CF3-pyridone (X) followed by further break-down of the glutathione conjugate. Fluazifop alcohol (XXXIV) appears to be formed only from the (S)-enantiomer of fluazifop-butyl by acid reduction, although trace levels have been found with the R-enantiomer in celery stems and leaves (4) and in many studies the presence of fluazifop alcohol (XXXIV) has not been verified. Hydroxyfluazifop acid (XL) is formed by hydroxylation of the pyridyl group.

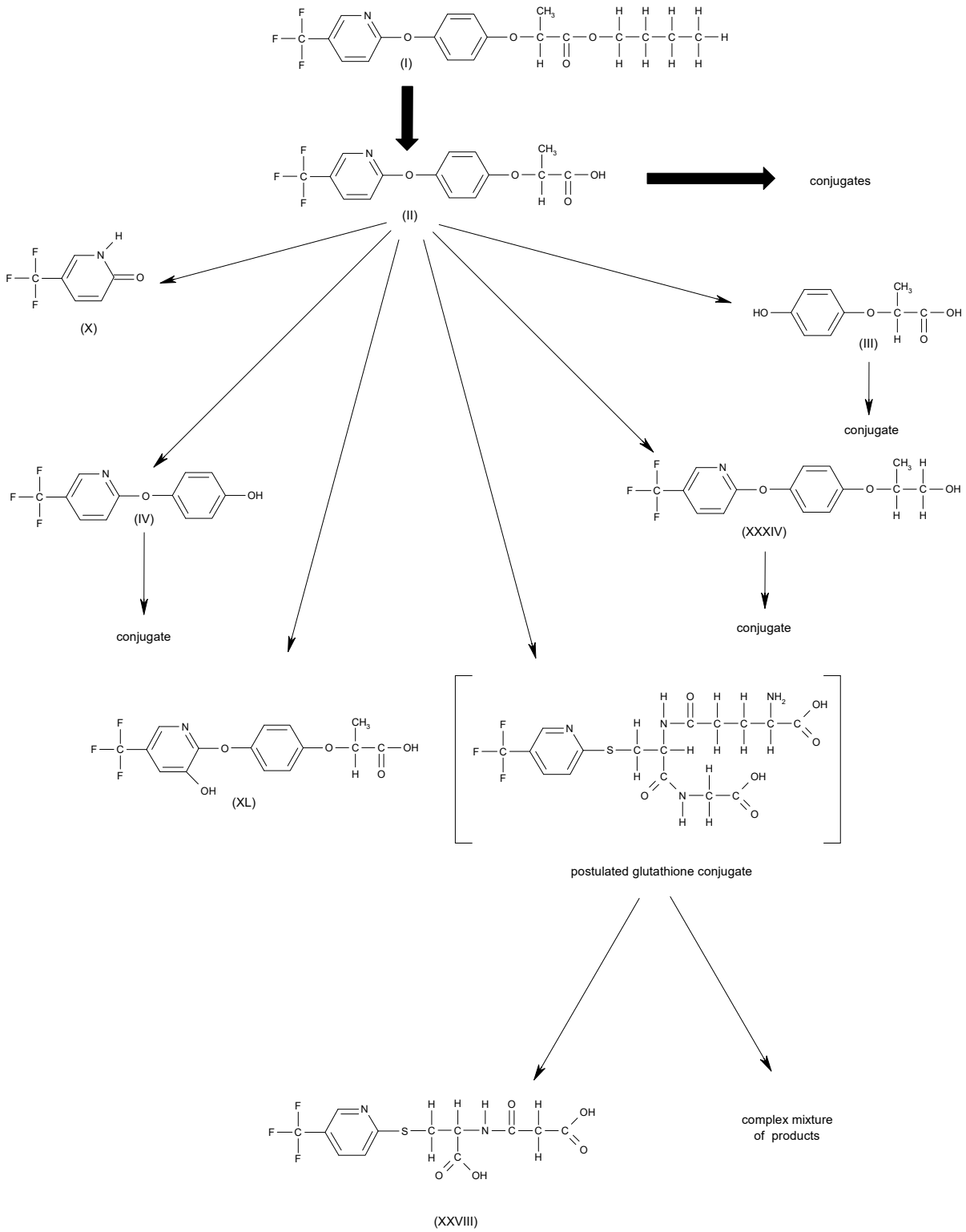


Figure 2 Proposed metabolic pathway of fluazifop-P-butyl in plants

Rotational crop studies

The Meeting received information on confined and field rotational crop studies. The fate and behaviour of fluazifop-P-butyl in the environment was investigated using [^{14}C-phenyl]-and ^{14}C-pyridyl labelled fluazifop-butyl (RS) or fluazifop-P-butyl (R-enantiomer) (see Figure 1).

Confined rotational crop study

Confined rotational study 1

^{14}C-Phenyl or ^{14}C-pyridyl labelled fluazifop-butyl (RS) were applied to a sandy loam soil and incorporated into the top 5 cm (in pots under greenhouse conditions) [Bell and Cavell, 1983, PP9/0175, report RJ0216B]. Soils were wetted to 40% of its maximum water holding capacity. Soil characteristics are shown in Table 42. The actual application rate was 0.25 kg ai/ha on 23 January 1980 (phenyl label) or 11 Sept 1980 (pyridyl label). Thouree different crops (spring wheat variety Sicco, sugar beet variety Amono and Cos lettuce variety Winter Density) were sown 30, 120, 327 days after soil treatment (phenyl label) or 60, 120, 365 days after soil treatment (pyridyl label). Two plants per pot were grown to maturity. Wheat and lettuce were cut just above the soil surface. Heads and straw from the mature wheat samples were separated. Grain was separated from the head and husks. Sugar beet roots and foliage were separated. Plant parts containing soil were washed with water (lettuce stems, sugar beet roots). Sugar beet roots were peeled (3 cm peel). Crop samples were stored at -20 °C or lower. Storage period is not stated but is maximally 32 months (first sampling to report date).

Crops were analysed for total radioactivity by combustion LSC. Residues were < 0.01 mg/kg eq in all crops grown in the ^{14}C-phenyl labelled fluazifop-butyl (RS) treated soil at all plant back intervals (30, 120 and 327 days after treatment). Results for crops grown in the ^{14}C-pyridyl labelled fluazifop-butyl treated soil are shown in Table 43. Residues were only detected in wheat straw (0.10–0.080–0.031 mg/kg eq at 60–120–365 PBI), wheat grain (0.011 mg/kg eq at 60 PBI) and sugarbeet foliage (0.027–0.018 mg/kg at 60–120 PBI).

Table 42 Soil characteristics

Origin	18 Acres, Bracknell, Berkshire, UK
Soil classification	Sandy loam
% Sand	56%
% Silt	19%
% Clay	25%
% Organic Matter	-
pH	6.7
Cation exchange capacity (meq/100g)	-
% Moisture holding capacity (Keen's box method)	68%

Table 43 Residues in crops planted 60, 120, 365 days after soil treatment with ^{14}C-pyridyl labelled fluazifop-butyl.

Crop	PBI (pyridyl) (days)	DAT (pyridyl)	DAS (pyridyl)	Control (pyridyl) mg/kg eq	Total ^{14}C residues mg/kg eq
Wheat, straw	60	182	122	0.005	**0.103**
	120	263	143	0.002	**0.080**
	365	494	129	0.004	**0.031**
Wheat, grain	60	182	122	0.005	**0.011**
	120	263	143	0.002	0.005
	365	494	129	0.002	0.003
Wheat, husk	60	182	122	**0.011**	**0.091**
	120	263	143	0.002	**0.023**
	365	494	129	0.006	**0.017**
Lettuce	60	182	122	0.001	0.005
	120	228	108	< 0.001	0.005
	365	482	117	0.001	0.004
Sugar beet tops	60	204	144	0.002	**0.027**
	120	263	143	0.001	**0.018**
	365	572	207	0.001	0.009
Sugar beet roots without peel	60	204	144	0.001	0.006
	120	263	143	< 0.001	0.003

Crop	PBI (pyridyl) (days)	DAT (pyridyl)	DAS (pyridyl)	Control (pyridyl) mg/kg eq	Total ^{14}C residues mg/kg eq
	365	572	207	0.001	0.002
Sugar beet peels	60	204	144	0.001	0.007
	120	263	143	< 0.001	0.003
	365	572	207	0.001	0.003

PBI = plant back interval; DAT = days after treatment; DAS = days after sowing of the crop

Confined rotational study 2

^{14}C-Phenyl or ^{14}C-pyridyl labelled fluazifop-P-butyl (R-enantiomer) were applied to a sandy loam soil (in pots under glasshouse conditions) [Bramley *et al*, 2004, PP5/0235, report RJ1457B]. Soil characteristics are shown in Table 44. The actual application rate was 0.44–0.50 kg ai/ha for a 30 day rotation interval and 0.85–1.1 kg ai/ha for a 90 and 270 day rotation interval (EC formulation). Thouree different crops (wheat variety Minaret, carrot variety Early Nantes and Cos lettuce variety Lobjoits Green) were sown 30, 60, and 270 days after soil treatment. All crops were harvested at maturity and in addition, immature wheat samples were taken. Wheat and lettuce were cut 2 cm above the soil surface. Heads and straw from the mature wheat samples were separated. Grain was separated from the head and husks and mixed thoroughly. Carrot roots and foliage were separated. Plant parts containing soil were washed and dried (wheat forage, outer leaves of lettuce, carrot roots and tops). Crop samples were stored frozen and were analysed within 6 months. Soil samples were taken at application, crop planting and at each crop harvest (5–7 cm in depth), were extracted within 24 hours and were analysed within 1–6 months.

Soil cores sampled at application were extracted by acetonitrile and radioactivity in extracts was measured by LSC. Those sampled at planting and at harvest were extracted with acetonitrile, and the post-extraction soil debris was subsequently extracted with acetonitrile:1M hydrochloric acid (1:1, v/v at 60 °C). Extracts were assayed by LSC and the residual soil debris were combusted for radioactivity quantification by LSC. In soil 28–90% of the applied radioactivity was recovered. Soil extracts were analysed by TLC (3 solvent systems). Results are shown in Table 45 and Table 46.

Wheat forage, lettuce, carrot tops and carrot roots were macerated in acetonitrile and the radioactivity was measured in extracts and in combusted post extraction solids. Wheat grain and wheat straw were analysed by combustion and LSC, or extracted with acetonitrile and acetonitrile:water (1:1, v/v) before radioactivity quantification in extracts and post-extraction solids. Following extraction of the plant samples, polar radioactive residues and post extraction solids were subjected to hydrolytic treatments including 1 M HCl at 60°C for 1 hour, 6 M HCl for 6 or 24 hours, cellulose and pectinase hydrolysis (51–73 hours at pH 4.5 at 37 °C) followed by papain hydrolysis (69 hours at pH 7 at 37 °C). Extracts were then partitioned with ethyl acetate and analysed by LSC, TLC (2 solvent systems) and/or HPLC with UV detection at 254 nm. Results are shown in Table 47 and Table 48.

Metabolites in the soil and plant extracts were identified using co-chouromatography of reference markers for fluazifop-butyl (I), fluazifop acid (II), Pyr-Ph ether (IV) and CF3-pyridone (X) in combination with autoradiography (TLC) or radio-detection (HPLC). The N-hexose-sugar conjugate of CF3-pyridone (X) was identified by positive ion thermospray MS and proton and fluorine NMR using an extra labelling experiment where wheat plants were grown during 24 hour in a solution containing ^{14}C-labelled CF3-pyridone (X) and where HPLC was used for isolation and purification.

Analysis of the soil extracts showed that only 1.2%TRR remained as parent after 30 days. In soil treated with ^{14}C-phenyl labelled fluazifop-P-butyl, fluazifop acid (II) was the main free metabolite. The amount of fluazifop acid (II) decreased from 23% TRR (30 days) to < 8% TRR (at harvest, 270 day period). In soil treated with ^{14}C-pyridyl labelled fluazifop-P-butyl, CF3-pyridone (X) was the main free metabolite ranging from 30% TRR at the 30 day PBI to 11% TRR at the later plant

back intervals. Both treatments resulted in high levels of polar material (12–33% TRR) and post extraction solids (35–81% TRR) in the soil samples.

All control crops had significant radioactive residues from natural incorporation of $^{14}CO_2$, which is a breakdown product of fluazifop-butyl in soil.

Crops grown in soil treated with ^{14}C-phenyl labelled fluazifop-P-butyl had very low residues. Residues in lettuce leaves and carrot roots were below 0.01 mg/kg eq, residues in other crops were below 0.04 mg/kg eq except wheat straw at the 60 day plant back interval (PBI) where the residue was 0.1 mg/kg eq. Therefore only cereal residues were characterised further. In wheat straw of the 60 day PBI 60% TRR was organo- and/or acid soluble. Individual extractable components of wheat straw did not exceed 0.014 mg/kg eq, post extraction solids represented a residue of 0.03 mg/kg eq. In the wheat grain of the 60 day PBI 27%TRR was organo- or aqueous soluble (0.01 mg/kg eq); the remainder (<0.03 mg/kg eq) was not tested for acid hydrolysis.

Crops grown in soil treated with ^{14}C-pyridyl fluazifop-P-butyl had radioactive residues >0.01 mg/kg eq. Residue levels in crops grown after 60 and 270 days rotations were very similar as were the metabolic profiles from soil cores taken at 60 and 270 day sowing. Characterisation and identification was therefore carried out on all crops grown after a 60-day rotation period, as representative of the residues from each rotation interval. Crops were fractionated in organo- and/or acid soluble and post extraction solids. Fluazifop-butyl (I), fluazifop acid (II) and Pyr-Ph ether (IV) were not detected. CF3-pyridone (X) represented > 60% TRR in most crop commodities (except 31%TRR in carrot roots and 45% TRR in wheat grain) both as free and conjugated forms. Other organo- and acid extractable compounds were < 0.05 mg/kg eq, except in immature wheat (5.4% TRR; 0.11 mg/kg eq), wheat straw (5.1% TRR, 0.47 mg/kg eq) and carrot tops (5.0% TRR; 0.07 mg/kg eq).

The initial profile of the 30 and 60 day rotation commodities was taken 6 months after sample harvest. Subsamples of the crops were kept frozen and were re-analysed at 10–11 months after the initial analysis. Storage stability during this period was confirmed by comparing the chouromatographic profile to that of the original sample.

Remark 1 by present reviewer: The application rates mentioned in the summary of the study report (0.85–0.98 kg ai/ha) differed from figures in the tables in the study report (0.85–1.10 kg ai/ha). Recovered radioactivity in soil mentioned in the summary of the study report (< 3% TRR for fluazifop acid (II) in ^{14}C-phenyl label) differed from figures in the tables in the study report (< 8% TRR for fluazifop in ^{14}C-phenyl label). The figures in the tables of the study report were taken.

Values for residue characterisation in the summary table of the study report differed from values given in the tables in the study report. The figures in the summary table were taken.

Reviewer's conclusion:

Fluazifop acid (II) is stable in 1 M HCl for 3 hours at 60 ºC (see hydrolytic stability section above). Stability of fluazifop acid (II) in 6 M HCl for 6 or 24 hours, cellulose and pectinase hydrolysis (51–73 hours at pH 4.5 at 37 °C) followed by papain hydrolysis (69 hours at pH 7 at 37 °C) has not been investigated. Since the identity of the CF3-pyridone N-hexose conjugate could be confirmed. CF3-pyridone (X) is assumed to be derived from uptake from the soil and subsequent metabolism within the plant. Verification of stability during enzymatic hydrolysis is desirable.

Table 44 Soil characteristics

Origin	Bracknell, Berkshire, UK
Soil classification	Sandy loam
% Sand	57.7
% Silt	25.1
% Clay	17.2
% Organic Matter	3.7
pH	7.0
Cation exchange capacity (meq/100g)	12.0
% Moisture holding capacity (15 Bar)	59.2

Table 45 Recovered radioactivity (^{14}C-phenyl label) and characterisation of residues in soil core extracts

PBI (days)	DAT	Soil sampling time	%TAR	Characterisation (% TRR in soil core)						
				Acetonitrile and acetonitrile/acid extracts						PES
				parent	fluazifop acid (II)	Pyr Ph ether (IV)	CF3-pyridone (X)	polar	unkn	
30	30	Sowing	54.9	1.2	22.8	6.0	NR	29.2	1.4	37.9
	56	Wheat forage harvest	47.2	1.3	4.3	0.4	NR	31.9	ND	56.8
	102	Lettuce harvest	61.9	ND	2.0	0.9	NR	22.0	ND	70.5
	149	Carrot harvest	56.2	ND	1.6	1.0	NR	13.6	0.75	81.0
	121	Wheat harvest	27.9	ND	1.8	1.9	NR	16.0	2.0	78.4
60	60	Sowing	67.8	0.9	22.6	3.6	NR	17.8	ND	52.7
	78	Wheat forage harvest	51.1	ND	7.9	2.3	NR	33.3	5.3	61.0
	130	Lettuce harvest	64.3	ND	2.8	1.8	NR	15.9	1.0	76.0
	197	Carrot harvest	45.2	ND	1.2	0.3	NR	15.0	0.42	80.1
	175	Wheat harvest	49.5	ND	1.5	1.0	NR	13.5	0.63	69.1
270	270	Sowing	53.0	ND	3.3	ND	NR	19.3	2.9	63.1
	294	Wheat forage harvest	39.2	ND	4.0	1.6	NR	21.0	2.0	71.3
	323	Lettuce harvest	48.7	ND	3.7	1.2	NR	24.2	2.0	68.8
	380	Carrot harvest	59.3	ND	7.1	4.2	NR	19.8	6.6	76.5
	393	Wheat harvest	59.1	ND	2.2	1.2	NR	23.6	1.2	72.0

NR = radioactive label not suitable to detect metabolite X

ND = not detected

Polar = material which remained very close to the origin in normal phase TLC

Unkn = low levels of radioactivity (no discrete bands in TLC or background levels)

Table 46 Recovered radioactivity (^{14}C-pyridyl label) and characterisation of residues in soil core extracts

PBI (days)	DAT	Soil sampling time	% TAR	Characterisation (% TRR in soil core)						
				Acetonitrile and acetonitrile/acid extracts						PES
				parent	fluazifop acid (II)	Pyr Ph ether (IV)	CF3-pyridone (X)	Polar	Unkn	
30	30	Sowing	76.1	0.89	10.1	4.7	27.1	13.0	3.0	40.9
	48	Wheat forage harvest	44.5	ND	3.2	2.6	30.2	15.5	2.4	43.4
	99	Lettuce harvest	49.4	ND	2.0	1.6	18.4	21.4	0.2	56.4
	147	Carrot harvest	44.3	ND	1.4	1.0	15.6	17.6	4.4	58.8
	121	Wheat harvest	42.5	ND	1.9	1.5	14.6	18.6	3.4	65.9
60	60	Sowing	72.4	ND	16.8	3.4	26.1	14.3	1.0	34.6
	79	Wheat forage harvest	51.9	ND	4.2	2.3	27.8	12.4	4.4	43.2
	130	Lettuce harvest	58.6	ND	2.1	1.4	16.0	17.8	1.8	58.7
	197	Carrot harvest	48.2	ND	2.3	1.5	21.7	20.2	4.2	47.7
	175	Wheat harvest	54.4	ND	1.5	1.0	10.8	16.4	0.2	68.9
270	270	Sowing	90.2	ND	32.0	3.3	13.4	17.5	4.6	34.6
	294	Wheat forage harvest	72.8	ND	10.8	5.4	27.7	21.4	2.4	35.7
	323	Lettuce harvest	53.3	ND	2.4	2.0	25.6	16.0	2.7	51.2
	380	Carrot harvest	81.6	ND	3.0	1.8	16.7	13.7	1.6	63.5
	393	Wheat harvest	76.3	ND	2.0	1.1	21.0	26.5	1.1	48.5

ND = not detected

Polar = material which remained very close to the origin in normal phase TLC

Unkn = low levels of radioactivity (no discrete bands in TLC or background levels)

Table 47 Total ^{14}C-labelled residues in mature crops planted 30, 60, 270 days after soil treatment.

Crop	PBI (days)	DAT (phenyl; pyridyl)	Control (phenyl) mg/kg eq	^{14}C-phenyl label mg/kg eq	Control (pyridyl) mg/kg eq	^{14}C-pyridyl label mg/kg eq
Wheat, forage[1]	30	56; 48	0.004	0.008 (n=3)		0.9 (n=3)
	60	78; 79	0.003	0.016 (n=3)	0.003	1.52* (n=4)
	270	294; 294	0.003	0.016 (n=3)	0.004	1.35 (n=3)
Wheat, straw[1]	30	121; 121	0.01	0.02* (n=6)	0.01	3.0 (n=3)
	60	175; 175	0.003	0.10* (n=8)		5.13* (n=10)
	270	393; 393	0.01	0.02 (n=9)		6.68 (n=3)
Wheat, grain[3]	30	121; 121	0.01	0.02 (n=4)		0.07 (n=2)
	60	175; 175	0.02	0.04* (n=7)		0.17* (n=8)
	270	393; 393	0.008	0.02 (n=8)		0.09* (n=4)
Lettuce[2]	30	102; 99	0.001	0.004 (n=9)	< 0.001	0.25 (n=10)
	60	130; 130	0.001	0.006 (n=6)		0.46* (n=7)
	270	323; 323	0.002	0.004 (n=9)	0.002	0.34 (n=9)
Carrot tops	30	149; 147	0.004	0.008 (n=3)		0.39 (n=4)
	60	197; 197	0.005	0.017 (n=3)	0.006	1.02* (n=4)
	270	380; 380	0.001	0.006 (n=3)		0.94 (n=3)
Carrot root	30	149; 147	0.002	0.003 (n=3)		0.01 (n=4)
	60	197; 197	0.003	0.007 (n=3)	0.003	0.03* (n=4)
	270	380; 380	< 0.001	0.003 (n=3)		0.02 (n=3)

1 = wheat cut at 2 cm above the soil surface
2 = lettuce cut at the base of the plant
3 = grain separated from the head and husks
* = characterised further (table 15)

Table 48 Characterisation of radioactive residues (% total residue) in crops based on acid hydrolysis

Commodity (PBI)	^{14}C Label	TRR (mg/kg eq)	CF3-pyridone (X, free +conj) [e] (%TRR)	CF3-pyridone N-hexose conjugate [e] (%TRR)	Polar compounds (total) (%TRR)	Extracted unknown (total) (%TRR)	PES (%TRR)
Wheat, straw (30)	Phenyl	0.02				54.1 g	45.9 [c]
Wheat, straw (60)	Phenyl	0.07				60 [b]	37.1
Wheat, grain (60)	Phenyl	0.04				27.2 h	72.8 [d]
Wheat, forage (60)	Pyridyl	1.99	45.0	24.4	7.8	18.1 [a]	4.7
Wheat, straw (60)	Pyridyl	9.27	46.1	14.3	12.0	18.9 [a]	8.7
Wheat, grain (60)	Pyridyl	0.18	2.3	28.6	7.1	48.5 [a]	13.5
Wheat, grain (270)	Pyridyl	0.11	4.3	N.A.	2.5	62.5 [a]	30.7
Lettuce (60)	Pyridyl	0.40	52.0	12.1	13.0	19.3 [a]	3.6
Carrot, tops (60)	Pyridyl	1.41	50.1	11.6	15.8	10.4 [a]	12.1
Carrot root (60)	Pyridyl	0.05	33.5	11.2	3.3	41.3 c	10.7

[a] contains several compounds, individual maximum organosoluble 5.4% TRR (0.11 mg/kg eq) in wheat forage (60 day PBI), 5.1% TRR (0.47 mg/kg eq) in wheat straw (60 day PBI), 1.3% TRR (0.002 mg/kg eq) in wheat grain (60 day PBI), 1.6% TRR (0.002 mg/kg eq) in wheat grain (270 day PBI), 3.9% TRR (0.02 mg/kg eq) in lettuce leaves, 5.0% TRR (0.07 mg/kg eq) in carrot tops (60 day PBI), 13.3% TRR (0.007 mg/kg eq) in carrot roots (60 day PBI)

[b] Consists of 8.8% TRR acid soluble debris (<0.01 mg/kg eq, after acid hydrolysis), an aqueous fraction (19.4% TRR, 0.014 mg/kg eq, after hydrolysis) and an organosoluble fraction (22.3% TRR, 0.015 mg/kg eq, after hydrolysis) . The organosoluble fraction contained 3 discrete compounds with a maximum of 7.3% TRR (0.005 mg/kg eq).

[c] based on acetonitrile extractions 54.1% (0.011 mg/kg eq) could be extracted; the remainder is 100-54.1%.

[d] based on hexane/acetonitrile/water extractions, 27.2% (0.011 mg/kg eq) from 1.2% hexane + 1.5% acetonitrile + 24.5% acetonitrile/water extractable; it was assumed that the remainder of 72.8% = (100–27.2%) was not extracted.

[e] N-hexose sugar conjugate of CF3-pyridone (identified by NMR and MS), cannot be hydrolysed by 1 M HCl, 1 hour 60 °C) CF3-pyridone (X, free + conjugates) includes conjugates of CF3-pyridone (X) which are hydrolysed.

Field rotational crop studies

Field rotational study 1

A field rotational crop study was conducted at four different locations in the USA in 1980 [Ussary, 1981, PP9/0176, TMU0671/B]. Fallow plots were treated with a single application of an EC formulation of fluazifop-butyl (RS) at 1.1 kg ai/ha. The trials were started in April and May 1980. The plots were then tilled to a depth of 10 cm. Soil characteristics are shown in Table 49. Crops were planted at intervals of 0–15–30–60–90–120 days after soil treatment (see Table 49). Whole plants, forage, grains or roots, as appropriate for the crop, were taken at maturity. In addition forage samples were taken from immature wheat and soya. Samples were taken at random from the plots. Samples sizes were 0.9 kg for the above ground portions of vegetables and foliar portions of grains and 2.2 kg of root crops. Root crops were separated into tops and roots. Separate samples of grains were collected from each grain crop that reached maturity. Samples were stored at -18 ºC or lower for approximately 12 months.

Weather at Champaign, Goldsboro and Visalia did not affect crop growth, but weather at Vicksburg was unusually hot and dry which caused many of the plants to either not grow at all or die prematurely. No phytotoxicity was observed for soya beans, sugar beets, broccoli, turnips, cotton, or sweet potatoes at any plant back interval. There was varying amounts of injury to wheat, maize, sorghum or sweet corn planted up to 30 days after the fluazifop-butyl application; however there was no observable damage to any of these crops when planted 60 days after the treatment.

Crop samples were analysed for residues of total fluazifop using HPLC-UV method PPRAM 53/1 (=PPRAM 62) with GC-MS confirmation for some immature wheat and soya forage. Mean method recoveries for fortification levels of 0.1–0.5 mg/kg fluazifop were 70–107%, except for sweet potato vine (69%), wheat forage (66%), cotton gin trash (64%).

Residues of total fluazifop were not detected at any of the plant back intervals: < 0.02 mg/kg in sugar beet roots, turnip roots and turnip tops, < 0.05 mg/kg in all other crop commodities. CF3-pyridone (X) was not analysed.

Table 49 Soil characteristics and planting schemes for each of the four locations in the USA

Trial no	RU1-80-01	RU4-80-004	RU2-80-001	HU5-80-14
Location	Goldsboro, NC, USA	Champaign, IL, USA	Visalia, CA, USA	Vicksburg, MS, USA
Soil type	loamy sand	silty clay loam	sandy loam	silty loam
% sand	90.2	38.1	74.8	38.0
% silt	9.4	35.7	17.4	51.2
% clay	0.4	26.3	7.8	10.8
% organic matter	0.8	5.2	0.8	1.9
pH	5.6	6.0	8.4	5.7
CEC (meq/100g)	-	-	-	-
MWHC	-	-	-	-
Broccoli PBI (days)	-	0-30-90; mature	120; mature	-
Sweet potato PBI (days)	15-60; vines, roots	-	-	-
Wheat PBI (days)	-	0; mature whole plant; 30-90-120; immature plant	-	-
Cotton PBI (days)	0-15; lint 15; stalks 60-90; forage	-	0-30-90; gin trash; 0-15; seeds	-
Maize PBI (days) (field corn)	-	0-30-60; forage 0-60-90; grains	-	-
Sweet corn PBI (days)	0-30; ears 30-90-120; stalks	-	0-30-90-120; forage	30; forage
Sorghum PBI (days)	15-30; grains 15-30- 60-90-120; forage	30-60; grains 30-60-90; forage	0-30; grains 30-90-120; forage	20-30; grains 20-30; forage
Soya bean PBI (days)	0-90; green stalks 60; stalks and seed	0-30; grains 0-30; stalks 90: immature plant	0-15-30-60-90; whole plant	-
Sugar beet PBI (days)	-	0-30-90; tops	0-30-60; tops	-

Trial no	RU1-80-01	RU4-80-004	RU2-80-001	HU5-80-14
Location	Goldsboro, NC, USA	Champaign, IL, USA	Visalia, CA, USA	Vicksburg, MS, USA
		0-30-90; roots	0-30-60; roots	
Turnips PBI (days)	120; tops	-	120; tops; 120; roots	0-30; tops 0-30; roots

PBI = plant back interval after soil treatment

Field rotational study 2

A field rotational crop study was conducted at two different locations in the UK between December 1993 and August 1995 [Atreya *et al*, 1997, PP5/0590, RJ2202B]. Fluazifop-P-butyl (R-enantiomer) was applied at 0.38–0.48 kg ai/ha as EC formulation in a spray volume of 300 L/ha using a hydraulic spray boom. A non-ionic surfactant (Agral 90) at 0.1% v/v was used as adjuvant. Application details and soil characteristics are shown in Table 50 and Table 51. After treatment plots 1-5 were harrowed ensuring no inversion of the soil occurred and 1, 2 and 4 months after treatment spring wheat, lettuce and carrots were sown. At 6 months after treatment, plots 4 and 5 were ploughed and harrowed and winter wheat was sown. Soil samples (0.5 kg) were taken within thouree hours after application (0–10 cm depth) and one week prior to planting of the rotational crops (0–10 and 10–20 cm depth). Crop samples were taken at harvest; in addition immature samples of both winter and spring wheat were taken. Samples sizes were 1 kg wheat forage, 1 kg wheat grain, 0.5 kg wheat straw, 12 plants of lettuce and 24 plants of carrots. Samples were stored frozen at -18 ºC until analysis for up to 239 days (soil) and up to 202 days (crops), respectively

Soil samples were analysed for residues of fluazifop-butyl using modification A of method RAM 054/02 and for residues of fluazifop acid (II, free) and CF3-pyridone (X, free) using method RAM 195/01. Residue levels were corrected for soil moisture and expressed as mg/kg dry soil. The results are presented in Table 52. Mean method recoveries for fortification levels of 0.01–0.2 mg/kg fluazifop-butyl, 0.01–0.05 mg/kg fluazifop acid (II) and 0.01–0.05 mg/kg CF3-pyridone (X) were 89±17% (n=14), 87±24% (n=17), 89±17% (n=17), respectively. Residues were corrected for method recovery; uncorrected results are not reported. Residues of fluazifop-butyl (0.02–0.05 mg/kg dry weight) and fluazifop acid (II, 0.11–0.24 mg/kg dry weight) measured in soil following application declined within one month to less than 0.01 mg/kg. The residues of fluazifop acid (II) in soil (0.11–0.12 mg/kg dry weight) were lowest in plot 1 where the application was made onto oilseed rape. Residues of CF3-pyridone (X) (0.01–0.03 mg/kg dry weight) were highest 1 month after application and declined to lower than 0.01 mg/kg dry weight 4 months after application and were not detectable 6 months after application.

Crop samples were analysed for residues of total fluazifop using GC-MS method RR 91-014B. Total CF3-pyridone (X, free plus conjugated) was analysed using a modification of GC-MS method RR 90-384B. The results are presented in Table 53. Mean method recoveries for fortification levels of 0.01–0.1 mg/kg fluazifop acid (II) and 0.01–0.1 mg/kg CF3-pyridone (X) were 91±18% (n=30) and 83±13% (n=59), respectively. Residues were corrected for method recovery; uncorrected results were not reported. Residues of total fluazifop were not detected (<0.01 mg/kg) in any of the rotational crops at any of the plant back intervals. The only residue found was CF3-pyridone (X). Residues of CF3-pyridone (X) were < 0.01 mg/kg (LOQ) in lettuce heads, carrot roots, immature winter wheat, mature grain and straw at any of the rotational periods (1, 2, 4 or 6 months PBI). CF3-pyridone (X) was only found in immature spring wheat samples at 0.02 mg/kg (4 month PBI) and carrot tops at 0.01–0.04 mg/kg (1, 2 month PBI) or 0.03–0.13 mg/kg (4 month PBI).

Table 50 Application scheme for five different field plots (each at two locations in the UK).

Plot	Target	Application (kg ai/ha)	Application time	Preparing of seed bed (after application)	Sowing time	Harvest time
1	Oilseed rape	1 × 0.38	Dec 1993	Apr 1994; desiccation of oilseed rape; harrow (10 cm)	Apr 1994[a,b,c]	Jun[b,e], Jul[c], Aug[1] 1994
2	Bare soil	1 × 0.38	Apr 1994	Apr 1994; harrow (10 cm)	Jun 1994[a,b,c]	Aug[b], Sept[a,c] 1994

Plot	Target	Application (kg ai/ha)	Application time	Preparing of seed bed (after application)	Sowing time	Harvest time
3	Bare soil	1 × 0.38	Apr 1994	Apr 1994; harrow (10 cm)	May 1994[a,b,c]	Jul[b], Aug[c], Sept[a] 1994
4	Bare soil	Untreated	-	Apr 1994; harrow (10 cm); Oct 1994; plough and press (20 cm); harrow (15 cm)	Apr 1994[a,b,c]; Oct 1994[d]	Jun[b,e], Jul[c], Aug[a] 1994; Jun[f], Aug[d] 1995
5	Bare soil	1 × 0.48	Apr 1994	Apr 1994; harrow (10 cm); Oct 1994; plough and press (20 cm); harrow (15 cm)	Oct 1994[d]	Jun[f], Aug[e] 1995

[a] Spring wheat, var Tonic (S004) or Cadenza (S005),

[b] lettuce, var Saladin, [c] carrots, var Nairobi,

[d] winter wheat, var Hussar (S004) or Cadenza (S005),

[e] spring wheat forage,

[f] winter wheat forage

Table 51 Soil characteristics for each of the two locations in the UK

Trial number	GB01-94-S004	GB02-94-S005
Location	Maidenhead (UK)	Eriswell (UK)
Soil type	Sandy loam	Sandy loam
% sand (top 0-30 cm)	65	75
% silt (top 0-30 cm)	18	13
% clay (top 0-30 cm)	17	12
% organic matter (top 0-30 cm)	1.8	0.6
pH (top 0-30 cm)	7.5	8.8
CEC (meq/100g) (top 0-30 cm)	11.1	3.4
MWHC at 0.33 bar (top 0-30 cm)	16.4	11.1

Table 52 Residue levels of fluazifop--butyl, fluazifop acid (II) and CF3-pyridone (X) in soil samples

Plot no.	Interval between application and sowing in months	Soil depth (cm)	Residues (mg/kg dry weight) [a]					
			fluazifop-butyl (acetonitrile/water and hexane)		Fluazifop acid (II, incl conjugates) (acetonitrile/water and ethylacetate/acid)		CF3-pyridone (X, incl conjugates) (acetonitrile/water and ethylacetate/acid)	
			Loc 1	Loc 2	Loc 1	Loc 2	Loc 1	Loc 2
4	0 (control, plot 1)	0-10	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	4 (control, plot 1)	0-10	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		10-20	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	6 (control, plot 5)	0-10	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		10-20	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
5	0 (at application)	0-10	0.04	0.03	0.24	0.22	< 0.01	< 0.01
	6 (at sowing)	0-10	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		10-20	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
1	0 (at application)	0-10	0.02	0.02	0.12	0.11	< 0.01	< 0.01
	4 (at sowing)	0-10	< 0.01	< 0.01	< 0.01	< 0.01	0.01	< 0.01
		10-20	< 0.01	< 0.01	0.01	< 0.01	< 0.01	< 0.01
2	0 (at application)	0-10	0.01	0.05	0.18	0.19	< 0.01	< 0.01
	2 (at sowing)	0-10	< 0.01	< 0.01	< 0.01	< 0.01	0.02	0.02
		10-20	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01
3	0 (at application)	0-10	0.02	0.03	0.22	0.13	< 0.01	< 0.01
	1 (at sowing)	0-10	< 0.01	< 0.01	0.01	< 0.01	0.03	0.01
		10-20	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01

Loc 1 = location GB01-94-S004

Loc 2 = location GB01-94-S005

[a] Residues were corrected for moisture content and method recovery; uncorrected results are not reported

Table 53 Residue levels of total fluazifop and CF3-pyridone (X) in rotational crops at harvest.

Crop	Plot no	Interval between treatment and planting (months)	Interval between planting and harvest (months)	Total fluazifop [b] (acetonitrile/acid)		CF3-pyridone (X) [b] (acetonitrile/water and acid hydrolysis)	
				Loc 1 [a]	Loc 2 [a]	Loc 1 [a]	Loc 2 [a]
Carrot, root	1	4	3	< 0.01	< 0.01	< 0.01	< 0.01
	2	2	3	< 0.01	< 0.01	< 0.01	< 0.01
	3	1	3	< 0.01	< 0.01	< 0.01	< 0.01
	4	Control	3	< 0.01	< 0.01	< 0.01	< 0.01
Carrot, tops	1	4	3	< 0.01	< 0.01	**0.03**	**0.13**
	2	2	3	< 0.01	< 0.01	**0.04**	**0.01**
	3	1	3	< 0.01	< 0.01	**0.04**	**0.04**
	4	Control	3	< 0.01	< 0.01	< 0.01	< 0.01
Lettuce, heads	1	4	2	< 0.01	< 0.01	< 0.01	< 0.01
	2	2	2	< 0.01	< 0.01	< 0.01	< 0.01
	3	1	2	< 0.01	< 0.01	< 0.01	< 0.01
	4	Control	2	< 0.01	< 0.01	< 0.01	< 0.01
Immature spring wheat[1]	1	4	2	< 0.01	< 0.01	**0.02**	< 0.01
	4	Control	2	< 0.01	< 0.01	< 0.01	< 0.01
Spring wheat, grain	1	4	4	< 0.01	< 0.01	< 0.01	< 0.01
	4	Control	4	< 0.01	< 0.01	< 0.01	< 0.01
Spring wheat, straw	1	4	4	< 0.05	< 0.05	< 0.01	< 0.01
	2	2	3	< 0.05	< 0.05	< 0.01	< 0.01
	3	1	4	< 0.05	< 0.05	< 0.01	< 0.01
	4	Control	4	< 0.05	< 0.05	< 0.01	< 0.01
Immature winter wheat[1]	5	6	6	< 0.01	< 0.01	< 0.01	< 0.01
	4	Control	6	< 0.01	< 0.01	< 0.01	< 0.01
Winter wheat, grain	5	6	7	< 0.01	< 0.01	< 0.01	< 0.01
	4	Control	7	< 0.01	< 0.01	< 0.01	< 0.01
Winter wheat, straw	5	6	7	< 0.05	< 0.05	< 0.01	< 0.01
	4	Control	7	< 0.05	< 0.05	< 0.01	< 0.01

[a] = plants cut at ground level (loc 1= GB01-94-S004) or at 5 cm above ground level (loc 2= GB02-94-S005)

[b] = residues were corrected for method recoveries; uncorrected results are not reported.

Animal metabolism

The Meeting received information on the fate of fluazifop-P-butyl in ruminants (lactating cow and lactating goat) and poultry (laying hens). The metabolism in laboratory animals was summarized and evaluated by the WHO panel of the JMPR in 2016.

Lactating cow

Livestock study 1

Metabolism of fluazifop-butyl (RS) was studied in a lactating cow [Evans *et al*, 1981, PP9/0180, report RJ0207B]. A lactating cow (Friesian, 3 year; 500 kg) was dosed orally with a 1:1 mixture of ^{14}C- phenyl-labelled (5.20 mCi, 41.5 mg) and ^{14}C- pyridyl-labelled (5.19 mCi, 45.2 mg) fluazifop-butyl (RS). The cow was dosed twice daily during 7 days via a gelatine capsule containing 18.7 mg fluazifop-butyl. With a mean feed intake of 15 kg/day this corresponds to 2.5 ppm dry weight feed. Urine, faeces and milk were collected during the test period (every 12 hours) and 4 hours after the last treatment the cow was killed and samples of tissues (fat, muscle, kidney and liver) were collected. Samples were stored at -20 ºC. The storage period is not indicated but does not exceed 4 months (study conduct to final report date).

Radioactivity in urine, feces, tissues and AM and PM milk was measured by (combustion) LSC. Total radioactivity recovered was 82% of the applied dose: 80% TAR was excreted in the urine and only 1.7% TAR in the faeces and 1.1% TAR in the milk. Average residue levels in milk on day 2 to 7 were 0.034 mg/kg eq (range 0.020–0.048 mg/kg eq), expressed as fluazifop-butyl; a plateau was already reached on day 2. Residue levels in the pm milk were higher than those in the am milk. Residues were also found in liver (0.024 mg/kg eq), kidney (0.039 mg/kg eq), muscle (0.001 mg/kg eq), omental fat (0.005 mg/kg eq), subcutaneous fat (0.002 mg/kg eq) and heart fat (0.005 mg/kg eq). The radioactive residues in milk (day 6, PM sample), liver, kidney, muscle and omental fat were characterised further.

The day 6 PM milk samples were extracted sequentially with acetone (10.1% TRR) and hexane (88.8% TRR) after which 1.1% TRR debris remained. The hexane fraction represents the butterfat fraction. The acetone extract (10.1% TRR) was diluted with water and residues were partitioned between hexane (5.4% TRR) and aqueous phase (4.7% TRR). The hexane phase (5.4% TRR) was combined with the primary hexane extract (88.8% TRR), rotary evaporated to dryness and redissolved in hexane. The combined hexane extract (94.2% TRR) was cleaned-up by Florisil column chouromatography and the 30% diethylether-70% hexane and 40% diethyl ether-60% hexane eluates were collected and combined (Eluate C: 84.2% TRR). The other eluates were not characterised further. The combined eluate C was cleaned-up further by preparative TLC and elution with diethylether in 3 fractions (x: 0.2% TRR, y: 77.0% TRR and z: 7.0% TRR). An aliquot of eluate y (77.0% TRR) was hydrolysed with aqueous 0.1 M NaOH for 1 hour under reflux conditions and partitioned between hexane (9.1% TRR), diethyl ether (5.7% TRR) and aqueous phase (62.2% TRR). The aqueous phase (62.2% TRR) was adjusted to pH 2 and partitioned between diethyl ether (fraction D; 61.8% TRR) and aqueous phase (0.4% TRR). The hexane (9.1% TRR) and diethyl ether phase (5.7% TRR) were combined with eluate z (7.0% TRR) from preparative TLC analysis. The combined phases (21.8% TRR) were rotary evaporated to dryness, hydrolysed with 0.1 M methanolic NaOH for 1 hour under reflux conditions and partitioned between diethyl ether (0.2% TRR) and aqueous phase. The aqueous phase was adjusted to pH 2 and partitioned between diethyl ether (fraction E: 21.5% TRR) and aqueous phase (0% TRR).

Liver samples were sequentially extracted with acetonitrile/water (1:1, v/v, 80.6% TRR), water (4.2% TRR) and diethyl ether (5.1% TRR), so that 10.1% TRR solids remained. The acetonitrile/water extract (80.6% TRR) was partitioned with hexane (0% TRR). The aqueous phase (80.6% TRR) was partitioned successively with diethylether, diethyl ether at pH 4 and diethylether at pH2. The thouree diethyl ether fractions were combined (fraction F; 71.1% TRR) for TLC analysis. The aqueous phase (9.5% TRR) was hydrolysed with 1 M HCl for 2 hours at 60 ºC and partitioned between diethyl ether (fraction G; 2.5% TRR) and aqueous phase (7.0% TRR).

Kidney samples were successively extracted by acetonitrile/water (1:1, v/v, 94.1% TRR) and diethyl ether (0.2% TRR) so that 5.7% TRR remained as solids. The acetonitrile/water extracts were partitioned with hexane (2.0% TRR). The remaining aqueous phase was sequentially partitioned with diethyl ether and diethyl ether at pH 4 and combined for TLC analysis (fraction H, 86.6% TRR). The remaining aqueous phase (5.5% TRR) was not analysed further.

Muscle samples were sequentially extracted with acetonitrile/water (73.3% TRR) and diethyl ether (16.2% TRR) so that 10.5% TRR solids remained. The acetonitrile/water extracts were diluted with 0.1 M sodium bicarbonate and partitioned with hexane (1.4% TRR). The remaining aqueous phase was partitioned with diethyl ether (5.5% TRR) and diethyl ether at pH 4 (fraction J: 54.2% TRR). The remaining aqueous phase was not analysed further (12.2% TRR).

Omental fat samples were heated at 50 ºC and subsequently extracted by hexane (61.8% TRR) and acetonitrile/water (35.9% TRR), so that 2.3% TRR remained in the solids. The hexane extract was partitioned between hexane (32.9% TRR) and acetonitrile (28.9% TRR). The acetonitrile phase was rotary evaporated to dryness and redissolved in diethyl ether (fraction A: 28.9% TRR). The primary acetonitrile/water extract was partitioned with hexane (1.1% TRR). The remaining acetonitrile/water phase was rotary evaporated to remove the acetonitrile and sequentially partitioned with diethyl ether (0.9% TRR) and diethyl ether at pH 2 (fraction B: 25.5% TRR). The remaining

water phase (8.4% TRR) was not analysed further. The diethyl ether phases (fraction A and B) were combined (54.4% TRR) and partitioned between diethyl ether (fraction C: 17.4% TRR) and aqueous 0.1 M NaHCO3 (fraction D: 37.0% TRR). The diethyl ether phase (fraction C) was partitioned between diethyl ether (3.8% TRR) and 0.1 M NaOH (13.6% TRR). The aqueous NaOH phase was adjusted to pH 2 and partitioned with diethyl ether (11.3% TRR) and water (2.3% TRR). The aqueous 0.1 M NaHCO3 pahse (fraction D) was adjusted to pH 2 and partitioned diethyl ether (37.0% TRR) and water (0% TRR).

Radioactivity in selected organic extracts was analysed by 1D- and 2D-TLC in parallel to reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X) and compounds 9 and 16. Results are shown in Table 54.

Parent was not detected. In milk most of the radioactive residue could be extracted by hexane (94.2% TRR) and represents the butterfat fraction. Most of this residue (70.9% TRR) co-chouromatographed with one of the isomeric fluazifop dipalmityl triglyceride (compound 16), which could be converted to fluazifop acid (II) on base hydrolysis (68% TRR). Fluazifop acid (II, free) was the only metabolite identified in muscle and omental fat at 37% and 32% TRR, respectively. Liver and kidney contained fluazifop acid (II, 60% and 61% TRR, respectively for liver and kidney) as well as Pyr-Ph ether (IV, free, 10% and 12% TRR, respectively for liver and kidney). The identity of fluazifop acid (II) was confirmed after methylation with diazomethane and MS analysis.

Reviewer's conclusion: Fluazifop acid (II) is stable in 0.1 M NaOH for 1 hours reflux and 1 M HCl for 2 hours at 60 ºC (see hydrolytic stability section above). Despyridinyl acid (III) and CF3-pyridone (X) were not detected. The presence of fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified. Hexane fractions that were not analysed may contain fluazifop-butyl (maximum 1.4–34% TRR). Fractions that were not subjected to hydrolysis (hexane, diethyl ether, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 16.0–68.2% TRR.

Table 54 Characterisation of ^{14}C residues in cow tissues and milk treated with fluazifop-butyl

Administered dose 2.5 ppm ^{14}C-phenyl + ^{14}C-pyridyl Fluazifop-butyl (RS)	milk, day 6 PM	liver	kidney	muscle	omental fat
TRR (mg/kg eq)	0.048	0.024	0.039	0.001	0.005
fluazifop-butyl	ND	ND	ND	ND	ND
Total fluazifop acid (II, free + conj) -- fluazifop acid (free) - fluazifop organo soluble conjugates -- fluazifop water soluble conjugates	67.7 - (67.7) -	61.7 (60.4) - (1.3)	61.0 (61.0) - -	36.9 (36.9) - -	31.8 (31.8) - -
Pyr-Ph ether (IV, free + conj) -- Pyr-Ph ether (free) -- Pyr-Ph ether water soluble conj	-	10.3 (9.9) (0.4)	11.8 (11.8) -	-	-
Despyridinyl acid (III)	ND	ND	ND	ND	ND
CF3-pyridone (X)	ND	ND	ND	ND	ND
Fluazifop alcohol (XXXIV)	no std	no std	no std	no std	no std
Hydroxyfluazifop acid (XL)	no std	no std	no std	no std	no std
Organo soluble unknowns hexane fraction – not hydrolysed diethyl ether soluble – not hydrolysed diethyl ether soluble – after hydrolysis	26.0 -10.2 - -15.8	5.9 - -5.1 -0.8	15.1 - 2.0 -13.1	40.4 -1.4 -39.0	55.2 - 34.0 -21.2-
Water soluble unknowns aqueous phases – not hydrolysed aqueous phases – after hydrolysis	5.1. -4.7 -0.4	11.2 - 4.2 - 7.0	5.5 -5.5 -	12.2 -12.2 -	10.7 -10.7
Solids: not hydrolysed	1.1	10.1	5.7	10.5	2.3
Total	99.9	99.2	99.1	100.0	100.0
Total identified	67.7	72.0	72.8	36.9	31.8
Total not hydrolysed	16.0	19.4	26.3	63.1	68.2

Lactating goats

Livestock study 2

Metabolism of fluazifop-P-butyl (R-enantiomer) was studied in lactating goats [Hand and Robertson, 1999, PP5/0593, report RJ2799B]. Phenyl- and Pyridyl-labeled fluazifop-P-butyl (R-enantiomer) were administered orally in gelatin capsules at a actual dose rate of 9.63 and 9.69 ppm dry feed, respectively, divided in two daily doses for 7 consecutive days to lactating goats (one per label, British Saanen, body weight 50 and 60 kg, 2 years old, daily milk production 1.3–1.8 L. The target dose rate of 10 ppm is equivalent to 13.93 mg ai/day (0.28 mg ai/kg bw/day) of phenyl-labeled fluazifop-P-butyl or 14.01 mg ai/day (0.23 mg ai/kg bw/day) of pyridyl-labeled fluazifop-P-butyl. Urine and feces samples and cage washes were collected predose, and thereafter at 24 hour intervals for the duration of the study (168 hours). The goats were milked twice daily (immediately before and 8 hours after each dose administration). At termination, 16 hours after last administration, tissue samples (including liver, kidneys, bile, fat (omental, perirenal and subcutaneous), gastro-intestinal tract contents, and forequarter and hindquarter skeletal muscle) were collected and stored at -10 °C until analysis (6 months).

Radioactivity in urine, feces, cage wash, gastro-intestinal tract, tissues and milk was measured by (combustion) LSC. The total recoveries of radioactivity from each of the treated goats were 87% and 99% TAR for the phenyl- and pyridyl label, respectively. The major route of excretion was the urine (70–82% TAR) and the remainder of the radioactivity was accounted for in faeces (10–11% TAR). Only minor levels of radioactivity were found in milk (0.83–0.86% TAR) and tissues (< 0.2% TAR).

Radioactivity levels in milk and tissues are summarized in Table 55. Radioactivity levels in milk peaked at 96-104 hours after the beginning of dosing at 0.15–0.16 mg/kg eq for both labels.

Omental and perirenal fat samples were extracted by dichloromethane (92–93% TRR). Fat and muscle samples were not analysed further because of low levels of radioactivity (< 0.01 mg/kg eq).

Liver samples were successively extracted by acetonitrile, acetonitrile/water, and acetone (total 62–66% TRR). These fractions were combined and evaporated until the aqueous phase remained. The aqueous remainder was partitioned to successively diethylether and diethylether at pH2. Both ether fractions were combined (55–69% TRR) for TLC analysis. The residual solids from liver (32–37% TRR) were extracted with 1N HCl (0.5–0.7%TRR) and then with 1N NaOH (8.7–11% TRR). A total of 21–25%TRR (0.010–0.012 mg/kg eq) remained as solids.

Kidney samples were successively extracted by acetonitrile, acetonitrile/water, and acetone (total 54–55% TRR). The acetonitrile fractions were combined (53–54% TRR) and analysed by TLC. The residual solids from kidney (45–46% TRR) were extracted with 1N HCl (0.6% TRR), then 0.1N NaOH (19–21% TRR), then acetone (3.8–12% TRR) and finally 2% sodium dodecyl sulfate (2.8–17% TRR). A total of 2.9–7.7% TRR (0.013–0.044 mg/kg eq) remained as solids. The NaOH and acetone extracts of kidney solids were combined, evaporated to the aqueous remainder and then partitioned with diethylether at pH9 and pH2. The aqueous phase was evaporated, saponified with 10% methanolic NaOH (2.5M) and partitioned with diethyl ether. The diethyl ether phase was combined with the other diethyl ethers extracts and analysed by TLC. The aqueous phase was centrifuged and the precipitate was refluxed with 6 M NaOH for 1 hour, acidified and partitioned with diethyl ether. The SDS extracts of the pyridyl-labelled kidney solids were further partitioned to diethyl ether at pH8 and pH2. The aqueous phase was centrifuged and the precipitates were refluxed with 6 M NaOH, acidified partitioned with diethyl ether.

The 96 hour milk samples were separated in skimmed milk (containing 30% and 8.1% TRR for the phenyl and pyridyl label) and butterfat (containing 70% and 92% TRR for the phenyl and pyridyl label). The skimmed milk fractions were partitioned with dichloromethane at pH2 and pH 10 (total 20.3%; 3.4% TRR, respectively, for phenyl and pyridyl label). The combined dichloromethane fractions form the phenyl label were evaporated, saponified with 10% methanolic NaOH (2.5 M

NaOH) and partitioned with diethyl ether. The butterfat milk fraction was extracted by acetonitrile (4.0%; 3.3% TRR for phenyl and pyridyl label) and dichloromethane (66%; 84% TRR). A total of 6.3% and 6.6% TRR (0.009; 0.010 mg/kg eq) remained as solids. The dichloromethane fraction was saponified with 10% methanolic NaOH (2.5 M) and finally partitioned with diethylether.

Radioactivity in extracts and diethylether phases was analysed by TLC in parallel to reference standards for fluazifop-butyl (I), fluazifop acid (II) and Pyr-Ph ether (IV). Where possible, the identity was confirmed by HPLC.

The nature of radioactive residues in milk and tissues is summarized in Table 56. Parent was not detected. Fluazifop acid (II) represented the main component in liver (22–25% TRR), kidney (50–51% TRR) and milk (67–69% TRR). Lipid conjugation was demonstrated for kidney and milk. This was the most prevalent in milk, in which the entire identified residue was due to lipid conjugates. Pyr-Ph ether (IV) was found at low levels in kidney (1.9% TRR or 0.01 mg/kg eq) and milk (< 2% TRR). A large number of unknown components were present in the liver and kidney samples, the largest of which represented 13% TRR (0.007 mg/kg eq) in liver 2.5% TRR (0.012 mg/kg eq) in kidney.

Tissues were extracted, fractionated and chouromatographically profiled within 6 months of necropsy. Further analysis was required on milk from the phenyl-label and liver and kidney from the pyridyl label. Samples were re-extracted and compared to the initial extraction profiles. No significant differences were observed for liver, kidney and milk.

Reviewer's conclusion: The study is considered not acceptable for milk and kidney because the hydrolysis conditions were too harsh. Fluazifop acid (II) degrades when refluxed with 6 M NaOH for 1 hour (see hydrolytic stability section above) as used to extract the kidney solids. Kidney and milk extracts were saponified with 2.5 M NaOH and since temperature and duration are not stated it is not clear whether this has an impact on fluazifop acid (II) levels. Compared to the cow study, fluazifop acid (II) residues are relatively lower in goat kidney than in cow kidney, suggesting the hydrolysis conditions for kidney are too harsh. Radiovalidation of SOP RAM 331/01 used 0.2 M NaOH in methanol (1 hour, 60 ºC) and was able to release more fluazifop acid (III) residues from milk, also suggesting that the hydrolysis conditions for milk and kidney were too harsh. Liver samples were not hydrolysed at all, but radiovalidation of SOP RAM 331/01 (with hydrolysis) was able to release the same amount of fluazifop acid (II). Therefore fluazifop acid (II) residues in liver are considered reliable. The presence of despyridinyl acid (III), CF3-pyridone (X), fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified. Butterfat fractions that were not analysed may contain som fluazifop-butyl (maximum 3.3–4.0% TRR). Fractions that were not subjected to hydrolysis (organic, water, solids) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 10.9–47.6% TRR.

Table 55 Levels of radioactivity by direct quantification in tissues and milk from goats treated with ^{14}C-fluazifop-butyl

Matrix	TRR mg/kg eq	
	Phenyl-label (R-enantiomer) 9.63 ppm	Pyridyl-label (R-enantiomer) 9.69 ppm
Milk, 8 hour – 168 hours	0.009-0.151 peak 94-104 hour: 0.151	0.011–0.161 peak 94-104 hours: 0.161
Omental fat	0.008	0.011
Perirenal fat	0.015	0.005
Subcutaneous fat	0.006	0.008
Kidneys	0.62	0.46
Liver	0.060	0.045
Forequarter muscle	0.004	0.003
Hindquarter muscle	0.004	0.002

Table 56 Nature of Residues in Liver, Kidney and milk from goats treated with ^{14}C-fluazifop-butyl

	Liver		Kidney		Milk 96 hours	
	^{14}C-phenyl R-enant 9.63 ppm	^{14}C-pyridyl R-enant 9.69 ppm	^{14}C-phenyl R-enant 9.63 ppm	^{14}C-pyridyl R-enant 9.69 ppm	^{14}C-phenyl R-enant 9.63 ppm	^{14}C-pyridyl R-enant 9.69 ppm
TRR by direct quantification	0.060	0.045	0.62	0.46	0.15	0.16
	% TRR	% TRR	% TRR	% TRR	% TRR	% TRR
Parent	ND	ND	ND	ND	ND	ND
Total fluazifop acid (II, free+conj) -- free -- conjugates	24.7 - 24.7 ND	21.5 - 21.5 ND	51.4 - 38.5 - 12.9	50.0 - 39.5 - 10.5	68.7 - ND - 68.7	67.1 - ND - 67.1
Pyr-Ph ether (IV, free + conj) -- Pyr-Ph ether (free) -- Pyr-Ph ether conjugated	ND	ND	1.6 - 1.3 - 0.3	1.9 - 1.5 - 0.4	1.7 - ND - 1.7	1.3 - ND - 1.3
Despyridinyl acid (III)	no std	no std	no std	no std	no std	no std
CF3-pyridone (X)	no std	no std	no std	no std	no std	no std
Fluazifop alcohol (XXXIV)	no std	no std	no std	no std	no std	no std
Hydroxyfluazifop acid (XL)	no std	no std	no std	no std	no std	no std
Organosoluble unknowns: - not hydrolysed (chouromatographed) - not hydrolysed (baseline and diffuse) - not hydrolysed (butterfat fraction) - not hydrolysed (not chouromatographed) - hydrolysed (chouromatographed) - hydrolysed (baseline and diffuse) - released by base from solids - released by SDS from solids	43.7 - 34.8 [a] - 8.9 - - - - - -	34.0 - 25.0 [a] - 8.3 - - 0.7 - - - -	21.3 - 5.3 [b] - 7.2 - - 0.9 - 0.2 - 0.9 -6.8 -	22.1 - 4.0 [b] - 6.0 - - 0.9 - 0.3 - 0.9 - 3.8 - 6.2 -	13.9 - - - 4.0 - - 7.1 [c] - 2.8	13.3 - - - 3.3 - 3.4 - 5.4 [c] - 1.2
Water soluble unknowns - not hydrolysed - released by acid from solids - released by base from solids - released by SDS from solids - left after saponification	15.8 - 3.9 - - 0.4 - 11.5	13.2 - 3.8 - 0.7 - 8.7	9.6 - - 0.6 - 6.2 - 2.8	16.6 - - 0.5 - 8.4 - 7.7	12.0 - 9.1 - - - - 2.9	8.8 - 4.3 - - - - 4.5
PES: - not hydrolysed - after hydrolysis	22.5 - -22.5	26.4 - -26.4	7.7 - -7.7	2.9 - - 2.9	6.3 -6.3 -	6.6 -6.6 -
Total	106.7	95.1	91.6	93.4	102.6	97.1
Total identified	24.7	21.5	53.0	51.9	70.4	68.4
Total not hydrolysed	47.6	34.0	13.4	10.9	19.4	17.6

TRR = Total Radioactive Residue (mg/kg fluazifop-butyl equivalents).

[a] Chouromatographed fractions consisting of at least 7 compounds, from which the largest unknown component represented 13.1% TRR (0.007 mg/kg eq) in the phenyl label, and of at least 4 compounds with none greater than 11.1% TRR (0.004 mg/kg eq) in the pyridyl label.

[b] Chouromatographed fractions consisting of at least 4 compounds, none greater than 2.2% TRR (0.013 mg/kg eq) in the ^{14}C-phenyl label and at least 2 compounds none greater than 2.5% TRR (0.012 mg/kg eq) for the ^{14}C-pyridyl label

[c] Chouromatographed fractions consisting of at least 5 compounds, none greater than 3.6% TRR (0.005 mg/kg eq) in the ^{14}C-phenyl label and at least 3 compounds none greater than 4.1% TRR (0.006 mg/kg eq) for the ^{14}C-pyridyl label

Laying hens

Livestock study 3

Metabolism of fluazifop-butyl (RS) was studied in laying hens [Day *et al*, 1981, PP9/0181, report RJ0212B]. Two laying hens (G-link hybrids, 9–18 months old, 1.9–2.3 kg) were dosed orally with either ^{14}C-phenyl-labelled or ^{14}C-pyridyl-labelled fluazifop-butyl (RS). The hens were dosed once daily during 14 days via gelatine capsules containing 0.47 and 0.28 mg phenyl- or pyridyl-labelled fluazifop-butyl (RS), respectively. With a mean feed intake of 0.150 kg/day this corresponds with 3.1 and 2.6 ppm dry feed. Excreta and eggs were collected daily during the test period. Four hours after the last treatment, the hens were killed and tissues (fat, muscle, liver and kidney) were collected. Eggs

were separated into egg yolk and egg white. Samples were stored at -20 ºC. The storage period does not exceed 2 months (study conduct to final report).

All samples were analysed for total radioactivity by combustion LSC and residues were expressed as fluazifop-butyl equivalents. Of the total administered radioactivity 97% and 98% was found in excreta after treatment with phenyl- and pyridyl-labelled fluazifop-butyl, respectively.

Total radioactive residues are shown in Table 57. The residues in egg yolk ranged between < 0.001–0.021 mg/kg eq (day 1-13) and reached plateau concentrations of 0.02 mg/kg eq in both groups after 6–7 days of dosing. The residues in egg albumen ranged between 0.001–0.008 mg/kg eq (day 1–13) and reached plateau concentrations of 0.002–0.003 mg/kg eq in both groups after 3 days of dosing. Plateau residues in egg yolk were higher than in the egg white. Residues found in tissues were: liver (0.027 and 0.077 mg/kg eq), kidney (0.056 and 0.44 mg/kg eq), leg muscle (0.005 and 0.011 mg/kg eq), breast muscle (0.004 and 0.008 mg/kg eq), subcutaneous fat (0.040 and 0.029 mg/kg eq) and peritoneal fat (0.045 and 0.039 mg/kg eq).

Egg yolks (phenyl label) were sequentially extracted with acetone/water (66.4% TRR) and hexane (26.0% TRR), so that 7.6% TRR solids remained. The acetone/water extract was partitioned with hexane (57.7% TRR). The acetone from the acetone/water phase was removed by evaporation. The remaining water phase was acidified to pH 1 and partitioned between diethyl ether (fraction A1: 7.3% TRR) and water (1.4% TRR). The hexane phase (57.7% TRR) was partitioned with acetonitrile and the acetonitrile phase was partitioned between acetonitrile (fraction C1; 14.9% TRR) and hexane (fraction B1; 37.2% TRR). The primary hexane extract was partitioned with acetonitrile and the acetonitrile phase was partitioned between acetonitrile (fraction C2: 8.0% TRR) and hexane (fraction B2; 14.0% TRR). Hexane fractions B1 and B2 were combined (51.2% TRR), evaporated to dryness, redissolved in hexane and fractionated on a Florisil column. The 50% diethyl ether in hexane eluate (eluate D: 44.5% TRR) was rotary evaporated to dryness and hydrolysed in 0.5 M NaOH in methanol for 3 hours under reflux conditions. Residues were sequentially partitioned with diethyl ether (2.4% TRR) and diethyl ether at pH 1 (fraction A2: 43.2% TRR), so that 1.4% TRR remained in the water phase. Acetonitrile fractions C1 and C2 were combined (22.9% TRR), mixed with water and partitioned between hexane (fraction B2: 18.1% TRR) and aqueous acetonitrile (5.6% TRR). The aqueous acetonitrile phase was rotary evaporated to remove the acetonitrile, adjusted to 1% NaHCO3 and sequentially partitioned with diethyl ether and diethylether at pH 1 (fraction A3: 4.4% TRR), until 0.6% TRR remained in the water phase. Diethyl ether fractions A1, A2, A3 hexane fraction B2 and eluate D were analysed by TLC.

Egg whites (phenyl label) were mixed with 1% aqueous NaHCO3 and sequentially partitioned with diethyl ether (1.9% TRR) and diethyl ether at pH 1 (fraction D1: 72.0% TRR). The water phase (2.3% TRR) was not analysed further. The remaining solid was extracted again with diethyl ether (fraction D2: 13.1% TRR), so that 10.7% TRR remained as solids. Diethyl ether fraction D1 and D2 were combined (85.1% TRR) and analysed by TLC.

Whole eggs (pyridyl label) were sequentially extracted with 2:1 acetone/water (22.3% TRR) and hexane (63.0% TRR), so that 14.7% TRR solids remained. The acetone/water extract was partitioned with hexane (2.1% TRR). The acetone from the acetone/water phase was removed by evaporation. The remaining water phase was acidified to pH 1 and partitioned between diethyl ether (fraction J1: 17.7% TRR) and water (3.1% TRR). The primary hexane extract (63.0% TRR) was evaporated, redissolved in hexane and fractionated on a Florisil column. The 50% diethyl ether in hexane eluate (eluate D: 46.4% TRR) was rotary evaporated to dryness and hydrolysed in 0.5 M NaOH in methanol for 1 hour under reflux conditions. Residues were sequentially partitioned with diethyl ether (1.4% TRR) and diethyl ether at pH 1 (fraction J2: 41.8% TRR), so that 3.2% TRR remained in the water phase. Diethyl ether fractions J1, J2 and eluate D were analysed by TLC.

Combined leg and breast muscle (phenyl label, pyridyl label) were sequentially extracted with acetone/water (72.7% and 85.7% TRR) and hexane (9.3% and 4.9% TRR), so that 18.0% and 9.4% TRR solids remained. The acetone/water extract was partitioned with hexane (0.9% and 1.9% TRR). The acetone was removed from the remaining acetone/water phase by evaporation. The water phase was sequentially partitioned with diethyl ether at pH 10 (0.9% and 0% TRR) and diethyl ether at pH 1

(fraction E1: 48.1% and 74.7% TRR). The remaining water phase (2.2% and 10.5% TRR) was not investigated further. The pellet formed during this extractions (phenyl label only) was mixed with water and partitioned between diethyl ether (fraction E2: 7.6% TRR) and water (13.4% TRR). Diethyl ether fractions E1 and E2 were combined (51.3% TRR and 74.7% TRR) and analysed by TLC.

Combined subcutaneous and peritoneal fat (phenyl and pyridyl label) was refluxed for 0.5–3 hours with hexane and partitioned between hexane (92.3% TRR and 86.5% TRR) and 1% $NaHCO_3$ solution (1.2% and 3.8% TRR), so that 7.7% and 9.7% TRR remained as solids. The hexane phase (92.3% and 86.5% TRR) was rotary evaporated until the oil remained, redissolved in hexane and fractionated on a Florisil column. The 30–50% ether in hexane eluate (eluate H: 89.5% TRR and 74.5% TRR) was rotary evaporated and refluxed in 0.1 M aqueous NaOH for 1–3 hours. The hydrolysate was sequentially partitioned with diethyl ether (2.9% and 1.9% TRR) and diethyl ether at pH 1 (fraction G: 74.7% and 72.6% TRR), so that 0.9% and 0% TRR remained in the water phase. Florisil eluate H (pyridyl label only) and diethyl ether fraction G was analysed by TLC.

Liver (phenyl and pyridyl label) was sequentially extracted with acetone/water (82.8% and 83.8% TRR) and hexane (12.1% and 9.2% TRR), so that 5.1% and 7.0% TRR solids remained. The hexane extract (12.1% and 9.2% TRR) was partitioned between acetonitrile (2.7% and 2.0% TRR) and hexane (8.8% and 7.3% TRR). The acetone/water extract (82.8% and 83.8% TRR) was partitioned between hexane (0.6% and 1.0% TRR) and aqueous acetone (78.4% and 75.9% TRR). Acetone was removed from the aqueous acetone phase by evaporation and this phase was sequentially partitioned with diethyl ether at pH 10 (0.6% and 0.6% TRR) and diethyl ether at pH 1 (fraction F1: 42.2% and 46.6% TRR). The aqueous phase from the phenyl label (1.2% TRR) was not investigated further. The aqueous phase from the pyridyl label (18.5% TRR) was acidified and partitioned between diethyl ether (fraction F3: 15.1% TRR) and water (3.5% TRR). The pellet resulting from these partitions was mixed with water, adjusted to pH 3 and partitioned with diethyl ether (fraction F2: 32.1% and 9.0% TRR). The aqueous phase (1.2% and 1.1% TRR) was not investigated further. Diethyl ether fractions F1, F2 and F3 were analysed by TLC.

Kidney (phenyl and pyridyl label) was sequentially extracted with acetone/water (90.1% and 88.4% TRR) and hexane (3.4% and 0% TRR), so that 6.5% and 11.6% TRR solids remained. The acetone/water extract was partitioned between hexane (4.0% and 0.9% TRR) and aqueous acetone. The aqueous acetone phase was rotary evaporated to remove the acetone and sequentially partitioned with diethyl ether at pH 10 (0% and 0% TRR) and pH 1 (fraction H: 78.9% and 63.7% TRR), so that 5.2% and 22.3% TRR remained in the water phase. The water phase from the pyridyl label was hydrolysed with 0.5 M HCl for 1 hour under reflux conditions. The hydrolysate was partitioned with diethyl ether (3.3% TRR). The remaining water phase was neutralised, evaporated to dryness and hydrolysed with 0.5 M NaOH in methanol for 1 hour under reflux conditions. The hydrolysate was adjusted to pH 1 and partitioned between diethyl ether (1.5% TRR) and water (13.8% TRR). Diethyl ether fraction H was analysed by TLC.

Selected organic fractions were subjected to 1D and 2D-TLC. Identification of residues was carried out by co-chouromatography against reference markers for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X) and compound 9 and 16. Results are shown in Tables 57 and 58.

Parent was not detected in eggs or any of the tissues. In eggs, the main metabolite was identied as fluazifop acid (II) either as free acid or its lipid conjugates (56–85% TRR). In liver, kidney and muscle, the main metabolite was fluazifop acid (II, free, 51–70% TRR) while in fat (subcutaneous/peritoneal) the major metabolite were fluazifop lipid conjugates (respectively 71% and 65% TRR). The lipid conjugates in egg yolk and fat tissue co-chouromatographed with the two isomeric dipalmityl triglyceride esters of fluazifop (compound 16) and fluazifop acid (II) could be released upon hydrolysis.

Reviewer's conclusion: Fluazifop acid (II) is stable when refluxed in 0.1 M NaOH for 1–3 hours or 0.5 M NaOH in methanol for 1 hour as used to cleave organo soluble conjugates in fat, kidney and whole eggs (see hydrolytic stability section above). It is not clear whether fluazifop acid (II) remains stable after 3 hours of reflux with 0.5 M NaOH in methanol as used to cleave

organosoluble conjugates in egg yolks or after 1 hour reflux in 0.5 M HCl used to cleave organosoluble conjugates in kidney. Despyridynyl acid, CF3-pyridone (X) and Pyr-Ph ether (IV) were not detected. The presence of fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified, but they may be present in the organic fractions. Fractions that were not subjected to hydrolysis (hexane, diethyl ether, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 8.9–49.1% TRR.

Table 57 Nature of residues in hen eggs treated with fluazifop-butyl (as % TRR).

	Egg yolk day 8; (phenyl; R-enantiomer) 3.1 ppm)	egg white day 8; (phenyl; R-enantiomer 3.1 ppm)	whole egg day 6 (pyridyl R-enantiomer 2.6 ppm)
TRR mg/kg eq	0.020	0.003	0.007 [b]
fluazifop-butyl	ND	ND	ND
Total fluazifop acid (II) (free acid) (lipid conjugates)	59.7 - 12.4 - 47.3 [a]	85.1 - 85.1 -	55.8 - 15.3 - 40.5 [a]
Pyr-Ph ether (IV)	ND	ND	ND
Despyridinyl acid (III)	ND	ND	ND
CF3-pyridone (X)	ND	ND	ND
Fluazifop alcohol (XXXIV)	no ref std	no ref std	no ref std
Hydroxyfluazifop acid (XL)	no ref std	no ref std	no ref std
Organo soluble unknowns - not hydrolysed (hexane) - not hydrolysed (diethyl ether) -after hydrolysis	20.5 - 10.9 - 5.3 - 4.3	1.9 - - 1.9 -	23.8 - 18.7 - 2.4 - 2.7
water soluble unknowns -not hydrolysed -after hydrolysis	3.4 - 2.0 - 1.4	2.3 - 2.3 -	6.3 - 3.1 - 3.2
post extraction solids - not hydrolysed	7.6	10.7	14.7
Total	91.2	100.0	100.6
Total identified	59.7	85.1	55.8
Total not hydrolysed	25.8	14.9	38.9

[a] Co-chouromatographed with compound 16 before hydrolysis

[b] Calculated based on mass fractions and residues in egg white (31.6 g; 0.002 mg/kg eq) and egg yolk (15.6 g; 0.016 mg/kg eq)

Table 58 Nature of residues in hen tissues treated with fluazifop-butyl (as % TRR)

	Liver (phenyl; R-enant) 3.1 ppm)	liver (pyridyl R-enant 2.6 ppm)	kidney; (phenyl; R-enant) 3.1 ppm)	kidney (pyridyl R-enant 2.6 ppm)	muscle; (phenyl; R-enant) 3.1 ppm)	muscle (pyridyl R-enant 2.6 ppm	fat; (phenyl; R-enant) 3.1 ppm)	fat (pyridyl R-enant 2.6 ppm
TRR mg/kg eq	0.027	0.077	0.056	0.44	0.005	0.010	0.043	0.034
fluazifop-butyl	ND	ND	ND	ND	ND	ND	ND	ND
Fluazifop acid (II) (free acid) (lipid conjugates)	69.7 - 69.7 -	65.9 - 65.9 -	57.6 - 57.6	54.1 - 54.1	51.3 - 51.3 -	68.0 - 68.0 -	70.8 - - 70.8	65.3 - - 65.3 [a]
Pyr-Ph ether (IV)	ND	ND	ND	ND	ND	ND	ND	ND
Despyridinyl acid (III)	ND	ND	ND	ND	ND	ND	ND	ND
CF3-pyridone (X)	ND	ND	ND	ND	ND	ND	ND	ND
Fluazifop alcohol (XXXIV)	no ref std	no ref std	no ref std	no ref std	no ref std	no ref std	no ref std	no ref std
Hydroxyfluazifop acid (XL)	no std	no std	no std	no std	no std	no std	no std	no std
organosoluble unknowns - metabolite M [b] - not hydrolysed (hexane)	17.9 - - 12.7	15.6 - -10.2	28.7 - 10.3 - 7.4	15.3 - - 0.9	15.5 - - 10.2	13.5 - - 6.8	6.8 - -	21.2 - - 12.0

	Liver (phenyl; R-enant) 3.1 ppm)	liver (pyridyl R-enant 2.6 ppm)	kidney; (phenyl; R-enant) 3.1 ppm)	kidney (pyridyl R-enant 2.6 ppm)	muscle; (phenyl; R-enant) 3.1 ppm)	muscle (pyridyl R-enant 2.6 ppm	fat; (phenyl; R-enant) 3.1 ppm)	fat (pyridyl R-enant 2.6 ppm
TRR mg/kg eq	0.027	0.077	0.056	0.44	0.005	0.010	0.043	0.034
- not hydrolysed (ether) - after hydrolysis	- 5.2 -	- 5.4 -	- 11 -	- 9.6 - 4.8	- 5.3 -	- 6.7 -	- - 6.8	- - 9.2
water soluble unknowns -not hydrolysed -after hydrolysis	2.4 - 2.4 -	4.6 - 4.6 -	5.2 - 5.2 -	13.8 - - 13.8	15.6 - 15.6 -	10.5 - 10.5 -	2.1 - 1.2 - 0.9	3.8 - 3.8 -
PES: not hydrolysed	5.1	7.0	6.5	11.6	18.0	9.4	7.7	9.7
Total	95.1	93.1	98.0	94.8	100.4	101.4	87.4	100.0
Total identified	69.7	65.9	57.6	54.1	51.3	68.0	70.8	65.3
Total not hydrolysed	25.4	27.2	40.4	22.1	49.1	33.4	8.9	25.5

[a] Co-chouromatographed with compound 16 before hydrolysis

[b] Partitioned into diethyl ether at pH 1 (without hydrolysis) and was also detected in faeces (phenyl label only)

Livestock study 4

Metabolism of fluazifop-P-butyl (R-enantiomer) was studied in laying hens [Robertson and Hand, 1999, PP5/0595, report RJ2839B]. Five laying hens (30 weeks old; 1.4-1.7 kg, Ross Hi-Sex) per label were treated with gelatine capsules containing ^{14}C- phenyl- or ^{14}C- pyridyl-labelled fluazifop-P-butyl (R-enantiomer). The hens were dosed twice daily during 10 consecutive days via a gelatine capsule. The actual mean daily dose level was 1.33 mg/hen/day (0.84 mg/kg bw/day), i.e. equivalent to 9 ppm dry feed (range 8–10 ppm). Excreta were collected prior to dosing and at 24 hour intervals thereafter until sacrifice. The cages were washed after each collection and the rinses retained for analysis. Eggs were collected prior to dosing and twice daily after dosing (24–240 hours). Hens were sacrificed 24 hours after the last dose to determine residues in tissues (liver, breast and thigh muscle, abdominal fat, skin with underlying fat). In addition the contents of the gastrointestinal tract and any eggs within the oviduct were taken for analysis. Carcass and partially-formed eggs were retained. Samples were stored at -10 °C or lower until analysis for a maximum of 9 months.

All samples were solubilized or combusted and total radioactivity was measured using LSC and expressed as fluazifop-butyl equivalents. The total recovery of the administered dose was 93% TAR for the phenyl-label, and 95% TAR for the pyridyl-label. The majority of the radioactivity was recovered in the excreta (90% TAR for the phenyl-label and 93% TAR for the pyridyl-label).

Residues in tissues are shown in Table 59. All tissues with residues > 0.01 mg/kg were investigated in order to characterize the nature of the residue.

Total radioactive residues in egg white for the phenyl label ranged from 0.007 to 0.011 mg/kg eq and had reached a plateau of 0.009 mg/kg eq after 24 hours of dosing. TRRs in egg yolk ranged from < 0.001 to 0.078 mg/kg eq and had reached a plateau of 0.072 mg/kg eq after 144 hours of dosing. Total radioactive residues in egg white for the pyridyl label ranged from 0.007 to 0.033 mg/kg eq and had reached a peak level of 0.033 mg/kg eq between 120–168 hours of dosing. TRRs in egg yolk ranged from < 0.001 to 0.231 mg/kg and had reached a plateau of 0.231 mg/kg eq after 216 hours of dosing.

Radioactivity (91–98% TRR) was extracted from egg yolk successively by dichloromethane (71–82% TRR), acetonitrile (13–14% TRR), acetonitrile/water and acetone. The dichloromethane extracts were partitioned with hexane and the hexane fraction saponified with methanolic base (temperature and duration not stated), partitioned with diethyl ether and then with acetonitrile and hexane. The acetonitrile extracts were combined, saponified with methanolic base, and partitioned with diethyl ether.

Radioactivity (88–99% TRR) was extracted from egg white by acetonitrile (81–93% TRR), acetonitrile/water and acetone. The acetonitrile extracts were combined and partitioned with diethyl ether and/or with diethyl ether at pH 2.

Radioactivity (23% TRR) was extracted from the thigh muscle (pyridyl label only) by acetonitrile (28% TRR), while 77% TRR remained in the solids. Muscle was not investigated further.

Radioactivity (48% TRR) was extracted from the liver (pyridyl label only) by acetonitrile (28% TRR), acetonitrile/water (18%) and acetone. The acetonitrile extracts were combined and partitioned with diethyl ether and with diethyl ether at pH 2. Remaining solids (52% TRR) were subjected to alkaline hydrolysis (0.1 M NaOH at 25 °C for 19 hours).

Radioactivity (92–100%TRR) was extracted from skin (with attached fat) and from abdominal fat by dichloromethane (78–99% TRR), acetonitrile (0.8–9.6% TRR), acetonitrile/water, and acetone. The dichloromethane extracts were then saponified with 10% methanol in 10 M NaOH solution (2–3 hours at 50 °C) and partitioned to diethyl ether and then to acetonitrile, and hexane.

Selected organic phases were subjected to normal and reverse phase TLC (7 solvent systems) and HPLC. Identification of residues was carried out by co-chouromatography against reference markers for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV) and CF3-pyridone (X). The nature of the TRRs in eggs is summarized in Table 60. The nature of the TRRs in fat and liver of hens is summarized in Table 61.

Tissues were extracted and chouromatographically profiled within 6 months of necropsy, but further analysis was conducted after this 6 month period. Chouromatographic profiles taken 7 months after the initial profile showed no significant changes in the nature of the residue.

The study demonstrates that fluazifop-P-butyl is de-esterified by the hen to generate fluazifop, which is readily conjugated to lipid moieties in fatty tissues. The residue in egg yolks and in the fat was mainly lipid-conjugated fluazifop, while it was mainly free fluazifop acid (II) in egg white and in the liver. The parent molecule was only detected at low levels in the liver and Pyr-Ph ether (IV) was also detected at low levels in egg white.

Reviewer's conclusion: Fluazifop acid (II) is stable during 1–3 hours reflux in 0.1 M NaOH and therefore it is assumed that fluazifop acid (II) is also stable in 0.1 M NaOH at 25 °C for 19 hours as used for liver. However, when the liver residues are compared to those found in the hen metabolism study 1, fluazifop acid (II) residues are much lower, suggesting that the hydrolysis conditions are too soft to release fluazifop acid (II). It is not clear whether fluazifop acid (II) is stable during saponification with methanolic base (temperature and duration not stated) as used for egg yolks and 10% methanol in 10 M NaOH solution (2–3 hours at 50 °C) as used for skin with fat. Relative fluazifop acid (II) levels in egg yolks are higher than in hen study 1 and relative fluazifop acid (II) levels in fat are similar to the levels in hen study 1, suggesting that fluazifop acid (II) is stable under the conditions used. Radiovalidation in SOP RAM 331/01 used 0.2 M NaOH in methanol (1 hour at 60 °C) and was able to cleave the same amount of fluazifop acid (II) from eggs, also suggesting that fluazifop acid (II) remains stable during saponification. The presence of fluazifop alcohol (XXXIV) and hydroxyfluazifop acid (XL) was not verified. Fractions that were not subjected to hydrolysis (organic, water, PES) may contain some additional fluazifop acid (II), Pyr-Ph ether (IV), despyridinyl acid (III)/CF3-pyridone (X), fluazifop alcohol (XXXIV) and/or hydroxyfluazifop acid (XL) at a maximum of 1.0–37.2% TRR

Table 59 Total radioactive residues in hens dosed with phenyl-labeled and pyridyl-labeled fluazifop-butyl

Tissue	Phenyl-label R-enantiomer (9 ppm) mg/kg eq		Pyridyl-label R-enantiomer (9 ppm) mg/kg eq	
	direct	summed	direct	summed
Egg Yolk, maximum level	0.078	0.076	0.231	0.267
Egg White, maximum level	0.011	0.011	0.033	0.034
Liver	0.007	-	0.028	0.027
Muscle (breast)	0.002	-	0.005	-
Muscle (Thigh)	0.009	-	0.012	0.011

Tissue	Phenyl-label R-enantiomer (9 ppm) mg/kg eq		Pyridyl-label R-enantiomer (9 ppm) mg/kg eq	
Skin with fat attached	0.041	0.042	0.064	0.054
Abdominal fat	0.138	0.149	0.236	0.156

direct = direct quantification of the sample by combustion LSC

summed = summation of radioactivity in extracts and post extracted solids.

Table 60 Nature of the residue in hen (eggs) dosed with fluazifop-butyl

	^{14}C-phenyl label R-enantiomer (9 ppm)		^{14}C-pyridyl label R-enantiomer (9 ppm)	
	Yolk (240 Hour) 0.077 mg/kg eq	White (192 Hour) 0.011 mg/kg eq	Yolk (240 Hour) 0.266 mg/kg eq	White (144 Hour) 0.034 mg/kg eq
	%TRR	%TRR	%TRR	%TRR
Parent	ND	ND	ND	ND
Total fluazifop acid (II, free + conj) Fluazifop (free) Fluazifop (lipid conjugates)	57.2 -10.0 (0.5+2.3+7.2) -47.2 (40.4+6.8)	85.6 -85.6 -	56.8 -8.4 (0.4+1.7+6.3) -48.4 (43.3+5.1)	73.3 -73.3 -
Pyr-Ph ether (IV) - free	ND	ND	ND	1.1
Despyridinyl acid (III)	ND	ND	ND	ND
CF3-pyridone (X)	ND	ND	ND	ND
Fluazifop alcohol (XXXIV)	no std	no std	no std	no std
Hydroxyfluazifop acid (XL)	no std	no std	no std	no std
Organo soluble unknowns - not hydrolysed [a] - not hydrolysed lipid conjugates - not hydrolysed baseline and streaks - after saponification ([hexane) - - - after saponification (MeCN) -	39.0 - 9.1 - 2.0 - 2.5 - 8.8 -10.6 - 3.1 - 1.1 - 1.8	 - - 6.7 - - - - -	23.8 - 4.7 - 1.4 - 1.2 - 3.9 - 4.5 - 3.5 - 0.5 - 4.1	1.7 - - 1.7 - - - - -
Water soluble unknowns - not hydrolysed - after saponification (hexane) - after saponification (MeCN)	3.4 - - 2.0 - 1.4	- - - -	5.3 - - 2.1 - 3.2	6.5 - 6.5 - -
PES: not hydrolysed [a]	1.9	0.7	9.0	11.9
Total	101.5	93.0	94.9	94.5
Total identified	57.2	85.6	56.8	73.3
Total not hydrolysed [a]	15.5	7.4	16.3	20.1

TRR = Total Radioactive Residue (mg/kg fluazifop-P-butyl equivalents).

ND = not detected

[a] Not hydrolysed: may contain some additional fluazifop acid (II), Pyr-Phour ether (IV) or despyridinyl acid (III)/CF3-pyridone (X)

Table 61 Nature of residues in tissues of hens dosed with ^{14}C-fluazifop-butyl

	Abdominal Fat		Fat and Skin		Liver
	^{14}C-Phenyl R-enantiomer (9 ppm in feed)	^{14}C-Pyridyl R-enantiomer (9 ppm in feed)	^{14}C-Phenyl R-enantiomer (9 ppm in feed)	^{14}C-Pyridyl R-enantiomer (9 pp in feed)	^{14}C-Pyridyl R-enantiomer (9 ppm feed)
TRR (mg/kg eq)	0.149	0.156	0.042	0.054	0.027
	%TRR	%TRR	%TRR	%TRR	%TRR
Parent	ND	ND	ND	ND	0.7
Total fluazifop (II, free + conj) - Fluazifop (free) - Fluazifop lipid conjugates	74.3 ND - 74.3	71.9 ND - 71.9	66.9 ND - 66.9	57.5 ND - 57.5	10.6 10.6 -
Pyr-Ph ether (IV) - free	ND	ND	ND	ND	ND
Despyridinyl acid (III)	ND	ND	ND	ND	ND

CF3-pyridone (X)	ND	ND	ND	ND	ND
Fluazifop alcohol (XXXIV)	no std	no std	no std	no std	no std
Hydroxyfluazifop acid (XL)	no std	no std	no std	no std	no std
Organo soluble unknowns - not hydrolysed - not hydrolysed lipid conj - not hydrolysed baseline - after saponification - - - saponified (baseline and streaks)	18.6 - 0.8 - - -14.9 [a] - - -2.9	21.3 - 1.3 - - 2.4 8.2 [a] - 9.4	24.4 - 8.6 [c] - - - 2.4 - 3.2 - 3.3 - 6.9	25.5 - 14.2 [c] - - - 2.6 - 2.0 -1.5 -5.2	15.1 - 10.9 [b] - 1.6 - 2.6 - - - -
Water soluble unknowns - not hydrolysed - after saponification - after hydrolysis (solids)	2.0 - -2.0 -	- - - -	1.6 - - 1.6 -	- - - -	47.2 - 21.8 - - 25.4
PES - not hydrolysed - after hydrolysis	0.2 - 0.2 -	1.4 - 1.4 -	2.6 - 2.6 -	8.0 - 8.0 -	22.7 - 22.7
Total	95.1	94.6	95.5	91.0	96.4
Total identified	74.3	71.9	66.9	57.5	11.3
Total not hydrolysed	1.0	2.7	11.2	22.2	37.2

TRR = Total Radioactive Residue (mg/kg fluazifop-butyl equivalents).

[a] At least 4 components, none greater than 7.9% TRR (0.012 mg/kg eq) in the ^{14}C-phenyl label and at least 2 components none greater than 7.3% TRR (0.011 mg/kg eq) for the ^{14}C-pyridyl label

[b] At least 8 components, none greater than 5.1% TRR (0.001 mg/kg eq)

[c] At least 4 components, none greater than 8.6% TRR (0.004 mg/kg eq) in the ^{14}-C phenyl label and at least 5 components none greater than 9.6% TRR (0.005 mg/kg eq) for the pyridyl label

Overview metabolic pathway of fluazifop-P-butyl in livestock

Metabolism of fluazifop-P-butyl in livestock after oral administration has been studied in ruminants and poultry. Results of these studies show that the route of degradation is similar.

The proposed metabolic pathway of fluazifop-P-butyl in livestock is shown in Figure 3. Fluazifop-P-butyl hydrolyses rapidly within the animal. The primary metabolite found in all animal commodities was fluazifop acid (II), formed by the hydrolysis of the parent ester bond and subsequent O-debutylation. Pyr-Ph ether (IV) was identified at trace levels and is derived from cleavage of the aliphatic ether bond. All metabolites undergo extensive conjugation.

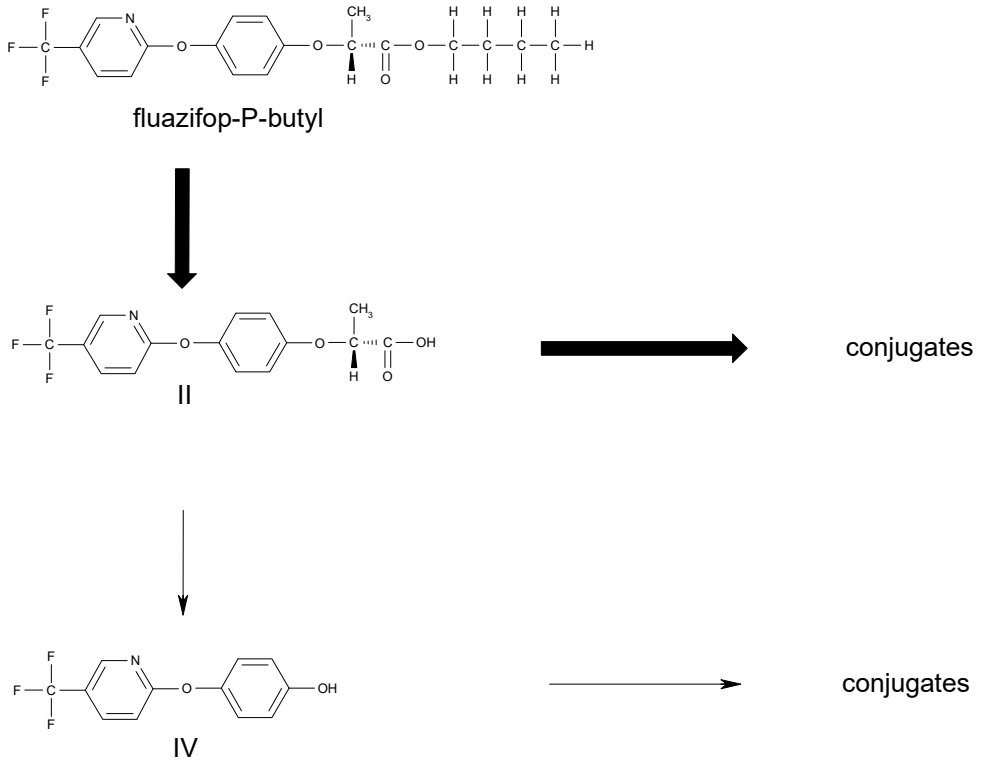


Figure 3 Proposed metabolic pathway of fluazifop-P-butyl in livestock

Environmental fate in soil

The Meeting received information on aerobic degradation and photolysis of fluazifop-P-butyl and its metabolites in soil under laboratory conditions and dissipation of fluazifop-P-butyl under field conditions. Available studies on anaerobic degradation, adsorption and desorption in/on soil were not taken into account. The fate and behaviour of fluazifop-P-butyl in the environment was investigated using [U-^{14}C-phenyl]-fluazifop-P-butyl and [U-^{14}C-pyridyl]-fluazifop-P-butyl (see Figure 1).

Aerobic degradation in soil - laboratory studies with fluazifop-butyl

Soil study 1

The rate of aerobic degradation of fluazifop-butyl (RS) was studied in two soils [Arnold *et al*., 1980, PP9/0270, report RJ0131B; Rapley *et al*., 1981, PP9/0272, report RJ0158B]. Soil characteristics are reported in Table 62. Soils were dug to a depth of 10 cm and stored at 25 °C overnight. Soils were sieved (2 mm mesh) and dispensed in glass pots (4 cm diameter, 3 cm high). The surface of a soil was treated with [U-^{14}C-phenyl]-fluazifop-butyl (RS) at a nominal equivalent field application rate of 1.0 kg ai/ha. Application to soil was 125 ug ai/30 g wet soil = 4.2 mg ai/kg wet soil. Soil samples were incubated under aerobic conditions in the dark at 25 ± 1 °C and soil moisture of 40% of the maximum water holding capacity for up to 21 weeks. Effluent air was passed thourough a series of trapping solutions (0.05 M H_2SO_4, 2-methoxyethanol and ethanol amine) to collect any volatile degradation products including CO_2. Soil and trapping solutions were sampled at 2 hours and 3, 8 and 21 weeks after treatment. Storage conditions for the samples are not reported.

Table 62 Soil characteristics

Soil name	**18 Acres**	**Lilyfield**
Location	Bracknell Berkshire, UK	Churt, Surrey, UK
Soil texture (New Jersey system)	sandy loam	sand

Soil name	18 Acres	Lilyfield
-- Sand	65.4%	90.3%
-- Silt	18.6%	2.4%
-- Clay	16.0%	7.3%
Organic Carbon (%)	-	-
Organic Matter (%) [b]	3.1%	1.4%
CEC (meq/100 g)	12.0	3.0
pH (water)	6.8	6.4
Moisture holding capacity at pF 0 (= MWHC) (%)	75.2%	27.6%
Moisture holding capacity at pF2.5 (0.33 bar) (%)	22.2%	5.6%
Moisture holding capacity at 10 bar (%)	15.8%	2.8%
% MWHC at incubation	40%	40%
Bulk density (g/mL)	-	
Microbial biomass (mg microbial carbon/kg soil)	-	

MWHC = Maximum Water Holding Capacity

Soil samples and trapping solutions were analysed by combustion LSC. Mass balances ranged from (93–102%).

Extraction efficiency for the ^{14}C treated soils was investigated in a separate study [Rapley *et al*., 1981, PP9/0272, report RJ0158B]. Six solvent mixtures were used to extract radioactive residues from the soils after 0, 3 and 8 weeks of incubation: hexane/isopropanol (50:25), dichoromethane/methanol (80:20), methanol/water (100:20), isopropanol/water (80:20), acetonitrile, acetone or combinations thereof. Soils were subjected to either cold (2 × 18 hours shaking at 25 °C) or hot (2 × 18 hours reflux) extraction. Thouroughout the periods of extraction, possible volatile products were trapped by passing a stream of air thourough the apparatus and into a series of trapping solutions containing 0.05 M H_2SO_4, 2-methoxyethanol and ethanolamine. Extraction with isopropanol/water (80:20) under reflux conditions (2 × 18 hours) resulted in slightly better extraction efficiencies for fluazifop-butyl and fluazifop acid (II). Fluazifop-butyl and fluazifop acid (II) were shown to be stable under these conditions as well as during storage at -20 °C for up to 5 weeks in the soil extracts.

The actual soil samples were twice reflux extracted with isopropanol/water (80:20, v/v) for 18 hours. All extracts were concentrated by evaporation. Soil extracts and extracted soils were analysed by (combustion) LSC. Selected extracts were analysed by TLC with 2 different solvent systems using reference standards for fluazifop-butyl (I), fluazifop acid (II), Pyr-Ph ether (IV), and compound 6.

Results are shown in Table 63. Efficiency of extraction was 99–100% TAR at 2 hours after application in both soils. As time of incubation increased, radioactivity became more difficult to extract from soil and after 21 weeks of incubation, only 8.6% TAR could be extracted from the 18 Acres soil, while still 76% TAR could be extracted from the Lilyfield soil. After 21 weeks 33% and 6% TAR evolved as $^{14}CO_2$ from 18 Acres and Lilyfield soils, respectively.

In both soils fluazifop-butyl degraded rapidly. The major degradation product resulting from hydrolysis of the butyl ester was fluazifop acid (II): peak maximum 45% TAR at 2 hours after application in 18 Acres soil and 72% TAR after 3 weeks of incubation in Lillyfield soil. Fluazifop acid (II) degraded rapidly in the “18 Acres” aerobic soil. A minor degradation product of fluazifop acid (II) occurred by hydrolysis of the propionic acid moiety to form Pyr-Ph ether (IV): peak maximum 2.3% TAR in 18 Acres soil after 3 weeks and 8.9% TAR in Lilyfield soil after 21 weeks of incubation.

Half lifes were not determined in this study.

Table 63 Distribution and characterisation of radioactivity (% TAR) in soils treated with 4.2 mg ai/kg (1.0 kg ai/ha) phenyl labelled fluazifop-butyl (RS) at 25 °C and 40% MWHC

	18 Acres				Lilyfield			
	2 hours	3 wks	8 wks	21 wks	2 hours	3 wks	8 wks	21 wks
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR

	18 Acres				Lilyfield			
	2 hours	3 wks	8 wks	21 wks	2 hours	3 wks	8 wks	21 wks
fluazifop-butyl (I)	31.1	3.1	0.7	0.9	78.8	2.7	2.4	ND
fluazifop acid (II)	44.9	6.8	1.1	1.5	11.5	71.7	63.3	61.3
Pyr-Ph ether (IV)	ND	2.3	0.8	0.7	ND	2.5	3.8	8.9
unknown extracted	22.9	7.4	8.2	5.5	9.7	12.5	13.6	6.3
post extracted solids	1.1	64.3	62.2	58.1	ND	8.4	12.8	17.5
$^{14}CO2$	ND	16.1	27.0	33.3	ND	2.2	4.1	6.0
Total	100	100	100	100	100	100	100	100

ND = not detected (<0.1% TAR)

Soil study 2

The rate of aerobic degradation of fluazifop-butyl (RS) was studied in two standard soils [Atreya and Houlden, 1981, PP9/0271, report RJ0183B]. Soil characteristics are reported in Table 64. Standard soils (equivalent to 100 g dry soil) were dispensed in conical flasks and soil moisture was adjusted to 40% maximum water holding capacity (MWHC). After equilibration for 1 week at 20 ± 2 °C, the soil was mixed with fluazifop-butyl (no radiolabel) at a rate equivalent to 1.0 mg ai/kg dry soil. The nominal equivalent field application rate in kg ai/ha was not indicated. Soil samples were incubated under aerobic conditions in the dark at 20 ± 2 °C for up to 32 weeks. Effluent air was not trapped. Soils were sampled at 0 and 3 days, and 1, 2, 4, 8, 16 and 32 weeks after treatment. Storage conditions for the samples are not reported.

Table 64 Soil characteristics

Soil name	**Speyer 2/2**	**Speyer 2/3**
Location	Germany standard soil (BBA)	Germany Standard soil (BBA)
Soil texture (New Jersey system)	Not reported	Not reported
-- Sand	84.1%	80.0%
-- Silt	4.0%	9.1%
-- Clay	12.0%	10.8%
Organic Carbon (%)	-	-
Organic Matter (%) [b]	5.7%	1.1%
CEC (meq/100 g)	11.9	6.0
pH (water)	6.4	7.7
Moisture holding capacity at pF 0 (= MWHC) (%)	43.0%	33.5%
Moisture holding capacity at pF2.5 (0.33 bar) (%)	-	-
Moisture holding capacity at 10 bar (%)	-	-
% MWHC at incubation	40%	40%
Bulk density (g/mL)	-	
Microbial biomass (mg microbial carbon/kg soil)	-	

MWHC = Maximum Water Holding Capacity

Soil samples were analysed for fluazifop-butyl by HPLC-UV method PPRAM 54 and fluazifop acid by PPRAM 55. Individual concurrent method recoveries ranged from 66–95% for fluazifop-butyl (0.1–1.0 mg/kg) and 68–95% for fluazifop acid (0.1–1.0 mg/kg). Levels in control samples were not indicated.

Results are shown in Table 65. Fluazifop-butyl degraded rapidly in both soils. Only small percentages of fluazifop-butyl remained in soils after 3 days and no fluazifop-butyl was detected after 1 week. The half-life for fluazifop-butyl was less than 3 days for both soils. Fluazifop-butyl degraded rapidly to its major metabolite fluazifop acid (II). Fluazifop acid (II) degraded in both soils with a half-life of 20 weeks in soil Speyer 2/2 and 5 weeks in soil Speyer 23 (as determined by graph). Degradation is more rapid under alkaline conditions.

Table 65 Degradation of fluazifop-butyl and fluazifop acid (II) in soils treated with 1 mg ai/kg fluazifop-butyl (RS) at 20 °C and 40% MWHC

	Speyer 2/2		Speyer 2/3	
Incubation time	Fluazifop-butyl	Fluazifop acid (II)	Fluazifop-butyl	Fluazifop acid (II)
0 day	0.88	ND	0.94	ND
3 days	0.25	0.71	0.04	0.84
1 week	< 0.01	0.64	< 0.01	0.82
2 weeks	ND	0.66	ND	0.62
4 weeks	ND	0.49	ND	0.51
8 weeks	ND	0.59	ND	0.19
16 weeks	ND	0.37	ND	0.08
32 weeks	ND	0.29	ND	0.04

ND = not detected (0.01 mg/kg)

Soil study 3a

The rate of aerobic degradation of fluazifop-butyl (RS) was studied in six soils [Harvey *et al*., 1981, PP9/0273, report RJ0197B]. Soil characteristics are reported in Table 66. Soils Speyer 2.2 and 2.3 were sampled in January 1979, then stored moist in open plastic bags until the degradation test was performed in May 1980. The other soils were sampled just before the degradation tests. Microbial biomass was not measured. Soils were sieved (2 mm mesh) and dispensed in glass pots (4 cm diameter, 3 cm high). The surface of a soil was treated with [U-^{14}C-phenyl]- or [U-^{14}C-pyridyl]-fluazifop-butyl at a nominal equivalent field application rate of 1.0 kg ai/ha (actual 0.98–1.2 kg ai/ha). Application as mg/kg soil was not reported. Soil samples were incubated under aerobic conditions in the dark at 20 ± 1 °C and soil moisture of 40% of the maximum water holding capacity for up to 45 weeks. In addition, two soils were also incubated with ^{14}C-fluazifop-butyl under other conditions: at 10× rate (actual 12 kg ai/ha), at low temperature (10 ºC), at low moisture content (15% MWHC) and sterilised by gamma irradiation or autoclaving (see Table 67). Sterile soils were treated in sterilised equipment with filter sterilised solutions. Effluent air was passed thourough a series of trapping solutions (0.05 M H_2SO_4, 2-methoxyethanol and ethanol amine) to collect any volatile degradation products including CO_2. Soil and trapping solutions were sampled at 0 and 2 days and 1, 3, 12, 24 and 45 weeks after treatment.

Table 66 Soil characteristics

Soil name	Speyer 2.2	Speyer 2.3	**18 Acres**	**Gore Hill**	**Frensham**	Rosedean
Location	Standard 1979	Standard 1979	Bracknell Berkshire, UK	Newbury, Berkshire, UK	Churt, Surrey, UK	Southery Norfolk, UK
Soil texture (USDA) [a]	loamy sand	sandy loam	sandy clay loam	calcareous clay loam	loamy sand	fen peat
-- Sand	84%	80%	58%	38%	84%	-
-- Silt	4%	9%	19%	22%	9%	-
-- Clay	12%	11%	23%	40%	7%	-
Organic Carbon (%)	-	-	-	-	-	-
Organic Matter (%)	5.7%	1.1%	4.6%	14%	2.1%	67%
CEC (meq/100 g)	12	6	16	33	7	96
pH [b]	6.4	7.7	6.0	7.4	5.4	6.7
Moisture holding capacity at pF 0 (= MWHC) (%)	52%	35%	74%	117%	45%	262%
Moisture holding capacity at pF2.5 (0.33 bar) (%)	13%	10%	18%	45%	10%	103%
Moisture holding capacity at 10 bar (%)	-	-	15%	29%	7%	90%
% MWHC at incubation	40%	40%	40%	40%	40%	40%
Bulk density (g/mL)	-	-	-	-	-	-
Microbial biomass (mg microbial carbon/kg soil)	-	-	-	-	-	-

[a] Classification according to United States Department of Agriculture (USDA)

[b] pH, not clear whether given as pH (water) or as pH ($CaCl_2$)
MWHC = Maximum Water Holding Capacity

Table 67 Other conditions

Soil	Label	Condition
18 Acres	phenyl	1 kg ai/ha, 10 ºC, 40% MWHC
	phenyl	1 kg ai/ha, 20 ºC, 15% MWHC
	phenyl	10 kg ai/ha, 20 ºC; 40% MWHC
	phenyl	1 kg ai/ha; 20 ºC; 40% MWHC, autoclaved for 15 min at 120 ºC for 3 consecutive days
	phenyl	1 kg ai/ha, 20 ºC; 40% MWHC, gamma-irradiated at one 5 M rad dose
Gore Hill	phenyl	1 kg ai/ha; 20 ºC; 40% MWHC, autoclaved for 15 min at 120 ºC for 3 consecutive days
	phenyl	1 kg ai/ha, 20 ºC; 40% MWHC, gamma-irradiated, one 5 M rad dose

Soil samples and trapping solutions were analysed by combustion LSC. Mass balances were on average 99% (87–108%) in the non-sterile soils and 155% (148–188%) in the sterile soils, which is thought to be due to an application error (1.5 kg ai/ha instead of 1.0 kg ai/ha).

The soil samples were sequentially extracted with isopropanol, isopropanol/water (80:20, v/v), acetone/concentrated HCl (98:2, v/v) and acetone/water/concentrated HCl (80:20:2, v/v). Each extraction was carried out for 18 hours by Soxhlet or reflux. Acetone containing extracts were rotary evaporated to remove the acetone and then partitioned either with diethylether or chloroform followed by diethylether. Some extracts required further clean-up on an anhydrous sodium sulphate column and elution with chloroform or methanol. All extracts were concentrated by evaporation. Soil extracts and extracted soils were analysed by (combustion) LSC. Selected extracts were analysed by 1D- and 2D-TLC with 3 different solvent systems using reference standards for fluazifop-butyl (I), fluazifop acid (II), despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X) and compounds 5, 6, 7, 8, 9. In addition, the identity of fluazifop acid (II), Pyr-Ph ether (IV), and CF3-pyridone (X) were further characterised by GC-MS, HPLC-UV and/or GC with paralel flame ionisation and radiogas detectors. Results are shown in Table 68 and Table 69.

Using the critera in FOCUS (2006) thouree soils are considered not representative:

- The soil Rosedean (peat soil, phenyl label) had a very high organic content of 67%.
- The soils Speyer 2.2 (loamy sand, phenyl label) and Speyer 2.3 (sandy loam, phenyl label) were stored for over a year before the beginning of the laboratory study and were excluded because they had too low microbial activity.

Although microbial mass was not measured, the remaining thouree soils are considered viable, because they were sampled just before the degradation tests started. Tests were conducted at 20 ºC with a moisture content of 40% MWHC. Using the critera in FOCUS (2006) the remaining thouree trials are performed at optimum moisture conditions.

In the thouree remaining soils (18 Acres, Gore Hill, Frensham), fluazifop-butyl was rapidly degraded: 1.2–2.4% TAR remained after 2 days. The major degradation product resulting from hydrolysis of fluazifop-butyl was fluazifop acid (II, maximum 78–83% TAR after 2 days of incubation). Degradation of fluazifop acid (II) occurred both by ether cleavage resulting in the formation of CF3-pyridone (X, maximum 22–25% TAR after 12 weeks of incubation, pyridyl label only), and by hydrolysis of the propionic acid moiety to form Pyr-Ph ether (IV, <4% TAR at all time points). In the phenyl label experiment, < 2% TAR co-chouromatographed with compound 7. Further decomposition occurred by cleavage of both the phenyl and pyridyl rings resulting in a steady increase of $^{14}CO_2$ evolution with time (up to 25–36% TAR at 45 weeks of incubation). The remaining unidentified extracted radioactivity (maximum 20–46% TAR after 12–45 weeks of incubation) remained at or near the origin of the TLC plates, even with more polar solvent systems on both normal and reverse phase TLC. As time of incubation increased, radioactivity became more difficult to extract from soil and 28–55% TAR remained unextracted after 12–45 weeks of incubation. In Gore

Hill soil with high organic matter, the unextracted residues tend to be higher than in other soils (48–55% TAR after 12–45 weeks).

In the thouree representative soils (18 Acres, Gore Hill, Frensham) the half life of fluazifop acid (II) ranged from less than 3 weeks to 3 weeks (based on graphical representation). In the other thouree soils (Rosedean, Speyer 2.2, Speyer 2.3) the half life of fluazifop acid (II) was much longer and ranged from less than 24 weeks (Rosedean, 74% peat; Speyer 2.3 non-viable soil) to more then 24 weeks (Speyer 2.2, non-viable soil). Half-lifes for Pyr-Ph ether (IV) and CF3-pyridone (X) were not reported.

Incubation of fluazifop-butyl under less favourable conditions (i.e. at high rate, at low temperature, at low moisture content) did not affect the rapid hydrolysis of the parent, but it decreased the rate of fluazifop acid (II) breakdown. The half-life of fluazifop acid (II) increased from less than 3 weeks at 20 °C (40% MWHC) to approximately 3 weeks at 10 °C (40% MWHC) and to more than 3 weeks at 15% MWHC (20 °C) or at higher dose of 10 kg ai/ha (20 °C, 40% MWHC). The degradation of fluazifop acid (II) was mediated by microbial activity as evidenced by the virtual absence of degradation in sterilised soils, where 81–93% TAR remained as fluazifop acid (II) after 12 weeks of incubation.

Table 68 Distribution and characterisation of radioactivity (% TAR) in soils treated with (1.0 kg ai/ha) phenyl or pyridyl labelled fluazifop-butyl (RS) at 20 °C and 40% MWHC

	18 Acres, pyridyl label					18 Acres, phenyl label						
Duration (wks)	0	3	12	24	45	0	0.3	1	3	12	24	45
fluazifop-butyl (I)	97.9	1.2	ND	ND	ND	95.1	2.4	7.6	1.6	ND	ND	ND
fluazifop acid (II)	ND	41.5	3.7	1.3	0.9	ND	78.0	61.3	19.9	1.2	1.1	0.4
Pyr-Ph ether (IV)	ND	2.4	2.7	1.2	1.2	ND	1.0	2.7	3.3	1.6	1.0	0.6
CF3-pyridone (X)	ND	13.2	25.1	12.0	9.8	-	-	-	-	-	-	-
unkn extracted	1.8	8.4	32.2	46.0 [b]	37.1	1.6	5.6	11.0	8.2	35.1	37.7 [b]	33.5
acidic extracts	[a]	4.6	[a]	[a]	[a]	3.2	2.4	4.5	4.8	[a]	[a]	[a]
PES	0.2	26.5	27.5	21.7	25.6 [c]	0.2	10.1	16.2	49.3	35.7	28.3	30.6 [c]
$^{14}CO2$	ND	2.3	8.9	17.7	25.4	ND	0.4	2.8	12.8	26.2	31.9	34.8
Total	99.9	100.1	100.1	99.9	100.0	100.1	99.9	106.1	99.9	99.8	100.0	99.9
	Gore Hill, pyridyl label					Gore Hill, phenyl label						
Duration (wks)	0	3	12	24	45	0	0.3	1	3	12	24	45
fluazifop-butyl (I)	92.0	ND	ND	ND	ND	94.9	1.2	0.7	ND	ND	ND	ND
fluazifop acid (II)	0.2	42.6	2.2	0.9	0.2	1.9	83.4	68.8	40.0	1.2	0.8	0.6
Pyr-Ph ether (IV)	ND	2.0	0.9	1.1	0.8	ND	0.9	1.7	2.7	1.0	1.7	1.0
CF3-pyridone (X)	ND	16.3	22.0	9.8	7.9	-	-	-	-	-	-	-
unkn extracted	7.3	6.7	25.5	28.5	21.3	2.7	5.2	7.7	8.2	20.2	14.4	13.2
acidic extracts	[a]	[a]	[a]	[a]	[a]	[a]	0.4	2.4	[a]	[a]	2.9	[a]
PES	0.5	31.0	39.6	40.9	43.6	0.4	8.8	16.4	42.6	55.4	48.0	49.4
$^{14}CO2$	ND	1.5	9.6	18.8	26.3	ND	0.1	2.3	6.5	22.5	32.2	35.8
Total	100.0	100.1	99.8	100.0	100.1	99.9	100.0	100.0	100.0	100.3	100.0	100.0
	Rosedean, phenyl label					Frensham, phenyl label						
Duration (wks)	0	3	12	24	45	0	0.3	1	3	12	24	45
fluazifop-butyl (I)	93.6	1.3	ND	ND	ND	96.3	-	-	0.6	ND	ND	-
fluazifop acid (II)	ND	83.7	68.3	8.0	2.5	ND	-	-	49.1	16.2	2.6	-
Pyr-Ph ether (IV)	ND	0.8	1.5	1.8	0.9	ND	-	-	2.2	1.2	1.2	-
unkn extracted	1.9	5.9	7.8	12.0	13.4	3.5	-	-	17.1	17.7	34.0	-
acidic extracts	4.3	4.5	2.6	0.5	[a]	[a]	-	-	[a]	11.9	[a]	-
PES	0.1	3.2	13.5	54.3	51.2	0.2	-	-	23.2	29.9	30.2	-
$^{14}CO2$	ND	0.6	6.2	23.3	32.0	ND	-	-	7.6	23.0	31.9	-
Total	99.9	100.0	99.9	99.9	100.0	100.0	-	-	99.8	99.9	99.9	-
	Speyer S2.2, phenyl label					Speyer S2.3, phenyl label						
Duration (wks)	0	3	12	24	45	0	0.3	1	3	12	24	45
fluazifop-butyl (I)	94.8	3.2	1.1	ND	-	93.1	-	-	0.7	0.2	ND	-
fluazifop acid (II)	ND	88.3	74.9	70.9	-	ND	-	-	80.0	51.2	31.5	-
Pyr-Ph ether (IV)	ND	1.0	2.5	1.9	-	ND	-	-	2.0	2.5	2.0	-
unkn extracted	1.6	2.6	13.4	11.9	-	2.1	-	-	3.0	12.5	10.3	-
acidic extracts	3.6	3.0	1.5	1.6	-	4.6	-	-	4.6	4.9	1.5	-

	18 Acres, pyridyl label					18 Acres, phenyl label						
Duration (wks)	0	3	12	24	45	0	0.3	1	3	12	24	45
PES	0.1	1.4	3.3	5.8	-	0.1	-	-	8.2	17.4	42.2	-
$^{14}CO2$	ND	0.5	3.2	7.9	-	ND	-	-	1.5	8.2	12.0	-
Total	100.1	100.0	99.9	100.0	-	99.9	-	-	100.0	96.9	99.5	-

[a] radioactivity in acidic extracts was greater than 5% TAR and was analysed by TLC and results were distributed over I, II, IVand unknown fractions.

[b] further analysed in [Harvey *et al.*, 1981, PP9/0737, no report number]. the majority of the radioactivity in these extracts is thought to be associated with extracted organic matter and is not easily separable from it.

[c] further analysed in [Harvey and Hill, 1983, PP9/0274, report RJ0336B]: the majorirty of the radioactivity was highly polar in nature and much of the extracted radiocarbon was incorporated into the soil organic matter.

Table 69 Distribution of radioactivity (% TAR) in soils treated at various conditions

	18 Acres, phenyl label, 1 kg ai/ha, at 10 °C, 40% MWHC					18 Acres, phenyl label, 1 kg ai/ha, at 20 °C, autoclaved			
	0	1	3	12	24	0	0.3	3	12
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
fluazifop-butyl (I)	97.9	-	ND	ND	ND	97.7	96.3	89.1	1.8
fluazifop acid (II)	ND	-	56.9	3.4	1.7	ND	ND	3.3	90.3
Pyr-Ph ether (IV)	ND	-	3.1	12.9	0.8	ND	ND	ND	ND
unknown extracted	1.8	-	12.7	34.7	45.2 [b]	2.1	3.5	6.6	2.7
acidic extracts	[a]	-	[a]	[a]	[a]	[a]	[a]	[a]	4.9
post extracted solids	0.2	-	23.6	38.0	33.4	0.2	0.2	0.9	0.3
$^{14}CO2$	ND	-	3.8	11.1	18.7	ND	ND	ND	ND
Total	99.9	-	100.1	100.1	99.8	100.0	100.0	99.9	100.0
	18 Acres, phenyl label, 1 kg ai/ha, at 20 °C, 15% MWHC					18 Acres, phenyl label, 1 kg ai/ha, at 20 °C, gamma irradiated			
	0	1	3	12	24	0	0.3	3	12
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
fluazifop-butyl (I)	97.1	1.8	1.1	-	ND	92.4	15.6	ND	ND
fluazifop acid (II)	0.7	92.0	86.1	-	20.8	ND	79.9	90.9	81.2
Pyr-Ph ether (IV)	ND	ND	ND	-	1.8	ND	ND	ND	1.3
unknown extracted	2.0	4.2	7.1	-	36.7	2.7	3.6	8.3	6.6
acidic extracts	[a]	0.9	[a]	-	[a]	4.7	[a]	[a]	4.7
post extracted solids	0.3	1.0	5.1	-	23.6	0.2	0.9	0.8	5.8
$^{14}CO2$	ND	0.1	0.5	-	17.1	ND	ND	ND	0.4
Total	100.1	100.0	99.9	-	100.0	100.0	100.0	100.0	100.0
	18 Acres, phenyl label, 10 kg ai/ha, at 20 °C, 40% MWHC					Gore Hill, phenyl label, 1 kg ai/ha, at 20 °C, autoclaved			
	0	1	3	12	24	0	0.3	3	12
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
fluazifop-butyl (I)	97.6	-	ND	-	ND	96.6	-	14.5	ND
fluazifop acid (II)	0.2	-	62.1	-	12.4	0.1	-	80.0	92.7
Pyr-Ph ether (IV)	ND	-	1.4	-	1.5	ND	-	ND	ND
unknown extracted	2.1	-	6.0	-	29.0	3.0	-	5.1	5.0
acidic extracts	[a]	-	4.4	-	5.6	[a]	-	[a]	0.8
post extracted solids	0.2	-	21.8	-	31.6	0.3	-	1.2	1.2
$^{14}CO2$	ND	-	4.3	-	20.1	ND	-	ND	ND
Total	100.1	-	100	-	100.2	100.0	-	100.8	99.7
						Gore Hill, phenyl label, 1 kg ai/ha, at 20 °C, gamma irradiated			
						0	0.3	3	12
Characterisation						%TAR	%TAR	%TAR	%TAR
fluazifop-butyl (I)						93.3	3.1	ND	ND
fluazifop acid (II)						3.2	91.6	92.7	85.4
Pyr-Ph ether (IV)						ND	ND	ND	ND
unknown extracted						2.8	3.8	6.6	5.0
acidic extracts						[a]	[a]	[a]	0.8
post extracted solids						0.6	1.5	0.7	1.2
$^{14}CO2$						ND	ND	ND	ND
Total						99.9	100.0	100.0	92.4

[a] radioactivity in acidic extracts was greater than 5% TAR and was analysed by TLC and results were distributed over I, II, IVand unknown fractions.

[b] further analysed in [Harvey *et al*., 1981, PP9/0737, no report number]. The majority of the radioactivity in these extracts is thought to be associated with extracted organic matter and is not easily separable from it.

Soil study 3b

In a first addendum [Harvey *et al*., 1981, PP9/0737, no report number] empirically based studies were conducted to establish if the unidentified extracted material, remaining at the origin of the TLC plates, was either a single component or a mixture. Thouree samples were chosen from the 24 weeks incubation timepoint of the Acres soil: phenyl label (38% TAR at origin), pyridyl label (46% TAR at origin), phenyl label low temperature (45% TAR at origin). Partition of the soil extracts with diethyl ether and/or chloroform and subsequent clean-up on an anhydrous sodium sulphate adsorption column (aqueous phase) or acid hydrolysis with 6 M HCl (60 ºC, 1 hour, organic phase) had no effect on the chouromatographic behaviour of the residues at the origin of the TLC plate (various solvent systems on normal and reverse phase TLC). Alkaline hydrolysis (2 M NaOH, 100 ºC, 1 hour) released a small amount of material (< 3% TAR) that moved away from the origin. Trace amounts co-chouromatographed with fluazifop acid (II), despyridinyl acid (III) and Pyr-Ph ether (IV).

The majority of the radioactivity in these extracts is therefore thought to be associated with extracted organic matter and not easily separable from it. The absence of movement on normal and reverse phase TLC, despite the use of a range of solvents suggests these materials will equally not be able to move within soil as they are tightly associated with the soil organic matter (i.e., bound).

In a second addendum [Harvey and Hill, 1983, PP9/0274, report RJ0336B] the remaining soil solids were re-extracted with isopropanol/water (80:20, v/v, 18 hour reflux), acetone/water/concentrated HCl (80:20:2, v/v, 18 hour reflux, twice) and 0.5 M NaOH (1 hour, ambient temperature, twice). Two samples were chosen from the 45 weeks incubation timepoint of the Acres soil: phenyl label (31% TAR solids), pyridyl label (26% TAR solids). A further 2% TAR could be released by isopropanol/water, 5–8% TAR could be released by acetone/water/HCl and a further 10–14% TAR could be released by 0.5 M NaOH. Fractionation by solvent partition and separation of the humin, humic and fulvic acid organic matter fractions, followed by analysis by TLC, showed the residues to be composed of a number of products, each of small proportions (< 5% TAR). The isopropanol/water extracts contained trace amounts of fluazifop acid (II), Pyr-Ph ether (IV) and CF3-pyridone (X). The remaining material was highly polar in nature and the distribution of the radioactivity over the different fractions suggested that much of the extracted radiocarbon was incorporated into the soil organic matter.

Soil study 4

The rate of aerobic degradation of fluazifop-P-butyl (R-enantiomer) was studied in a sandy clay loam soil at a temperature of 20 °C [Graham and Gilbert, 2009, PP5/10033, report 1983/104-D2149(2)]. Soil characteristics are reported in Table 70. The soil was treated with [U-^{14}C-phenyl]- or [U-^{14}C-pyridyl]-fluazifop-P-butyl at an actual rate of 0.67–0.68 mg ai/kg dry soil, corresponding with a field application rate of 0.50 kg ai/ha. The active substance was incorporated into the soil to a depth of 5 cm. Soil samples were incubated under aerobic conditions in the dark at 20 ± 2°C under moist conditions (pF2) for up to 120 days. Samples were taken at DAT = 0, 1, 4, 7, 28, 61, 90, 120. Any volatile radioactivity was continously flushed from the vessels and collected in ethanediol, 2% liquid paraffin in xylene and 2 M NaOH traps. All samples generated during the study were extracted at the time of sampling.

Table 70 Soil characteristics

Soil name	18 Acres
Location	Bracknell, Berkshire, UK
Soil texture (USDA) [a]	Sandy clay loam
Sand (0.05-2 mm)	51%
Silt (0.002-0.05 mm)	24%

Clay (< 0.002m m)	25%
Organic Carbon (%)	2.7%
Organic Matter (%) [b]	4.7%
CEC (meq/100 g)	18.9
pH (water)	6.5
pH ($CaCl_2$)	6.2
Maximum water holding capacity (MWHC, %) (at pF0)	55.9%
Moisture holding capacity @ 0.33 bar (w/w %) (at pF 2.5)	23.6%
Moisture holding capacity @ 0.1bar (w/w %) (at pF 2.0)	29.8%
Moisture holding capacity at incubation (w/w %) (at pF 2.0)	29.8%
Bulk density (g/mL)	1.1
Microbial biomass (mg microbial carbon/kg soil)	
Initial	539.5 (i.e. 2.0% of soil organic carbon)
After 120 days	-

[a] Classification according to United States Department of Agriculture (USDA)

[b] Organic matter = organic carbon content × 1.7

The soil samples were extracted at ambient temperature using neutral/mild acidic extractions (acetonitrile alone or combined with water and either acetic acid or formic acid in various combinations). Both cold shake and reflux conditions were utilized to ensure optimum extractability. Remaining solids were subjected to mild basic extraction (0.5 M ammonia) followed by a basic extraction (1 M NaOH, reflux) and/or a harsh acidic extraction (acetonitrile/water/concentrated HCl, 80:20:2, v/v/v). The 1 M NaOH extract was acidified to pH1 and separated into precipitate (humic acid fraction) and supernatant (fulvic acid fraction). The precipitate was reconstituted in 0.5 M NaOH.

Radioactivity in soil extracts and remaining solids was determined by (combustion) LSC. The mass balance was determined by summation of the radioactivity recovered in the soil extracts, remaining solids and liquid traps. Mass balances ranged between 92–104% of the applied radioactivity. Soil extracts were analysed by HPLC-UV with radio-detection, TLC and HPLC-MS using reference standards for fluazifop-butyl (I), fluazifop acid (II), Pyr-Ph ether (IV) and CF3-pyridone (X).

Results are shown in Tables 71 and 72. Fluazifop-P-butyl was degraded rapidly (3–4% TAR remained after 1 day) with the formation of two major identified metabolites (fluazifop acid (II) and CF3-pyridone (X)) and one minor metabolite (Pyr-Ph ether (IV)). Fluazifop acid (II) accounted for a maximum of 67–69% TAR at DAT 1 and then decreased to < 2% TAR at DAT 120. CF3-pyridone (X) accounted for a maximum of 25% TAR at DAT 28 and then decreased to 17% TAR at DAT 120. Pyr-Ph ether (IV) accounted for a maximum of 4% TAR at DAT 1 and then decreased to 1% TAR at DAT 120. Carbon dioxide evolution increased to 17% and 35% TAR at DAT 120 for the pyridyl and pheny label respectively.

In the mild basic extracts, additional fluazifop acid (II), Pyr-Ph ether (IV) and CF3-pyridone (X) could be released, accounting for a maximum of 2%, 1% and 7% TAR at DAT 28. In the fulvic acid fractions derived from the sodium hydroxide extracts, additional fluazifop acid (II), Pyr-Ph ether (IV) and CF3-pyridone (X) could be released, accounting for a maximum of 7%, 3% and 2% TAR at DAT 28. Furthermore, an unknown metabolite G was detected from the pyridyl label at a maximum of 6% TAR at DAT 4. In the harsh acidic extracts, additional metabolites could be released, accounting for a maximum of 6–9% TAR at DAT 28 (fluazifop acid, II), 2% TAR at any timepoint (Pyr-Ph ether, IV) or a maximum of 10% TAR at DAT 61 (CF3-pyridone, X). All other metabolites detected in the various extracts accounted for less than 5% TAR at all times, except for one polar fraction at 7% TAR. As time of incubation increased, radioactivity became more difficult to extract from soil. Unextracted radioactivity increased from 3% TAR at DAT 0 to 29–35% TAR at DAT 61-120.

A half-life for fluazifop acid (II), Pyr-Ph ether (IV) or CF3-pyridone (X) was not determined.

Table 71 Distribution of radioactivity (% TAR) in 0.67–0.68 mg ai/kg dry soil at 20 °C and pF2

	18 Acres, pyridinyl label							
Sampling (DAT)	0	1	4	7	28	61	90	120
fluazifop-butyl (I)								
- mild acid	90.8	4.1	1.5	0.7	0.4	0.2	0.2	0.1
- mild basic	-	-	-	-	ND	ND	ND	ND
- fulvic acid fraction	-	-	ND	-	ND	-	-	-
- harsh acid	-	ND	-	ND	ND	ND	ND	ND
fluazifop acid (II)								
- mild acid	6.0	67.3	37.2	26.3	4.1	2.5	1.7	2.0
- mild basic	-	-	-	-	1.7	0.9	1.3	0.9
- fulvic acid fraction	-	-	7.0	-	3.0	-	-	-
- harsh acid	-	1.7	-	1.2	6.4	2.3	6.5	2.7
Pyr-Ph ether (IV)								
- mild acid	ND	4.3	ND	3.4	ND	0.2	ND	0.5
- mild basic	-	-	-	-	0.9	0.2	0.6	1.0
- fulvic acid fraction	-	-	0.7	-	0.6	-	-	-
- harsh acid	-	0.3	-	1.0	1.0	1.7	1.7	1.7
CF3-pyridone (X)								
- mild acid	ND	2.9	15.5	22.7	24.9	18.3	14.1	17.2
- mild basic	-	-	-	-	6.8	3.7	7.6	6.0
- fulvic acid fraction	-	-	1.2	-	1.7	-	-	-
- harsh acid	-	0.5	-	1.2	7.7	10.0	2.4	6.2
unknown extracted								
- mild acid [a]	ND	1.9	4.8	0.4	0.8	0.7	0.4	1.7
- mild basic [b]	-	-	-	-	6.5	4.3	4.0	2.7
- fulvic acid fraction [c]	-	-	9.0	-	6.6	-	-	-
- harsh acid [d]	-	1.9	-	7.7	6.3	9.1	10.6	5.5
Extracts not analysed	0.1	5.0	13.7	4.1	10.5	0.6	1.3	1.4
- mild acid	0.1	3.9	0.1	0.2	0.2	0.1	0.1	0.5
- mild basic	-	1.0	3.3	3.7	0.2	0.2	0.5	0.4
- 1 M NaOH	-	-	10.4	-	9.6	-	-	-
- harsh acid	-	0.1	-	0.2	0.6	0.3	0.8	0.6
Subtotal								
- mild acid	96.8	80.5	59.0	53.5	30.3	21.8	16.4	21.4
- mild basic	-	-	-	-	15.8	9.0	13.5	10.7
- fulvic acid fraction	-	-	17.9	-	12.0	-	-	-
- harsh acid	-	4.4	-	11.1	21.4	23.1	21.2	16.1
- not analysed	0.1	5.0	13.7	4.1	10.5	0.6	1.3	1.4
Total extracted	96.9	89.9	90.6	68.7	90.0	54.5	52.4	49.6
Post extracted solids	3.3	7.0	4.7	22.7	5.0	29.5	28.8	28.9
NaOH trap (CO2)	-	0.1	3.2	4.9	9.4	13.0	15.8	16.7
Mass Balance	100.2	97.0	98.5	96.3	104.3	96.9	97.0	95.1

[a] unknowns extracted with neutral/mild acid, largest unknown 2.9% TAR (pyridyl); 3.3% TAR (phenyl)

[b] unknown extracted with ammonia/mild basic, largest unknown 2.5% TAR (pyridyl; unkn G); 5.8% TAR (phenyl; polar material)

[c] unknown extracted in fulvic acid fraction, largest unknown 6.4% TAR (pyridyl; unkn G); 7.7% TAR (phenyl, polar material)

[d] unknown extracted with harsh acid, largest unknown 4.6% TAR (pyridyl); 7.1% TAR (phenyl, polar material)

Table 72 Distribution of radioactivity (% TAR) in 0.67–0.68 mg ai/kg dry soil at 20 °C and pF2

	18 Acres, phenyl label							
Sampling (DAT)	0	1	4	7	28	61	90	120
fluazifop-butyl (I)								
- mild acid	90.1	3.4	1.2	0.5	0.4	0.2	0.2	ND
- mild basic	-	-	-	-	ND	ND	ND	ND
- fulvic acid fraction	-	-	ND	-	ND	-	-	-
- harsh acid	-	ND	-	ND	ND	ND	ND	ND
fluazifop acid (II)								
- mild acid	6.0	69.1	37.8	33.8	7.2	4.9	3.2	0.4
- mild basic	-	-	-	-	2.3	1.9	1.5	1.5
- fulvic acid fraction	-	-	7.4	-	6.9	-	-	-

	18 Acres, phenyl label							
Sampling (DAT)	0	1	4	7	28	61	90	120
- harsh acid	-	1.9	-	2.9	8.6	3.2	4.0	3.3
Pyr-Ph ether (IV)								
- mild acid	ND	4.2	0.6	3.4	0.3	ND	ND	1.0
- mild basic	-	-	-	-	1.3	0.9	1.4	0.9
- fulvic acid fraction	-	-	2.7	-	0.4	-	-	-
- harsh acid	-	0.4	-	2.0	1.4	2.1	1.6	1.8
unknown extracted								
- mild acid [a]	ND	1.3	5.4	2.2	1.3	ND	0.4	2.7
- mild basic [b]	-	-	-	-	9.4	3.2	7.0	4.0
- fulvic acid fraction [c]	-	-	10.5	-	8.2	-	-	-
- harsh acid [d]	-	3.7	-	6.9	8.2	12.1	12.6	7.1
Subtotal								
- mild acid	96.2	78.0	44.9	39.9	9.2	5.1	3.8	4.0
- mild basic	-	-	-	-	13.0	6.0	9.9	6.2
- fulvic acid fraction	-	-	20.5	-	15.4	-	-	-
- harsh acid	-	6.0	-	11.8	18.2	17.3	18.2	12.2
Extracts not analysed	0.1	3.8	17.6	3.9	14.5	0.2	-	3.3
- mild acid	0.1	3.2	0.3	0.2	0.1	-	-	2.8
- mild basic	-	0.4	3.2	3.7	0.3	0.1	-	0.2
- 1 M NaOH	-	-	14.2	-	13.5	-	-	-
- harsh acid	-	0.2	-	0.1	0.8	0.2	-	0.4
Total extracted	96.3	87.8	83.0	55.6	70.3	28.6	31.2	25.7
Post extracted solids	3.3	7.2	6.5	26.0	7.1	34.5	31.9	32.1
NaOH trap (CO2)	-	1.5	9.0	11.8	22.3	29.1	32.8	34.8
Mass Balance	99.5	96.5	98.5	93.3	99.7	92.2	95.8	92.5

[a] unknowns extracted with neutral/mild acid, largest unknown 2.9% TAR (pyridyl); 3.3% TAR (phenyl)

[b] unknown extracted with ammonia/mild basic, largest unknown 2.5% TAR (pyridyl; unkn G); 5.8% TAR (phenyl; polar material)

[c] unknown extracted in fulvic acid fraction, largest unknown 6.4% TAR (pyridyl; unkn G); 7.7% TAR (phenyl, polar material)

[d] unknown extracted with harsh acid, largest unknown 4.6% TAR (pyridyl); 7.1% TAR (phenyl, polar material)

Aerobic degradation in soil – enantiomer analysis

Soil study 5

Bewick, 1982 [PP9/0277, RJ0270B; Bewick, 1986, Pest.Sci] performed enantiomer analysis on selected extracts from thouree soils of [Harvey *et al.*, 1981, PP9/0273, report RJ0197B]. The isopropanol or isopropanol/water extracts from 18 Acres (at 0, 2, 7, 21 DAT), Gore Hill (at 0, 2, 7, 21, 84 DAT) and Frensham (at 0, 21, 84 DAT) were chosen for this enantiomer analysis, since they contained the bulk of the extractable residues. LSC and TLC analysis of the soil extracts showed that the concentration and composition of the radioactive residues had not changed markedly on storage, for any soil type during storage (-20 °C, 2 years). The enantiomer ratio of the fluazifop-butyl residue in the zero time extract had not changed from the expected 50:50 R:S ratio.

Selected soil extracts were fortified with non-labelled reference compounds. An aliquot (1 mL) of the Acres soil extract at zero time was fortified with 0.25 mg fluazifop-butyl (RS); all other extracts were fortified with 0.38 mg (RS) fluazifop acid (II). Fluazifop-butyl and fluazifop acid (II) were then isolated from the soil extracts. The fortified soil extracts were separated by TLC and the areas on the TLC plate corresponding to each compound were scraped and eluted with methanol. The methanol eluates were concentrated. The solutions containing fluazifop acid (II) were methylated by mixing with an ethereal diazomethane solution for 1 hour, and then evaporated. From these methylated fluazifop acid and fluazifop-butyl solutions, hexane solutions were prepared and concentrated, then analysed by LSC. To determine enantiomer ratios, the hexane solutions were separated by HPLC (chiral column at 10 ºC, consisting of Sperisorb S5 NH_2, modified with the N-3,5-dinitrobenzoyl derivative of D-phenyl glycine; non-chiral mobile phase consisting of 0.09% methanol in hexane). Fractions corresponding to the UV response (at 230 nm) of the R and S enantiomers of

either compound, were collected manually and analysed by LSC. Non-radiolabelled fortified compounds were analysed using peak area measurement on the HPLC-UV chouromatograms.

The ratio of R:S isomers in the radiolabeled residues decreased with the degradation of fluazifop acid (II) (Table 73). This decrease could not be demonstrated for the non-labeled residues.

The fortification levels of non-labeled fluazifop acid (II) were more than 100 times the radioactivity residue levels in the sample in order to get a clear UV response. Therefore, the enantiomer ratios measured from the peak areas on the HPLC-UV chouromatograms were unaffected by the presence of radioactive residue. The recoveries of the radioactive compounds thouroughout the isolation and derivations procedure ranged between 93% for fluazifop-butyl and 66–105% for fluazifop. Since the recoveries of the total fortified non-labelled residues thourough the entire method were shown to be non-stereoselective, the recoveries of the radioactive residues must also have been non-stereoselective. Therefore, although the radioactive residues are not quite quantitative, the recovered material is considered representative of the radioactive residue in the soil extract.

The distribution of R and S enantiomers of ^{14}C-fluazifop acid (II) in the soil extracts, expressed as percentage of the total recovered radioactivity (extracted + unextracted + evolved CO_2) was calculated (Table 73). These show that the R enantiomer predominates in the fluazifop acid (II) residues which result from the hydrolysis of the fluazifop-butyl racemate; the S enantiomer appears to degrade faster than the R enantiomer.

Table 73 Ratios and percentage of fluazifop acid (II) enantiomers in soil extracts.

Soil name	DAT	R / S ratio of radioactive residues*	R / S ratio of fortified residues**	% TAR R-fluazifop	% TAR S-fluazifop
18 Acres	2	84.8 / 18.2	52.0 / 48.0	63.6	14.2
	7	94.6 / 5.4	52.0 / 48.0	55.9	3.2
	21	95.3 / 4.7	51.5 / 48.5	22.2	1.1
Gore Hill	2	81.2 / 18.8	51.3 / 48.7	66.9	15.5
	7	94.0 / 6.0	52.0 / 48.0	56.5	3.6
	21	93.0 / 7.0	51.3 / 48.7	33.3	2.5
Frensham	21	63.5 / 36.5	51.3 / 48.7	30.9	17.8
	84	92.3 / 7.7	50.5 / 49.5	5.0	0.4

* determined by LSC of eluted column fractions

** determined by integrals of peak areas on HPLC-UV chouromatograms

Soil study 6

R/S conversion was studied in a sandy loam soil [Bewick, 1983, PP9/0276, report RJ0306B; Bewick, 1986, Pest. Sci]. Soil characteristics are summarized in Table 74. The soil was sieved (2 mm mesh), moisturized to 40% MWHC and dispensed into glass pots (3.8 cm diameter, 3 cm high, 30 g moist soil). The separate R and S enantiomers of uniformly ^{14}C-phenyl-labelled fluazifop-butyl (RS) were applied separately to the surface of separate soil samples at a dose corresponding to an application rate 1.0 kg ai/ha, for each enantiomer. Application was 114–115 ug/30 g moist soil = 3.8 mg/kg moist soil. The R:S enantiomer ratio was 97.5:2.5 and 2.6:97.4 for ^{14}C-fluazifop-butyl (R-enantiomer) and ^{14}C-fluazifop-butyl (S-enantiomer), respectively. The samples (40% MWHC) were incubated at 20± 1 °C under CO_2-free air, for 7 days. Evolved CO_2 was trapped in ethanolamine. Samples were taken at 0, 2, 6, 12, 24 hours and 2 and 7 days.

Table 74 Soil characteristics

Soil name	18 Acres
Location	Bracknell Berkshire, UK
Soil texture (USDA)	sandy clay loam
-- Sand	61%

-- Silt	10%
-- Clay	29%
Organic Carbon (%)	-
Organic Matter (%)	5.3%
CEC (meq/100 g)	19.4
pH	6.8
MWHC (%) (at pF 0)	87%
Moisture holding capacity at 0.33 bar (%) (at pF2.5)	21%
Moisture holding capacity at 10 bar (%)	-
% MWHC at incubation	40%
Bulk density (g/mL)	-
Microbial biomass (mg microbial carbon/kg soil)	-

At each sampling time, samples were extracted by Soxhlet with propan-2-ol, followed by a further reflux with propan-2-ol:water. The liquid traps, extracts and remaining solids were analysed by (combustion) LSC. The total recovery of applied radioactivity ranged between 94–102%. The evolved CO_2 was low; 4.5% TAR after 7 days. The first propanol extract contained the bulk of the residues: 89–99% TAR at 0–12 hours after treatment to 51–53% TAR at 7 days after treatment. The second propanol/water extract contained less than 5% TAR at all timepoints. Unextracted residues increased to 35–36% TAR after 7 days.

Only the propanol extracts of the soils were analysed by TLC, as the propanol/water extracts contained less than 5% TAR. Soil extracts were fortified with non-labelled reference compounds for fluazifop-butyl (RS) or fluazifop acid (II) and then separated by TLC. The areas on the TLC plate corresponding to each compound were scraped and eluted with methanol. The methanol eluates were concentrated, mixed with hexane, cleaned-up on an anhydrous sodium sulphate column and then analysed by LSC. To determine enantiomer ratios, the hexane solutions were separated by HPLC (chiral column at 10 ºC, consisting of Sperisorb S5 NH_2, modified with the N-3,5-dinitrobenzoyl derivative of D-phenyl glycine; non-chiral mobile phase consisting of 0.09% methanol in hexane). Fractions corresponding to the UV response (at 230 nm) of the R and S enantiomers of either compound, were collected manually and analysed by LSC. Non-radiolabelled fortified compounds were analysed using peak area measurement on the HPLC-UV chouromatograms.

The recoveries of the ^{14}C-fluazifop-butyl and ^{14}C-fluazifop acid (II) residues thouroughout the isolation procedure prior to enantiomer analysis ranged from 76–91% TAR and 95–114%, respectively. As a consequence, the procedure is not entirely quantitative but was demonstrated in previous work to be non-stereo-selective.

Table 75 shows that both the R and S enantiomers of fluazifop-butyl are hydrolysed in the soil over 7 days to yield fluazifop acid (II). The R:S ratio of both enantiomers of fluazifop-butyl was nearly constant during the hydrolysis. The degradation compound, fluazifop acid (II), reached a maximum at 6 hours after treatment (80% and 81% TAR for R and S enantiomer respectively) and decreased to 43–45% by 7 days. The R:S ratio of the R fluazifop acid (II) remained constant from 0 to 7 days. This is in contrast to the S fluazifop, which was steadily R enriched (ratio 5.3:94.7 at time zero changing to 97.8:2.2 after 7 days).

These results show that the R and S enantiomers of fluazifop-butyl are hydrolysed in the soil over 7 days to yield fluazifop acid (II) with the R configuration.

Table 75 Radioactive compounds (% TAR) and their enantiomer ratios during incubation of soil with ^{14}C-R enantiomer or ^{14}C-S enantiomer fluazifop-butyl

Time	^{14}C-R fluazifop-butyl				^{14}C-S fluazifop-butyl			
	Parent	R: S	Fluazifop	R: S	Parent	R : S	Fluazifop	R: S
0	95	98.3 : 1.7	2.3	ND	92	1.6 : 98.4	2.9	ND
2 h	40	97.7 : 2.3	53	94.6 : 5.4	40	2.7 : 97.3	53	5.3 : 94.7
6 h	12	98.2 : 1.8	80	97.4 : 2.6	14	3.0 : 97.0	81	11.3 :88.7
12 h	4.8	91.4 : 8.6	77	96.3 : 3.7	5.6	8.1 : 91.9	79	20.6 :79.4
24 h	2.8	ND	78	94.9 : 5.1	3.1	ND	74	36.9 :63.1

Time	^{14}C-R fluazifop-butyl				^{14}C-S fluazifop-butyl			
	Parent	R: S	Fluazifop	R: S	Parent	R : S	Fluazifop	R: S
2 d	1.9	ND	67	94.1 : 5.9	2.0	ND	66	63.6 :36.4
7 d	0.9	ND	43	95.6 : 4.4	1.0	ND	45	97.8 :2.2

Aerobic degradation in soil - laboratory studies with fluazifop acid (II)

Soil study 7

The rate of degradation of ^{14}C-pyridyl-fluazifop-P acid (II) (R-enantiomer) was determined in six soils [Goodyear, 1998, PP5/0808, report 38/200-D2142]. The characteristics of the soils are shown in Table 76. Soils were sieved (2 mm mesh). ^{14}C-pyridyl-fluazifop-P acid (II) was applied at a rate of 1 mg/kg dry soil, corresponding to 0.50 kg/ha. The treated soil was mixed thoroughly and was incubated at 20 °C ± 2 °C and moisture content pF2 in the dark under aerobic conditions. Microbial biomass was measured at the beginning and at the end of the study and did not decrease significantly during the tests, except for the Hall sample. Duplicate samples were analysed at 0, 1, 3, 7, 14 and 59 DAT. The incubation was terminated at 59 days, when less than 5% TAR remained in the soil extracts.

Table 76 Characteristics of soils and kinetic data for ^{14}C-pyridyl fluazifop-P acid (II) degradation (aerobic conditions)

Soil Identity	Malham SK104691	Wick SK342287	Wix PT102	Hall PT103	Enborne SK343286	Elmton SK961089
Location	Chelmorton, Derbyshire, UK, 1998	Barrow on Trent, Derbyshire, UK, 1998	UK, 1998	UK, 1998	Barrow on Trent, Derbyshire, UK, 1998	Empingham, Rutland, UK, 1998
Textural class (USDA)	Silt loam	Sandy clay loam	Sandy loam	sandy loam	Sandy clay loam	Clay loam / loam
--sand %	22	58	56	76	49	40
--Silt %	59	21	34	12	26	33
--Clay %	19	21	10	12	25	27
Organic C / OM % [a]	1.9 / 3.3	2.1 / 3.6	2.2 / 3.8	0.9 / 1.6	3.1 / 5.3	4.3 / 7.4
pH H_2O/KCl	7.0 / 6.2	5.8 / 4.9	7.2 / 6.6	5.3 / 4.3	7.1 / 6.3	7.7 / 7.1
CEC (meq/100g)	20.8	18.2	19.0	9.9	30.6	42.9
WHC at pF0 (MWHC) %	76.2	69.7	64.2	41.5	84.8	88.6
WHC at pF2.0 (0.1 Bar) %	35.2	29.7	31.5	15.4	41.8	41.3
WHC at pF2.5 (0.33 Bar) %	28.3	22.8	21.5	10.1	33.3	34.9
Microbial biomass (mg C/kg soil) at zero time end of incubation	252.7 112.8	459.2 221.1	250.0 284.7	286.0 82.7	935.0 875.6	1054.1 1077.1

[a] OM = Organic C x 1.724

WHC = water holding capacity

Soil samples were extracted with acetonitrile:water (1:1). Soil extracts were analysed by LSC. Volatiles were not trapped during the trial and unextracted residues were not measured. Therefore, mass balance was not assessed. Extracted radioactivity in acetonitrile:water decreased from 88%–94% TAR immediately after application to 20–41% TAR after 59 days of incubation.

The acetonitrile/water extract was rotary evaporated to low volume, reconstituted in 0.01 M HCl and concentrated on an SPE cartridge. Analysis of extracted radioactivity by HPLC showed that the amount of fluazifop acid (II) decreased steadily in every soil, from 80–93% TAR at day 0 to 0.4–4.7% TAR after 59 days of incubation. The identity of fluazifop acid (II) was confirmed by TLC analysis. Other degradation products were not analysed.

Results are shown in Table 77. DT_{50} and DT_{90} values were calculated by graphical representation and using single first order best fit in Lotus Excel 97 software. DT_{50} values for

fluazifop acid (II) ranged from 2.3–8.3 days; DT_{90} values for fluazifop acid (II) ranged from 7.7–27.5 days.

Table 77 Fluazifop acid (II) extracted from soils treated with 1 mg/kg fluazifop acid (II) and kinetic endpoints

DAT	Malham SK104691	Wick SK342287	Wix PT102	Hall PT103	Enborne SK343286	Elmton SK961089
0	88.5	84.9	86.8	93.3	83.3	79.6
1	74.7	74.4	68.1	81.8	58.3	53.4
3	59.5	59.7	29.4	64.5	32.2	22.8
7	41.4	44.8	24.0	51.4	24.3	21.2
14	34.0	25.1	6.5	29.7	14.5	8.9
30	4.8	10.9	1.9	15.0	2.5	4.8
59	2.1	1.2	0.4	4.7	1.2	0.7
DT_{50} (SFO)	8.3	8.2	2.7	9.1	3.3	2.3
DT_{90} (SFO)	27.5	27.3	9.0	30.3	11.1	7.7
R-squared	0.966	0.987	0.962	0.979	0.931	0.923

Aerobic degradation in soil - laboratory studies with CF3-pyridone (X)

Soil study 8

The rate of degradation of non-radiolabelled CF3-pyridone (X) was determined in four soils [Emburey, 2002, R154719/0002, report RJ3259B]. Soil characteristics are shown in Table 78. Soils were sieved (2 mm mesh). CF3-pyridone (X) was applied at a rate of 0.2 mg/kg dry soil and the soil was thoroughly mixed. This rate was based on a fluazifop-P-butyl field application rate of 0.375 kg ai/ha with 5 cm incorporation into soil and a soil bulk density of 1.5 g/cm^3. The rate was also adjusted according to the molecular weight of the molecule. The soils were were incubated in the dark, under moist (pF2) aerobic conditions, at a temperature of 20 ± 2 ºC. Microbial biomass was measured at the beginning and at the end of the study. Duplicate samples were removed after 0, 3, 7, 14, 30, 59, 86, 93 and 115 days of incubation. CF3-pyridone (X) was quantified by GC-MS method RAM 354/02 and expressed as mg/kg dry soil. Residues were corrected for concurrent recoveries. The average concurrent recoveries ranged from 71–76% at 0.05–0.20 mg/kg. Control samples were < 0.01 mg/kg.

Table 78 Characteristics of soils and kinetic data for CF3-pyridone (X) degradation (aerobic conditions)

Soil name	18 Acres	Frensham	Kenny Hill	Wisborough Green
Location	Bracknell, Berkshire, UK	Churt, Surrey, UK	Mildenhall, Suffolk, UK	Billingshurst, West Sussex, UK,
Soil texture (USDA)	Sandy clay loam	Sandy loam	Loamy sand	Silty clay loam
Sand %	50	78	84	12
Silt %	22	8	4	52
Clay %	28	14	12	36
Organic Matter %	5.9	2.6	7.4	5.3
pH (H2O)	6.0	6.1	8.0	5.3
Cation exchange capacity (meq/100g)	17.0	7.9	13.9	14.2
WHC at pF2 (% moisture content)	35.5	21.5	19.7	41.3
WHC at pF2.5 (0.33 Bar) %	27.0	13.4	14.0	37.4
WHC (15 Bar) %	13.1	4.7	7.7	25.3
% organic carbon as active biomass at 9 days of incubation at 123-130 days of incubation	 1.91 0.92	 3.33 0.49	 1.14 0.46	 1.04 0.46

Results are shown in Table 79. DT_{50} values were calculated using ModelManager version 1.1. The Acres soil was the only soil in which CF3-pyridone (X) decreased to less than 10% TAR within the timeframe of the study and for which a DT_{90} could be calculated. In the other soils, the final

percentage of residues, after 115 days, was 14% TAR (Wisborough), 26% (Frensham) and 38% (Kenny Hill), which indicates a moderate degradation. This is not the consequence of an abnormal decrease of the biomass.

Table 79 CF3-pyridone (X, mg/kg dw) in soils treated with 0.2 mg/kg CF3-pyridone (X) and its kinetic endpoints

DAT	18 Acres	Frensham	Kenny Hill	Wisborough Green
0	0.20	0.19	0.21	0.21
3	0.16	0.18	0.19	0.16
7	0.10	0.15	0.16	0.12
14	0.08	0.11	0.15	0.10
30	0.05	0.08	0.13	0.06
59	0.03	0.07	0.10	0.04
86	-	0.06	0.10	-
93	0.02	-	-	0.03
115	0.01	0.05	0.08	0.03
DT_{50} (SFO)	13	43	82	22
DT_{50} (FOMC)	9.0	22	60	12
DT_{90} (FOMC)	85	-	-	-
R-squared	-	-	-	-

Aerobic degradation in soil - laboratory studies with Pyr-Ph ether (IV)

Soil study 9

The rate of degradation of ^{14}C-phenyl-Pyr-Ph ether (IV) was determined in thouree soils [Oddy and Doble, 2011, CGA181847_50001, report NC/09/015]. Soil characteristics are shown in Table 80. Soils were sieved (2 mm mesh). All soils were stored in accordance with ISO/DISS 10381-9 to maintain viability and were allowed to acclimatize to the study conditions for 25 days prior to application. ^{14}C-Phenyl labelled Pyr-Ph ether (IV) was applied at a rate of 0.267 mg/kg dry soil and the soil was thoroughly mixed. Actual rates were 0.272, 0.271, 0.272 mg/kg dry soil for Marsillargues, Gartenacker and 18 Acres soils (102%, 101%, 102% of target). This rate was equivalent to 0.2 kg/ha with 5 cm incorporation into soil and a soil bulk density of 1.5 g/cm^3. The soils were incubated in the dark, under moist (pF2) aerobic conditions, at a temperature of 20 ± 2 ºC for up to 14 days. Any volatile radioactivity was continuously flushed from the vessels and collected in liquid traps containing ethylene glycol and 2 M KOH. Duplicate samples were removed after 0, 3, 6 hours, 1, 2, 4, 7, 14 days of incubation. Microbial biomass was measured at the beginning and at the end of the study and confirmed that all of the soils were microbiologically active thouroughout the course of the study.

Table 80 Characteristics of soils for Pyr-Ph (IV) degradation (aerobic conditions)

Soil name	Marsillargues	Gartenacker	18 Acres
Location	La Paulette Marsillargues; France	Les Barges, Vouvry, Switzerland	Warfield, Bracknell, UK
Soil texture (USDA)	Silty Clay	Loam	Sandy clay
Sand % (50-2000 um)	11	42	53
Silt % (2-50 um)	42	50	24
Clay % (< 2 um)	47	8	23
Organic carbon %	1.0	1.8	2.8
Organic Matter %	1.7	3.1	4.8
pH (water, 1:1 soil/water) pH (KCl, 1:1) pH (CaCl2, 1:2)	7.9 7.2 7.7	7.3 7.0 7.1	6.1 5.4 5.7
Cation exchange capacity (meq/100g)	18.4	8.3	15.3
WHC at pF2 (0.1 bar, g/100 g dry soil)	22.7	39.0	29.8
WHC at pF2.5 (0.33 Bar, g/100 g dry soil)	25.8	23.5	21.2
WHC (15 Bar) %	-	-	-
active biomass in mg C/kg soil (% org C) 1 Dec 2009: initial	159.6 (1.6%)	229.5 (1.3%)	504.0 (1.8%)

23 Febr 2010: final	220.2 (2.3%)	472.1 (2.6%	601.3 (2.2)
Deg T50 (SFO)	7.16	2.31	4.62
Deg T90 (SFO)	23.80	7.68	15.35
R-squared	0.961	0.977	0.980
Chi-square (err%)	20.7	23.4	14.0
Deg T50 (FOMC)	**4.94**	**0.97**	**4.10**
Deg T90 (FOMC)	**72.5**	**46.2**	**22.9**
R-squared	**0.986**	**0.995**	**0.984**
Chi-square (err%)	**9.1**	**6.4**	**11.9**
Deg T50 (DFOP)	5.39	2.00	4.46
Deg T90 (DFOP)	107.1	73.6	17.2
R-squared	0.979	0.989	0.981
Chi-square (err%)	12.5	10.8	14.4

Soil samples were extracted on the day that they were collected. Samples were extracted thouree times with acetonitrile/water (80:20, v/v) for 20 min. The remaining solids were extracted with acetonitrile/water/concentrated HCl (80:20:2 v/v/v) at reflux for 6 hours. The liquid traps, extracts and remaining solids were analysed by (combustion) LSC. The ambient and soxhlet extracts were combined and concentrated to near dryness, redissolved in acetonitrile/water (1:1, v/v) and analysed by HPLC-UV (224 nm) and TLC against a reference standard for Pyr-Ph ether (IV).

The total recovery of applied radioactivity ranged between 82–99% (Marsillagues), 91–106% (Gartenacker) and 92–100% (18 Acres). Extracted radioactivity decreased from 95–97% TAR immediately after application to 11–15% TAR after 14 days of incubation. Over this period there was a corresponding increase in the levels of unextractable and volatile radioactivity. Levels of CO_2 reached maximum values between 9.5–17.0% TAR by the end of the incubation period (14 days).

The amount of ^{14}C-Pyr-Ph ether (IV) decreased steadily in every soil, from 95–98% TAR at day 0 to 0.7–7.8% TAR after 14 days of incubation. The identity of Pyr-Ph ether (IV) was confirmed by HPLC- and TLC analysis. Other degradation products were not analysed.

Results are shown in Table 81. Deg T_{50} and Deg T_{90} values were calculated according to FOCUS 2006 recommendations using MATLAB 7.0.4.365 software. Single first order (SFO), first order multi compartment (FOMC) and bi-exponential (double first order in parallel, DFOP) models were evaluated. The FOMC model was found to give the best fit to the experimental data. Using the FOMC model, DT_{50} values for Pyr-Ph ether (IV) ranged between 0.97–4.7 days and DT_{90} values ranged between 23–72 days.

Table 81 Characterisation of residues in soils treated with 0.267 mg/kg Pyr-Ph ether (IV)

Marsillargues								
Duration (hours; days)	0	3 hours	7 hours	1 day	2 days	4 days	7 days	14 days
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
Pyr-Ph ether (IV)	96.41	55.21	51.84	17.68	10.13	9.04	7.52	4.44
unknown extracted	ND	3.27	1.89	7.89	9.00	7.77	6.85	6.20
post extracted solids	2.31	34.05	37.87	60.57	72.89	69.01	68.30	70.74
$^{14}CO2$	ND	0.05	0.11	1.91	4.58	2.93	10.45	14.60
Total	98.72	92.58	91.71	88.05	96.60	88.75	93.12	95.98
Gartenacker								
Duration (days)	0	3 hours	7 hours	1 day	2 days	4 days	7 days	14 days
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
Pyr-Ph ether (IV)	95.33	32.58	24.83	9.70	9.06	8.63	2.41	7.83
unknown extracted	ND	7.19	8.70	9.68	10.88	9.74	13.04	7.18
post extracted solids	3.87	54.32	61.77	74.09	77.61	81.50	77.74	75.45
$^{14}CO2$	ND	0.07	0.28	2.45	3.65	5.35	7.55	9.52
Total	99.20	94.16	95.58	95.92	101.20	105.22	100.74	99.98
18 Acres								
Duration (days)	0	3 hours	7 hours	1 day	2 days	4 days	7 days	14 days
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
Pyr-Ph ether (IV)	97.53	51.61	45.49	5.47	2.81	2.13	2.50	0.71
unknown extracted	ND	14.34	14.25	20.52	17.66	20.13	8.38	14.21

Marsillargues								
Duration (hours; days)	0	3 hours	7 hours	1 day	2 days	4 days	7 days	14 days
post extracted solids	1.42	28.80	35.47	65.71	70.65	62.81	74.25	64.92
$^{14}CO2$	ND	0.38	0.83	1.84	7.19	10.78	13.85	17.03
Total	98.95	95.13	96.04	93.54	98.31	95.85	98.98	96.87

Kinetic endpoints for aerobic soil degradation studies

Soil study 10

[Leahey, 1990, no code available, M5148B] derived kinetic endpoints for fluazifop-butyl, fluazifop acid (II), Pyr-Ph ether (IV) and CF3-pyridone (X) based on the laboratory trials described in [Harvey *et al*., 1981, PP5/0273, report RJ0197B]. In this aerobic soil degradation study with fluazifop-butyl degradation data at 20 ºC and 40% MWHC were available for eight trials (two soils, each with pyridyl and phenyl label; four soils with phenyl label only) and degradation data at 20 ºC and 15% MWHC (1 soil with phenyl label). Two trials were not included in the kinetic evaluation:

- The two trials with the soils Speyer 2.2 (loamy sand, phenyl label) and Speyer 2.3 (sandy loam, phenyl label) were stored for over a year before the beginning of the laboratory study and were excluded because they had too low microbial activity.

Half-lives (Table 82) were calculated manually by graphical representation of the results.

Table 82 Half-lives calculated for study [Harvey *et al*., 1981, PP5/0273, report RJ0197B]

	18 Acres	Gore Hill	Frensham	Rosedean
	sandy clay loam	calcareous clay loam	loamy sand	peat
	40% MWHC phenyl 40% MWHC pyridyl 15% MWHC phenyl	40% MWHC phenyl 40% MWHC pyridyl	40% MWHC phenyl	40% MWHC phenyl
fluazifop-P-butyl (I)	< 1 day	< 1 day	< 1 day	< 1 day
fluazifop acid (II);	14 days	19 days	19 days	12 weeks
Pyr-Ph ether (IV)	10-20 wks	10-20 wks	10-20 wks	10-20 wks
CF3-pyridone (X)	12 weeks	12 wks	NA	NA

Soil study 11

[Jones, 2003, PP5/1219, report RAJ0161B] derived kinetic endpoints for fluazifop acid (II) from the laboratory trials described in two study reports. In the aerobic soil degradation study with fluazifop-butyl [Harvey *et al*., 1981, PP5/0273, report RJ0197B] degradation data at 20 ºC and 40% MWHC were available for eight trials (two soils, each with pyridyl and phenyl label; four soils with phenyl label only). Thouree trials were not included in the kinetic evaluation for fluazifop acid (II):

- The trial with the soil Rosedean (fen peat soil, phenyl label) was excluded due to the very high organic content of 67%.
- The two trials with the soils Speyer 2.2 (loamy sand, phenyl label) and Speyer 2.3 (sandy loam, phenyl label) were stored for over a year before the beginning of the laboratory study and were excluded because they had too low microbial activity.
- The study had a limited number of sampling intervals directly after application for soils treated with the pyridyl label. Due to the rapid conversion of fluazifop-butyl to fluazifop acid (II) (DT_{90} < 2 days) and continued rapid degradation of fluazifop acid (II), the peak concentration of fluazifop acid (II) production and its decline are missed by these chosen sampling intervals. Therefore, in order to model the disappearance of fluazifop acid (II), it is assumed that 100% conversion of fluazifop-butyl to fluazifop acid (II) has occurred after 2 days. Therefore this additional datapoint has been entered in the DT_{50} calculation and the decline of fluazifop acid (II) started from this assumed peak concentration at 2 days after treatment.

In the aerobic soil degradation study with fluazifop-P acid (II) [Goodyear, 1998, PP5/0808, report 38/200-D2142] degradation data at 20 ºC and moisture content pF2 were available for 6 trials (6 soils, pyridyl label only).

In summary, degradation data were available for fluazifop-P acid (II) (11 trials) in soil under aerobic conditions (see Table 83). In the original reports, these data were modelled using either simple graphical assessment or first-order methods. These data were remodelled using ModelManager (version 1.1). For each dataset a statistical assessment was performed to establish the most appropriate model to fit to the data: simple first order (SFO) or first-order multi-compartment model (FOMC). FOMC describes the decline in residues using an ensemble of first-order compartments. Remodelled half life values for fluazifop acid (II) ranged from 2.3–38.4 days (see Table 83). The median laboratory half-life determined for fluazifop acid (II) was 8.3 days (n=11).

Table 83 Trigger Endpoints for fluazifop acid (II) for aerobic laboratory soil degradation studies

Soil name	Soil type	Kinetic model (best fit)	DT_{50} (days)	DT_{90} (days)	Code no Report no
Gore Hill (phenyl)	calcareous clay loam	SFO	17.5	60.0	PP5/0273, RJ0197B
Gore Hill (pyridyl)	idem	SFO	14.8	49.2	idem
18 Acres (phenyl)	sandy clay loam	SFO	10.4	34.6	PP5/0273, RJ0197B
18 Acres (pyridyl)	idem	SFO	18.3	60.8	idem
Frensham (phenyl)	loamy sand	SFO	38.4	127.5	PP5/0273, RJ0197B
Malham (pyridyl)	silt loam	FOMC	8.3	27.5	PP5/0808, 38/200-D2142
Wick (pyridyl)	sandy clay loam	FOMC	7.4	33.7	PP5/0808, 38/200-D2142
Wix (pyridyl)	sandy loam	FOMC	2.3	12.8	PP5/0808, 38/200-D2142
Hall (pyridyl)	sandy loam	FOMC	7.7	43.5	PP5/0808, 38/200-D2142
Enborne (pyridyl)	sandy clay loam	FOMC	2.2	21.0	PP5/0808, 38/200-D2142
Elmton (pyridyl)	sandy clay loam	FOMC	1.7	16.1	PP5/0808, 38/200-D2142
Overall median (n = 11)			8.3	34.6	

Soil study 12

[Wang, 2009, PP5/10008, report R-09049-2; Wang, 2009, PP5/10009, report R-09049-1] derived kinetic endpoints for fluazifop-P-butyl, fluazifop acid (II) and CF3-pyridone (X) from thouree study reports [Harvey *et al*., 1981, PP5/0273, report RJ0197B; Goodyear, 1998, PP5/0808, report 38/200-D2142; Emburey, 2002, R154719/0002, report RJ3259B]. Due to the rapid degradation of the parent and relatively slower degradation of the metabolites, the degradation kinetics of fluazifop acid (II) and CF3-pyridone (X) were estimated with the metabolites as parent compounds in the kinetic models.

In the aerobic soil degradation study [Harvey *et al*., 1981, PP5/0273, report RJ0197B] degradation data at 20 ºC and 40% MWHC were available for eight trials (two soils, each with pyridyl and phenyl label; four soils with phenyl label only). Since CF3-pyridone (X) was measured in only two soils at only 3 timepoints, it was not possible to estimate reliable kinetic parameters for CF3-pyridone (X) from this study. Thouree trials were not included in the kinetic evaluation for fluazifop-P-butyl and fluazifop-P:

- The trial with the soil Rosedean (fen peat soil, phenyl label) was excluded due to the very high organic content of 67%.

- The two trials with the soils Speyer 2.2 (loamy sand, phenyl label) and Speyer 2.3 (sandy loam, phenyl label) were stored for over a year before the beginning of the laboratory study and were excluded because they had too low microbial activity.
- For the parent substance, one trial was excluded (Gore Hill, pyridyl label) from the analysis because only two data points were available for a kinetic analysis.

In the aerobic soil degradation study with fluazifop-P acid (II) [Goodyear, 1998, PP5/0808, report 38/200-D2142] degradation data at 20 ºC and moisture content pF2 were available for six trials (six soils, pyridyl label only). In the aerobic soil degradation study with CF3-pyridone (X) [Emburey, 2002, R154719/0002, report RJ3259B] degradation data at 20 ºC and moisture content pF2 were available for four trials (4 soils, no radiolabel).

In summary, degradation data were available for fluazifop-butyl (4 trials), fluazifop acid (II) (11 trials) and CF3-pyridone (X) (four trials) in soil under aerobic conditions (see Tables 84–86 . Kinetic models for the derivation of trigger endpoints were applied in a stepwise approach proposed by FOCUS using software ModelMaker 3.1 [FOCUS, 2006]. All trials were conducted at FOCUS reference temperatures (20 ºC). Only the laboratory degradation study 1 [Harvey *et al*., 1981, PP5/0273, report RJ0197B] was not conducted under FOCUS reference moisture conditions (pF2) and therefore moisture correction factors were calculated. Since all moisture correction factors were > 1, trials were performed at optimum moisture conditions and normalisation to FOCUS reference conditions was not necessary. Best model fits were evaluated with chi-square (χ^2) error tests and a visual assessment. The estimation of model parameters was evaluated by a t-test. Data sets were first run with SFO (Single First Order) and FOMC (First Order Multi Compartment) models. If the FOMC model showed a better fit, a bi-phasic (DFOP) model was run. Only $DegT_{50}$ values obtained from reliable fits were used for the derivation of trigger endpoints.

For the parent fluazifop-butyl, an analysis of four laboratory degradation trials resulted in a worst-case $DegT_{50}$ of 2.9 days and a corresponding $DegT_{90}$ of 9.6 days as only thouree values were available. The geometric mean $DegT_{50}$ and $DegT_{90}$ values (n = 3) are calculated to be 1.0 and 3.4 days.

For the metabolite fluazifop acid (II), an analysis of 11 laboratory trials, covering nine different soils, resulted in a geometric mean $DegT_{50}$ = 6.5 days and $DegT_{90}$ = 32 days (n = 9).

For CF3-pyridone (X), data from four degradation trials revealed a geometric mean of $DegT_{50}$ = 12 days and $DegT_{90}$ = 134 days (n = 4).

Table 84 Kinetic endpoints for fluazifop-butyl

Trial	Soil name	Soil type	Kinetic model (best fit)	χ^2 error (%)	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Code no Report no
1	Gore Hill (phenyl)	calcareous clay loam	SFO	1.3	0.3	1.1	[PP5/0273, RJ0197B]
2	Acres (phenyl)	sandy clay loam	SFO	14.2	0.4	1.3	[PP5/0273, RJ0197B]
3	Acres (pyridyl)	idem	SFO	0.2	3.3	11.1	idem
	Acres (geometric mean)				1.1	3.8	
4	Frensham (phenyl)	loamy sand	SFO	0.1	2.9	9.6	[PP5/0273, RJ0197B]
	Overall geometric mean [a] (n = 3)				1.0	3.4	
	Worst-case (n = 3 for parent)				2.9	9.6	

[a] Where multiple DegT50 values for individual soils were available these were averaged (geometric mean, values in bold) before taking the overall geometric mean.

Table 85 Kinetic endpoints for fluazifop acid (II)

Trial	Soil name	Soil type	Kinetic model (best fit)	χ^2 error (%)	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Code no Report no
6	Gore Hill (phenyl)	calcareous	SFO	2.3	17.5	58.1	[PP5/0273,

Trial	Soil name	Soil type	Kinetic model (best fit)	χ^2 error (%)	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Code no Report no
		clay loam					RJ0197B]
7	Gore Hill (pyridyl)	idem	FOMC	0.8	5.1	35.8	idem
	Gore Hill (average)				9.4	45.6	
8	Acres (phenyl)	sandy clay loam	SFO	6.5	10.4	34.7	[PP5/0273, RJ0197B]
9	Acres (pyridyl)	idem	SFO	5.1	17.8	59.0	idem
	Acres (geometric mean)				13.6	45.2	
10	Frensham (phenyl)	loamy sand	SFO	2.2	38.6	128.1	[PP5/0273, RJ0197B]
11	Malham (pyridyl)	silt loam	SFO	8.7	8.3	27.6	[PP5/0808, 38/200-D2142]
12	Wick (pyridyl)	sandy clay loam	DFOP	2.3	7.3	32.0	[PP5/0808, 38/200-D2142]
13	Wix (pyridyl)	sandy loam	SFO	14.6	2.7	9.1	[PP5/0808, 38/200-D2142]
14	Hall (pyridyl)	sandy loam	FOMC	3.4	7.7	43.7	[PP5/0808, 38/200-D2142]
15	Enborne (pyridyl)	sandy clay loam	DFOP	4.8	2.1	21	[PP5/0808, 38/200-D2142]
16	Elmton (pyridyl)	sandy clay loam	DFOP	9.4	1.6	19.7	[PP5/0808, 38/200-D2142]
	Overall geometric mean [a] (n = 9)				6.5	32.3	

[a] Where multiple DegT50 values for individual soils were available these were averaged (geometric mean) before taking the overall geometric mean.

Table 86 Kinetic endpoints for CF3-Pyridone (X)

Trial	Soil name	Soil type	Kinetic model (best fit)	χ^2 error (%)	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Code no Report no
17	Acres	sandy clay loam	DFOP	7.5	5.1	38.6	[R154719/0002; RJ3259B]
18	Frensham	sandy loam	DFOP	2.5	11.6	158.8	[R154719/0002; RJ3259B]
19	Kenny Hill	loamy sand	DFOP	2.5	29.1	208.1	[R154719/0002; RJ3259B]
20	Wisborough	silty clay loam	FOMC	3.9	11.9	255.2	[R154719/0002; RJ3259B]
	Overall geometric mean [a] (n = 4)				12.0	134.3	

[a] Where multiple DegT50 values for individual soils were available these were averaged (geometric mean) before taking the overall geometric mean.

Soil study 13

[Greener, 2009, PP5/10019, report RAJ0708B] derived kinetic endpoints for Pyr-Ph ether (IV) from one study report [Harvey *et al*., 1981, PP5/0273, report RJ0197B]. The calculations of $DegT_{50}$, $DegT_{90}$ and formation fraction values followed [FOCUS, 2006] for modelling endpoints using [KINGUI, 2006] software. The degradation pathway considered for the calculations was: fluazifop-P-butyl (I) to fluazifop acid (II) to Pyr-Ph ether (IV). Fluazifop-P-butyl and fluazifop acid (II) were fitted first, using parameters determined in study 1 for SFO [Wang, 2009, PP5/10009, report R-09049-1]. Since there were only 3–5 datapoints for Pyr-Ph ether (IV), only the SFO kinetic endpoints were used, because the other models (FOMC, DFOP) had too many unknown parameters and χ^2-errors could not be calculated for these models.

Table 87 shows that for Pyr-Ph ether (IV), data from 6 degradation trials (3 soils) revealed an arithmethic mean formation fraction of 0.1 and a geometric mean of $DegT_{50}$ = 31 days.

Table 87 Modelling Endpoints for Pyr-Ph ether (IV)

Soil	Soil type	temp (°C); %MWHC	Kinetic model (best fit)	χ^2 % error	Formation fraction	DT_{50} (days)	DT_{90} (days)	Code no Report no
18 Acres (phenyl)	calcareous clay loam	20 °C; 40%	SFO	27	0.07	39.6	132	[PP5/0273, RJ0197B]
18 Acres (pyridyl)	idem	20 °C; 40%	SFO	25	0.06	82.9	275	idem
18 Acres (phenyl)	idem	10 °C; 40%	SFO	75	0.18	28.2 [b]	45.3 [b]	idem
18 Acres average [a]					0.09	45.3		
Gore Hill (phenyl)	sandy clay loam	20 °C; 40%	SFO	49	0.08	26.5	87.9	[PP5/0273, RJ0197B]
Gore Hil (pyridyl)	idem	20 °C; 40%	SFO	49	0.03	105	348	idem
Gore Hill average [a]					0.05	52.2		
Frensham (phenyl)	loamy sand	20 °C; 40%	SFO	39	0.15	12.6	41.8	[PP5/0273, RJ0197B]
Arithmetic mean					0.10 (n=3)			
Geometric mean						31 (n=3)		

[a] Arithmetic mean for formation fractions; geometric mean for DT_{50}

[b] normalised to 20 °C using Q10 = 2.2

Comparison of UK soils with USA soils

Soil study 14

Microflora and physico-chemical properties of the UK soils were compared to freshly sampled soils from USA field sites and with data from the literature on USA soils [Askew and Hill, 1985, 444863, report RJ0429B; Leahey, 1990, code not available, report M5148B]. The UK soils were Frensham (loamy sand, Surrey, UK) and 18 Acres (sandy loam, Berkshire UK) as used in the aerobic soil degradation studies. The eight USA soils came from Champaign (Illinois), White Heath (Illinois), Yanceyville (North Carolina), Proctor (Arkansas), Tallulah (Louisiana), Thomastown (Louisiana), Lebeau (Louisiana), Dothan (Alabama). The physico-chemical properties of the UK soils fall within the range determined for all USA soils. The total microbial counts in all UK and USA soils were 10^9 cells/g dry weight soil. In soils from both countries, counts from bacteria were about 200 times higher than the fungi and the actinomycetes were intermediate to those of bacteria and fungi.

Soil study 15

[Bang, 2013, PP5_50403, report TK0058358] concluded that the SK104691, SK342287, PT102, PT103, SK343286, Old Paddock, Frensham, East Jubilee and Lilly Field UK study soils used in laboratory environmental fate and behaviour studies of fluazifop-butyl can be found in label use area soils for fluazifop-P-butyl in the USA based on soil classification and physico-chemical properties of the soils.

Soil study 16

[Ghebremichael and Bang, 2014, R156172_50003, report TK0256375] concluded that the '18 Acres', 'Gore Hill', and 'Rosedean' UK study soils used in laboratory environmental fate and behavior studies of fluazifop-butyl can be found in label use area soils for fluazifop-P-butyl in the USA based on soil classification and physico-chemical properties of the soils.

Field dissipation studies

Several field dissipation studies were submitted where only the residues of fluazifop-butyl and/or fluazifop acid (II) were investigated [Ussary, 1981, PP9/0284, TMU0657/B; Ussary, 1981, 405714, TMU0676B; Harradine, 1984, PP5/0815, report M3858B; Pay and Harradine, 1986, PP5/0816, report

RJ0539B, Wiebe, 1989, PP5/0811, report 89-066B; Wiebe, 1989, PP5/0812, report RR 89-067B; Bolygo, 1992, PP5/0817, RJ1323B; Bolygo, 1993, PP5/0818, report RJ1386B; Bolygo, 1993, PP5/0819, report RJ1512B]. These studies were not summarized because no information on the persistent soil metabolite CF3-pyridone (X) was provided. [Jones, 2003, PP5/1291, report RAJ0161B] derived kinetic trigger endpoints for fluazifop acid (II) from field dissipation studies performed in Germany in 1988 (4 locations) and the USA in 1989 (2 locations). This study was not summarized because no kinetic trigger endpoints were derived for the persistent soil metabolite CF3-pyridone (X).

Soil study 17

Four trials were carried out in Germany during 1988 and 1989 to study behaviour of fluazifop-P-butyl (R-enantiomer), following either autum or spring applications [Jones and Atreya, 1991, PP5/0814, RJ0952B]. Fluazifop-P-butyl (R-enantiomer) was sprayed at an actual rate of 0.50–0.52 kg ai/ha, as an EC formulation, on bare soil. Soil characteristics for the four locations are given in Table 88. Twenty soil cores (0–30 cm, only 0–10 cm on the day of application) in each plot were removed just before spraying, on the day of treatment and at different times up to 18 months, depending on the site. The cores were divided in 2 segments (0–5 and 5–10 cm) at day 0 and 4 segments (0–5, 5–10, 10–20 and 20–30 cm) for subsequent times. Composite samples were prepared for each soil layer. Cores were stored at -18 °C or lower for a maximum period of 700 days (spring application) or 930 days (autumn application) based on application date and report date.

Table 88 Soil characteristics for a field dissipation in Germany

Site	Depth cm	OM%	Soil Type	pH	CEC meq/100 g	MHC% 0.33bar	MHC% 15 bar
Krukow	0-5	3.1	sandy loam	6.8	8.5	15.32	8.27
	5-10	4.1	sandy loam	6.7	8.0	15.39	9.13
	10-20	3.8	sandy loam	6.7	8.2	15.99	9.45
	20-30	2.6	sandy loam	6.7	7.3	13.72	7.64
Varendorf	0-5	2.1	Loam	6.7	8.9	16.49	8.14
	5-10	2.9	Loam	6.7	9.1	18.03	6.58
	10-20	2.7	Loam	7.3	8.9	17.39	10.9
	20-30	1.5	Sandy Loam	7.2	6.9	15.96	5.56
Pallhausen	0-5	2.5	clay loam	7.8	14.4	23.72	9.61
	5-10	2.8	clay loam	7.9	14.4	24.01	10.11
	10-20	2.4	Loam	7.8	14.7	23.43	9.82
	20-30	0.8	clay loam	7.7	14.1	21.20	8.34
Mechtersheim	0-5	1.2	Silty clay loam	7.3	13.8	21.89	8.29
	5-10	1.3	Silty clay loam	7.4	13.4	21.86	8.81
	10-20	1.3	clay loam	7.5	13.0	21.13	9.19
	20-30	1.2	Silty clay loam	7.6	14.1	21.98	9.19

Soil samples were analysed for free fluazifop-butyl using GC-NPD method RAM 054/01. Samples were also analysed for free fluazifop acid (II) and free CF3-pyridone (X) using HPLC-UV and GC-MS method ARAM 195. Soils were not corrected for average concurrent recoveries (133% at 0.01 mg/kg fluazifop-butyl; 94–112% at 0.05–0.1 mg/kg fluazifop-butyl). Control samples were < 0.01 mg/kg for each analyte.

Zero day recoveries of fluazifop-butyl (i.e. the sum of free fluazifop-butyl, free fluazifop acid (II) and free CF3-pyridone (X), expressed in parent equivalents) were 49% (Pallhausen), 76% (Krukow), 80% (Varendorf) and 96% (Mechtersheim) of the nominal applied dose. No explanation was provided why the recovery for PallHausen was soo low.

Results for 0–5 cm and 5–10 cm soil cores are shown in Table 89. The parent compound was rapidly hydrolysed and was detected up to 14 days after treatment. Fluazifop acid (II) was detected up to 61 days after treatment with a maximum of 0.38–1.0 mg/kg dry soil at 0–14 days after treatment. CF3-pyridone (X) was no longer detected at 83–118 days at Krukow, Pallhausen and Varendorf sites, but was detected until the end of the study period (182 days) at the Mechtersheim site. CF3-pyridone (X) maximum concentrations of 0.05–0.13 mg/kg dry soil were found between 7–63 days.

No residues (<0.01 mg/kg dry soil) of free fluazifop-butyl, free fluazifop acid (II) or free CF3-pyridone (X) were found below the 10 cm soil horizon, except for one time point (Varendorf, 14 DAT, 0.01 mg/kg free fluazifop-butyl). This indicates no apparent leaching of fluazifop-butyl and its metabolites to deeper soil layers. No significant differences in the behaviour of fluazifop-butyl in various soils was observed when the autumn and spring applications are compared.

DT_{50} and DT_{90} values for dissipation of fluazifop acid (II) are shown in Table 90. DT_{50} and DT_{90} were in the range 3–25 days and 33–60 days**,** respectively. No kinetic endpoints were reported for fluazifop-butyl or CF3-pyridone (X).

Table 89 Soil residues (mg/kg dry soil) in field dissipation studies

Krukow, bare soil; 0.50 kg ai/ha; 17 Sept 1988					Pallhausen; bare soil, 0.50 kg ai/ha, 16 Sept 1988				
DAT	Depth (cm)	fluazifop-butyl mg/kg dry soil	fluazifop acid (II) mg/kg dry soil	CF3-pyridone (X) mg/kg dry soil	DAT	Depth	fluazifop-butyl mg/kg dry soil	fluazifop acid (II) mg/kg dry soil	CF3-pyridone (X) mg/kg dry soil
0	0-5	< 0.01	0.38	0.01	0	0-5	< 0.01	0.30	0.01
	5-10	< 0.01	< 0.01	< 0.01		5-10	< 0.01	< 0.01	< 0.01
14	0-5	< 0.01	0.12	0.06	14	0-5	< 0.01	0.39	0.02
	5-10	< 0.01	< 0.01	< 0.01		5-10	< 0.01	< 0.01	< 0.01
27	0-5	< 0.01	0.03	0.05	28	0-5	< 0.01	0.07	0.05
	5-10	< 0.01	0.01	0.02		5-10	< 0.01	< 0.01	< 0.01
61	0-5	< 0.01	0.02	0.03	63	0-5	NA	< 0.01	0.05
	5-10	< 0.01	< 0.01	0.02		5-10	NA	< 0.01	< 0.01
117	0-5	< 0.01	< 0.01	< 0.01	118	0-5	NA	< 0.01	0.01
	5-10	< 0.01	< 0.01	< 0.01		5-10	NA	< 0.01	0.01
					244	0-5	NA	NA	< 0.01
						5-10	NA	NA	< 0.01
Varendorf, bare soil, 0.50 kg ai/ha; 3 May 1989					Mechtersheim, bare soil, 0.52 kg ai/ha, 24 April 1989				
0	0-5	0.06	0.51	< 0.01	0	0-5	0.07	0.69	< 0.01
	5-10	< 0.01	< 0.01	< 0.01		5-10	< 0.01	0.02	< 0.01
7	0-5	0.26	0.82	0.11	7	0-5	< 0.01	0.39	< 0.01
	5-10	0.01	0.19	0.02		5-10	< 0.01	< 0.01	< 0.01
14	0-5	0.02	0.20	0.04	14	0-5	< 0.01	0.35	0.04
	5-10	< 0.01	< 0.01	< 0.01		5-10	< 0.01	< 0.01	< 0.01
	5-20	0.01	< 0.01	< 0.01		5-20	< 0.01	< 0.01	< 0.01
28	0-5	< 0.01	0.11	0.03	28	0-5	NA	0.09	0.08
	5-10	< 0.01	< 0.01	< 0.01		5-10	NA	< 0.01	0.01
55	0-5	NA	0.05	0.04	55	0-5	NA	< 0.01	0.07
	5-10	NA	< 0.01	< 0.01		5-10	NA	< 0.01	0.01
83	0-5	NA	< 0.01	< 0.01	83	0-5	NA	< 0.01	0.05
	5-10	NA	< 0.01	< 0.01		10-20	NA	< 0.01	< 0.01
112	0-5	NA	< 0.01	< 0.01	112	0-5	NA	< 0.01	0.04
	5-10	NA	< 0.01	< 0.01		5-10	NA	< 0.01	< 0.01
180	0-5	NA	< 0.01	< 0.01	182	0-5	NA	NA	0.02
	5-10	NA	< 0.01	< 0.01		5-10	NA	NA	< 0.01

NA: No analysis performed as residues in the previous samples were < 0.01 mg/kg

Table 90 Trigger Endpoints for fluazifop acid (II) for field dissipation studies on four locations in Germany

Location	Soil	Model	DT_{50} (days)	DT_{90} (days)
Krukow, Germany, bare soil, autumn 1988	sandy loam	Timme and Frehse	3	33
Pallhausen, Germany, bare soil, autumn 1988	clay loam	manual from graphs	25	60
Varendorf, Germany, bare soil, spring 1989	loam	Timme and Frehse	3	30
Mechtershemi, Germany,	silty clay loam	Timme and Frehse	3	17

Location	Soil	Model	DT_{50} (days)	DT_{90} (days)
bare soil, spring 1989				

Soil study 18

A field soil dissipation study was conducted in the USA in 1989 [Wiebe, 1990, RR 90-337B, PP5/0813]. An EC formulation of fluazifop-P-butyl (R-enantiomer) was applied to a cotton plot at 2 × 0.84 kg ai/ha with an interval of 28 days. The first application (31 May 1989) was on a 3 week old cotton (20% ground cover), the second application was on cotton with the first squares developing (28 June 1989, 60% ground cover). The soil characteristics are given in Table 91. Soil cores were removed at 4 dates after the first application (up to 28 days after the first application), then at 12 dates after the second application (last sampling 9 months after the second application). Cores were collected at 0–15 cm, 15–76 cm and 76–137 cm. The cores were divided in segments (0–15, 15–30, 30–46, 46–61, 61–76, 76–91, 91–122 cm) and stored at -10 °C or lower until extraction and analysis for a maximum of 243 days (CF3-pyridone (X)) or 126 days (fluazifop-butyl and fluazifop acid (II)). No crop samples were collected from this trial.

Table 91 Soil characteristics for a field dissipation study in Visalia, CA, USA

Site	Depth (cm)	OM%	Soil Type (USDA)	pH	CEC (meq/100g)	MHC% 0.33bar	MHC% 15 bar
Visalia, CA, USA, 1989	0-15	1.1	loam	7.3	8.4	20.6	6.16
	15-30	1.1	loam	7.1	8.7	-	-
	30-61	0.7	loam	8.1	11.7	-	-
	61-91	0.5	loam	8.3	15.6		-
	91-122	0.3	loam	8.3	19.1	-	-

Samples were analysed for fluazifop-butyl and fluazifop acid (II) using GC-MS method RR 89-072B; they were also analysed for CF3-pyridone (X), using GC-MS method RR 90-076B. Results were not corrected for average concurrent recoveries (91–111% at 0.01–1.0 mg/kg fluazifop-butyl, 89–100% at 0.01–1.0 mg/kg fluazifop acid (II) and 93–98% at 0.01–1.0 mg/kg CF3-pyridone (X). Control samples were < 0.01 mg/kg. Results in the samples were reported as received basis; results as mg/kg dry soil were not reported.

Residues of fluazifop-P-butyl, fluazifop acid (II) or CF3-pyridone (X) were never detected below 15 cm soil depth, at any time, indicating no apparent leaching of fluazifop-butyl and its metabolites.

Residues of free fluazifop-butyl, free fluazifop acid (II) and free CF3-pyridone (X) measured in the 0–15 cm soil layers at different times are reported in Table 92. The results are the average of 3 subplots (when one subplot residue is < 0.01 mg/kg, it is counted as 0.005 mg/kg in the calculation of the average).

The theoretical zero-day residue for a 1.7 kg ai/ha (2 × 0.84 kg ai/ha) application is 0.67 mg/kg soil. Since 2 applications were made with an interval of 28 days, the theoretical post-first application zero-day residue will give a more reasonable estimate and is 50% of 0.67 mg/kg soil or 0.34 mg/kg. The actual recovered amount (i.e. sum of free fluazifop-butyl, free fluazifop acid (II) and free CF3-pyridone (X), in parent equivalents) at zero-day is 0.20 mg/kg or 59% of the theoretical amount. As fluazifop-P-butyl was applied to cotton, rather than directly to bare soil, one would not expect to recover the theoretical amount due to foliar interception of the applied herbicide.

Fluazifop-butyl residues (0.07 and 0.15 mg/kg, as received, after the first and second application respectively) decreased to undetectable levels after 7 days of each application. After the second application, residues were determined at 6 time points between 0 to 7 days, which allowed estimation of a half-life of dissipation of 1.5 day for fluazifop-P-butyl.

The first maximum residue of fluazifop acid (II, 0.13 mg/kg, as received) was found at 7 days after the first application; the second maximum of fluazifop acid (II, 0.16 mg/kg, as received), was found at 7 days after the second application. Using the data from the 7 to 90 days after the second application, the half-life of fluazifop acid (II) was estimated as 18 days.

CF3-pyridone (X) was detected at low levels thouroughout the study period. Maximum levels of 0.03 mg/kg were found at 7–14 days after the second application and these decreased slowly to less than the LOQ (< 0.01 mg/kg, as received) at 270 days after the second application. Using the data from 7 to 270 days after the second application, the half-life of CF3-pyridone (X) was estimated as 108 days (see Table 93).

Table 92 Average residues (3 subplots) in 0-15 cm soil layers

Sampling	DAFT	fluazifop-butyl (mg/kg as received)	Fluazifop acid (II) (mg/kg as received)	CF3-pyridone (X) (mg/kg as received)
0 DAFT	0	0.07	0.11	< 0.01
7	7	< 0.01	0.13	0.01
14	14	< 0.01	0.09	0.02
28	28	< 0.01	0.04	0.02
0 DALT		0.15	0.14	0.02
4 hours		0.10	-	-
8 hours		0.12	-	-
1 day	29	0.09	-	-
2	30	0.10	-	-
7	35	< 0.01	0.16	0.03
14	42	< 0.01	0.08	0.03
28	56	-	0.05	0.02
60	88	-	0.02	0.02
90	118	-	< 0.01	0.01
180	208	-	< 0.01	0.01
270	298	-	-	< 0.01

DAFT: days after first treatment

DALT: days after last treatment; the second and last application was at 28 DAFT

- Not analysed

Table 93 Field dissipation half-lives for fluazifop-P-butyl, fluazifop acid (II) and CF3-pyridone (X)

	Fluazifop-P-butyl	Fluazifop acid (II)	CF3-pyridone (X)
	SFO	SFO	SFO
DT_{50}	1.5 days	18 days	108 days
DT_{90}	NC	NC	NC
r	0.972	0.990	0.946

NC: not calculated

Soil study 19

A field soil dissipation study was conducted in the USA in 1989 [Wiebe, 1990, RR 90-338B, PP5/1110]. An EC formulation of fluazifop-P-butyl (R-enantiomer) was applied to a cotton plot at 2 × 0.84 kg ai/ha with an interval of 28 days. The first application (27 June 1989) was on a 3 week old cotton (20% ground cover), the second application was on cotton with the peak squares (25 July 1989, 50% ground cover). As the rainfall was negligible in this arid region, irrigation was applied. The soil characteristics are given in Table 94. Soil cores were removed at 4 dates after the first application (up to 28 days after the first application), then at 12 dates after the second application (last sampling 9 months after the second application). Samples of soil at 0–15, 15–30, 30–61, 61–91 and 91–122 cm depths were collected for most of the sampling intervals. Soil Samples were stored at -10°C or lower until analysis for a maximum of 249 days (CF3-pyridone (X)) or 126 days (fluazifop-butyl and fluazifop acid (II)). No crop samples were collected from this trial.

Table 94 Field dissipation trial in Porterville (California) – Soil characteristics

Site	Depth (cm)	OM%	Soil Type (USDA)	pH	CEC (meq/100g)	MHC% 0.33bar	MHC% 15 bar
Porterville, CA, USA, 1989	0-15	1.7	sandy loam	7.4	7.5	14.5	10.7
	15-30	0.7	sandy loam	7.7	7.2	-	
	30-61	0.3	sandy loam	8.0	11	-	
	61-91	0.1	sandy loam	8.1	8.9	-	
	91-122	0.2	sandy loam	8.1	9.1	-	

Samples were analysed for fluazifop-butyl and fluazifop acid (II) using GC-MS method RR 89-072B; they were also analysed for CF3-pyridone (X), using GC-MS method RR 90-076B. Results were not corrected for average concurrent recoveries (91–111% at 0.01–1.0 mg/kg fluazifop-butyl, 89–100% at 0.01–1.0 mg/kg fluazifop acid (II) and 93–98% at 0.01–1.0 mg/kg CF3-pyridone (X). Control samples were < 0.01 mg/kg. Results in the samples were reported as received basis; results as mg/kg dry soil were not reported.

No residues (< 0.01 mg/kg as received) were detected below 15 cm, except at 28 days after the second application, when fluazifop acid (II) was measured in the 15–30 cm layer (0.02 mg/kg, as received) and in the 76–91 cm layer (0.01 mg/kg, as received). This indicates that vertical fluazifop acid (II) movement in soil is minimal.

Residues of fluazifop-butyl, fluazifop acid (II) and CF3-pyridone (X), measured in the 0–15 cm soil layers at different times, are reported in Table 95. The results are the average of 3 subplots (when one subplot residue is < 0.01, it is counted as 0.005 mg/kg in the calculations).

The theoretical zero-day residue for a 1.7 kg ai/ha (2 × 0.84 kg ai/ha) application is 0.72 mg/kg soil. Since 2 applications were made with an interval of 28 days, the theoretical post-first application zero-day residue will give a more reasonable estimate and is 50% of 0.67 mg/kg soil or 0.36 mg/kg. The actual recovered amount (i.e. sum of free fluazifop-butyl, free fluazifop acid (II) and free CF3-pyridone (X), in parent equivalents) at zero-day is 0.225 mg/kg or 63% of the theoretical amount. As fluazifop-P-butyl was applied to cotton, rather than directly to bare soil, one would not expect to recover the theoretical amount due to foliar interception of the applied herbicide.

Fluazifop-butyl residues (0.12 and 0.13 mg/kg, as received, after the first and second application respectively) decreased to undetectable levels after 28 days after the first application and 60 days after the second application. Using the data from the 0 to 60 day post second application, the half-life of dissipation for fluazifop-P-butyl was estimated as 13 days.

The first maximum residue of fluazifop acid (II, 0.14 mg/kg, as received) was found at 7 days after the first application; the second maximum of fluazifop acid (II, 0.19 mg/kg, as received), was found at 7-14 days after the second application. The maximum measured in individual subplots was 0.29 mg/kg, as received. Fluazifop acid (II) was no longer detected at 270 days after the second application. Using the data from the 7 to 270 days after the second application, the half-life of fluazifop acid (II) was estimated as 48 days.

CF3-pyridone (X) was detected at low levels only after the second application. Maximum levels of 0.02 mg/kg were found at 7, 28 and 180 days after the second application and these decreased to less than the LOQ (< 0.01 mg/kg, as received) at 270 days after the second application. Using the data from 7 to 270 days after the second application, the half-life of CF3-pyridone (X) was estimated as 241 days (see Table 96).

Table 95 Average residues (3 subplots) measured in 0-15 cm soil layer (mg/kg, as received)

Sampling	DAFT	fluazifop-butyl mg/kg as received	Fluazifop acid (II) mg/kg as received	CF3-pyridone (X) mg/kg as received
0 DAFT	0	**0.12**	0.09	< 0.01
7	7	0.04	**0.14**	< 0.01
14	14	0.02	0.12	< 0.01
28	28	< 0.01	**0.13**	< 0.01
0 DALT	-	**0.13**	0.12	< 0.01

Sampling	DAFT	fluazifop-butyl mg/kg as received	Fluazifop acid (II) mg/kg as received	CF3-pyridone (X) mg/kg as received
4 hours	-	**0.13**	-	-
8 hours	-	0.10	-	-
1 day	29	**0.12**	-	-
2	30	0.08	-	-
7	35	**0.19**	**0.19**	**0.02**
14	42	0.03	**0.19**	0.01
28	56	0.03	0.15	**0.02**
60	88	< 0.01	0.04	0.01
90	118	< 0.01	0.07	0.01
180	208	-	0.01	**0.02**
270	298	-	< 0.01	< 0.01

DAFT: days after first treatment

DALT: days after last treatment; the second and last application was at 28 DAFT

- Not analysed

Table 96 Field dissipation half lives for fluazifop-P-butyl, fluazifop acid (II) and CF3-pyridone (X)

	Fluazifop-P-butyl	fluazifop acid (II)	CF3-pyridone (X)
DT_{50}	13 days	48 days	241 days
DT_{90}	NC	NC	NC
r	0.942	0.967	0.539

NC: not calculated

Soil study 20

A field dissipation study was conducted in the USA from 21 July 2010 to 11 November 2011 [Wiepke *et al*., 2013, A12460A_50023, report TK0015266]. Thouree broadcast applications at 3×0.46 kg ai/ha with an EC formulation of fluazifop-P-butyl (R-enantiomer) were made with a 14-day interval to a bare soil plot. No tillage occurred in the bare soil plot. A soya bean plot received a broadcast applications with 0.46 kg ai/ha and a broadcast application of 0.12 kg ai/ha with an EC formulation of fluazifop-P-butyl with a 14-day interval. The soya beans were harvested on 30 November 2010 and the plant material was left on the plot. The plot remained fallow for the remainder of the study. Study characteristics are indicated in Table 97. Soil cores were taken at various intervals and were extracted at a depth of 0–7.5 cm (manual) and 7.5–91 cm (tractor mounted soil probe). Each soil core was sectioned into 0–7.5; 7.5–15; 15–30; 30–46; 46–61; 61–76 and 76–91 cm segments. Soil samples were stored at -10 °C for a maximum storage interval of 480 days. Whole soya bean plants and "simulated rainwater" samples were also collected from this trial; but these results are not summarized.

Table 97 Experimental conditions for field dissipation studies on fluazifop-P-Butyl

Location	Application	Soil type (USDA)	pH	% OM	CEC meq/ 100 g	% MHC at 1 bar	% MHC at 15 bar	Bulk density – disturbed (g/cc)
Chula, GA, USA, 2010 bare soil	3×0.46 kg ai/ha (interval 14 days) 1st.: 21 July 2nd: 4 Aug 3rd: 18 Aug	Sa (0-7.5 cm)	6.3	0.55	3.5	3.6	1.7	1.52
		Sa (7.5-15 cm)	6.0	0.59	3.9	4.6	1.9	1.49
		LSa (15-30cm)	6.1	0.38	3.7	3.8	1.9	1.50
		SaL (30-46 cm)	5.4	0.46	5.2	12.3	6.1	1.36
		SaCL (46-61 cm)	4.8	0.51	7.1	21.2	11.2	1.19
Chula, GA, USA, 2010 soya bean plot planted: 23 June harvest: 30 Nov	0.46 kg ai/ha (1st) 0.12 kg ai/ha (2nd) (interval 14 days) 1st.: 21 July (25% SC) 2nd: 4 Aug (60% SC)	Sa (0-7.5 cm)	7.2	0.59	4.9	6.5	2.1	1.51
		Sa (7.5-15 cm)	6.5	0.30	4.1	5.7	2.2	1.49
		Sa (15-30cm)	6.3	0.46	3.6	5.3	2.5	1.47
		SaL (30-46 cm)	6.4	0.30	5.3	16.2	7.7	1.31
		SaCL (46-61 cm)	5.8	0.08	6.1	15.5	8.3	1.34

1st application in the soya bean plot was at growth stage V3-V5 = 3-5 trifoliate leaf stage of soya bean plants

Sa = Sand, LSa = Loamy Sand; SaL = Sandy Loam, SaCL = sandy clay loam

SC = soil coverage: percentage of the crop that covers the soil

Potassium bromide was applied to a replicate area of the treated soya bean plot and the treated bare soil plot at a target rate of 0.11 kg KBr/ha. The bromide data demonstrated the movement of the wetting front (i.e., water) thourough the soil profile down to the 76–91 cm depth. This indicates that the plots received adequate moisture to evaluate the downward movement potential of fluazifop-P-butyl and its degradation products.

The collected soil samples were analysed for free fluazifop-butyl, free fluazifop acid (II) and free CF3-pyridone (X) using HPLC-MS/MS Method GRM044.03A. Results on dry weight basis are shown in Table 98. Results are not corrected for mean concurrent recoveries (83–107%, each analyte). Control samples were < 0.001 mg/kg dry soil.

The actual recovered amount (i.e. sum of fluazifop-butyl, fluazifop acid (II) and CF3-pyridone (X), in parent equivalents) at zero-day on the bare soil plot is 0.458, 0.456 and 0.514 kg ai/ha for the 3 applications, respectively or 109%, 106% or 121% of the theoretical amount. The actual recovered amount at zero-day on the soya bean plot is 0.431 and 0.089 kg ai/ha for the 2 applications, respectivly, or 103 and 72% of the theoretical amount. Since the percentage foliage coverage was 60% at the second application, a considerable amount of the spray solution was intercepted by the soya bean crop canopy.

In the bare soil and soya bean plot, fluazifop-butyl remained in the 0–7.6 cm soil layer, while fluazifop acid (II) distributed over the 0–15 cm soil layer with the exception of one single fluazifop acid (II) residue (0.0012 mg/kg dry soil) in the 15–30 cm soil layer at 7 days after the first treatment in the soya bean plot. In the bare soil and soya bean plot, CF3-pyridone (X) distributed over the 0–46 cm soil layers, with the exception of one single CF3-pyridone (X) residue (0.0010 mg/kg) in the 46–61 cm layer at 387 days after first treatment in the bare soil plot. No residues were found below 61 cm soil depth.

The highest mean concentrations of fluazifop-butyl and fluazifop acid (II) were found at the day of each application in both the bare soil plot and the soya bean plot.

In the bare soil plot with 3 applications of 0.46 kg ai/ha and 14 day intervals, thouree maxima were found for CF3-pyridone (X). The first maximum was found 5 days after the first application (0.022 mg/kg dry soil); the second maximum was found 7 days after the second application (0.059 mg/kg dry soil) and the third maximum was found 5 days after the third application (0.075 mg/kg dry soil). In the bare soil plot, CF3-pyridone (X) was still found at levels of 0.010 mg/kg dry soil at the end of the study period (478 days after the first application and 450 days after the first application).

In the soya bean plot with 1 application of 0.46 kg ai/ha and 1 application of 0.12 kg ai/ha and a 14 day interval, two maxima were found for CF3-pyridone (X). The first maximum was found 5 days after the first application (0.032 mg/kg dry soil) and the second maximum was found 7 days after the second application (0.043 mg/kg dry soil). In the soya bean plot, CF3-pyridone (X) was no longer detected at 478 days after the first application and 464 days after the second application.

Residue data for all soil depths were summed to get the total residues per analyte per sampling day. Untransformed replicate (n=3) residue data or mean residue data versus time data were subjected to non-linear regression analysis using the SFO kinetics model CAKE version 1.3. CAKE is a kinetic analysis tool designed to fit data to various kinetic models in accordance with the FOCUS degradation kinetics guideline. Regression on the mean residue data was performed if the replicates had large standard deviations.

Half lives are represented in Table 99. The results demonstrate that parent fluazifop-P-butyl degrades very rapidly (DT_{50} 1–2 days) in soil to its major degradation product fluazifop acid (II). Fluazifop acid (II) further degrades very rapidly (DT_{50} 1–3 days) to CF3-pyridone (X). Dissipation of CF3-pyridone (X) was slower with a DT_{50} of 100 days in the soya bean plot (2 applications; total rate

0.58 kg ai/ha) or 140 days in the bare soil plot (3 applications; total rate 1.4 kg ai/ha). CF3-pyridone (X) showed some mobility potential into the deeper soil layers.

Table 98 Residues in soil after bare soil treatment or soya bean treatment

Bare soil treatment; 3×0.46 kg ai/ha, interval 14 days					Soya bean treatment; 1 × 0.46 + 1 × 0.12 kg ai/ha, interval 14 days				
DALT – DAFT	Depth (cm)	fluazifop-butyl mg/kg dry soil	fluazifop acid (II) mg/kg dry soil	CF3-pyridone (X) mg/kg dry soil	DALT-DAFT	Depth (cm)	fluazifop-butyl mg/kg dry soil	fluazifop acid (II) mg/kg dry soil	CF3-pyridone (X) mg/kg dry soil
0-0; 1st	0-7.6	0.0068	0.351	0.0022	0-0; 1st	0-7.6	0.0056	0.337	0.0069
	7.6-15	-	-	-		7.6-15	-	-	-
1-1	0-7.6	0.0037	0.193	0.0078	1-1	0-7.6	0.0020	0.203	0.014
	7.6-15	< 0.001	< 0.001	< 0.001		7.6-15	< 0.001	0.016	0.0011
2-2	0-7.6	0.0015	0.185	0.015	2-2	0-7.6	< 0.001	0.218	0.020
	7.6-15	< 0.001	< 0.001	< 0.001		7.6-15	< 0.001	0.0031	0.0011
3-3	0-7.6	0.0012	0.122	0.017	3-3	0-7.6	< 0.001	0.185	0.022
	7.6-15	< 0.001	< 0.001	< 0.001		7.6-15	< 0.001	0.0019	< 0.001
5-5	0-7.6	0.0011	0.070	0.022	5-5	0-7.6	< 0.001	0.063	0.025
	7.6-15	< 0.001	0.0014	< 0.001		7.6-15	< 0.001	0.018	0.0072
7-7	0-7.6	< 0.001	0.032	0.020	7-7	0-7.6	< 0.001	0.022	0.019
	7.6-15	< 0.001	0.0096	0.0063		7.6-15	< 0.001	0.0096	0.0089
	15-30	< 0.001	< 0.001	< 0.001		15-30	< 0.001	0.0012	< 0.001
13-13	0-7.6	< 0.001	0.077	0.020	13-13	0-7.6	< 0.001	0.0065	0.018
	7.6-15	< 0.001	0.001	0.0093		7.6-15	< 0.001	0.0032	0.016
	15-30	< 0.001	< 0.001	< 0.001		15-30	< 0.001	< 0.001	0.0017
0-14; 2nd	0-7.6	0.0022	0.325	0.034	0-14; 2nd	0-7.6	< 0.001	0.064	0.016
	7.6-15	< 0.001	0.0011	0.0080		7.6-15	< 0.001	0.0011	0.014
	15-30	< 0.001	< 0.001	< 0.001		15-30	< 0.001	< 0.001	0.0012
1-15	0-7.6	0.0011	0.189	0.047	1-15	0-7.6	< 0.001	0.049	0.023
	7.6-15	< 0.001	0.0011	0.0090		7.6-15	< 0.001	0.0011	0.020
	15-30	< 0.001	< 0.001	< 0.001		15-30	< 0.001	< 0.001	0.0018
2-16	0-7.6	< 0.001	0.102	0.038	2-16	0-7.6	< 0.001	0.032	0.022
	7.6-15	< 0.001	0.0011	0.0086		7.6-15	< 0.001	0.0015	0.017
	15-30	< 0.001	< 0.001	< 0.001		15-30	< 0.001	< 0.001	0.0016
3-17	0-7.6	< 0.001	0.081	0.045	3-17	0-7.6	< 0.001	0.028	0.017
	7.6-15	< 0.001	0.001	0.0092		7.6-15	< 0.001	0.002	0.013
	15-30	< 0.001	< 0.001	< 0.001		15-30	< 0.001	< 0.001	0.0017
5-19	0-7.6	< 0.001	0.026	0.039	5-19	0-7.6	< 0.001	0.0089	0.018
	7.6-15	< 0.001	0.0075	0.015		7.6-15	< 0.001	0.0011	0.017
	15-30	< 0.001	< 0.001	< 0.001		15-30	< 0.001	< 0.001	0.0016
7-21	0-7.6	< 0.001	0.015	0.041	7-21	0-7.6	< 0.001	0.0084	0.021
	7.6-15	< 0.001	0.0021	0.017		7.6-15	< 0.001	0.0018	0.020
	15-30	< 0.001	< 0.001	0.0010		15-30	< 0.001	< 0.001	0.0018
13-27	0-7.6	< 0.001	0.0041	0.029					
	7.6-15	< 0.001	0.0011	0.021					
	15-30	< 0.001	< 0.001	0.0018					
0 – 28; 3rd	0-7.6	0.0038	0.366	0.029	14-28	0-7.6	< 0.001	0.0038	0.013
	7.6-15	< 0.001	0.0013	0.019		7.6-15	< 0.001	< 0.001	0.012
	15-30	< 0.001	< 0.001	0.0014		15-30	< 0.001	< 0.001	< 0.001
1-29	0-7.6	0.0010	0.173	0.043					
	7.6-15	< 0.001	< 0.001	0.020					
	15-30	< 0.001	< 0.001	0.0019					
2-30	0-7.6	0.0029	0.135	0.040					
	7.6-15	< 0.001	< 0.001	0.022					
	15-30	< 0.001	< 0.001	0.0020					
3-31	0-7.6	< 0.001	0.102	0.035					
	7.6-15	< 0.001	< 0.001	0.022					
	15-30	< 0.001	< 0.001	0.0016					
5-33	0-7.6	< 0.001	0.053	0.040					
	7.6-15	< 0.001	0.011	0.032					
	15-30	< 0.001	< 0.001	0.0030					

Bare soil treatment; 3×0.46 kg ai/ha, interval 14 days					Soya bean treatment; 1 × 0.46 + 1 × 0.12 kg ai/ha, interval 14 days				
DALT – DAFT	Depth (cm)	fluazifop-butyl mg/kg dry soil	fluazifop acid (II) mg/kg dry soil	CF3-pyridone (X) mg/kg dry soil	DALT-DAFT	Depth (cm)	fluazifop-butyl mg/kg dry soil	fluazifop acid (II) mg/kg dry soil	CF3-pyridone (X) mg/kg dry soil
7-35	0-7.6	< 0.001	0.027	0.042					
	7.6-15	< 0.001	0.0014	0.022					
	15-30	< 0.001	< 0.001	0.0022					
14-42	0-7.6	< 0.001	0.0075	0.043	28-42	0-7.6	< 0.001	0.0024	0.0090
	7.6-15	< 0.001	0.0011	0.022		7.6-15	< 0.001	0.001	0.018
	15-30	< 0.001	< 0.001	0.0018		15-30	< 0.001	< 0.001	0.0051
30-58	0-7.6	< 0.001	0.0060	0.038	44-58	0-7.6	< 0.001	0.0025	0.0055
	7.6-15	< 0.001	< 0.001	0.019		7.6-15	< 0.001	< 0.001	0.013
	15-30	< 0.001	< 0.001	0.0014		15-30	< 0.001	< 0.001	0.0017
61-89	0-7.6	< 0.001	0.0038	0.030	75-89	0-7.6	< 0.001	0.0021	0.0051
	7.6-15	< 0.001	< 0.001	0.018		7.6-15	< 0.001	< 0.001	0.0086
	15-30	< 0.001	< 0.001	0.0031		15-30	< 0.001	< 0.001	0.0031
86-114	0-7.6	< 0.001	0.0029	0.019	100-114	0-7.6	< 0.001	0.0020	0.0037
	7.6-15	< 0.001	< 0.001	0.013		7.6-15	< 0.001	< 0.001	0.0071
	15-30	< 0.001	< 0.001	0.0025		15-30	< 0.001	< 0.001	0.0037
124-152	0-7.6	< 0.001	0.001	0.0087	138-152	0-7.6	< 0.001	< 0.001	0.0020
	7.6-15	< 0.001	< 0.001	0.013		7.6-15	< 0.001	< 0.001	0.0059
	15-30	< 0.001	< 0.001	0.0047		15-30	< 0.001	< 0.001	0.0025
180-208	0-7.6	< 0.001	< 0.001	0.0045	194-208	0-7.6	< 0.001	< 0.001	0.0011
	7.6-15	< 0.001	< 0.001	0.0080		7.6-15	< 0.001	< 0.001	0.0040
	15-30	< 0.001	< 0.001	0.0052		15-30	< 0.001	< 0.001	0.0030
	30-46	< 0.001	< 0.001	0.0010		30-46	< 0.001	< 0.001	< 0.001
273-301	0-7.6	< 0.001	< 0.001	0.0051	287-301	0-7.6	< 0.001	< 0.001	0.0018
	7.6-15	< 0.001	< 0.001	0.0091		7.6-15	< 0.001	< 0.001	0.0043
	15-30	< 0.001	< 0.001	0.0051		15-30	< 0.001	0.001	0.0021
	30-46	< 0.001	< 0.001	0.0010		30-46	< 0.001	< 0.001	< 0.001
					292-306	0-7.6	< 0.001	< 0.001	0.0018
						7.6-15	< 0.001	< 0.001	0.0030
						15-30	< 0.001	< 0.001	0.0034
						30-46	< 0.001	< 0.001	< 0.001
359-387	0-7.6	< 0.001	< 0.001	0.0012	373-387	0-7.6	< 0.001	< 0.001	< 0.001
	7.6-15	< 0.001	< 0.001	0.0044		7.6-15	< 0.001	< 0.001	0.0011
	15-30	< 0.001	< 0.001	0.0047		15-30	< 0.001	< 0.001	0.0013
	30-46	< 0.001	< 0.001	0.0013		30-46	< 0.001	< 0.001	< 0.001
	46-61	< 0.001	< 0.001	0.0010		46-61	< 0.001	< 0.001	< 0.001
450-478	0-7.6	< 0.001	< 0.001	0.0014	464-478	0-7.6	< 0.001	< 0.001	< 0.001
	7.6-15	< 0.001	< 0.001	0.0036		7.6-15	< 0.001	< 0.001	< 0.001
	15-30	< 0.001	< 0.001	0.0040		15-30	< 0.001	< 0.001	< 0.001
	30-46	< 0.001	< 0.001	0.0013		30-46	< 0.001	< 0.001	< 0.001
	46-61	< 0.001	< 0.001	< 0.001		46-61	< 0.001	< 0.001	< 0.001

DAFT: days after first treatment

DALT: days after last treatment

Table 99 DT_{50} and DT_{90} values calculated by the CAKE model

Location	Compound	Application and date	Model	DT_{50} (days)	DT_{90} (days)	$\chi 2$ error %	r2
Chula, GA, USA, 2010 bare soil	fluazifop-P-butyl	1st 21 July 2nd 4 Aug 3rd 18 Aug	SFO	1 1- 2	4 NC	13.38 -	0.8057 -
	Fluazifop acid (II)	1st 21 July 2nd 4 Aug 3rd 18 Aug	SFO	2 1 1.5	7 5 5	11.04 6.447 19.41	0.891 0.9804 0.957
	CF3-pyridone (X)	1st 21 July 2nd 4 Aug 3rd 18 Aug	SFO	- - 140	- - 470	- - 11.17	- - 0.8392

Location	Compound	Application and date	Model	DT_{50} (days)	DT_{90} (days)	$\chi 2$ error %	r2
Chula, GA, USA, 2010 soya bean plot	fluazifop-P-butyl	1st 21 July 2nd 4 Aug	SFO	~ 1 NC	NC NC	- -	- -
	fluazifop acid (II)	1st 21 July 2nd 4 Aug	SFO	3 2	9 8	10.20 12.53	0.8787 0.9305
	CF3-pyridone (X)	1st 21 July 2nd 4 Aug	SFO	- 100	- 330	- 15.19	- 0.7849

NC= not calculated/ unsufficient data. After the second and third application, there were insufficient time-points with parent residues tocalculate a meaningful half-life or DT_{50}

Soil photolysis

Soil study 21

The rate of photolysis was investigated on a loam soil [MacNeil *et al*., 1981, PP9/0278, report RJ0191B]. ^{14}C-phenyl or ^{14}C-pyridyl labelled fluazifop-P-butyl (RS) was applied to thin layers (0.5 mm) at a rate equivalent to 0.25 kg ai/ha (for the phenyl label) and 0.23 k gai/ha (for the pyridyl label). Soil characteristics are shown in Table 100. Soil was exposed to natural daylight (6 June to 8 July 1980) for a period of 32 days (Bracknell, Berkshire, UK, at latitude 51°23'N and longitude 0°47'W, temperatures 8.8–23.0 °C between 9.00–21.00 hour). Volatiles were not collected. Soil was sampled at times equivalent to 0, 1, 2, 4, 16 and 32 days. Dark control plates (placed in quartz flasks or borosilicate flasks wrapped with silver foil) were also incubated under these conditions for 32 days.

Soil was extracted with acetonitrile and acetonitrile/water. Radioactivity in the extracts was quantified by (combustion) LSC; solids were not analysed. Recoveries ranged from 100–86% TAR (0–32 days) in the exposed samples and 88–89% TAR in the dark controls (32 days).

Extracts were analysed by TLC using reference standards for fluazifop-butyl (I), fluazifop acid (II), Pyr-Ph ether (IV) and CF3-pyridone (X). The degradation was negligible in the dark controls where fluazifop-butyl was still present at 81–82% TAR at the study end. In the irradiated samples a small decrease of fluazifop-butyl was observed at the end of the test: from 88–92% TAR at time zero to 71–80% TAR at the end of the test. At least 6 photodegradates were formed. Fluazifop acid (II, 1–2% TAR) and an uncharacterized photoproduct (2–4% TAR) were one of these. CF3-pyridone (X, 2% TAR) was additionally characterized from the ^{14}C-pyridyl label. All other radioactive photoprodcuts were present in smaller quantities and accounted in total for less than 10% TAR.

The estimated half-life of fluazifop-butyl was greater than 70 days.

Soil study 22

The rate of photolysis was investigated on a loam soil [French and Matharu, 1989, PP5/0801, report RJ0795B]. ^{14}C-phenyl or ^{14}C-pyridyl labelled fluazifop-P-butyl (R-enantiomer) was applied to thin layers (1 mm) of Acres soil at a rate equivalent to 0.42 kg ai/ha. Soil characteristics are shown in Table 100. The R:S enantiomer ratio was 92.9:7.1 and 96.7:3.3 for the phenyl and pyridyl labels respectively. Plates were exposed to a xenon arc lamp, filtered to emit a spectrum similar to that of natural sunlight, for a time period equivalent to 30 days of Florida summer light (25–35 ºN, 12 hours of light per day), at 25 ± 5 °C. Volatiles were trapped in a series of traps (sulphuric acid, 2-methoxyethanol, ethanolamine, potassium hydroxide). Soil was sampled at times equivalent to 7, 14, 22 and 30 days of Florida summer sunlight. Dark control plates were also incubated at 25 ± 5 °C.

Soil was extracted with acetonitrile, acetonitrile/water and 1 M HCl. Radioactivity in the extracts and remaining solids was quantified by (combustion) LSC. Recoveries ranged from 96–110% TAR.

After partitioning with dichloromethane, extracts were analysed by TLC using reference standards for fluazifop-butyl (I), fluazifop acid (II), Pyr-Ph ether (IV) and CF3-pyridone (X). The degradation was negligible in the dark controls where fluazifop-butyl was still present at 92–93%

TAR at the study end. In the irradiated samples a slight decrease of fluazifop-butyl was observed at the end of the test: from 98% TAR at time zero to 75–85% TAR at the end of the test. Small amounts of fluazifop acid (II), Pyr-Ph ether (IV) and CF3-pyridone (X), each less than 10% TAR, were formed at the end of the test. Unextracted residues were < 5.7% TAR and the evolved CO_2 was < 1.1% TAR at the end of the test.

Degradation of fluazifop-P-butyl follows first order kinetics. The half-life of degradation was calculated by linear regression analysis of the log of the average residue percentage against sampling time. In irradiated samples the average half-life was calculated as 116 days (r2=0.89, k=5.92 $\times 10^{-3}$/day) of Florida summer sunlight. In the dark control samples, it was 272 days (r2=0.93, k=2.37$\times 10^{-3}$/day). Photodegradation is therefore not a major route of degradation for fluazifop-P-butyl.

Table 100 Soil characteristics

	Study 21	Study 22
Soil name		Acres
Location		Bracknell Berkshire, UK
Soil texture [a]	loam	loam
-- Sand	60.0%	49%
-- Silt	16.0%	28%
-- Clay	24.0%	22%
Organic Carbon (%)	-	2.5%
Organic Matter (%)	4.3%	4.3%
CEC (meq/100 g)	19.0	16.4
pH I	7.25	6.5
Moisture holding capacity at 0.33 bar (%)	-	22%
Moisture holding capacity at 10 bar (%)	-	-
MWHC (%)	-	-
% MWHC at incubation	-	-
Bulk density (g/mL)	-	-
Microbial biomass (mg microbial carbon/kg soil)	-	-

[a] Soil texture based on New Jersey Scale for study 1 and USDA characterisation for study 2

Proposed degradation pathway of fluazifop-P-butyl in/on aerobic soil

The proposed degradation pathway of fluazifop-P-butyl in/on aerobic soil is shown in Figure 4. Under aerobic soil conditions, fluazifop-P-butyl (I) is rapidly degraded into the primary degradation product fluazifop acid (II). Degradation of fluazifop acid (II) occurred both by ether cleavage resulting in the formation of CF3-pyridone (X) and by hydrolysis of the propionic acid moiety to form Pyr-Ph ether (IV) and compound 7 (VII). Further decomposition occurred by cleavage of both the phenyl and pyridyl rings resulting in a steady increase of $^{14}CO2$ evolution with time.

Photodegradation is not a major route of degradation for fluazifop-P-butyl.

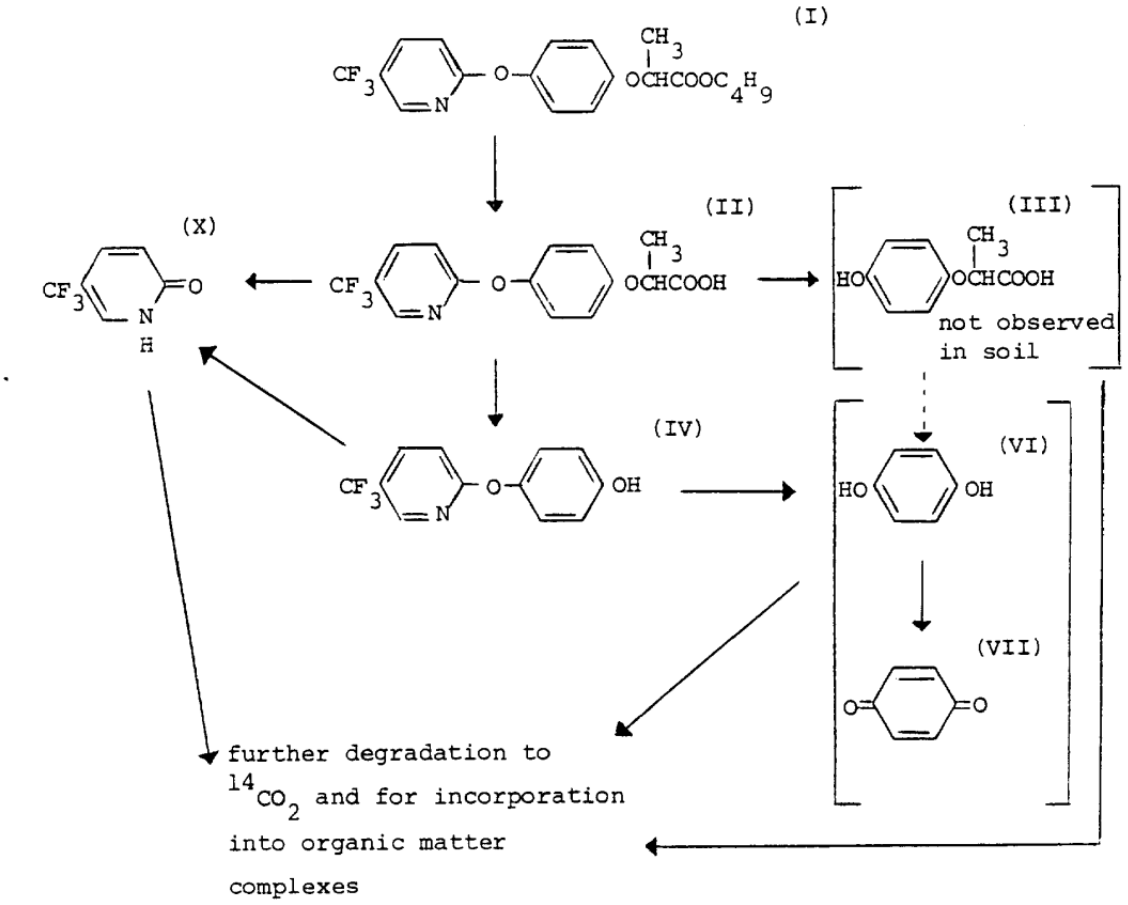


Figure 4 Proposed degradation pathway of fluazifop-P-butyl in/on aerobic soil

Environmental fate in water/sediment systems

No data submitted. Not relevant for the present intended use.

RESIDUE ANALYSIS

The Meeting received information on enforcement/monitoring methods for the determination of total fluazifop (i.e. fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant and animal commodities. In addition the Meeting received information on analytical methods for the determination of total fluazifop or CF3-pyridone (X) as used in the various study reports (supervised residue trials, storage stability studies, processing studies, feeding studies).

Fluazifop-butyl and fluazifop acid (II) occur in two isomeric forms: the R- and S-isomer. The R-isomer is responsible for the herbicide activity and is the main component of fluazifop-P-butyl and fluazifop-P acid. The R- and S-isomer cannot be separated by the chouromatographic techniques applied in the analytical methods described below.

[Moore, 2014, PP5/50544, report TK0251855] provided information on the suitability of the analytical methods to evaluate residues of fluazifop-P-butyl. In soil [Bewick, 1983, PP5/0276, report RJ0306B] only 5% inverted from R to S following dosing with the R-enantiomer (i.e. fluazifop-P-butyl), while 98% inverted from S to R by day 7 following dosing with the S-enantiomer. In another soil study [Bewick, 1982, PP9/0277, RJ0270B] fluazifop acid (II) enantiomer ratio changed to > 4:1

of R:S by day 2 following dosing with 51:49 R:S racemate of fluazifop-butyl. In two separate studies in plants [Evans and Cavell, 1984, PP9/0048, report RJ0356B; Evans and Cavell, 1984, PP9/0043, report RJ0353B] little or no epimerization occurred in plants when fluazifop-butyl transformed to fluazifop acid (II) or its conjugates. These studies indicated very low probability of R forms inverting to S forms in either soil or plants. Therefore studies with fluazifop-butyl racemate can be used to evaluate residue levels for fluazifop-P-butyl in soil and plants without knowledge of the enantiomeric form of fluazifop-butyl or its degradates.

The analytical residue methods have been evaluated according to the guidance provided by OECD (Series on Pesticides number 39) as indicated on page 25 of the FAO manual 2009.

Validation results are required for every commodity submitted for MRL-setting: at least one full validation for a commodity within the five defined crop groups (high acid content, high water content, high oil content, high protein content, high starch content) and a reduced validation for every other commodity within a certain crop group. Where validation results do not meet the criteria given below, this is indicated.

When the analytical method is validated according to a full validation scheme, it means that

- at least 5 recovery experiments per level were conducted on at least 2 levels (LOQ and 10× LOQ) and average recovery per level was shown to be between 70–120% and the relative standard deviation (RSD_r or CV) per level was shown to be < 20%,
- at least two control samples were analysed and were shown to be below 0.3×LOQ and
- the calibration was conducted with at least 5 single points or at least 3 duplicate points and was shown to be linear (either standards in solvent or matrix matched standards).
- When the analytical method is validated according to a reduced validation scheme, it means that
- a full validation is available for a crop in the same crop group (high acid content, high water content, high oil content, high protein content, high starch content);
- at least 3 recovery experiments per level were conducted on at least 2 levels (LOQ and 10× LOQ) and the average recovery per level was shown to be between 70–120% and the relative standard deviation (RSD_r or CV) per level was < 20%;
- at least two control samples were analysed and shown to be below 0.3× LOQ
- the calibration was conducted with at least 5 single points or at least 3 duplicate points and was shown to be linear (only relevant for matrix matched standards; standards in solvent are already covered by full validation).

When a method is intended for enforcement and monitoring, a full validation is required for one crop within each of the representative crop groups ((high acid content, high water content, high oil content, high protein content, high starch content), a validation is required for a confirmation method and the method needs to be validated by an independent laboratory.

Optimisation of extraction and hydrolysis conditions

The Meeting received several additional studies to show extraction efficiency for fluazifop acid (II) and total fluazifop, stability of fluazifop acid (II) or CF3-pyridone (X) under the hydrolysis conditions applied and hydrolysis efficiency of fluazifop acid (II) from supervised residue trial samples.

Study 1

Extractability of **fluazifop acid** (II) was investigated in soya bean seeds from trial CA/ON/HE/79/511 conducted in 1979 in Canada [Atreya and Collis, 1980, PP9/0497, report PP009B003]. The soya bean seeds (ADJ 5009/79) were obtained from soya bean plants that were treated on 7 July 1979 at the growth stage of 1 trifoliate leaf with an EC formulation of fluazifop-butyl (racemate) at a rate of 0.75 kg ai/ha and from which the seeds were harvested 111 days later.

To choose the suitable extraction procedure for fluazifop acid (II), the homogenised soya bean seeds (10 g) underwent a pre-treatment and extraction as listed in Table 101. Samples were filtered and than analysed. The extracts were cleaned-up and residues were derivatised to their methylated brominated fluazifop acid (II) derivative and analysed by GC-ECD.

Recovery of the analytical method was verified by fortifying untreated soya bean seeds with 0.2 mg/kg fluazifop acid (II), prior to sample pre-treatment. Recoveries were satisfactory (results not shown).

Extractability of fluazifop acid (II) is shown in Table 101. Soaking of the soya bean seeds prior to extraction is essential as well as an extraction under acid conditions. The sample pre-treatment may be either soaking with water overnight or soaking with 1 M HCl for a minimum of 2 hours. The best extraction conditions for quantitative recovery of fluazifop acid (II) residues from soya bean seeds are:

- overnight soaking in water and extraction with acetonitrile/concentrated HCl
- overnight soaking in 1 M HCl and cold extraction with acetonitrile
- 2 hours soaking in 1 M HCl and hot extraction with acetonitrile (reflux, 1hour).

Table 101 Extractability of fluazifop acid (II) from treated soya bean seeds

Sample pre-treatment (10 g homogenised sample)	Extraction solvent (100 mL)	Average result (n=1-3)
none	acetonitrile/concentrated HCl (98:2, v/v)	0.07
2 hours soaking in 20 mL water	acetonitrile/concentrated HCl (98:2, v/v)	0.15
2 hours soaking in 20 mL water	acetonitrile	0.11
2 hours soaking in 20 mL 1 M HCl	acetonitrile	0.17
2 hours soaking in 20 ml 1 M HCl	acetonitrile, reflux for 1 hour	0.19
overnight soaking in 20 mL water	acetonitrile	0.10
overnight soaking in 20 mL water	acetonitrile/concentrated HCl (98:2, v/v)	0.19
overnight soaking in 30 mL water	acetonitrile/concentrated HCl (98:2, v/v)	0.21
overnight soaking in 20 ml 1 M HCl	acetonitrile	0.20
overnight soaking in 10% TCA	acetonitrile	0.18
overnight soaking in 10% TCA	acetonitrile/concentrated HCl (98:2, v/v)	0.18
overnight soaking in 20% TCA	acetonitrile/concentrated HCl (98:2, v/v)	0.22

TCA = trichloroacetic acid

Study 2

Extractability of total fluazifop (i.e. fluazifop-butyl, fluazifop acid (II) its conjugates) was investigated in lettuce and fodder beets from trials conducted in 1981-1982 [Atreya and Upton, 1984, PP9/0102, report PP009B281]. The fodderbeet roots (5081/81) and lettuce leaves (sample ID unknown) were obtained from plants treated with fluazifop-butyl (racemate) with a single rate of 0.75 or 0.50 kg ai/ha, respectively, and from which the roots or leaves were harvested 21 days later.

To choose the suitable extraction procedure for total fluazifop, homogenised samples of lettuce and fodder beets containing residues of total fluazifop underwent a pre-treatment and extraction as listed in Table 102. An internal standard was added to each sample prior to extraction. The internal standard 2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy)butyric acid, MW=341, is a synthetic analogue of fluazifop acid (II) in which the methyl is replaced by the ethyl on the side chain. An aliquot of the extract was evaporated to the aqueous volume and hydrolysed by heating at 60 ºC for 1 hour in 6 M HCl. The hydrolysate was cleaned-up as described for Method PPRAM 62/1 and than analysed by HPLC-UV, using internal standard calibration.

Recovery was verified by fortifying the treated fodderbeet and lettuce samples with the internal standard. Recoveries were satisfactory (see Table 102). Therefore, extraction, hydrolysis and clean-up steps used in the analytical method do not result in significant losses of fluazifop acid (II).

Extractability of total fluazifop is shown in Table 102. Soaking of fodder beet roots or lettuce leaves prior to extraction is not necessary, but extraction under acid conditions is essential. The best extraction conditions for quantitative recovery of total fluazifop from fodderbeet roots or lettuce leaves were:

- direct extraction with acetonitrile/concentrated HCl (98:2, v/v)
- direct extraction with hot acetonitrile/concentrated HCl (98:2, v/v, 1 hour, reflux)

Table 102 Extractability of total fluazifop from treated lettuce and fodderbeet roots

Sample pre-treatment (homogenised sample)	Extraction solvent	Fodderbeet roots Average result (n=3)	Lettuce leaves Average result (n=3)	Average Concurrent recovery internal std fodderbeet (n=3)	Average Concurrent recovery internal std lettuce (n=3)
none	acetonitrile/water (1:1, v/v)	0.34	2.5	87	81
none	**acetonitrile/conc HCl (98:2, v/v)**	**0.42**	**2.4**	77	80
2 hours soaking in 20 mL water	acetonitrile	0.41	2.5	83	84
2 hours soaking in 20 mL 1 M HCl	acetonitrile	0.39	2.3	84	84
none	**acetonitrile/conc HCl (98:2, v/v), 1 hour reflux**	**0.43**	**2.8**	72	na

na = not analysed

Study 3

Efficiency of hydrolysis for cleavage of fluazifop (II) conjugates was investigated using supervised residue trial samples of soya bean seeds [Atreya *et al*., 1981, 462775, report PP009B030]. The soya bean seeds were obtained from ADJ No 5562/80 (2 kg ai/ha, DAT 128).

Total fluazifop was determined by soaking homogenised soya bean seeds (10 g) with 1 M HCl or water as indicated in Table 103. Samples were extracted by maceration with acetonitrile (acetonitrile/1M HCl = 100:20, v/v). An aliquot of the extract, equivalent to 5 g sample, was evaporated to remove the acetonitrile. The remaining acqueous phase was hydrolysed by various conditions as indicated in Table 103 to cleave fluazifop (II) conjugates to fluazifop acid (II). The hydrolysate was partitioned into dichloromethane and fluazifop acid (II) was determined as for PPRAM 62 (by coagulation and HPLC-UV detection).

Hydrolytic efficiency under various conditions is shown in Table 103. No residue of fluazifop-butyl has been found in mature crops. Fluazifop-butyl is extracted with acetonitrile and hydrolysed to fluazifop acid (II) (100%) with 6 M HCl at 60 ºC for 1 hour (data not shown).

There was no significant difference between residues obtained from any of the systems used. Most analytical methods on plant commodities used 6 M HCl for 1 hour at 60 ºC. Under these conditions the extracted fluazifop acid (II) remains stable and is cleaved efficiently from its conjugates.

Table 103 Hydrolysis efficiency of fluazifop acid (II) in soya bean seeds from supervised residue trials

Presoak	Hydrolysis conditions	Average total fluazifop mg/kg n=2-3
1 M HCl, 2 hours	1 M HCl, 3 hours, 60 ºC	0.25
1 M HCl, 2 hours	**6 M HCl, 1 hour, 60 ºC**	**0.33**
water, 2 hours	6 M HCl, 3 hours, 60 ºC	0.28

Presoak	Hydrolysis conditions	Average total fluazifop mg/kg n=2-3
1 M HCl, 2 hours	6 M HCl, 3 hours, 60 ºC	0.31
1 M HCl, 2 hours	6 M HCl, 6 hours, 60 ºC	0.27
1 M HCl, 2 hours	6 M HCl, 3 hours, 80 ºC	0.31

Analytical methods for enforcement in plant commodities

HPLC-MS/MS Method GRM044.02A was submitted as enforcement/monitoring methods for the determination of total fluazifop (i.e. fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant commodities. The method is described below, since it was also used in some study reports. Total fluazifop cannot be determined by existing multi-residue method, since hydrolysis is needed to release fluazifop acid (II) from its conjugates.

Analytical methods for enforcement in animal commodities

GC-MS Method RAM 331/01 was submitted as enforcement/monitoring methods for the determination of total fluazifop (i.e. fluazifop-butyl, fluazifop acid (II) and its conjugates, expressed as fluazifop acid (II)) in animal commodities. The method is described below, since it was also used in some study reports. Total fluazifop cannot be determined by existing multi-residue method, since hydrolysis is needed to release fluazifop acid (II) from its conjugates.

Analytical methods used in study reports in plant commodities

Several analytical methods were submitted for use in supervised residue trials, processing studies, storage stability studies on plant commodities and rotational crop studies.

HPLC-UV Method PPRAM 51

HPLC-UV Method PPRAM 51 (1979-1980) determines fluazifop-butyl in various plant commodities. Fluazifop acid (II) or its conjugates are not included. The reported LOQ is 0.01 mg/kg

A method description is available [Atreya *et al*, 1980, PP9/0385, PPRAM 51] for dry and wet plant commodities. Samples (25 g) are extracted by homogenisation with acetonitrile/water (90:10, v/v). After filtration and dilution with water, fluazifop-butyl is partitioned into hexane. The hexane extract is dried with anhydrous sodium sulfate and concentrated by evaporation. Florisil adsorption column chouromatography is used to remove interfering crop co-extractives. Fluazifop-butyl is eluted from the column by 30% ether in hexane and then evaporated to dryness. The residuum is redissolved in acetonitrile/water (65:35 v/v). Quantitative determination is performed by HPLC using UV-detection.

Qualitative and quantitative confirmation is carried out by combined GC-MS. An alternative method utilising the bromination of fluazifop-butyl may be used for confirmation.

A validation report is not available. HPLC-UV Method PPRAM 51 was used in supervised residue trials on various crops [RJ0226B; no validation reported] and potatoes [PP009B010, PP009B013]. Concurrent method validation results extracted from supervised trial reports are summarized in Table 104.

Reviewer's conclusion: Method PPRAM 51 is considered not valid for determination of total fluazifop in plant commodities, since fluazifop acid (II) or its conjugates are not included. Method PPRAM 51 is considered valid (reduced validation) for the determination of fluazifop-butyl in potatoes (at 0.05 mg/kg only). The valid LOQ is 0.02 mg/kg (no validations below this level).

Table 104 Validation results for HPLC-UV method PPRAM 51 (free fluazifop-butyl only)

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
potatoes	0.02	0.02 (Fb) 0.05 (Fb)	81 75	71-91 69-83	- 9.4	2 3	< 0.02	no data	PP9/0507; PP009B013
potatoes	0.02	0.02 (Fb) 0.05 (Fb)	88 88	70-105 81-110	- 7.2	2 4	< 0.02	no data	PP9/0504; PP009B010

Legends to this and all further tables:

LOQ limit of quantification, proposed by study author, may not be validated.

Control maximum or range of concentrations in given number of untreated (control) samples.

RSD relative standard deviation of recoveries found.

n number of samples used for validation of the analytical method

ns not stated (i.e. not reported)

Fb fortified with fluazifop-butyl, results are for total fluazifop recovery

F fortified with fluazifop acid (II), results are for fluazifop acid (II) recovery

IS fortified with internal standard, results are for internal standard recovery

X fortified with CF3-pyridone (X), results are for CF3-pyridone (X) recovery

GC methods TRAM and PPRAM 52

GC methods TRAM and PPRAM 52 (1981-1982) determine free fluazifop acid (II) in various plant commodities. Fluazifop-butyl or the fluazifop (II) conjugates are not included. A method description is not available [Syngenta, Response to Questions 02, March 2015].

GC methods TRAM and PPRAM 52 (1981-1982) were used in supervised residue trials on tomatoes [RIC2816], potatoes [PP009B013, PP009B10], witloof [RIC2816] and in a storage stability study on soya bean [PP009B017; no validation reported]. Concurrent method validation results extracted from these reports are summarized in Table 105.

Reviewer's conclusion: Methods TRAM and PPRAM 52 are considered not valid for determination of total fluazifop in plant commodities since fluazifop-butyl and the fluazifop (II) conjugates are not included. GC methods TRAM and PPRAM 52 are considered:

- valid (reduced validation) for the determination of free fluazifop acid (II) in potatoes (0.02 0.05 mg/kg);
- insufficiently validated for the determination of free fluazifop acid (II) in witloof roots and tomatoes.
- The valid LOQ is 0.02 mg/kg (no validations below this level)

Table 105 Validation results for GC method PPRAM 52 (free fluazifop acid (II) only)

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
tomatoes	0.02	0.02- 0.08 (F) separate levels not indicated	70	ns	8.6%	6	< 0.02 (1)	no data	PP5/0280; RIC2816
potatoes	0.02	0.02 (F) 0.05 (F)	77 117	64-90 96-140	13% 17%	4 5	< 0.02	no data	PP9/0507; PP009B013
potatoes	0.02	0.05 (F) 0.1 (F) 0.2 (F)	94 76 -	90-100 72-80 70	5.2% - -	4 2 1	< 0.02	no data	PP9/0504; PP009B010
witloof roots	0.02	0.02-0.08 (F) separate levels not indicated	80	ns	ns	ns	< 0.02 (1)	no data	PP5/0280; RIC2816

HPLC-UV Method PPRAM 53

HPLC-UV Method PPRAM 53 determines free fluazifop acid (II) in various plant commodities. Fluazifop-butyl or fluazifop (II) conjugates are not included. A method description is not available [Syngenta, Response to Questions 02, March 2015].

HPLC-UV method PPRAM 53 was used in supervised residue trials on various crops [RJ0226B], but validation results were not reported.

HPLC-UV method PPRAM 53/1 is a modification of method PPRAM 53 and includes a hydrolysis step. Method PPRAM 53/1 is identical to PPRAM 62 and validation results are described under PPRAM 62.

Reviewer's conclusion: Method PPRAM 53 is considered not valid for determination of total fluazifop in plant commodities, since fluazifop-butyl or fluazifop (II) conjugates are not included and validation results are not available. An LOQ is not indicated.

GC-MS or HPLC-UV Method ref III for determination of total despyridinyl acid (III)

Method ref III determines total despyridinyl acid (III) (free and conjugates as one single analyte) in various plant commodities. The reported LOQ is 0.05 mg/kg total despyridinyl acid (III).

GC-MS ref III is described by [Francis and Kennedy, 1981, 407582, PP009B042] for cottonseed. An internal standard ((4-hydroxy phenoxy) acetic acid) is added to homogenised cottonseeds prior to extraction. Cotton seeds are soaked in 1 M HCl for at least 2 hours. Samples were extracted by maceration with acetonitrile/1M HCl (50:50, v/v) and then filtered. The acetonitrile is removed from the filtrate by rotary evaporation and the remaining aqueous extract is partitioned with dichloromethane to remove the oils. The aqueous phase is refluxed for 1 hour in 6 M HCl to hydrolyse the conjugates of despyridinyl acid (III). Despyridinyl acid (III) is partitioned from the hydrolysate into diethyl ether. The diethyl ether phase was shaken with 0.1 M NaOH solution. The aqueous phase is acidified and despyridinyl acid (III) is partitioned into ethyl acetate. The ethyl acetate extract is concentrated and despyridinyl acid (III) is derivatised with 4 M HCl in butanol for 1 hour at 105 °C to the butyl ester of despyridinyl acid (III). The solution is cleaned-up by adsorption chouromatography (Fractosil). The eluate is evaporated to near dryness and treated with heptafluorobutyric anhydride for 30 min at 70 °C to convert the butyl ester of despyridinyl acid (III) into the C_3F_7CO derivative. The extracts are analysed by GC-MS.

GC-MS Method ref III was used in supervised residue trials on cottonseed [Ussary, 1981, 405793, TMU0680/B]. Control samples were < 0.05 mg/kg in cottonseeds but further validation results were not reported.

Modification A of GC-MS method Ref III was used on lettuce, cucumber, carrots, dry harvested bulb onions and green onions [Atreya and Dick, 1984, PP9/0728, PP009B272]. Extraction for crops with high water content is changed and an HPLC-UV detection method is introduced as alternative for GC-MS. An internal standard (4-hydroxyphenoxy) acetic acid is added to each sample prior to extraction. Samples (50 g) were extracted by maceration with acetonitrile/water (50:50, v/v). An aliquot of the extract is evaporated to near dryness and refluxed in 6 M HCl for an unstated time to hydrolyse the despyridinyl acid (III) conjugates. The hydrolysate is then diluted with water and washed with dichloromethane to remove some crop coextractives. The remaining water phase is then partitioned with ethyl acetate to recover despyridinyl acid (III). The ethyl acetate phase is in turn partitioned with 1 M NaOH. The aqueous phase is acidified and despyridinyl acid (III) is back extracted from the aqueous phase using ethyl acetate. The extract is concentrated to dryness and despyridinyl acid (III) residues are either quantified by HPLC with electrochemical detection or GS-MS after derivatisation of the extracts. For determination by GC-MS the extracts were esterified with acetylchloride:butanol reagent for 1 hour at 100 °C. The eluate is evaporated to near dryness and treated with heptafluorobutyric anhydride for 1 hour at 70 °C to convert the butyl ester of despyridinyl acid (III) into the C_3F_7CO derivative. The extracts are analysed by GC-MS at m/z 434 (Internal standard at m/z 420).

HPLC-UV and GC-MS method Ref III was used in supervised trials on lettuce, cucumber, carrots, dry harvested bulb onions and green onions [PP009B272]. Control samples were < 0.05 mg/kg in cucumbers, lettuce, carrot roots and 0.10 mg/kg in onions, but further validation results were not reported.

Modification B of ref III was used in supervised trials on carrots [Atreya *et al*., 1984, PP9/0065, report PP009B300]. Total fluazifop and total despyridinyl acid (III) were determined in the same sample. An aliquot of the acetonitrile/water extract is evaporated to near dryness and hydrolysed for 1 hour in 6 M HCl at 60 °C to hydrolyse the fluazifop (II) conjugates and despyridinyl acid (III) conjugates. The hydrolysate is then diluted with water and fluazifop acid (II) is extracted with dichloromethane (follow up as for PPRAM 62). The remaining water phase is then partitioned with ethyl acetate to recover despyridinyl acid (III) (follow up as for modification A).

Reviewer's conclusion: Method GC-MS ref III original and modification A is considered not acceptable for the determination of total despyridinyl acid (III) in cottonseed, lettuce, cucumber, carrots, dry harvested bulb onions and green onions. Fluazifop acid (II) degrades under the hydrolysis conditions used in this analytical method and therefore levels of despyridinyl acid (III) are overestimated. The hydrolysis procedure in modification B is acceptable for fluazifop acid (II), but no validation results are available to confirm quantitative determination of despyridinyl acid (III).

NMR Method PP009B061 for determination of free CF3-pyridone (X)

NMR method PP009B061 determines free CF3-pyridone (X) in various crops. Residues are expressed CF3-pyridone (X). The reported LOQ is 0.02–0.05 mg/kg depending on the matrix.

NMR Method PP009B061 is described by [Atreya *et al*., 1981, PP9/0733, report PP009B061]. Crop samples (soya seeds, cotton seeds) are soaked for at least 2 hours in water. Samples are extracted by homogenisation with acetonitrile. After filtration, the extract is evaporated to dryness and the residue is redissolved in 50% deutero-acetone/acetone with PP199 as internal standard (= 2-chloro-4',6'-dinitro 2'5 di (trifluoromethyl) diphenylamine). CF3-pyridone (X) is then quantified by ^{19}F NMR. Samples fortified with CF3-pyridone (X) gave 80% recovery (concentration levels not stated).

Reviewer's conclusion: NMR Method PP009B061 is considered not valid for the determination of CF3-pyridone (X) in cottonseeds or soya beans, since CF3-pyridone conjugates are not included.

HPLC-UV method PR 1878

Method PR 1878 determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and it conjugates) as one single analyte (common moiety) in cucumbers and residues are expressed as fluazifop acid (II). The reported LOQ is 0.05 mg/kg.

HPLC-UV Method PR 1878 is described by [IR-4, 1984, no code, report PR1878 (NC); IR-4, 1984, no code, report PR1878 (DE) and Baron, 1987, no code, report IR-4 PR 1878]. Homogenised cucumbers (15 g) are extracted by homogenisation with acetonitrile/concentrated HCl (98:2, v/v). After filtration, the extract is evaporated until the aqueous fraction remains. The aqueous remainder is then hydrolysed with 6 M HCl (1 hour, 60 ºC) so that any fluazifop-butyl or fluazifop (II conjugates are converted to fluazifop acid (II). Samples are then diluted with water (pH < 1) and fluazifop acid (II) is partitioned into diethyl-ether. Fluazifop acid (II) is then partitioned into an aqueous 1% pH 8 sodium bicarbonate solution and after pH adjustment to pH<1, fluazifop acid (II) is partitioned into dichloromethane from the aqueous solution. The dichloromethane fraction is evaporated to dryness and redissolved into hexane. The hexane solution is cleaned-up with adsorption column chouromatography (Silica Seppak) to remove interfering co-extractives. Fluazifop acid (II) is eluted with methanol. Total fluazifop is quantified by reversed phase HPLC-UV at 230 nm using external standard calibration.

A method validation report for HPLC-UV detection is available for cucumbers [IR-4, 1984, no code, report PR 1878 (NC)]. Results are summarized in Table 106.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-UV Method PR 1878 is considered valid for the determination of total total fluazifop in cucumber (0.7-1.0 mg/kg). The reported LOQ is 0.05 mg/kg (but this level was not validated).

Table 106 Validation results for total fluazifop using HPLC-UV method PR 1878

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
cucumber	0.05	0.68 (F) 1.37 (F)	75 96	78-81 94, 99	7.8% -	4 2	no data	external std linear R2=0.994	no code; PR1878 (NC)
cucumber	0.05	1.03 (F)	92	82-103	11.5%	3	no data	external std linearity R^2=0.994	No code; PR 1878 (DE)]

NMR Method TMU3418B for determination of total fluazifop and total CF3-pyridone

Method TMU 3418B determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates as one single analyte) as well as total CF3-pyridone (CF3-pyridone (X) and its conjugates as one single analyte) in celery commodities and residues are expressed as fluazifop acid (II) or CF3-pyridone (X). The reported LOQ is 0.04 mg/kg for each analyte.

NMR Method TMU3418 is described by [Watford and Francis, 1988, PP5/0323**,** TMU3418/B]. Celery samples were fortified with an unstated internal standard, and extracted by homogenization with acetonitrile:concHCl solution (98:2). The extracts were evaporated to aqueous volume and hydrolyzed with 6 M HCl at 60°C for an unstated time period. The samples were then diluted with distilled water and partitioned into diethyl ether. The diethyl ether extract was dried over anhydrous sodium sulphate and then evaporated to dryness by rotary evaporation. The dried residuum was re-dissolved in deuterated acetone. The final determination of fluazifop acid (II) and CF3-pyridone (X) was by ^{19}F-NMR spectroscopy operating at 282 MHz with a dedicated ^{1}H/^{19}F probe.

Validation results obtained from supervised residue trials are presented in Table 107.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: NMR Method TMU3418B is considered not valid for the determination of total CF3-pyridone (no radiovalidation) and valid for the determination of total fluazifop in celery (0.5 mg/kg only). The valid LOQ is 0.05 mg/kg (no validations below this level).

Table 107 Validation results for total fluazifop and total CF3-pyridone (X) using NMR method TMU3418B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
celery	0.04	0.05 (F) 0.5 (F) 0.05 (X) 0.1 (X) 0.5 (IS)	80 90 62 58 80	71-88 89-91 52-73 52-64 67-92	- - - - 10%	2 2 2 2 15	< 0.04 (11) for F < 0.04 (11) for X	no data	PP5/0323; TMU3418B

GC-MS method PP009B152

Method PP009B152 determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) in oilseed commodities and residues are expressed as fluazifop acid (II). The reported LOQ is 0.1 mg/kg.

A summary description is available in [Atreya *et al*, 1982, PP9/0700, PP009B152]. Depending on the samples two different approaches were used:

- Oilseed soapstock, crude and refined oil samples were hydrolysed by boiling under reflux with 0.2 M NaOH in methanol for 1 hour.
- Oilseed whole-seed, cake, meal and hull samples were soaked with 1M hydrochloric acid overnight prior to extraction with acetonitrile. The extracts were evaporated to remove acetonitrile and leave a small aqueous volume which was neutralised with sodium hydroxide. Samples were then hydrolysed by boiling under reflux with a solution of 0.2 M NaOH in methanol for 1 hour.

Hydrolysates were diluted with water and pH adjusted to 1. Fluazifop acid (II) was then extracted by partition with diethyl-ether. After subsequent partition and coagulation clean-up procedures fluazifop acid (II) was derivatised to its methyl ester with diazomethane. Total fluazifop was then quantitatively determined (as methyl-ester) by GC-MS operated in the selective ion monitoring (SIM) mode.

GC-MS Method PP009B152 was used in processing studies on soya beans [PP009B152]. Validation results are shown in Table 108.

No radiovalidation was conducted.

Reviewer's conclusion: Method GC-MS PP009B152 is considered

- Insufficiently validated for the determination of fluazifop-butyl and fluazifop acid (II) in cotton seed and cottonseed commodities.
- not valid for the determination of total fluazifop in various soya commodities (low recovery in dry beans; high control values in dry bean seeds and oil).
- Not valid for the determination of fluazifop (II) conjugates (no radiovalidation).
- The valid LOQ is 0.1 mg/kg (no validations below this level)

Table 108 Validation results for method PP009B152

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
dry soya bean seeds	0.1	1.0 (Fb) 1.0 (F)	89 50	- -	- -	1 1	0.21	no data	PP9/0700; PP009B152
soya bean hulls	0.1	0.1 (Fb) 0.1 (F)	78 93	- -	- -	1 1	0.04	no data	PP9/0700; PP009B152
soya bean meal	0.1	1.0 (Fb) 1.0 (F)	98 92	- -	- -	1 1	0.08	no data	PP9/0700; PP009B152
soya bean crude oil	0.1	0.5 (Fb) 0.5 (F)	134 99	- -	- -	1 1	0.25	no data	PP9/0700; PP009B152
soya cake	0.1	1.0 (Fb) 1.0 (F)	110 102	- -	- -	1 1	0.30	no data	PP9/0700; PP009B152
soya refined oil	0.1	0.5 (Fb) 0.5 (F)	110 103	- -	- -	1 1	0.25	no data	PP9/0700; PP009B152
soya soapstock	0.1	0.1 (Fb) 0.5 (F)	78 72	- -	- -	1 1	0.22	no data	PP9/0700; PP009B152
cotton whole seeds	0.1	0.1 (Fb) 1.0 (F)	72 86	- -	- -	1 1	0.02	no data	PP9/0700; PP009B152
cotton hulls	0.1	0.1 (Fb) 0.1 (F)	138 142	- -	- -	1 1	0.07	no data	PP9/0700; PP009B152
cotton meal	0.1	1.0 (Fb) 1.0 (F)	101 98	- -	- -	1 1	< 0.02	no data	PP9/0700; PP009B152
cotton crude oil	0.1	0.2 (Fb) 0.2 (F)	72 68	- -	- -	1 1	0.03	no data	PP9/0700; PP009B152
cotton cake	0.1	0.1 (Fb) 1.0 (F)	88 95	- -	- -	1 1	0.05	no data	PP9/0700; PP009B152
cotton refined oil	0.1	0.2 (Fb) 0.2 (F)	105 79	- -	- -	1 1	0.02	no data	PP9/0700; PP009B152

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
cotton soapstock	0.1	0.1 (Fb)	117	-	-	1	0.04	no data	PP9/0700; PP009B152

HPLC-UV and GC-MS Method PPRAM 62 and its modifications

Method PPRAM and its modifications determine total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) in various crops and residues are expressed as fluazifop acid (II). The reported LOQ is 0.01–0.1 mg/kg depending on the level of co-extractives in different crops.

HPLC-UV Method PPRAM 62 (24 August 1981) is described by [Atreya *et al*., 1981, PP5/0606, report PPRAM62]. Homogenised wet crops (e.g. roots, tubers, vegetables, fruits, 25 g) are extracted by homogenisation with acetonitrile/concentrated HCl (98:2, v/v). Dry or oily crops (e.g. cereal grains, oilseeds, pulses, coffee beans, 25 g) are soaked in water overnight or 1 M HCl for 2 hours prior to extraction of residues by homogenisation with acetonitrile/concentrated HCl (98:2, v/v) or acetonitrile, respectively. After filtration, an aliquot of the extract is evaporated until the aqueous fraction remains. The aqueous remainder is then hydrolysed with 6 M HCl (1 hour, 60 ºC) so that any fluazifop-butyl or fluazifop (II) conjugates are converted to fluazifop acid (II). Samples are then diluted with water (pH < 1) and fluazifop acid (II) is partitioned into diethyl-ether. The diethyl ether extract is concentrated to dryness and redissolved in acetone. A coagulation solution and celite is added to remove some of the proteins, fats and oils. After this, the pH is adjusted to pH < 1 and fluazifop acid (II) is partitioned into dichloromethane from the aqueous solution, then partitioned into an aqueous 1% sodium bicarbonate solution from the dichloromethane fraction and after pH adjustment to pH<1, fluazifop acid (II) is partitioned into dichloromethane from the aqueous solution. The dichloromethane fraction is evaporated to dryness and redissolved into chloroform. The chloroform solution is cleaned-up with adsorption column chouromatography (Fractosil, silica) to remove interfering co-extractives. Fluazifop acid (II) is eluted with 30% methanol in chloroform, concentrated to dryness and redissolved in HPLC mobile phase. Total fluazifop is quantified by reversed phase HPLC-UV at 270 nm using external standard calibration. In some cases (e.g. apples, grapes) quantification was conducted at 230 nm.

GC-MS is used for the quantitative confirmation of residues of derivatised fluazifop. Derivatisation of fluazifop acid (II) to its methyl ester is done by methylation with diazomethane. The chloroform solution from the extraction procedure described above is evaporated to dryness and diazomethane is added to react for 30 min. The solution is evaporated to dryness and the residuum is redissolved in hexane for GC-MS determination.

A method validation report for HPLC-UV and GC-MS detection is available for cotton seed and soya beans [Atreya and Collis, 1982, PP9/0697, report PP009B151]. Results are summarized in Table 110.

HPLC-UV Method PPRAM 62 was used in supervised trials on oranges [PP009/B117], apples [PP009B120], pears [PP009B127], peaches [PP009B132], grapes [PP009B139], onions [TMU1815/B], dry pea seeds [PP009B070], carrots [TMU1231/B], rhubarb [IR-4 PR 2404 (1987)], asparagus [IR-4 PR 3944], cotton seed [PP009B035, TMU0987/B, TMU1027/B], coffee [PP009B122], sugarbeets [RJ0226B (no validations)], a storage stability study on strawberries, cauliflower, green beans with pods, sugarbeet roots and oilseed rape seeds [PP009B157, TMU1257/B], a storage stability study on celery [TMU3074] and processing studies on sugarbeets [PP009B089] and potatoes [PP009B153]. Concurrent method validation results extracted from supervised trial reports are summarized in Table 110.

HPLC-UV method PPRAM 53/1, a modification of method PPRAM 53 including a hydrolysis step, is identical to PPRAM 62. This method was used in a supervised residue trial on cottonseed [PP9/0734; PP009B035], field rotational crop study [TMU0671/B] and in a soya bean processing study [PP009B040, no validation]. Validation results are described in Table 110.

Modification A of method PPRAM 62 was used on juice and syrup samples from sugarbeet [PP009B089, no validation]. Samples were diluted with water, acidified to pH 1 and extracted with dichloromethane. An aliquot of the extract is evaporated until the aqueous fraction remains and this fraction is then hydrolysed and cleaned-up as described for method PPRAM 62. No validation results are available for this modification.

Method PPRAM 62/1 (December 1982) is a modification of method PPRAM 62 and is described by [Atreya, 1982, PP5/0935, report PPRAM 62/1]. Where indicated in the table below, an internal standard is added after soaking but prior to extraction. The internal standard 2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy)butyric acid, MW=341, is a synthetic analogue of fluazifop acid (II) in which the methyl is replaced by the ethyl on the side chain. Extraction, hydrolysis and liquid-liquid partition clean-up are as described for PPRAM 62, except that only 10 g of homogenised sample is used and extraction volumes are changed. The final dichloromethane fraction is evaporated to dryness and redissolved into acetone. The acetone solution is cleaned-up with adsorption column chouromatography (VacElut, disposable silica) to remove interfering co-extractives. Fluazifop acid (II) is eluted with dichloromethane/n-hexane/acetic acid/methanol (40:60:0.5:1.5), concentrated to dryness and redissolved in HPLC mobile phase. For some crops (e.g. hops, oilseed rape, cotton, coffee beans) a further HPLC clean-up (Silica Zorbax Sil) may be necessary before analysis by HPLC-UV. Total fluazifop acid (II) is quantified using internal standard calibration.

GC-MS is used for the quantitative confirmation of fluazifop acid (II) (m/z = 341, 322, 282 and 254). Derivatisation of fluazifop acid (II) is done by methylation with diazomethane on the sample eluent from the silica adsorption column as described for PPRAM 62.

HPLC-UV method PPRAM 62/1 was used in supervised residue trials on limes [RIC1933], apples [PP009B167], pears [PP009B163], peaches [PP009B159, PP009B187], grapes [PP009B180], bananas [RIC1933], dry soya beans (seeds, fodder) [PP009B229, PP009B265, PP009B176], carrots [RIC1913], swedes [PP009B169], hazelnuts [PP009B194], and cottonseeds [TMU1401/B]. Concurrent method validation results extracted from supervised trial reports are summarized in Table 110.

Modification B of HPLC-UV method PPRAM 62/1 was used in supervised residue trials on sugarbeets [D26-EP] and sunflower seeds [H19/834/P]. A coagulation solution is added directly to the hydrolysate to remove fats and oils. After this, fluazifop acid (II) is cleaned-up by liquid liquid partition and adsorption chouromatography as described for PPRAM 62/1. Total fluazifop was quantified by HPLC-UV using external standard calibration. Concurrent method validation results extracted from supervised trial reports are summarized in Table 110.

HPLC-UV Method PPRAM 62/2 (28 March 1983) is identical to method PPRAM 62 and is described by [Atreya *et al*, 1983, PP5/1047, report PPRAM62/2].

HPLC-UV and GC-MS Method PPRAM 62 and 62/2 has been published in the Pesticide Analytical Manual, Volume II (PAM II) and has been tested on broccoli, spinach, carrots, potatoes, cottonseed, oilseed rape seed and oilseed rape cake [Bussey, 1990, 407594, report RR 90-098B]. Validation results are shown in Table 110.

HPLC-UV Method PPRAM 62/2 was used in supervised residue trials of apples [TMU3119/B], cherries [TMU3181/B], peaches [TMU3168/B], blackberries [M4779B], raspberries [M3847B, M4779B], bilberries [M4779B], black currants [M3870B, M4197B], gooseberries [M3869B, M4186B], grapes [TMU3144/B; TMU3330/B], strawberries [M4883B], bananas [M4388B; RIC1934], onions [M3872B, M3975B, M4205B], head cabbage [M3681B], green peas (seeds, pods, forage) [M3754B; M3976B, M4008B], dry peas (seeds, fodder) [M3754B; M3759B; M3724B, M4209B (no validation)], dry soya beans [M4010B], carrots [M3954B], potatoes [M3676B; M3694B, M3977B], swede (roots, tops) [M4001B, M4204B, M4052B], sugar beet (roots, tops) [M3701B], fodderbeet (roots, tops) [M3701B], witloof roots [M3690B, M4058B], macadamia nut [PR 3431], oilseed rape [M3685B]. For dry beans [TMU3094B, RR 89-046B], the method was slightly modified by using acetonitrile/water (35:65, v/v, pH 3) mobile phase and detection at 230 nm.

Concurrent method validation results extracted from supervised trial reports are summarized in Table 110.

HPLC-UV Method PPRAM 62/2 with the additional clean-up step by HPLC was used in supervised trials on apples [AZ8466A/91], cherries [AZ83558/91], plums [AZ83558/91], coffee beans [PR 03432 (1988)], a storage stability study on tomatoes [TMU3079] and processing studies on Brussels sprouts, cauliflower, head cabbage and kale [AZ83592/91]. Concurrent method validation results extracted from supervised trial reports are summarized in Table 110.

Modification C of method PPRAM 62/2 (9 November, 2011) was used in a residue trial on coffee [Barney, 2011; PP5_50291, report PR 03432 (2011)]. Coffee samples were soaked in water or 1 M HCl for a minium of 2 hours. Internal standard is added (optional) and extraction, hydrolysis, liquid-liquid partition, clean-up by adsorption chouromatography and additional HPLC clean-up are as described for PPRAM 62/1. Alternate mobile phase and gradient programming was required to maximize separation of fluazifop acid (II) and co-extractives. Concurrent method validation results extracted from supervised trial reports are summarized in Table 110.

Method PPRAM 62/2 was radiovalidated using radiolabelled carrot and endive metabolism samples [Lin, 2009, PP005_50017, report T009022-08]. In the metabolism studies, carrots and endive were treated with phenyl labelled ^{14}C-fluazifop-P-butyl (R-enantiomer) at 2 × 0.42 kg ai/ha. Control samples spiked with fluazifop acid (II) at 0.01 and 1 mg/kg were analysed concurrently with the radiolabelled samples for each of the matrices. The radiolabelled samples were analysed using a simplified version of method PPRAM 62/2. Samples were extracted with acetonitrile/concentrated HCl (98/2, vv) without soaking followed by hydrolysis with 6 M HCl for 1 hour at 60 °C. The samples were purified using an Oasis HLB SPE cartridge. The analyte was eluted with 0.1% formic acid/acetonitrile (60/40, v/v) and then analysed by HPLC-MS/MS at m/z 328 and 254. Concurrent recoveries ranged between 91–104% (n=1 per level and sample) and control samples were < 0.01 mg/kg. The residues in the radiolabelled samples found by method PPRAM62/2 agreed well with the sum of free fluazifop acid (II) and its conjugates reported in the metabolism studies (see Table 109). The accountabilities of the method ranged from 69% for endive and 99% for carrot roots, demonstrating that the conjugates can be converted to free fluazifop acid (II) under the reflux conditions specified in the method.

Reviewer's conclusion: HPLC-UV and GC-MS method PPRAM62 and its modifications are considered:

- valid (full validation) for determination of fluazifop acid (II) by HPLC-UV in oilseed rape forage (at 1.0–10 mg/kg),
- valid (reduced validation) for the determination of fluazifop acid (II) by HPLC-UV in apples (at 0.5 mg/kg only), pears (at 0.5 mg/kg only), cherries (at 0.5 mg/kg only), peaches (at 0.5 mg/kg only), grapes (0.05–0.5 mg/kg), bulb onions (at 0.05 mg/kg only), broccoli (0.05 mg/kg only); dry bean seeds (0.05–0.2 mg/kg); dry pea seeds (0.5–1.0 mg/kg), dry pea haulms (0.5-1.0 mg/kg); dry soya bean seeds (0.05–0.5 mg/kg); carrots (0.6 mg/kg only), potato tubers (0.1–1.0 mg/kg), boiled potatoes (1.0 mg/kg only), potato peels (1.0 mg/kg only), asparagus (0.1 mg/kg), sorghum forage (0.1–0.5 mg/kg), sorghum fodder (0.1 mg/kg only); macadamia nutmeat (at 0.5 mg/kg only), cotton seeds (0.1–0.5 mg/kg), oilseed rape seeds (at 1–2 mg/kg), coffee green beans (0.05–2.0 mg/kg), coffee roasted beans (at 0.05 mg/kg only) and coffee freeze dried (at 0.05 mg/kg only).
- insufficiently validated for the determination of fluazifop acid (II) by HPLC-UV in orange flesh, orange peel, lime, black currants, gooseberries, raspberries, blackberries, bilberries, strawberries, banana, cabbage, cabbage cooked, Brussels sprouts, cauliflower, tomatoes, kale, kale cooked, kale canned, spinach, green beans with pods, green pea seeds, green pea pods, green pea forage, carrots, fodder beet roots, fodderbeet tops, sugarbeet roots, sugarbeet tops, sugarbeet sugar, swede roots, swede tops, sweet potato roots, sweet potato vines, turnip tops, turnip roots, witloof roots, celery, rhubarb, maize ears, maize forage, sorghum grains, wheat forage, hazelnuts, cotton gin trash, oilseed rape seeds, peanut kernels, sunflower seeds.

- valid (reduced validation) for the determination of fluazifop acid (II) by GC-MS in soya bean seeds (at 0.2 mg/kg only).
- insufficiently validated for the determination of fluazifop acid (II) by GC-MS in cottonseeds, oilseed rape seeds and oilseed rape cake
- valid for the HPLC-UV and GC-MS determination of fluazifop-butyl (verified in dry soya bean seeds, sugarbeet tops, sorghum fodder stalks, cotton seeds, sunflower seeds)
- valid for the HPLC-UV or GC-MS determination of fluazifop acid (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).

The valid LOQ for HPLC-UV method PPRAM 62, 62/1 and 62/2 is 0.05 mg/kg (no validations below this point).

Table 109 Radiovalidation results for method PPRAM 62/2

Matrix (sample ID)	Description	Metabolism study [a] 6 M HCl overnight at room temp	PPRAM 62/2 6 M HCl 1 hour 60 °C	RSD (%)	n	Trueness (Method/^{14}C)	Code no; Report no
Mature carrot roots (1689W-020)	^{14}C phenyl; 2 × 0.42 kg ai/ha; DAT 45	TRR = 0.091 mg/kg Fb eq; Fluazifop acid (II, free + conj): 0.058 mg/kg Fb eq	Total fluazifop, mg/kg F [a] mean 0.049 range 0.048-0.050	2.0%	3	99% (as F)	PP005_50017; T009022-08
Mature endive foliage (1690W-022)	^{14}C phenyl; 2 × 0.42 kg ai/ha; DAT 28	TRR = 1.44 mg/kg Fb eq; Fluazifop acid (II, free + conj): 0.71 mg/kg Fb eq	Total fluazifop, mg/kg F [a] mean 0.42 range 0.39-0.46	8.3%	3	69% (as F)	PP005_50017; T009022-08

[a] Residues in fluazifop-butyl equivalents (Fb eq, from metabolism studies) need to be multiplied by 0.85 to get fluazifop acid (II) equivalents, since results from method PPRAM 62/2 are expressed as fluazifop.

Table 110 Validation results for HPLC-UV method PPRAM 62 and its modifications

Matrix	LOQ	Forti fication level (mg/kg)	Recovery (%) Mean range		RSD (%)	n	control mg/kg	calibration	Code no; Report no
PPRAM 53/1 – HPLC-UV detection (PPRAM 53 including hydrolysis step = PPRAM 62)									
broccoli	0.05	0.5 (F)	83	82-84	1.2%	3	ns	no data	PP9/0176; TMU0671/B (rotational)
sugar beet roots	0.02	0.1 (F)	97	97-97	-	2	ns	no data	PP9/0176; TMU0671/B (rotational)
sugar beet tops	0.05	0.2 (F) 0.5 (F) 0.5 (Fb)	79 70 80	75-83 - -	- - -	2 1 1	ns	no data	PP9/0176; TMU0671/B (rotational)
turnip tops	0.02	0.2 (F)	84	81-87	-	2	ns	no data	PP9/0176; TMU0671/B (rotational)
turnip roots	0.02	0.2 (F)	93	-	-	1	ns	no data	PP9/0176; TMU0671/B (rotational)
sweet potato vine	0.05	0.1 (F) 0.5 (F)	69 82	- -	- -	1 1	ns	no data	PP9/0176; TMU0671/B (rotational)
sweet potato roots	0.05	0.1 (F) 0.5 (F)	78 84	- -	- -	1 1	ns	no data	PP9/0176; TMU0671/B (rotational)
wheat forage	0.05	0.1 (F) 0.5 (F)	66 64	- -	- -	1 1	ns	no data	PP9/0176; TMU0671/B (rotational)

Matrix	LOQ	Forti fication level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
maize ears	0.05	0.1 (F) 0.5 (F)	101 79	89-113 -	- -	2 1	ns	no data	PP9/0176; TMU0671/B (rotational)
maize forage	0.05	0.5 (F)	71	-	-	1	ns	no data	PP9/0176; TMU0671/B (rotational)
sorghum grains	0.05	0.1 (F) 0.5 (F)	84 78	- 78-78	- -	1 2	ns	no data	PP9/0176; TMU0671/B (rotational)
sorghum forage	0.05	0.1 (F) 0.5 (F)	81 55	57-110 47-60-	29% 13%	5 3	ns	no data	PP9/0176; TMU0671/B (rotational)
sorghum fodder (stalks)	0.05	0.1 (F) 0.1 (Fb)	86 96	71-108 -	21% -	4 1	ns	no data	PP9/0176; TMU0671/B (rotational)
cotton gin trash	0.05	0.5 (F)	64	61-68	-	2	ns	no data	PP9/0176; TMU0671/B (rotational)
cotton seeds	0.05	0.5 (F)	73	71-76	3.6%	3	ns	no data	PP9/0176; TMU0671/B (rotational)
cottonseed	0.02	0.1 (F)	99	91-109	9.1%	3	< 0.02 (4)	no data	405792; TMU0679B; PP9/0734; PP009B035
PPRAM 62 – HPLC-UV detection (270 nm or 230 nm)									
oranges flesh	0.05	2.0 (IS)	88	-	-	1	< 0.05 (2)	internal std linearity ns	PP9/0613; PP009/B117
oranges peel	0.05	2.0 (IS)	71	-	-	1	< 0.05 (2)	internal std linearity ns	PP9/0613; PP009/B117
apples	0.03	0.1 (F)	93	-	-	1	0.04 (1)	internal std linearity ns	PP9/0433; PP009B120
pears	0.03	-	-		-	-	< 0.03 (1)	internal std linearity ns	PP9/0434; PP009B127
peaches	0.05	0.1 (F)	93	-	-	1	0.05 (1)	external std linearity ns	PP9/0644; PP009B132
grapes	0.02	0.05 (F) 0.1 (F) 0.2 (F) 0.5 (F)	86 88 88 86	73-99 79-97 85-91 -	- 10% - -	2 3 2 1	< 0.02 (8)	external std linearity ns	PP9/0436; PP009B139
strawberry	0.05	0.1-5.0 (F)	86	78-95	6%	8	ns	external std linearity ns	PP9/0039; PP009B157 stor stab
bulb onion	0.03	0.03 (F) 0.05 (F) 0.1 (F) 0.12 (F) 0.2 (F) 0.6 (F) 0.8 (F)	81 81 94 88 84 90 91	- 70-91 81-108 - 84-84 90-91 -	- 11% - - - - -	1 4 2 1 2 2 1	< 0.03 (8)	no data	434142; TMU1815/B
cauliflower	0.05	0.1-5 (F)	80	71-98	8%	8	ns	external std linearity ns	PP9/0039; PP009B157 stor stab
green beans with pods	0.05	0.1-5 (F) combined	89	77-111	13%	7	ns	external std linearity ns	PP9/0039; PP009B157 stor stab
dry pea seeds	0.02	0.1–0.3 (F)	77	ns	ns	ns	< 0.02-0.03 (2)	no data	PP9/0554 PP009B070
dry soya bean seeds	ns	0.05 (Fb) 0.05 (F) 0.1 (Fb) 0.1 (F)	84 74 78 84	74-96 67-80 74-82 75-94	10% - - -	5 2 2 2	no data	no data	PP9/0697; PP009B151 validation

Matrix	LOQ	Forti fication level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
		0.2 (Fb) 0.2 (F) 0.5 (Fb)	66 72 72	64-67 62-84 62-79	- 13% 12%	2 4 3			
carrots	ns	0.04 (F) 0.05 (F) 0.12 (F) 0.16 (F) 0.6 (F)	113 94 92 92 75	- - 92-92 - 70-81	- - - - 7.3%	1 1 2 1 3	no data	no data	407594; RR 90-098B PAM II validation
carrots	0.04	0.04-0.8 (F) ns (Fb)	82% 91%	ns ns	ns ns	6 ns	< 0.04 (5)	no data	406309; TMU1231/B
potato raw flesh	0.01	1.0 (IS)	80	72-85	6.7%	7	< 0.01	internal std no data	PP9/0702; PP009B153 processing
potato cooked flesh	0.01	1.0 (IS)	85	84-100	6.0%	7	< 0.01	internal std no data	PP9/0702; PP009B153 processing
potato skins	0.01	1.0 (IS)	88	86-90	2.3%	3	< 0.01	internal std no data	PP9/0702; PP009B153 processing
sugarbeet roots	0.05	0.1-5 (F) combined	83	62-95	11%	10	ns	external std linearity ns	PP9/0039; PP009B157 stor stab
sugarbeet roots	0.01	0.1 (F) 0.5 (F) 1.0 (F)	82 91 87	- - -	- - -	1 1 1	ns	external std linearity ns	PP9/0366; PP009B089 processing
Sugarbeet roots	0.01	0.05-1.0 (F)	98	-	ns	13	ns	No data	406215 TMU1257/B
sugarbeet sugar	0.01	0.1 (F) 0.2 (F)	68 97	- -	- -	1 1	ns	external std linearity ns	PP9/0366; PP009B089 processing
asparagus	0.1	0.085 (Fb) 0.10 (F)	62 77	61-64 65-84	2.1% 11%	4 5	<0.1	no data	No code;I IR-4 PR 3944;
celery	0.03	0.5 (F)	87	-	-	1	ns	no data	PP9/0037 TMU3074; (stor stab)
rhubarb	0.1	0.4 (F)	73	-	-	1	<0.1 (4)	no data	464387; IR-4 PR 2404 (1987)
cottonseed	ns	0.1 (F) 0.2 (Fb) 0.2 (F) 0.4 (F) 0.5 (Fb)	99 84 78 82 77	91-119 82-86 - 75-90 77-78	10% - - - -	6 2 1 2 2	no data	no data	PP9/0697; PP009B151 validation
cottonseed	0.03	1.0 (F) 1.5 (F) 2.4 (F) 4.9 (F) 1.0 (Fb) 2.0 (Fb)	107 111 114 110 90 112 106 88	90-124 108- 92-125 64-116 117- 89-88	- - 15% - - -	2 2 3 2 2 2	ns	no data	405794; TMU0987/B
cottonseed	0.04	1.0 (F) 1.5 (F) 2.5 (F)	81 91 88	72-98 - 77-114	14% - 18%	4 1 5	< 0.04	no data	405795; TMU1207/B
oilseed rape seeds	0.05	0.1-5 (F) combined	85	77-90	5%	7	ns	external std linearity ns	PP9/0039; PP009B157
coffee	0.03	0.5-5.0 (F) combined	83	-	-	1	< 0.03	no data	PP9/0633 PP009B122
PPRAM 62, GC-MS confirmation									
soya bean seed	ns	0.2 (Fb) 0.2 (F) 0.5 (Fb)	96 92 93	85-109 90-95 90-96	11% - -	4 2 2	no data	no data	PP9/0697; PP009B151 validation

Matrix	LOQ	Forti fication level (mg/kg)	Recovery (%) Mean range		RSD (%)	n	control mg/kg	calibration	Code no; Report no
cottonseed	ns	0.2 (Fb) 0.2 (F) 0.4 (F) 0.5 (Fb)	100 100 86 100 88	100- - 98-102 88-89	- - - - 	2 1 2 2	no data	no data	PP9/0697; PP009B151 validation
PPRAM 62/1 – HPLC-UV detection									
lime	0.05	0.1–0.3 (F)	75	ns	12%	ns	< 0.05	external std linearity ns	PP9/0130; RIC1933
apples	0.05	0.50 (IS)	76	70-87	5%	13	< 0.05 (3)	internal std linearity ns	PP9/0432; PP009B167
pears	0.04	0.50 (IS)	84	74-95	8%	14	< 0.04 (3)	internal std linearity ns	PP9/0435; PP009B163
peaches	0.04	0.50 (IS)	85	83-110	18%	6	< 0.05 (2)	internal std linearity ns	PP9/0710; PP009B159
peaches	0.04	0.50 (IS)	79	64-88	8%	6	< 0.04 (2)	internal std linearity ns	PP9/0621; PP009B187
grapes	0.03 0.04 0.05	0.50 (IS)	76	69-84	5%	26	< 0.03 (2) < 0.04 (2) < 0.05 (4)	internal std linearity ns	PP9/0437; PP009B180
banana	0.05	0.1–0.3 (F)	85	ns	9.4%	ns	< 0.05	external std linearity ns	PP9/0130; RIC1933
dry soya beans	0.05	ns (IS)	95	ns	5%	ns	< 0.05	internal std linearity ns	PP9/0669; PP009B229
dry soya beans	0.05	0.5 (IS)	78	71-92	8.5%	14	< 0.05 (3); 0.14	internal std linearity ns	PP9/0726; PP009B265
dry soya beans dry soya fodder	0.05	ns	ns		ns	ns	0.12 (seed) 0.14 (fodder)	internal std linearity ns	PP9/0606; PP009B176
carrots	0.05	0.2-0.5 (F)	90	ns	3.3%	9	< 0.05 (5)	no data	PP9/0050; RIC1913
swedes	0.02	1.0-5.0 (F)	76	ns	ns	ns	< 0.02 (1)	no data	ASF64_10000; PP009B169
hazelnuts	0.05	0.50 (IS)	no data		-	-	< 0.05-0.05	internal std linearity ns	PP9/0628; PP009B194
cottonseeds	0.04	ns (Fb) ns (F)	94 69	ns ns	12% 8.7%	10 15	-	no data	405796; TMU1401/B
PPRAM 62/1, modification B, HPLC-UV detection									
sugarbeet roots	0.05	0.1–0.2 (F)	76	ns	7%	ns	< 0.05 (4)	external std linearity ns	PP5/0096; D26-EP
sunflower seeds	0.05	0.2 (Fb)	70	-	85	8	< 0.05 (5)	no data	PP5/0540; H19/834-P
PPRAM 62/2 HPLC-UV detection at 270 nm or 230 nm									
apples	0.03	0.05-0.1 (F) combined	96	ns	4%	ns	< 0.03 (6)	no data 230 nm	405749; TMU3119/B

Matrix	LOQ	Forti fication level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
cherries	0.03	0.5 (IS)	78	ns	8%	5	< 0.03	internal std linearity ns	PP5/0468; TMU3181
peaches	0.03	0.5 (IS)	80	ns	6%	4	< 0.03	internal std; linearity ns	PP5/0476; TMU3168/B
blackberries	0.01	0.10 (IS)	78	ns	7.7%	ns	< 0.01 (6)	internal std linearity ns	PP5/0462; M4779B
raspberries	0.03	0.50 (IS)	78	ns	3.3%	ns	< 0.03 (2)	internal std linearity ns	PP5/0488; M3847B
raspberries	0.01	0.10 (IS)	78	ns	7.7%	ns	< 0.01 (6)	internal std linearity ns	PP5/0462; M4779B
bilberries	0.01	0.10 (IS)	78	ns	7.7%	ns	< 0.01 (6)	internal std linearity ns	PP5/0462; M4779B
black currants	0.03	0.2-1.0 (IS)	73	ns	4.1%	ns	< 0.03 (3)	internal std linearity ns	PP5/0458; M3870B
black currants	0.03	0.25 (IS)	75	ns	10%	ns	< 0.03 (4)	internal std linearity ns	PP5/0457; M4197B;
gooseberries	0.03	0.2-1.0 (IS)	77	ns	3.9%	ns	< 0.03 (3)	internal std linearity ns	PP5/0473; M3869B
gooseberries	0.02	0.1 (IS)	97	ns	6.2%	ns	< 0.02 (5)	internal std linearity ns	PP5/0472; M4186B
grapes	0.03	0.5 (IS)	80	ns	10%	ns	< 0.03 (6)	internal std linearity ns 230 nm	PP5/0471; TMU3144B
grapes	0.03	0.025 (F) 0.05 (F) 0.1 (F)	88 92 85	- 82-106 78-94	- 12% 8.0%	1 4 4	< 0.03 (4)	linearity ns 230 nm	PP5/1113 TMU3330B
strawberries	0.01	0.2 (IS)	95	ns	27%	ns	< 0.01 (2)	internal std linearity ns	PP5/0194; M4883B
bananas	0.04	0.2-0.5 (IS)	83	ns	6.3%	ns	< 0.04 (6)	internal std; linearity ns	PP5/0185; M4388B
bananas	0.02	0.5 (F)	80	ns	ns	ns	< 0.02 (1)	no data	PP5/0184; RIC1934
bulb onions	0.03	0.5-1.0 (IS)	80	ns	8%	ns	< 0.03 (1)	internal std linearity ns	PP5/0088; M3872B
bulb onions	0.02	0.5 (IS)	83	ns	5%	ns	< 0.02 (4)	internal std linearity ns	PP5/0089; M3975B
bulb onions	0.03	0.1 (IS)	79	ns	13%	ns	< 0.03 (2)	internal std linearity ns	PP5/0090; M4205B
broccoli	ns	0.12 (F)	84	-	-	1	no data	no data	407594; RR 90-098B PAM II validation
head cabbage	0.02	0.1 (IS) 1.0 (IS) 2.0 (IS) 5.0 (IS)	81 81 80 86	76-87 75-83 79-81 84-87	4.6% 4.8% - -	12 4 2 2	< 0.02 (4)	no data	PP9/0057; M3681B
spinach	ns	0.15 (F)	77	-	-	1	no data	no data	407594; RR 90-098B PAM II validation
green pea seeds	0.03	0.5 (IS)	64	ns	10%	ns	< 0.03 (1)	internal std linearity ns	PP5/0412; M3976B
green pea seeds	0.03	0.5-1.0 (IS)	78	ns	4.7%	ns	< 0.03 (1)	internal std linearity ns	PP5/0397; M4008B
green pea seeds	0.02	1.0 (IS)	76	58-86	11%	13	< 0.02 (1); < 0.05 (1)	internal std linearity ns	PP9/0116; M3754B
green pea pods	0.02	1.0 (IS)	82	78-86	-	2	ns	internal std linearity ns	PP9/0116; M3754B
green pea forage	0.03	0.5-1.0 (IS)	72	ns	8.9%	ns	0.08 (1)	internal std linearity ns	PP5/0397 M4008B
dry bean seeds	0.03	0.05 (F) 0.1 (F)	77 77	69-82 75-82	9.4% 5.2%	3 3	< 0.03 (4)	no data	PP5/0378; TMU3094/B

Matrix	LOQ	Forti fication level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
		0.2 (F)	69	64-74	7.3%	3			
dry bean seeds	0.03	0.05 (F) 0.1 (F) 0.2 (F) 1.0 (F) 3.0 (F)	83 80 78 85 73	69-93 75-85 64-104 - -	9.0% 5.2% 20% - -	7 6 5 1 1	< 0.03 (7); < 0.05 (3)	no data	405660; RR 89-046B
dry pea seeds, dry pea pods dry pea fodder	0.02- 0.04	0.5 (IS) 1.0 (IS)	81 76	72-85 64-90	7.6% 12%	4 8	< 0.02- 0.05 (seeds) 0.17-0.28 (pods) < 0.04 (fodder) n=2, each	no data	PP9/0119; M3724B
dry pea seeds	0.05	1.0 (IS)	73	61-86	14%	4	< 0.05 (1)	internal std linearity ns	PP9/0116; M3754B
dry pea seeds	0.05	0.5 (IS) 1.0 (IS)	74 61	67-80 55-71	6.2% 9.0%	8 8	< 0.05 (1)	no data	PP9/0117; M3759B
dry pea straw	0.05	0.5 (IS) 1.0 (IS)	84 80	78-90 76-86	5.8% 4.3%	6 6	< 0.05 (1)	no data	PP9/0117; M3759B
dry soya bean seeds	0.04	0.25 (IS)	77	ns	5.4%	ns	< 0.01 (1)	internal std	PP5/0408; M4010B
carrots	0.03	0.5 (IS)	77	ns	9.9%	ns	< 0.03 (5)	internal std linearity ns	PP5/0084; M3954B,
potato tubers	ns	0.1 (F) 0.4-0.6 (F)	79 72	59-110 44-82	ns ns	10 4	no data	no data	407594; RR 90-098B PAM II validation
potato tubers	0.04	0.5 (IS)	82	57-106	12%	24	< 0.04 (8); 0.04 (1)	internal std linearity ns	PP9/0052; M3676B
potato tubers	0.03	1.0 (IS) 2.0 (IS)	97 83	83-121 81-86	12% 2.6%	13 4	< 0.03 (12); 0.03 (1); 0.05 (1)	internal std linearity ns	PP5/0092; M3694B
potato tubers	0.03	0.5 (IS)	83	ns	7%	ns	< 0.03 (8)	internal std linearity ns	PP5/0094; M3977B
swede roots	0.03	0.5 (IS)	76	ns	9.1%	ns	< 0.03 (5)	internal std linearity ns	PP5/0100; M4001B
swede roots	0.02	0.5 (IS)	85	ns	ns	ns	< 0.02	internal std linearity ns	PP5/0273; M4052B
swede roots	0.02	0.2 (IS)	86	ns	ns	ns	< 0.02 (1)	internal std linearity ns	PP5/0272; M4204B
swede tops	0.03	0.5 (IS)	77	ns	4.4%	ns	< 0.03 (5)	internal std linearity ns	PP5/0100; M4001B
sugarbeet tops	0.05	0.5 (IS) 1.0 (IS) 2.0 (IS) 5.0 (IS) 10 (IS)	76 78 76 90 86	61-92 76-82 70-80 89-91 80-90	9.0% 3.5% 5.7% 1.1% 5.6%	24 4 4 4 4	< 0.05 (4)	internal std linearity ns	PP9/0054; M3701B
fodderbeet tops	0.05	0.5 (IS) 1.0 (IS) 2.0 (IS) 5.0 (IS) 10 (IS)	76 78 76 90 86	61-92 76-82 70-80 89-91 80-90	9.0% 3.5% 5.7% 1.1% 5.6%	24 4 4 4 4	< 0.05 (4)	internal std linearity ns	PP9/0054; M3701B
sugarbeet roots	0.02	0.5 (IS)	83	37-96	11%	24	< 0.02 (4)	internal std linearity ns	PP9/0054; M3701B
fodderbeet roots	0.02	0.5 (IS)	83	37-96	11%	24	< 0.02 (4)	internal std linearity ns	PP9/0054; M3701B
witloof roots	0.02	0.5 (IS)	81	66-85	6.6%	12	< 0.02 (1)	internal std; linearity ns	PP9/0071; M3690B
witloof roots	0.02	0.5 (S)	85	ns	4.3%	ns	< 0.02 (2)	internal std; linearity ns	PP9/0089; M4058B

Matrix	LOQ	Forti fication level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
macadamia, nutmeat	0.1	0.1 (F) 0.2 (F) 0.25 (F) 0.5 (F)	61 90 68 71	42-86 90 64-72 62-94	29% - 8.3% 18%	5 1 2 5	<0.1	external std linearity ns	464386; PR 3431
cottonseed	ns	0.05 (F) 0.1 (F)	94 87	92-96 86-88	- -	2 2	no data	no data	407594; RR 90-098B PAM II validation
oilseed rape seeds	0.05	1 (IS) 2 (IS)	75 72	71-83 70-73	7.2% 2.0%	4 4	< 0.05 (4)	internal std	PP9/0399; M3685B
oilseed rape forage	0.05	0.5 (IS) 1.0 (IS) 2.0 (IS) 5.0 (IS) 10 (IS)	66 76 84 72 71	63-70 70-85 78-89 65-79 65-82	4.5% 7.1% 3.6% 8.3% 11%	4 9 12 4 4	< 0.05 (4)	internal std	PP9/0399; M3685B
PPRAM 62/2 GC/MS confirmation									
oilseed rape seed	ns	0.5 (F)	78	-	-	1	no data	no data	407594; RR 90-098B PAM II validation
oilseed rape cake	ns	0.5 (F)	96	-	-	1	no data	no data	407594; RR 90-098B PAM II validation
PPRAM 62/2 with additional HPLC-cleanup and HPLC-UV detection (270 nm or 230 nm)									
apples	0.02	0.02 (F) 0.2 (F)	110 102	- -	- -	1 1	< 0.02 (2)	external std linearity ns	PP5/0813; AZ8466A/91
cherries	0.02	0.02 (F) 0.2 (F)	116 86	- -	- -	1 1	< 0.02 (1)	external std linearity ns	PP5/0192; AZ83558/91
plums	0.02	0.02 (F) 0.2 (F)	117 111	- -	- -	1 1	< 0.02 (1)	external std linearity ns	PP5/0192; AZ83558/91
Brussels sprouts	0.05	0.05 (F) 0.50 (F) 5.0 (F)	74 107 114	- - -	- - -	1 1 1	< 0.05 (1)	external std linearity ns	PP5/0129; AZ83592/91
Brussels sprouts cooked	0.05	0.05 (F)	98	-	-	1	< 0.05 (1)	external std linearity ns	PP5/0129; AZ83592/91 processing
cauliflower	0.05	0.05 (F) 0.50 (F)	78 87	- -	- -	1 1	< 0.05 (1)	external std linearity ns	PP5/0129; AZ83592/91 processing
head cabbage	0.05	0.05 (F) 0.50 (F) 1.0 (F)	112 71 92	- - -	- - -	1 1 1	0.06 (1)	external std linearity ns	PP5/0129; AZ83592/91 processing
head cabbage cooked	0.05	0.05 (F)	88	-	-	1	< 0.05 (1)	external std linearity ns	PP5/0129; AZ83592/91 processing
tomatoes	0.03	0.3 (F)	82	-	-	1	-	external std linearity ns	PP9/0036; TMU3079 stor stab
kale	0.05	0.05 (F) 0.5 (F) 1.0 (F)	114 70 75	- - -	- - -	1 1 1	< 0.05 (1)	external std linearity ns	PP5/0129; AZ83592/91 processing
kale cooked	0.05	0.05 (F)	114	-	-	1	< 0.05 (1)	external std linearity ns	PP5/0129; AZ83592/91 processing
kale canned	0.05	0.05 (F)	80	-	-	1	< 0.05 (1)	external std linearity ns	PP5/0129; AZ83592/91 processing
coffee green beans		0.1 (F) 0.5 (F)	62 70	56-67 56-80	- 15%	2 4	<0.1 (4)	external std; 0.5-10 ug/L; linear by graph;	471695; PR 03432 (1988)

Matrix	LOQ	Forti fication level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
Modification C of HPLC-UV method PPRAM 62/2 with additional HPLC clean-up									
coffee green beans	0.05	0.05 (F) 0.2 (F) 2.0 (F)	81 83 81	66-94 79-85 80-81	17 4.2 0.7	6 3 3	< 0.05	external std linear, 0.5-2.0 mg/kg	PP5/50291; PR 03432 (2011)
coffee, roasted	0.05	0.05 (F) 0.2 (F) 2.0 (F)	82 67 63	76-90 63-70 59-67	6.5 5.3 6.4	6 3 3	< 0.05	external std linearity ns	PP5/50291; PR 03432 (2011)
coffee, freeze dried	0.05	0.05 (F) 0.2 (F) 2.0 (F)	77 67 74	75-81 66-68 69-75	2.1 1.0 5.7	6 3 3	< 0.05	external std linearity ns	PP5/50291; PR 03432 (2011)

HPLC-UV Method Yokomizo and Carvalho

HPLC-UV method Yokomizo and Carvalho determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) in seeds, grains, roots, tubers, vegetables and fruits and residues are expressed as fluazifop. The reported LOQ was 0.01 mg/kg.

An English summary of this method was provided [Yokomizo and Carvalho, 1984, no code available, report AM0006]. Homogenised dry seed crop commodities (25 g) are soaked for at least 2 hours (or overnight) in 1 M HCl and then acetonitrile is added (1 M HCl/acetonitrile, 50:100 v/v). Homogenised wet crop commodities (25 g) are directly mixed with acetonitrile/concentrated HCl (98:2, v/v). After homogenisation of the sample-extract mixtures, the samples are filtered and an aliquot of the extract (equivalent to 10 g sample) is concentrated until all the acetonitrile has evaporated. The aqueous remainder is dissolved in 6 M HCl. Fluazifop-butyl and fluazifop (II) conjugates are converted to fluazifop acid (II) during a 1 hour hydrolysis with 6 M HCl at 60 °C. Fluazifop acid (II) is then partitioned into diethylether. The diethylether phase is evaporated to dryness, redissolved in acetone and coagulation solution (6.25 g/L ammonium chloride and 1% v/v phosporic acid) with Celite 545 (10:50, v/v), mixed and left for 10 min. The solution is filtered, whereby coagulated proteins, oils and other matrix interferences are removed. The pH of the filtrate is adjusted to pH<1 by adding concentrated HCl and fluazifop acid (II) is then partitioned into dichloromethane. The dichloromethane extract is extracted with 1% sodium bicarbonate solution. The aqueous solution is kept and acidified to pH < 1 with concentrated HCl. Fluazifop acid (II) is then extracted with dichloromethane. The dichloromethane phase is evaporated to dryness and redissolved in chloroform or acetone, depending on the clean-up system. The chloroform solution is cleaned-up by Fractosil 200 adsorption chouromatography and fluazifop acid (II) is eluted with 30% methanol in chloroform. Alternatively the acetone solution is cleaned-up by a Bond-Elut silica column and fluazifop acid (II) is eluted with dichloromethane/hexane/acetic acid/methanol (40:60:0.5:1.5, v/v/v/v). The eluate is concentrated to dryness, redissolved in acetonitrile/water (50:50) and analysed by HPLC-UV at 270 nm.

A method validation report is available for pears, dry soya beans, cotton seeds, carrot roots, beet roots [Yokomizo and Carvalho, 1984, no code, report AM0006]. Results are summarized in Table 111.

HPLC-UV Method Yokomizo and Carvalho is used in supervised trials on dry beans [TECPAR 81975/92, TECPAR 81980/92, TECPAR 81981/92, TECPAR 83030/92, which all have identical validations] and soya beans [TECPAR 81976/92, TECPAR 81978/92, TECPAR 81979/92, which all have identical validations]. Concurrent method validation results extracted from supervised trial reports are summarized in Table 111.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-UV method Yokomizo and Carvalho is considered:

- insufficiently validated for the determination of total fluazifop in pears, dry soya beans, cotton seeds, carrot roots and beet roots.
- valid for the determination of fluazifop acid (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).

The valid LOQ is 0.08 mg/kg (no validations below this point).

Table 111 Validation results for method Yokomizo and Carvalho

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
pears	0.01	0.08[a]	85	-	ns	ns	-	no data	no code; AM0006; validation
dry beans	0.01	ns	75		ns	ns	< 0.01 (1)	no data	PP5/1028; TECPAR 81975/92
dry soya beans	0.01	0.08 [a]	95	-	ns	ns	-	no data	no code; AM0006; validation
dry soya bean seeds	0.01	ns	78		ns	ns	< 0.01 (1)	no data	PP5/0411; TECPAR 81976/92;
cotton seeds	0.01	0.08 [a]	92	-	ns	ns	-	no data	no code; AM0006; validation
carrot roots	0.01	0.08 [a]	78	-	ns	ns	-	no data	no code; AM0006; validation
beet roots	0.01	0.08 [a]	83	-	ns	ns	-	no data	no code; AM0006; validation

[a] 2 ųg added (assumption added to 25 g homogenised crop commodity; 2 ug/25 g = 0.08 mg/kg)

19F-NMR Method PPRAM 83

19F-NMR method PPRAM 83 determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) and residues are expressed as fluazifop. The reported LOQ was 0.01–0.05 mg/kg, depending on the number of coextractives in the sample.

Method PPRAM 83 (16 January 1984) was described by [Harradine and Atreya, 1984, no code, report PPRAM 83; Bussey, 1990, 407595, report RR 90-103B]. Dry or oily samples, including green pea commodities (10 g) are soaked in water or 1 M HCl for a minimum of 2 hours, prior to fortification with internal standard and extraction of residues with acetonitrile/concentrated HCl (98:2, v/v) or acetonitrile, respectively. Other "wet" commodities (50–100 g, e.g. sugarbeet, potato) are extracted directly by acetonitrile/concentrated HCl (98:2, v/v) without presoak. After filtration, the acetonitrile was removed by rotary evaporation (below 40 ºC). Any ester or conjugates were converted to fluazifop acid (II) by hydrolysis in 6 M HCl (1 hour, 60 ºC). The samples were diluted with water, acidified to pH < 1 and partitioned with dichloromethane. The dichloromethane is evaporated to dryness and redissolved in 50% acetone/deuterated acetone. The final determination is by ^{19}F NMR using an internal standard or external standard method.

Method PPRAM 83 was used in supervised residue trials on oranges [M4533B], apples [TMU3291/B], plums [TMU3311/B] blackcurrants [M5091B], gooseberries [M5092B], raspberries [M5320B], strawberries [M5319B], olives [M4526B], onions [M4799B, M5264B,], green onions [M4799B], leeks [M4217B], head cabbage [M4799B], green beans with pods [M4799B], green peas (seeds, pods, forage) [M4234B, M4261B, M5347B, RJ1059B], dry beans [M4130B, M4799B,], dry broad beans (seeds, pods) [M4233B, M4994B, M5002Badd, M5316B], dry harvested soya beans [M4140B, M4141B] carrots [M5317B], swedes (roots, tops) [M5318B], turnips (roots, tops)

[RJ0997B], fodder beets (roots, tops) [M4870B], cotton seeds [M4799B, RJ1131B] and sugarcane [TMU3310/B]. Concurrent method validation results extracted from supervised trial reports are summarized in Table 112.

NMR Method PPRAM 83 modification A was used in a supervised trial on citrusfruits [RR89-051B] and in a processing study with oranges [RR 89-052B]. The hydrolysate was partitioned with diethylether, instead of dichloromethane. Validation results are shown in Table 112.

NMR Method PPRAM 83 modification B was used for the analysis of orange oil [RR 89-052B], a modification of method PPRAM 83 was required. Samples were first hydrolysed with 6 M HCl, and then the hydrolysed extracts were partitioned with dichloromethane. The aqueous extracts were discarded. Then the residual oil was dissolved in a 1% sodium bicarbonate solution and partitioned with hexane. The aqueous extracts were adjusted to pH1 with HCl. After acidification the extracts were partitioned with dichloromethane. The aqueous layer was discarded. The residuum was dissolved in acetone and then analysed as described for PPRAM 83. Validation results are shown in Table 112.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: NMR method PPRAM83 is considered:

- valid (reduced validation) for the determination of fluazifop acid (II) in orange (at 0.05 mg/kg only), strawberries (at 0.5 mg/kg only), bulb onions (at 0.5 mg/kg only), dry broad bean seeds (at 0.5 mg/kg only), swede roots (at 0.5 mg/kg only), swede tops (at 0.5 mg/kg only)
- insufficiently validated for the determination of fluazifop acid (II) in lemon, grapefruit, orange peels, orange dried pulp, orange molassess, orange juice, orange oil, apples, plums, black currants, gooseberries, raspberries, olives, green onions, cabbage, green beans with pods, green pea seeds, green pea pods, green pea forage, dry bean seeds, dry soya bean seeds, turnip roots, turnip tops, cotton seeds, fodderbeet roots, fodderbeet tops, leeks, cotton seeds.
- not valid for the determination of fluazifop-butyl (no validation results)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive)

The valid LOQ for NMR method PPRAM 83 is 0.05 mg/kg (no validations below this level).

Table 112 Validation results for method PPRAM 83

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
orange flesh or peel	0.01	0.5 (IS)	85	ns	6.3%	ns	< 0.01 (2)	internal std; linearity ns	PP5/0191; M4533B
apples	0.02	0.05 (F) 0.1 (IS)	89 118 138	- 89-	- 13%	1 9	< 0.02 (3)	internal std; linearity ns	405746; TMU3291/B
Plums	0.02	0.05 (F) 0.1 (F)	124 113	- -	- -	1 1	< 0.02	internal std; linearity ns	PP5/0480; TMU3311/B
black currants	0.05	0.5 (F)	98	ns	10%	ns	< 0.05 (2)	external std; linearity ns	PP5/0460; M5091B
gooseberries	0.05	0.5 (IS)	99	ns	19%	ns	< 0.05 (2)	internal std; linearity ns	PP5/0474; M5092B
raspberries	0.05	0.5 (IS)	77	ns	12%	ns	< 0.05 (2)	internal std; linearity ns	PP5/0193; M5320B
strawberries	0.05	0.5 (IS) 0.5 (F)	89 96 116	73-99 79-	10% 19%	12 3	< 0.05 (4)	internal std; linearity ns	PP5/0195; M5319B;
olive flesh	0.03	0.5 (IS)	92	ns	6.4%	ns	< 0.03 (2)	internal std; linearity ns	PP5/0485 M4526B
bulb onions	0.01	0.1 (IS)	98	ns	15%	ns	< 0.01 (2)	internal std; linearity ns	PP5/0380; M4799B

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
bulb onions	0.05	0.5 (IS) 0.5 (F)	90 104 99 118	73- 82-	12% 19%	6 4	< 0.05 (1)	internal std; linearity ns	PP5/0091; M5264B
green onion tops	0.01	0.1 (IS)	98	ns	15%	ns	0.02 (1)	internal std; linearity ns	PP5/0380; M4799B
leeks	0.05	0.1 (IS) 0.1 (IS)	86 89	ns ns	21% 20%	ns ns	< 0.05 (7)	internal std. linearity ns	PP5/0087; M4217B
cabbage	0.01	0.1 (IS)	98	ns	15%	ns	0.02 (1)	internal std; linearity ns	PP5/0380; M4799B
green beans with pods	0.01	0.1 (IS)	98	ns	15%	ns	< 0.01 (1)	internal std; linearity ns	PP5/0380; M4799B
green pea seeds	0.02	0.25 (IS)	77	ns	13%	ns	< 0.02 (3)	internal std; linearity ns	PP5/0396 M4234B;
green pea seeds	0.05	0.1–0.5 (IS)	95	ns	27%	ns	< 0.05 (10)	internal std; linearity ns	PP5/0161; M4261B
green pea seeds	0.05	0.5 (F)	80	ns	18%	ns	< 0.05 (2)	internal std; linearity ns	PP5/0150; M5347B
green pea forage	0.05	0.5 (IS)	75	ns	17%	ns	< 0.05 (2)	internal std; linearity ns	PP5/0161 M4261B;
green pea forage	0.05	0.5 (F)	80	ns	18%	ns	0.10 (1); 0.24 (1)	internal std; linearity ns	PP5/0150; M5347B
green pea forage or pods	0.05	0.5 (IS) 0.5 (F)	91 86	ns ns	17% 7.1%	ns ns	< 0.05 (3)	internal std; linearity ns	PP5/0405; RJ1059B
dry bean seeds	0.02	0.5 (IS)	74	ns	4.7%	ns	0.06 (1), 0.26 (1)	internal std; linearity ns	PP5/0376; M4130B
dry bean seeds	0.02	0.1 (IS)	98	ns	15%	ns	< 0.02 (1)	internal std; linearity ns	PP5/0380; M4799B
dry broad bean seeds	0.05	0.25 (IS)	71	ns	14%	ns	< 0.02 (3)	internal std; linearity ns	PP5/0374; M4233B
dry broad bean seeds	0.05	0.05 (IS) 0.05 (F)	78 81	- -	- -	1 1	< 0.05 (4)	internal std linearity ns	PP5/0384; M4994B
dry broad bean seeds	0.05	0.5 (IS) 0.5 (F)	76 82	59-94 72-91	12% 8.5%	17 6	< 0.05 (6)	internal std linearity ns	PP5/0381; M5002add;
dry broad bean seeds	0.05	0.5 (IS) 0.5 (F)	88 110 81	72- 66-96	15% 16%	13 4	< 0.05 (3)	internal std; linearity ns	PP5/0387; M5316B
dry broad bean pods	0.05	0.5 (IS) 0.5 (F)	63 88	52-73 84-92	14% 6.4%	5 2	< 0.05 (1)	internal std; linearity ns	PP5/0387; M5316B
dry soya bean seeds	0.03	0.05 (IS)	75	ns	14%	ns	< 0.03 (3), 0.20 (1)	internal std; linearity ns	PP9/0120 M4140B;
dry soya bean seeds	0.03	0.5 (IS)	80	ns	13%	ns	< 0.03 (1)	internal std; linearity ns	PP5/0407 M4141B;
carrots	0.05	0.5 (IS) 0.5 (F)	95 101 96 101	88- 91-	5.4% -	8 2	< 0.05 (2)	internal std linearity ns	PP5/0085 M5317B
swede roots and tops	0.05	0.5 (IS) 0.5 (F)	105 127 106 113	49- 98-	18% 6.1%	16 5	R: < 0.05 (2) T: < 0.05 (2)	internal std; linearity ns	PP5/0101; M5318B
turnip roots and tops	0.05	0.5 (IS)	99	ns	22%	ns	R: < 0.05 (2) T: < 0.05 (2)	internal std; linearity ns	PP5/0099; RJ0997B
fodderbeet roots and tops	0.01	ns (IS)	91	ns	25%	ns	R: < 0.01 (2) T: < 0.01 (2)	internal std linearity ns	PP5/0519; M4870B
cotton seeds	0.03	0.1 (IS)	98	ns	15%	ns	< 0.03 (1)	internal std; linearity ns	PP5/0380; M4799B
cotton seed	0.05	0.05 (F) 0.5 (F)	86 88	- 86-89	- -	1 2	< 0.05 (1)	no data	PP5/0576 RJ1131B;
sugarcane stalks	0.02	0.1 (F) 0.1 (IS)	108 110	ns ns	ns 10%	ns ns	< 0.02	internal std; linearity ns	405720 ; TMU3310/B

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
PPRAM 83 modification A									
Lemon	0.03	0.05 (F) 0.1 (F)	96 91 101	93-98 81-	- -	2 2	0.03	internal std; linearity ns	PP5/0466; RR89-051B
Grapefruit	0.03	0.05 (F) 0.1 (F)	93 100 93	86- 92-94	- -	2 2	0.03	internal std; linearity ns	PP5/0466; RR89-051B
Orange	0.03	0.05 (F) 0.1 (F)	89 93 100 [1]	88-90 86-	11% -	3 2	0.03	internal std; linearity ns	PP5/0466; RR89-051B
Orange	0.03	0.05 (F) 0.1 (F)	88 157	- -	- -	1 1	< 0.03	Internal std; linearity ns	PP5/0586 RR 89-052B (processing)
Orange peel	0.03	0.05 (F)	82	-	-	1	< 0.03	internal std; linearity ns	PP5/0586 RR 89-052B (processing)
Orange dried pulp	0.03	0.05 (F)	77	-	-	1	< 0.03	internal std; linearity ns	PP5/0586 RR 89-052B (processing)
Orange molasses	0.03	0.05 (F)	94	-	-	1	< 0.03	internal std; linearity ns	PP5/0586 RR 89-052B (processing)
Orange juice	0.03	0.05 (F)	89	-	-	1	< 0.03	internal std; linearity ns	PP5/0586 RR 89-052B (processing)
Orange pulp (finisher)	0.03	0.05 (F)	96	-	-	1	< 0.03	internal std; linearity ns	PP5/0586 RR 89-052B (processing)
PPRAM 83 modification B									
Orange oil	0.03	0.05 (F)	70	-	-	1	< 0.03	internal std; linearity ns	PP5/0586 RR 89-052B (processing)

HPLC-UV Method PCY 86-1

Method PCY 86-1 determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) in asparagus and residues are expressed as fluazifop acid (II). The reported LOQ is 0.1 mg/kg.

Method PCY 86-1 (12 March 1986), is described by [Baron, 1987, 464389, report IR-4 PR 2201]. Homogenised asparagus spears (50 g) are extracted by homogenisation with acetone/dichloromethane/concentrated HCl (25:75:0.01). After addition and shaking with water, the dichloromethane phase was collected. The remaining water phase was extracted twice with dichloromethane and the dichloromethane phases were collected. All thouree dichloromethane phases were combined and extracted once with water. The dichloromethane phase was retained, evaporated to dryness and then hydrolysed with 6 M HCl (1 hour, 60 ºC) so that any fluazifop-butyl or fluazifop conjugates are converted to fluazifop acid (II). Samples are then diluted with water, filtered and fluazifop acid (II) is partitioned into dichloromethane (3 times). The dichloromethane phase is then extracted twice with 1% sodium bicarbonate solution. The bicarbonate solution is acidified, diluted with water and then re-extracted with dichloromethane (3 times). The dichloromethane phase was filtered thourough anhydrous sodium sulfate, evaporated to dryness and re-dissolved in acetonitrile. Total fluazifop is quantified by reversed phase HPLC-UV/VIS at 230–540 nm using external standard calibration.

Method PCY 86-1 was used in supervised residue trials on asparagus [IR-4 PR 2201]. Concurrent method validation results extracted from supervised trial reports are summarized in Table 113.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-UV method PCY 86-1 is considered insufficiently validated for the determination of total fluazifop in asparagus (limited recovery data). The valid LOQ is 0.2 mg/kg (no validations below this point).

Table 113 Validation results for method PCY 86-1

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
Asparagus spears	0.1	0.20	85	-	-	1	<0.1 (1)	no data	464389; PR-2201
		0.25	96	-	-	1			
		0.50	78	ns	-	2			
		0.75	98	-	-	1			
		1.0	90	ns	-	2			

NMR Method PPRAM 103 for determination of total CF3-pyridone

NMR method PPRAM 103 and its modifications determine total CF3-pyridone (i.e. CF3-pyridone (X) and its acid cleavable conjugates) in various crops. Residues are expressed as CF3-pyridone (X). The reported LOQ is 0.02–0.05 mg/kg depending on the matrix.

NMR Method PPRAM 103 (9 May 1986) is described by [Davy, 1986, PP9/0390, report PPRAM 103]. Homogenised crop samples are extracted by homogenisation with acetonitrile/water (50:50, v/v). After filtration, the extract is evaporated until the aqueous fraction remains. The aqueous remainder is adjusted to 1 M HCl and CF3-pyridone (X, free form) and fluazifop acid (II, free form) are partitioned into diethyl-ether. The remaining aqueous phase is refluxed for 1 hour (1 M HCl) to convert CF3-pyridone conjugates into CF3-pyridone (X). CF3-pyridone (X) is then partitioned into diethyl-ether. The diethyl ether fractions from both partitions are combined and dried over anhydrous sodium sulphate. The diethyl ether extract is evaporated to dryness and the residue is redissolved in 50% deutero-acetone/acetone. CF3-pyridone (X) is then quantified by ^{19}F NMR.

A summary report on validations performed for method PPRAM 103 is available in [Atreya, 1990, PP9/0390, report M5166B]. A residue analytical method validation is available in [Upton, 1986, PP9/0035 report M4239B]. Validation results are summarized in Table 114. The method was used in a supervised trial on carrots [Atreya *et al*., 1984, PP9/0065, report PP009B300, no validations].

Modification A of NMR Method PPRAM 103 was used in a storage stability study on onions [M4843B], apples, lettuce, soya bean [M4842B] and peanut kernels [M4841B] to determine fluazifop-butyl and/or CF3-pyridone (X). For peanut kernels and soya bean seeds, the oil was extracted by partitioning with hexane and discarding the hexane prior to hydrolysis. Since the samples for the storage stability studies were fortified with fluazifop-butyl and/or CF3-pyridone (X), the hydrolysis step was omitted and the final residue was dissolved in deutero-acetone. The final determination was by ^{19}F Fourier Transformed NMR. Validation results are summarized in Table 114.

Modification B of NMR method PPRAM 103 was used in a supervised residue trial on onions [M4545B]. The final residue was dissolved in deutero-acetone and final determination was by ^{19}F Fourier Transformed NMR. Validation results are summarized in Table 114.

Modification C of NMR method PPRAM 103 was used in a supervised residue trial on onions [M4266B]. Onions were extracted by macerating for 5 minutes in 98:2 acetonitrile: concentrated HCl. After reflux (as in PPRAM 103), the aqueous solution was cooled and adjusted to pH 5–7 and then partitioned with diethyl ether. The aqueous remainder was then re-acidified and extracted again with diethyl ether. The diethyl ether fractions were combined. Analysis as in PPRAM 103. Validation results are summarized in Table 114.

No radiovalidation was conducted.

Reviewer's conclusion: NMR method PPRAM103 and its modifications are considered not acceptable for determination of total CF3-pyridone (X). Fluazifop acid (II) degrades under the hydrolysis conditions used in this analytical method and therefore levels of CF3-pyridone (X) are overestimated.

Modification A of NMR method PPRAM 103 (without hydrolysis step) is considered

- valid (reduced validation) for the determination of free CF3-pyridone (X) in apples (at 1.0 mg/kg only), onion bulb (at 0.2–1.0 mg/kg), lettuce (at 1.0 mg/kg only), peanut kernels (at 1.0 mg/kg only) as used in the storage stability study
- is not valid for the determination of free CF3-pyridone (X) in soya bean seeds (low recovery, high RSD, high levels in control samples).
- valid for the detemination of fluazifop-butyl in onion (at 1.0 mg/kg only)

The valid LOQ is 0.05 mg/kg for CF3-pyridone (X) or 1.0 mg/kg for fluazifop-butyl (no validations below this level).

Table 114 Validation results for NMR method PPRAM 103 and its modifications

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
onion bulb	0.05	0.05 (X) 0.2 (X) 0.75 (X)	62 87 64	- 80-93 -	- 7.5% -	1 3 1	ns	ns	PP9/0390 M5166B (validation)
carrots	0.05	0.5 (X)	100	-	-	1	ns	ns	PP9/0390 M5166B (validation)
carrots	0.05	0.05 (X) 0.1 (X) 0.2 (X)	95 83 79	84-109 76, 90 74, 83	12% - -	4 2 2	ns	ns	PP9/0035; M4239B (validation)
sugarbeet root	0.05	0.2 (X)	129	-	-	1	ns	ns	PP9/0390 M5166B (validation)
peanut meal	0.05	0.2 (X)	74	66-83	9.5%	4	ns	ns	PP9/0390 M5166B (validation)
peanut hull	0.05	0.2 (X) 1.0 (X)	93 73	64-118 65-93	24% 13%	4 7	ns	ns	PP9/0390 M5166B (validation)
Modification A (no hydrolysis step; storage stability studies only)									
apples	0.01	1.0 (X)	84	68-96	13%	6	< 0.01 (1)	no data	PP5/0076; M4842B (stor stab)
onion bulb	0.05	1.0 (Fb) 1.0 (X)	89 75	75-106 64-94	9.4% 14%	10 14	< 0.01 (1)	no data	PP5/0077; M4843B; (stor stab)
lettuce	0.01	1.0 (X)	71	58-91	17%	6	< 0.01 (1)	no data	PP5/0076; M4842B (stor stab)
soya bean seeds	0.4	1.0 (X)	64	45-90	27%	6	<0.4 (1)	no data	PP5/0076; M4842B (stor stab)
peanut kernels	0.05	1.0 (X)	73	65-93	12%	7	< 0.05 (1)	ns	462746; M4841B (stor stab)
Modification B (detection with 19F-FT-NMR									
bulb onions	ns	ns	ns		ns	ns	ns	ns	PP5/0251; M4545B
Modification C (extraction with 98:2 acetonitrile:concentrated HCl)									
bulb onions	0.05	0.05 (X) 0.2 (X)	62 86	- 80-93	- -	1 2	< 0.05 (1)	linear by graph	PP5/0250; M4266B

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
		0.75 (X) 1.0 (X)	64 78	- 75-82	- -	1 2			

HPLC-UV Method TMU3251

HPLC-UV method TMU3251 determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) in pecans and residues are expressed as fluazifop acid (II). The reported LOQ was 0.03 mg/kg.

A summary of this method was provided [Watford and Francis, 1987, 434208, report TMU3251/B]. The pecan meats (weight not stated) were extracted by homogenization with acetonitrile:conc. hydrochloric acid solution (volume ratio not stated). The extracts were evaporated to aqueous volume and hydrolyzed with 6M hydrochloric acid solution at 60°C for 1 hour. Samples were then diluted with water and partitioned into diethylether. The extracts were partitioned into a 1% sodium bicarbonate solution, washed thouree times with hexane, acidified, and partitioned into dichloromethane. The extracts were evaporated to dryness, taken thourough a coagulation procedure, and partitioned into dichloromethane. Remaining co-extractives were removed by adsorption chouromatography using disposable silica columns. The final determination was HPLC-UV with detection at 230 nm .

The method was used in supervised residue trials on pecans [TMU3251/B]. Validation results are summarized in Table 115.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-UV method TMU3251 is considered:

- insufficiently validated for the determination of total fluazifop in pecans.
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).

The valid LOQ is 0.05 mg/kg (no validations below this point).

Table 115 Validation results for method TMU3251

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report no
pecans	0.03	0.05-0.1	68	-	2%	ns	< 0.03 (3)	no data	434208; TMU3251

GC-MS Method RR89-073B

GC-MS Method RR89-073B determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) and residues are expressed as fluazifop. The reported LOQ is 0.01 mg/kg.

Method RR 89-073B (8 May 1990) was described by [Alferness, 1990, PP5/0607, report RR 89-073B]. Dry matrices (hay, dry pomace, raisins, grains, seeds, 10 g) are soaked for at least 2 hours in 1 M HCl and are then macerated by addition of acetonitrile (40+15, v/v, acetonitrile/1 M HCl). Soaking can be omitted for other matrices. Juice can directly be hydrolysed. An aliquot of the extract is evaporated to the aqueous volume and hydrolysed with 6 M HCl (60 °C, 1 hours). The hydrolysate is diluted with water and partitioned with diethyl ether. The diethyl ether extract is cleaned by solid-phase extraction using disposable silica columns. The column eluate is evaporated to dryness, and an

aliquot of ethereal diazomethane reagent is added to derivatize the fluazifop acid (II) to its methyl ester. After the derivatisation reaction is complete, the solvent iss evaporated, and the residuum is dissolved in toluene. The methyl ester of fluazifop acid (II) is quantified by GC-MS at m/z 341 using an external standard for fluazifop-methyl (in-situ derivatised).

Confirmation can be achieved by GC-NPD or by using alternate m/z ions (146, 227, 254, 282) for GC-MS or by comparing ratios for two or more ions.

A validation study for method RR 89-073B is available in [Alferness, 1990, PP5/0607, report RR 89-073B]. Validation results are summarized in Table 116.

Modification A of method RR 89-073 B was used in a processing study on sunflower seed [Alferness *et al*, 1991, PP5/0233, report RR91-010B]. The extraction for oil was changed and extraction and hydrolysis for other sunflower commodities was slightly changed. Sunflower seeds, hulls, meal (10 g) were extracted by maceration in acetonitrile/1 M HCl (27:73, v/v) without soaking. The extract was hydrolysed with 6 M HCl for 2 hours at 60 °C. Further as for method RR 89-073B. Sunflower oil (5 g) was partitioned with acetonitrile and hexane. The organic phase was discarded and the remaining phase was diluted with water. The extract was then evaporated to the aqueous volume. The aqueous phas was hydrolysed with 6 M HCl (1 hour, 60 °C). The hydrolysate is diluted with water and partitioned with dichloromethane. The aqueous phase is discarded, and the organic phase is partitioned with 1% sodium bicarbonate solution. The organic phase is discarded and the aqueous phase is acidified to pH 1 and then partitioned with dichloromethane. The dichloromethane phase is cleaned by solid-phase extraction and derivatised as described for method RR 89-073B. Validation results are summarized in Table 116.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: GC-MS method RR 89-073B and its modifications is considered:

- valid (reduced validation) for the determination of fluazifop acid (II) in apple fruit (at 0.01–0.2 mg/kg) and grape berries (at 0.01 mg/kg only)
- insufficiently validated for the determination of fluazifop acid (II) in apple juice, apple wet pomace, apple dry pomace, grape juice, grape raisins, grape wet pomace, grape dry pomace, sugarbeet roots, sugarbeet dry pulp, sugarbeet white sugar, sugarbeet molasses, alfalfa seed, alfalfa forage, alfalfa hay, sunflower seed, sunflower meal, sunflower hulls, sunflower crude oils, sunflower refined oil.
- not valid for the determination of fluazifop-butyl (no validation results)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive)

The valid LOQ is 0.01 mg/kg (no validations below this point).

Table 116 Validation results for GC-MS method RR-89-073B and its modifications

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
RR 89-073B (original method)									
apple fruit	0.01	0.01 (F) 0.05 (F) 0.2 (F)	102 88 102	88-116 - 82-115	14% - 17%	3 1 3	no data	external std in solvent 0.01-10 mg/L linear	PP5/0607; RR 89-073B (validation)
apple juice	0.01	0.01 (F) 0.5 (F)	102 99	- -	- -	1 1	no data	idem	PP5/0607; RR 89-073B (validation)
apple wet pomace	0.01	0.01 (F) 0.1 (F)	109 99	- -	- -	1 1	no data	idem	PP5/0607; RR 89-073B (validation)

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
apple dry pomace	0.01	0.01 (F) 0.02 (F)	90 120	- -	- -	1 1	no data	idem	PP5/0607; RR 89-073B (validation)
grape berries	0.01	0.01 (F) 0.05 (F) 0.2 (F)	85 89 101	65-97 - 87-115	21% - -	3 1 2	no data	idem	PP5/0607; RR 89-073B (validation)
grape juice	0.01	0.01 (F) 0.5 (F)	102 91	- -	- -	1 1	no data	idem	PP5/0607; RR 89-073B (validation)
grape raisin	0.01	0.01 (F) 0.5 (F)	85 110	- -	- -	1 1	no data	idem	PP5/0607; RR 89-073B (validation)
grape wet pomace	0.01	0.01 (F)	70	-	-	1	no data	idem	PP5/0607; RR 89-073B (validation)
grape dry pomace	0.01	0.01 (F)	76	60-91	-	2	no data	idem	PP5/0607; RR 89-073B (validation)
sugarbeet roots	0.01	0.01 (F) 1.0 (F)	104 123	- -	- -	1 1	no data	idem	PP5/0607; RR 89-073B (validation)
sugarbeet dry pulp	0.01	0.01 (F) 1.0 (F)	119 111	- -	- -	1 1	no data	idem	PP5/0607; RR 89-073B (validation)
sugarbeet white sugar	0.01	0.01 (F) 1.0 (F)	96 81	85-106 -	- -	1 1	no data	idem	PP5/0607; RR 89-073B (validation)
sugarbeet molasses	0.01	0.01 (F) 1.0 (F) 20 (F)	81 75 102	- - -	- - -	1 1 1	no data	idem	PP5/0607; RR 89-073B (validation)
alfalfa seed	0.01	0.1	103	-	-	1	no data	idem	PP5/0607; RR 89-073B (validation)
alfalfa forage	0.01	0.1 (F) 9.4 (F) 11 (F)	98 96 107	97-100 81-111 -	- - -	2 2 1	no data	idem	PP5/0607; RR 89-073B (validation)
alfalfa hay	0.01	0.2 (F) 37 (F)	108 66	97-118 -	- -	2 1	no data	idem	PP5/0607; RR 89-073B (validation)
sunflower seed	0.01	0.1 (F)	107	-	-	1	no data	idem	PP5/0607; RR 89-073B (validation)
Modification A (longer hydrolysis, different extraction for oil)									
sunflower seed	0.01	0.01 (F) 0.1 (F)	81 92	76-86 90-92	- -	2 2	< 0.01 (4)	no data	PP5/0233, RR91-010B (processing)
sunflower meal	0.01	0.01 (F) 0.1 (F)	96 101	91-102 98-104	- -	2 2	< 0.01 (2)	no data	PP5/0233, RR91-010B (processing)
sunflower hulls	0.01	0.01 (F) 0.1 (F)	97 76	- -	- -	1 1	< 0.01 (2)	no data	PP5/0233, RR91-010B (processing)
sunflower crude oil	0.01	0.01 (F) 0.1 (F)	100 91	- -	- -	1 1	< 0.01 (2)	no data	PP5/0233, RR91-010B (processing)
sunflower refined oil	0.01	0.01 (F)	78	-	-	1	< 0.01 (2)	no data	PP5/0233, RR91-010B (processing)

GC-MS Method RR 90-384B for determination of total CF3-pyridone

GC-MS Method RR 90-384B determines total CF3-pyridone (i.e. CF3-pyridone (X) and its conjugates) as one single analyte and residues are expressed as CF3-pyridone (X). The reported LOQ is 0.01 mg/kg.

GC-MS Method RR 90-384B (25 July 1991) was described by [Kukla, 1991, PP5/0608, RR 90-384B] Samples (5-25 g) are extracted by macerating in acetonitrile/water (50:50, v/v). After filtering the supernatant liquid, the acetonitrile is removed by rotary evaporation and the remaining aqueous solution is acidified. Free CF3-pyridone (X) is partitioned into diethyl ether. The remaining aqueous phase is hydrolysed with 1 M HCl (reflux, 1 hour) to convert CF3-pyridone conjugates to CF3-pyridone (X). CF3-pyridone (X) is partitioned into diethyl ether. The two diethyl ether fractions are combined and dried over anhydrous sodium sulphate. The diethyl ether is evaporated off and the residuum is dissolved in acetonitrile and then derivatised with N-tert-buyldimethylsilyl-N-methyl-trifluoroacetamide containing 1% tert-butyldimethylchlorosilane to form the tert-butyldimethylsilyl derivative of CF3-pyridone (X). The derivative is analysed by GC-MS at m/z=220 against external standards in solvent for in-situ prepared TBDMS derivative of CF3-pyridone (X).

Confirmation can be achieved by GC-NPD or by using alternate m/z ions (190, 221, 228) for GC-MS or by comparing ratios for two or more ions.

A validation study for method RR 90-384B is available in [Kukla, 1991, PP5/0608, RR 90-384B. GC-MS method RR 90-384B was used in a processing study on sunflowers [RR 91-010B]. Validation results are shown in Table 117.

Modification A of Method RR 90-384B was used in processing studies on sunflower seed [Alferness *et al*, 1991, PP5/0233, report RR91-010B]. The extraction for oil was changed. Oil samples were partitioned with acetonitrile/hexane. The aqueous phase was diluted with water. After evaporation to the aqueous volume, the extract was hydrolysed, partitioned and derivatised as described for method RR 90-384B. Validation results are shown in Table 117.

Modification B of method RR 90-384 was used in a field rotational crop study [Atreya *et al*, 1997, PP5/0590, report RJ2202B]. Commodities were extracted 3 times with extraction solvent instead of a single extraction specified in the method. CF3-pyridone conjugates were hydrolysed using 1 M HCl for 1 hour at 60 °C instead of refluxing for 1 hour. Validation results are shown in Table 117.

No radiovalidation was conducted.

Reviewer's conclusion: GC-MS method RR 90-384B and its modifications is considered not acceptable. Fluazifop acid (II) degrades under the hydrolysis conditions used in this analytical method (reflux) and therefore levels of CF3-pyridone (X) are overestimated. Fluazifop acid (II) remains intact under the hydrolysis conditions used in modification B.

Modification B of method RR 90-384B is considered:

- valid (full validation) for the determination of free CF3-pyridone (X) in wheat straw (0.01–0.05 mg/kg).
- valid (reduced validation) for the determination of free CF3-pyridone (X) in carrot tops (0.02-0.1 mg/kg), carrot roots (0.01–0.02 mg/kg), wheat grain (0.01–0.02 mg/kg) and wheat forage (0.01–0.02 mg/kg)
- not valid for the determination of CF3-conjugates (no radiovalidation).

The valid LOQ is 0.01 mg/kg.

Table 117 Validation results GC-MS method RR 90-384B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
RR 90-384B (original method)									
sugarbeet	0.01	0.01 (X)	92	-	-	1	no data	external std	PP5/0608,

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
roots		0.04 (X)	96	-	-	1		in solvent 0.01-1 mg/L linear	RR 90-384B (validation)
sugarbeet dry pulp	0.01	0.01 (X) 0.04 (X)	113 100	- -	- -	1 1	no data	idem	PP5/0608, RR 90-384B (validation)
sugarbeet molasses	0.01	0.01 (X) 0.4 (X)	83 85	- -	- -	1 1	no data	idem	PP5/0608, RR 90-384B (validation)
sugarbeet sugar	0.01	0.01 (X) 0.02 (X) 0.04 (X)	95 97 78	87-103 - -	- - -	2 1 1	no data	idem	PP5/0608, RR 90-384B (validation)
sunflower seed	0.01	0.01 (X) 0.1 (X)	92 83	82-101 75-91	- -	2 2	< 0.01 (4)	no data	PP5/0233, RR 91-010B (processing)
sunflower meal	0.01	0.01 (X) 0.1 (X)	93 83	72-114 79-87	- -	2 2	< 0.01 (2)	no data	PP5/0233, RR 91-010B (processing)
sunflower hulls	0.01	0.01 (X) 0.1 (X)	88 89	- -	- -	1 1	< 0.01 (2)	no data	PP5/0233, RR 91-010B (processing)
Modification A (different extraction for oil)									
sunflower crude oil	0.01	0.01 (X) 0.1 (X)	88 71	- -	- -	1 1	< 0.01 (2)	no data	PP5/0233, RR 91-010B (processing)
sunflower refined oil	0.01	0.01 (X) 0.1 (X)	89 73	- -	- -	1 1	< 0.01 (2)	no data	PP5/0233, RR 91-010B (processing)
Modification B (lower hydrolysis temperature)									
lettuce	0.01	0.01 (X) 0.02 (X) 0.05 (X)	82 89 72	78-85 87-91 -	- - -	2 2 1	< 0.01 (2)	no data	PP5/0590; RJ2202B (rotational)
carrot tops	0.01	0.02 (X) 0.1 (X)	75 83	57-90 75-89	21% 7.1%	4 4	< 0.01 (2)	no data	PP5/0590; RJ2202B (rotational)
carrot roots	0.01	0.01 (X) 0.02 (X) 0.04 (X)	94 96 74	84-103 93-100 74-75	10% 3.1% -	4 4 1	< 0.01 (2)	no data	PP5/0590; RJ2202B (rotational)
wheat grain	0.01	0.01 (X) 0.02 (X)	92 94	76-108 92-97	19% 2.4%	4 4	< 0.01 (4)	no data	PP5/0590; RJ2202B (rotational)
wheat straw	0.01	0.01 (X) 0.02 (X) 0.05 (X)	74 72 76	66-83 65-78 76-77	9.0% 7.5% -	8 6 2	< 0.01 (4)	no data	PP5/0590; RJ2202B (rotational)
wheat forage	0.01	0.01 (X) 0.02 (X)	86 86	73-97 82-91	8.5% 4.4%	8 4	< 0.01 (4)	no data	PP5/0590; RJ2202B (rotational)

GC-MS Method RR91-014B

GC-MS Method RR91-014B determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) in various crop commodities as one single analyte (common moiety) and residues are expressed as fluazifop acid (II). The reported LOQ is 0.01 mg/kg.

Method RR91-014B (25 February 1992) is described by [Alferness, 1992, PP5/0066, report RR 91-014B]. Homogenised samples (10 g) are macerated with a mixture of 1 M HCl and acetonitrile (20+40, v/v). Dry crops (e.g. straw, hay, dry pomace, grain, seeds) require preliminary soaking in 1 M HCl for a minimum of 2 hours followed by maceration in acetonitrile (acetonitrile : 1M HCl = 20+40, v/v). Depending on the matrix, additional acetonitrile may be required to facilitate maceration. High sugar matrices (e.g. raisins) may require additional water instead of acetonitrile during maceration.

After filtration, an aliquot of the extract is evaporated to remove the acetonitrile. The remaining aqueous phase is hydrolysed after addition of 6 M HCl (1 hour at 60 °C). Any ester or acid conjugates of fluazifop are thereby converted to fluazifop. Samples are then diluted with water, the pH is adjusted to <1 and fluazifop acid (II) is partitioned twice into diethyl ether. Both diethyl ether fractions are combined and then dried with anhydrous sodium sulphate, followed by evaporation to (near) dryness. The residuum is redissolved in hexane/dichloromethane/methanol/acetic acid (60+40+1.5+0.5, v/v). Remaining co-extractives are removed by adsorption chouromatography on a disposable silica SPE column. The eluate is evaporated to dryness and redissolved in methanol/HCl derivatizing reagent and treated for 30 min at 60 °C to form the methyl ester derivative of fluazifop. The mixture is cooled, diluted with water and fluazifop acid (II) is then partitioned into toluene. After addition of citral to the toluene phase, fluazifop acid (II) is quantified by GC-MS (m/z=341) using external standards for fluazifop-methyl in solvent. Matrix effects are reduced to acceptable levels by diluting the sample or using peak area instead of peak height. Final residues are expressed as fluazifop.

Confirmation can be achieved by comparing ratios of two or more ions (m/z = 146, 227, 254, 282, 341) or by using a GC-NPD.

Some crops and processed fractions require deviations to the standard extraction procedure.

- If a sample absorbs excessive solvent (e.g. dry beans or dry pomace) the sample size may be reduced or alternatively the quantities of solvents increased.
- Liquid homogenous processed fractions (e.g. fruit juice) are diluted with water and hydrolysed directly.
- Oil fractions (5 g) are macerated with hexane/acetonitrile (15+10, v/v) and the lower acetonitrile layer is isolated and diluted with water. The extract is evaporated to remove the acetonitrile. The remaining aqueous phase is hydrolysed. Hydrolysates from oil fractions are partitioned into dichloromethane instead of diethyl ether. The dichloromethane fraction is partitioned into 1% sodium bicarbonate solution. The aqueous phase is acidified to pH=1 and partitioned into dichloromethane and evaporated to dryness. The residuum is derivatised.

A validation study [Alferness, 1992, PP5/0066, report RR 91-014B] and an independent laboratory validation study [Devine, 1999, PP5/0080, report CEMR-1159] are available. Validation results are shown in Table 118.

The method has been used in supervised residue trials on almonds (nutmeat, hulls) [RR 92-041B], walnuts [RR 92-009B], soya bean seeds [RR 99-021B], a processing studies on asparagus [RR 92-057B] and field rotational crop study [RJ2202B]. Validation results extracted from these study reports are shown in Table 118.

GC-MS method ABC 45820-M-1 is identical to RR91-014B, but was issued under a new name by another laboratory. The method was used in supervised trials on banana [RR 00-043B]. Validation results are shown in Table 118.

Modification A of method RR91-014B was used in supervised trials on citrus fruit [RR 00-063B], grapes [RR 00-062B], dry beans [RR 00-061B], dry soya beans [RR 00-065B], sugar beets (roots, tops) [RR 00-066B], a processing study on sugar beets [RR 00-070B], a processing study on grapes [RR 00-067B] and a processing study on soya bean [RR 00-069B]. The residuum was dissolved in BF3 in methanol in stead of methanolic/HCl reagent to improve derivatization efficiency. In addition to this modification, for the studies on dry beans [RR 00-061B] and soya bean [RR 00-069B and RR 00-065B] the anhydrous sodium sulfate filtration step was omitted to improve recoveries for soya bean seed, meal and hulls. For analysis of peanut and soya bean oil [RR 00-069B], a second dichloromethane partition was added, the use of 1% sodium bicarbonate was omitted and the pH was not adjusted. Validation results are shown in Table 118.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: GC-MS method RR 91-014B and its modifications is considered:

- valid (full validation) for the determination of fluazifop acid (II) in head cabbage (0.01–0.2 mg/kg), tomatoes (0.01–0.5 mg/kg), dry bean seeds (0.01–3.0 mg/kg), dry soya bean seeds (0.01–0.5 mg/kg), asparagus (0.01-5.0 mg/kg)
- valid (reduced validation) for the determination of fluazifop
- acid (II) in dry apple pomace (0.01–0.1 mg/kg), grapes (0.01-1.0 mg/kg), grape raisins (0.01–0.1 mg/kg), banana (0.01–0.5 mg/kg), spinach (0.01–0.1 mg/kg), soya bean oil (0.01–0.1 mg/kg), sugarbeet roots and tops (0.01–1.0 mg/kg), asparagus cooked (at 5.0 mg/kg only), wheat straw (0.05–0.1 mg/kg), wheat forage (at 0.02 mg/kg only), pecan nutmeat (0.01–0.1 mg/kg) and peanut oil (0.01–1.0 mg/kg)
- insufficiently validated for the determination of fluazifop acid (II) in orange, grape juice, lettuce, soya bean meal, soya bean hulls, carrot tops, carrot roots, sugarbeet sugar, sugarbeet dry pulp, sugarbeet molasses, wheat grain, almond nutmeat, almond hulls, walnut nutmeat.
- valid (reduced validation) for the determination of fluazifop-butyl in grapes (0.01–1.0 mg/kg; full validation), dry beans (0.01–1.0 mg/kg), dry soya bean seeds (0.01–1.0 mg/kg), sugarbeet roots (0.01–1.0 mg/kg) and peanut oil (0.01–1.0 mg/kg).
- insufficiently validated for the determination of fluazifop-butyl in orange, dry apple pomace, grape raisins, banana, head cabbage, tomato, lettuce, spinach, dry bean seeds, soya bean meal, soya bean hulls, soya bean oil, carrot tops, carrot roots, asparagus, asparagus cooked, wheat grain, wheat straw, wheat forage, pecan nutmeat, almond nutmeat, almond hulls, walnut nutmeat
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive)

The valid LOQ is 0.01 mg/kg.

Table 118 Validation results for method RR91-014B and its modifications

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Ref.
RR 91-014B original method									
dry apple pomace	0.01	0.01 (F) 0.1 (F)	88 90 90 93	86- 86-	2.4% 3.9%	3 3	ns	external std in solvent, linear, r^2=1.000 0.002-0.05 mg/L	PP5/0066; RR91-014B (validation)
grape raisin	0.01	0.01 (F) 0.1 (F)	122 129 130 132	111- 127-	7.8% 2.0%	3 3	ns	external std in solvent, linear, r^2=1.000 0.002-0.05 mg/L	PP5/0066; RR91-014B (validation)
banana	0.01	0.01 (F) 0.5 (F)	95 110 89 109	77- 73-	18% 9.4%	3 3	< 0.01 (10)	external std in solvent linear, r>0.999 0.002-0.05 mg//L	PP5/0454; RR 00-043B and 405683; RR 00-043B
head cabbage	0.01	0.01 (F) 2.0 (F)	84 100 91 98	76- 86-	11% 4.9%	5 5	<0.3LOQ (2)	external std in solvent, linear r>0.999; 0.005-1.0 mg/L	PP5/0080; CEMR-1159 (ILV)
tomato	0.01	0.01 (F) 0.5 (F)	90 91 86 92	85- 83-	4.4% 4.4%	5 5	<0.3LOQ (2)	external std in solvent, linear r>0.999; 0.005-1.0 mg/L	PP5/0080; CEMR-1159 (ILV)
lettuce	0.01	0.01 (F) 0.02 (F) 0.05 (F)	102 112 106 113	92- 98-	- - -	2 2 2	< 0.01 (2)	no data	PP5/0590; RJ2202B (rotational)

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Ref.
			98 100	95-					
spinach	0.01	0.01 (F) 0.1 (F)	89 92 102 108	86- 94-	3.4% 7.1%	3 3	ns	external std in solvent, linear, r^2=1.000 0.002-0.05 mg/L	PP5/0066; RR91-014B (validation)
dry bean seeds	0.01	0.01 (F) 3.0 (F)	98 108 78 87	89- 74-	7.4% 6.8%	5 5	<0.3LOQ (2)	external std in solvent, linear r>0.999; 0.005-1.0 mg/L	PP5/0080; CEMR-1159 (ILV)
dry soya bean seeds	0.01	0.01 (F) 2.0 (F)	93 95 88 94	91- 81-	2.0% 5.7%	5 5	<0.3LOQ (2)	external std in solvent, linear r>0.999; 0.005-1.0 mg/L	PP5/0080; CEMR-1159 (ILV)
dry soya bean seed	0.01	0.01 (F) 0.5 (F) 2.0 (F) 5.0 (F)	83 81 82 100 88 82	85- 71- - -	- 19 - -	2 3 1 1	< 0.01 (4)	external std, linearity ns	PP5/0368; RR 99-021B
Soya bean oil	0.01	0.01 (F) 0.1 (F)	72 73 75 76	70- 74-	2.1% 1.3%	3 3	ns	external std in solvent, linear, r^2=1.000 0.002-0.05 mg/L	PP5/0066; RR91-014B (validation)
carrot tops	0.01	0.01 (F) 0.02 (F)	73 83	- -	- -	1 1	< 0.01 (2)	no data	PP5/0590; RJ2202B (rotational)
carrot roots	0.01	0.01 (F) 0.05 (F)	115 102	- -	- -	1 1	< 0.01 (2)	no data	PP5/0590; RJ2202B (rotational)
Asparagus fresh or washed	0.01	0.01 (F) 0.1 (F) 1.0 (F) 5.0 (F)	92 117 98 108 102 115 90 109	78- 88- 88- 73-	15 - - 16	7 2 2 8	< 0.01 (2)	external std, linearity ns	PP5/0584; RR92-057B (processing)
Asparagus boiled, steamed or micro waved	0.01	0.01 (F) 5.0 (F)	100 113 78 80	88- 77-	- 2.0%	2 3	< 0.01 (1)	external std, linearity ns	PP5/0584; RR92-057B (processing)
wheat grain	0.01	0.02 (F) 0.05 (F) 0.1 (F)	89 103 96 109 106 133	75- 82- 80- 	- - -	2 2 2	< 0.01 (4)	no data	PP5/0590; RJ2202B (rotational)
wheat straw	0.05	0.05 (F) 0.1 (F)	86 92 90 100	71- 70-	12% 19%	4 3	< 0.05 (4)	no data	PP5/0590; RJ2202B (rotational)
wheat forage	0.01	0.01 (F) 0.02 (F) 0.05 (F)	70 74 81 100 84	65- 73- -	- 16% -	2 4 1	< 0.01 (4)	no data	PP5/0590; RJ2202B (rotational)
pecan nutmeat	0.01	0.01 (F) 0.1 (F)	101 117 109	88- 108-	15% 2.1%	3 3	ns	external std in solvent, linear, r^2=1.000	PP5/0066; RR91-014B (validation)

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Ref.
			112					0.002-0.05 mg/L	
almond, nutmeat	0.01	0.01 (F)	86 91	80-	-	2	< 0.01	external std linearity ns	PP5/0572; RR92-041B
almond, hulls	0.01	0.01 (F) 0.3 (F) 0.5 (F)	73 80 80 91 102	66- - 79-	- - n-	2 1 2	< 0.01	external std, linearity ns	PP5/0572 RR92-041B
walnut, nutmeat	0.01	0.01 (F)	107	-	-	1	< 0.01	external std, linearity ns	PP5/0582; RR92-009B
Modification A of RR 91-014B									
orange	0.01	0.01 (Fb) 1.0 (Fb)	81 85 68 72	78- 65-	- -	2 2	< 0.01 (8)	no data	406466; RR 00-063B
grapes	0.01	0.01 (Fb) 1.0 (Fb) 0.01 (F) 1.0 (F)	96 104 91 92 97 103 84 86	91- 91- 94- 82-	7.4% 0.83% 5.5% 2.0%	3 3 3 3	< 0.01 (12)	no data	406504; RR 00-062B (validation)
grapes	0.01	0.01 (Fb) 1.0 (Fb)	90 99 88 109	81- 74-	7.3% 15%	5 5	< 0.01 (12)	no data	406504; RR 00-062B
grapes	0.01	0.01 (F) 1.0 (F)	99 92	- -	-	1 1	< 0.01	external std in solvent, linearity ns	406498; RR 00-067B (processing)
grape juice	0.01	0.01 (F) 1.0 (F)	86 84	- -	-	1 1	< 0.01	external std in solvent, linearity ns	406498; RR 00-067B (processing)
grape raisins	0.01	0.01 (F) 1.0 (F)	93 86	- -	-	1 1	< 0.01	external std in solvent, linearity ns	406498; RR 00-067B (processing)
dry bean seeds	0.01	0.01 (Fb) 1.0 (Fb) 0.01 (F) 1.0 (F) 25 (F)	108 112 92 94 87 95 89 93 84	105- 89- 80- 87- -	3.4% 2.4% 8.5% 3.4% -	3 3 3 3 1	< 0.01 (12)	no data	PP5/1069; RR 00-061B (validation)
dry soya bean, seed	0.01	0.01 (Fb) 1.0 (Fb) 0.01 (F) 1.0 (F)	104 110 82 85 80 83 84 85	94- 78- 76- 84-	8.4% 4.2% 4.1% 0.7%	3 3 3 3	< 0.01 (17)	external std linearity ns	406507; RR 00-065B (validation)
dry soya bean seed	0.01	0.01 (Fb) 1.0 (Fb) 2.0 (Fb)	88 92 79 77	85- - -	- - -	2 1 1	< 0.01 (2)	external std linearity ns	406508; RR 00-069B (processing)
soya bean meal	0.01	0.01 (Fb) 1.0 (Fb) 50 (Fb)	89 79 100	- - -	- - -	1 1 1	< 0.01 (2)	external std linearity ns	406508; RR 00-069B (processing)
soya bean	0.01	0.01 (Fb)	88	84-	-	2	< 0.01 (2)	external std	406508;

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Ref.
hulls		1.0 (Fb) 2.0 (Fb)	92 87 74	- -	- -	1 1		linearity ns	RR 00-069B (processing)
soya bean oil	0.01	0.01 (Fb) 10.0 (Fb)	84 83	- -	- -	1 1	< 0.01 (2)	external std linearity ns	406508; RR 00-069B (processing)
Sugarbeet root	0.01	0.01 (Fb) 1.0 (Fb) 0.01 (F) 1.0 (F)	98 108 86 89 91 93 82 84	92- 80- 90- 79-	9.2% 5.7% 1.9% 3.5%	3 3 3 3	< 0.01	external std in solvent, linear r2>0.99 0.002-0.050 µg/mL	PP5/1070; RR 00-066B (validation)
Sugarbeet root	0.01	0.01 (F) 1.0 (F)	86 118 74 78	76- 67-	18.5% 5.9%	5 5	< 0.01	external std in untreated controls, method recoveries	PP5/1070; RR 00-066B
Sugarbeet top	0.01	0.01 (Fb) 1.0 (Fb) 0.01 (F) 1.0 (F)	99 107 76 81 81 84 78 79	91- 74- 74- 77-	7.8% 5.7% 1.9% 1.5%	3 3 3 3	< 0.01	external std in solvent, linear r2>0.99 0.002-0.050 µg/mL	PP5/1070; RR 00-066B (validation)
Sugarbeet top	0.01	0.01 (F) 10.0 (F)	97 110 77 86	80- 67-	12.7% 11.8%	5 5	< 0.01	external std in untreated controls, method recoveries	PP5/1070; RR 00-066B
Sugar beet root	0.01	0.01 (F) 1.0 (F)	102 82	- -	-	1 1	< 0.01	ext std in solvent, linear r2>0.99 0.002-0.050 µg/mL	406493; RR 00-070B (processing)
Sugar beet sugar (refined)	0.01	0.01 (F) 1.0 (F)	108 85	- -	-	1 1	< 0.01	extl std in solvent, linear r2>0.99 0.002-0.050 ug/mL	406493; RR 00-070B (processing)
Sugar beet dry pulp	0.01	0.01 (F) 1.0 (F)	92 74	- -	-	1 1	< 0.01	ext std in solvent, linear r2>0.99 0.002-0.050 ug/mL	406493; RR 00-070B (processing)
Sugar beet molasses	0.01	0.01 (F) 10.0 (F) 15.0 (F)	106 79 69	- - -	-	1 1 1	< 0.01	external std in solvent, linearity r2>0.99 0.002-0.050 ug/mL	406493; RR 00-070B (processing)
peanut oil	0.01	0.01 (Fb) 1.0 (Fb) 0.01 (F) 1.0 (F)	92 98 74 79 81 91 75 78	84- 70- 73- 73-	7.9% 5.9% 11% 4.0%	3 3 3 3	no data	no data	406508; RR 00-069B (validation)

GC-MS Method P-14.077

GC-MS method P-14.077 determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) and residues are expressed as fluazifop. The reported LOQ is 0.01 mg/kg. A full description of this method is not available [Syngenta, 2015, Response to questions 02].

Method P-14.077 (version 01) was used in a processing studies on potatoes [Weeren, 1994, PP5/0102, report AZ13430/93 = Pelz, 1994, PP5_50062, report AZ13430/93]. A summary description was available in this report. Samples were hydrolysed with NaOH to hydrolyse any ester or acid conjugates to fluazifop (details not indicated). The mixture is acidified using sulphuric acid and extracted with a mixture of acetone/dichloromethane. The organic extract was evaporated to dryness and cleaned-up by gel permeation chouromatography on Bio Beads S-X3 polystyrene gel, using a mixture of cyclohexane/ethylacetate (1+1, v/v) as eluant. The eluate is evaporated to dryness. The residue is redissolved in acetone and methyled by ion pair alkylation with tetrabutylammonium hydroxide (TBAH) and iodomethane. The final determination was by GC-MS at m/z = 341 (quantification) and m/z 282 and 254 (confirmation) using external standards for fluazifop-methyl in n-hexane. Validation results are shown in Table 119.

No radiovalidation was conducted.

Reviewer's conclusion: GC-MS method P14.077 is considered:

- valid (reduced validation) for the determination of fluazifop acid (II) in potatoes (0.1 mg/kg only).
- insufficiently validated for the determination of fluazifop acid (II) in potato peels, cooked potatoes, potato crisps and dried potatoes.
- insufficiently validated for the determination of fluazifop-butyl in potatoes, potato peels, cooked potatoes, potato crisps and dried potatoes.)
- not valid for the determination of fluazifop (II) conjugates (no radiovalidation)

The valid LOQ is 0.01 mg/kg.

Table 119 Validation results for GC-MS method P14.077

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
potato (RAC)	0.01	0.01 (F) 0.1 (F) 0.01 (Fb) 0.1 (Fb)	90 85 101 82 74	- 72- - -	- 17% - -	1 4 1 1	< 0.01 (2)	external std calibration ns	PP5/0102; AZ13430/93 (processing)
potato peel	0.01	0.01 (Fb) 0.1 (F)	69 102	- -	- -	1	< 0.01–0.01 (2)	external std calibration ns	PP5/0102; AZ13430/93 (processing)
potato cooked without peel	0.01	0.01 (F) 0.1 (Fb)	109 102	- -	- -	1 1	< 0.01 (2)	external std calibration ns	PP5/0102; AZ13430/93 (processing)
potato chips [a]	0.01	0.01 (F) 0.1 (F)	101 85	- -	- -	1 1	< 0.01–0.01 (2)	external std calibration ns	PP5/0102; AZ13430/93 (processing)
potato dried	0.01	0.01 (Fb) 0.1 (F)	87 77	- -	- -	1 1	< 0.01 (2)	external std calibration ns	PP5/0102; AZ13430/93 (processing)

[a] Since this concerns thinly sliced potatoes, the reviewer assumes that chips refer to crisps and not to French fries.

NMR, HPLC-MS/MS and GC-MS Method PPRAM 122 and its modifications

NMR, HPLC-MS/MS and GC-MS Method PPRAM 122 and its modifications determine total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) and residues are expressed as fluazifop. The reported LOQ is 0.02–0.06 mg/kg for NMR detection, 0.01 mg/kg for HPLC-MS/MS detection and 0.01 mg/kg for GC-MS detection.

NMR Method PPRAM 122 (15 October 1987) is described by [Hayward and Atreya, 1987, 462761, report PPRAM 122]. Oil or oily commodities (e.g. peanuts, oilseed rape seeds, 20 g) are refluxed for 1 hour in 0.2 M NaOH in methanol to extract the free fluazifop acid (II) and hydrolyse any ester or acid conjugates to fluazifop. After centrifugation, the extract is diluted with water, acidified to pH<1 and partitioned into diethylether. The analyte is back-partitioned into 1% aqueous sodium hydrogen carbonate solution to leave behind the majority of the oil co-extractives in the diethyl ether. Further oily co-extractives were separated by partitioning into n-hexane. The remaining aqueous extract is acidified to pH<1 and then partitioned with dichloromethane. The dichloromethane extract is evaporated to dryness and redissolved in a mixture of acetone/deuterated acetone for analysis by ^{19}F-NMR Fourier Transformed. Halosafen (5-[2-chloro-4-trifluoromethyl-6-fluoro-phenoxy]-N-ethylsulphonyl-2-nitrobenzamide or 5-[2-chloro-α,α,α,6-tetrafluoro-p-tolyloxy]-N-ethylsulphonyl-2-nitrobenzamide, MW 471.5) is added to the final extract as internal standard for calibration.

GC-MS may be used for confirmation of residues of fluazifop. An aliquot of the dichloromethane extract is evparated to dryness. Diazomethane is added and left to react for 30 min at ambient temperature. The mixture is evaporated to dryness, redissolved in hexane. Derivatised fluazifop acid (II) is determined by GC-MS (m/z 341, 282). Calibration by matrix matched standards for fluazifop-methyl.

A limited validation report is available for NMR method PPRAM 122 [Mak and Atreya, 1987, no code available, report PPRAM 122 addendum; Bussey, 1990, 407594, RR 90-098B]. Method PPRAM 122 was used in a storage stability study on peanuts [M4841B]. Validation results are shown in Table 120.

NMR and GC-MS Method RAM 122/02 (20 January 1994) is described by [Bolygo and Kipps, 1994, PP9/0357, SOP RAM122/02]. Halosafen (5-[2-chloro-4-trifluoromethyl-6-fluoro-phenoxy]-N-ethylsulphonyl-2-nitrobenzamide or 5-[2-chloro-α,α,α,6-tetrafluoro-p-tolyloxy]-N-ethylsulphonyl-2-nitrobenzamide, MW 471.5) is added to the final extract as internal standard for calibration. A limited validation report is available. Method RAM 122/02 (NMR detection) was used in a processing study on oilseed rape [RJ1684B]. Validation results are shown in Table 120.

NMR and GC-MS Method RAM 122/04 (11 August 1997) is described by [Bolygo, 1998, PP9/0391, SOP RAM 122/04] and is identical to method RAM 122/02. Method RAM 122/04 was used in a processing study with soya bean [RJ2914B], however, final detection was with HPLC-MS/MS instead of NMR. Validation results are shown in Table 120.

NMR and GC-MS and HPLC-MS/MS Method RAM 122/05 (21 September 2000) is described by [Bolygo *et al*., 2000, PP9/0188, SOP RAM 122/05]. The method is identical to RAM 122/02 and RAM 122/04, except for the following revisions. An alternative final determination technique (HPLC-MS/MS) is added. The final dichloromethane extract is evaporated to dryness and redissolved in mobile phase acetonitrile/0.4% aqueous acetic acid, 10/90, v/v) for analysis by HPLC-MS/MS (m/z 328 to m/z 282). Calibration by matrix matched standards for fluazifop acid (II). An alternative methylation is added for GC-MS confirmation, using 3 M HCl/methanol as derivatising agent. An aliquot of the dichloromethane extract is evaporated to dryness. A 3 M HCl/methanol derivatising reagent is added and left to react for 30 min at 60 ºC. After cooling, water is added and the derivatised fluazifop acid (II) is partitioned into hexane. The hexane phase of either diazomethane methylation or 3M HCl/methanol methylation is dried over anhydrous sodium sulphate and then cleaned-up using a Si cartridge. The derivatised fluazifop acid (II) is eluted using hexane/ethyl acetate (80:20, v/v) and determined in the eluent by GC-MS (m/z 341, 282, 254). Calibration by matrix matched standards for fluazifop-methyl (fluazifop acid (II) is derivatised in situ and then added to a control sample that has undergone the extraction and clean-up procedure). HPLC-MS/MS detector response was found to be linear in the range 0.005-1 mg/L using matrix matched standards [Bolygo *et al*., 2000, PP9/0188, SOP RAM 122/05]. Method RAM 122/05 (HPLC-MS/MS detection) was used in a storage stability study with soya bean oil [RJ3087B] and processing studies on soya bean [RJ3149B, RJ3208B]. Validation results are shown in Table 120.

No radiovalidation was conducted.

Reviewer's conclusion: NMR Method PPRAM 122 and its modifications is considered:

- valid (reduced validation) for the determination of fluazifop acid (II) in oilseed rape oil (0.5 mg/kg only) and peanut kernels (at 0.4 mg/kg only).
- insufficiently validated for the determination of fluazifop acid (II) in lettuce, spinach, oilseed rape seeds.
- valid for the determination of fluazifop-butyl (as shown by peanut kernels) in the same commodities as for fluazifop acid (II)
- not valid for the determination of fluazifop (II) conjugates (no radiovalidation)

The valid LOQ is 0.4 mg/kg (too high RSD at 0.1 mg/kg, no validations below 0.4 mg/kg)

HPLC-MS/MS Method 122/04 and 122/05 is considered:

- valid (full validation) for the determination of fluazifop acid (II) in soya bean oil (0.01–0.25 mg/kg)
- not valid for the determination of fluazifop-butyl (no validations)
- not valid for the determination of fluazifop (II) conjugates (no radiovalidation)

The valid LOQ is 0.01 mg/kg (no validations below this point).

GC-MS Method PPRAM 122 and its modifications is considered :

- not valid for the determination of fluazifop acid (II) (no validations)
- not valid for the determination of fluazifop-butyl (no validations)
- not valid for the determination of fluazifop (II) conjugates (no radiovalidation)

Table 120 Validation results for method PPRAM 122 and its modifications

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
NMR method PPRAM 122 (no internal standard)									
lettuce	0.05	0.1 (ns) 0.2 (ns)	83 69	- -	ns	ns	< 0.05 (1)	NMR; linearity ns	no code; PPRAM 122 add (validation)
spinach	0.05	0.1 (ns) 0.2 (ns)	92 82	- -	ns	ns	< 0.05 (1)	NMR; linearity ns	no code; PPRAM 122 add (validation)
peanut kernels	ns	0.2 (F)	85	-	-	1	no data	no data	407594; RR90-098B (validation)
peanut kernels	0.06	0.4 (Fb)	92	89-96	3.9%	3	< 0.06 (1)	NMR; linearity ns	462746; M4841B (stor stab)
NMR method RAM 122/02, RAM 122/04 and RAM 122/05 (with internal standard)									
oilseed rape seed	0.02	0.05 (F) 0.1 (F) 0.5 (F)	103 82 86	- - -	- - -	1 1 1	ns	NMR; linearity ns	PP9/0357 RAM 122/02 (validation)
oilseed rape oil	0.02	0.02 (F) 0.05 (F) 0.1 (F) 0.5 (F)	99 100 98 90	- 98-103 90-105 81-100	- - - -	1 2 2 2	ns	NMR linearity ns	PP9/0357 RAM 122/02 (validation)
oilseed rape oil	0.02	0.1 (F) 0.2 (F) 0.5 (F)	95 98 99	71-119 - 90-117	24% - 11%	4 1 5	< 0.02 (2)	NMR linearity ns	PP5/1105; RJ1684B processing
HPLC-MS/MS method RAM 122/04 and 122/05									
soya bean oil	0.01	0.25 (F)	85	68-102	12%	11	< 0.01 (1)	HPLC-MS-MS	PP5/1281; RJ3087B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
								linearity ns	storage stab
soya bean crude oil	0.01	0.01 (F) 0.1 (F)	74 76	65-77 70-81	6.6% 5.3%	5 5	< 0.01	HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
Soya bean crude oil	0.01	0.01 (F) 1.0 (F) 10.0 (F)	68 78 97	- - -	- - -	1 1 1	< 0.01	HPLC-MS-MS Linearity ns	PP5_50435, RJ2914B (processing)
Soya bean crude oil	0.01	0.05 (F) 0.1 (F)	94 88	92-98 76-96	3.4% 12%	3 3	< 0.01	HPLC-MS/MS linearity ns	PP5/1122, RJ3208B (processing)
soya refined oil	0.01	0.01 (F) 0.1 (F)	85 79	74-93 73-89	8.1% 8.0%	5 5	< 0.01	HPLC-MS/MS linearity ns	PP5/1144; RJ3149B processing
Soya refined oil	0.01	0.01 (F) 1.0 (F) 5.0 (F)	99 111 108	- - -	- - -	1 1 1	< 0.01	HPLC-MS-MS Linearity ns	PP5_50435, RJ2914B processing
Soya refined oil	0.01	0.05 (F) 0.1 (F)	78 90	74-85 89-91	7.8% 1.1%	3 3	< 0.01	HPLC-MS/MS linearity ns	PP5/1122, RJ3208B
Soya residual oil	0.01	0.05 (F) 0.1 (F)	105 112	- -	- -	1 1	< 0.01	HPLC-MS/MS linearity ns	PP5/1144 RJ3149B processing
Soya fatty acid	0.01	0.01 (F)	75	-	-	1	< 0.01	HPLC-MS-MS Linearity ns	PP5-50435, RJ2914B processing

F, fortification with fluazifop acid (II)

^{19}F-NMR method (A)RAM 197 and its modifications, non-GLP

^{19}F-NMR method (A)RAM 197 and it modifications determine total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant commodities as one single analyte (common moiety) and residues are expressed as fluazifop. The reported LOQ is 0.05-0.07 mg/kg depending on the matrix.

NMR Method ARAM 197 (6 June 1991) is described by [Davy *et al.*, 1991, PP9/0356, ARAM 197]. Oily and dry crop commodities (e.g. oilseeds, 10 g) are soaked for a minimum of 2 hours in 1 M HCl prior to extraction. Crops with high water content (e.g. roots and tubers, 20 g) are macerated with acetonitrile/concentrated HCl (98:2, v/v). Soaked crops are macerated with acetonitrile and celite. An internal standard halosafen (5-[2-chloro-4-trifluoromethyl-6-fluoro-phenoxy]-N-ethylsulphonyl-2-nitrobenzamide or 5-[2-chloro-α,α,α,6-tetrafluoro-p-tolyloxy]-N-ethylsulphonyl-2-nitrobenzamide) is added before maceration. After filtration, the acetonitrile is removed by evaporation until the aqueous phase remains. Any fluazifop-butyl or fluazifop (II) conjugates in extract are converted to fluazifop acid (II) by hydrolysis with 6 M HCl (1 hour, 60 ºC). The samples are then diluted with water (pH < 1) and partitioned into dichloromethane. Where heavy emulsions are experienced (e.g. oilseed rape seeds) samples were passed thourough diatomaceous earth (e.g. Celite). Where high levels of plant co-extractives are present (e.g. oilseeds, oil, oilseeds meal, cabbage) an additional back-partitioning with 1% aqueous sodium hydrogen carbonate solution is employed to separate the oil and plant co-extractives from the analyte. The aqueous extract is acidified to pH <1 and fluazifop acid (II) are again partitioned into dichloromethane. The dichloromethane is evaporated to dryness and redissolved in acetone. Any particulate matter is removed by centrifugation or filtration. The samples are again evaporated to dryness and reconstituted in a mixture of acetone:deuterated acetone (1:1, v/v) for analysis by ^{19}F NMR. Calibration by internal standardisation.

NMR Method RAM 197/01 is summarized by [Patel and Robinson, 1994, PP5/0130, report RJ1583B] and Method RAM 197/02 (21 January 1994) is described by [Davy *et al.*, 1994, PP9/0358, SOP RAM 197/02]. Both methods are identical to method ARAM 197.

A method validation report is available [Davy *et al.*, 1991, PP9/0356, ARAM 197]. The NMR detector response ratio for fluazifop/internal standard was linear at levels between 0.1-10 mg/kg fluazifop and 0.5 mg/kg internal standard. Validation results are shown in Table 121.

NMR Method ARAM 197 was used in supervised residue trials on summer squash [RJ1085B], green soya bean forage [TMJ3065B], potatoes [RJ1405B], sugar beets (roots and tops) [RJ1424B] and oilseed rape (seeds, forage) [RJ1456B, RJ1846B] and alfalfa [RJ1068B, RJ1338B]. Method RAM 197/01 was used in supervised residue trials on head cabbage [RJ1583B], oilseed rape (seeds, forage) [RJ1660B] and sunflower (seeds, forage) [RJ1656B]. Method RAM 197/02 was used in supervised trials on strawberries [RJ1817B], dry bean seeds [RJ1894B], carrots [RJ1884B] and processing studies on oilseed rape [RJ1684B]. Method validation results extracted from supervised residue trials are summarized in Table 121.

Modification A of method ARAM 197 is used for dry soya bean (seeds, fodder) [TMJ3065B]. Solvent volumes are doubled for fodder. With both fodder and seed the filter cake and pads were re-extracted with acetonitrile and combining the extracts.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: Method ARAM 197 and RAM 197/01 and its modifications are considered:

- valid (reduced validation) for the determination of fluazifop acid (II) in kale (0.5 mg/kg only), green pea seeds (0.5 mg/kg only), dry pea seeds (0.5 mg/kg only), dry pea straw (0.5 mg/kg only), dry bean seeds (0.5 mg/kg only), dry lentil seeds (0.5 mg/kg only), turnip roots (0.5 mg/kg only), sugarbeet tops (0.1–0.5 mg/kg), sugarbeet roots (0.1–0.5 mg/kg), carrot roots (0.2–0.5 mg/kg), oilseed rape seeds (0.1 mg/kg only), oilseed rape forage (1 mg/kg only), sunflower seeds (5.0 mg/kg only).
- insufficiently validated for the determination of fluazifop acid (II) in strawberries, summer squash, head cabbage, soya bean forage, soya bean fodder, dry soya been seeds, turnip tops, linseed, oilseed rape meal, sunflower forage, medic pasture
- not valid for the determination of fluazifop-butyl (no validation)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive))

The valid LOQ is 0.05 mg/kg (no validations below this point).

Table 121 Validation results for NMR method ARAM 197 and RAM197/01 and its modifications

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Ref.
NMR method ARAM 197									
summer squash	0.05	0.05 (F) 0.5 (F) 0.5 (IS)	100 107 112 126 109 139	92- 98- 63-	- - 18%	2 2 12	< 0.05 (4)	internal std linearity ns	PP5/0422; RJ1085B
kale	0.05	0.5 (F) 0.5 (IS)	92 108 92 102	62- 62-	22% 13%	3 10	ns	no data	PP9/0356; ARAM 197; validation
green pea seeds	0.05	0.5 (F) 0.5 (IS)	87 82	83-92 67-96	5.4% 10%	4 16	ns	no data	PP9/0356; ARAM 197; validation
dry pea seeds	0.05	0.5 (F) 0.5 (IS)	83 108	44-	18% 19%	14 36	ns	no data	PP9/0356; ARAM 197;

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Ref.
			85 118	46-					validation
dry pea straw	0.05	0.5 (F) 0.5 (IS)	81 95 128	71-92 58-	13% 20%	3 9	ns	no data	PP9/0356; ARAM 197; validation
dry lentil seeds	0.05	0.5 (F)	87	83-94	6.7%	3	ns	no data	PP9/0356; ARAM 197; validation
green soya bean forage	0.05	0.05 (F) 0.1 (F) 0.2 (F) 0.5 (F) 0.5 (IS)	117 124 115 110 114 107 118	- - - 107- 96-	- - - - 5.1%	1 1 1 2 21	ns	no data	PP5/1031; TMJ3065B
potatoes	0.05	0.05 (F) 0.1 (F) 0.5 (F) 0.5 (IS)	76 90 97 101 98 118	62-89 79-99 92- 72-	- 9.4% 3.8% 12%	2 4 5 11	< 0.05 (6)	no data	PP5/0095; RJ1405B
turnip root	0.05	0.5 (F) 0.5 (IS)	94 84 104	94-95 69-	- 15%	2 9	ns	no data	PP9/0356; ARAM 197; validation
turnip tops	0.05	0.5 (F) 0.5 (IS)	100 101 111 131	99- 65-	- 21%	2 7	ns	no data	PP9/0356; ARAM 197; validation
sugarbeet tops	0.05	0.5 (IS) 1 (IS) 5 (IS) 0.05 (F) 0.1 (F) 0.5 (F) 1 (F) 5 (F)	98 109 81 89 108 116 98 103 100 102 99 109	75- - - 99- 94- 98- - -	11% - - - 4.7% 1.7% - -	9 1 1 2 3 4 1 1	< 0.05 (17)	by internal std linearity ns	PP5/0098; RJ1424B
sugarbeet roots	0.05	0.5 (IS) 0.05 (F) 0.1 (F) 0.5 (F)	94 107 87 102 86 97 111	80- 67- 75-99 84-	7.0% 21% 12% 10%	12 3 4 5	< 0.05 (15)	by internal std linearity ns	PP5/0098; RJ1424B
linseed	0.05	1.0 (F)	78	76-81	-	2	ns	no data	PP9/0356; ARAM 197; validation
oilseed rape seed	0.05	0.5 (F)	92 123	64-	26%	7	ns	no data	PP9/0356; ARAM 197; validation
oilseed rape seed	0.05	0.1 (F) 0.5 (F) 0.5 (IS)	88 85 91	- 76-94 86-98	- - 6.6%	1 2 3	< 0.05 (3); 0.23 (1)	no data	PP5/0564; RJ1456B
Alfalfa forage	0.05	0.5 (IS) 0.5 (F)	82 101 88	61- 84-94	13% 5.1%	18 4	< 0.05 (3); 0.24 (1); 0.27 (1), 0.49 (1); 2.2 (1)	no data	PP5/0521; RJ1068B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Ref.
Alfalfa forage	0.05	0.05 (F) 0.1 (F) 0.5 (F)	91 121 92 101	- - 84-	- - 9.4%	1 1 3	< 0.05 (6)	No data	No code; RJ1338B
NMR method ARAM 197 modification A									
dry soya bean fodder	0.05	0.1 (F) 0.5 (F) 0.5 (IS)	92 75 81	- 74-76 77-84	- - 2.2%	1 2 6	ns	no data	PP5/1031; TMJ3065B
dry soya bean seeds	0.05	0.05 (F) 0.5 (F) 0.5 (IS)	89 82 79	- 78-85 74-88	- - 4.9%	1 2 6	ns	no data	PP5/1031 TMJ3065B
NMR method RAM 197/01									
head cabbage	0.05	2.0 (IS) 1.0 (F) int 2.0 (F) int 0.1 (F) ext 1.0 (F) ext	90 106 94 99 88 93	63- - - - -	29% - - - - 	3 1 1 1 1 	< 0.05 (5)	by external or internal std [a] linearity ns	PP5/0130; RJ1583B
oilseed rape forage	0.05	1 (IS) 5 (IS) 10 (IS) 0.05 (F) 1 (F) 5 (F) 10 (F)	90 82 77 100 101 91 81 76	85-95 - - 98- 85-97 - -	5.0% - - - 6.6% - - 	5 1 1 2 3 1 1 	< 0.05 (11)	by internal std linearity ns	PP5/0217; RJ1660B
oilseed rape seed	0.05	0.05 (F) 0.5 (F) 0.1 (F)	109 80 95	- - -	- - -	1 1 1	< 0.05 (2)	by internal std linearity ns	PP5/0217; RJ1660B
sunflower seed	0.05	1.0 (IS) 0.1 (F) 0.5 (F) 1.0 (F) 2.0 (F) 5.0 (F)	82 92 102 106 110 91 103 118	81-83 92-92 - 101- - 83-	0.9% - - - - 14% 	8 2 1 2 1 4 	< 0.05 (4)	by internal std linearity ns	PP5/0218; RJ1656B
sunflower forage	0.05	1.0 (IS)	99 121	75-	11%	32	< 0.05 (4)	by internal std linearity ns	PP5/0218; RJ1656B
NMR method RAM 197/02									
strawberries	0.05	0.5 (IS) 0.1 (F) 0.2 (F) 0.5 (F)	87 109 88 83 90	71- 82-94 - -	8.1% - - - 	21 2 1 1 	< 0.05 (4)	internal std linearity ns	PP5/0196; RJ1817B
dry bean seed	0.07	0.50 (IS) 0.25 (F) 0.50 (F)	92 80 86	90-95 - -	2.2% - -	4 1 1	< 0.07-0.09 (4)	by internal std linearity ns	PP5/0416; RJ1894B
carrots	0.05	0.50 (IS) 0.1 (F) 0.2 (F) 0.5 (F)	93 108 89 96 101 98	79- 86-92 90- -	9.7% - 5.8% - 	20 2 3 1 	< 0.05 (4)	by internal std linearity ns	PP5/0103; RJ1884B
oilseed rape seed	0.05	0.1 (F) 0.5 (F) 1.0 (F)	117 133 109 116 90	109- 102- -	12% - - 	3 2 1 	< 0.05 (2)	by internal std linearity ns	PP5/1105; RJ1684B (processing)
Oilseed rape seed	0.05	0.5 (F) 1.0 (F)	88 79	84-91 -	- -	2 1	< 0.05	by internal std linearity ns	PP5/0220; RJ1846B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Ref.
oilseed rape meal	0.05	0.5 (F)	74	-	-	2	< 0.05 (2)	by internal std linearity ns	PP5/1105; RJ1684B (processing)
Oilseed rape forage	0.05	1.0 (F) 2.5 (F) 5.0 (F) 8.0 (F) 10 (F) 15 (F)	84 90 91 110 118 121 97 72	- - 72- 114- - -	- - 21% - - - 	1 1 3 1 1 1 	< 0.05	By internal std Linearity ns	PP5/0220; RJ1846B

[a] Internal standard if a single dichloromethane partition was performed. External standard if an additional sodium bicarbonate partition was performed. The sodium bicarbonate partition has the effect of reducing the internal standard recovery (to 55%).

HPLC-MS/MS and HPLC-UV Method RAM 287/01 and it modifications

HPLC-MS-MS and HPLC-UV method RAM 287/01 and it modifications determine total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) in crop commodities as one single analyte (common moiety) and residues are expressed as fluazifop. The reported LOQ is 0.01–0.05 mg/kg for HPLC-MS/MS detection and 0.05–0.2 mg/kg for HPLC-UV detection depending on the matrices.

Method RAM 287/01 (17 December 1996) is described by [Walter, 1996, PP5/0079, report RJ2172B]. When calculating the required amount of extraction solvent, the natural water content of the plant commodities is taken into account. Crop commodities with ≥ 60% water content (10 g) are extracted by maceration in acetonitrile: concentrated HCl (98:2 v/v). Dry or oily crop commodities (olives, cereal grains, oilseeds, dry pulses, tree nuts) (10 g) are soaked in 1M HCl for at least 2 hours followed by maceration in acetonitrile (acetonitrile : 1M HCl = 50:50 v/v). From an aliquot of the extract, the acetonitrile is removed by evaporation and the aqueous remainder is hydrolysed at 6 M HCl (1 hour 60 ºC) to convert any ester or acid conjugates to fluazifop acid (II). Endogenous plant co-extractives are separated off by adsorption chouromatographic clean-up using C_2 (End Capped) SPE cartridges followed by silica SPE extraction cartridges. Fluazifop acid (II) is eluted with dichloromethane containing 5% acetone from the C_2 SPE cartridge onto a Si SPE cartridge. Fluazifop acid (II) is eluted with methanol from the Si SPE cartridge. The eluate is evaporated to dryness and resuspended in HPLC mobile phase. The extracts are analysed by HPLC-MS-MS (m/z 328 to 282) or HPLC-UV (270 nm). The residues are quantified against a fluazifop acid (II) external standard in the appropriate matrix. Results obtained by HPLC-UV can be confirmed by HPLC-MS/MS and vice versa.

A validation study is available [Walter, 1996, PP5/0079, report RJ2172B]. Results are shown in Table 122. Since all supervised residue trials have been conducted with HPLC-MS-MS detection, validation results for HPLC-UV detection were not summarized.

Method RAM 287/02 (26 June 1998) is described by [Bolygo, 1998, PP5/0067, SOP RAM 287/02]. Method RAM 287/02 is identical to method RAM 287/01 and therefore validation results for each method can be exchanged.

Method RAM 287/01 and 287/02 were used in supervised trials on lemons [RJ2241B], apples [RJ2319B], raspberries [RJ3210B], strawberries [RJ3074B, CEMR-2306], grapes [RJ2636B], olives [RJ2634B], bulb onions [RJ2728B, RJ2827B], leeks [RJ3278B, 02-7035, 02-7083, 02-21401, 03-7029, CEMR-2687], head cabbage [RJ2306B, RJ2312B, RJ2645B, RJ2794B, RJ2834B, RJ2992B, RJ3232B, 03-7068, 03-7076], cucumber [RJ2265B, RJ2380B, RJ2507B, RJ3058B], summer squash [RJ2265B], tomatoes [RJ2268B, RJ2370B, RJ2657B, RJ2780B], kale [RJ2654B, RJ2759B], lettuce [RJ2302B, RJ2363B, RJ2631B, RJ2782B, RJ2786B], spinach [RJ2632B], green beans (pods, haulms) [RJ2287B, RJ2290B, RJ2611B, RJ2629B, RJ2993B, RJ3294B, RJ3299B, CEMR-3014, T009248-07-

REG], green peas (seeds, pods, forage) [RJ2254B, RJ3336B, 03-7031, 03-7032, CEMR-3009, CEMR-3012], dry bean seeds [RJ2610B, RJ2826B, RJ2994B], dry fava beans (seeds, haulms) [CEMR-3008], dry peas (seeds, straw) [RJ2510B, RJ2785B, RJ3209B, RJ3211B, RJ3266B, RJ3300B, 03-7058, 03-7059, T009247-07-REG], dry soya bean seeds [RJ2368B, RJ2405B, RJ2442B, RJ2481B, RJ2720B, RJ2781B, 03-7026, 03-7072, 03-7073, 03-7074], carrots [RJ2638B, RJ2659B, RJ2772B, RJ3065B], celeriac [RJ2630B, RJ2804B], potatoes [gpo11501, gpo31501, gpo41501, gpo91501, gpo023103, gpo079002, 02-7044, 02-7045, 02-7068, 02-7069, 03-7027, 03-7028, 03-7030, 03-7037, 03-7038, 03-7047, 03-7048, 03-7056; 03-7057, 03-7079, 03-7080, CEMR-2309, CEMR-2688, CEMR-2689, CEMR-3374, CEMR-3375, RJ3200B; RJ3222B, RJ3295B], sugar beets (roots, tops) [RJ2779B, RJ2553B, RJ2833B, RJ2995B, CEMR-2310, gsb064002, gsb064202], asparagus [RJ2281B, RJ2673B, RJ2701B], witloof (roots, sprouts) [RJ2646B], hazelnuts [RJ2656B], oilseed rape seeds [RJ2758B, RJ2765B, RJ2766B, RJ2771B, RJ2806B, , 02-7015, 03-7004, 03-7005], sunflower seeds [RJ2284B, RJ2303B, RJ2726B, RJ2940B, RJ3234B, RJ3252B, CEMR-2690], and grass (forage, hay) [RJ2496B] in a storage stability study [RJ3087B] and in a processing studies with soya bean [RJ2914B, RJ3149B, RJ3208B].Validation results are listed in Table 122.

Modification A of method RAM 287/02. Dry pea straw [CEMR-3373] and grass hay [RJ2764B] were extracted by homogenisation with acetonitrile: concentrated HCl (98:2) without the normal soaking step. Validation results are shown in Table 122.

Modification B of method RAM 287/02. The hydrolysate of green pea forage [CEMR-3009] and dry peas seeds [T009247-07-REG] was diluted with mobile phase directly after the hydrolysis stage without clean-up. Validation results are shown in Table 122.

Modification C of method RAM 287/02. Dry pea seeds [CEMR-3373] were extracted by homogenisation with acetonitrile: concentrated HCl (98:2), without the normal soaking step. The extracts were diluted with mobile phase directly after the hydrolysis stage without clean-up. Validation results are shown in Table 122.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer’s conclusion: HPLC-MS/MS Method RAM 287/01 and its modifications are considered:

- valid (full validation) for the determination of fluazifop acid (II) in raspberries (0.01–0.10 mg/kg), head cabbage (0.05-2.0 mg/kg), tomatoes (0.01-2.0 mg/kg), lettuce (0.1–0.5 mg/kg), green pea forage (0.01–0.1 mg/kg), dry pea straw (0.01–0.1 mg/kg), soya bean meal (0.01–0.25 mg/kg), soya bean flour (0.01–0.1 mg/kg), soya bean flocs (0.01–0.1 mg/kg) and potatoes (0.01–0.1 mg/kg);
- valid (reduced validation) for the determination of fluazifop acid (II) in orange (0.01–0.05 mg/kg), apple (0.05-0.1 mg/kg), bulb onions (0.05 mg/kg only), cucumber (0.05-0.1 mg/kg), kale (0.1-1.0 mg/kg), spinach (0.05-0.1 mg/kg), green peas with pods (0.01–0.1 mg/kg), dry bean seeds (0.1-2.5 mg/kg), dry soya bean seeds (0.05-5.0 mg/kg), soya bean hulls (at 0.25 mg/kg only), sugarbeet roots (at 0.1 mg/kg only), sugarbeet tops (at 0.1 mg/kg only)
- insufficiently validated for the determination of fluazifop acid (II) in lemon, grapes, strawberries, olives, summer squash, green pea seeds, green beans with pods, green bean haulms, dry fava bean seeds, dry fava bean haulms, dry pea seeds, witloof roots, witloof endives, carrots, celeriac, leeks, asparagus, hazelnuts, oilseed rape seeds, sunflower seeds, grass, grass hay
- not valid for the determination of fluazifop-butyl (no validation)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive)

- modification C, where the soaking step has been omitted, is considered not valid for dry pea seeds, since a processing study with dry peas [Devine, 2013, A12791B_11068, report CEMR-5037-REG] has shown that soaking is essential for complete hydrolysis of the fluazifop (II) conjugates

The valid LOQ is 0.01 mg/kg.

Table 122 Validation results for the determination of total fluazifop using method 287/01 and its modifications

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
HPLC MS/MS method 287/01									
oranges	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.2 (F) 0.5 (F)	101 110 89 95 98 100 94 96 100 102	93- 83- 96- 92- 99-	6.9% 5.8% - - -	4 4 2 2 2	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, not shown 0.005-1.0 mg/L	PP5/0079; RJ2172B validation
lemon	0.01	0.1 (F)	63 64	62-	-	2	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0197; RJ2241B
apples	0.01	0.1 (F)	94 110	75-	16%	4	< 0.01– 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0198; RJ2319B
apples	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.2 (F) 0.5 (F)	94 101 92 95 96 96 81 92 96 96	91- 90- 95- 70- 95-	5.1% 2.7% - - -	4 4 2 2 2	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, r>0.999 0.005-1.0 mg/L	PP5/0079; RJ2172B validation
grapes	0.01	0.05 (F) 0.1 (F)	80 86 76 82	75- 71-	- -	2 2	< 0.01 (4)	HPLC-MS/MS linearity ns	PP5/0189; RJ2636B
olives	0.01	0.05 (F) 0.5 (F)	98 77	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0188; RJ2634B
bulb onions	0.01	0.05 (F) 0.5 (F)	101 106	- -	- -	1 1	< 0.01 (4)	HPLC-MS/MS linearity ns	PP5/0295; RJ2728B
head cabbage	0.01	0.1 (F)	95 101	88-	-	2	< 0.01 (5); 0.01 (1)	HPLC-MS/MS linearity ns	PP5/0135; RJ2306B
head cabbage	0.01	0.1 (F) 0.3 (F)	107 112 111 118	102- 103-	- -	2 2	< 0.01 (4) 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0133; RJ2312B
head cabbage	0.01	0.05 (F) 0.5 (F)	99 103	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0137; RJ2645B
head cabbage	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.2 (F) 0.5 (F) 1.0 (F) 2.0 (F)	103 119 92 97 94 97 89 90 104	90- 84- 89- 88- 103-	12% 6.6% 3.8% - - - -	4 4 4 2 2 2 2	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, not shown 0.005-1.0 mg/L	PP5/0079; RJ2172B validation

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
			104 92 94	91-92 93-95					
cucumber	0.01	0.1 (F) 0.25 (F) 0.5 (F)	80 84 83 92	75- - -	- - -	2 1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0168; RJ2265B
cucumber	0.01	0.1 (F)	100 100	99-	-	2	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0171; RJ2380B
cucumber	0.01	0.05 (F) 0.10 (F) 0.50 (F)	101 110 98 113 111	96- 86-	10% 7.5% -	3 4 1	< 0.01 (5)	HPLC-MS/MS linearity ns	PP5/0173; RJ2507B
summer squash	0.01	0.1 (F) 0.25 (F) 0.5 (F)	80 84 83 92	75- - -	- - -	2 1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0168; RJ2265B
tomatoes	0.05	0.1 (F)	103 113	93-	-	2	< 0.05 (4)	HPLC-MS/MS linearity ns	PP5/0170; RJ2370B
tomatoes	0.01	0.1 (F) 0.5 (F)	89 102 92	67-	21% -	3 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0169; RJ2268B
tomato	0.05	0.05 (F) 0.1 (F) 0.2 (F) 1.0 (F) 2.0 (F)	88 95 78 82 81 84 88 88 90 90	84- 74- 78- 87- 89-	5.6% 4.7% - - -	4 4 2 2 2	< 0.05 (1)	HPLC-MS/MS matrix matched; linear, not shown 0.005-1.0 mg/L	PP5/0079; RJ2172B validation
kale	0.01	0.05 (F) 0.10 (F)	82 77	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0140; RJ2654B;
lettuce	0.01	0.1 (F) 0.5 (F)	85 83	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0138; RJ2302B
lettuce	0.01	0.1 (F) 0.5 (F)	80 95 83 84	64- 82-	19% -	4 1	< 0.01 (11)	HPLC-MS/MS linearity ns	PP5/0134; RJ2363B
lettuce	0.01	0.05 (F) 0.5 (F)	106 114	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0340; RJ2631B
spinach	0.01	0.05 (F) 0.5 (F)	103 108 106 112	98- 101-	- -	2 2	< 0.01 (4)	HPLC-MS/MS linearity ns	PP5/0139; RJ2632B
spinach	0.05	0.05 (F) 0.1 (F) 0.2 (F) 1.0 (F) 2.0 (F)	104 121 97 100 93 94 97 99 98 98	94- 90- 92- 95- 97-	11% 4.9% - - -	4 4 2 2 2	< 0.05 (1)	HPLC-MS/MS matrix matched; linear, not shown 0.005-1.0 mg/L	PP5/0079; RJ2172B validation
green beans with pods	0.01	0.3 (F)	99 99	98-	-	2	< 0.01 (1);	HPLC-MS/MS linearity ns	PP5/0152; RJ2287B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
							0.01 (1)		
green beans with pods	0.01	0.10 (F)	86 91	81-	-	2	< 0.01 (1); 0.01 (1)	HPLC-MS/MS; linearity ns	PP5/0151; RJ2290B
green beans with pods	0.01	0.05 (F) 0.25 (F)	90 84	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0154; RJ2611B
green beans with pods	0.01	0.05 (F) 0.5 (F)	100 101	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0156; RJ2629B
green pea seeds	0.01	0.1 (F) 0.5 (F)	80 92 82	68- 81-3	- -	2 2	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0162; RJ2254B
green peas with pods	0.05	0.01 (F) 0.05 (F) 0.1 (F) 0.2 (F) 0.5 (F) 1.0 (F) 2.0 (F)	115 119 97 99 91 95 92 96 88 90 92 94 96 96	111- 95- 86- 88- 86- 90- 96- 	2.9% 1.7% 4.9% - - - -	4 4 4 2 2 2 2	< 0.05 (1)	HPLC-MS/MS matrix matched; linear, not shown 0.005-1.0 mg/L	PP5/0079; RJ2172B validation
dry bean seeds	0.01	0.05 (F) 2.0 (F)	90 94	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0153; RJ2610B
dry fava bean seeds	0.05	0.05 (F) 0.50 (F)	99 108	- -	- -	1 1	< 0.05 (5)	HPLC-MS/MS matrix matched 0.005-0.5 mg/L linear, R2>0.99	PP5/1545; CEMR-3008
dry fava bean haulm	0.05	0.05 (F) 0.50 (F)	105 109	- -	- -	1 1	< 0.05 (5)	HPLC-MS/MS matrix matched 0.005-0.5 mg/L linear, R2>0.99	PP5/1545; CEMR-3008
dry pea seeds	0.01	0.05 (F) 0.10 (F)	75 78 77	72- -	- -	2 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0157; RJ2510B
soya bean seed	0.05	0.05 (F) 0.1 (F) 0.2 (F) 1.0 (F) 2.0 (F)	91 101 97 104 81 82 91 97 91 93	80- 86- 80- 85- 89- 	9.5% 8.3% - - -	4 4 2 2 2	< 0.05 (1)	HPLC-MS/MS matrix matched; linear, not shown 0.005-1.0 mg/L	PP5/0075; RJ2172B validation
dry soya bean seeds	0.05	0.1 (F)	99 100	97-	-	2	< 0.05 (2)	HPLC-MS/MS linearity ns	PP5/0163; RJ2368B
dry soya bean seeds	0.01	0.5 (F) 5.0 (F)	83 91	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS; linearity ns	PP5/0164; RJ2405B
dry soya bean seeds	0.01	0.05 (F) 0.1 (F) 0.5 (F)	91 113 115 95	- 111- -	- - -	1 2 1	< 0.01 (4)	HPLC-MS/MS linearity ns	PP5/1026; RJ2720B
dry soya bean seeds	0.05	0.1 (F)	98 100	96-	-	2	< 0.05 (4)	HPLC-MS/MS linearity ns	PP5/1024; RJ2442B
dry soya bean seeds	0.05	0.05 (F) 0.10 (F) 0.50 (F)	107 119 107	- - 91-	- - 16%	1 1 3	< 0.05 (3); 0.10 (1)	HPLC-MS/MS; linearity ns	PP5/0165; RJ2481B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
		2.0 (F)	124 130 132	128-	-	2			
dry soya bean seeds	0.01	0.01 (F) 0.1 (F) 5.0 (F)	108 113 116	- - -	- - -	1 1 1	< 0.01 (4)	HPLC-MS/MS linearity ns	PP5/0159; RJ2781B
dry soya bean seeds	0.01	1.0 (F) 4.0 (F)	76 85	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS; matrix matched; linear, R2>0.999 0.005-1.0 mg/L	PP5/1398; 03-7026
dry soya bean seeds	0.01	1.0 (F) 4.0 (F)	103 107 104 111	99- 97-	- -	2 2	< 0.01 (1)	HPLC-MS/MS matrix matched linear, R2>0.999; 0.0005-1.0 mg/L	PP5/1396; 03-7072
dry soya bean seeds	0.01	1.0 (F) 4.0 (F)	104 107 100 108	102- 96-	- 6.6%	2 3	< 0.01 (2)	HPLC-MS/MS matrix matched linear, R2>0.999; 0.0005-1.0 mg/L	PP5/1397; 03-7073
dry soya bean seeds	0.01	1.0 (F) 4.0 (F)	99 103 108	95- -	- -	2 1	< 0.01 (2)	HPLC-MS/MS matrix matched linear, R2>0.999; 0.0005-1.0 mg/L	PP5/1399; 03-7074
potato	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.2 (F) 0.5 (F)	96 100 88 93 100 107 77 82 96 100	89- 76- 92- 73- 92-	5.0% 9.3% - - -	4 4 2 2 2	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, not shown 0.005-1.0 mg/L	PP5/0075; RJ2172B validation
carrots	0.01	0.05 (F) 0.1 (F) 0.5 (F)	106 108 115 110	105- - -	- - -	2 1 1	< 0.01 (3)	HPLC-MS/MS; linearity ns	PP5/0112; RJ2638B
carrots	0.01	0.05 (F) 0.50 (F)	110 110	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0125; RJ2659B
celeriac	0.01	0.025 (F) 0.25 (F)	69 97	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0116; RJ2630B
sugarbeet roots	0.01	0.05 (F) 0.10 (F) 0.50 (F)	117 124 114 110	110- - -	- - -	2 1 1	< 0.01 (4)	HPLC-MS/MS linearity ns	PP5/0115; RJ2553B
asparagus	0.01	0.1 (F)	108 108	108-	-	2	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/0105; RJ2281B
asparagus	0.01	0.1 (F)	99 100	97-	-	2	< 0.01 (3)	HPLC-MS/MS linearity ns	PP5/0110; RJ2673B
asparagus	0.01	0.1 (F) 0.2 (F)	95 95	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0113; RJ2701B
witloof roots witloof endives	0.01	0.1 (F)	101 110	93-	-	2	R: < 0.01 (2) E: < 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0111; RJ2646B
hazelnuts	0.01	0.05 (F) 0.5 (F)	71 84	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0223; RJ2656B
sunflower seed	0.01	0.1 (Fb)	84 86	81-	-	2	< 0.01 (1); 0.01 (1)	HPLC-MS-MS linearity ns	PP5/0221; RJ2284B
sunflower seed	0.01	0.1 (Fb)	71 76	66-	-	2	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0207; RJ2303B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
sunflower seed	0.01	0.05 (F) 0.5 (F) 2.0 (F)	97 88 90 104	- 86- -	- - - 	1 2 1	< 0.01 (4)	HPLC-MS-MS linearity ns	PP5/0534; RJ2726B
sunflower seed	0.01	0.1 (F) 0.2 (F)	108 116	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0542; RJ2940B
sunflower seed	0.01	0.01 (F) 0.05 (F)	89 91	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0005; RJ3234B
sunflower seed	0.01	0.01 (F) 0.05 (F)	109 93	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/1118; RJ3252B
sunflower seed	0.05	0.05 (F) 0.50 (F)	78 75	- -	- -	1 1	< 0.05 (2)	HPLC-MS/MS matrix matched linear, R2>0.99 0.03-15.00 ng	PP5/1488; CEMR-2690
grass forage	0.01	0.1 (F)	100 101	99-	-	2	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0201; RJ2496B
HPLC MS/MS method 287/02									
raspberries	0.01	0.01 (F) 0.10 (F)	85 100 91 93	74- 89-	11% 2%	5 5	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/1111; RJ3210B
strawberries	0.01	0.05 (F) 0.1 (F)	85 91	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0455; RJ3074B
strawberries	0.01	0.01 (F) 0.1 (F)	64 81	- -	- -	1 1	< 0.01 (6)	HPLC-MS/MS matrix matched linear, r>0.999 0.001–0.5 mg/L	PP5/1438; CEMR-2306
bulb onions	0.01	0.05 (F) 0.1 (F)	112 125 103	104- -	10% -	3 1	< 0.01 (4)	HPLC-MS/MS linearity ns	PP5/0126; RJ2827B
leeks	0.01	0.1 (F) 1.0 (F)	94 97 86 91	90- 81-	- -	2 2	< 0.01 (2)	HPLC-MS/MS; linearity ns	PP5/1222; RJ3278B
leeks	0.01	0.01 (F) 0.05 (F) 0.1 (F)	108 113 108 108 109 111	102- 107- 107-	- - -	2 2 2	< 0.01 (2)	HPLC-MS-MS matrix matched linear, R2>0.999 0.0005-0.1 mg/L	PP5/1377; 02-7035; PP5/1376; 02-7083; PP5/1405; 02-21401
leeks	0.01	0.01 (F) 0.05 (F)	93 82	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS matrix matched linear, R2>0.999 0.0005-1 mg/L	PP5/1409; 03-7029
leeks	0.01	0.01 (F) 0.10 (F)	74 80 85 86	69- 84-	- -	2 2	< 0.01 (3)	HPLC-MS-MS matrix matched linear, R2>0.99 0.03-15 mg/L	PP5/1489 CEMR-2687
head cabbage	0.01	0.5 (F) 1.0 (F)	111 107	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/0147; RJ2834B
head cabbage	0.01	0.2 (F) 0.5 (F)	102 105	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0356; RJ2992B
head cabbage	0.01	0.01 (F) 0.05 (F)	105 92	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0006; RJ3232B
head cabbage	0.01	0.1 (F) 0.5 (F)	111 100	- -	- -	1 1	< 0.01 (3)	HPLC-MS/MS matrix matched; linear, R2>0.9999 0.0005-1.0 mg/L	PP5/1394; 03-7068
head cabbage	0.01	0.01 (F) 0.20 (F)	110 107	- -	- -	1 1	< 0.01 (3)	HPLC-MS/MS matrix matched; linear, R2>0.9999	PP5/1395; 03-7076

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
								0.0005-1.0 mg/L	
head cabbage	0.01	0.1 (F) 0.2 (F)	112 114	100- 	- -	2 2	< 0.01 (4)	HPLC-MS-MS linearity ns	PP5/0146; RJ2794B
			108 116	101-					
head cabbage	0.01	1.0 (F) 2.0 (F)	103 113	93- 	7.7% 4.8%	7 7	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1281; RJ3087B stor stab
			106 113	98-					
cucumbers	0.01	0.05 (F) 0.1 (F)	110 116	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/0447; RJ3058B
tomatoes	0.01	0.05 (F) 0.1 (F)	80 86	75- 	- 9.2%	2 4	< 0.01 (4)	HPLC-MS-MS; linearity ns	PP5/0175; RJ2657B
			88 100	79-					
tomatoes	0.01	0.01 (F) 0.1 (F)	87 106	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0177; RJ2780B
tomatoes	0.01	0.1 (F) 0.2 (F)	103 116	88- 	10% 7.2%	7 7	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1281; RJ3087B stor stab
			107 119	97-					
kale	0.01	0.01 (F) 0.1 (F) 1.0 (F)	106 99 102	 97- 	- 2.7% 2.7%	1 3 3	< 0.01 (4)	HPLC-MS-MS linearity ns	PP5/0143; RJ2759B
			97 100	95-					
lettuce	0.01	0.25 (F) 0.50 (F)	94 106	78- 	9.8% 10%	10 6	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1281; RJ3087B stor stab
			97 109	84-					
lettuce	0.01	0.01 (F) 0.05 (F)	107 80	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0145; RJ2782B
lettuce	0.01	0.05 (F) 0.10 (F) 0.20 (F) 0.50 (F)	84 89 83 93 86	79- - - -	- - - - -	2 1 1 1	< 0.01 (4)	HPLC-MS/MS linearity ns	PP5/0351; RJ2786B
green beans with pods	0.01	0.1 (F) 0.5 (F)	118 105	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/0372; RJ2993B
green beans with pods	0.01	0.1 (F) 1.0 (F)	111 108	- -	- -	1 1	< 0.01 (3)	HPLC-MS/MS linearity ns	PP5/1333; RJ3294B
green beans with pods	0.01	0.1 (F) 0.2 (F)	104 102	- -	- -	1 1	< 0.01 (3)	HPLC-MS/MS linearity ns	PP5/1232; RJ3299B
green beans with pods	0.01	0.01 (F) 0.1 (F)	104 88	- -	- -	1 1	< 0.01 (5)	HPLC-MS/MS matrix matched linear, R2>0.999 0.0005-0.1 mg/L	A1279B_10430 CEMR-3014
green beans with pods	0.01	0.01 (F) 0.1 (F) 4.0 (F)	82 84 100 106 95	80- 93- -	- - -	2 2 1	< 0.01 (2)	HPLC-MS/MS matrix matched linear, R2>0.999 0.0005-0.5 mg/L	A12791B_10788: TK009248-07-REG;
green bean haulms	0.01	0.01 (F) 0.1 (F)	98 94	- -	- -	1 1	< 0.01 (5)	HPLC-MS/MS matrix matched linear, R2>0.999 0.0005-0.1 mg/L	A1279B_10430 CEMR-3014
green bean haulms	0.01	0.01 (F) 0.1 (F)	99 91	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS matrix matched linear, R2>0.999 0.0005-0.5 mg/L	A12791B_10788; T009248-07-REG
green pea seeds	0.01	0.1 (F)	107	-	-	1	< 0.01 (1)	HPLC-MS/MS	PP5/1260;

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
		1.0 (F)	104	-	-	1		linearity ns	RJ3336B
green pea seeds	0.01	0.1 (F) 0.5 (F)	112 109	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.0005-1 mg/L	PP5/1412; 03-7031
green pea seeds	0.01	0.05 (F) 0.10 (F)	109 106	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.0005-1 mg/L	PP5/1413; 03-7032
green pea seeds	0.01	0.01 (F) 0.10 (F)	103 103	- -	- -	1 1	< 0.01 (4)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.005-1.0 mg/L	PP5/1552; CEMR-3009
green pea pods	0.01	0.01 (F) 0.10 (F)	76 71	- -	- -	1 1	< 0.01 (4)	HPLC-MS/MS matrix matched; linear, R2>0.99 0.005-0.5 mg/L	PP5/1552; CEMR-3009
green pea seeds	0.01	0.01 (F) 0.10 (F)	95 107	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.99 0.005-0.5 mg/L	PP5/1550; CEMR-3012
green pea forage	0.01	0.01 (F) 0.10 (F) 1.0 (F)	99 108 102 104 112	91- 99- -	7% 2% -	5 5 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/1260; RJ3336B
green pea forage	0.01	0.05 (F) 0.10 (F)	105 110	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/1412; 03-7031
green pea forage	0.01	0.05 (F) 0.10 (F)	100 70	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/1413; 03-7032
green pea forage	0.01	0.01 (F) 0.10 (F)	79 90	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.99 0.005-0.5 mg/L	PP5/1550; CEMR-3012
green pea pods	0.01	0.01 (F) 0.10 (F)	85 99	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.99 0.005-0.5 mg/L	PP5/1550; CEMR-3012
dry bean seeds	0.01	0.01 (F) 0.05 (F) 0.1 (F)	129 101 107 114	- - 100-	- - 6.6%	1 1 3	< 0.01 – 0.03 (2)	HPLC-MS-MS; linearity ns	PP5/0160; RJ2826B
dry bean seeds	0.01	0.1 (F) 1.0 (F)	108 113	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/0373; RJ2994B
dry bean seeds	0.01	2.5 (F)	108 121	94-	7.3%	10	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1281; RJ3087B stor stab
dry pea seeds	0.01	0.01 (F) 0.10 (F)	105 79	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0158; RJ2785B
dry pea seeds	0.01	0.05 (F) 0.5 (F)	85 97	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/1112; RJ3209B
dry pea seeds	0.01	0.05 (F) 0.5 (F)	91 97	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS; linearity ns	PP5/1090; RJ3211B
dry pea seeds	0.01	0.2 (F) 0.5 (F)	94 97	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1227; RJ3266B
dry pea seeds	0.01	0.2 (F) 0.5 (F)	107 106	- -	- -	1 1	< 0.01 (3)	HPLC-MS/MS linearity ns	PP5/1233; RJ3300B
dry pea seeds	0.01	1.0 (F) 5.0 (F)	104 100	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.0005-1.0 mg/L	PP5/1425; 03-7058

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
dry pea seeds	0.01	1.0 (F) 5.0 (F)	104 100	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.999; 0.0005-1 mg/L	PP5/1426; 03-7059
dry pea straw	0.01	0.01 (F) 0.1 (F) 0.2 (F) 0.5 (F)	90 101 90 104 91 85	81- 72- - -	8.4% 13% - -	5 5 1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1227; RJ3266B
dry pea straw	0.01	0.5 (F) 1.0 (F)	108 104	- -	- -	1 1	< 0.01 (3)	HPLC-MS/MS linearity ns	PP5/1233; RJ3300B
dry pea straw	0.01	0.01 (F) 0.10 (F)	83 82	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS; matrix matched; linear, R2>0.9999 0.0025-0.5	A12791B_10830; T009247-07-REG
dry soya bean seeds	0.01	0.01 (F) 3.0 (F) 5.0 (F)	93 105 109 102 116	80- - 95-	- - 12%	2 1 3	< 0.01	HPLC-MS-MS linearity ns	PP5/50435, RJ2914B processing
dry soya bean seeds	0.01	2.5 (F)	103 115	94-	7.1%	11	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1281; RJ3087B stor stab
dry soya bean seeds	0.01	0.1 (F) 0.5 (F) 1.0 (F)	103 93 100 109 98 107	113, 84- 89-	- 8.6 -	2 4 2	< 0.01	HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
dry soya bean seed	0.01	0.01 (F) 0.1 (F) 0.5 (F) 1.0 (F)	89 99 99 114 104 115 97	80- 89- 94- -	11% 9.1% 8.3% -	3 5 4 1	< 0.01	HPLC-MS/MS linearity ns	PP5/1122, RJ3208B processing
Soya bean hulls	0.01	0.1 (F) 0.5 (F)	115 117	- -	- -	1 1	0.02	HPLC-MS-MS linearity ns	PP5/50435, RJ2914B processing
soya bean hulls	0.01	0.25 (F)	104 113	88-	7.0%	12	0.03 (1)	HPLC-MS-MS linearity ns	PP5/1281; RJ3087B stor stab
Soya bean hulls	0.01	0.1 (F) 0.5 (F)	107 103 94 84	111. 105-	- -	2 2	< 0.01	HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
soya bean hulls	0.01	0.01 (F) 0.1 (F) 1.0 (F)	104 113 86 93 83 94	93- 72- 72-	8.6% 8.8% -	5 7 2	< 0.01	HPLC-MS/MS linearity ns	PP5/1122, RJ3208B processing
soya bean meal	0.01	0.25 (F)	106 117	92-	7.1%	12	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1281; RJ3087B stor stab
Soya bean meal	0.01	0.01 (F) 0.1 (F)	90 99 75 86	83- 62-	7% 14%	5 5	< 0.01	HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
soya bean meal	0.01	0.1 (F) 1.0 (F)	98 105 98 107	91- 89-	- -	2 2	< 0.01	HPLC-MS/MS linearity ns	PP5/1122, RJ3208B processing
Soya bean flour	0.01	0.01 (F) 0.1 (F)	93 112 99 104	75- 90-	14% 5.5%	5 5	< 0.01	HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
Soya bean flour	0.01	0.1 (F) 1.0 (F)	84 93 97 101	76- 93-	- -	2 2	< 0.01	HPLC-MS/MS linearity ns	PP5/1122, RJ3208B processing
Soya bean, flocs	0.01	0.01 (F) 0.1 (F)	107 111 108 113	103- 90-	3.0% 9.4%	5 5	< 0.01	HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
carrots	0.01	0.05 (F) 0.1 (F) 0.2 (F) 0.5 (F)	90 92 98 82 89	89- - - -	- - - -	2 1 1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0118; RJ2772B
carrots	0.01	0.05 (F) 0.2 (F)	105 102	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/0309; RJ3065B
celeriac	0.01	0.01 (F) 0.05 (F) 0.1 (F)	98 102 103	- - -	- - -	1 1 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0120; RJ2804B
potato	0.01	0.25 (F) 0.50 (F)	96 106 101 110	91- 93-	8.7% 5.7%	3 8	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1281; RJ3087B stor stab
potato	0.01	0.05 (F) 0.2 (F)	99 110	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1148; gpo11501
potato	0.01	0.05 (F) 0.2 (F)	104 105	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1147; gpo31501
potato	0.01	0.05 (F) 0.2 (F)	103 104	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1145; gpo41505
potato	0.01	0.05 (F) 0.2 (F)	95 99	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1146; gpo91501
potato	0.01	0.02 (F) 0.05 (F)	90 99	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1342; gpo079002
potato	0.01	0.05 (F) 0.2 (F)	99 103	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS matrix matched linear, R2>0.999 0.0005-1 mg/L	PP5/1427; gpo023103
potato	0.01	0.1 (F) 0.2 (F)	106 102	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1355; 02-7044
potato	0.01	0.02 (F) 0.05 (F)	111 109	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1353; 02-7045
potato	0.01	0.02 (F) 0.05 (F)	98 86	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1357; 02-7068
potato	0.01	0.1 (F) 0.2 (F)	100 105	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS linearity ns	PP5/1359; 02-7069
potato	0.01	0.05 (F) 0.20 (F)	91 106	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS matrix matched linear, R2>0.999 0.0005-1 mg/L	PP5/1418; 03-7027
potato	0.01	0.05 (F) 0.2 (F)	86 103	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS matrix matched linear, R2>0.999 0.0005-1 mg/L	PP5/1416; 03-7028
potato	0.01	0.05 (F)	73	-	-	1	< 0.01 (1)	HPLC-MS/MS	PP5/1417;

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
		0.20 (F)	94	-	-	1		matrix matched; linear, R2>0.999 0.0005-1.0 mg/L	03-7030
potato	0.01	0.05 (F) 0.2 (F)	95 92	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS matrix matched linear, R2>0.999 0.0005-1 mg/L	PP5/1410; 03-7037
potato	0.01	0.05 (F) 0.2 (F)	92 91	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS matrix matched linear, R2>0.999 0.0005-1 mg/L	PP5/1408; 03-7038
potato	0.01	0.05 (F) 0.20 (F)	109 100	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.0005-1.0 mg/L	PP5/1419; 03-7047
potato	0.01	0.05 (F) 0.20 (F)	93 104	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.0005-1.0 mg/L	PP5/1420; 03-7048
potato	0.01	0.05 (F) 0.2 (F)	81 104	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.0005-1.0 mg/L	PP5/1424; 03-7056
potato	0.01	0.05 (F) 0.2 (F)	94 87	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS matrix matched linear, R2>0.999 0.0005-1 mg/L	PP5/1411; 03-7057
potato	0.01	0.05 (F) 0.20 (F)	107 76	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.0005-1.0 mg/L	PP5/1414; 03-7079
potato	0.01	0.05 (F) 0.20 (F)	88 91	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.0005-1.0 mg/L	PP5/1415; 03-7080
potato	0.01	0.01 (F) 0.10 (F)	90 92	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS matrix matched linear, r>0.99 0.001–0.1 mg/L	PP5/1440; CEMR-2309
potato	0.01	0.01 (F) 0.10 (F)	89 102	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS matrix matched linear, R2>0.99 0.03-15 ng	PP5/1487; CEMR-2688
potato	0.01	0.01 (F) 0.10 (F)	89 102	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS matrix matched linear, R2>0.99 0.03-15.0 ng	PP5/1486; CEMR-2689
potato	0.01	0.01 (F) 0.10 (F)	72 81	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS matrix matched linear, R2>0.99 0.005-0.5 mg/L	A12791B_10432; CEMR-3374
potato	0.01	0.01 (F) 0.10 (F)	72 81	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS matrix matched linear, R2>0.99 0.005-0.5 mg/L	PP5/1555; CEMR-3375
potato	0.01	0.05 (F) 0.5 (F)	85 87 102 105	83- 99-	- -	2 2	< 0.01 (3)	HPLC-MS/MS linearity ns	PP5/1091; RJ3200B
potato	0.01	0.05 (F) 0.1 (F)	98 101	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/1149; RJ3222B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
potato	0.01	0.05 (F) 0.20 (F)	100 104 104 104	97- 103-	- -	2 2	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/1241; RJ3295B
sugarbeet roots	0.01	0.1 (F)	104 108	91-	8.3%	4	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0124; RJ2779B
sugarbeet roots	0.01	0.05 (F) 0.1 (F) 0.5 (F)	96 104 112 100	- 95- -	- - -	1 2 1	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0121; RJ2833B
sugarbeet roots	0.01	0.05 (F) 0.1(F)	104 104	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/0308; RJ2995B
sugarbeet roots	0.01	0.01 (F) 0.10 (F)	103 113	- -	- -	1 1	< 0.01 (3)	HPLC-MS/MS matrix matched; linear, r>0.9999 0.001–0.5 mg/L	PP5/1441; CEMR-2310
sugarbeet roots	0.01	0.01 (F) 0.1 (F)	112 114	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/1337; gsb064002
sugarbeet roots	0.01	0.01 (F) 0.1 (F)	109 115	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/1336 gsb064202
sugarbeet tops	0.01	0.1 (F)	104 108	91-	8.3%	4	< 0.01 (2)	HPLC-MS/MS linearity ns	PP5/0124; RJ2779B
sugarbeet tops	0.01	0.25 (F) 0.5 (F) 1 (F)	120 117 125 129	- 109- -	- - -	1 2 1	0.01 (2)	HPLC-MS/MS linearity ns	PP5/0121; RJ2833B
sugarbeet tops	0.01	0.5 (F) 1.0 (F)	104 89	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/0308; RJ2995B
sugarbeet tops	0.01	0.01 (F) 0.10 (F)	85 88	- -	- -	1 1	< 0.01 (3)	HPLC-MS/MS matrix matched; linear, r>0.9999 0.001–0.5 mg/L	PP5/1441; CEMR-2310
sugarbeet tops	0.01	0.01 (F) 0.5 (F) 1.0 (F)	113 107 107	- - -	- - -	1 1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/1337; gsb064002
sugarbeet tops	0.01	0.01 (F) 0.5 (F) 1.0 (F)	100 111 92	- - -	- - -	1 1 1	< 0.01 (1)	HPLC-MS/MS linearity ns	PP5/1336 gsb064202
oilseed rape seeds	0.01	1.0 (F) 5.0 (F)	81 86	- -	- -	1 1	< 0.01 (1); 0.05 (1)	HPLC-MS-MS linearity ns	PP5/0212; RJ2758B
oilseed rape seeds	0.01	0.1 (F) 1.0 (F)	90 99	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0209; RJ2765B
oilseed rape seeds	0.01	0.01 (F) 0.1 (F) 1.0 (F)	97 99 102	- - -	- - -	1 1 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0120; RJ2766B
oilseed rape seeds	0.01	0.1 (F) 0.5 (F)	101 94	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0208; RJ2771B
oilseed rape seeds	0.01	1.0 (F) 5.0 (F)	78 91	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS linearity ns	PP5/0211; RJ2806B
oilseed rape seeds	0.05	0.5 (F) 2.0 (F)	98 100	- -	- -	1 1	< 0.05 (1)	HPLC-MS-MS linearity ns	PP5/1256; 02-7015
oilseed rape seeds	0.01	0.1 (F) 0.5 (F)	99 102	-	- -	1 1	0.02 (1)	HPLC-MS-MS linearity ns	PP5/1365; 03-7004
oilseed rape seeds	0.01	0.1 (F) 0.5 (F)	92 98	- -	- -	1 1	< 0.01 (1)	HPLC-MS-MS matrix matched linear, R2>0.99 0.0005-1.0 mg/L	PP5/1367; 03-7005
HPLC MS/MS method 287/02 modification A (no soaking)									
dry pea straw	0.01	0.05 (F) 0.50 (F)	95 99	- -	- -	1 1	< 0.01 (1)	HPLC-MS/MS matrix matched;	PP5/1544; CEMR-3373

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	detection; calibration	Code no; Report no
							linear, R2>0.99 0.005-0.5 mg/L	
grass hay	0.01	0.01 (F) 0.1 (F)	103 - 95 -	- -	1 1	< 0.01 (1) 0.01 (1)	HPLC-MS/MS linearity ns	PP5/0203; RJ2764B
HPLC MS/MS method 287/02 modification B (no clean-up)								
green pea forage	0.01	0.01 (F) 0.1 (F)	98 - 100 -	- -	1 1	< 0.01 (4)	HPLC-MS/MS matrix matched; linear, R2>0.999 0.0005-0.1 mg/L	PP5/1553; CEMR-3009
dry pea seeds	0.01	0.01 (F) 0.10 (F)	85 - 90 -	- -	1 1	< 0.01 (1)	HPLC-MS/MS; matrix matched; linear, R2>0.9999 0.0025-0.5 mg/L	A12791B_10830; T009247-07-REG
HPLC MS/MS method 287/02 modifications C (no soaking, no clean-up)								
dry pea seeds	0.05	0.01 (F) 0.05 (F) 0.50 (F)	106 - 87 - 94 -	- - -	1 1 1	< 0.05 (5)	HPLC-MS/MS matrix matched; linear, R2>0.9999 0.00025-0.1 mg/L	PP5/1544; CEMR-3373

GC-NPD Method R606/BAZ/1

GC-NPD method R606/BAZ/1 determines total fluazifop (fluazifop-butyl, free fluazifop acid (II) and its conjugates) sunflower seeds as one single analyte (common moiety) and residues are expressed as fluazifop-butyl. The method is based on HPLC-MS/MS method RAM/287/02 and RR91-014B. The reported LOQ is 0.01 mg/kg.

Method R606/BAZ/1 (2003) is described by [Suszter, 2003, PP5/1497, report 02SYNAA0505]. Homogenised sunflower seeds (20 g) are soaked in 1 M HCl for at least 2 hours (or overnight) at room temperature followed by maceration in acetonitrile (acetonitrile/1M HCl = 100:20, v/v). After filtration, the extract is shaken with n-hexane and the hexane layer containing the oils is discarded. The remaining extract is reduced to aqueous volume by rotary evaporation. A 6 M HCl solution was added to induce hydrolysis (final concentration 3 M HCl, 60 °C, 1 hour) to convert any ester or acid conjugates to fluazifop acid (II). The hydrolysate (pH < 1) was partitioned against dichloromethane. The aqueous phase was discarded and the organic phase was extracted with 1% sodium bicarbonate solution. The organic phase was discarded and the aqueous phase was acidified with HCl (pH < 1) and then partitioned against dichloromethane. The aqueous phase was discarded and the organic phase was dried with anhydrous sodum sulfate and than evaporated to dryness. Fluazifop acid (II) was dissolved in acetone and derivatised to the methyl ester using 0.8 M tetrabutyl ammonium hydroxide and methyl iodide at 40 °C for 1.5 hours. Afterwards, the solution is diluted with n-hexane/isooctane (50:5) and then evaporated to near dryness. The residue is diluted with n-hexane and than shaken with water (hexane:water = 6:2, v/v) to dissolve the precipitate. The aqueous phase is discarded and the organic phase is dried with anhydrous sodium sulfate. The final extract is analysed by GC-NPD against in-situ hydrolysed and derivatised fluazifop-butyl standards. Confirmation by GC-MS at m/z 146, 254, 282, 341. Residues are expressed as fluazifop-butyl and need to be corrected with a factor 327.3/383.4=0.85 to get the total fluazifop residues, expressed as fluazifop acid (II) [Syngenta, 2016, Response to questions 14].

A validation report was available [Suszter, 2003, PP5/1497, report 02SYNAA0505]. Validation results are shown in Table 123. Method R606/BAZ/1 was used in supervised trials on sunflower seeds [02SYNAA0505].

No radiovalidation was conducted, but experiments with samples from supervised residue trials suggest incomplete hydrolysis in 3 M HCl 1 hour 60 °C (see Table 103).

Reviewer's conclusion: GC-NPD Method R606/BAZ/1 is considered:

- not valid for the determination of fluazifop acid (II) in sunflower seeds (no validation)
- valid (reduced validation) for the determination of fluazifop-butyl in sunflower seeds (0.05 0.25 mg/kg)
- not valid for the determination of fluazifop (II) conjugates (no radiovalidation and extraction experiments with samples from supervised residue trials suggest incomplete hydrolysis in 3 M HCl 1 hour 60 °C)

The valid LOQ is 0.05 mg/kg (no validations below this point).

Table 123 Validation results for GC-NPD method R606/BAZ/1

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
sunflower seeds	0.05	0.05 (Fb) 0.25 (Fb)	94 98	86-106 92-102	9.4% 4.95	5 3	< 0.05 (5)	external std 0.1-2.0 ng 1/× weighted linear R2>0.9999	PP5/1497; 02SYNAA0505

GC-MS method IT 125

GC-MS method IT 125, rev 01, determines free fluazifop acid (II). The reported LOQ is 0.05 mg/kg.

The method is described by [Baptista and Bahia, 2006, A13680A_10002, report M04064]. Soya bean seeds (10 g) were soaked in 1 M HCl for 2 hours. Samples were extracted with acetonitrile/1 M HCl (50:50, v/v) in the presence of sodium sulphate for 1 hour. A hydrolysis step is not indicated. An aliquot of the extract is evaporated to dryness, redissolved in methyl ether and left to react for 15 min. After derivatization, the mixture is evaporated to dryness and redissolved in hexane/ethyl acetate (50:50 v/v). The mixture is cleaned-up by GPC and the fraction containing derivatised fluazifop acid (II) is evaporated to dryness and redissolved in hexane. Derivatised fluazifop acid (II) is determined by GC-MS (m/z 282).

Neither the summary nor the more detailed description of the method specifically refer to a hydrolysis step. Appendix 1 indicates that the method is based on method RAM 287/02, which does include a hydrolysis step. However, specific confirmation of this point is not available in the report, nor could it be provided by the manufacturer [Syngenta, Response to Questions 11, 2 May 2016].

The method was used in a supervised trial on green and dry soya beans seeds [M04064]. Validation results are shown in Table 124.

Reviewer's conclusion: Method GC-MS method IT 125 is considered:

- valid (full validation) for the determination of free fluazifop acid (II) in soya bean seeds (at 0.02-0.2 mg/kg)
- not valid for the determination of fluazifop-butyl (no validation)
- not valid for the determination of fluazifop (II) conjugates (no hydrolysis step included in the method)

The valid LOQ is 0.05 mg/kg (no validations below this point).

Table 124 Validation results for GC-MS method IT 125

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
dry soya bean seeds	0.05	0.05 (F) 0.5 (F)	80 77	72-86 72-82	8% 6%	5 5	< 0.05 (2)	external std in matrix linear, r>0.999 0.25-15 ng/mL	A13680A_10002; M04064

HPLC-MS/MS method CER 2605

HPLC-MS/MS method CER 2605, determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant commodities as one single analyte (common moiety) and residues are expressed as fluazifop. The reported LOQ is 0.01 mg/kg.

The method is described by [Sagan, 2008, A12791B_50003, report CER 02605/07; Sagan, 2008, A12791B_50005, report CER 02606/07; Sagan, 2008, A12791B_50001, report CER 02607/07]. Potatoes and dry seeds (10 g) are soaked in 1M HCl overnight followed by maceration in acetonitrile (acetonitrile:1M HCl = 50:50 v/v). The extract is collected and the solid remains are extracted again with acetonitrile. The acidic acetonitrile extracts are combined and the acetonitrile is evaporated off. The aqueous remainder is diluted 1:1 with concentrated 12 M HCl and than hydrolysed at 6 M HCl for 1 hour at 60 °C. The hydrolysate is neutralized with concentrated ammonium hydroxide and then partitioned twice against hexane. The hexane fractions are discarded. The aqueous fraction is acidified to pH 2 with concentrated HCl and then partitioned twice against dichloromethane. The dichloromethane fractions are collected and evaporated to dryness and then redissolved in acetonitrile or acetontrile/water (30:70, vv). Residues are quantified by HPLC-MS/MS (m/z 329 to 283 and 328 to 282; positive ion mode).

Method CER 2605 is used on dry bean seeds [CER 02607/07], dry soya beans (seeds, forage, hay) [CER 02605/07, CER 02401/06] and potatoes [CER 02606/07]. Validation results are presented in Table 125.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-MS/MS Method CER 2605 is considered:

- valid (full validation) for the determination of fluazifop acid (II) in dry bean seeds (0.01–0.1 mg/kg); dry soya bean seeds (0.01–0.1 mg/kg), dry soya bean hay (0.01–0.1 mg/kg), dry soya bean forage (0.01–0.1 mg/kg) and potatoes (0.01–1.0 mg/kg)
- not valid for the determination of fluazifop-butyl (no validation)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).

The valid LOQ is 0.01 mg/kg.

Table 125 Validation results for HPLC-MS/MS method CER2605

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
dry bean seeds	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.5 (F) 5.5 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	94 120 86 99 114 100 88 109 104 99	70- 77-94 87- - 88-89 - - -	20% 8.7% 10% - - - - -	8 5 5 1 2 1 1 1	<0.3 LOQ (3)	0.5-15 ng/mL in solvent; linear 1/x: r>0.99	A12791B_50001; CER 02607/07
dry soya bean seeds	0.01	0.01 (F) 0.05 (F) 0.1 (F) 1.5 (F) 2.5 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	84 94 108 106 114 102 90 106 109	76-93 87- 94- - - - -	7.9% 8.7% 8.0% - - - - -	7 5 4 1 1 1 1 1	< 0.01 (7)	matrix matched 0.5-15 ng/mL linear 1/x; r>0.99	A12791B_50003; CER 02605/07

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
dry bean seeds	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.5 (F) 5.5 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	94 120 86 99 114 100 88 109 104 99	70- 77-94 87- - 88-89 - - -	20% 8.7% 10% - - - - - -	8 5 5 1 2 1 1 1 1	<0.3 LOQ (3)	0.5-15 ng/mL in solvent; linear 1/x: r>0.99	A12791B_50001; CER 02607/07
			85	-					
soya bean seeds	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	90 91 102 94 104 87 100 75	86-99 79- 77- - - -	5.9% 10% 16% - - - - -	6 4 3 1 1 1 1 1	< 0.005 (3)	matrix matched 0.5-15 ng/mL linear 1/x; r>0.99	A12791B_50006; CER 02401/06
soya bean hay	0.01	0.01 (F) 0.05 (F) 0.1 (F) 4.0 (F) 5.0 (F) 6.0 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	103 114 92 108 86 93 95 102 116 88 89	84- 75- 77-97 - - - - - -	9.2% 14% 8.8% - - - - - - -	8 5 5 1 1 1 1 1 1 1	< 0.01 (8)	matrix matched 0.5-15 ng/mL linear 1/x; r>0.99	A12791B_50003; CER 02605/07
soya bean hay	0.01	0.01 (F) 0.05 (F) 0.1 (F) 1.0 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	89 102 78 82 114 86 92 86	78- 76-82 79-85 - - - -	9.0% 3.4% 3.7% - - - - -	6 4 3 1 1 1 1 1	< 0.005 (3)	matrix matched 0.5-15 ng/mL linear 1/x; r>0.99	A12791B_50006; CER 02401/06
soya bean forage	0.01	0.01 (F) 0.05 (F) 0.1 (F) 2.5 (F) 3.0 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	110 120 102 107 102 109 99 106 90 119 102	98- 99- 92- - - - - -	7.6% 3.7% 6.8% - - - - - - -	7 5 5 1 1 1 1 1 1 1	< 0.01 (7)	matrix matched 0.5-15 ng/mL linear 1/x; r>0.99	A12791B_50003; CER 02605/07
soya bean forage	0.01	0.01 (F) 0.05 (F) 0.1 (F) 1.0 (F) 2.0 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	81 81 84 91 85 78 91 84	72-89 70-92 78-90 - - - - -	7.9% 12% 7.2% - - - - -	6 4 3 1 1 1 1 1	< 0.005 (3)	matrix matched 0.5-15 ng/mL linear 1/x; r>0.99	A12791B_50006; CER 02401/06
potato	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.25 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	94 106 107 113 107 109 102	76- 102- 104- 101-	12% 5.3% 2.4% - - - -	5 3 3 2 1 1 1	< 0.0033 (3)	no data	A12791B_50005; CER 02606/07

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
dry bean seeds	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.5 (F) 5.5 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	94 120 86 99 114 100 88 109 104 99	70- 77-94 87- - 88-89 - - -	20% 8.7% 10% - - - - - -	8 5 5 1 2 1 1 1 1	<0.3 LOQ (3)	0.5-15 ng/mL in solvent; linear 1/x: r>0.99	A12791B_50001; CER 02607/07
			102 101 108 84	 - - -					

HPLC-MS/MS method CER 2608

HPLC-MS/MS method CER 2608, determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant commodities as one single analyte (common moiety) and residues are expressed as fluazifop acid (II). The reported LOQ is 0.01 mg/kg.

The method is described by [Sagan, 2009, A12791N_50004, report CER 02608/08]. Potatoes (20 g) are extracted by maceratin with acetonitrile (acetonitrile : concentrated HCl = 98:2 v/v). The acidic acetonitrile extract is collected and the acetonitrile is evaporated off. The aqueous remainder is diluted with water and 6 M HCl is added (water/6M HCl = 2:1, v/v; final concentration 2 M HCl) and then hydrolysed for 1 hour at 60 °C. The hydrolysate (pH of 2-3) is partitioned thouree times against dichloromethane. The dichloromethane fractions are collected and evaporated to dryness and then redissolved in acetontrile/water (30:70, vv). Residues are quantified by HPLC-MS/MS (m/z 328 to 282; positive ion mode).

Method CER 2608 is used on potatoes [CER 02608/08]. Validation results are presented in Table 126.

Although no radiovalidation was conducted at 2 M HCl, residue levels in the potato trials in report [CER 02608/08] contained the highest residue levels with that dose rate. Therefore hydrolysis conditions are considered sufficient for potatoes.

Reviewer's conclusion: HPLC-MS/MS Method CER 2608 is considered:

- valid (full validation) for the determination of fluazifop acid (II) in potatoes (0.01–1.0 mg/kg)
- valid for the determination of fluazifop-butyl (potatoes)
- valid for the determination of fluazifop (II) conjugates in potatoes.

The valid LOQ is 0.01 mg/kg.

Table 126 Validation results for HPLC-MS/MS method CER2608

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
potato	0.01	0.01 (F) 0.05 (F) 0.1 (F) 1.0 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	84 93 89 95 101 116 107 115 78	71- 80- 88- 102- -	8.4% 7.2% 11%6.5% - - - 	7 4 5 3 1 1 1 	< 0.0033	no data	A12791N_50004; CER 02608/08

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	rang e	RSD (%)	n	control mg/kg	calibration	Code no Report no
			91 101	- -					

HPLC-MS/MS method CER 2609

HPLC-MS/MS method CER 2609, determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant commodities as one single analyte (common moiety) and residues are expressed as fluazifop acid (II). The reported LOQ is 0.01 mg/kg.

The method is described by [Sagan, 2009, A12791N_50001, report CER 02609/08]. Dry bean seeds (10 g) are soaked in 1M HCl overnight followed by maceration in acetonitrile (acetonitrile : 1M HCl = 50:50 v/v). The extract is collected and the solid remains are extracted again with acetonitrile. The acidic acetonitrile extracts are combined and the acetonitrile is evaporated off. The aqueous remainder is diluted 1:1 with concentrated 12 M HCl and than hydrolysed at 6 M HCl for 1 hour at 60 °C. The hydrolysate is partitioned twice against dichloromethane. The dichloromethane fractions are collected and evaporated to dryness and then redissolved in acetonitrile/water (70:30, v/v). Residues are quantified by HPLC-MS/MS (m/z 328 to 282; positive ion mode).

Method CER 2609 is used on dry bean seeds [CER 02609/08]. Validation results are presented in Table 127.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-MS/MS Method CER 02609 is considered:

- valid (full validation) for the determination of fluazifop acid (II) in dry bean seeds (0.01–0.1 mg/kg)
- valid for the determination of fluazifop-butyl (recovery verification in dry bean seeds)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).

The valid LOQ is 0.01 mg/kg.

Table 127 Validation results for HPLC-MS/MS method CER 2609

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
dry bean seeds	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.5 (F) 0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	96 98 98 100 88 102 83	78-118 79-118 88-110 - - - -	16% 15% 8.9% - - - -	7 8 5 1 1 1 1	<0.3 LOQ (3)	0.1-10 ng/mL 1/x linear; R2>0.999	A12791N_50001 CER 02609/08;

HPLC-MS/MS Method PLMV-027-C

HPLC-MS/MS method PLMV-027-C determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant commodities as one single analyte (common moiety) and residues are expressed as fluazifop acid (II). The reported LOQ is 0.02 mg/kg.

HPLC-MS/MS method PLMV-027-C (30 September 2008) is described by [Tomaz, 2008, A12530B_10010, report 027-003-07B]. Homogenised samples (sunflower seeds, 7.0 g) are macerated with 1 M HCl and then soaked overnight. After addition of acetonitrile (50:50, v/v) samples are

macerated and centrifuged. From an aliquot of the extract, the acetonitrile is removed by evaporation. Concentrated HCl is added to the aqueous remainder to give 6 M HCl and then hydrolysed for 1 hour at 60 ºC to convert any ester or acid conjugates to fluazifop. The samples are then diluted with water and analysed by HPLC-MS/MS. Transitions are not indicated. Calibration is by matrix matched standards for fluazifop acid (II). Validation results are shown in Table 128.

A validation report was available [Tomaz, 2008, A12530B_10010, report 027-003-07B]. Validation results are shown in Table 128. Method PLMV-027C was used in supervised trials on sunflower seeds [027-003-07B].

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: Method PLMV-027-C is considered:

- valid (full validation) for the determination of fluazifop acid (II) in sunflower seeds (0.02–0.2 mg/kg)
- not valid for the determination of fluazifop-butyl (no validation)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).

The valid LOQ is 0.02 mg/kg (no validations below this point).

Table 128 Validation results for method PLMV-027-C and its modifications

Matrix	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
sunflower seed	0.02	0.02 (F) 0.2 (F)	81 87	79-83 84-92	2.2% 3.8%	5 5	<0.3LOQ (4)	external std in matrix linear, R2 >0.999 0.25-15 ng/mL	A12530B_10010, 027-003-07B

HPLC-MS-MS Method RAM 336/01

HPLC-MS-MS method RAM 336/01 determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) in soya bean milk as one single analyte (common moiety) and residues are expressed as fluazifop acid (II). The reported LOQ is 0.01 mg/kg.

Method RAM 336/01 (20 March 2009) is described by [Kwiatkowski and Crook, 2009, no code, SOP RAM 336/01]. Concentrated HCl is added to soya bean milk, diluted soya based infant formula or other emulsion containing liquids to give 6 M HCl. Samples are hydrolysed in 6 M HCl (1 hour, 60 ºC) to convert any esters or acid conjugates to fluazifop acid (II). The hydrolysate is diluted with water to give 1 M HCl and any particulates are filtered out by centrifugation using Vecta Spin tubes. Any remaining residues of fluazifop acid (II) on the filter cake are extracted from the Vecta Spin tubes by shaking with 0.5 M $NaHCO_3$ followed by centrifugation. The $NaHCO_3$ filtrate is combined with the acid fraction. Endogenous co-extractives are separated by adsorption chouromatographic clean-up using C2 (End-Capped) SPE columns. Fluazifop acid (II) is eluted with dichloromethane/acetone (95:5, v/v). The eluent is evaporated to dryness and redissolved in HPLC mobile phase (acetonitrile/water, 10:90, v/v). Samples are then analysed by HPLC-MS/MS (m/z 328 to m/z 282). The residues are quantified against a matrix matched fluazifop acid (II) external standard.

A validation study is available [Mason, 2009, PP5/10004, report RJ3110B]. Additional validation data are generated in a storage stability study on soya bean milk [RJ3087B] and processing studies on various processed soya commodities [RJ2914B, RJ3149B, RJ3208B]. Validation results are shown in Table 129.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-MS/MS Method RAM 336/01 is considered:

- valid (full validation) for the determination of fluazifop acid (II) in soya bean milk (0.01–0.25 mg/kg), soya based infant formula (0.01–0.1 mg/kg), soya bean wash water (0.01–0.1 mg/kg), soya bean aqueous phase (0.01–0.1 mg/kg), soya bean filter cake (0.01–0.1 mg/kg),
- insufficiently validated for the determination of fluazifop acid (II) in soya bean diluted protein insolate
- not valid for the determination of fluazifop-butyl (no validation)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).

The valid LOQ is 0.01 mg/kg.

Table 129 Validation results for HPLC-MS/MS method RAM 336/01

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration/detection	Code no; Report no
Soya bean milk	0.001	0.001 (F) 0.5 (F) 1.0 (F)	118 95 101	- - -	- - -	1 1 1	< 0.001	HPLC-MS-MS Linearity ns	PP5/50435, RJ2914B processing
soya bean milk	0.01	0.25 (F)	92	74-113	14%	15	< 0.01 (1)	HPLC-MS-MS calibration ns	PP5/1281; RJ3087B stor stab
soya bean milk	0.01	0.01 (F) 0.1 (F)	103 88	93-108 81-99	6% 8%	5 5	<0.3 LOQ (2)	matrix matched linear, r2>0.999 0.0005-0.2 mg/L	PP5/10004 RJ3110B validation
Soya bean milk	0.01	0.05 (F) 0.1 (F)	95 97	- -	- -	1 1	< 0.01	by HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
soya bean milk	0.01	0.01 (F)	104	-	-	2	< 0.01	HPLC-MS/MS Linearity ns	PP5/1122, RJ3208B processing
soya based infant formula	0.01	0.01 (F) 0.1 (F)	97 96	71-107 88-110	15% 10%	5 5	<0.3 LOQ (2)	matrix matched linear, r2>0.99999 0.0005-0.2 mg/L	PP5/10004 RJ3110B validation
Soya bean, wash water	0.01	0.01 (F) 0.1 (F)	90 91	77-105 68-100	11% 11%	10 10	< 0.01	by HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
Soya bean, aqueous phase	0.01	0.01 (F) 0.1 (F)	103 103	85-102 98-111	12% 5%	5 5	< 0.01	by HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
Soya bean, filter cake	0.01	0.01 (F) 0.1 (F)	71 72	63-78 50-92	7.6% 23%	5 5	< 0.01	by HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
Soya bean, diluted protein isolate	0.01	0.05 (F) 0.1 (F)	83 84	- -	- -	1 1	< 0.01	by HPLC-MS-MS linearity ns	PP5/1144; RJ3149B processing
soya bean, diluted protein isolate	0.01	0.01 (F)	86	-	-	2	< 0.01	HPLC-MS/MS linearity ns	PP5/1122, RJ3208B processing

HPLC-MS/MS Method GRM044.01A

HPLC-MS/MS method GRM044.01A determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant commodities as one single analyte (common moiety) and residues are expressed as fluazifop acid (II). The reported LOQ is 0.01 mg/kg.

Method GRM044.01A (10 March 2009) is described by [Mayer, 2009, PP5_50036, report GRM044.01A]. Homogenised samples (5 g) were macerated in 1 M NaOH: acetonitrile (90:10, v/v) and then refluxed for 1 hour to convert fluazifop-butyl or fluazifop conjugates into fluazifop acid (II). The centrifuged extract is acidified (pH<2) and loaded onto an SPE cartridge (OASIS HLB). Fluazifop acid (II) is eluted with 0.1% formic acid/acetonitrile (60/40, v/v) and quantified by HPLC-MS/MS at m/z 328 to 254 (quantification) or 328 to 282 (confirmation). Calibration is performed with fluazifop acid (II) as external standard in solvent.

A validation study as well as an independent laboratory validation is available [Mayer, 2009, PP5_50029, report T002220-07; Brown, 2009, PP5_50066, report ML09-1552-SYN]. Results are shown in Table 131. Recoveries for the confirmation ions were within mean recovery range of 82%-107%. No significant enhancement or suppression of detector response was observed.

Method GRM044.01A was used in supervised trials on dry soya beans (seeds, aspirated grain fractions) [TK0016832], carrots [T002222-07], cotton (seeds, gin trash) [T002224-07], a storage stability study [13SYN331REP] and a processing study on citrus [TK0058357]. Validation results are shown in Table 131.

Modification A of method GRM044.01A was used on potato flakes [13SYN331REP]. Samples were pre-soaked in a small amount of water overnight to hydrate the matrix prior to extraction. Validation results are shown in Table 131.

Modification B of method GRM044.01A was used on caneberries [IR-4 PR 03947], blueberries [IR-4 PR 02083], strawberries [IR-4 PR A2085] and rhubarb [IR-4 PR A2404 (2013)]. The SPE clean-up step was substituted by a dichloromethane partition: After hydrolysis, the sample was filtered, concentrated on a rotary evaporator and acidified using concentrated HCl. The hydrolysate was partitioned twice against dichloromethane. The dichloromethane phases were combined, evaporated to dryness and redissolved in 0.1% formic acid/acetonitrile (60/40, v/v). The extract was analysed by HPLC-MS/MS. Validation results are shown in Table 131.

Method GRM044.01A was radiovalidated using radiolabelled carrot and endive metabolism samples [Lin, 2009, PP5_50001, report T002223-07]. In the metabolism studies, carrots and endive were treated with phenyl labelled ^{14}C-fluazifop-P-butyl (R-enatiomer) at 2 × 0.42 kg ai/ha. Control samples spiked with fluazifop acid (II) at 0.01 and 1 mg/kg were analysed concurrently with the radiolabelled samples for each of the matrices. Concurrent recoveries ranged between 78%-107% (n=1 per level and sample). The residues in the radiolabelled samples found by method GRM044.01A agreed well with the sum of free fluazifop acid (II) and its conjugates reported in the metabolism studies (see Table 130). The accountabilities of the method ranged from 79% for endive, 110% for carrot roots to 131% for carrot foliage, demonstrating that the conjugates can be effectively converted to free fluazifop acid (II) under the alkaline reflux conditions specified in the method.

Reviewer's conclusion: HPLC-MS/MS Method GRM044.01A is considered:

- valid (full validation) for the determination of fluazifop acid (II) in blueberries (0.02-2.0 mg/kg), strawberries (0.01-5.0 mg/kg), tomato (0.01–0.10 mg/kg), spinach (0.01–0.10 mg/kg), carrot (0.01–0.10 mg/kg), undelinted cotton seed (0.01–0.10 mg/kg), cotton gin trash (0.01–0.10 mg/kg), green coffee beans (0.01–0.10 mg/kg)
- valid (reduced validation) for the determination of fluazifop acid (II) in orange juice (0.01-1.0 mg/kg), orange oil (0.01-1.0 mg/kg), orange dried pulp (0.01-1.0 mg/kg), caneberries (0.02-5.0 mg/kg), tomato paste (at 0.2 mg/kg only), tomato puree (at 0.2 mg/kg only), dry soya bean seed (0.01-1.0 mg/kg), soya bean meal (at 0.2 mg/kg only), soya bean hull (at 0.2 mg/kg only), soya bean oil (at 0.2 mg/kg only), potato flakes (at 0.2 mg/kg only), potato wet peel (at 0.2 mg/kg only), potato chips (at 0.2 mg/kg only), rhubarb (0.02-5.0 mg/kg), wheat flour (at 0.2 mg/kg only), wheat middlings (at 0.2 mg/kg only), wheat shorts (at 0.2 mg/kg only).

- insufficiently validated for the determination of fluazifop acid (II) in orange and soya bean aspirated grain fraction
- not valid for the determination of fluazifop-butyl (no validation)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 M NaOH: acetonitrile (90:10, v/v) and then refluxed for 1 hour shows 110% trueness for carrots and 79% trueness for endive and 131% trueness for carrot foliage).

The valid LOQ is 0.01 mg/kg.

Earlier studies indicated that 14-phenyl labelled fluazifop acid (II) degraded slightly to 81% in solvent [RJ0171B, RJ0196B] or 72% in the presence of soya bean extract (RJ0171B) after 1 hour reflux in aqueous 1 M NaOH. Validation of method GRM044.01A showed that on average fluazifop acid (II) is stable under these conditions.

Table 130 Radiovalidation results for method GRM044.01A

Matrix (sample ID)	Description	Metabolism study [a] 6 M HCl overnight at room temp	GRM044.01A 1 M NaOH: acetonitrile (90:10, v/v) reflux for 1 hour	RSD (%)	n	Trueness (Method/^{14}C)	Code no; Report no
Mature carrot foliage (1689W-019)	^{14}C phenyl; 2 × 0.42 kg ai/ha; DAT 45	TRR, 1.00 mg/kg Fb eq Fluazifop acid (II, free + conj): 0.82 mg/kg Fb eq	Total fluazifop, mg/kg F mean 0.91 range 0.90-0.92	1.1%	3	130% (as F)	PP5_50001; T002223-07
Mature carrot roots (1689W-020)	1^{14}C phenyl; 2 × 0.42 kg ai/ha; DAT 45	TRR = 0.091 mg/kg Fb eq; Fluazifop acid (II, free + conj): 0.058 mg/kg Fb eq	Total fluazifop, mg/kg F mean 0.054 range 0.053-0.057	4.3%	3	109% (as F)	PP5_50001; T002223-07
Mature endive foliage (1690W-022)	^{14}C phenyl; 2 × 0.42 kg ai/ha; DAT 28	TRR = 1.44 mg/kg Fb eq; Fluazifop acid (II, free + conj): 0.71 mg/kg Fb eq	Total fluazifop, mg/kg F mean 0.48 range 0.44-0.51	7.4%	3	79% (as F)	PP5_50001; T002223-07

[a] Residues in fluazifop-butyl equivalents (Fb eq, from metabolism studies) need to be multiplied by 0.856 to get fluazifop acid (II) equivalents, since results from method GRM44.01A are expressed as fluazifop acid (II)

Table 131 Validation results for method GRM044.01A

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration /detection	Code no; Report no
GRM044.01A (no presoak)									
Orange, RAC	0.01	0.01 (F) 1.0 (F)	113 103	- -	- -	1 1	< 0.01	external std in solvent; linear, r>0.9999 0.02-2 ng/mL	A12460A_50019; TK0058357 (processing)
Orange, juice	0.01	0.01 (F) 1.0 (F)	98 99	93-102 96-104	4.7% 4.4%	3 3	< 0.01	external std in solvent; linear, r>0.9999 0.02-2 ng/mL	A12460A_50019; TK0058357 (processing)
Orange, oil	0.01	0.01 (F) 1.0 (F)	91 97	87-93 94-101	3.5% 3.9%	3 3	< 0.01	external std in solvent; linear, r>0.9999 0.02-2 ng/mL	A12460A_50019; TK0058357 (processing)
Orange, dried pulp	0.01	0.01 (F) 1.0 (F)	107 98	101-112 95-101	5.2% 3.1%	3 3	< 0.01	external std in solvent; linear, r>0.9999 0.02-2 ng/mL	A12460A_50019; TK0058357 (processing)

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration /detection	Code no; Report no
tomato	0.01	0.01 (F) 0.10 (F)	85 88	79-99 82-95	9.2% 6.1%	5 5	no data	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5_50029; T002220-07 (validation)
tomato	0.01	0.01 (F) 0.1 (F)	111 107	100-123 102-109	8.5% 2.6%	5 5	< 0.01	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5_50066; ML09-1552-SYN (ILV)
tomato paste	0.01	0.2 (F)	95	83-117	12%	10	< 0.025 (5)	external std in matrix 1/x, r>0.999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
tomato puree	0.01	0.2 (F)	94	73-117	14%	10	< 0.025 (5)	external std in matrix 1/x, r>0.999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
spinach	0.01	0.01 (F) 0.10 (F)	85 104	79-91 98-108	5.5% 3.6%	5 5	no data	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5_50029; T002220-07 (validation)
dry soya bean seed	0.01	0.01 (F) 0.1 (F) 10 (F)	108 108 114	95-121 - 106-121	10% - 6.6%	4 1 3	< 0.01 (5)	external std in solvent; linear, r> 0.99 0.02-2 ng/mL	A12460A_50026; TK0016832
Soya bean, aspirated grain fraction	0.01	0.01 (F) 10 (F)	104 120	- -	- -	1 1	< 0.01 (1)	external std in solvent; linear, r> 0.99 0.02-2 ng/mL	A12460A_50026; TK0016832
soya bean meal	0.01	0.2 (F)	81	71-94	9.6%	10	< 0.025 (5)	external std in matrix 1/x, r>0.999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
soya bean hull	0.01	0.2 (F)	90	71-106	11%	10	< 0.025 (5)	external std in matrix 1/x, r>0.999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
soya bean oil	0.01	0.2 (F)	87	84-91	3.7%	10	< 0.025 (5)	external std in matrix 1/x, r>0.999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
carrots	0.01	0.01 (F) 0.10 (F)	90 105	80-99 100-113	7.5% 5.0%	5 5	no data	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5_50029; T002220-07 (validation)
carrots	0.01	0.01 (F) 0.10 (F)	96 88	91-102 83-94	- -	2 2	< 0.005 (4)	external std in solvent linear, r>0.999 range ns	PP5_50071; T002222-07
potato wet peel	0.01	0.2 (F)	89	80-95	5.6%	10	< 0.025 (5)	external std in matrix 1/x, r>0.9999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
potato chips	0.01	0.2 (F)	77	65-85	7.9%	10	< 0.025 (5)	external std in matrix 1/x, r>0.9999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
wheat flour	0.01	0.2 (F)	75	62-85	9.5%	10	< 0.025 (5)	external std in matrix 1/x, r>0.999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
wheat	0.01	0.2 (F)	75	62-80	7.4%	10	< 0.025	external std	R156172_50001;

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration /detection	Code no; Report no
middlings							(5)	in matrix 1/x, r>0.99999 0.02-2 ng/mL	13SYN331REP stor stab
wheat shorts	0.01	0.2 (F)	83	74-98	8.8%	10	< 0.025 (5)	external std in matrix 1/x, r>0.999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
Cotton seed undelinted	0.01	0.01 (F) 0.10 (F)	89 82	82-93 79-84	5.4% 2.2%	5 5	no data	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5_50029; T002220-07 (validation)
Cotton seed undelinted	0.01	0.01 (F) 0.1 (F) 1.0 (F)	92 90 102	69-113 75-109 86-118	16% 14% -	8 6 2	<0.3LOQ (11)	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5/50076; T002224-07 (procedural validation)
Cotton seed undelinted	0.01	0.01 (F) 0.1 (F)	95 103	83-105 96-113	12% 8.6%	3 3	no data	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5/50076; T002224-07 (method validation)
Cotton seed undelinted	0.01	0.01 (F) 0.1 (F)	78 87	73-88 81-89	7.7 3.9	5 5	< 0.01	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5_50066; ML09-1552-SYN (ILV)
Cotton gin trash	0.01	0.01 (F) 0.1 (F) 1.0 (F)	105 99 110	96-115 96-102 108-112	9.0% 3.4% -	4 3 2	<0.3LOQ (6)	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5/50076; T002224-07 (procedural validation)
Cotton gin trash	0.01	0.01 (F) 0.1 (F)	105 103	102-108 101-106	2.9% 2.1%	3 3	no data	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5/50076; T002224-07 (method validation)
cotton gin trash	0.01	0.01 (F) 0.10 (F)	89 87	82-99 82-95	8.0% 6.0%	5 5	no data	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5_50029; T002220-07 (validation)
cotton gin trash	0.01	0.01 (F) 0.1 (F)	104 105	94-112 101-109	7.6% 3.0%	5 5	< 0.01	external std in solvent; linear, r>0.99 0.02-2 ng/mL	PP5_50066; ML09-1552-SYN (ILV)
green coffee beans	0.01	0.01 (F) 0.10 (F)	81 102	72-95 100-104	11% 1.4%	5 5	no data	external std in solvent; linear, r>0.99 0.02-2 ng/mL	No code; T002220-07 (validation)
GRM044.01A Modification A (including presoak)									
potato flakes	0.01	0.2 (F)	75	64-94	13%	10	< 0.025 (5)	external std in matrix 1/x, r>0.999 0.02-2 ng/mL	R156172_50001; 13SYN331REP stor stab
GRM044.01A Modification B: clean-up by partitioning against dichloromethane									
caneberries	0.02	0.02 (F) 0.2 (F) 0.5 (F) 5.0 (F)	86 91 88 95	77-94 88-96 - 93-97	7.0% 4.4% - 2.1%	8 4 1 3	< 0.02 (6)	external std in solvent, linear, R^2>0.99 2-10 ng/mL	PP5_50556; IR-4 PR 03947
blueberries	0.02	0.02 (F) 0.2 (F) 0.5 (F) 5.0 (F)	91 94 102 89 97	88-96 90- - 97-97	3.3% 4.2% - 0.0%	7 9 1 3	< 0.02 (10)	external std in solvent, linear, R^2>0.99 1-8 ng/mL	PP5_50557; IR-4 PR 02083
strawberries	0.01	0.01 (F) 0.1 (F)	82 85	77-88 77-90	4.9% 5.9%	6 5	< 0.01 (6)	external std in solvent,	PP5_50553; IR-4 PR A2085

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration /detection	Code no; Report no
		1.0 (F) 5.0 (F)	99 104 94 106	96- 88-	3.0% 8.5%	7 4		linear, R^2>0.99 0.25-6 ng/mL	
rhubarb	0.02	0.02 (F) 0.2 (F) 0.5 (F) 5.0 (F)	87 90 92 96	82-90 82-96 - 95-97	3.4% 6.7% - 1.0%	6 4 1 3	< 0.02 (1)	external std in solvent, linear, R^2>0.99 1-8 ng/mL	PP5_50552; IR-4 PR A2404 (2013)

HPLC-MS/MS Method GRM044.02A

Method GRM044.02A is proposed for use as enforcement method. Method GRM044.02A determines total fluazifop (i.e. fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant commodities as one single analyte (common moiety) and residues are expressed as fluazifop acid. The reported LOQ is 0.01 mg/kg.

Method GRM044.02A is described by [Edwards and Braid, 2010, PP5_10112, report GRM044.02A]. Crop commodities with high (>60%) water content (e.g. apples, strawberries, lettuce, 20 g) are extracted by maceration with acetonitrile/concentrated HCl (98:2 v/v). An aliquot of the extract is evaporated to dryness. Oily and dry crop commodities (e.g. oilseeds, pulses, 10 g) are soaked in 1 M HCl for at least 2 hours and are then extracted by maceration in acetonitrile/1 M HCl (50:50, v/v). The acetonitrile is removed from an aliquot of the extract by evaporation until the oily/aqueous solution remains. Concentrated HCl is added to the oily/aqueous remainder (from dry or oily crops) to give 6 M HCl or the dry residuum (from crops with high water content) is dissolved in 6 M HCl. The acid solution (6 M HCl) is hydrolysed for 1 hour at 60 ºC to convert any ester or acid conjugates to total fluazifop. Endogenous plant co-extractives are separated by adsorption chouromatographic clean-up using reverse phase SPE cartridges and methyl-t-butyl ether/methanol (90:10, v/v) as eluent. The eluate is evaporated to dryness and reconstituted in HPLC mobile phase (water/acetonitrile/formic acid, 95/5/0.4, v/v/v). Total fluazifop is determined by HPLC-MS/MS at mz/ 328 to 282 (primary transition) and m/z 328 to 254 (confirmatory transition). Calibration is by fluazifop acid (II) external standardization.

A validation study as well as an independent laboratory validation is available [Marshal, 2010, PP5_10084, report CEMR-4218-REG, Gemrot, 2010, PP5_10101, report S10-01917-REG]. Results are shown in Table 132. For the primary transition (m/z 328 to 282). Acceptable mean recoveries between 70% and 120% with a relative standard deviation lower than 20% were also found for the confirmatory transition (m/z 328 to 254) in all matrices tested. No significant enhancement or suppression of detector response (<10%) was observed in the presence of potato, apple, tomato, cabbage, onion, artichoke, green pea seeds or soya bean seed matrices. Therefore standards in solvent may be used for calibration. Significant enhancement or suppression of detector response was observed in the presence of orange fruit (+4% to +14%), raspberry (+12%), dried peas (-68%), and oilseed rape seed (-11% to +27%) matrices. Therefore matrix matched standards need to be used for calibration for these matrices.

Method GRM044.02A was used in a supervised residue trial on apples [CEMR-4968], strawberries [CEMR-5448], lettuce [CEMR-5451], green beans (pods, haulms) [CEMR-4384-REG], green peas (pods, seeds, forage) [CEMR-4658-REG, CEMR-5453], dry peas [CEMR-4385-REG], oilseed rape seeds [CEMR-5449] and in a processing study on green peas [CEMR-4751-REG] and dry peas [CEMR-5037-REG]. Validation results are shown in Table 132. Quantification was performed by fluazifop acid (II) external standardization using single point (apple, green peas (pods, seeds, haulms)) or multi point calibration with 1/x weighted regression (strawberries, oilseeds, green peas (seeds, haulms), pulses). For strawberries [CEMR-5448] no significant enhancement or suppression of detector response (+9.6%) was found.

Modification A of method GRM044.02A was used in supervised trials on strawberries [CEMR-6043]. Samples were sonicated after addition of HCl to increase the recovery. The SPE cartridge was eluted with methyl tert-butyl ether/methanol (90:10 v/v) + 4% (volume) ammonium hydroxide (20% solution, 5.7 M) as the original solvent resulted in poor recovery. Validation results are shown in Table 132.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-MS/MS Method GRM044.02A is considered:

- valid (full validation) for the determination of fluazifop acid (II) in orange (0.01–0.1 mg/kg), apple (0.01–0.2 mg/kg), raspberry (0.01–0.2 mg/kg), strawberry (0.01–0.1 mg/kg), onion (0.01–0.3 mg/kg), cabbage (0.01–0.3 mg/kg), tomato (0.01–0.3 mg/kg), green pea seeds (0.01–1.0 mg/kg), green pea seeds canned, cooked or blanched (0.01–0.1 mg/kg), dry soya bean seeds (0.01–5.0 mg/kg), dry pea seeds (0.01–5.0 mg/kg), dry pea seeds cooked, steeped, blanched or canned (0.01–0.5 mg/kg), potato (0.01–0.1 mg/kg), artichoke (0.01–0.1 mg/kg), oilseed rape seeds (0.01–15 mg/kg),
- valid (reduced validation) for the determination of fluazifop acid (II) in lettuce (0.1 mg/kg only),
- insufficiently validated for the determination of fluazifop acid (II) in green beans with pods, green bean haulms, green peas with pods, green pea forage,
- not valid for the determination of fluazifop-butyl (no validation)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).
- suitable for use as enforcement method for the determination of fluazifop acid (II) in commodities with high acid, commodities with high water, commodities with high protein, commodities with high starch and commodities with high oil content, since validation for a confirmatory method as well as an independent laboratory validation as well as a radiovalidation study has been submitted.

The valid LOQ is 0.01 mg/kg.

Table 132 Validation results for method GRM044.02A

Matrix	LOQ	Spike level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	m/z 328 to 282 calibration	Code no; Report no
orange (whole fruit)	0.01	0.01 (F) 0.1 (F)	83 89	79-86 84-94	3.1% 4.9%	5 5	<0.3 LOQ (2)	HPLC-MS/MS matrix matched 0.0025-0.5 mg/L linear, R2>0.999	PP5_10084; CEMR-4218-REG; validation
orange (whole fruit)	0.01	0.01 (F) 0.10 (F)	95 89	86-106 82-94	8% 6%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent; 0.0025-0.1 mg/L 0.5-20× LOQ R2>0.999	PP5_10101; S10-01917-REG; (ILV)
apple	0.01	0.01 (F) 0.2 (F)	102 106	83-115 97-139	11% 17%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent 0.0025-0.5 mg/L linear, R2>0.999	PP5_10084; CEMR-4218-REG; validation
apple	0.01	0.01 (F) 0.2 (F)	113 96	- -	- -	1 1	< 0.01 (2)	HPLC-MS/MS matrix matched linear, R2>0.99 0.0025-0.5 mg/L	A12791B_10841; CEMR-4968
raspberry	0.01	0.01 (F) 0.2 (F)	80 90	73-92 76-101	9.3% 11%	5 5	<0.3 LOQ (2)	HPLC-MS/MS matrix matched; 0.0025-0.5 mg/L linear, R2>0.999	PP5_10084; CEMR-4218-REG; validation

Matrix	LOQ	Spike level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	m/z 328 to 282 calibration	Code no; Report no
straw berries	0.01	0.01 (F) 0.1 (F)	87 88	82-92 83-93	4.6% 4.9%	5 5	< 0.01 (4)	HPLC-MS/MS solvent; linear, R2>0.999 0.0015-0.5 mg/L	A12791A_10077; CEMR-5448
onion	0.01	0.01 (F) 0.3 (F)	71 84	68-74 80-89	3.7% 3.9%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent 0.0025-0.5 mg/L linear, R2>0.999	PP5_10084; CEMR-4218-REG; validation
cabbage	0.01	0.01 (F) 0.3 (F)	78 92	75-80 85-96	2.8% 4.5%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent 0.0025-0.5 mg/L linear, R2>0.999	PP5_10084; CEMR-4218-REG; validation
tomato	0.01	0.01 (F) 0.3 (F)	74 85	70-81 82-88	6.1% 2.8%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent 0.0025-0.5 mg/L linear, R2>0.999	PP5_10084; CEMR-4218-REG; validation
tomato	0.01	0.01 (F) 0.30 (F)	80 85	70-87 80-89	8% 4%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent; 0.0025-0.1 mg/L 0.5-20× LOQ R2>0.99	PP5_10101; S10-01917-REG; (ILV)
lettuce	0.01	0.01 (F) 0.10 (F) 5.0 (F) 30 (F)	71 74 103 103	71-71 70-81 103-103 -	- 8.2% - -	2 3 2 1	< 0.01 (6)	HPLC-MS/MS in solvent linear, R2>0.999 0.0015-0.15 mg/L	A12791B_11028 CEMR-5451
green bean pods	0.01	0.01 (F) 2.0 (F)	64 80	- -	- -	1 1	< 0.01 (4)	HPLC-MS/MS in solvent linear, R2>0.999 0.0025-0.5 mg/L	A12791B_10829; CEMR-4384-REG
green bean haulms	0.01	0.01 (F) 2.0 (F)	74 87	- -	- -	1 1	< 0.01 (4)	HPLC-MS/MS in solvent linear, R2>0.999 0.0025-0.5 mg/L	A12791B_10829; CEMR-4384-REG
green pea seeds	0.01	0.01 (F) 1.0 (F)	71 78	67-78 70-87	6.2% 8.2%	5 5	<0.3 LOQ (2)	HPLC-MS/MS matrix matched; 0.0025-0.5 mg/L linear, R2>0.999	PP5_10084; CEMR-4218-REG; validation
green pea seeds	0.01	0.01 (F) 0.1 (F)	70 92	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS Matrix matched linear, R2>0.999 0.0025-0.5 mg/L	A12791B_10837; CEMR-4658-REG
green pea seeds	0.01	0.01 (F) 0.10 (F) 0.50 (F)	113 95 100	- - -	- - -	1 1 1	< 0.01 (4)	HPLC-MS-MS Matrix matched linear, R2>0.99 0.0015-0.5 mg/L	A12791B_11035 CEMR-5453
green pea seeds	0.01	0.01 (F) 0.5 (F)	87 75	- -	- -	1 1	< 0.01–0.32 (8)	external std in solvent; linear, r2>0.99; 0.02-2 ng/mL	A12791B_11029; CEMR-4751-REG (processing)
green pea seeds, canned	0.01	0.01 (F) 0.1 (F)	94 89	83-103 80-95	8.3% 6.4%	5 5	< 0.01–0.21 (6)	external std in solvent; linear, r2>0.999; 0.02-2 ng/mL	A12791B_11029; CEMR-4751-REG (processing)
green pea seeds, cooked	0.01	0.01 (F) 0.1 (F)	91 87	84-98 86-90	5.6% 1.7%	5 5	< 0.01–0.24 (2)	external std in solvent; linear, r2>0.999; 0.02-2 ng/mL	A12791B_11029; CEMR-4751-REG (processing)
green pea seeds, blanched	0.01	0.01 (F) 0.1 (F)	75 86	64-90 75-94	13 % 8.7%	5 5	< 0.01–0.34 (2)	external std in solvent; linear, r2>0.999; 0.02-2 ng/mL	A12791B_11029; CEMR-4751-REG (processing)
green pea pods	0.01	0.01 (F) 0.5 (F)	71 76	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS Matrix matched	A12791B_10837; CEMR-4658-REG

Matrix	LOQ	Spike level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	m/z 328 to 282 calibration	Code no; Report no
								linear, R2>0.999 0.0025-0.5 mg/L	
green pea forage	0.01	0.01 (F) 0.5 (F)	90 93	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS Matrix matched linear, R2>0.99 0.0025-0.5 mg/L	A12791B_10837; CEMR-4658-REG
green pea forage	0.01	0.01 (F) 0.10 (F) 0.5 (F) 2.0 (F)	96 98 77 69	81-110 - - -	- - - -	2 1 1 1	< 0.01 (4)	HPLC-MS-MS Matrix matched linear, R2>0.99999 0.0015-0.5 mg/L	A12791B_11035 CEMR-5453
dry soya bean seeds	0.01	0.01 (F) 5.0 (F)	91 100	87-99 96-103	5.6% 3.0%	5 5	<0.3 LOQ (2)	HPLC-MS/MS 0.0025-0.5 mg/L in solvent R2>0.999	PP5_10084; CEMR-4218-REG; validation
dry pea seeds	0.01	0.01 (F) 5.0 (F)	86 76	70-103 69-82	14% 6.9%	5 5	<0.3 LOQ (2)	HPLC-MS/MS matrix matched 0.0025-0.5 mg/L R2>0.99	PP5_10084; CEMR-4218-REG; validation
dry pea seeds	0.01	0.01 (F) 1.0 (F)	87 74	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS matrix matched; linear, R2=1.0000 0.0025-0.5 mg/L	A12791B-10831; CEMR-4385-REG
dry pea seeds (RAC, cleaned or washed)	0.01	0.01 (F) 0.1 (F) 0.5 (F) 1.0 (F) 5.0 (F)	85 71 79 89 121	74-92 - 77-81 - -	9.1% - - - -	4 1 2 1 1	< 0.01	external std in solvent; linear, r2>0.99 0.02-2 ng/mL	A12791B_11068; CEMR-5037-REG; (processsing)
dry pea seed (cooked, steeped, blanched, canned)	0.01	0.01 (F) 0.5 (F) 1.0 (F) 5.0 (F)	84 81 88 99	74-97 73-88 - -	11% 7.6% - -	6 5 1 1	< 0.01	external std in solvent; linear, r2>0.99 0.02-2 ng/mL	A12791B_11068; CEMR-5037-REG; (processsing)
dry pea straw	0.01	0.01 (F) 1.0 (F)	68 89	- -	- -	1 1	< 0.01 (2)	HPLC-MS-MS matrix matched; linear, R2>0.99 0.0025-0.5 mg/L	A12791B-10831; CEMR-4385-REG
potato	0.01	0.01 (F) 0.1 (F)	71 78	70-72 75-82	1.1% 3.4%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent; 0.0025-0.5 mg/L linear, R2>0.999	PP5_10084; CEMR-4218-REG; validation
potato	0.01	0.01 (F) 0.10 (F)	94 77	88-102 69-83	7% 7%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent; 0.0025-0.1 mg/L 0.5-20× LOQ R2>0.9999	PP5_10101; S10-01917-REG; (ILV)
artichoke	0.01	0.01 (F) 0.5 (F)	79 94	76-83 90-97	3.4% 2.8%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent 0.0025-0.5 mg/L linear, R2>0.999	PP5_10084; CEMR-4218-REG; validation
oilseed rape seeds	0.01	0.01 (F) 15.0 (F)	76 73	65-85 68-76	9.7% 4.9%	5 5	<0.3 LOQ (2)	HPLC-MS/MS matrix matched; 0.0025-0.5 mg/L R2>0.999	PP5_10084; CEMR-4218-REG; validation
oilseed rape seed	0.01	0.01 (F) 15 (F)	78 72	63-92 64-75	16% 6%	5 5	<0.3 LOQ (2)	HPLC-MS/MS in solvent; 0.0025-0.1 mg/L 0.5-20× LOQ R2>0.999	PP5_10101; S10-01917-REG; (ILV)
oilseed rape seeds	0.01	0.01 (F) 0.1 (F) 5.0 (F) 7.5 (F)	86 85 68 94	83-93 77-91 - -	4.5% 6.3% - -	5 5 1 1	<0.3 LOQ (2)	HPLC-MS-MS Matrix matched linear, R2>0.99 0.0015-0.5 mg/L	A12791B_11249; CEMR-5449

Matrix	LOQ	Spike level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	m/z 328 to 282 calibration	Code no; Report no
GRM044.02A Modification A									
straw berries	0.01	0.01 (F) 0.5 (F)	95 104	- -	- -	1 1	< 0.01 (4	HPLC-MS/MS matrix matched; linear, R2>0.9999 0.0015-0.5 mg/L	A12791B_11992; CEMR-6043

HPLC-MS/MS Method POPIT MET.138.Rev.00 and its modifications

Method POPIT.MET.138.Rev.00 and its modifications determine total fluazifop (i.e. fluazifop-butyl, fluazifop acid (II) and its conjugates) in plant commodities as one single analyte (common moiety) and residues are expressed as fluazifop acid (II). The reported LOQ is 0.01 mg/kg.

Method POPIT.MET.138.Rev.00 (26 October 2011) is described by [Suzuki, 2011, no code, report POPIT.MET.138.Rev.00]. Samples (5 g) with moisture content ≥ 60% (potato, sugarcane) are extracted by maceration in acetonitrile: conc. HCl (98:2 v/v). Samples with a moisture content of less than 60% were first kept in 1 M HCl for at least 2 hours or overnight, followed by maceration with acetonitrile (acetonitrile/1 M HCl, 50:50, v/v). From an aliquot of the extract, the acetonitrile is removed by evaporation.The remainder is hydrolysed with 6 M HCl (1 hour, 60 ºC) to convert fluazifop-butyl or fluazifop (II) conjugates to fluazifop acid (II). The sample is loaded onto an SPE (Oasis HLB) cartridge, eluted with acetonitrile and evaporated to dryness. The residue is redissolved in a solution of water:acetonitrile:formic acid (95:5:0.2, v/v/v) and filtered thourough 0.22 µm. The extracts are analysed by HPLC-MS/MS at m/z 328 to 282. The residues are quantified against a fluazifop acid (II) external standard.

Method POPIT.MET.138.Rev.02 (18 January 2012) is described by [Maslowski, 2012, no code, report POPIT.MET.138.Rev.02] and POPIT.MET.138.Rev.08 (21 October 2013) is described by [Weissenberg, 2013, 18664401MDC2, report POPIT.MET.138.Rev.08]. In total, 8 revisions of the method were reported, but they included only addition of data on new crop matrices: rev 01 (carrot, tomato, lettuce), rev 02 (additional carrot data), rev 03 (onion), rev 04 (cotton seeds, dry soya bean seeds, dry beans), rev 05 (additional soya bean and bean data) rev 06 (manioc and cabbage), rev 07 (additional cabbage data), rev 08 (textual changes). The wet sample protocol is followed for: potato sugarcane, carrot, tomato, lettuce, onion, manioc and cabbage. The dry and oily sample protocol is followed for cotton, dry soya beans and dry beans. Validation results derived from supervised residue trials are included in the method description and are shown in Table 133.

Method POPIT.MET.138.Rev 00 to 08 were used on supervised trials on bulb onions [M11026], head cabbage [M12060], tomatoes [M11033], lettuce [M11028], dry beans [M11034], dry soya beans [M13030, M11032,], carrots [M11030], potatoes [M11031], cottonseed [M11027] and sugar cane [M11029]. Validation results are shown in Table 133.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-MS/MS Method POPIT.MET.138.Rev 00-08 is considered:

- valid (full validation) for the determination of fluazifop acid (II) in bulb onion (0.01–0.1 mg/kg), cabbage (0.01–2.0 mg/kg), tomato (0.01–2.0 mg/kg), lettuce (0.01–0.1 mg/kg), dry soya bean seeds (0.01–4.0 mg/kg), dry bean seeds (0.01–2.0 mg/kg), potato (0.01–0.1 mg/kg), carrot (0.01–2.0 mg/kg), manioc (0.01–0.1 mg/kg), sugar cane (0.01–0.1 mg/kg) and cotton seed (0.01–2.0 mg/kg)
- not valid for the determination of fluazifop-butyl (no validation)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).

The valid LOQ is 0.01 mg/kg.

Table 133 Validation results for method POPIT MET 138 (Revisions 00 to 08)

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no Report no
POPIT.MET.138.Rev 00 to 08									
Onion	0.01	0.01 (F) 0.1 (F)	80 85	71- 90	7.1% 2.2%	7 5	< 0.01 (4)	Linear, R^2 >0.999	A12530B_10016; M11026
				87-92					
Head cabbage	0.01	0.01 (F) 0.1 (F) 2.0 (F)	79 85 82	70- 89 72-	7.3% 8.0% 6.8%	7 5 5	< 0.01 (4)	Linear, R^2 >0.99	A12530D_10013; M12060
			89	80-96					
Tomato	0.01	0.01 (F) 0.1 (F)	89 95	86-	3.6% 10%	7 5	< 0.01 (5)	Linear, R^2 >0.999	M11033; A12530B_10020
			81	71-89					
Lettuce	0.01	0.01 (F) 0.1 (F)	87 99	77-	7.8% 3.4%	7 5	< 0.01 (4)	Linear, R^2 >0.999	M11028; A12530B-10013
			83	80-87					
Dry Bean seeds	0.01	0.01 (F) 0.1 (F) 2.0 (F)	75 78 89	72- 86-	2.6% 2.2% 6.6%	7 5 5	< 0.01 (4)	Linear, R^2 >0.99	M11034; A12530B_10018
			91 93	86-103					
Dry soya bean seed	0.01	0.01 (F) 0.1 (F) 4.0 (F)	90 92 85	78- 71-	6.0% 10% 4.0%	7 5 5	<0.3 LOQ (4)	Linear, R^2 >0.99	A13680D_ 10051; M13030;
			93 119	112-125					
Dry soya bean seed	0.01	2.0 (F)	106	104-110	2.5%	5	<0.3 LOQ (4)	Linear, R^2 >0.99	A12530B_10015; M11032
Carrot	0.01	0.01 (F) 0.1 (F) 0.2 (F)	93 98 92	82- 88-	6.1% 2.9% 0.5%	7 5 5	< 0.01	Linear, R^2 >0.99	A12530B_10012; M11030
			95 103	103-104					
Manioc (cassava)	0.01	0.01 (F) 0.1 (F)	100 107	92-	6.0% 7.1%	7 5	< 0.01	Linear, R^2 >0.99	18664401MDC2; POPIT.MET.138.Rev.08
			96	86-105					
Potato	0.01	0.01 (F) 0.1 (F)	83 92	78-	6.1% 7.7%	7 5	< 0.01	Linear, R^2 > 0.9999	A12530B_10019; M11031
			79	70-94					
Cotton seed	0.01	0.01 (F) 0.1 (F)	78 86	73-	5.6% 4.4%	7 5	<0.3 LOQ (4)	Linear, R^2 >0.999	A12530B_10014; M11027
			86	80-89					
Sugar cane	0.01	0.01 (F) 0.1 (F)	84 90	72-	8.4% 7.1%	7 5	< 0.01	Linear, R^2 > 0.99	A12530B_10011; M11029
			77	73-86					

HPLC-MS/MS method MRID 40831305

HPLC-MS/MS method MRID 40831305 determines total fluazifop (fluazifop-butyl, fluazifop acid (II) and its conjugates) as one single analyte (common moiety) in sweet potatoes and residues are expressed as fluazifop acid (II). The reported LOQ is 0.02 mg/kg.

HPLC-MS/MS Method MRID 40831305 is a modification of HPLC-UV method PPRAM 62/2. HPLC-MS/MS method MRID 40831305 (26 May 2011) was used and described in the residue trial on sweet potato [Barney, 2011, PP5_50290, report IR-4 PR 02328] and grass (forage, hay) [PP5_50554, IR-4 PR 09825]. Homogenized samples (10 g) are soaked for 10 min with acetonitrile/concentrated hydrochloric acid (98:2, v/v) and then blended. The extract is evaporated to remove all acetonitrile. The aqueous extract is hydrolysed to convert fluazifop-butyl or fluazifop (II) conjugates to fluazifop acid (II) by addition of concentrated HCl to get 6 M HCl (60°C, 1 hour). The hydrolysate is partitioned once or twice with dichloromethane. The dichloromethane solution is evaporated to dryness and the residue is reconstituted in acetonitrile/water (40:60, v/v; sweet potato) or 0.1% formic acid and acetonitrile (grass) for HPLC-MS/MS analysis (m/z 326 to 254). Concurrent method validation results extracted from supervised trial reports are summarized in Table 134.

No radiovalidation was conducted, but extraction and hydrolytic efficiency for total fluazifop have been evaluated in the radiovalidation study for PPRAM 62.

Reviewer's conclusion: HPLC-MS/MS method MRID 40831305 is considered:

- valid (full validation) for determination of fluazifop acid (II) in sweet potatoes (at 0.02–5.0 mg/kg) grass forage (0.02–5.0 mg/kg) and grass hay (0.02–5.0 mg/kg).
- not valid for the determination of fluazifop-butyl (no validation results)
- valid for the determination of fluazifop (II) conjugates (radiovalidation for 1 hour 6 M HCl at 60 °C shows 99% trueness for carrots and 69% trueness for endive).

The valid LOQ is 0.02 mg/kg (no validations below this point).

Table 134 Validation results for HPLC-MS/MS method MRID 40831305

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Ref.
sweet potato	0.02	0.02 (F) 0.05 (F) 5.0 (F)	84 94 89 94 100 103	71- 84- 96- 	12% 5.5% 3.1%	6 9 4	< 0.02	external std; linear, $r^2 > 0.97$	PP5_50290; IR-4 PR 02328
grass, forage	0.02	0.02 (F) 0.2 (F) 0.5 (F) 5.0 (F)	80 82 83 84 70 91 98	77- 81- 87- 	2.1% 1.6% - 4.5%	6 5 1 6	< 0.02 (6)	linear by graph	PP5_50554; IR-4 PR 09825
grass, hay	0.02	0.02 (F) 0.2 (F) 0.5 (F) 5.0 (F) 10 (F)	73 76 77 82 77 85 93 92 96	70- 69- 80- 87- 	3.4% 7.6% - 6.3% 4.4%	6 4 1 7 4	< 0.02 (6)	linear by graph	PP5_50554; IR-4 PR 09825

Analytical methods used in study reports in animal commodities

Several analytical methods were submitted for use in feeding studies and storage stability studies on animal commodities.

GC-MS and HPLC-UV method PPRAM 58

GC-MS and HPLC-UV method PPRAM 58 determines total fluazifop (fluazifop-butyl, free fluazifop acid (II) and its conjugates) in animal commodities as one single analyte (common moiety) and residues are expressed as fluazifop. The reported LOQ is 0.01 mg/kg for the GC-MS detection and 0.02 mg/kg for the HPLC-UV detection.

Method PPRAM 58 was summarized by [Atreya, 1990, 463114, report M5159B]. Samples are extracted with chloroform: methanol (1:1, v/v). For tissue samples the extracts are diluted with water, acidified to pH 1 and partitioned into chloroform. The chloroform phase is evaporated to dryness and the residue taken up into 0.2 M NaOH in methanol. Samples of eggs are extracted in the same manner but the total extract is evaporated to dryness directly prior to taking up into methanolic NaOH. The methanolic 0.2 M NaOH is heated under reflux for at least 1 hour in order to hydrolyse fluazifop-butyl and fluazifop (II) conjugates into fluazifop. The hydrolysate is diluted with water, acidified to pH 1 and the fluazifop acid (II) partitioned into chloroform. The chloroform is shaken with 1% w/v sodium bicarbonate solution. After discarding the chloroform and after acidification of the aqueous layer, the fluazifop acid (II) is back extracted into chloroform. Fluazifop acid (II) is then treated with diazomethane to form the methyl ester. The methylated extracts are then subjected to Florisil adsorption column chouromatography to remove co-extractives. The methyl ester of fluazifop acid (II) is quantitatively determined using GC-MS (m/z 341) using an external standard for fluazifop-methyl.

As confirmation, fluazifop acid (II) can be quantified by HPLC-UV (270 nm) using an external standard for fluazifop. Residues are expressed as fluazifop acid (II) in both cases.

Method PPRAM 58 was used in a feeding study on poultry [Swaine and Francis, PP9/0183, report RJ0217B]. Validation results are shown in Table 135.

A radiovalidation study is summarized in [Atreya, 1990, 463114, report M5159B]. Actual results are not shown. The extraction and hydrolysis procedures from PPRAM 58 have been used on radiolabelled samples from a metabolism study on cow and chicken [Evans *et al*, 1981, PP9/0180, report RJ0207B; Day *et al*, 1981, PP9/0181, report RJ0212B].

- The solvent extraction system (chloroform/methanol, 50:50, v/v) as used in PPRAM 58 removed 100% of the radioactivity from chicken whole eggs (TRR = 0.007 mg/kg Fb eq) and 95% of the radioactivity from cow liver tissue (TRR = 0.024 mg/kg Fb eq).
- The hydrolysis procedure (0.2 M methanolic NaOH, 1hour reflux) as used in PPRAM 58 cleaved >90 % of the lipophilic conjugates of fluazifop acid (II) in egg yolk and bovine milk. Results in mg/kg are not shown.
- Fluazifop-butyl was demonstrated to be converted 100% to fluazifop acid (II) by the adopted hydrolysis conditions.

Reviewer's remark: In the metabolism studies 47% TRR of 0.020 mg/kg eq in egg yolk and 68% TRR of 0.048 mg/kg Fb eq in milk are characterised as lipophilic conjugates using 0.5 M methanolic NaOH (3 hours reflux) for egg yolks or 0.1 M aqueous NaOH (1 hour reflux) for milk, respectively. Significant levels of polar conjugates of fluazifop acid (II) were not present in these matrices. The hydrolysis conditions as used in the hen metabolism study (0.5 M methanolic NaOH, 3 hours reflux) slightly cleave the fluazifop acid (II, recovery 88%). Since the hydrolysis conditions as used in PPRAM 58 (0.2 M methanolic NaOH, 1 hour reflux) result in similar total fluazifop residues, it is concluded that the cleavage of fluazifop acid (II) under these conditions remains within acceptable levels (minimum 0.90×88=79%). Extraction and hydrolytic efficiency are considered acceptable.

Reviewer's conclusion: GC-MS Method PPRAM 58 is considered:

- valid (full validation) for the determination of fluazifop acid (II) in hen eggs (0.02–0.1 mg/kg), hen tissues (0.02–0.1 mg/kg) and hen liver (0.05–0.1 mg/kg)
- valid for the determination of fluazifop-butyl (radiovalidation) in the same matrices
- valid for the determination of fluazifop lipophilic conjugates (radiovalidation in hen eggs and cow liver); validation of polar conjugates is not relevant, since levels of polar conjugates are very low in animal tissues, milk and eggs (< 5% TRR).

The valid LOQ is 0.02 mg/kg (no validations below this level).

Table 135 Validation results for GC-MS method PPRAM58

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
mixed hen tissues (muscle with fat and skin)	0.01	0.02 F 0.05 Fb 0.1 F	81 92 84	68-94 72-117- 64-111	16% 17% 16%	3 6 9	< 0.01 (1)	no data	PP9/0813; RJ0217B
hen liver	0.02	0.05 Fb 0.05 F 0.1 F	78 101 89	64-86 - 62-110	16% - 18%	3 1 7	< 0.02 (1)	no data	PP9/0813; RJ0217B
hen eggs	0.02	0.02 F 0.05 F 0.05 Fb 0.1 F 0.2 F	94 79 99 78 72	73-109 65-91 94-104 67-85 -	20% 13% - 9.5% -	3 5 2 5 1	< 0.02 (3)	no data	PP9/0813; RJ0217B

LOQ = LOQ reported in the study report

Fb = fortification with fluazifop-butyl;

F = fortification with fluazifop

RSD = relative standard deviation

Control = residue levels measured in untreated control samples

HPLC-UV and GC-MS Method PPRAM 61

HPLC-UV and GC-MS method PPRAM 61 describe the separate determination of the polar fluazifop conjugates and the lipophilic fluazifop conjugates in animal commodities. Residues are expressed as fluazifop acid (II). The reported LOQ is 0.01 mg/kg for milk and 0.02 mg/kg for tissues.

Method PPRAM 61/1 (28 March 1983) is described by [Atreya *et al*, 1983, PP9/0034, report PPRAM 61/1] and method PPRAM 61 was summarized by [Atreya, 1990, 463113, report M5164B]. The method differs for milk, tissues and fat:

- Milk samples (20 mL) are extracted by blending with acetonitrile: acetone: hexane (1:1:1 v/v). The acetonitrile: acetone fraction contains fluazifop-butyl, free fluazifop acid (II) and polar fluazifop conjugates (not present in milk) and while the hexane fraction contains lipophilic conjugates of fluazifop. The hexane fraction is cleaned-up by 'Florisil' column chouromatography and subjected to hydrolysis with 0.2 M methanolic NaOH (1hour reflux) to convert lipophilic conjugates to fluazifop acid (II). The acetonitrile/acetone fraction and the hydrolysed fraction are cleaned-up separately. They are diluted with water, adjusted to pH 1 and fluazifop-butyl and fluazifop acid (II) are partitioned into dichloromethane. The dichloromethane phase from the hydrolysed fraction is partitioned into 1% (w/v) sodium bicarbonate solution. The sodium bicarbonate aqueous phase is acidified to pH 1 and then back-partitioned into dichloromethane. The two dichloromethane fractions (corresponding to fluazifop acid (II) and lipophilic fluazifop conjugates) are evaporated to dryness and redissolved in acetonitrile/water (65:35 or 50:50, v/v) and then analysed separately by HPLC-UV (270 nm) using external standards for fluazifop acid (II) and fluazifop-butyl.

- Tissue samples (10 g) are extracted by blending with acetonitrile: acetone: hexane (1:1:1 v/v). The acetonitrile: acetone fraction contains fluazifop-butyl, fluazifop acid (II) and its polar conjugates, while the hexane fraction contains lipophilic conjugates of fluazifop acid (II). The acetonitrile/acetone fraction is diluted with water, adjusted to pH 1 and partitioned into dichloromethane. The hexane fraction is cleaned-up by 'Florisil' column chouromatography. The dichloromethane phase and the Florisil eluate are evaporated to dryness and than subjected (separately) to hydrolysis with 0.2 M methanolic NaOH (1hour reflux) to convert fluazifop-butyl and/or fluazifop (II) conjugates to fluazifop acid (II). Hydrolysates from the acetonitrile/acetone and hexane fractions are cleaned-up separately. The hydrolysates are diluted with water, adjusted to pH 1 and partitioned into dichloromethane. The dichloromethane phase is partitioned into 1% (w/v) sodium bicarbonate solution. The sodium bicarbonate aqueous phase is acidified to pH 1 and then back-partitioned into dichloromethane. The dichloromethane phase from the acetonitrile/acetone extract is evaporated to dryness, redissolved in chloroform and then cleaned-up by adsorption chouromatography (Fractosil). The eluate is evaporated to dryness and then redissolved in diethyl ether. Fluazifop acid (II) is converted to the methyl ester with diazomethane (2 hour, room temperature). The extract is evaporated to dryness, redissolved in hexane and analysed by GC-MS at m/z 341, 322, 282, 254 using in-situ derivatised fluazifop-methyl external standards (representing fluazifop-butyl, free fluazifop acid (II) and polar fluazifop conjugates).The dichloromethane phase from the hexane extracts is evaporated to dryness, redissolved in acetonitrile/water (50:50, v/v) and analysed by HPLC-UV using external standards for fluazifop acid (II) (representing lipophilic conjugates).
- Fat samples (10 g) are extracted by homogenisation in 50% methanol in chloroform, followed by boiling under reflux for 2 hours. The extract is evaporated to dryness and then hydrolysed with 0.2 M methanolic NaOH (1hour reflux). The hydrolysed fraction is diluted with water, adjusted to pH 1 and fluazifop acid (II) is partitioned into dichloromethane. The dichloromethane phase is partitioned into 1% (w/v) sodium bicarbonate solution. The sodium bicarbonate aqueous phase is acidified to pH 1 and then back-partitioned into dichloromethane. The dichloromethane phase is evaporated to dryness, redissolved in chloroform and then cleaned-up by adsorption chouromatography (Fractosil). The eluate is evaporated to dryness and then redissolved in diethyl ether. Fluazifop acid (II) is converted to the methyl ester with diazomethane (2 hours, room temperature). The extract is evaporated to dryness, redissolved in hexane and analysed by GC-MS using in-situ derivatised fluazifop-methyl external standards (representing fluazifop-butyl, free fluazifop acid (II) and its conjugates).

Method PPRAM 61 was used in a feeding study on cows [Atreya *et al*., 1981, PP9/0182, report RJ0215B]. Validation results are shown in Table 136.

A radiovalidation study is summarized in [Atreya, 1990, 463113, report M5164B]. Actual results are not shown. The extraction procedures from PPRAM 61 have been used on radiolabelled samples from a cow metabolism study [Evans *et al*, 1981, PP9/0180, report RJ0207B].

- Milk (day 5, approximately 0.034 mg/kg Fb eq) and cow liver (0.024 mg/kg Fb eq) were extracted with acetonitrile/acetone/hexane (1:1:1, v/v). The residuum from both types of samples was re-extracted with the same solvent system and solid debris was combusted. The solvent extraction system removed >95% TRR from the milk, of which 89% TRR was in the hexane fraction and 7% remained in the acetonitrile/acetone fraction. The solvent extraction system removed >95% TRR from liver tissue of which 89% TRR was in the acetonitrile/acetone fraction and 6% TRR remained in the hexane fraction. Both fractions are analysed in method PPRAM 61.
- Hydrolysis efficiency for lipophilic conjugates in milk using caustic alcohol solution has been described for PPRAM 58.

Reviewer's conclusion: HPLC-UV Method PPRAM 61 is considered:

- valid (reduced validation) for the determination of fluazifop acid (II) in cow milk (0.05–0.1 mg/kg) and cow muscle (0.05–0.1 mg/kg), cow liver (0.05–0.1 mg/kg), cow kidney (0.05–0.1 mg/kg), cow fat (0.05 mg/kg only)
- valid for the determination of fluazifop-butyl in the same matrices
- valid for the determination of fluazifop lipophilic conjugates (radiovalidation for milk and cow liver showed acceptable extraction and hydrolytic efficiency).

The valid LOQ is 0.05 mg/kg (no radiovalidations below this point).

Note: It is not clear whether the method is capable of extraction and analysis of fluazifop acid (II) derived from polar conjugates. Since levels of fluazifop polar conjugates are very low in animal tissues, milk and eggs (<5% TRR), this is considered to have no impact on the residue results.

Table 136 Validation results for HPLC-UV method PPRAM61

Matrix	LOQ	Fortification level (mg/kg) [b]	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
cow milk	0.01	0.05-0.1 (Fb) 0.05-0.1 (F) ^{14}C lipophilic [a]	90 92 89	56-108 80-109 80-100	15% 11% 7.6%	15 8 9	< 0.01 (10)	no data	PP9/0182; RJ0215B
cow muscle	0.02	0.05-0.1 (Fb) 0.05-0.1 (F)	95 100	75-108 88-111	19% 12%	3 7	< 0.02-0.02 (3)	no data	PP9/0182; RJ0215B
cow liver	0.02	0.05-0.1 (Fb) 0.05-0.1 (F)	88 96	- -	- ns	2 4	< 0.02 (2)	no data	PP9/0182; RJ0215B
cow kidney	0.02	0.05-0.1 (Fb) 0.05-0.1 (F)	109 121	- -	- ns	2 4	< 0.02-0.02 (2)	no data	PP9/0182; RJ0215B
cow fat	0.02	0.05 (Fb) 0.05 (F)	118 94	110-126 92-96	- ns	2 5	< 0.02 (3)	no data	PP9/0182; RJ0215B

[a] Milk sample from cow metabolism study [Evans *et al*, 1981, PP9-0180], sample reference PP009AA01 containing ^{14}C lipophilic fluazifop conjugates

[b] individual fortification levels are not stated in the study report

GC-MS Method RAM 331/01

GC-MS method RAM 331/01 is proposed for use as enforcement metod. GC-MS method RAM 331/01 determines total fluazifop (fluazifop-butyl, free fluazifop acid (II) and its conjugates) in animal commodities as one single analyte (common moiety) and residues are expressed as fluazifop acid (II). The reported LOQ is 0.01 mg/kg.

Method RAM 331/01 (6 April 2000) is described by [Robinson *et al*., 2000, PP5/0611, SOP RAM 331/01]. Samples (10 g) are extracted by maceration with dichloromethane:methanol (50:50, v/v). After centrifugation, aliquots are concentrated by evaporation and hydrolysed with 0.2 M NaOH in methanol (1 hour at 60 °C). The hydrolysate is concentrated by evaporation, the pH is adjusted to < 1 and the hydrolysate is partitioned with dichloromethane. The dichloromethane layer is partitioned with 0.5 M sodium bicarbonate. The aqueous layer is isolated and the pH is adjusted to < 1 followed by a C_2 solid phase extraction (SPE) procedure using dichloromethane/acetone (95:5, v/v) as eluent. Eluates are evaporated to dryness and are then derivatised to the methyl ester with 3 M HCl in methanol (30 min 60 °C). The mixture is diluted with water and then partitioned with hexane. The hexane is dried on anhydrous sodium sulphate and then cleaned-up on a silica SPE cartridge using hexane/ethyl acetate (80:20, v/v) as eluent. Fluazifop-methyl is determined in the eluate by GC-MS (m/z=341, 282, 284) using matrix matched fluazifop-methyl as external standard (fluazifop acid (II) derivatised in situ). Confirmation can be achieved by using a different GC column (BPX 50).

A validation study is available [Crook, 2000, PP5/0613, report TMJ4388B]. Validation results are shown in Table 138. No significant enhancement or suppression of detector response (<10%) was observed in the presence of milk, fat, eggs and muscle, indicating that no matrix matched standards are required for these matrices. Significant matrix effects were found for liver (+23%) and kidney

(+17%), indicating that these matrices need to be quantitated using matrix matched standards. Validation of the confirmation method was not conducted.

An independent laboratory validation is available [Croucher, 2000, PP5/0612, report 38/263-D2140]. Validation results are shown in Table 138. Validation of matrices using matrix matched standards was unsuccessful, because mean recoveries were very often below 70%. Satisfactory results were obtained with standards in solvent. Since the preparation of matrix matched standards is a critical step in the procedure, this needs further investigation to avoid unexpected matrix effects.

The method was used in a storage stability study on animal products [1983/045-D2149]. The method was slightly modified: quantification was at m/z=254 as this ion was less prone to interferences than ions at m/z 341. Some samples were analysed on a different GC column (ZB5).

A radiovalidation study is available on efficiency of extraction and hydrolysis [Ryan and Kenny, 1999, PP5/0610, report RJ2873B]. Samples of hen eggs, cow milk and cow liver from a hen and cow metabolism study [Robertson and Hand, 1999, PP5/0595, report RJ2839B; Hand and Robertson, 1999, PP5/0593, report RJ2799B] were extracted using dichloromethane:methanol (50:50, v/v) as used in SOP RAM 331/01. The extract is hydrolysed in a solution of 0.2 M NaOH in methanol (1 hour, 60 ºC) as used in SOP RAM 331/01. Level of radioactivity in extract solutions were measured by LSC. Qualitative analysis of the hydrolysates was performed by TLC. Results are shown in Table 137.

Reviewer's remark: Total fluazifop residues in milk in the goat metabolism study may be underestimated because of the harsh hydrolysis conditions used. Hydrolysis in 0.2 M NaOH in methanol (1 hour, 60 ºC) as used in SOP RAM 331/01 released more residues from milk, confirming this hypothesis. Similar residues were released from eggs and liver. The extractability and hydrolytic efficiency is considered acceptable.

Reviewer's conclusion: GC-MS Method RAM 331/01 is considered:

- valid (full validation) for the determination of fluazifop acid (II) in cow milk (0.01–0.1 mg/kg), hen eggs (0.01–0.1 mg/kg), bovine muscle (0.01–0.1 mg/kg), bovine liver (0.01–0.1 mg/kg), bovine kidney (0.01–0.1 mg/kg), bovine fat (0.01–0.1 mg/kg)
- not valid for the determination of fluazifop-butyl (no validation results); since fluazifop-butyl is not found in the metabolism studies with goat, cow and hens, this is considered not to have any effect on the total fluazifop residues
- valid for the determination of fluazifop (II) conjugates (radiovalidation for milk, eggs and liver)
- suitable for use as enforcement method for the determination of total fluazifop in milk, eggs, muscle, liver, kidney and fat, since an independent laboratory validation has been submitted. The confirmation method still needs to be validated.

The valid LOQ is 0.01 mg/kg.

Table 137 Comparative extractability and hydrolysis data from radioactive samples from goat and hen metabolism studies

	Comparative extractability data			Comparative hydrolysis data		
Matrix	% extracted, in report RJ2873B	% extracted, metabolism study	comparison %	% total fluazifop, in report RJ2873B	% total fluazifop, metabolism study	comparison %
^{14}C pyridyl 96 hours milk	94%	92% [a]	102%	86%	67% [a]	127%
^{14}C phenyl goat liver	58%	66% [a]	87%	23%	25% [a]	92%
whole egg (not listed in metabolism study)	83%	89% [b]	93%	67%	72% [b]	93%

[a] Goat metabolism study [Hand and Robertson, 1999, PP5/0593, report RJ2799B], sample ID not specified. Skimmed milk and milk fat were extracted separately with (acetonitrile and) dichloromethane and then saponified with 2.5 M NaOH in

methanol (unknown conditions). In the goat metabolism study, fluazifop acid (II) in milk may be underestimated because of the hydrolysis conditions used. Liver was extracted with acetonitrile, acetonitrile/water and acetone, 1 M HCl and 1 M NaOH. Liver extracts were not hydrolysed.

[b] Hen metabolism study [Robertson and Hand, 1999, PP5/0595, report RJ2839B], sample ID not specified. Egg yolks and egg whites extracted separately with (dichloromethane), acetonitrile, acetonitrile/water and acetone. Only the egg yolk extracts were saponified with methanolic base (unknown conditions). In the hen metabolism study, fluazifop acid (II) in egg yolks may be underestimated because of the hydrolysis conditions used.

Table 138 Validation results for method RAM 331/01

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report
cow milk	0.01	0.01 (F) 0.1 (F)	88 83	82-94 68-99	6% 12%	5 5	<0.3LOQ (2)	in solvent 0.005-0.1 mg/L linear r2>0.99	PP5/0613; TMJ4388B validation
cow milk	0.01	0.01 (F) 0.1 (F)	73 82	66-82 73-95	9.0% 12%	5 5	<0.3LOQ (2)	matrix matched 0.004-0.2 mg/L R2>0.999	PP5/0612; 38/263-D2140; (ILV)
cow milk	0.01	0.01 (F) 0.1 (F)	101 110	89-111 90-127	8.9% 14%	5 5	<0.3LOQ (2)	in solvent	PP5/0612; 38/263-D2140; (ILV)
cow milk	0.01	0.1 (F)	99	71-131	21%	7 [b]	<0.3LOQ (4)	in solvent 0.005-0.125 mg/L; r>0.999	PP5/1243; 1983/045-D2149 stor stab
hen egg	0.01	0.01 (F) 0.1 (F)	98 84	86-110 79-89	10% 5%	5 5	<0.3LOQ (2)	idem	PP5/0613; TMJ4388B validation
hen egg	0.01	0.01 (F) 0.1 (F)	62 79	54-68 72-88	8.6% 8.6%	5 4 [a]	<0.3LOQ (2)	matrix matched 0.004-0.2 mg/L R2>0.999	PP5/0612; 38/263-D2140; (ILV)
hen egg	0.01	0.01 (F) 0.1 (F)	100 116	89-115 99-127	12% 11%	5 5	<0.3LOQ (2)	in solvent	PP5/0612; 38/263-D2140; (ILV)
hen egg	0.01	0.1 (F)	89	72-104	13%	8	<0.3LOQ (4)	in solvent 0.005-0.125 mg/L; r>0.999	PP5/1243; 1983/045-D2149 stor stab
bovine muscle	0.01	0.01 (F) 0.1 (F)	108 112 102	103- 94-109	5% 7%	5 5	<0.3LOQ (2)	idem	PP5/0613; TMJ4388B validation
bovine muscle	0.01	0.1 (F)	81	68-94	11%	8	<0.3LOQ (4)	in solvent 0.005-0.125 mg/L; r>0.999	PP5/1243; 1983/045-D2149 stor stab
bovine fat	0.01	0.01 (F) 0.1 (F)	109 88	97-113 83-91	6% 4%	5 5	<0.3LOQ (2)	idem	PP5/0613; TMJ4388B validation
bovine fat	0.01	0.01 (F) 0.1 (F)	66 63	52-80 51-71	16% 12%	5 5	<0.3LOQ (2)	matrix matched 0.004-0.2 mg/L R2>0.97	PP5/0612; 38/263-D2140; (ILV)
bovine fat	0.01	0.01 (F) 0.1 (F)	97 92	83-112 84-102	12% 9%	5 5	<0.3LOQ (2)	in solvent	PP5/0612; 38/263-D2140; (ILV)
bovine fat	0.01	0.1 (F)	71	55-82	15%	8	<0.3LOQ (3); 0.0035 (1)	in solvent 0.005-0.125 mg/L; r>0.999	PP5/1243; 1983/045-D2149 stor stab
bovine liver	0.01	0.01 (F) 0.1 (F)	108 110 107	105- 99-111	2% 5%	5 5	<0.3LOQ (2)	idem	PP5/0613; TMJ4388B validation
bovine liver	0.01	0.01 (F) 0.1 (F)	63 69	58-76 64-79	12% 9.4%	5 5	<0.3LOQ (2)	matrix matched 0.004-0.08 mg/L R2>0.999	PP5/0612; 38/263-D2140; (ILV)

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code; Report
bovine liver	0.01	0.01 (F) 0.1 (F)	86 101	84-90 92-123	2.9% 13%	5 5	<0.3LOQ (2)	in solvent	PP5/0612; 38/263-D2140; (ILV)
bovine liver	0.01	0.1 (F)	91	72-112	16%	8	<0.3LOQ (4)	in solvent 0.005-0.125 mg/L; r>0.999	PP5/1243; 1983/045-D2149 stor stab
bovine kidney	0.01	0.01 (F) 0.1 (F)	95 96	91-98 91-98	3% 3%	5 5	<0.3LOQ (2)	idem	PP5/0613; TMJ4388B validation
bovine kidney	0.01	0.1 (F)	91	51-121	24%	8	<0.3LOQ (3); 0.0033 (1)	in solvent 0.005-0.125 mg/L; r>0.999	PP5/1243; 1983/045-D2149 stor stab

[a] one outlier with a recovery of 0% was excluded from evaluation

[b] one outlier with a recovery of 250% was excluded from evaluation

Analytical methods used in study reports in soil samples

Several analytical methods were submitted for use in field rotational crop studies and storage stability studies on soil.

Extractabilility of fluazifop-butyl related residues from soil

Extractability of fluazifop-butyl from soil was investigated [Atreya and Houlden, 1980, PP9/0355, report PPRAM 54] on a sample from a treated soil using acetonitrile/water (90:10, v/v), acetone/hexane (20:80, v/v), methanol/dichloromethane (20:80, v/v) or boiling acetonitrile/water (90:10, v/v) under reflux for 1 hour as extraction solvents. None of the solvent systems gave positive residues of fluazifop-butyl in soil, because fluazifop-butyl degrades rapidly in soil and therefore no residues of fluazifop-butyl are likely to be present in soil. The acetonitrile/water (90:10, v/v) was chosen as extraction solvent for fluazifop-butyl in method PPRAM 54 because it was shown to remove less co-extractive material than the other solvents.

Extractability of fluazifop acid (II) from soil was investigated [Bolygo *et al*, 1991, PP5/0776, report ARAM 197] on a sample from a treated soil using acetone/concentrated HCl/water (98:2:20, v/v) for 30 min or acetonitrile/water (50:50, v/v) for 30 min. The average recovery of a fortified soil sample at 0.5 mg/kg was 85% (n=4, RSD 4.6%) for the acid extraction mixture and 94% (n=4, RSD 2.2%) for the non-acid extraction mixture. In a sample from a treated soil (taken from study 88JH384, trial KRS 8830-G1, clay loam soil) the average fluazifop acid (II) concentration was measured as 0.19 mg/kg (n=5, RSD 2.8%) using the acid extraction mixture and 0.20 mg/kg (n=5, RSD 4.4%) for the non-acid extraction mixture. Both extraction methods gave similar residue results.

Extractability of CF3-pyridone (X) from soil was investigated [Wiebe, 1990, PP5/0778, report RR 90-076B] on a sample from a treated soil using acetonitrile/water (50:50, v/v) for 30 min or 2-propanol/water (80:20, v/v) for 18 hours reflux. In sample from a treated soil (E211-46AM, 0-6 inch soil horizon, 1× 0.84 kg ai/ha, DAT 4 weeks) the average fluazifop acid (II) concentration was measured as 0.021–0.022 mg/kg using the acetonitrile/water mixture and 0.011 mg/kg using reflux conditions. The acetonitrile/water extraction resulted in higher levels of CF3-pyridone (X) than when reflux was used. This could indicate degradation of CF3-pyridone (X) under reflux conditions, but recovery of CF3-pyridone (X) was not verified.

Extractability of Pyr-Ph ether (IV) from soil was investigated [Huang, 2012, R150397_50000, report TK0172993]. Various attempts to improve the extraction efficiency for Pyr-Ph ether (IV) were unsuccessful. When soil samples were extracted by shaking the soil with 50/50 acetonitrile/ammonium acetate buffer (10 mM; pH 5.5) (v/v) for 20 minutes, acceptable recovery and minimum instrumental interference for HPLC-MS/MS were found for fluazifop-butyl, fluazifop acid

(II) and CF3-pyridone (X), but not for Pyr-Ph ether (IV). Further work showed that the recovery of Pyr-Ph ether (IV) was inconsistent and depended on the time between fortification of the sample and extraction. To investigate this, soil samples fortified with Pyr-Ph ether (IV) were left for 10, 20, 30, 60, 120 and 180 minutes prior to extraction. The recovery of Pyr-Ph ether (IV) was determined and the results are presented in Table 139.

In addition, different ratios of acetonitrile to buffer were tested. Furthermore, various other extraction conditions were evaluated (see Table 139).

From the recovery experiments conducted it appears that Pyr-Ph ether (IV) either binds strongly to soil or is degraded. From the work using the harsher extraction conditions, binding is not likely the cause of the low recoveries of Pyr-Ph ether (IV). The compound appears to be degraded on extended contact with soil. While adequate recoveries can be obtained from samples of soil fortified and extracted within 20 minutes, residues of Pyr-Ph ether in actual samples from treated soils would unlikely to be found.

Table 139 Recovery of Pyr-Ph ether (IV) after various extraction conditions

Extraction solvent	Time prior to extraction (min)	% recovery,range		n
50/50 acetonitrile/ ammonium acetate buffer (10 mM; pH 5.5)	10 20 30 60 120 180	89 83 60 69 53 41	85-99 65-89 46-79 65-75 53 39-43	5 32[a] 9[d] 6[b] 1 2
30/70 acetonitrile/ ammonium acetate buffer (10 mM; pH 5.5)	20	70	59-81[c]	4
40/60 acetonitrile/ ammonium acetate buffer (10 mM; pH 5.5)	20	92	86-103[c]	4
45/55 acetonitrile/ ammonium acetate buffer (10 mM; pH 5.5)	20	80	77-84	4
60/40 acetonitrile/ ammonium acetate buffer (10 mM; pH 5.5)	20	86	81-90[d]	4
70/30 acetonitrile/ ammonium acetate buffer (10 mM; pH 5.5)	20	75	71-80[d]	4
50/50 acetonitrile/ pH 3.5 buffer	10 12 hour 20 hour 40 hour	88 26 15 6		
50/50 acetonitrile/ pH 7.0 buffer	10 12 hour 20 hour 40 hour	88 26 18 7		
50/50 acetonitrile/ 0.5% NH4OH	10 12 hour	82 27		
50/50 acetonitrile/ 2% NH4OH	1 hour	58		
50/50 methanol/ pH 5.5 buffer	20 3 hour	80 49		
50/50 methanol/ pH 10 buffer	20 70 hour	51 4		
70/30 acetonitrile/ 0.3% formic acid	20 aged (>1 week)	74 2		
30/70 acetone/ pH 3.5 buffer	20 hour	14		
methanol	20 hour	16		
methanol with 2% formic acid	20 hour	16		
50/50 acetonitrile/ 0.01 M NaOH	12 hour	18		
50/50 acetonitrile/ 0.1 M NaOH	12 hour	8		
80/20 acetone/ 0.1 M HCl	2 hour	47		
80/20 acetone/ 0.2 M HCl	2 hour	38		
80/20 acetone/ 0.5 M HCl	2 hour	42		
80/20 acetone/ 2% HCl	2 hour	41		
100/20 methanol/ 0.1 M HCl, reflux 1 hour	2 hour	0		

Extraction solvent	Time prior to extraction (min)	% recovery,range	n
100/20 methanol/ 1.0 M HCl, reflux 1 hour	2 hour	0	
50/50 acetonitrile/ 0.1 M HCl, reflux 1 hour	20 hour	0	
50/50 acetonitrile/ 1.0 M HCl, reflux 1 hour	20 hour	5	
50/50 acetonitrile/ 2% formic acid, reflux 1 hour	20 hour	15	
One cycle with 50/50 acetonitrile/ buffer, 80 °C, glass filter	2 hour	25	
One cycle with 50/50 acetonitrile/ buffer, 80 °C, cellulose filter	2 hour	33	
Two cycles with acetonitrile, 80 °C, cellulose filter	2 hour	42	
Two cycles with 50/50 acetonitrile/ buffer, 80°C, cellulose filter	2 hour	45	
Two cycles with 20/80 acetonitrile/ buffer, 80°C, cellulose filter	2 hour	39	
Two cycles with buffer, 80 °C, cellulose filter	2 hour	36	

[a] Four types of soils were evaluated.

[b] Two types of soils were used; sandy soils generally obtain higher recoveries than clay loam soils.

[c] Significant matrix interference (signal suppression; severity increases as buffer content increases) observed and a low recovery for other targeted analytes.

[d] Significant matrix interference (signal enhancement as acetonitrile content increases) observed for fluazifop-butyl.

HPLC-UV, GC-NPD and GC-MS method PPRAM 54 and its modifications

Method PPRAM 54 and its modifications describe the determination of fluazifop-butyl in soil. The reported LOQ was 0.01 mg/kg for HPLC-UV, GC-NPD and GC-MS.

Method PPRAM 54 (December 1980) is described by [Atreya and Houlden, 1980, PP9/0355, report PPRAM 54; Jones, 1991, no code, report PPRAM 54 addendum]. Soil samples (20–50 g) are extracted with acetonitrile/water (90/10, v/v) for 30 min. After filtration an aliquot of the extract, equivalent to 10 g soil, is diluted with water and fluazifop-butyl is selectively partitioned into hexane. The hexane extract is dried by filtration thourough anhydrous sodium sulphate and cleaned-up on a Florisil adsorption column to remove interfering co-extractives. Fluazifop-butyl is eluated with 30% ether in hexane. The eluate is evaporated to dryness and redissolved in acetonitrile/water (65:35, v/v) for quantitative determination by HPLC-UV (270 nm). Calibration is by external standards in solvent.

GC-MS may be used for the confirmation of residues of fluazifop-butyl. The final solution of acetonitrile/water (65:35, vv) is diluted with water and then partitioned into hexane for quantitative determination by GC-MS. Calibration is by external standards in solvent. A validation report is not available.

HPLC-UV method PPRAM 54 was used in an aerobic soil degradation study [RJ0183B]. Validation results are shown in Table 140.

Method RAM 054/01 (June 1991) is described by [Jones, 1991, no code, report PPRAM54 addendum]. The method is identical to PPRAM 54, except that an alternative detection technique is introduced. The final solution of acetonitrile/water (65:35, vv) is diluted with water and then partitioned into hexane for quantitative determination by GC-NPD. Calibration is by external standards in solvent. The method was used in a field dissipation study [RJ0952B]. Validation results are shown in Table 140.

Method RAM 054/02 (4 August 1995) is described by [Atreya and Jones, 1995, PP5/0779, SOP RAM 054/02]. The Florisil eluate is evaporated to dryness and then redissolved in acetonitrile/water (65/35, vv) for quantitiave determination by HPLC-UV (270 nm) or redissolved in acetone for quantititative determination by GC-NPD or confirmation by GC-MS at m/z 282 and 383. A validation report is not available.

Modification A of Method RAM 054/02 was used in a field rotational crop study [RJ2202B]. The florisil clean-up column was omitted and a quantitative determination of fluazifop-butyl was carried out by GC-MS instead of GC-NPD. Validation results are shown in Table 140.

Method RAM 054/03 (30 October 2000) is described by [Atreya *et al*., 2000, PP5/1178, SOP RAM 054/03]. Sample amounts and extraction volumes are changed, the florisil clean-up column is omitted and GC-MS is used as primary detection. Soil samples (10 g) are extracted with acetonitrile/water (90/10, v/v) for 30 min. After centrifugation an aliquot of the extract, equivalent to 1 g soil is diluted with water and fluazifop-butyl is selectively partitioned into hexane. The hexane extract is dried by filtration thourough anhydrous sodium sulphate, evaporated to dryness and redissolved in acetone for quantitative determination by GC-MS at m/z 282 (target), 383 and 254 (qualifiers). Calibration is by external standards in solvent. An enhancement for fluazifop-butyl of 7% is observed in the presence of soil matrix. A matrix matched standard may be used to compensate for this effect. Validation results are shown in Table 140.

Reviewer's conclusion: Method PPRAM 54 and RAM054/01 and its modifications are considered:

- valid (full validation) for the determination of fluazifop-butyl in soil (0.01–0.1 mg/kg) using GC-NPD or GC-MS as detection;
- not valid for the determination of fluazifop-butyl in soil using HPLC-UV as detection (no validation results).

Table 140 Validation of fluazifop-butyl using method PPRAM54 or its modifications

Soil type	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection calibration	Code no; Report no
PPRAM 54, HPLC-UV detection									
Speyer 2/2 soil	0.01	0.1 (Fb) 1.0 (Fb)	92 68	- 66-69	- -	1 2	ns	HPLC-UV Calibration ns	PP9/0271; RJ0183B
Speyer 2/3 soil	0.01	0.1 (Fb) 1.0 (Fb)	95 79	- 75-83	- -	1 2	ns	HPLC-UV Calibration ns	PP9/0271; RJ0183B
RAM054/01, GC-NPD detection									
soil ns	0.01	0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	97 88 85	69-127 60-124 66-114	19% 24% 17%	7 7 7	-	GC-NPD calibration ns	no code; PPRAM 54 addendum (validation)
soil ns	0.01	0.01 (Fb) 0.05 (Fb) 0.1 (Fb)	133 112 94	112-152 100-119 70-129	12% 5.4% 22%	7 7 7	< 0.01 (8)	GC-NPD; calibration ns	PP9/0814; RJ0952B (dissipation)
RAM054/02, modification A, omission of Florisil Clean-up column, GC-MS detection									
sandy loam	0.01	0.01 (Fb) 0.02 (Fb) 0.05 (Fb) 0.1 (Fb) 0.2 (Fb)	92 79 95 95 88	75-109 59-101 86-109 77-115 -	- 23% 11% 20% -	2 4 4 3 1	< 0.01 (10)	GC-MS calibration ns	PP5/0590; RJ2202B (rotational)
RAM 054/03									
sandy loam	0.01	0.01 (Fb) 0.1 (Fb)	91 89	88-95 87-94	3% 3%	5 5	<0.3 LOQ (2)	GC-MS 0-1 mg/L; in matrix r2>0.999; linear	PP5/1178; RAM054/03 (validation)
sandy clay loam	0.01	0.01 (Fb) 0.1 (Fb)	93 93	90-98 92-95	3% 1%	5 5	<0.3 LOQ (2)	GC-MS 0-1 mg/L in matrix r2>0.999; linear	PP5/1178; RAM054/03 (validation)

HPLC-UV method PPRAM 55

Method PPRAM 55 describes the determination of fluazifop acid (II) in soil. The reported LOQ was 0.01 mg/kg for HPLC-UV detection.

Method PPRAM 55 (December 1980) is summarized by [Atreya and Houlden, 1981, PP9/0271, report RJ0183B]. Soil samples (50 g) are extracted with aqueous acetone/HCl solution (time and concentrations not reported). The extract is diluted with water and fluazifop acid (II) is selectively partitioned into diethyl ether. The diethyl ether extract is cleaned-up on a Fractosil adsorption column to remove interfering co-extractives. Fluazifop acid (II) is quantified by HPLC-UV (270 nm). Calibration is by external standards in solvent.

HPLC-UV method PPRAM 55 was used in an aerobic soil degradation study [RJ0183B]. Validation results are shown in Table 141.

Reviewer's conclusion: Method PPRAM 55 is considered:

- valid for the determination of fluazifop acid (II) in soil (at 0.5 mg/kg).

The valid LOQ is 0.1 mg/kg (no validations below this point).

Table 141 Validation of fluazifop-butyl using method PPRAM54 or its modifications

Soil type	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	detection calibration	Code no; Report no
PPRAM 54, HPLC-UV detection									
Speyer 2/2 soil	0.01	0.1 (F) 0.2 (F) 0.5 (F) 1.0 (F)	82 77 83 76	68-95 - 73-92 76-77	- - 10% -	2 1 4 2	ns	HPLC-UV Calibration ns	PP9/0271; RJ0183B
Speyer 2/3 soil	0.01	0.1 (F) 0.2 (F) 0.5 (F) 1.0 (F)	100 94 84 84	99-102 - 71-106 79-89	- - 20% -	2 1 4 2	ns	HPLC-UV Calibration ns	PP9/0271; RJ0183B

GC-MS method RR 89-072B

GC-MS method RR 89-072B determines fluazifop-butyl and fluazifop acid (II) separately in soil samples. The reported LOQ is 0.01 mg/kg soil (as received basis) for each analyte.

Method RR 89-072B (30 November 1989) is described by [Wiebe, 1989, PP5/0777, report RR 89-072B]. Fluazifop-butyl is extracted from the soil (40 g) with acetonitrile/water (90:10, v/v) for 30 min. The extract is evaporated (below 40 ºC) to remove the acetonitrile and to concentrate the extract. The extract is diluted with 0.05 M HCl and fluazifop-butyl is then partitioned into dichloromethane. The dichloromethane phase is evaporated to dryness (below 30 ºC) and redissolved in toluene. Fluazifop-butyl is quantified by GC-MS at m/z 383. Confirmation can be achieved by using a different m/z ion (254, 255, 282) or GC-NPD. Validation results are shown in Table 142.

Fluazifop acid (II) is extracted from the soil (40 g) with acetone/water/concentrated HCl (98:20:2, v/v/v) for 30 min. The extract is evaporated (below 40 ºC) to remove the acetone and to concentrate the extract. The extract is diluted 0.05 M HCl and fluazifop acid (II) is then partitioned into dichloromethane. The dichloromethane phase is dried over an anhydrous sodium sulphate column and then evaporated (below 30 ºC) to just dryness. The residuum is dissolved in methanol and treated with a diazomethane solution for 30 min to form the methyl ester derivative of fluazifop acid (II). The diazomethane solution is evaporated to just dryness and redissolved in toluene. Fluazifop-methyl is quantified by GC-MS at m/z 341. Confirmation can be achieved by using a different m/z ion (254, 255, 281) or GC-NPD.

A validation report is available [Wiebe, 1989, PP5/0777, report RR 89-072B]. GC-MS method RR 89-072B was used in a soil storage stability study [RR 95-002B] and field dissipation studies [90-337B, 90-338B]. Validation results are shown in Table 142.

Modification A of GC-MS method 89-072B was used to quantify fluazifop acid (II) in a storage stability study in soil [RR 95-002B]. The dichloromethane residuum was treated with a methanol/HCl solution to form the methyl ester derivative of fluazifop. After derivatisation, the fluazifop-methyl is partitioned into toluene and quantified by GC-MS. Validation results are shown in Table 142.

Reviewer's conclusion: Method RR 89-072B and its modifications are considered:

- valid (full validation) for the determination of fluazifop-butyl in soil (0.01–0.4 mg/kg);
- valid (full validation) for the determination of free fluazifop acid (II) in soil (0.01–0.4 mg/kg).

Table 142 Validation of fluazifop-butyl and fluazifop acid (II) using method RR 89-072B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no; soil type
Method RR 89-072B									
loam	0.01	0.01 (Fb) 0.1 (Fb) 1.0 (Fb)	111 104 96	91-128 93-113 95-97	10% 9.2% -	8 4 2	no data	0.1-10 mg/L; in solvent linear by graph	PP5/0777; RR 89-072B (validation); PP5/0813; RR 90-337B (dissipation) PP5/1110 RR 90-338B (dissipation)
sandy loam	0.01	0.01 (Fb) 0.1 (Fb)	95 84	74-125 73-101	18% 14%	6 4	no data	0.1-10 mg/L; in solvent linear by graph	PP5/0777; RR 89-072B (validation)
loam	0.01	0.04 (Fb) 0.4 (Fb) 4.0 (Fb)	91 93 117	79-116 76-107 112-122	15% 12% -	7 7 2	< 0.01 (7) – 0.018 (1)	no data	PP5/0798; RR 95-002B; (stor stab) PP5/0813; RR 90-337B (dissipation)
loam	0.01	0.01 F) 0.1 (F) 1.0 (F)	99 100 98	77-118 78-121 -	14% 14% -	6 7 -	no data	0.1-10 mg/L; in solvent linear by graph	PP5/0777; RR 89-072B (validation); PP5/0813; RR 90-337B (dissipation) PP5/1110 RR 90-338B (dissipation)
sandy loam	0.01	0.01 (F) 0.1 (F)	112 90	101-129 73-104	8.5% 16%	8 4	no data	0.1-10 mg/L; in solvent linear by graph	PP5/0777; RR 89-072B (validation)
loam	0.01	0.04 (F) 0.4 (F)	89 96	66-104 75-113	13% 14%	10 10	< 0.01 (10)	no data	PP5/0798; RR 95-002B; (stor stab) PP5/0813; RR 90-337B (dissipation)
Method RR 89-072B, modification A									
loam	0.01	0.04 (F) 0.4 (F)	66 68	- -	- -	1 1	< 0.01 (1)	no data	PP5/0798; RR 95-002B (stor stab)

GC-MS method RR 90-076B

GC-MS method RR 90-076B determines free CF3-pyridone (X) in soil samples. The reported LOQ is 0.01 mg/kg soil (as received basis).

Method RR 90-076B (30 April 1990) is described by [Wiebe, 1990, PP5/0778, report RR 90-076B]. CF3-pyridone (X) is extracted from the soil (40 g) with acetonitrile/water (50:50, v/v) for 30 min. An aliquot equivalent to 5 g soil is taken and evaporated (below 40 ºC) to remove the acetonitrile and to concentrate the extract. The extract is acidified with concentrated HCl, NaCl is added and CF3-pyridone (X) is then partitioned into ethylacetate. The ethyl acetate phase is dried over anhydrous sodium sulphate and the ethyl acetate phase is then evaporated to dryness (below 30 ºC). The residuum is redissolved in acetonitrile and then treated with N-methyl-N-t-butyldimethylsilyltrifluoroacetamide with 1%-t-butyldimethylchlorosilane to form the t-butyldimethylsilyl derivatives. The derivatives are quantified by GC-MS at m/z 220 using external standards in solvent or matrix. Confirmation can be achieved by using a different m/z ion (190, 221, 258) or GC-NPD.

A validation report is available [Wiebe, 1990, PP5/0778, report RR 90-076B]. GC-MS method RR 90-076B was used in a soil storage stability study [RR 95-002B] and field dissipation studies [RR 90-337B, RR 90-338B]. Validation results are shown in Table 143.

Reviewer's conclusion: Method RR 90-076B is considered:

- valid (full validation) for the determination of free CF3-pyridone (X) in soil (0.01–0.4 mg/kg)

Table 143 Validation of fluazifop-butyl and fluazifop acid (II) using method RR 90-076B

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no; soil type
loam	0.01	0.01 (X) 0.1 (X) 1 (X)	94 94 93	87-101 90-102 -	6.0% 4.4% -	6 6 1	no data	0.05-5.0 mg/L; in solvent; linear by graph	PP5/0778; RR 90-076B (validation); PP5/0813; RR 90-337B (dissipation) PP5/1110 RR 90-338B (dissipation)
sandy loam	0.01	0.01 (X) 0.1 (X)	81 87	67-99 71-109	14% 18%	6 5	no data	0.05-5.0 mg/L; in solvent; linear by graph	PP5/0778; RR 90-076B; (validation)
loam	0.01	0.04 (X) 0.4 (X)	96 98	82-115 87-109	13% 8.75	6 6	< 0.01 (6)	GC-MS calibration ns	PP5/0798; RR 95-002B; (stor stab) PP5/0813; RR 90-337B (dissipation)

HPLC-UV and GC-MS method ARAM 195 (1991) and its modifications

HPLC-UV and GC-MS method ARAM 195 and its modifications describe the separate determination of fluazifop acid (II, free) and CF3-pyridone (X, free) in soil samples and residues are expressed as the respective analyte. The reported LOQ is 0.01 mg/kg for each analyte.

HPLC-UV and GC-MS Method ARAM 195 (June 1991) is described by [Bolygo *et al*, 1991, PP5/0776, report ARAM 195]. Soil samples (40 g) are extracted with acetonitrile/water (50/50, v/v) for 30 min. The acetonitrile is removed by rotary evaporation. The aqueous solution is acidified with HCl (pH < 1) and NaCl is added (1 g/10 mL extract). Fluazifop acid (II, free) and CF3-pyridone (X, free) are both partitioned into ethyl acetate (3 times). The combined ethyl acetate extracts are evaporated to dryness and redissolved in acetone and divided in two aliquots: one for fluazifop acid (II) and one for CF3-pyridone (X). The aliquot for fluazifop acid (II) work-up, is evaporated to

dryness, redissolved in dichloromethane/acetic acid/methanol (100+0.5+1.5, v/v), cleaned-up on a silica gel column and eluted with dichloromethane/hexane/acetic acid/methanol (40+60+0.5+1.5, v/v) for quantitative determination by HPLC-UV (224 nm). The aliquot for CF3-pyridone (X) work-up is evaporated to dryness, redissolved in acetonitrile and derivatised with N-tert-butyldimethylsilyl-N-methyl-trifluoroacetamide containing 1% tert-butyldimethylchlorosilane (30 min, 90 ºC) to form the tert-butyldimethylsilyl ether derivative of CF3-pyridone (X) for quantitative determination by GC-MS (m/z = 220). Both analytes are quantified by external standards (fluazifop acid (II) and in-situ derivatised CF3-pyridone (X)) in solvent.

Method RAM 195/01 (January 1993) [Atreya, 1993, PP5/0776, report RAM 195/01] is identical to method ARAM 195.

A method validation report for method ARAM 195 and RAM 195/01 is available [Bolygo *et al*, 1991, PP5/0776, report ARAM 195]. Additional validation results are available from a field rotational crop study [RJ2202B] and a field dissipation study [RJ0952B]. Results are shown in Table 144 and Table 145.

Reviewer's conclusion: Method ARAM195 and RAM 195/01 are considered:

- valid (full validation) for the determination of free fluazifop acid (II) in soil (0.01–0.5 mg/kg) using HPLC-UV as detection;
- valid (full validation) for the determination of free CF3-pypridone in soil (0.01–0.5 mg/kg) using GC-MS as detection;

Table 144 Validation of the determination of fluazifop acid (II) in soil using method ARAM 195 (=RAM 195/01)

Soil type	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
soil ns	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.5 (F)	80 85 106 85 108 91 118	70-90 71- 74- 74-	9.5% 12% 14% 18%	6 11 7 9	-	HPLC-UV in solvent 0.025-10 mg/L linear, R2>0.9999	PP5/0776; ARAM 195; validation
sandy loam	0.01	0.01 (F) 0.02 (F) 0.05 (F)	92 126 86 104 84 126	69- 74- 44-	25% 19% 28%	6 3 8	< 0.01 (10)	HPLC-UV calibration ns	PP5/0590; RJ2202B (rotational)
soil ns	0.01	0.01 (F) 0.05 (F) 0.1 (F) 0.2 (F)	87 110 98 169 96 155 94 140	70- 50- 74- 74-	19% 32% 27% 22%	7 17 8 12	< 0.01 (8)	HPLC-UV calibration ns	PP9/0814; RJ0952B (dissipation)

Table 145 Validation of the determination of CF3-pyridone (X) in soil using method ARAM 195 (=RAM 195/01)

Soil type	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
Soil ns)	0.01	0.01 (X) 0.05 (X) 0.1 (X) 0.5 (X)	87 86 82 78	71-106 72-105 69-95 62-89	18% 11% 8.2% 13%	14 13 14 7	-	GC-MS in solvent 0.05-0.5 mg/L linear, R2>0.99	PP5/0776; ARAM 195; (validation)
Sandy loam	0.01	0.01 (X) 0.02 (X)	95 88	75-117 74-103	19% 14%	5 4	< 0.01 (10)	GC-MS calibration ns	PP5/0590; RJ2202B

		0.05 (X)	85	72-121	18%	8			(rotational)
Soil ns	0.01	0.01 (X) 0.05 (X) 0.1 (X) 0.5 (X)	87 83 81 76	71-106 72-97 69-95 62-86	15% 9.3% 8.8% 12%	11 12 11 7	< 0.01 (8)	GC-MS calibration ns	PP9/0814; RJ0952B (dissipation)

GC-MS method RAM 354/01 (2000) and its modifications

GC-MS method RAM 354/01 describes the separate determination of fluazifop acid (II, free) and CF3-pyridone (X, free) in soil samples and residues are expressed as the respective analyte. The reported LOQ is 0.01 mg/kg dry soil for each analyte.

Method RAM 354/01 (30 October 2000) is described by [Hargreaves, 2000, PP5/1062, SOP RAM 354/01]. Soil samples (10 g) are extracted with acetonitrile/water (50/50, v/v) for 30 min. After centrifugation, aliquots equivalent to 2.0 g soil are taken. The acetonitrile is removed by rotary evaporation at temperatures below 30 ºC to avoid evaporation of CF3-pyridone (X). Samples are acidified. Fuazifop acid (II, free) and CF3-pyridone (X, free) are partitioned into ethyl acetate (twice). The combined ethyl acetate extracts are dried by filtering thourough sodium sulphate. The filtrate is evaporated just to dryness (to avoid CF3-pyridone (X) losses) and redissolved in acetone. This process is repeated twice to remove all acid and moisture. The acetone solution is divided in two aliquots: one for fluazifop acid (II) and one for CF3-pyridone (X). The fluazifop acid (II) aliquot is derivatised with bis(trimethylsilyl)trifluoroacetamide containing 1% trimethylsilylchloride (30 min, 80 ºC) to form the trimethylsilyl ester derivative of fluazifop acid (II) for quantitative determination by GC-MS (m/z = 282 (target), 399 and 254 (qualifiers). The CF3-pyridone (X) aliquot is derivatised with N-(tert-butyldimethylsilyl)-N-methyl-trifluoroacetamide containing 1% tert-butyldimethylchlorosilane (30 min, 80 ºC) to form the tert-butyldimethylsilyl ether derivative of CF3-pyridone (X) for quantitative determination by GC-MS (m/z = 220). Analytes are quantified separately by external standards (in-situ derivatised) in solvent.

A validation report is available for GC-MS method RAM 354/01 [Hargreaves, 2000, PP5/1062, SOP RAM 354/01]. Results are shown in Table 146. An enhancement of the GC-MS respons for fluazifop acid (II) and CF3-pyridone (X) of 7% and 9%, respectively, is observed in the presence of soil matrix. A matrix matched standard may be use to compensate for these effects.

Method RAM 354/02 (30 March 2001) is described by [Hargreaves, 2001, PP5/1061, SOP RAM 354/02]. Recoveries of fluazifop acid (II) and CF3-pyridone (X) are improved by a) acidification of samples by addition of concentrated HCl prior to partitioning with ethyl acetate and b) by not evaporating samples to complete dryness at any point, with the exception of the final acetone evaporation before derivatisation.

GC-MS method RAM 354/02 was used in an aerobic soil degradation study with CF3-pyridone (X) [RJ3259B]. Validation results are shown in Table 146.

Reviewer's conclusion: GC-MS Method RAM 354/01 and its modifications are considered:

- valid (full validation) for the determination of free fluazifop acid (II) in soil (0.01–0.5 mg/kg);
- valid (full validation) for the determination of free CF3-pyridone (X) in soil (0.01–0.5 mg/kg).

Table 146 Validation of fluazifop acid (II) and CF3-pyridone (X) using method RAM 354/01

Soil type	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no; soil type
RAM 354/01									
sandy loam	0.01	0.01 (F) 0.1 (F)	88 93	85-92 89-96	3% 3%	5 5	<0.3LOQ (2)	0.002-1 mg/L; in matrix linear, r2>0.999	PP5/1062; RAM 354/01 (validation)

Soil type	LOQ	Fortifi cation level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no; soil type
sandy clay loam	0.01	0.01 (F) 0.1 (F)	93 96	87-103 91-105	7% 6%	5 5	<0.3LOQ (2)	0.002-1 mg/L; in matrix linear, r2>0.999	PP5/1062; RAM 354/01 (validation)
sandy loam	0.01	0.01 (X) 0.1 (X)	71 71	70-73 69-73	2% 2%	5 5	<0.3LOQ (2)	0.002-1 mg/L; in matrix linear, r2>0.999	PP5/1062; RAM 354/01 (validation)
sandy clay loam	0.01	0.01 (X) 0.1 (X)	77 81	68-82 80-82	75 1%		<0.3LOQ (2)	0.002-1 mg/L; in matrix linear, r2>0.999	PP5/1062; RAM 354/01 (validation)
RAM 354/02									
soil ns	0.01	0.05 (X) 0.08 (X) 0.10 (X) 0.12 (X) 0.14 (X) 0.18 (X) 0.20 (X)	72 76 73 71 71 74 74	66-78 72-80 68-76 67-76 67-78 70-80 69-81	5% 5% 4% 4% 5% 5% 6%	16 8 8 8 8 8 8	< 0.01 (1)	GC-MS calibration ns	R154719/0002; RJ3259B (aerobic soil)

HPLC-MS/MS method GRM044.03A

HPLC-MS/MS method GRM044.03A describes the separate determination of fluazifop-butyl, fluazifop acid (II, free) and CF3-pyridone (X, free) in soil samples and residues are expressed as the respective analyte. The reported LOQ is 0.001 mg/kg dry soil for each analyte.

Method GRM044.03A (2013) is described by [Huang, 2010, PP5_50103, report GRM044.03A; Hagan and Bertrand, 2013, no code, report 12SYN323]. Soil samples (20 g) are extracted twice with acetonitrile/buffer pH 5.5 (50/50, v/v) for 30 min. The buffer consisted of aqueous acetic acid adjusted to pH 5.5 with ammonium hydroxide. Extracts were combined. An aliquot was filtered thourough an 0.2 or 0.45 um PTFE filter disc, diluted with pH 5.5 buffer and fluazifop-butyl, fluazifop acid (II) and CF3-pyridone (X) were quantified by HPLC-MS/MS using electrospray ionisation. Fluazifop-butyl was determined at m/z 384 to 328 (quantification) or 384 to 282 (confirmation); fluazifop acid (II) at m/z 326 to 254 (quantification) or 326 to 226 (confirmation); CF3-pyridone (X) at m/z 164 to 146 (quantification) or 164 to 75 (confirmation). Residue quantification is carried out using external standard calibrations in solvent.

Method GRM044.03A is validated by [Huang, 2010, PP5_50103, report GRM044.03A] and also an independent laboratory validation is available [Schmitt and Perez, 2013, PP5_50339, TK0114928]. Validation results are shown in Table 147. Acceptable mean recoveries between 70% and 120% with a relative standard deviation lower than 20% were also found for the confirmatory transition. Samples were fortified either with fluazifop-butyl alone or with a mixture containing both fluazifop acid (II) and CF3-pyridone (X).

Method GRM044.03A was used in a soil storage stability study [Pyles and Hagan, 2013, PP5_50411, TK0015285] and a field dissipation study [Wiepke *et al*., 2013, A12460A_50023, report TK0015266]. Validation results are shown in Table 147.

Reviewer's conclusion: GC-MS Method GRM044.03A and its modifications are considered:

- valid (full validation) for the determination of fluazifop-butyl in soil (0.001–0.5 mg/kg)
- valid (full validation) for the determination of free fluazifop acid (II) in soil (0.001–0.5 mg/kg);
- valid (full validation) for the determination of free CF3-pypridone in soil (0.001–0.5 mg/kg).

Table 147 Validation of fluazifop-butyl, fluazifop acid (II) and CF3-pyridone (X) using method GRM044.03A

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
bare soil; sand – loamy sand	0.001	0.001 (Fb) 0.010 (Fb) 0.100 (Fb)	99 97 103	79-130 73-124 96-116	15% 14% 7.4%	44 35 9	<0.3LOQ (48)	0.01-2.0 ng/mL 1/x weighted linear; r>0.99	A12460A_50023; TK0015266 (dissipation)
bare soil; sand – loamy sand	0.001	0.001 (F) 0.010 (F) 0.100 (F) 0.500 (F)	87 96 103 107	60-106 75-114 83-126 102-112	13% 10% 12% 4.3%	46 37 9 3	<0.3LOQ (48)	0.01-2.0 ng/mL 1/x weighted linear; r>0.99	A12460A_50023; TK0015266 (dissipation)
bare soil; sand – loamy sand	0.001	0.001 (X) 0.010 (X) 0.100 (X) 0.500 (X)	91 93 98 83	71-109 70-109 88-105 80-85	12% 11% 5.4% 2.8%	46 37 9 3	<0.3LOQ (48)	0.01-2.0 ng/mL 1/x weighted linear; r>0.99	A12460A_50023; TK0015266 (dissipation)
crop grown soil; sand – loamy sand	0.001	0.001 (Fb) 0.010 (Fb) 0.100 (Fb)	94 92 103	70-118 69-112 90-118	12% 12% 12%	37 32 5	<0.3LOQ (41)	0.01-2.0 ng/mL 1/x weighted linear; r>0.99	A12460A_50023; TK0015266 (dissipation)
crop grown soil; sand – loamy sand	0.001	0.001 (F) 0.010 (F) 0.100 (F)	86 94 95	61-100 72-114 85-101	12% 12% 7.1%	41 36 5	<0.3LOQ (41)	0.01-2.0 ng/mL 1/x weighted linear; r>0.99	A12460A_50023; TK0015266 (dissipation)
crop grown soil; sand – loamy sand	0.001	0.001 (X) 0.010 (X) 0.100 (X)	90 92 97	74-109 81-109 87-102	8.3% 6.6% 6.2%	41 36 5	<0.3LOQ (41)	0.01-2.0 ng/mL 1/x weighted linear; r>0.99	A12460A_50023; TK0015266 (dissipation)
loam (soil 1)	0.001	0.001 (Fb) 0.01 (Fb) 0.05 (Fb) 0.001 (F) 0.01 (F) 0.05 (F) 0.001 (X) 0.01 (X) 0.05 (X)	96 92 93 89 91 92 92 87 89	91-111 88-96 90-94 86-90 90-93 90-94 86-97 84-90 87-91	8.8% 3.7% 2.2% 2.1% 1.4% 1.6% 5.9% 2.8% 1.8%	5 5 5 5 5 5 5 5 5	No data	External std in solvent; Linear, r2>0.99 0.05-10 ng/mL	PP5_50103, GRM044.03A (validation)
loam (soil 2)	0.001	0.001 (Fb) 0.01 (Fb) 0.05 (Fb) 0.001 (F) 0.01 (F) 0.05 (F) 0.001 (X) 0.01 (X) 0.05 (X)	97 95 95 94 95 93 89 88 88	94-99 92-99 92-98 89-98 93-97 91-97 82-93 85-92 85-90	2.8% 2.9% 2.9% 3.7% 1.6% 2.4% 4.8% 2.9% 2.4%	5 5 5 5 5 5 5 5 5	No data	External std in solvent; Linear, r2>0.99 0.05-10 ng/mL	PP5_50103, GRM044.03A (validation)
Sandy loam (soil 3)	0.001	0.001 (Fb) 0.01 (Fb) 0.05 (Fb) 0.001 (F) 0.01 (F) 0.05 (F) 0.001 (X) 0.01 (X)	106 107 106 96 95 95 92 90	103-111 103-111 103-110 94-101 93-97 93-98 84-103 88-93	3.5% 2.8% 2.5% 2.8% 1.9% 1.6% 8.6% 2.5%	5 5 5 5 5 5 5 5	No data	External std in solvent; Linear, r2>0.99 0.05-10 ng/mL	PP5_50103, GRM044.03A (validation)

Matrix	LOQ	Fortification level (mg/kg)	Recovery (%) mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
		0.05 (X)	92	91-94	1.1%	5			
Sand (soil 4)	0.001	0.001 (Fb)	103	99-112	5.1%	5	No data	External std in solvent; Linear, r2>0.99 0.05-10 ng/mL	PP5_50103, GRM044.03A (validation)
		0.01 (Fb)	104	99-106	2.8%	5			
		0.05 (Fb)	105	100-107	2.6%	5			
		0.001 (F)	88	85-91	2.2%	5			
		0.01 (F)	93	91-95	1.8%	5			
		0.05 (F)	99	97-101	1.7%	5			
		0.001 (X)	89	83-95	4.7%	5			
		0.01 (X)	84	79-90	5.6%	5			
		0.05 (X)	91	89-92	1.2%	5			
Soil ns	0.001	0.001 (Fb)	114	110-117	2.4%	5	< 0.001	External std in solvent; Linear, r2>0.99 0.05-10 ng/mL	PP5_50339, TK0114928 (ILV)
		0.01 (Fb)	109	101-116	5.1%	5			
		0.001 (F)	75	67-83	7.5%	5			
		0.01 (F)	85	84-88	2.0%	5			
		0.001 (X)	83	81-89	4.2%	5			
		0.01 (X)	85	82-89	3.3%	5			
Sand	0.001	0.01 (Fb)	92	76-111	11%	12	< 0.001	External std in solvent; Linear, r>0.99 0.05-2.0 ng/mL	PP5_50411; TK0015285 (stor stab)
		0.01 (F)	93	69-118	13%	14			
		0.01 (X)	89	78-101	7.6%	14			
		0.01 (X)	92	83-106	8.0%	10			

Overview of analytical methods

Given the large number of analytical methods utilized, an overview of the analytical methods in plant and animal commodities was prepared to aid in assessing the suitability of each method for use in supervised residue trials (see Table 148).

Table 148 Overview of analytical methods used in supervised residue trials

Method code	Hydrolysis	Valid LOQ	Radio validated	Accept	Remark
Methods for total fluazifop in plant commodities					
PPRAM 51	none	0.02	NA	No	conjugates not taken into account
PPRAM 52	none	0.02	NA	No	conjugates not taken into account
PPRAM 53	none	-	NA	No	conjugates not taken into account
PR 1878	6 M HCl, 60 C, 1 hour	0.05	Yes	Yes	
TMU3418B	6 M HCl, 60 C, duration ns	0.05	no	Yes	
PP009B152	0.2 M NaOH in MeOH, reflux, 1 hour	0.1	no	No	no radiovalidation
PPRAM 62	6 M HCl, 60 C, 1 hour	0.05	yes	Yes	higher LOQs for some commodities
Yokomizo	6 M HCl, 60 C, 1 hour	0.08	yes	Yes	
PPRAM 83	6 M HCl, 60 C, 1 hour	0.05	yes	Yes	higher LOQs for some commodities
PCY 86-1	6 M HCl, 60 C, 1 hour	**0.2**	yes	yes	high LOQ, but most residues >0.2 mg/kg
TMU3251	6 M HCl, 60 C, 1hour	0.05	yes	yes	
RR89-073B	6 M HCl, 60 C, 1 hour	0.01	yes	Yes	
RR91-014B – GC-MS	6 M HCl, 60 C, 1 hour	0.01	yes	Yes	
P-14.077	NaOH, details not available	0.01	no	No	no radiovalidation; higher LOQ for some commodities
PPRAM 122 – NMR	0.2 M NaOH in MeOH, reflux, 1 hour	0.4	no	No	no radiovalidation; high LOQ
PPRAM 122 – HPLC-MS/MS	0.2 M NaOH in MeOH, reflux, 1 hour	0.01	no	No	no radiovalidation

Method code	Hydrolysis	Valid LOQ	Radio validated	Accept	Remark
(A)RAM 197-NMR	6 M HCl, 60 C, 1 hour	0.05	yes	Yes	higher LOQs for some commodities
RAM 287-HPLC-MS/MS	6 M HCl, 60 C, 1 hour	0.01	yes	Yes	higher LOQs for some commodities
R606/BAZ/1	**3 M** HCl, 60 C, 1 hour	0.05	no	No	no radiovalidation incomplete hydrolysis
IT 125	none	0.05	NA	No	conjugates not taken into account
CER 2605	6 M HCl, 60 C, 1 hour	0.01	yes	Yes	
CER 2608	**2 M** HCl, 60 C, 1hour	0.01	no	No	no radiovalidation incomplete hydrolysis
CER 2609	6 M HCl, 60 C, 1 hour	0.01	yes	Yes	
PLMV-027-C	6 M HCl, 60 C, 1 hour	0.02	yes	yes	
RAM 336/01	6 M HCl, 60 C, 1 hour	0.01	yes	yes	
GRM044.01A	1 M NaOH+MeCN (90:10, v/v), reflux 1 hour	0.01	yes	yes	
GRM044.02A	6 M HCl, 60 C, 1 hour	0.01	yes	yes	
POPIT MET.138	6 M HCl, 60 C, 1 hour	0.01	yes	yes	
MRID 40831350-HPLC-MS/MS	6 M HCl, 60 C, 1 hour	0.02	yes	yes	
Analytical methods for total fluazifop in animal commodities					
PPRAM 58	0.2 M NaOH in MeOH, reflux, 1 hour	0.02	yes	yes	
PPRAM 61	0.2 M NaOH in MeOH, reflux, 1 hour	0.05	yes	yes	
RAM 331/01	0.2 M NaOH in MeOH, 60C, 1 hour	0.01	yes	yes	

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability in plant, animal or soil commodities fortified with fluazifop-butyl, fluazifop acid (II) or CF3-pyridone (X) to verify storage stability of the free compounds. The Meeting also received information of storage stability of total fluazifop in plant and animal commodities with incurred residues to verify storage stability of fluazifop (II) conjugates.

Storage stability studies with fortified plant and animal commodities and soil

Fortification study 1 – Fluazifop-butyl.

Homogenised cucumbers were fortified with 0.34 or 0.68 mg/kg fluazifop-P-butyl [IR-4, 1984, PR 1878 (NC)]. Samples were stored for 290 days at -17 °C and then analysed. Total Fluazifop was quantified using HPLC-UV method PR 1878. The analytical method is considered valid for the determination of total fluazifop in cucumber (0.7 mg/kg only).

Storage stability results are presented in Table 149. Samples were reported as uncorrected for average concurrent method recoveries (75% at 0.68 mg/kg).

Reviewer's conclusion: The results indicate that fluazifop-butyl (analysed as total fluazifop) is stable for a period of at least 290 days months at -17 ºC in commodities representative for high water content (cucumbers).

Table 149 Storage stability in cucumber fortified with fluazifop-P-butyl

Commodity	Storage time (days)	Spike level (mg/kg)	Total fluazifop mg/kg	%remaining	concurrent recovery
Cucumber	290	0.68	0.74, 0.69, 0.68	107, 102, 100	75
Cucumber	290	0.34	0.36, 0.39, 0.31	106, 113, 91	na

Fortification study 2 – CF3-pyridone (X)

Duplicate samples of minced onions were fortified with 1.0 mg/kg CF3-pyridone (X) [Morgan and Crook, 1986, PP5/0250, report M4266B]. Samples were stored for 3 months at -18 ºC and were analysed at several intervals. CF3-pyridone (X) was quantified using a modification C of NMR method PPRAM 103. The analytical method is considered valid for the determination of CF3-pyridone (X) in onion bulbs (0.2–1.0 mg/kg).

Storage stability results are presented in Table 150. Samples were not corrected for individual concurrent method recoveries (64–94% at 1.0 mg/kg).

Reviewer's conclusion: The results indicate that CF3-pyridone (X) is stable for a period of at least 3 months at -18 ºC in commodities representative for high water content (onions).

Table 150 Storage stability in bulb onions fortified with 1 mg/kg CF3-pyridone (X)

Analyte	Storage time (months)	Analyte mg/kg [a]	% remaining [b] mean	range	RSD_r	concurrent recovery
CF3-pyridone (X)	0	1.0, 1.0	100	-		64, 70
	1	0.93, 0.95	94	93-95		64, 68
	3	0.93, 1.0	97	93-101		90, 94

[a] residues have been corrected for their concurrent recoveries; uncorrected results are not reported.

[b] %remaining is calculated as mg/kg residue in storage stability sample, divided by mg/kg residue in day 0 sample.

Fortification study 3 – Fluazifop acid (II) and fluazifop-butyl

Homogenised asparagus spears were fortified with 0.20 mg/kg fluazifop acid (II) or 0.2 mg/kg fluazifop-butyl [Baron, 1987, 464389, report IR-4 PR 2201]. Samples were stored for 1 year at -17 °C and then analysed. Total Fluazifop (in this case the sum of fluazifop-butyl and fluazifop acid (II)) was quantified using HPLC-UV method PCY 86-1. The analytical method is considered insufficiently validated for the determination of total fluazifop in asparagus (limited recovery data).

Storage stability results are presented in Table 151. Samples were reported as uncorrected and corrected for average concurrent method recoveries (85% at 0.2 mg/kg).

Reviewer's conclusion: The information from this study cannot used to evaluate storage stability for asparagus spears, because the analytical method has not been shown to be fit for purpose.

Table 151 Storage stability in asparagus spears fortified at 0.2 mg/kg fluazifop acid (II) or 0.2 mg/kg fluazifop-butyl

Analyte	Storage time (yrs)	Total fluazifop mg/kg	% remaining mean (n=2)	concurrent recovery
Fluazifop-butyl	0	NA	-	-
	1	-	58	85
Fluazifop acid (II)	0	NA	-	-
	1	-	62	85

NA not analysed; starting residue level is assumed to be 0.2 mg/kg fluazifop acid (II)

Fortification study 4 – CF3-pyridone (X)

Duplicate homogenised samples of apples, lettuce and soya bean seeds were fortified with 1.00 mg/kg CF3-pyridone (X) [Hayward, 1988, PP5/0076, report M4842B]. Apple and lettuce samples were composite samples obtained from a local market. Soya bean samples were a composite of untreated samples from studies submitted in 1984 and 1985. Samples were stored for 25 months at -18 ºC and were analysed after several intervals. At each interval a recovery experiment was carried out to determine method recovery. CF3-pyridone (X) was extracted using a modification A of NMR method PPRAM 103. The analytical method is considered not valid for the determination of CF3-pyridone (X) in soya bean seeds (low recovery, high RSD, high levels in control samples). The analytical

method is considered valid for the determination of CF3-pyridone (X) in apples (at 1.0 mg/kg only) and lettuce (at 1.0 mg/kg only).

Samples were corrected for concurrent method recovery. Uncorrected results were not reported. Storage stability results are presented in Table 152.

Reviewer's conclusion: The results indicate that CF3-pyridone (X) is stable for a period of at least 25 months at -18 ºC in commodities representative for high water content (apples, lettuce). The information from this study cannot used to evaluate storage stability for soya bean seeds.

Table 152 Storage stability in plant commodities fortified with 1 mg/kg CF3-pyridone (X)

Matrix	Storage time (months)	CF3-pyridone (X) mg/kg [a]	% remaining [b] mean	range	RSD_r	concurrent recovery
Apples	0	1.00, 1.00	100	-		-
	8	0.94, 0.94	94	94-94	-	78, 78
	14	0.89, 0.93	91	89-93	-	96, 92
	25	1.23, 1.09	116	109-123	-	68, 70
Lettuce	0	1.00, 1.00	100	-		-
	8	1.04, 1.14	109	104-114		66, 73
	14	1.13, 1.06	110	106-113		58, 61
	25	0.90, 0.87	88	87-90		78, 91
Soya bean seeds	0	1.00, 1.00	100	-	-	-
	8	0.83, 1.04	94	83-104	-	45, 60
	14	0.75, 0.69	72	69-75	-	50, 59
	25	0.76, 0.96	86	76-96	-	80, 90

[a] residues have been corrected for their concurrent recoveries; uncorrected results are not reported.

[b] %remaining is calculated as mg/kg residue in storage stability sample, divided by mg/kg residue in day 0 sample.

Fortification study 5 – Fluazifop-P-butyl and CF3-pyridone (X)

Duplicate samples of minced onions were fortified with 1.00 mg/kg fluazifop-P-butyl or 1.00 mg/kg CF3-pyridone (X) [Hayward, 1988, PP5/0077, report M4843B, Atreya, 1990, PP5/0799, report M5163B summary]. Samples were stored for 28 months at -18 ºC and were analysed at several intervals. At each interval a recovery experiment was carried out to determine method recovery. Fluazifop-butyl and CF3-pyridone (X) were quantified using a modification A of NMR method PPRAM 103 (hydrolysis step omitted). The analytical method is considered valid for the determination of fluazifop-butyl in onion bulbs (1.0 mg/kg only) and CF3-pyridone (X) in onion bulbs (0.2–1.0 mg/kg).

Samples were corrected for concurrent method recovery. Uncorrected results were not reported. Storage stability results are presented in Table 153.

Since NMR method PPRAM 103 uses a non-acid extraction (acetone/water) and the hydrolysis step was omitted, fluazifop-butyl remains intact as the butyl ester in this study. The NMR method detects shifts in the different components and the peak height at a specific shift is proportional to the residue of a specific component. The shift of the F peaks will depend on the nature of the molecule attached to it. Therefore it would be expected that different shifts might be expected for CF3-pyridone (X), parent and their breakdown products (as long as they contain F). The presented NMR chouromatograms only show two peaks, corresponding to the parent and CF3-pyridone (X). The significantly different shifts for parent and CF3-pyridone (X) suggest that possible breakdown products of fluazifop-butyl that contain F (eg. Pyr-Ph ether (IV) and fluazifop acid (II)) would be observed, but were not seen, as no peaks at different shifts were seen. The storage stability therefore relates to fluazifop-butyl (as parent) and to CF3-pyridone (X, free) [Syngenta, Response to questions 14, 26 August, 2016]

Reviewer's conclusion: The results indicate that fluazifop-butyl (as parent) and CF3-pyridone (X) are stable for a period of at least 28 months at -18 ºC in commodities representative for commodities with high water content (bulb onions).

Table 153 Storage stability in onions fortified with 1 mg/kg fluazifop-P-butyl and 1 mg/kg CF3-pyridone (X)

Analyte	Storage time (months)	Analyte mg/kg [a]	% remaining [b] mean	range	RSD_r	concurrent recovery
Fluazifop-butyl	0	1.10, 1.00	100	-	-	75, 83
	1	0.96, 0.95	91	87-95	-	87, 88
	3	1.08, 1.02	100	98-102	-	85, 88
	12	1.03, 1.04	99	94-104	-	87, 90
	28	0.88, 0.84	82	80-84	-	98,106
CF3-pyridone (X)	0	1.03, 0.96	100	-		64, 70
	1	0.93, 0.95	94	90-99	-	64, 68
	3	0.88, 1.01	95	85-105	-	90, 94
	10	0.81, 0.89, 1.04, 1.01	94	79-108	15%	74, 68, 73,66
	15	1.02, 1.10	106	99-115	-	76, 67
	28	0.90, 0.88	89	87-92	-	83, 88

[a] residues have been corrected for their concurrent recoveries; uncorrected results are not reported.

[b] %remaining is calculated as mg/kg residue in storage stability sample, divided by mg/kg residue in day 0 sample.

Fortification study 6 – CF3-pyridone (X)

Duplicate samples of homogenised peanut kernels were fortified with 1.00 mg/kg CF3-pyridone (X) [Hayward, 1988, 462746, report M4841B]. Samples were a composite of samples obtained from a local market. Samples were stored for 25 months at –18 ºC and were analysed at several intervals. At each interval a recovery experiment was carried out to determine method recovery. CF3-pyridone (X) were extracted using a modification A of NMR method PPRAM 103. The analytical method is considered valid for the determination of CF3-pyridone (X) in peanut kernels (at 1.0 mg/kg only).

Samples were corrected for concurrent method recovery. Uncorrected results were not reported. Storage stability results are presented in Table 154.

Reviewer's conclusion: The results indicate that CF3-pyridone (X) is stable for a period of at least 24 months at -18 ºC in commodities representive for commodities with high oil content (peanut kernels).

Table 154 Storage stability in peanut kernels fortified with 1 mg/kg CF3-pyridone (X)

Analyte	Storage time (months)	CF3-pyridone mg/kg [a]	% remaining [b] mean	range	RSD_r	concurrent recovery
CF3-pyridone (X)	0	1.00, 1.00	100	-		70, 70
	8	1.00, 0.88	94	88-100	-	65
	14	0.84, 0.86	85	84-86	-	70, 70
	24	1.13, 1.38	126	113-138	-	93, 73

[a] residues have been corrected for their concurrent recoveries; uncorrected results are not reported.

[b] %remaining is calculated as mg/kg residue in storage stability sample, divided by mg/kg residue in day 0 sample.

Fortification study 7 – Fluazifop-butyl

Homogenised asparagus spears were fortified with 0.50 or 0.10 mg/kg fluazifop acid (II) [Baron, 1989, no code, IR-4 PR 3944]. Samples were stored for 235 days at -20 °C and then analysed. Total Fluazifop was quantified using HPLC-UV method PPRAM 62. The analytical method is considered valid for the determination of total fluazifop in asparagus (at 0.1 mg/kg).

Storage stability results are presented in Table 155. Samples were reported as uncorrected for average concurrent method recoveries (77% at 0.10 mg/kg).

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a period of at least 235 days months at -20 ºC in commodities representative for high water content asparagus).

Table 155 Storage stability in asparagus fortified with fluazifop acid (II)

Commodity	Storage time (days)	Spike level (mg/kg)	Total fluazifop mg/kg	%remaining	concurrent recovery
Asparagus	235	0.50	0.437, 0.405	87, 81	NA
Asparagus	235	0.10	0.101, 0.087	101, 87	77

NA = not analysed

Fortification study 8 – Fluazifop acid (II)

Homogenised green coffee beans were fortified with 0.50 mg/kg fluazifop acid (II) [Baron, 1988, 471695, report IR-4 PR 03432 (1988)]. Samples were stored for 6 months at -15 °C and then analysed. Fluazifop acid (II) was quantified using HPLC-UV method PPRAM 62/2. The analytical method is considered valid for the determination of fluazifop acid (II) in green coffee beans (0.05–2.0 mg/kg).

Samples were not corrected for average concurrent method recoveries (70% at 0.5 mg/kg). Storage stability results are presented in Table 156.

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a period of at least 198 days (7 months) at -15 °C or lower in commodities with high oil content (coffee beans).

Table 156 Storage stability in green coffee berries fortified with 0.5 mg/kg fluazifop acid (II)

Analyte	Storage time (days)	Fluazifop acid (II) mg/kg	% remaining			concurrent recovery		
			mean	range	RSD_r	mean	range	RSD
Fluazifop acid (II)	0	NA	-			-		
	198	-	75	53-88	18%	70	56-80	15%

NA not analysed; starting residue level is assumed to be 0.5 mg/kg fluazifop acid

Fortification study 9 – Fluazifop acid (II)

Homogenised macadamia nuts were fortified with 0.50 mg/kg fluazifop acid (II) [Baron, 1989; 464386, report IR-4 PR 3431]. Samples were stored for 8 months at -15 °C and then analysed. Total fluazifop was quantified using HPLC-UV method PAM II (i.e. PPRAM 62/2). The analytical method is considered valid for the determination of fluazifop acid (II) in macadamia nutmeat (at 0.5 mg/kg only).

Storage stability results are presented in Table 157. Samples were reported uncorrected and corrected for average concurrent method recoveries (62–94%).

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a period of at least 8 months at -15°C in commodities representative of high oil content (macadamia nuts).

Table 157 Storage stability in macadamia nutmeat fortified with 0.2 mg/kg fluazifop acid (II)

Analyte	Storage time (months)	Total fluazifop mg/kg	% remaining (n=3)			concurrent recovery (n=5)		
			mean	range	RSD	mean	range	RSD
Fluazifop acid (II)	0	NA	-			-		
	8	-	62	60-66	5.6%	71	62-94	18%

NA not analysed; starting residue level is assumed to be 0.5 mg/kg fluazifop acid (II)

Fortification study 10 – Fluazifop acid (II)

Homogenised sweet potatoes were fortified with 0.20 mg/kg fluazifop acid (II) [Barney, 2011, PP5_50290, report IR-4 PR 02328]. Thouree subsamples were stored for 922 days at -20 °C and then

analysed. Fluazifop acid (II) was quantified using HPLC-MS/MS method MRID 40831305. The analytical method is considered valid for the determination of fluazifop acid (II) in sweet potatoes (0.02–5.0 mg/kg).

Samples were not corrected for concurrent method recovery (71–103% at 0.02–5.0 mg/kg) for fluazifop acid (II). Storage stability results are presented in Table 158.

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a period of at least 922 days (31 months) at -20 °C in commodities representative of high starch content (sweet potato tubers).

Table 158 Storage stability in sweet potato fortified with 0.2 mg/kg fluazifop acid (II)

Analyte	Storage time (days)	Fluazifop acid (II) mg/kg	% remaining mean	range	RSD_r	concurrent recovery
Fluazifop acid (II)	0 922	NA 0.18	- 94	- -	- -	- 71%-103%

NA not analysed; starting residue level is assumed to be 0.2 mg/kg fluazifop acid (II)

Fortification study 11 – Fluazifop acid (II)

Homogenised raspberries were fortified with 0.50 mg/kg fluazifop acid (II) [Arsenovic and Jolly, 2013, PP5_50556, report IR-4 PR 03947]. Thouree subsamples were stored at -38 °C to -2 °C for 687 days then analysed. Fluazifop acid (II) was quantified using HPLC-MS/MS method GRM044.01A modification B. The analytical method is considered valid for the determination of fluazifop acid (II) in caneberries (0.02–5.0 mg/kg).

Storage stability results are presented in Table 159. Samples were not corrected for average concurrent method recoveries (88% at 0.5 mg/kg).

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a period of at least 687 days (23 months) at -2 °C or lower in commodities representative of high acid content (caneberries).

Table 159 Storage stability in caneberries fortified with 0.5 mg/kg fluazifop acid (II)

Analyte	Storage time (days)	Total fluazifop mg/kg	% remaining mean	range	RSD_r	concurrent recovery
Fluazifop acid (II)	0 687	NA -	- 89	- 88-90	- 1.3%	- 88

NA not analysed; starting residue level is assumed to be 0.5 mg/kg fluazifop acid (II)

Fortification study 12 – Fluazifop acid (II)

Homogenised blueberries were fortified with 0.50 mg/kg fluazifop acid (II) [Arsenovic and Jolly, 2013, PP5_50557, report IR-4 PR 02083]. Thouree subsamples were stored at -35 °C to -1 °C for 795 days then analysed. Fluazifop acid (II) was quantified using HPLC-MS/MS method GRM044.01A modification B. The analytical method is considered valid for the determination of fluazifop acid (II) in blueberries (0.02–2.0 mg/kg).

Storage stability results are presented in Table 160.Samples were not corrected for average concurrent method recoveries (89% at 0.5 mg/kg).

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a period of at least 795 days (27 months) at -1 °C or lower in commodities representative of high acid content (blueberries).

Table 160 Storage stability in caneberries fortified with 0.5 mg/kg fluazifop acid (II)

Analyte	Storage time	Fluazifop acid (II)	% remaining	concurrent

	(days)	mg/kg	mean	range	RSD_r	recovery
Fluazifop acid (II)	0	NA	-			-
	795	-	95	94-96	1.1%	89

NA not analysed; starting residue level is assumed to be 05 mg/kg fluazifop acid (II)

Fortification study 13 – Fluazifop acid (II)

Homogenised rhubarb was fortified with 0.50 mg/kg fluazifop acid (II) [Arsenovic, 2013, PP5_50552, report IR-4 PR A2404 (2013)]. Samples were stored at -38 °C to -1 °C for 744 days then analysed. Fluazifop acid (II) was quantified using HPLC-MS/MS method GRM044.01A modification B. The analytical method is considered valid for the determination of fluazifop acid (II) in rhubarb (0.02–5.0 mg/kg).

Samples were not corrected for average concurrent method recoveries (92% at 0.5 mg/kg). Storage stability results are presented in Table 161.

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a period of at least 744 days (25 months) at -1 °C or lower in commodities representative of high acid content (rhubarb).

Table 161 Storage stability in rhubarb fortified with 0.5 mg/kg fluazifop acid (II)

Analyte	Storage time (days)	Fluazifop acid (II) mg/kg	% remaining mean	range	RSD_r	concurrent recovery
Fluazifop acid (II)	0	NA	-			-
	744	-	92	89-95	3.3%	92

NA not analysed; starting residue level is assumed to be 0.5 mg/kg fluazifop acid (II)

Fortification study 14 – Fluazifop acid (II)

Homogenised strawberries were fortified with 0.10 mg/kg fluazifop acid (II) [Arsenovic, 2014, PP5_50553, report PR A2085]. Thouree subsamples were stored for 693 days at -22°C then analysed. Fluazifop acid was quantified using HPLC-MS/MS method GRM044.01A modification B. The analytical method is considered valid for the determination of fluazifop acid (II) in strawberries (0.01–5.0 mg/kg).

Samples were not corrected for average concurrent method recoveries (85% at 0.1 mg/kg). Storage stability results are presented in Table 162.

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a period of at least 693 days (23 months) at -22 °C or lower in commodities representative of high acid content (strawberries).

Table 162 Storage stability in strawberries fortified with 0.1 mg/kg fluazifop acid (II)

Analyte	Storage time (days)	Fluazifop acid (II) mg/kg	% remaining mean	range	RSD_r	concurrent recovery mean	range	RSD_r
Fluazifop acid (II)	0	NA	-			-		
	693	-	85	83-89	3.8%	85	77-90	5.9%

NA not analysed; starting residue level is assumed to be 0.1 mg/kg fluazifop acid (II)

Fortification study 15 – Fluazifop acid (II)

Homogenised forage and hay samples of fine fescue grass were fortified with 0.5 mg/kg fluazifop acid (II) [Jolly, 2014, PP5_50554, report IR-4 PR 09825]. Samples were stored for 1043 (forage) and 1047 (hay) days at -22 °C then analysed. Fluazifop acid (II) was quantified using HPLC-MS/MS method MRID 40831305. The analytical method is considered valid for the determination of fluazifop acid (II) in grass forage (0.02–5.0 mg/kg) and grass hay (0.02–5.0 mg/kg).

Samples were not corrected for average concurrent method recoveries (70% (forage) and 70% (hay) at 0.5 mg/kg). Storage stability results are presented in Table 163.

Reviewer's conclusion: The results indicate that fluazifop acid (II) is not stable in grass forage (high water content) and grass hay (dry commodities). The reason for this is not clear, since fluazifop acid (II) is very resistent to analytical hydrolysis procedures (acid, base, enzymatic).

Table 163 Storage stability in fine fescue grass forage and hay fortified with 0.5 mg/kg fluazifop acid (II)

Commodity	Storage time (days)	Fluazifop acid (II) mg/kg	% remaining			concurrent recovery
			mean	range	RSD	
	0	NA	-			-
- forage	1043	-	55	49-59	9.6%	70
- hay	1047	-	67	65-69	3.1%	77

NA not analysed; starting residue level is assumed to be 0.5 mg/kg fluazifop acid (II)

Fortification study 16 – Fluazifop acid (II)

Samples of soya bean hulls, soya bean meal, soya bean milk and soya bean oil were fortified with fluazifop acid (II) at 0.25 mg/kg and stored at < -18 °C for 18 months [McGill, 2003, PP5/1281, report RJ3087B]. Soya bean hulls and soya bean meal control samples were obtained from from a processing study (98JH142). Soya bean milk and soya bean oil were obtained from a local supermarket. Duplicate samples of each matrix were analysed before storage and after 3, 6, 9, 12 and 18 months for residues of total fluazifop (i.e., sum of fluazifop-butyl, fluazifop acid (II) and its conjugates, expressed as fluazifop acid (II)). The total fluazifop in soya bean hull and soya bean meal was determined by HPLC-MS-MS method RAM 287/02. The total fluazifop in soya bean milk was determined by HPLC-MS-MS method RAM 336/01. Total fluazifop in soya bean oil was determined by HPLC-MS/MS methods RAM 122/04 and RAM 122/05. The analytical methods are considered valid for the determination of fluazifop acid (II) in soya bean hulls (at 0.25 mg/kg only), soya bean meal (0.01–0.25 mg/kg), soya bean milk (0.01–0.25 mg/kg), and soya bean oil (0.01–0.25 mg/kg).

Storage stability results are presented in Table 164. Average concurrent recoveries ranged between 71–119% for each matrix. Control samples had residues < 0.01 mg/kg eq total fluazifop, except soya bean hulls. An average total fluazifop residue of 0.03 mg/kg eq was detected in the control soya bean hull samples. All total fluazifop residues detected in soya bean hulls were corrected for the contamination measured in the untreated sample in each batch.

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a at least 12 months at -18 °C in soya bean meal and at least 18 months -18 °C in soya bean hulls, soya bean oil and soya bean milk (processed commodities).

Table 164 Storage stability in soya commodities fortified with 0.25 mg/kg fluazifop acid (II)

Commodity	Storage time (months, days)	Fluazifop acid (II) mg/kg (n=2-3, mean)	% remaining (n=2-3) [a] mean	Mean concurrent recovery
Soya bean hulls	0	0.27	100	109
	3 (121 days)	0.26	96	111
	6 (198 days)	0.25	93	92
	9 (296 days)	0.28	104	107
	12 (394 days)	0.26	96	107
	18 (569 days)	0.24	89	99
Soya bean meal	0	0.26	100	102
	3 (121 days)	0.21	81	111
	6 (198 days)	0.30	115	113
	9 (296 days)	0.26	100	100
	12 (394 days)	0.20	77	96
Soya bean milk	0	0.22	100	98
	3 (122 days)	0.27	123	109

	6 (189 days)	0.23	105	73
	9 (300-310 days)	0.25	114	78
	12 (358 days)	0.23	105	89
	18 (549 days)	0.30	136	99
Soya bean oil	0 (19 days)	0.20	100	84
	3 (96 days)	0.16	80	81
	6 (183 days)	0.20	100	89
	9 (277 days)	0.22	110	101
	12 (491 days)	0.23	115	71
	18 (545 days)	0.23	115	87

[a] %remaining is calculated as mg/kg residue in storage stability sample, divided by mg/kg residue in day 0 sample.

Fortification study 17 – Fluazifop acid (II)

Samples of various processed commodities were fortified with fluazifop acid (II) at 0.20 mg/kg and stored at < -16 °C for 12 months [Tauber and Hagan, 2013, R156172_50001, report 13SYN331REP]. Untreated potato flakes, potato wet peel, soya bean hulls, soya bean meal, soya bean oil, wheat flour, wheat middlings and wheat shorts were obtained from processing studies. Organic potato chips, tomato paste and tomato puree were purchased locally. Duplicate samples of each matrix were analysed before storage and after 3, 6, 9 and 12 months for residues of total fluazifop (i.e., sum of fluazifop-butyl, fluazifop acid (II) and its conjugates, expressed as fluazifop acid (II)) using HPLC-MS/MS method GRM044.01A and its modification A (potato flakes only). The analytical method is considered valid for the determination of fluazifop acid (II) in potato flakes, potato wet peel, potato chips, soya bean meal, soya bean hull, soya bean oil, wheat flour, wheat middlings, wheat shorts, tomato paste and tomato puree (each at 0.2 mg/kg only).

Storage stability results are presented in Table 165. Average concurrent recoveries ranged between 75–95% for each matrix. Control samples had residues < 0.025 mg/kg eq total fluazifop.

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for a at least 12 months at -16 °C in potato flakes, potato wet peel, potato chips, soya bean meal, soya bean hulls, soya bean oil, wheat flour, wheat middlings, wheat shorts, tomato paste and tomato puree (processed commodities).

Table 165 Storage stability in processed commodities fortified with 0.20 mg/kg fluazifop acid (II)

Commodity	Storage time (months	Fluazifop acid (II) mg/kg (n=2)	%remaining (n=2) [a] mean	% remaining (n=2) [b] mean, corrected	Concurrent recovery mean
Pototo flakes	0	149, 141	73	100	70, 76
	3	130, 131	66	99	64, 67
	6	139, 141	71	97	74, 72
	9	144, 141	72	97	70, 77
	12	170, 156	82	89	94, 90
Potato wet peel	0	186, 182	92	101	90, 92
	3	182, 183	92	98	95, 93
	6	161, 174	84	102	80, 86
	9	182, 177	90	106	85, 85
	12	179, 170	88	94	95, 91
Potato chips	0	177, 173	88	105	83, 84
	3	140, 145	72	94	75, 77
	6	125, 137	66	96	65, 72
	9	151, 155	77	102	76, 75
	12	154, 168	81	97	85, 80
Soya bean meal	0	176, 177	89	103	83, 89
	3	156, 151	77	99	76, 79
	6	144, 141	72	98	75, 71
	9	159, 143	76	98	74, 81
	12	160, 169	83	89	91, 94
Soya bean hulls	0	186, 187	94	99	96, 94
	3	187, 186	94	92	106, 98
	6	153, 149	76	102	71, 79

Commodity	Storage time (months	Fluazifop acid (II) mg/kg (n=2)	%remaining (n=2) [a] mean	% remaining (n=2) [b] mean, corrected	Concurrent recovery mean
	9	178, 176	89	100	88, 90
	12	170, 159	83	95	87, 86
Soya bean oil	0	188, 181	93	103	88, 91
	3	181, 179	91	102	87, 91
	6	201, 178	95	113	84, 83
	9	171, 180	88	105	83, 85
	12	165, 159	82	94	90, 84
Wheat flour	0	170, 158	82	114	62, 81
	3	155, 146	76	97	71, 85
	6	153, 148	76	103	70. 76
	9	158, 142	75	105	70, 73
	12	141, 146	72	88	81, 82
Wheat middlings	0	166, 173	85	111	76, 78
	3	156, 152	77	104	73, 75
	6	148, 140	72	99	73, 73
	9	145, 142	72	90	80, 80
	12	149, 154	76	90	76, 62
Wheat shorts	0	157, 165	81	103	77, 81
	3	172, 170	86	107	77, 83
	6	152, 162	79	101	82, 74
	9	140, 143	71	82	81, 92
	12	150, 169	80	89	81, 98
Tomato paste	0	189, 185	94	109	88, 83
	3	178, 180	90	95	97, 92
	6	183, 188	93	111	85, 83
	9	161, 172	84	86	99, 97
	12	219, 198	105	92	111, 117
Tomato puree	0	192, 191	96	116	73, 92
	3	181, 188	93	100	95, 91
	6	176, 175	88	100	91, 84
	9	182, 174	89	96	99, 86
	12	204, 204	102	87	117, 116

[a] %remaining is calculated as mg/kg residue in storage stability sample, divided by the spike level (0.2 mg/kg).

[b] %remaining is calculated as a, but corrected for concurrent method recovery.

Fortification study 18 – Fluazifop acid (II)

Portions of bovine muscle, fat, liver, kidney, milk and hen eggs were fortified with 0.1 mg/kg fluazifop acid (II) [Wimbush, 2003, PP5/1243, report 1983/045-D2149]. Samples were kept frozen at -20 °C for 12 months. Samples were analysed for total fluazifop using GC-MS method RAM 331/01. The method was slightly modified: quantification was at m/z=254 as this ion was less prone to interferences than ions at m/z 341. Further the 0 and 3 month samples were analysed on a different GC column (ZB5). The analytical method is considered valid for the determination of fluazifop acid (II) in bovine muscle (0.05–0.1 mg/kg), bovine fat (0.05 mg/kg only), bovine liver (0.05–0.1 mg/kg), bovine kidney (0.01–0.1 mg/kg), cow milk (0.05–0.1 mg/kg) and hen eggs (0.01–0.1 mg/kg).

Results are shown in Table 166. Samples were not corrected for matrix interferences (< 0.3 LOQ), nor for concurrent recovery values (61–131%).

Reviewer's conclusion: The results indicate that fluazifop acid (II) is stable for at least 344–354 days (12 months) at -20 °C in animal commodities (eggs, milk, tissues).

Table 166 Storage stability in animal commodities fortified with 0.1 mg/kg fluazifop acid (II) and stored at -20 °C

Matrix	Storage time (days)	Fluazifop acid (II) (mg/kg) mean	range	%remaining relative to day 0 mean	%remaining relative to nominal value mean (range, n=3)	Concurrent method recovery mean (range, n=2)
Bovine liver	0	0.088	0.083-0.096	100	88 (83-96)	82 (78-86)

Matrix	Storage time (days)	Fluazifop acid (II) (mg/kg) mean	range	%remaining relative to day 0 mean	%remaining relative to nominal value mean (range, n=3)	Concurrent method recovery mean (range, n=2)
	96	0.073	0.071–0.076	83	73 (71-76)	76 (72-81)
	187	0.119	0.11–0.13	135	119 (112-126)	99 (86-112)
	354	0.105	0.10-0.11	119	105 (101-110)	104 (102-106)
Bovine kidney	0	0.102	0.091–0.12	100	102 (91-120)	110 (99-122)
	89	0.069	0.055-0.086	68	69 (55-86)	60 (51-70)
	180	0.109	0.11–0.11	107	109 (107-113)	104 (98-109)
	347	0.096	0.094-0.098	94	96 (94-98)	91 (90-92)
Bovine muscle	0	0.084	0.078-0.088	100	84 (78-88)	83 (78-88)
	95	0.061	0.058-0.063	73	61 (58-63)	68 (68-68)
	182	0.086	0.080-0.098	102	86 (80-98)	82 (82-83)
	349	0.094	0.092-0.096	112	94 (92-96)	90 (86-94)
Bovine fat	0	0.083	0.080-0.086	100	83 (80-86)	69 (55-82)
	90	0.056	0.050-0.059	67	56 (50-59)	62 (57-66)
	177	0.071	0.055-0.081	86	71 (55-81)	76 (70-81)
	344	0.080	0.078-0.083	96	80 (78-83)	77 (77-77)
Bovine milk	0	0.096	0.094-0.098	100	96 (94-98)	94 (94-95)
	90	0.132	0.12-0.14	137	132 (118-143)	131 (131-250[a])
	177	0.112	0.11–0.12	117	112 (108-116)	111 (111-112)
	344	0.087	0.076-0.097	91	87 (76-97)	76 (71-80)
Hen eggs	0	0.088	0.080-0.093	100	88 (80-93)	80 (75-86)
	90	0.115	0.11–0.12	131	115 (112-120)	101 (98-104)
	179	0.086	0.083-0.091	98	86 (83-91)	96 (91-101)
	344	0.079	0.077-0.081	90	79 (77-81)	78 (73-82)

[a] value excluded from the calculation of the mean recovery

Fortification study 19 – Fluazifop-P-butyl, fluazifop acid (II), CF3-pyridone (X)

Loam soil samples (Visalia, CA, USA, pH 7.3, CEC 8.4 meq/100 g, 1.1% organic matter, moisture content 10%) were individually fortified each with 0.4 mg/kg fluazifop-P-butyl, fluazifop acid (II) or CF3-pyridone (X) [Wiebe, 1995, PP5/0798, report RR95-002B]. Samples were stored for up to 36 months at -20 ºC ± 10 ºC and were analysed (n = 3 per sample) at several intervals. At each interval two control samples were fortified with 0.04 and 0.4 mg/kg for each analyte to determine concurrent method recovery. Fluazifop-butyl and fluazifop acid (II) were quantified using GC-MS method RR 89-072B. CF3-pyridone (X) was quantified using GC-MS method RR 90-076B. Modification A of GC-MS method 89-072B was used to quantify fluazifop acid (II) in the 36 months storage samples. The analytical methods are considered valid for the determination of fluazifop-butyl in soil (0.01–0.4 mg/kg), fluazifop acid (II) (0.01–0.4 mg/kg), and CF3-pyridone (X) (0.01–0.4 mg/kg).

Storage stability results are presented in Table 167. Samples were not corrected for average concurrent method recovery: 95% for fluazifop-butyl, 90% for fluazifop acid (II) and 97% for CF3-pyridone (X), respectively. Control samples were < 0.01 mg/kg for each analyte, except for one sample containing 0.018 mg/kg fluazifop-butyl.

Reviewer's conclusion: The results indicate that fluazifop-butyl is stable in frozen soil for a maximum of 12 months, fluazifop acid (II) for at least 24 months and CF3-pyridone (X) for at least 36 months. Fluazifop-butyl degraded to fluazifop acid (II) after 12 months of storage.

Table 167 Storage stability in loam soil fortified with 0.4 mg/kg fluazifop-P-butyl, fluazifop acid (II) or CF3-pyridone (X)

Analyte	Storage time (months)	% remaining (n=3) [a] mean	range	RSD_r	concurrent recovery
Fluazifop-butyl	0	98	94-101	3.6%	90, 87
	1 week	78	76-80	2.6%	76, 79
	1 month	85	68-96	18%	101, 101
	3	83	75-90	9.2%	86, 81
	6	97	80-108	16%	122, 112

	12	70	57-93	28%	90, 92
	18	53	43-58	16%	107, 116
	24	44 [b]	41-47	7.0%	103, 81
Fluazifop acid (II)	0	89	76-96	12%	75, 80
	1 week	93	90-97	3.8%	84, 90
	1 month	78	30-103	53%	113, 90
	1.25	101	87-111	12%	-
	3	88	83-91	4.7%	92, 75
	6	108	103-118	7.7%	108, 104
	12	90	76-114	24%	108, 95;
	18	86	71-95	15%	102, 96;
	24	85	74-98	14%	97, 100
	36	63 [c]	59-66	5.6%	68, 66
CF3-pyridone (X)	6	87	84-90	3.4%	90, 82
	8	92	91-94	1.7%	-
	12	84	83-85	1.2%	87, 85
	18	97	95-98	1.6%	109, 115
	24	93	79-101	13%	95, 94
	36	95	90-98	4.4%	104, 106

[a] %remaining is the amount found in the samples compared to the nominal amount added (0.4 mg/kg).

[b] These samples were also analysed for fluazifop. A 62% remaining (as fluazifop-butyl equivalents) was found, showing that, within experimental error, all of the missing fluazifop-butyl (100%-44%=56% missing) was recovered as fluazifop acid (II).

[c] A modified analytical method was used, whereby the methylation step was modified and a different standard was used. The concurrent method recovery (68%, 66%) indicates that a lower recovery is obtained with this method, making the result not reliable.

Fortification study 20 – Fluazifop-butyl, fluazifop acid (II), CF3-pyridone (X)

Composite soil samples were fortified with 0.01 mg/kg of fluazifop-P-butyl or 0.01 mg/kg each of a mix of fluazifop acid (II) and CF3-pyridone (X) [Pyles and Hagan, 2013, PP5_50411, TK0015285]. Soil characteristics were: sand, pH 6.3, 0.55% organic matter, CEC 3.5, Georgia, USA, 0-3 inch soil depth. Samples were stored at -15 °C for 19 months.

A new storage stability experiment for CF3-pyridone (X) was also initiated approximately 11 months into the initial study to confirm the recovery trend observed in the samples from the initial study. These samples were fortified with 0.01 mg/kg CF3-pyridone (X) and stored frozen at -15 °C.

Residue levels in soil were determined by using HPLC-MS/MS method GRM044.03A with some minor modifications. The analytical method is considered valid for the determination of fluazifop-butyl in soil (0.001–0.5 mg/kg), fluazifop acid (II) (0.001–0.5 mg/kg), and CF3-pyridone (X) (0.001–0.5 mg/kg).

Storage stability results are presented in Table 168 and Table 169. Samples were not corrected for average concurrent method recovery (76–118%). In samples fortified with fluazifop-P-butyl, fluazifop acid (II) was found in low levels after 1 month of storage. It is likely that fluazifop-P-butyl degrades into fluazifop- acid during frozen storage.

The results from this study demonstrate that parent fluazifop-P-butyl, fluazifop acid (II) and CF3-pyridone (X) are stable under freezer storage conditions in soil for maximally 19, 15 and 15-18 months, respectively. The results for fluazifop acid (II) and CF3-pyridone (X) are not consistent with data from a previous storage stability study [RR 95-002B] where these substances were reported to be stable for up to 24 and 36 months, respectively. According to the study author, the lower storage stability for fluazifop acid (II) and CF3-pyridone (X) observed in the present study may be due to partially irreversible adsorption (bound residues) over time, not degradation.

Table 168 Storage stability in soil fortified with 0.01 mg/kg fluazifop-P-butyl, fluazifop acid (II) or CF3-pyridone (X)

Analyte	Storage time	% remaining (n=2) [a]	concurrent	Fluazifop acid (II)

	(months)	mean	range	recovery (n=2) mean (%)	(mg/kg)	Recalculated to fluazifop-butyl mg/kg	%
Fluazifop-P-butyl	0	101	96-106	111	< 0.0005	< 0.00059	<5.9
	0 [b]	112	110-114	98	< 0.0005	< 0.00059	<5.9
	1	85	84-86	90	0.00154[d]	0.0018	18
	3	85	84-86	89	0.00056	0.00066	6.6
	6	85	83-87	86	0.00058	0.00068	6.8
	7	67	67-67	90	0.00064	0.00075	7.5
	9	66	64-68	76	0.00059	0.00069	6.9
	11	75	73-77	88	0.00085 [d]	0.001	10
	12	97	93-100	110	0.00103	0.0012	12
	15	93	92-95	93	0.00080	0.00094	9.4
	18	76	76-77	85	0.00098	0.0012	12
	19	86	85-86	89	0.00084	0.00098	9.8
Fluazifop acid (II)	0	86	84-88	86			
	0 [b]	88	87-90	83			
	1	85	83-88	93			
	3	76	74-79	78			
	6	109	105-113	111			
	7	79	79-80	92			
	9	69	68-70	91			
	11	113	112-114	118			
	12	102	101-103	101			
	15	75	74-76	98			
	16	65	63-68	69			
	18	56	51-60	92			
	18 [c]	59	57-62	94			
	19	61	61-61	93			
CF3-pyridone (X)	0	82	81-83	93			
	0 [b]	73	71-75	85			
	1	79	75-82	89			
	3	78	76-80	81			
	6	83	80-87	90			
	7	75	74-77	88			
	7 [c]	73	73-73	95			
	9	78	78-79	85			
	11	88	88-88	94			
	12	83	83-83	101			
	15	83	83-83	97			
	16	84	78-90	91			
	18	70	66-74	80			
	19	38	37-38	78			

[a] %remaining is the amount found in the samples compared to the nominal amount added (0.01 mg/kg).

[b] samples were extracted at same date as the first day-0 sample, but analysed again 2 days later

[c] about 1-2 weeks later, the sample was extracted and analysed again

[d] also in fresh fortified sample with fluazifop-P-butyl, fluazifop-acid (II) is measured

Table 169 Storage stability in soil fortified with 0.01 mg/kg CF3-pyridone (X)

Analyte	Storage time (months)	% remaining (n=2) [a] mean	range	concurrent recovery (n=2) mean (%)
CF3-pyridone (X)	0	88	87-89	87
	1	72	70-73	89
	3	81	74-89	95
	6	64	62-65	89
	7	72	72-72	83
	9	88	85-91	93
	13	72	72-73	100
	15	78	77-80	106
	18	63	63-63	98
	19	59	59-60	84

[a] %remaining is the amount found in the samples compared to the nominal amount added (0.01 mg/kg).

Storage stability studies with incurred residues in plant and animal commodities

Incurred residue study 1

Soya bean plants were treated in the field with an EC fluazifop-butyl (racemate) formulation at a growth stage of one trifoliate leaf at a rate of 0.750 kg ai/ha [Atreya and Collis, 1981, PP9/0431, report 496/PP009B017; Atreya, 1990, PP5/0799, M5163B summary]. Soya bean seeds were treated on 7 July 1979 and were sampled 111 days later on 26 October 1979. Seeds were stored for 16 months at -18 ± 2 °C. The seed sample was first analysed on 8 February 1980 (3.5 months after harvest). The initial residue level and the residues after a further 3 months of storage were quantified with method TRAM. The residues after a further 4.5 and 12.5 months storage were quantified with method PPRAM 52. Method TRAM and method PPRAM 52 determine fluazifop (fluazifop-P-butyl and free fluazifop (II)) but not the fluazifop (II) conjugates.

The concurrent recoveries are not mentioned in the report. The mean measured concentrations, corrected for concurrent method recoveries, are reported in Table 170. Uncorrected residue levels were not reported.

Reviewer's conclusion: The information from this study cannot be used to evaluate storage stability for the following reasons:

- Methods TRAM and PPRAM 52 are considered not valid for quantification of total fluazifop residues in soya bean seeds.
- The study report indicated that lower residues were obtained with the TRAM method (3.5 and 6.5 month storage) than with the PPRAM 52 method (8 and 16 month storage), because of a different extraction method. Based on this information any degradation remains unnoticed, because the extraction efficiency is increased for the later samples.
- Residues were not analysed at harvest. Since the rate of degradation is generally highest in the first 3 months after harvest and thereafter stabilizes, % remaining since harvest cannot be calculated.

Table 170 Storage stability of residues in soya bean seeds with incurred residues at -18 °C

commodity	Treatment (kg ai/ha) (fluazifop-butyl)	Harvest (DALT)	Storage time (months)	Total fluazifop (mg/kg)	% remaining	concurrent recovery (%)
Soya bean seeds; study 5009/79	1 × 0.75	111	0 (at harvest)	not analysed	-	-
			3.5	0.12 *	cannot be calculated	80
			3.5+3.0=6.5	0.11 *	cannot be calculated	78
			3.5+4.5=8.0	0.20 **	cannot be calculated	-
			3.5+12.5=16	0.19 **	cannot be calculated	77; 79

* quantified by method TRAM, which determined fluazifop-butyl and free fluazifop acid (II), but not fluazifop (II) conjugates

** quantified by method PPRAM 52, which determined fluazifop-butyl and free fluazifop acid (II), but not fluazifop (II) conjugates

Incurred residue study 2

Strawberries, green beans, cauliflower, oilseed rape and sugar beets were treated in the field with an EC formulation of fluazifop-butyl [Atreya and Froggatt, 1983, PP9/0039, report PP009B157; Atreya, 1990, PP5/0799, report M5163B summary]. The crops were stored at -20 ± 2 ºC and analysed at an unknown period after harvest. Samples were re-analysed at 3 monthly intervals after the first analysis. At each interval method recovery was verified for each sample type. Total fluazifop was determined using HPLC-UV method PPRAM 62. The analytical method is considered valid for the determination

of total fluazifop in oilseed rape seeds (1–2 mg/kg), but not at higher concentration levels. The method is considered insufficiently validated for quantification of total fluazifop in strawberries, green cauliflower, beans with pods, sugarbeet roots.

Residue concentrations were corrected for recovery; uncorrected results were not reported. The storage stability results are shown in Table 171.

Reviewer's conclusion: The information from this study cannot used to evaluate storage stability for the following reasons:

- Analytical method fitness for purpose is not shown for any of the commodities.
- The time between harvest and first analysis (zero storage time) is not provided (confirmed by the manufacturer). Since the rate of degradation is generally highest in the first 3 months after harvest and thereafter stabilizes, % remaining since harvest cannot be calculated.

Table 171 Storage stability of residues in various crops with incurred residues at -20 °C

commodity	Treatment (kg ai/ha) (fluazifop-butyl)	Harvest (DALT)	Storage time (months)	Total fluazifop (mg/kg)	% remaining [a]	concurrent recovery
strawberries sample 3762/81	1×1.0	7	unknown (zero time)	2.0 (n=1)	harvest to first analysis unknown	90, 95
			+3	2.7 (n=3)	cannot be calculated	82, 84
			+6	2.6 (n=3)	cannot be calculated	78, 79
			+9	2.2 (n=3)	cannot be calculated	87, 89
Green beans sample 5245/80	1×1.0	60	unknown (zero time)	1.0 (n=1)	harvest to first analysis unknown	91, 79
			+ 6	0.86 (n=3)	cannot be calculated	77, 79
			+ 9	0.95 (n=3)	cannot be calculated	100, 111
			+ 12	0.96 (n=3)	cannot be calculated	89
Cauliflower sample 5531/81	1 × 0.375	2 hours	unknown (zero time)	2.9 (n=1)	harvest to first analysis unknown	81, 98
			+3	3.7 (n=3)	cannot be calculated	83, 77
			+6	4.2 (n=3)	cannot be calculated	78, 78
			+9	4.6 (n=3)	cannot be calculated	71, 75
Oilseed rape seeds sample 2860/81	1 × 0.5+1×1.0	115	unknown (zero time)	4.5 (n=1)	harvest to first analysis unknown	89
			+3	5.0 (n=3)	cannot be calculated	79, 90
			+6	4.8 (n=3)	cannot be calculated	85, 86
			+9	5.5 (n=3)	cannot be calculated	77, 91
Sugarbeet roots sample 3816/81	1×1.5	65	unknown (zero time)	0.24 (n=1)	harvest to first analysis unknown	90, 95
			+3	0.19 (n=2)	cannot be calculated	71, 62
			+6	0.26 (n=3)	cannot be calculated	89, 87
			+9	0.25 (n=3)	cannot be calculated	81, 73
			+12	0.19 (n=3)	cannot be calculated	91, 94

[a] = calculated by the reviewer by dividing the mean concentration after storage by the concentration at harvest * 100%;

Residue values in the study report were reported as corrected for concurrent recovery; uncorrected results were not available.

Incurred residue study 3

Tomato plants were treated in the field with two different EC fluazifop-butyl formulations at a rate of 2 × 0.42 kg ai/ha with an interval of 15 days [Trumbo and Francis, 1986, PP9/0036, report TMU3079; Atreya, 1990, PP5/0799, M5163B summary]. Tomatoes were harvested 45 days after the last application in June 1984. The tomatoes from trial 71TX84-004 (samples 84-1582 and 84-1583) were stored at -20 ± 2 ºC and analysed in October 1984 for total fluazifop residues using HPLC-UV method PPRAM 62. Samples were re-analysed in June 1986 for total fluazifop residues using HPLC-UV method PPRAM 62/2. In method PPRAM 62/2 a second HPLC clean-up was used and analysis

without internal standard. Concurrent method recovery was reported to be 82% at 0.3 mg/kg fluazifop acid (II, free) for method PPRAM 62/2. The analytical method is considered insufficiently validated for quantification of total fluazifop in tomatoes. Storage stability results are presented in Table 172.

Reviewer's conclusion: The information from this study cannot used to evaluate storage stability for the following reasons:

- Analytical method fitness for purpose is not shown for tomatoes.
- Residues were not analysed at harvest. Since the rate of degradation is generally highest in the first 3 months after harvest and thereafter stabilizes, % remaining since harvest cannot be calculated.

Table 172 Storage stability of residues in tomatoes with incurred residues at -20 °C

commodity	Treatment (kg ai/ha) (fluazifop-butyl)	Harvest (DALT)	Storage time (days; months)	Total fluazifop (mg/kg)	% remaining	concurrent recovery
Tomato study 84-1582	2 * 0.42	45	0 (at harvest)	not analysed	-	-
			110 (4 months)	0.45 (n=1)	cannot be calculated	-
			744 (25 months)	0.52 (n=3)	cannot be calculated	-
Tomato study 84-1583	2 * 0.42	45	0 (at harvest)	not analysed	-	-
			+110 (4 months)	0.30 (n=1)	cannot be calculated	-
			+744 (25 months)	0.30 (n=3)	cannot be calculated	-

Incurred residue study 4

Peanut plants were treated in the field (USA, 1986) with fluazifop-P-butyl and frozen immediately after harvest [Hayward, 1988, 462746, report M4841B; Atreya, 1990, PP5/0799, M5163B summary]. The harvest date is not stated, but trials were carried out in 1986. Hulls were separated from the kernels. Duplicate samples of peanut kernels were stored at -18 ºC immediately after harvest and analysed for the first time in May 1987. Samples were re-analysed 16 months later in September 1988. Total fluazifop was determined using NMR method PPRAM 122. Storage stability results are presented in Table 173. Average concurrent method recovery was 92% at 0.4 mg/kg fluazifop-butyl.

Reviewer's conclusion: The information from this study cannot used to evaluate storage stability for the following reasons:

- Method PPRAM 122 is considered not valid for quantification fluazifop conjugates in any commodity (valid for fluazifop-butyl and fluazifop acid (II) at 0.4 mg/kg in peanut kernels)
- Residues were not analysed at harvest. Since the rate of degradation is generally highest in the first 3 months after harvest and thereafter stabilizes, % remaining since harvest cannot be calculated.

Table 173 Storage stability of residues in peanut kernels with incurred residues at -18 °C

commodity	Treatment (kg ai/ha) (fluazifop-butyl)	Harvest (DALT)	Storage time (months)	Total fluazifop (mg/kg)	% remaining	concurrent recovery
peanut kernel sample 1005/87	2 × 1.2	92	unknown (zero time)	0.23	harvest to first analysis unknown	96, 89
			+16	0.24, 0.27, mean 0.26	cannot be calculated	91
peanut kernel sample 1009/87	2 × 0.25	68	unknown (zero time)	0.23	harvest to first analysis unknown	96, 89
			+16	0.21, 0.22, mean 0.22	cannot be calculated	91

Incurred residue study 5

Celery plants were treated in the field with two different EC formulations of fluazifop-P-butyl at a rate of 2 × 0.42 kg ai/ha [Trumbo and Francis, 1986, PP9/0037, report TMU3074; Atreya, 1990, PP5/0799, report M5163B summary]. Celery was harvested 30 days after the last application, on 13 February 1984. Duplicate samples of celery were stored at -20 ºC. Samples were analysed for the first time on 12 June 1984, 4 months after harvest, for total fluazifop residues using HPLC-UV method PPRAM 62. Samples were re-analysed 24 months later, on 11 June 1986, for total fluazifop residues using HPLC-UV method PPRAM 62/2. Method recovery was verified. Storage stability results are presented in Table 174.

Reviewer's conclusion: The information from this study cannot used to evaluate storage stability for the following reasons:

- Method PPRAM 62 is considered not valid for quantification of fluazifop acid (II) (and total fluazifop) residues in celery.
- Residues were not analysed at harvest. Since the rate of degradation is generally highest in the first 3 months after harvest and thereafter stabilizes, % remaining since harvest cannot be calculated.

Table 174 Storage stability of residues in celery with incurred residues at -20 °C

commodity	Treatment (kg ai/ha) (fluazifop-butyl)	Harvest (DALT)	Storage time (months)	Total fluazifop (mg/kg) mean range RSD_r	% remaining	concurrent recovery
celery trial 75FL84-007 sample 84-5523	2 × 0.42 interval 53 days	30	0 (harvest)	not analysed	-	-
			4	0.37	cannot be calculated	87
			4 + 24 =28	0.38 0.35-0.42 7.9%	cannot be calculated	87
celery trial 75FL84-007 sample 84-5524	2 × 0.42 interval 53 days	30	0 (harvest)	not analysed		
			4	0.41	cannot be calculated	87
			4+ 24 =28	0.39 0.37-0.42 5.1%	cannot be calculated	87

ns = not stated

Incurred residue study 6

Storage stability was investigated in potato, lettuce, cabbage, dried bean, tomato and soya bean seeds with incurred fluazifop residues [McGill, 2003, PP5/1281, report RJ3087B]. The ramples were stored at -18 °C. Bulked samples of crops treated with fluazifop-P-butyl during supervised field trials were first analysed on 13–22 Sept 1999. Time between harvest and first analysis is not indicated in the study report. Samples were re-analysed after 3, 6, 9, 12 and 18 months of storage for residues of total fluazifop (i.e. sum of fluazifop-butyl, fluazifop acid (II) and it conjugates, expressed as fluazifop acid (II)) by HPLC-MS-MS method RAM 287/02. The method is considered valid for the determination of total fluazifop in dry soya bean seeds (0.05–5.0 mg/kg), potatoes (0.01–0.1 mg/kg), lettuce (0.1–0.5 mg/kg), head cabbage (0.05–2.0 mg/kg), tomatoes (0.01–2.0 mg/kg) and dry bean seeds (0.1–2.5 mg/kg).

Average concurrent recoveries ranged between 71–119% for each matrix. Control samples had residues < 0.01 mg/kg eq total fluazifop. The residue levels measured in samples are summarized in Table 175. Residue levels are reported as average residue, uncorrected for concurrent recoveries.

Reviewer's conclusion: The information from this study cannot be used to evaluate storage stability because the time between harvest and first analysis (zero storage time) is not provided. Based on the sampling information from the supervised residue trials samples were stored for 48–125 days before first analysis in the supervised residue trials. First analysis in the supervised residue trials was

carried out between 20 November 1997 and 4 March 1999, depending on the commodity. The experimental phase of the storage stability study was conducted between 1 September 1999 and 15 May 2001, indicating that the first sample analysis of the storage stability study was several months to years after harvest, depending on the commodity. Since the rate of degradation is generally highest in the first 3 months after harvest and thereafter stabilizes, % remaining since harvest cannot be calculated.

Table 175 Storage stability of residues in various crops with incurred residues at -18 °C

commodity	Treatment (kg ai/ha)	Storage time (months, days)	Total fluazifop (mg/kg) (n=2, mean)	% remaining [a]	concurrent recovery
soya bean seeds study 98JH055, trial IT20-98-H314 sample 3/0 [PP5/0159; RJ2781B]	1× 0.31 kg ai/ha; Harvest DAT 73 Supervised residue trial 2.7 mg/kg Sampled 22 Sept 1998 Extracted 4 Nov 1998 Analysed 9 Nov 1998 Stored 43+5= 48 days	0 (unknown zero time	2.8	cannot be calculated	-
		+ 3 (121 days)	2.8	idem	96
		+ 6 (200 days)	2.6	idem	100
		+ 9 (337 days)	2.6	idem	104
		+ 12 (392 days)	2.6	idem	112
		+ 18 (603 days)	2.6	idem	106
potato tubers study 98JH072 trial IT20-98-H326 sample 2/0, 5/0 [PP5/0119; RJ2757B]	1 × 0.31 kg ai/ha; Harvest DAT 42, 45 Supervised residue trial Mean 0.42 mg/kg (0.32, 0.52 mg/kg) Sampled 24-27 Jul 1998; Extracted 5 Nov 1998; Analysed 9 Nove 1998; Stored 104+4=108 days	0 (unknown zero time	0.53	cannot be calculated	-
		+ 3 (121 days)	0.48	idem	99
		+ 6 (181 days)	0.46	idem	107
		+ 9 (288 days)	0.42	idem	97
		+ 12 (393 days)	0.30	idem	84
		+ 18 (548 days)	0.48	idem	105
lettuce study 98JH069 trial S340.99 sample 2/0, 3/0 [PP5/0145; RJ2782B]	1× 0.31 kg ai/ha; Harvest DAT 31 Supervised residue trial mean 0.44 mg/kg (0.22, 0.66 mg/kg) Sampled 20 Nov 1998 Extracted 25 Jan 1999 Analysed 28 Jan 1999 Stored 66+3 = 69 days	0 (unknown zero time	0.37	cannot be calculated	-
		+3 (121 days)	0.35	idem	96
		+6 (181 days)	0.30	idem	87
		+9 (278 days)	0.29	idem	87
		+12 (475 days)	0.37	idem	108
		+18 (607 days)	0.40	idem	91
Savoy cabbage study 98JH133 trial S401.99, sample 2/0 and trial S641.99, sample 2/0 [PP5/0147; RJ2834B]	1× 0.38 kg ai/ha Harvest DAT 49 Supervised residue trial 0.85 mg/kg Sampled 30 Oct 1998 Extracted 3 Mar 1999 Analysed 4 Mar 1999 Stored 124+1 = 125 days	0 (unknown zero time	1.7	cannot be calculated	-
		+3 (121 days)	1.8	idem	113
		+6 (181 days)	1.6	idem	105
		+9 (327 days)	1.6	idem	101
		+12 (435 days)	1.8	idem	96
		+18 (561 days)	1.8	idem	102
tomatoes study 98JH052 trial IT30-98-H322 sample 3/0 [PP5/0177; RJ2780B]	1 × 0.31 kg ai/ha Harvest DAT 42 Supervised residue trial 0.25 mg/kg Sampled 3 Aug 1998 Extracted 12 Nov 1998 Analysed 19 Nov 1998 Stored 101+7=108 days	0 (unknown zero time	0.20	100	-
		+3 (121 days)	0.23	cannot be calculated	113
		+6 (181 days)	0.22	idem	104
		+9 (278 days)	0.20	idem	101
		+12 (425 days)	0.20	idem	93
		+18 (566 days)	0.24	idem	108
dry bean seeds study 97JH097 trial ES10-97-SH010, sample 2/0 and trial ES10-97-SH110, sample 2/0 [PP5/0153; RJ2610B]	1 × 0.32 kg ai/ha [SH010] DAT 66, 0.89 mg/kg 1 × 0.34 kg ai/ha [SH110] DAT 66, 1.6 mg/kg Sampled 1 Aug 1997 Extracted 12 Nov 1997 Analysed 20 Nov 1997 [SH010] 27 Nov 1997 [SH110] Stored 103+8 = 111 days Stored 103+15=118 days	0 (unknown zero time	2.7	cannot be calculated	-
		+3 (112 days)	2.7	idem	110
		+6 (191 days)	2.6	idem	105
		+9 (315 days)	2.3	idem	106
		+12 (385 days)	2.3	idem	99
		+18 (575 days)	2.7	idem	119

[a] = calculated by the reviewer by dividing the mean concentration after storage by the concentration at harvest * 100%;

Incurred residue study 7

Storage stability was investigated in bovine milk and bovine kidney with incurred fluazifop residues [Atreya *et al*, 1981, PP9/0182, report RJ0215B]. Milk and kidney from a cow fed with fluazifop-butyl were stored at -20 ± 2 °C. Milk samples were analysed for the first time after maximally 4 months of storage; kidney samples after maximally 4 months [Swain, 2009, PP9_50000, report T008915/08]. Milk only contained lipophilic fluazifop conjugates. Kidney only contained free fluazifop acid (II). Milk and kidney samples from this study were re-analysed after 3 and 6 weeks of storage. Residues of parent compound and free fluazifop acid (II) are analysed separately from lipophilic conjugates of fluazifop acid (II) in milk and kidney (each expressed as fluazifop) by HPLC-UV and GC-MS method PPRAM 61. The method is considered valid for the determination of free fluazifop acid (II) in cow kidney (0.01–0.1 mg/kg) and lipophilic fluazifop conjugates in cow milk (0.05–0.1 mg/kg).

Concurrent recoveries were not available. The residue levels measured in samples are summarized in Table 176.

Reviewer's conclusion: The information from this study cannot used to evaluate storage stability since the first analysis was not at collection of the samples, but after after maximally 4 months. Therefore, the stability during storage cannot be calculated in this study.

Table 176 Storage stability of residues in animal commodities with incurred residues at -20 °C

commodity; analyte	Treatment (fluazifop-butyl)	Storage time (weeks)	Analyte (as mg/kg fluazifop) mean	range	RSD	% remaining	concurrent recovery
bovine milk, day 23; sample 340/81 lipophilic fluazifop conjugates	12 ppm 29 days	0 (at collection)	Not analysed			-	-
		Max 4 months	0.15			Cannot be calculated	no data
		Max 4 months + 6 weeks	0.18	0.17-0.18	2.8%	Cannot be calculated	no data
bovine kidney; sample 452/81 fluazifop acid (II, free)	12 ppm; 29 days	0 (collection)	Not analysed				
		Max 4 months	0.12			Cannot be calculated	no data
		Max 4 months + 3 weeks	0.13	-	-	Cannot be calculated	no data

USE PATTERN

The systemic herbicide fluazifop-P-butyl is used for the post-emergence control of annual and perennial grass (graminaceous) weeds in a wide range of crops. It can be used at growth stage BBCH 12–14 of the weeds. Fluazifop-P-butyl belongs to the chemical group of aryloxyphenoxypropionate and is quickly absorbed through the leaf surface, hydrolysed to fluazifop-P (fluazifop-P acid, II) and translocated through the phloem and xylem, focusing on the growing points of grass weeds causing his death. Fluazifop-P-butyl can also be used as a desiccant in the culture of grass seeds [Jolly, 2014, PP5_50554, report IR-4 PR 09825] and as ripener of sugarcane thereby increasing its sucrose concentration significantly [Draetta, 2012, A12530B_10011, report M11029].

Fluazifop-P-butyl is a registered herbicide in several countries and the original registered labels in the original language as well as their English translation were submitted for a limited number

of countries: Brazil, USA, France, Belgium, Germany, the Netherlands, and the United Kingdom. Use patterns were submitted for an extensive list of crops. An overview is presented in Table 177.

Table 177 Registered pre-harvest uses of fluazifop-P-butyl

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
001 Citrus fruits							
Citrus fruit[x]	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Weed directed ground application, strip band or spot treatment, in the interspace and around base of tree	0.42 (tot. max 1.26/season)	0.11–0.90	1-3 [z] (21 days)	14
Citrus fruit	EU (FR)	EC, 125	Weed directed spray	0.250	0.063–0.250	1	21
Lemons and limes	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Mandarins	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Oranges	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Pummelos	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
002 Pome fruit							
Apple	EU (NL)	EC, 125	Weed directed spray	0.125-0.375	0.025-0.094	1	28
Apple	EU (BE)	EC, 125	Weed directed spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	28
Apple	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Pear	EU (NL)	EC, 125	Weed directed spray	0.125-0.375	0.025-0.094	1	28
Pear	EU (BE)	EC, 125	Weed directed spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	28
(Oriental) Pear	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Quinces	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
003 Stone fruit							
003A Cherries							
Cherries	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Ground application, strip band or spot treatment, in the interspace and around base of tree	0.14-0.42 (tot. max 1.26/season)	0.34-2.7	1-3 [z] (interval not reported)	14 [h]
Cherries	EU (NL)	EC, 125	Weed directed spray; in the interspace between trees	0.125-0.375	0.025-0.094	1	28
Cherries (sweet and sour)	EU (BE)	EC, 125	Weed directed spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2	28

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
Cherries	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
003B Plums							
Plums (and prunes)	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Ground application, strip band or spot treatment, in the interspace and around base of tree	0.14-0.42 (tot. max 1.26/season)	0.34-2.7	1-3 [z] (interval not reported)	14 [h]
Plums	EU (NL)	EC, 125	Weed directed spray; in the interspace between trees	0.125-0.375	0.025-0.094	1	28
Plums	EU (BE)	EC, 125	Weed directed spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2	28
Plums	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
003C Peaches							
Apricots	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Ground application, strip band or spot treatment, in the interspace and around base of tree	0.14-0.42 (tot. max 1.26/season)	0.34-2.7	1-3 [z] (interval not reported)	14 [h]
Apricot	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Nectarines	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Ground application, strip band or spot treatment, in the interspace and around base of tree	0.14-0.42 (tot. max 1.26/season)	0.34-2.7	1-3 [z] (interval not reported)	14 [h]
Nectarines	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Peaches	USA (as stone fruit)	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Ground application, strip band or spot treatment, in the interspace and around base of tree	0.14-0.42 (tot. max 1.26/season)	0.34-2.7	1-3 [z] (interval not reported)	14 [h]
Peaches	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
004 Berries and other small fruits							
004A Cane berries							
Raspberries	EU (UK)	EC, 125	Where possible weed directed spray, before bloom or after harvest	0.125-0.375	0.025-0.469	1	Growth stage driven
Raspberries+ blackberries	EU (NL)	EC, 125	weed directed spray; between bushes	0.125-0.375	0.025-0.094	1	45
Raspberries+ blackberries	EU (BE)	EC, 125	Unspecified spray, before bloom or after harvest.	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported)	Growth stage driven

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
						[y]	
Raspberries+ blackberries	EU (FR)	SC, 125	Unspecified spray	0.250	Not reported	1	42
004B Bush berries							
Airelles and Myrtillier: Vaccinium berries	EU (FR)	SC, 125	Unspecified spray	0.250	Not reported	1	42
(Black) Currants	EU (NL)	EC, 125	weed directed spray; between bushes	0.125-0.375	0.025-0.094	1	45
Blackcurrants	EU (UK)	EC, 125	Weed directed spray, before bloom or after harvest	0.125-0.375	0.025-0.469	1	Growth stage driven
Cassissier and groseillier: (black) currants	EU (FR)	SC, 125	Unspecified spray	0.250	Not reported	1	42
Gooseberries	EU (NL)	EC, 125	weed directed spray; between bushes	0.125-0.375	0.025-0.094	1	45
Gooseberries	EU (BE)	EC, 125	Unspecified spray, before bloom or after harvest.	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	Growth stage driven
Gooseberries	EU (UK)	EC, 125	Weed directed spray, before bloom or after harvest	0.125-0.375	0.025-0.469	1	Growth stage driven
Groseillier a macquereau: gooseberries	EU (FR)	SC, 125	Unspecified spray	0.250	Not reported	1	42
Rose hip	EU (FR)	SC, 125	Unspecified spray	0.250	Not reported	1	42
004C Large shrub/tree berries							
Elderberry	EU (FR)	SC, 125	Unspecified spray	0.250	Not reported	1	42
Mulberries	EU (FR)	SC, 125	Unspecified spray	0.250	Not reported	1	42
004D Small fruit vine climbing							
Grapes	EU (BE)	EC, 125	Unspecified spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	28
Grape (table- and wine)	EU (FR)	SC, 125	Unspecified spray	0.250	Not reported	1	28
Grapes	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Ground application, strip band or spot treatment, in the interspace and around base of tree	0.42 (tot. max 1.26/season)	0.056-0.90	1-3 [z] (14 days)	50
004E Low growing berries							
Cranberry	EU (FR)	SC, 125	Foliar spray	0.250	Not reported	1	42
Strawberries	EU (NL)	EC, 125	over-the-top-spray	0.125-0.375	0.025-0.094	1	42
Strawberries	EU (BE)	EC, 125	Foliar spray,	0.125-0.375	Not reported	1-2	Growth

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
			before bloom or after harvest	(tot. max 0.375/season)			stage driven
Strawberries	EU (FR)	SC, 125	Foliar spray	0.1875-0.375 (tot. max 0.375/season)	Not reported	1	42
Strawberries	EU (UK)	EC, 125	Foliar spray, before bloom or after harvest	0.125-0.375	0.025-0.469	1	Growth stage driven
005 Assorted tropical and sub-tropical fruits–edible peel							
Olives	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Fig	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
006 Assorted tropical and sub-tropical fruits–inedible peel–(subgroup B: large)							
Banana	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Ground application, strip band or spot treatment, in the interspace and around base of tree	0.42 (tot. max 1.26/season)	0.11–0.90	1-3 [z] (30 days)	0
Banana	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	0
009 Bulb vegetables							
009A Bulb onions							
Garlic	EU (BE)	EC, 125	Foliar spray	0.125-0.375 Max 0.375/season	Not reported	1-2 (interval not reported) [y]	28
Garlic	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	28
Onion, bulb	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Foliar spray/aerial spray	0.14-0.42 (tot. max 0.84/season)	0.22-1.8 (aerial 0.90-1.8)	1-2 (interval not reported)	45
Onion	Brazil	EW, 250	Foliar spray/aerial spray	0.125-0.250 (tot. Max 0.250 /season)	0.042–0.25 [a] 0.16-0.31 [b] 0.31–0.63 [c]	1-2 (5-10 days)	28
Onion	EU (NL)	EC, 125	Foliar spray, post emergence	0.125-0.375	0.025-0.094	1	28
Onion	EU (UK)	EC, 125	Foliar spray	0.125-0.375	0.025-0.469	1	28
Onion	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	28
Onion	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2	28
Shallot	EU (NL)	EC, 125	Foliar spray, post emergence	0.125-0.375	0.025-0.094	1	28
Shallot	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2	28
Shallot	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	42
009B Green onions							
Leek	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (max 0.375/season)	Not reported	1-2 (interval not reported) [y]	42
Leek	EU (FR)	SC, 125	Foliar spray	0.1875-0.375 (max	Not reported	1	42

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
				0.375/season)			
Onion, spring	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	42
010 Brassicas							
010A Head brassica's							
Cabbage, white head cabbage, head	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Brussels sprouts	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Cabbage, head	EU (BE)	EC, 125	Foliar spray, until BBCH 18	0.125-0.1875	Not reported	1	Growth stage driven
Cabbage	Brazil	EW, 250	Foliar spray/aerial spray	0.125-0.250 (tot. max 0.250 /season)	0.042–0.25 [a] 0.16-0.31 [b] 0.31–0.63 [c]	1-2 [d] (5-10 days)	28
010B Flowerhead brassicas							
Broccoli	Brazil	EW, 250	Foliar spray/aerial spray	0.125-0.250 (tot. max 0.250/season)	0.042–0.25 [a] 0.16-0.31 [b] 0.31–0.63 [c]	1-2 [d] (5-10 days)	28
Broccoli	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Cauliflower	Brazil	EW, 250	Foliar spray/aerial spray	0.125-0.250 (tot. max 0.250 /season)	0.042–0.25 [a] 0.16-0.31 [b] 0.31–0.63 [c]	1-2 [d] (5-10 days)	28
Cauliflower	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
011 Fruiting vegetales, cucurbits–subgroup A: edible peel							
Courgette (Summer squash)	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	28
Cucumber	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	28
Gherkin	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	28
012 Fruiting vegetables, other than cucurbits							
Aubergine (eggplant)	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	35
Tomato	EU (FR)	EC, 125	Foliar spray	0.1875-0.375	0.047-0.375	1	35
Peppers	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	35
Peppers (tabasco) (Louisiana only)	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Foliar spray/aerial spray	0.14-0.42 (tot. max 0.84/season)	0.22-1.8 (aerial 0.90-1.8)	1-2 (interval not reported)	45
Peppers (chili)	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	35
013 Leafy vegetables (including Brassica leafy vegetables)							
Chard	EU (BE)	EC, 125	Foliar spray	0.125-0.375	Not reported	1	21
Chard	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	28
Chervil	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	21
Chervil	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Chicory leaves	EU (BE)	EC, 125	Foliar spray	0.125-0.188	Not reported	1	42
Chicory leaves	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Cress varieties	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Dandelion	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Endive	EU (BE)	EC, 125	Foliar spray	0.125-0.188	Not reported	1	42
Kale	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Kale for	EU (UK)	EC, 125	Foliar spray	0.125-0.375	0.025-0.469	1	56

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
animal fodder							
Lamb's lettuce	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Lettuce	EU (BE)	EC, 125	Foliar spray	0.125-0.188	0.025-0.094	1 [x]	42
Lettuce	EU (FR)	SC, 125	Foliar spray	0.1875	0.0469-0.1875	1	42
Lettuce	Brazil	EW, 250	Foliar spray/aerial spray	0.125-0.250 (tot. Max 0.250 /season)	0.042–0.25[a] 0.16-0.31[b] 0.31–0.63[c]	1-2 (5-10 days)	28
Purslane	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	28
Rocket (Rucola)	EU (BE)	EC, 125	Foliar spray	0.125-0.188	Not reported	1	42
Rocket (Rucola)	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Spinach	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Spinach	EU (BE)	EC, 125	Foliar spray	0.125-0.375	Not reported	1 [x]	42
Spinach	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	28
014 Legume vegetables							
Beans (with pods)	EU (NL)	EC, 125	Foliar spray, before bloom	0.125-0.375	0.025-0.094	1	56
Beans (without pods)	EU (NL)	EC, 125	Foliar spray, before bloom	0.125-0.375	0.025-0.094	1	56
Peas (green with and without pods), peas for ensilage, green Phaseolus beans, green Vicia beans	EU (BE)	EC, 125	Foliar spray, 2-4 leaves growth stage, before bloom	0.125-0.375 (tot. max 0.375 /season)	Not reported	1-2 (interval not reported) [y]	28
Peas for ensilage	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	42
Peas (without pods)	EU (NL)	EC, 125	Foliar spray, before bloom	0.125-0.375	0.025-0.094	1	56
Peas (vining)	EU (UK)	EC, 125	Foliar spray, before visible flower bud stage	0.125-0.1875	0.025-0.23	1	Growth stage driven
015 Pulses							
Dry beans (do not apply to cow peas)	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Foliar spray	0.420 (tot. max 0.840/season)	0.11–0.90 (aerial 0.45-0.90)	1-2 (14 days)	60
Beans	Brazil	EW, 250	Foliar spray	0.125-0.250 (tot. max 0.250 /season)	0.042–0.25 [a] 0.16-0.31 [b] 0.31–0.63 [c]	1-2 (5-10 days)	60
Pulses (dry) (g)	EU (NL)	EC, 125	Foliar spray	0.125-0.375	0.025-0.094	1	90
Dry harvested peas	EU (BE)	EC, 125	Foliar spray, before bloom	0.125-0.375 (tot. max 0.375 /season)	Not reported	1-2	Growth stage driven
Dry harvested peas: peas for ensilage; field peas for protein production (winter/spring); lupin	EU (FR)	SC, 125	Foliar spray; before bud formation	0.1875-0.375	not reported	1	56
Lupins	EU (FR)	SC, 125	Foliar spray; before bud formation	0.1875-0.375	not reported	1	56

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
Field beans (i.e. dry *Vicia* spp)	EU (UK)	EC, 125	Foliar spray, before first visible flower buds	0.125-0.375	0.025-0.469	1	Growth stage driven
Field beans (i.e. dry *Vicia* spp)	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	56
Peas (dried)	EU (UK)	EC, 125	Foliar spray, before visible flower buds	0.125-0.1875	0.025-0.23	1	Growth stage driven
Soya bean	Brazil	SC, 125	Foliar spray/aerial spray	0.200-0.250	0.067-0.13 [a] 0.25-0.31 [b] 0.50-0.83 [c]	1	60
Soya bean	Brazil	EW, 250	Foliar spray/aerial spray	0.125-0.250 (tot. max 0.250 /season)	0.042–0.25 [a] 0.16-0.31 [b] 0.31–0.63 [c]	1-2 (5-10 days)	60
Soya bean	EU (FR)	SC, 125	Foliar spray	0.1875	not reported	1	90
Soya bean	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Foliar spray/aerial spray	0.53 /season max 0.42 prebloom (up to V5 growth stage) and max 0.11 bloom through post-bloom (R1 growth stage or later)	0.028-0.90 (aerial 0.11–0.90)	1-2	60
016 Root and tuber vegetables							
Beetroot	EU (BE)	EC, 125	Foliar spray, post emergence	0.125-0.375	0.025-0.094	1	56
Carrots	EU (BE)	EC, 125	Foliar spray, post emergence	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	56
Carrots	EU (UK)	EC, 125	Foliar spray, before bloom or after harvest	0.125-0.375	0.025-0.469	1	56
Carrots	EU (NL)	EC, 125	Foliar spray,	0.125-0.375	0.025-0.094	1	56
Carrots	EU (FR)	SC, 125	Foliar spray	0.1875-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported)	42
Carrots	Brazil	EW, 250	Foliar spray/aerial spray	0.125-0.250 (tot. max 0.250 /season)	0.042–0.25[a] 0.16-0.31[b] 0.31–0.63[c]	1-2 (5-10 days)	30
Carrots	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Foliar spray/aerial spray	0.14-0.42 (tot. max 0.84/season)	0.22-1.8 (aerial 0.90-1.8)	1-2 (interval not reported)	45
Celeriac	EU (BE)	EC, 125	Foliar spray, post emergence	0.125-0.375	Not reported	1	56
Celeriac	EU (NL)	EC, 125	Foliar spray	0.125-0.375	0.025-0.094	1	56
Cichory, roots	EU (BE)	EC, 125	Foliar spray, post emergence	0.125-0.375	Not reported	1	56
Chicory roots	EU (NL)	EC, 125	Foliar spray, post emergence	0.125-0.375	0.025-0.094	1	56

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
Horseradish	EU (FR)	SC, 125	Foliar spray	0.188	Not reported	1	42
Parsnip	EU (BE)	EC, 125	Foliar spray, post emergence	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	56
Parsnip	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Potato	Brazil	EW, 250	Foliar spray/aerial spray	0.12–0.25 (max 0.250 /season)	0.042–0.25[a] 0.16-0.31[b] 0.31–0.63[c]	1-2 (5-10 days)	28
Potato	EU (DE)	EC, 125	Foliar spray at BBCH 29 (max 40% of soil covered by potato plants)	0.25	0.063–0.13	1	90 (before tuber formation)
Potato	EU (NL)	EC, 125	Foliar spray, post emergence	0.125-0.250	0.025-0.063	1	75
Radish	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported)	56
Radish	EU (FR)	SC, 125	Foliar spray	0.1875-0.375 (tot. max 0.375/season)	Not reported	1	42
Red beet	EU (NL)	EC, 125	Foliar spray, post emergence	0.125-0.375	0.025-0.094	1	56
Red beet	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported)	56
Red beet	EU (FR)	SC, 125	Foliar spray	0.1875-0.375 (tot. max 0.375/season)	Not reported	1	56
Rettich (Japanese radish)	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	56
Salsify	EU (NL)	EC, 125	Foliar spray	0.125-0.375	0.025-0.094	1	56
Salsify	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375 /season)	Not reported	1-2 (interval not reported)	56
Salsify	EU (FR)	SC, 125	Foliar spray	0.1875-0.375 (tot. max 0.375/season)	Not reported	1	42
Sugar beet	EU (NL)	EC, 125	Foliar spray	0.125-0.375	0.025-0.094	1	56
Sugar beet	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375 /season)	Not reported	1-2 (interval not reported) [y]	56
Sugar beet	EU (FR)	EC, 125	Foliar spray	0.1875-0.375 (tot. max 0.375 /season)	Not reported	1	56
Sugar beet	EU (UK)	EC, 125	Foliar spray	0.38	0.19-0.47[e] 0.075-0.19[f]	1	56
Sugar beet	USA	EC, 250 +0.5-1% v/v COC or	Foliar spray	0.42 (tot. max 0.84/season)	0.22-1.8 (aerial 0.90- 1.8)	1-2 (14 days)	90

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
		0.25-1% v/v NIS					
Sweet potato (and yam)	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Foliar spray	0.21 (tot. max 0.84/season)	0.22-1.8 (aerial 0.90-1.8)	1-4 [z] (14 days)	14
Swede	EU (NL)	EC, 125	Foliar spray, post emergence	0.125-0.375	0.025-0.094	1	56
Swede	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	56
Swede	EU (FR)	SC, 125	Foliar spray	0.1875-0.375 (tot. max 0.375/season)	Not reported	1	42
Swede (stockfeed only)	EU (UK)	EC, 125	Foliar spray, before 50% ground cover	0.125-0.375	0.025-0.469	1	56
Turnip	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	56
Turnip	EU (FR)	SEC, 125	Foliar spray	0.1875-0.375 (tot. max 0.375/season)	Not reported	1	42
Turnip (stock feed only)	EU (UK)	EC, 125	Foliar spray, before 50% ground cover	0.125-0.375	0.025-0.469	1	56
Witloof roots, for sprout production	EU (NL)	EC, 125	Foliar spray, post emergence	0.125-0.375	0.025-0.094	1	56
Witloof roots, for sprout production	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (max 0.375/season)	Not reported	1-2 (interval not reported) [y]	56
Witloof roots, for sprout production	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	56
017 Stalk and stem vegetables							
Asparagus	EU (NL)	EC, 125	Foliar spray, after harvest, post emergence	0.125-0.375	0.025-0.094	1	Growth stage driven
Asparagus	EU (BE)	EC, 125	Foliar spray, after harvest	0.125-0.1875	Not reported	1	42
Asparagus	EU (FR)	SC, 125	Foliar spray	0.1875-0.375 (tot. max 0.375/season)	Not reported	1	42
Asparagus	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Foliar spray	0.21–0.42 (tot. max 0.84/season)	0.056-0.90 (aerial 0.22–0.45)	1-2 (14-21 days)	1
Cardoon	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Celery (stem)	EU (BE)	EC, 125	Foliar spray, post emergence	0.125-0.250	0.025-0.063	1	42
Celery (stem)	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
Rhubarb	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
021 Grasses for sugar or syrup production							
Sugar cane	Brazil	EW, 250	Foliar spray	0.025-0.075	0.0083–0.75 [a] 0.031–0.094 b] 0.063–0.25 [c]	1	35
022 Tree nuts							
Almond	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Chestnut	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Hazelnut	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21
Macadamia	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Weed directed ground application, strip band or spot treatment, in the interspace and around base of tree	0.14-0.42 (tot. max 0.84/season)	0.22-1.8	1-2 (interval not reported)	1 [h]
Macadamia	EU (FR)	SC, 125	Weed directed spray	0.250	Not reported	1	21 [g]
Pecans	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Weed directed ground application, strip band or spot treatment, in the interspace and around base of tree	0.14-0.42 (tot. max 1.26/season	0.34-2.7	1-3 [z] (interval not reported)	30 [h]
Walnuts	EU (FR)	SC, 125	Weed directed spray, around the base of the tree	0.125-0.250	Not reported	1	21
023 Oilseeds							
Cotton seed	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Foliar spray/aerial spray Do not apply after boll set	0.14-0.42 (tot. max 0.84/season)	0.22-1.8 (aerial 0.90-1.8)	1-2 (interval not reported)	90 [h]
Cotton seed	Brazil	EW, 250	Foliar spray/Aerial spraying	0.125-0.250 (tot. max 0.250 kg a.i./ha)	0.042–0.25 [a] 0.16-0.31 [b] 0.31–0.63 [c]	1-2 [d] (5-10 days)	60
Mustard seed	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	90
Linseed (flax seed)	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	90
Linseed (including flax)	EU (UK)	EC, 125	Foliar spray, before visible (flower) bud stage	0.125-0.1875	0.025-0.23	1	-
Oilseed, Rape	Brazil	EC, 125	Foliar spray	0.19	0.094-0.23 [e] 0.038-0.094 [f]	1	14
Oilseed, Rape	EU (NL)	EC, 125	Foliar spray, post emergence, before winter	0.125-0.375	0.025-0.094	1	-
Oilseed, Rape	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	0.047-0.375	1	90
Oilseed, Rape	EU (BE)	EC, 125	Foliar spray, post emergence, before winter, and 15 cm crop height	0.125-0.1875	Not reported	1	-

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
Oilseed, Rape (spring and winter)	EU (UK)	EC, 125	Foliar spray, from 1 true leave to before 5 true leaves or before visible (flower) bud stage	0.125-0.1875	0.025-0.23	1	-
Oilseed, Rape (spring and winter, industrial use)	EU (UK)	EC, 125	Foliar spray	0.125-0.1875	0.025-0.23	1	14
Peanuts	USA	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Foliar spray/aerial spray	0.14-0.42 (tot. max 0.84/season)	0.34-2.7 (aerial 1.3-2.7)	1-2 (14 days	40 [h]
Sunflower	Brazil	EW, 250	Foliar spray	0.125-0.250 (tot. max 0.250 kg a.i./ha)	0.042–0.25[a] 0.16-0.31[b] 0.31–0.63[c]	1-2[d] (5-10 days)	59
Sunflower	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	90
024 Seed for beverages and sweets							
Coffee	USA (Hawaii only)	EC, 250 +0.5-1% v/v COC or 0.25-1% v/v NIS	Weed directed ground application, strip band or spot treatment, in the interspace and around base of tree	0.28-0.42 (tot. max 0.84/season)	0.22-1.8	1-2 (interval not reported)	1
027 Herbs and spices							
Celery leaves	EU (FR)	EC, 125	Foliar spray	0.1875	Not reported	1	42
Celery leaves	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported)[y]	21
Fennel	EU (BE)	EC, 125	Foliar spray	0.125-0.1875	Not reported	1	42
Fresh herbs	EU (FR)	SC, 125	Foliar spray	0.1875-0.375	Not reported	1	28
Hops	EU (UK)	EC, 125	Foliar spray, before bloom or after harvest	0.125-0.375	0.025-0.469	1	Growth stage driven
Parsley	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	21
Parsley	EU (FR)	SC, 125	Foliar spray	0.1875	Not reported	1	42
051 Straw, fodder and forage of cereal grains and grasses, except grasses for sugar production							
Clover	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	28
Fescue (red and hard fescue)	EU (NL)	EC, 125	Foliar spray, post emergence	0.125-0.250	0.025-0.094	1	49
Lucerne (alfalfa)	EU (BE)	EC, 125	Foliar spray	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	28

Crop	Country	Form g ai/L g ai/kg	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (interval in days)	
052 Miscellaneous Fodder and Forage crops							
Fodder beet	EU (BE)	EC, 125	Foliar spray, post emergence	0.125-0.375 (tot. max 0.375/season)	Not reported	1-2 (interval not reported) [y]	56
Fodder beet	EU (UK)	EC, 125	Foliar spray	0.125-0.375	0.025-0.469	1	56

COC = Crop Oil Concentrate; NIS = non ionic surfactant

[z] The registered label is not clear on the number of application allowed. In general a total number of 2 applications are allowed. However to achieve the maximum seasonal rate for several commodities 3 (and for sweet potatoes even 4) applications are needed.

[x] Citrus fruit in this USA label includes calamondin, citrus citron, citrus hybrids, grapefruit, kumquat, lemon, lime, mandarine (tangerine), orange (all), pummelo, and satsuma mandarin.

[y] For a variety of crops the interval between multiple applications was not included in the label. A reregistration is currently (2016) running, restricting the GAP to 1 application only. According to the applicant, the interval used in agricultural practice is 1 week. Furthermore, it is noted that the applications for fruit trees and currants and berries are directed at the black strips beneath the trees and not directed at the fruits.

[a] Back-pack or tractor mounted boom: spray volume 200-300 L/ha (125 SC) and 100-300 L/ha (250 EW)

[b] CDA (controlled droplet application): spray volume 80 L/ha

[c] Aerial spray: spray volume 30-40 L/ha

[d] Either a single dose (usually up to 0.19 kg ai/ha) or a sequential application not exceeding the maximum dose for each culture and weed.

[e] light infestations (80 to 200 L/ha).

[f in dense crop and dense weed situations

[g] do not graze treated area

[h] do not graze animals in treated areas (apricots, cherries, nectarines, peaches, plums, prunes, cotton, macadamia, pecans); do not harvest for forage or hay (cotton), do not harvest immature growing peanut plants to livestock or harvest for livestock feed (peanuts); do not feed cover crops of treated macadamia groves (macadamia)

RESULTS OF SUPERVISED RESIDUE ON CROPS

The Meeting received information on supervised residue trials for the following crops for weed directed or foliar spray applications:

(Sub)group	Table	Commodity
Weed directed spray applications at the base of trees, shrubs and vines		
citrus fruits	178	grapefruits
	179	lemons
	180	limes
	181	oranges
pome fruits	182	apples
	183	pears
stone fruits	184	cherries
	185	plums
	186	peaches
small fruit vine climbing	187	grapes

(Sub)group	Table	Commodity
assorted tropical and sub-tropical fruits-edible peel	188	olives
assorted tropical and sub-tropical fruits-inedible peel	189	banana
	-	mango
	-	pineapple
tree nuts	190	almonds
	191	hazelnuts
	192	macadamia nuts
	193	pecans
	194	walnuts
Seed for beverages and sweets	195	coffee beans
<u>Broadcast or banded foliar applications or weed directed inter-row applications:</u>		
caneberries	196	blackberries
	197	raspberries
bushberries	198	bilberries
	-	blueberries
	199	currants
	200	gooseberries
low growing berries	201	strawberries
assorted tropical and sub-tropical fruits-inedible peel	-	pineapple
bulb vegetables	202	dry harvested bulb onions
	-	garlic
	-	green onions
	203	leeks
Brassica (cole or cabbage) vegetables	-	broccoli
	-	Brussels sprouts
	-	cauliflower
	204	head cabbages
fruiting vegetables -cucurbits	205	indoor cucumbers
	206	field cucumbers
	207	summer squash
	-	melons
fruiting vegetables other than cucurbits	-	chili peppers
	208	tomato
leafy vegetables	-	endive
	209	kale
	210	head lettuce

(Sub)group	Table	Commodity
	211	leaf lettuce
	212	cos lettuce
	-	spinach
	213	turnip greens
legume vegetables	214	green beans with pods
	215	green peas with pods
	216	green pea seeds
	-	green soya bean seeds
pulses	217	beans, dry (Phaseolus spp)
	218	broad beans, dry (Vicia spp)
	-	cowpeas, dry (Vigna spp)
	219	field peas, dry (Pisum spp)
	-	lupins, dry
	220	soya beans, dry
root and tuber vegetables	221	carrots
	222	celeriac
	-	manioc (cassava)
	224	potatoes
	225	radish
	226	sugar beets
	227	fodder beets
	228	swedes
	229	sweet potatoes
	230	turnips
stem vegetables	-	artichokes
	231	asparagus
	-	celery
	232	rhubarb
	233	witloof roots and sprouts
grasses for sugar or syrup production	234	sugar cane
Oilseeds	235	cotton seed
	-	linseed
	236	oilseed rape seeed
	-	peanuts
	237	sunflower seed
Herbs	-	parsley
Legume animal feeds	238	bean forage

(Sub)group	Table	Commodity
	239	phaseolus bean straw
	240	Vicia bean straw
	241	pea forage
	242	pea straw
	243	soya bean forage
	244	soya hay
	245	soya straw
	246	clover, trefoil, medic pasture
Miscellaneous fodder and forage crops	247	sugar beet tops
	248	fodderbeet tops
	249	swede tops
	250	oilseed rape forage
	-	sunflower forage
Fodder and forage of grasses	251	grass forage
	252	grass hay
Byproducts	253	almond hulls
	-	cabbage wrapper leaves
	254	cotton gin trash

Application rates, spray concentrations and residues have been rounded to two figures. Residue data are recorded unadjusted for percentage recoveries or for residue values in control samples unless otherwise stated. Unquantifiable residues are shown as below the reported LOQ (e.g. < 0.01 mg/kg). Where multiple analyses were conducted on a single sample, the average value is reported. Where multiple samples were taken from a single plot, the individual and average values are reported.Where results from separate plots with distinguishing characteristics such as different formulations, crop varieties or treatment schedules were reported, results are listed separately for each plot. Residues from the trials conducted according to the critical GAP, which has been used for the estimation of maximum residue levels, STMR and HR values are underlined.

Some of the trials were performed with the racemate fluazifop-butyl and some were performed with fluazifop-P-butyl (R-enantiomer), this is indicated by (rac) or (P) in the tables below. Analytical methods cannot discriminate between the R- and S-enantiomers of fluazifop-butyl and for this reason the residues resulting from the racemate can be used to evaluate residues resulting from fluazifop-P-butyl (R-enantiomer). The residues presented in the tables are given as total fluazifop. Total fluazifop represents the sum of fluazifop-butyl plus fluazifop acid (II) and its conjugates and is expressed as fluazifop acid (II).

The field and analytical data were generally sufficiently described, except when marked by "[QU]" in the tables below, indicating that sample sizes, storage conditions and analytical method performance were not described. Samples marked with "[RT]", "[GS]' or "[WC]" indicate that the samples were not of commercial standards, since the samples were rotten (RT) or the harvest was too early (GS) or the growing conditions of the plants were affected by the weather (WC). Sample sizes were in accordance with the FAO manual 2016 appendix V, except when marked by "[SS]" in the table. Since residues are measured as total fluazifop, storage conditions are not an issue, except when marked with "[ST]" indicating that samples were defrosted are kept at ambient temperature for a

considerable time. The analytical methods used are considered fit for purpose, except when marked by "[AM]", "[RV"] or "[SK"] indicating that no hydrolysis step was included in the analytical method (AM), no radiovalidation (RV) was available to confirm effective hydrolysis of the fluazifop (II) conjugates or no soaking (SK) step was used for dry pulses before extraction. When control samples contained residues above 25% of the residues in the treated sample, this is marked with "[CT]". Results marked with "[QU]", "[WC]", "[RT]", "[GS]", "[SS]", "[ST]", "[RV]", "[AM]" or "[SK]" are not selected for derivation of the MRL, if according to cGAP. Results marked with [LOQ = nn] need to be increased to the valid LOQ of the respective method

In the tables the following abbreviations were used:

- ns = not stated, not reported, not specified
- Form = formulations: (rac) is racemate; (P) = fluazifop-P-butyl; EC = emulsifiable concentrate; EW = oil in water emulsion; SL = soluble concentrate; ME = micro-emulsion; WG = water disperible granule; NIS = non ionic surfactant as adjuvant; COC = crop oil concentrate as adjuvant
- Soil types: C = clay (PT: argiloso), CL = clay loam, Csa = clay sand, CSi = clay silt, L=loam, LC = loamy clay, LSa = loamy sand, Sa = sand, SaC = sandy clay, SaCL = sandy clay loam (PT: argilo arenoso), SaL = Sandy Loam, SaLC = sandy loam clay, SaSi = sandy silt, SaSiL = sandy silt loam, Si = silt, SiC = silty clay, SiCL = silty clay loam, SiCSa = silty clay sand, SiL = Silty Loam, SiSaC = Silt sandy clay and in addition: CaC = calcareous clay, GrL = gravelly loa
- GS = growth stage at last application
- GSH = growth stage at harvest: CH = commercial harvest, MAT = mature, IMM = immature, RP = ripe fruits, BR = beginning of ripening, i.e. full grown fruits but not yet ripe or expressed as BBCH code
- DAT = days after last application

Weed directed spray applications at the base of trees shrubs and vines

Grapefruits

Two cGAPs for grapefruits are available:

- cGAP from the USA with 3 ×0.42 kg ai/ha with a PHI of 14 days for citrusfruits
- cGAP from France with 1 ×0.25 kg ai/ha with a PHI of 21 days for citrusfruits

Trials that could be matched to these cGAPs were summarized.

Table 178 lists trials conducted in the USA (1986–1987, 2000). A weed directed spray application with fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 178. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[LOQ = 0.05] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for NMR method PPRAM 83.

Table 178 Supervised field trials on grapefruits (whole fruit), treated with fluazifop-butyl at the base of the trees

GRAPE FRUITS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [reference]
Yuma,	EC	3	0.56	0.15	3-4 inch	ns	MAT	14	≤ 0.03	RR 89-051B;

GRAPE FRUITS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [reference]
AZ, USA, 1986, Grapefruit (Ruby Red)	120 (P) + 1% COC	(21, 21)	0.56 0.56	0.15 0.15	diameter, BBCH not reported; 22 Oct				[LOQ=0.05]	38AZ86-902R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.23 0.23 0.23	3-4 inch diameter, BBCH not reported; 22 Oct	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
Tulare, CA, USA, 1986, Grapefruit (variety ns)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	ns	4-6 in. diameter, BBCH not reported; 31 July	ns	MAT	14	<u>≤ 0.03</u> [LOQ=0.05]	RR 89-051B; US02-86-S10H; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	ns	4-6 in. diameter, BBCH not reported; 31 July	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
La Feria, Texas, USA, 1986-87, Grapefruit (Ruby Red)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	0.35 0.35 0.35	mature, BBCH not reported; 23 Jan 1987	ns	MAT	14	<u>≤ 0.03</u> [LOQ=0.05]	RR 89-051B; 60TX86-902R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.53 0.53 0.53	mature, BBCH not reported; 23 Jan 1987	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
Avon Park, FL, USA, 1986-87, Grapefruit (Ruby Red)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	0.19 0.19 0.19	4.3-5.1 inch diameter, BBCH not reported; 5 Jan 1987	ns	MAT	14	<u>≤ 0.03</u> [LOQ=0.05]	RR 89-051B; 75FL86-911R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.29 0.29 0.29	4.3-5.1 inch diameter, BBCH not reported; 5 Jan 1987	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
Ft. Pierce, FL, USA, 1986-87, Grapefruit (Pink)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	0.11 0.11 0.11	3.2-4 in. diameter, BBCH not reported; 5 Jan 1987	ns	MAT	14	<u>≤ 0.03</u> [LOQ=0.05]	RR 89-051B; 75FL86-912R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.17 0.17 0.17	3.2-4 inch diameter; BBCH not reported; 5 Jan 1987	ns	MAT	14	< 0.03 [LOQ=0.05]	idem

GRAPE FRUITS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [reference]
Mt. Dora, FL, USA, 2000, Grapefruit (Ruby Red)	EC 240 (P) +0.5% NIS	3 (19, 21)	0.42 0.43 0.43	0.81 0.18 0.18	GS not reported; 22 Oct	Sa	MAT	12	< 0.01, < 0.01 mean < 0.01	RR 00-063B; 202(CTFL2); [Stewart, 2001, 406466]

Additional trial information

RR-89-051B: GLP study. No unusual weather conditions were reported. Plots consisted of 3 trees, except trial US02-86-S10H (18 trees) and 75FL86-911R (12 trees). Application by boom sprayer. Spray volume not reported. Grapefruits (12 units/sample, 4.5-6.9 kg) were collected manually and randomly, taking care to avoid the plot boundaries (outer part of external trees). Storage at -18 °C for 510-686 days (18-25 months). Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent recovery (range 86-100%, mean 93%; fortification levels 0.05 and 0.1 mg/kg, n = 2/fortification level). Control samples were < 0.03 mg/kg.

RR-00-063B: GLP study. No unusual weather conditions were reported. Plot size was 610 ft2 with 250-425 ft2/tree, which indicates 1-2 trees/plot. Soil type reported was sand. Application by tractor mounted sprayer. Grapefruits (24 units/sample, 3.9-7.3 kg) were collected manually and randomly, taking care to avoid the plot boundaries (outer part of external trees). Storage at –19 to -17 °C for 48 days (1.5 months). Samples were analysed for total fluazifop using **GC-MS method RR91-014B with a valid LOQof 0.01 mg/kg**. Samples were not corrected for concurrent recovery. No concurrent recoveries were given for grapefruits. Control samples were < 0.01 mg/kg.

Lemons

Two cGAPs for lemons and limes are available:

- cGAP from USA with 3 × 0.42 kg ai/ha with a PHI of 14 days for citrusfruits
- cGAP from France with 1 × 0.25 kg ai/ha with a PHI of 21 days for citrusfruits

Trials that could be matched to these cGAPs were summarized.

Table 179 lists trials conducted in the USA (1986) and Southern France (1996). A weed directed spray application with fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 179. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[LOQ = 0.05] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for NMR method PPRAM 83.

Table 179 Supervised field trials on lemons (whole fruit), treated with fluazifop-butyl at the base of the trees

LEMONS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Yuma, AZ, USA, 1986, (Lisbon)	EC 120 (P) +1% COC	3 (21, 21)	0.56 0.56 0.56	ns	2 inch diameter, BBCH not reported; 18 Aug	ns	MAT	14	< 0.03; < 0.03 [a] [LOQ=0.05]	RR 89-051B; 38AZ86-900R and 901R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1%	3 (21, 21)	0.84 0.84 0.84	ns	2 inch diameter, BBCH not reported;	ns	MAT	14	< 0.03 < 0.03; [a]	idem

LEMONS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	COC				18 Aug				[LOQ=0.05]	
Bard, CA, USA, 1986, (Lisbon)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	ns	2 inch diameter, BBCH not reported; 30 Aug	ns	MAT	14	< 0.03 [LOQ=0.05]	RR 89-051B; 38CA86-904R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	ns	2 inch diameter, BBCH not reported; 30 Aug	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
Loxahatchee, FL,USA, 1986, (Bearrs)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	0.19 0.19 0.19	2.25-2.6 inch diameter, BBCH not reported; 28 Oct	ns	MAT	14	< 0.03 [LOQ=0.05]	RR 89-051B; 75FL86-910R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.29 0.29 0.29	2.25-2.6 inch diameter, BBCH not reported; 28 Oct	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
Visalia, CA, USA, 1986, (Lisbon)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	0.20 0.20 0.20	mature; BBCH not reported; 2 Dec	ns	MAT	14	< 0.03 [LOQ=0.05]	RR 89-051B; US02-S09H; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.30 0.30 0.30	mature; BBCH not reported; 2 Dec	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
Cagnes sur Mer; Alpes-Maritimes; S-France 1996, (Eureka)	EC 124 (P)	1	0.76	0.21	BBCH 89; 11 April	Si	MAT	7	< 0.01	RJ2241B; 96H ARSAP05; [Miles and Nassoy, 1997, PP5/0197]
Vallauris; Alpes-Maritimes; S-France 1996; (Eureka)	EC 124 (P)	1	0.67	0.18	BBCH 89; 11 April	Si	MAT	7	< 0.01	RJ2241B; 96H ARSAP05; [Miles and Nassoy, 1997, PP5/0197]

[a] Results came from two replicate plots; highest value is taken for MRL derivation, if according to cGAP

Additional trial information

RR 89-051B: GLP study. No unusual weather conditions were reported. Plots consisted of 3 trees, except trial U02S09H (6 trees). Application by boom sprayer. Spray volume not reported. Lemons (12 units/sample, > 2 kg) were collected manually and randomly, taking care to avoid the plot boundaries (outer part of external trees). Storage at -18 °C for 524-648 days (19-23 months). Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent recovery (range 88.180.9-100.9%, mean 93%; fortification levels 0.05 and 0.1 mg/kg, n = 2/fortification level. Control samples were < 0.03 mg/kg.

RJ2241B: GLP study. No unusual weather conditions were reported. Plots consisted of 4 trees. Soil type "limoneux" was translated as "silt'. Application by 3 m boom sprayer with a spray volume 362-363 L/ha. Lemons (12 units/sample) were collected manually and randomly, taking care to avoid the plot boundaries (outer part of external trees). Storage at -18 °C for 153 days (5 months). HPLC-MS/MS method **RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery (63%, 0.1 mg/kg, n = 2). Control samples were < 0.01 mg/kg.

Limes

Two cGAPs for lemons and limes are available:

- cGAP from USA with 3 ×0.42 kg ai/ha with a PHI of 14 days for citrusfruits
- cGAP from France (and it overseas areas like Martinique) with 1 ×0.25 kg ai/ha with a PHI of 21 days for citrusfruits

Trials that could be matched to these cGAPs were summarized.

Table 180 lists trials conducted in Martinique (1984). A weed directed spray application with fluazifop-butyl (racemate) was conducted at the base of the trees/shrubs under the conditions listed in Table 180. Results marked with "[QU]" are not selected for derivation of the MRL, if according to cGAP.

[QU] indicates that the quality of the study was very poor since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

Table 180 Supervised field trials on limes (whole fruit), treated with fluazifop-butyl at the base of the trees

LIMES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Irfa; Martinique, 1984, (Citrus latifolia)	EC 250 (rac)	1	0.50	ns	GS ns; 23 May	ns	ns	15 30	< 0.05 < 0.05 [QU]	RIC1933 trial ns [Culoto and Mallmann, 1985, PP9/0130]
Irfa; Martinique, 1984, (Citrus latifolia)	EC, 250 (rac)	1	1.0	ns	GS ns; 23 May	ns	ns	15 30	< 0.05 < 0.05 [QU]	idem

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

Additional trial information

RIC 1933, non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Storage at -18 °C for unknown period. Peel and flesh were analysed separately. Since each commodity was < 0.05 mg/kg, also the RAC is < 0.05 mg/kg. **HPLC-UV method PPRAM 62/1 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent recovery 75% (0.1–0.3 mg/kg). Control samples were < 0.05 mg/kg.

Oranges

Two cGAPs for oranges are available:

- cGAP from USA with 3 ×0.42 kg ai/ha with a PHI of 14 days for citrusfruits
- cGAP from France with 1 ×0.25 kg ai/ha with a PHI of 21 days for citrusfruits

Trials that could be matched to these cGAPs were summarized.

Table 181 lists trials conducted in the USA (1986-1987, 2000, 2011), Brazil (1981) and Italy (1986-1987). A weed directed spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 181. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[SS] indicates that the sample sizes were not reported or less than 12 fruits (or more to yield 2 kg).

[LOQ = 0.05] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for NMR method PPRAM 83.

Table 181 Supervised field trials on oranges (whole fruit), treated with fluazifop-butyl at the base of the trees

ORANGES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Lake Placid, FL, USA, 1986, (Hamilin)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	0.19 0.19 0.19	2.5- 2.8 inch diameter, 18 Nov	ns	MAT	14	< 0.03, < 0.03; mean < 0.03 [LOQ=0.05]	RR 89-051B; 75FL86-906R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.29 0.29 0.29	2.5-2.8 inch diameter, 18 Nov	ns	MAT	14	< 0.03, < 0.03; mean < 0.03 [LOQ=0.05]	idem
Avon Park, FL, USA, 1986/87, (Pineapple)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	0.19 0.19 0.19	2.7-3 inch diameter, 5 Jan 1987	ns	MAT	14	< 0.03, < 0.03 mean < 0.03 [LOQ=0.05]	RR 89-051B; 75FL86-907R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.29 0.29 0.29	2.7-3 inch diameter, 5 Jan 1987	ns	MAT	14	< 0.03, < 0.03 mean < 0.03 [LOQ=0.05]	idem
Merritt Island, FL, USA, 1986, (Naval)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	0.19 0.19 0.19	3-3.2 inch diameter, 19 Nov	ns	MAT	14	< 0.03 [LOQ=0.05]	RR 89-051B; 75FL86-908R; [Francis, 1989, PP5/0466]
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.29 0.29 0.29	3-3.2 inch diameter, 19 Nov	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
Wauchula, FL, USA, 1986, (Hamilin)	EC 120 (P) + 1% COC	3 (21, 21)	0.56 0.56 0.56	0.11 0.11 0.11	2.9-3.2 in. diameter, 11 Nov	ns	MAT	14	< 0.03 [LOQ=0.05]	RR 89-051B; 75FL86-929R; [Francis, 1989, PP5/0466] and RR 89-052B 75FL86-929R [Francis, 1989, PP5/0586].
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.17 0.17 0.17	2.9-3.2 in. diameter, 11 Nov	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
idem	EC 120 (P) + 1% COC	3 (21, 21)	4.2 4.2 4.2	0.85 0.85 0.85	2.9-3.2 in. diameter, 11 Nov	ns	MAT	14	< 0.03 [LOQ=0.05]	idem processing
Lake Placid, FL, USA, 1987, (Valencia)	EC 120 (P) + 1%	3 (21, 21)	0.56 0.56 0.56	0.19 0.19 0.19	2.6-3.3 inch diameter, 24 Mar	ns	MAT	14	< 0.03 [LOQ=0.05]	RR 89-051B; 75FL86-909R; [Francis, 1989, PP5/0466]

ORANGES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	COC									
idem	EC 120 (P) + 1% COC	3 (21, 21)	0.84 0.84 0.84	0.29 0.29 0.29	2.6-3.3 inch diameter, 24 Mar	ns	MAT	14	< 0.03 [LOQ=0.05]	idem
Mt. Dora, Florida, USA, 2000, (Hamilin)	EC 240 (P) +0.5% NIS	3 (19, 21)	0.42 0.43 0.42	0.18 0.18 0.18	GS ns; 22 Oct	Sa	MAT	12	< 0.01, < 0.01 mean < 0.01	RR 00-063B; 201 (CTFL1); [Stewart, 2001, 406466]
Oviedo, Florida, USA, 2000, (Navel)	EC 240 (P) +0.5% NIS	3 (19, 21)	0.41 0.41 0.42	0.18 0.18 0.18	GS ns; 13 Nov	Sa	MAT	14	< 0.01, < 0.01 mean < 0.01	RR 00-063B; 203(CTFL3); [Stewart, 2001, 406466]
Oviedo, Florida, USA 2000, (Hamlin)	EC 240 (P) +0.5% NIS	3 (21, 21)	0.41 0.42 0.42	0.18 0.18 0.19	GS ns, 13 Nov	Sa	MAT	14	< 0.01, < 0.01 mean < 0.01	RR 00-063B; 204(CTFL4); [Stewart, 2001, 406466]
Raymond-ville, Texas, USA, 2000, (N-33)	EC 240 (P) +0.5% NIS	3 (21, 21)	0.43 0.43 0.43	0.18 0.23 0.23	GS ns; 1 Nov	SaL	MAT	14	< 0.01, < 0.01 mean < 0.01	RR 00-063B; 205(CTTX1); [Stewart, 2001, 406466]
Sacaton, Arizona, USA, 2000 (Washington navel)	EC 240 (P) +1% COC	3 (21, 20)	0.41 0.41 0.41	0.21 0.23 0.23	GS ns; 31 Oct	SaL	MAT	13	< 0.01, < 0.01 mean < 0.01	RR 00-063B; 206(CTAZ1); [Stewart, 2001, 406466]
Minkler, California, USA (10), 2000, (TI Navel)	EC 240 (P) +0.5% NIS	3 (21, 21)	0.41 0.42 0.41	0.23 0.24 0.23	GS ns; 9 Nov	C	MAT	14	< 0.01, < 0.01 mean < 0.01	RR 00-063B; 207(CTCA1); [Stewart, 2001, 406466]
Sanger, California, USA, 2000, Beck)	EC 240 (P) +0.5% NIS	3 (21, 21)	0.42 0.42 0.42	0.23 0.23 0.22	GS ns; 1 Nov	SaL	MAT	14	< 0.01, < 0.01 mean < 0.01	RR 00-063B; 208(CTCA2); [Stewart, 2001, 406466]
Chuluota, FL, USA, 2011, (Hamilin)	EC 240 (P) + 0.75 % COC	3 (21, 21)	2.1 2.1 2.1	0.75 0.75 0.75	BBCH 81; 23 Oct	Sa	MAT	14	< 0.01, < 0.01, < 0.01 mean < 0.01	TK0058357, TK0058357-01 [Mazlo, 2013, A12460A_50019] (processing)
Porterville, CA, USA, 2011, (Valencia)	EC 240 (P) + 0.75 % COC	3 (21, 21)	2.1 2.1 2.1	0.88 0.85 0.77	BBCH 89; 3 Aug	SaCL	MAT	14	0.014, 0.016, 0.015; mean 0.015	TK0058357, TK0058357-02 [Mazlo, 2013, A12460A_50019] (processing)
Jaguariuna; Holamba Brazil, 1981; (Natal)	EC 250 (rac)	2 (ns)	1.0	0.33	GS ns; 16 Oct	ns	ns	7 14 28	< 0.05[a] < 0.05[a] < 0.05 [a] [SS]	PP009B117 1Ct/81 ou 6LS [Atreya and Harradine, 1981, PP9/0613] and RJ0291B summary

ORANGES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
										[Atreya and Harradine, 1982, PP9/0062] (see edible portion section)
idem	EC 250 (rac)	2 (ns)	2.0	0.67	GS ns 16 Oct	ns	ns	7 14 21 28	0.068 [a] < 0.05 [a] < 0.05 [a] < 0.05 [a] [SS]	idem (see edible portion section)
location ns Italy 1986/87 (Bionda)	EC 125 (P)	1	0.69	0.094	Pre-ripening; 27 Nov	ns	Ripening	20 40	< 0.01 < 0.01 [SS] [LOQ=0.05]	M4533B; 37-86-1; [O'Brien and Harradine, 1987, PP5/0191]
location ns Italy 1986/87 (Bionda)	EC 125 (P)	1	1.4	0.18	Pre-ripening; 27 Nov	ns	Ripening	20 40	< 0.01 < 0.01 [SS] [LOQ=0.05]	M4533B; 37-86-1; [O'Brien and Harradine, 1987, PP5/0191]

[SS] Sample size not stated; results are considered not representative for MRL derivation.

[a] Residues in the whole orange were calculated assuming 30% weight as peel and 70% weight as flesh

Additional trial information

RR-89-051B GLP. No unusual weather conditions were reported. Plots consisted of 3 trees, except trial 75FL86-907R (12 trees) and 75FL86-909R (4 trees). Application by boom sprayer. Spray volume not reported. Oranges (11.5-15.4 lbs = >5 kg) were collected manually and randomly, taking care to avoid the plot boundaries (outer part of external trees). Storage at -18 °C for 510-686 days (18-25 months). Samples were analysed for total fluazifop using **NMR method PPRAM 83 modification A with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent recovery (range 86-100%, mean 93%; fortification levels 0.05 and 0.1 mg/kg, n = 2/fortification level). Control samples were < 0.03 mg/kg.

RR-89-052B GLP. See RR-89-051B

RR 00-063B: GLP study. No unusual weather conditions were reported. Plot size was 768-3000 ft2 with 200-440 ft2/tree, which corresponds to 4 trees/plot except in trials 201 and 204 and 208 (3 trees) and 207 (2 trees). Application by tractor mounted sprayer. Oranges (24 units/sample, 5.4-7.9 kg) were collected manually and randomly, taking care to avoid the plot boundaries (outer part of external trees). Storage at -28 to -10 °C for 24-71 days (1-2.5 months). Samples were analysed for total fluazifop using **GC-MS method RR91-014B with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery. Concurrent recoveries for 0.01 and 1.0 mg/kg in oranges were established (overall mean recovery 74.9, range 65-85%, n = 2/fortification level). Control samples were < 0.01 mg/kg.

TK0058357: GLP study. No unusual weather conditions were reported. Plot size not stated. Soil directed broadcast spray with a spray on each side of the tree row.. Spray volume 10-40 GPA. Bulk samples of 189-215 kg were taken for processing. Sampling strategy not stated. Storage at -10 °C or lower for 6.9-9.7 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.01A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery (103-113% at 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg.

PP009/B117: non-GLP study. Weather conditions and application equipment not stated. Plot size or number of trees/plot not stated. Spray volume 300 L/ha. Sample sizes are not stated. The peel was removed from the orange and the flesh and peel were analysed separately. Residue levels in the whole orange were calculated from flesh and peel, assuming 30% weight as peel and 70% weight as flesh. Residues in the peel and the flesh are reported in the edible portion section. Storage for a maximum of 158 days at unstated conditions. Results are the average of four replicate analytical samples. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with in internal standard calibration and a valid LOQ of 0.05 mg/kg.** Individual internal standard recovery 71-81% at 0.2 mg/kg for peel or flesh. Control samples were < 0.05 mg/kg.

M4553B: non-GLP study. No unusual weather conditions were reported. Plot size 24 m2, which is considered to be 1 tree. Application by motorpump at the base of the trees with a spray volume 729-737 L/ha. Oranges were collected manually. Sample sizes are not stated. Storage at -18 °C for a maximum of 7 months. The peel was removed from the orange and the flesh and peel were analysed separately. Residue levels in the whole orange were calculated from flesh and peel. Residues in flesh and peel were < 0.01 mg/kg each. Samples were analysed for total fluazifop using **NMR method PPRAM 83**

with a valid LOQ of 0.05 mg/kg. An internal standard was used for calibration (mean internal standard recovery 85%). Control samples were < 0.01 mg/kg.

Apples

Two cGAPs for apples are available:

- cGAP from the Netherlands or Belgium with 1 ×0.38 kg ai/ha with PHI 28 days on apple, pear
- cGAP from France with 1 ×0.25 kg ai/ha with PHI 21 days on apple, pear, quince

Trials that could be matched to this GAP were summarized.

Table 182 lists trials conducted in the USA (1984, 1985, 1986), Germany (1981, 1982, 1991), France (1996, 2011) and Italy (2011). A weed directed spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 182. Results marked with "[QU]", "[SS]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 12 fruits (or more to yield 2 kg).

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62, 62/1 or 62/2 or NMR method PPRAM 83.

Additional trials from Australia (1980) were available on apples with an application of 1 × 0.50–3.0 kg ai/ha and harvest at 137 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from South Africa (1980) were available on apples with an application of 1 × 0.25–0.60 kg ai/ha and harvest at 119 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from Canada (1979, 1980) were available on apples with an application of 1 × 0.50–1.0 kg ai/ha and harvest at 149 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from France (1982) were available on apples with an application of 1 ×1.5 kg ai/ha and harvest at 118, 152, 159, 162 DAT and in decline trials at 0, 15, 30/33, 60/62 and 95/98/130 DAT [Culoto and Mallman, 1983, Report RIC2815]. These trials were not summarized, because they would not assist in MRL setting.

Table 182 Supervised field trials on apples (whole fruit), treated with fluazifop-butyl at the base of the trees

APPLES Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Blacksburg, VA, USA, 1984 (Rome Beauty and Red Delicious)	EC 125 (P) +1% COC	2 (19)	0.42 0.42	0.18 0.18	GS ns; 5 Sept	ns	MAT	13	< 0.03 [SS] [LOQ=0.05]	TMU3119/B; 15VA84-034 [Watford and Francis, 1986, 405749]
Wooster, OH, USA, 1984	EC 125 (P) +1%	2 (87)	0.42 0.42	0.22 0.22	GS ns; 28 Aug	ns	MAT	14	< 0.03 [LOQ=0.05]	TMU3119/B; 04OH84-037 [Watford and

APPLES Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(McIntosh)	COC									Francis, 1986, 405749]
Snelling, CA, USA, 1984; (Granny Smith)	EC 125 (P) + 0.25% NIS	2 (96)	0.42 0.42	0.18 0.18	GS ns; 21 Aug	ns	MAT	14	< 0.03 [LOQ=0.05]	TMU3119/B; 69CA84-025 [Watford and Francis, 1986, 405749]
Prosser, WA, USA, 1984 (variety ns)	EC 125 (P)	2 (ns)	0.42	ns	ns	ns	MAT	14	< 0.03 [LOQ=0.05]	TMU3119/B; 32WA84-031 [Watford and Francis, 1986, 405749]
Kearneysville, WV, USA, 1985; (Golden Delicious)	EC 125 (P) 0.25% NIS	2 (123)	0.42	0.15	GS ns 11 Oct	ns	MAT	14	< 0.03 [LOQ=0.05]	TMU3119/B; 15WV85-037 [Watford and Francis, 1986, 405749]
Haslett, MI, USA, 1985 (Ida Red)	EC 125 (P)	2 (75)	0.42	0.12	GS ns; 22 Aug	ns	MAT	14	< 0.03 [LOQ=0.05]	TMU3119/B; 49MI85-020 [Watford and Francis, 1986, 405749]
Sunnyside, WA, USA, 1986 (Bisbee Red Delicious)	EC 125 (P) +0.25% NIS	2 (125)	0.42	0.11	3.5-4.0 inch fruit; 2 Sept	ns	MAT	14	< 0.02 [LOQ=0.05]	TMU3291/B; 32WA86-918R; [Watford and Francis, 1987, 405746]
idem	EC 125 (P) + 0.25% NIS	3 (9, 116)	0.42	0.11	3.5-4.0 inch fruit; 2 Sept	ns	MAT	14	< 0.02 [LOQ=0.05]	idem
Placerville, CA, USA, 1986 (Golden Delicious)	EC 125 (P) +0.25% NIS	2 (100)	0.42	0.18	fruit 6 cm diameter; 6 Aug	ns	MAT	14	< 0.02 [LOQ=0.05]	TMU3291/B; 69CA86-909R; [Watford and Francis, 1987, 405746]
idem	EC 125 (P) +0.25% NIS	3 (19, 81)	0.42	0.18	fruit 6 cm diameter; 6 Aug	ns	MAT	14	< 0.02 [LOQ=0.05]	idem
Williamson, NY, USA, 1986 (McIntosh)	EC 125 (P)	2 (95)	0.42	0.14	fruit 2.5 inch diameter; 29 Aug	ns	MAT	14	< 0.02 [LOQ=0.05]	TMU3291/B; 34NY86-909R; [Watford and Francis, 1987, 405746]
idem	EC 125 (P)	3 (14, 81)	0.42	0.14	fruit 2.5 inch diameter; 29 Aug	ns	MAT	14	< 0.02 [LOQ=0.05]	idem
idem	EC 125 (P)	3 (14, 81)	2.1	0.14	fruit 2.5 inch diameter; 29 Aug	ns	MAT	14	< 0.02 [LOQ=0.05]	idem
2059 Wotersen Roseburg, Germany;	EC 250 (rac)	1	1.0	0.1	GS ns; 8 Sept	ns	ns	0 7 14 21	0.07 < 0.03 < 0.03 < 0.03	PP009B120, 8104-RS-II-4 [Atreya *et al*, 1982, PP9/0433]

APPLES Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
1981; (Golden Delicious)								28	< 0.03 [QU] [CT] [cntrl=0.04]	and RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
6749 Gleiszellen-Gleishorbach Germany; 1981; (Boskoop)	EC 250 (rac)	1	1.0	0.1	GS ns; 4 Sept	ns	ns	7 14 21 28	< 0.05 < 0.05 < 0.05 < 0.05 [QU]	PP009B120, 8164-RS-VI [Atreya, 1982, PP9/0433] and RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
6741 Winden, Germany; 1982; (Goldrenette Freiherr)	EC 250 (rac)	1	1.0	0.1	BBA 27-31; 28 August	ns	ns	0 7 14 21 28	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04 [QU] [LOQ=0.05]	PP009B167 RS8277E1 [Atreya *et al*, 1982, PP9/0432]
2059 Wotersen Roseburg Germany; 1982; (Holstein Cox)	EC 250 (rac)	1	1.0	0.1	GS ns; 1 Sept	ns	ns	6 13 20 27	< 0.05 < 0.05 < 0.05 < 0.05 [QU]	PP009B167 RS8277B1 [Atreya *et al*., 1982, PP9/0432]
Location ns; Germany, 1991; (Idared)	EC 125 (P)	1	0.50	ns	GS ns; 19 Aug	ns	ns	29	< 0.02 [QU] [LOQ=0.05]	AZ84661A/91; 91JH069E1 [Gardyan, 1992, PP5/0183]
Location ns; Germany 1991; (Cox Orange)	EC 125 (P)	1	0.50	ns	GS ns; 23 Aug	ns	ns	25	< 0.02 [QU] [LOQ=0.05]	AZ84661A/91; 91JH069G1 [Gardyan, 1992, PP5/0183]
Grossoeuvre; Normandy, N-FR, 1996, (Belle des Reinettes)	EC 125 (P)	1	0.96	0.32	near maturity; 10 Sept	Si	MAT	7	< 0.01 [CT] [cntrl=0.01]	RJ2319B, S218.96 [Jones *et al*., 1998, PP5/0198]
Villers, Coterets; Picardy, N-FR, 1996; (Braeburn)	EC 125 (P)	1	0.75	0.25	maturity; 11 Oct	C	MAT	7	< 0.01	RJ2319B, S406.96 [Jones *et al*., 1998; PP5/0198]
Grenade; Haute Garonne; Midi-Pyrenees; S-France; 2011; (Golden)	EC 125 (P)	1	0.38	0.12	BBCH 77; 22 July	L	89	28	<u>< 0.01</u>	CEMR-4968; SRFR11-005-37HR; [Devine, 2012, A12791B_10841]
Verzuolo; Italy; 2011; (Itred)	EC 125 (P)	1	0.39	0.12	BBCH 83; 17 Aug	ns	89	28	<u>< 0.01</u>	CEMR-4968; SRIT11-1058-37HR; [Devine, 2012, A12791B_10841]

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported.

[SS] Sample size not stated or less than the required 2 kg and/or 12 units; sample considered not representative for MRL setting

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

Additional trial information

TMU3119/B: GLP. Weather conditions, spray equipment not stated. Application to the ground. Plot size not stated (VA, MI, WA), except OH (12 trees, 1000 ft2/80ft2), CA (1 tree, 144 ft2/216 ft2), and WV (1 tree). Spray volume 20-36 GPA (190-336 L/ha). Mature apples (>5 lbs = 2.2 kg) were collected) were sampled. Storage at -20 °C for a maximum of 21 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Residues were not corrected for average concurrent recoveries (96% at 0.05 and 0.1 mg/kg). Control samples were < 0.03 mg/kg.

TMU3291/B: GLP. No unusual weather conditions. Back pack sprayer with 25-41 GPA (234-383 L/ha). Application to the ground. Plot size 3 trees/plot (WA, NY), 5 trees/plot (CA). Mature apples (>5 lbs = 2.2 kg) were collected. Storage at -20 °C for a maximum of 5 months. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Residues were not corrected for individual concurrent recoveries (89% at 0.05 mg/kg). Control samples were < 0.02 mg/kg.

PP009/B120. Non-GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume 1000 L/ha. Storage conditions not stated (maximum 251 days). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. An internal standard was used in trial RS-II-4, but not in trial RS-VI. Concurrent method recovery was 93% at 0.1 mg/kg (n = 1). Control samples were not reported or were 0.04 mg/kg (8104-RS-II-4).

PP009/B167 Non-GLP Study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported Spray volume 1000 L/ha. Storage conditions not stated (maximum 92 days). Samples were analysed for total fluazifop using HPLC-UV method **PPRAM 62/1 with a valid LOQ of 0.05 mg/kg**. Average concurrent internal standard recoveries were 76% at 0.5 mg/kg (n = 13) and control samples were < 0.04 mg/kg or < 0.05 mg/kg.

AZ84661A/91GLP. A field report was not available (weather, equipment, soil type, plot size, sample sizes, growth stage at harvest were not reported). Storage at -20 °C (storage time not stated). Samples were analysed for total fluazifop using HPLC-UV method **PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. A second HPLC clean-up was used and analysis without internal standard. Samples were not corrected for indivual concurrent method recoveries (102-110% at 0.02 and 0.20 mg/kg). Control samples were < 0.02 mg/kg.

RJ2319B GLP. No unusual weather conditions. Plot size 4-8 trees (10-20 m with 2 apple trees/5 m) Application by hand held small boom connected to a gas pressurised knapsack sprayer. Spray volume 300 L/ha. Mature apples (5.7 kg fruit or at least 24 apples) were sampled by hand taking care to avoid plot boundaries. Storage time 87-115 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Residues were not corrected for average concurrent recoveries (94% at 0.1 mg/kg). Control samples were < 0.01 mg/kg fluazifop, except 0.01 mg/kg in trial S218.96.

CEMR-4968: GLP. No unusual weather conditions. Plot size 6-10 trees. Application by back pack sprayer (France) or boom sprayer (Italy). Spray volume 308-314 L/ha. Mature apples (> 2.0 kg fruit, equivalent to 12-14 units) were sampled by hand. Storage time up to 138 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Residues were not corrected for average concurrent recoveries (96-113% at 0.01 and 0.2 mg/kg). Control samples were < 0.01 mg/kg fluazifop.

Pears

Two cGAPs for pears are available:

- cGAP from the Netherlands or Belgium with 1 ×0.38 kg ai/ha with PHI 28 days on apple, pear
- cGAP from France with 1 ×0.25 kg ai/ha with PHI 21 days on apple, pear, quince

Trials that could be matched to this GAP were summarized.

Table 183 lists trials conducted in Germany (1981, 1982). A weed directed spray application with fluazifop-butyl (racemate) was conducted at the base of the trees under the conditions listed in Table 183. Results marked with “[QU]” are not selected for derivation of the MRL, if according to cGAP. Results marked with “LOQ = nn”, need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/1.

Additional trials from Canada (1979) were available on pears with 1 × 0.50–1.0 kg ai/ha and harvest at 135 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from France (1982) were available on pears with an application of 1 ×1.5 kg ai/ha and harvest at 93, 101, 127 DAT and in decline trials at 0, 15, 30, 60 and 64 DAT [Culoto and Mallman, 1983, Report RIC2815]. These trials were not summarized, because they would not assist in MRL setting.

Table 183 Supervised field trials on pears (whole fruit), treated with fluazifop-butyl at the base of the trees

PEARS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
2059 Wotersen Roseburg Germany; 1981; (Huise)	EC 250 (rac)	1	1.0	0.10	(ns); 1 Sept	ns	ns	0 7 14 21 28	< 0.03 0.05 < 0.03 < 0.03 0.03 [QU] [LOQ=0.05]	PP009B127; 8164 RS-II-3 [Atreya *et al*, 1982, PP9/0434] and and RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
6740 Landau Godramstein Germany; 1981; (Williams Christ)	EC 250 (rac)	1	1.0	0.10	(ns); 18 July	ns	ns	0 6 13 20 27 34	< 0.03 < 0.03 0.07 < 0.03 < 0.03 < 0.03 [QU] [LOQ=0.05]	PP009B127; 8164 RS-V-2 [Atreya *et al*., 1982, PP9/0434] and and RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
2152 Neuenkirchen Horneburg Germany; 1982; (Bürgermeister)	EC 250 (rac)	1	1.0	0.10	(ns); 1 Sept	ns	ns	0 7 14 21 28	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04 [QU] [LOQ=0.05]	PP009B163; RS8277B2 [Atreya 1982, PP9/0435]
6741 Winden Germany; 1982; ns; (Conference)	EC 250 (rac)	1	1.0	0.10	BBA 27-31; 23 August	ns	ns	0 7 14 21 28	< 0.04 0.05 < 0.04 < 0.04 < 0.04 [QU] [LOQ=0.05]	PP009B163; RS8277E2 [Atreya, 1982, PP9/0435]

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported.

Additional trial information

PP009/B127. Non-GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Spray volume 1000 L/ha. Storage conditions not stated (maximum 264 days). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. An internal standard was used for calibration. Information on concurrent method recovery is not reported. Control samples were < 0.03 mg/kg.

PP009/B163 Non-GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume 1000 L/ha. Storage conditions not stated (maximum 88 days). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/1 with a valid LOQ of 0.05 mg/kg**. Average concurrent internal standard recoveries were 84% at 0.5 mg/kg (n = 14). Control samples were < 0.04 mg/kg.

Cherries

Three cGAPs for cherries are available:

- cGAP from the USA with 3 ×0.42 kg ai/ha with PHI 14 days on cherries, plums, apricots, nectarines and peaches
- cGAP from the Netherlands or Belgium with 1 ×0.38 kg ai/ha with PHI 28 days on cherries, plums, peaches
- cGAP from France with 1 ×0.25 kg ai/ha with PHI 21 days on cherries, plums, apricots, nectarines and peaches

Trials that could be matched to this GAP were summarized.

Table 184 lists trials conducted in the USA (1986) and Germany (1981, 1991). A weed directed spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 184. Results marked with "[QU]" and/or "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2.

As the cherries from the trials in the USA and Germany were pitted before analysis, the residue data do not represent the RAC and would generally not be considered suitable for MRL derivation. Though no information on the weight fractions of stones and flesh is given either, this is not considered to affect the result, since residues were below LOQ.

Table 184 Supervised field trials on cherries (flesh only), treated with fluazifop-butyl at the base of the trees

CHERRIES Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Prosser, WA, USA, 1986 (Bing)	EC 120 (P) + 0.25% NIS	3; (9, 25)	0.42 0.42 0.42	0.11	0.75 inch fruit; 23 June	ns	MAT	14	<u>< 0.03</u> [LOQ=0.05]	TMU3181/B 32WA86-909R [Watford andFrancis, 1987, PP5/0468]
Williamson, NY, USA, 1986 (Montmorency)	EC 120 (P) + 1% COC	3; (14, 14)	0.42 0.42 0.42	0.14	2 cm fruit; 03 June	ns	MAT	14	<u>< 0.03</u> [LOQ=0.05]	TMU3181/B 34NY86-902R [Watford andFrancis, 1987, PP5/0468]
Brentwood,	EC	3;	0.42	0.18	1–2 cm fruit,	ns	MAT	15	<u>< 0.03</u>	TMU3181/B

CHERRIES Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
CA, USA, 1986, (Bing–tart cherry)	120 (P) + 0.25% NIS	(14, 5)	0.42 0.42		beginning to turn red; 22 April				[LOQ=0.05]	69CA86-903R [Watford andFrancis, 1987, PP5/0468]
Orem, UT, USA, 1986 (Bing)	EC 120 (P) + 0.25% NIS	3; (19, 7)	0.42 0.42 0.42	0.15	2–2.5 cm fruit, 10 June	ns	MAT	14	< 0.03 [LOQ=0.05]	TMU3181/B 69UT86-904R [Watford andFrancis, 1987, PP5/0468]
Haslett, MI, USA, 1986 (Napoleon)	EC 120 (P) + 1% COC	3; (14, 25)	0.42 0.42 0.42	0.13	colour developing, 16 June	ns	MAT	14	< 0.03 [SS] [LOQ=0.05]	TMU3181/B 71MI86-904R [Watford andFrancis, 1987, PP5/0468]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	0 7 14 21 30	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany; 1991; (ns)	EC 125 (P)	1	0.5	ns	GS ns; 12 June	ns	ns	36	< 0.02 [QU] [LOQ=0.05]	AZ83558/91; 91JH070E1 [Gardyan, 1992, PP5/0192] (processing)

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

Additional trial information

TMU3181B. GLP study. Soil type were not reported. Spray volume not stated, but all spray applications were directed at the base of the trunk without contact to the trunk. Plots consisted of 3 trees/plot. Growth stage at harvest not stated, but all stated to be marketable. Sample sizes 2.5-5.0 lbs = >1.1 kg, except in MI where 2.0 lbs=0.90 kg was sampled. Cherries, not washed, but pitted and ground. Storage at -20 °C and analysed within 7 months. Samples were analysed for total fluazifop using **HPLC-UV PPRAM 62/2 with minor modifications with a valid LOQ of 0.05 mg/kg**. Internal standard were used (0.1 mg/kg), with a mean recovery of 124% ± 5.3%. Control samples fortified with 0.05 mg/kg resulted in a recovery of 78% ± 6%. Samples were not corrected for indivual concurrent method recoveries. Control samples were < 0.03 mg/kg.

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.05 mg/kg.

AZ83558/91. GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. Cherries were washed and pitted. Storage at -20 °C (storage time not stated). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. No internal standard was used. Samples were not corrected for indivual concurrent method recoveries (86-116% at 0.02 and 0.20 mg/kg). Control samples were

Plums

Three cGAP for plums are available:

- cGAP from the USA with 3 ×0.42 kg ai/ha with PHI 14 days on cherries, plums, apricots, nectarines and peaches
- cGAP from the Netherlands or Belgium with 1 ×0.38 kg ai/ha with PHI 28 days on cherries, plums, peaches

- cGAP from France with 1 ×0.25 kg ai/ha with PHI 21 days on cherries, plums, apricots, nectarines and peaches

Trials that could be matched to these cGAPs were summarized.

Table 185 lists trials conducted in the USA (1986) and Germany (1981, 1991). A weed directed spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 185. Results marked with "[QU]" or "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 12 fruits (or more to yield 2 kg).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2 or NMR method PPRAM 83.

As the plums from the trials in the USA and Germany were pitted before analysis, the residue data do not represent the RAC and would generally not be considered suitable for MRL derivation. Though no information on the weight fractions of stones and flesh is given either, this is not considered to affect the result, since residues were below LOQ.

Table 185 Supervised field trials on plums (flesh only), treated with fluazifop-butyl at the base of the trees

PLUMS Country; year; location; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Hillsboro, OR, USA, 1986 (Italian Prune)	EC 120 (P) +0.25% NIS	3; (14, 119)	0.42 0.42 0.42	0.15 0.15 0.15	Purple/firm; 18 August	ns	MAT	15	< 0.02 [SS] [LOQ=0.05]	TMU3311/B 320R86-911R [Watford and Francis, 1987, PP5/0480]
Traver, CA, USA, 1986 (Black Beauty)	EC 120 (P) + 1% COC	3; (28, 12)	0.42 0.42 0.42	ns	GS ns; 14 May	ns	MAT	14	< 0.02 [LOQ=0.05]	TMU3311/B 45CA86-906R [Watford and Francis, 1987, PP5/0480]
Haslett, MI, USA, 1986 (Stanley)	EC 120 (P) + 1% COC	3; (14, 59)	0.42 0.42 0.42	0.13 0.13 0.13	Fruit colouring; 16 August	ns	MAT	14	< 0.02 [LOQ=0.05]	TMU3311/B 71MI86-905R [Watford and Francis, 1987, PP5/0480]
Sunnyside, WA, USA, 1986 (Italian Prune)	EC 120 (P) +0.25% NIS	3; (12, 76)	0.42 0.42 0.42	0.11 0.11 0.11	1.25–1.5 inch fruit, 5 August	ns	MAT	14	< 0.02 [LOQ=0.05]	TMU3311/B 32WA86-910R [Watford and Francis, 1987, PP5/0480] (processing)
Exeter, CA, USA, 1986 (French)	EC 120 (P) + 1% COC	3; (14, 34)	0.42 0.42 0.42	0.20 0.20 0.20	Fruit 8-10 cm, 21 July	ns	MAT	14	< 0.02 [LOQ=0.05]	TMU3311/B 45CA86-910R [Watford and Francis, 1987, PP5/0480]
idem	EC 120 (P) + 1% COC	3; (14, 34)	2.1 2.1 2.1	1.1 1.1 1.1	Fruit 8-10 cm, 21 July	ns	MAT	14	< 0.02 [LOQ=0.05]	idem (processing)
Location ns; Germany, 1981	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	0 7 14	0.04 < 0.03 < 0.03	RJ0291B summary [Atreya and

PLUMS Country; year; location; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(yellow plums)								21 28	< 0.03 < 0.03 [QU] [LOQ=0.05]	Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (yellow plums)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	0 6 13 20 28	< 0.03 < 0.03 < 0.03 < 0.03 < 0.03 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany; 1991; ns; (ns)	EC 125 (P)	1	0.5	ns	GS ns; 17 July	ns	ns	27	< 0.02 [QU] [LOQ=0.05]	AZ83558/91; 91JH070E2; (Gardyan, 1992, PP5/0192] (processing)

[SS] Sample size not stated or less than the required 2 kg and/or 12 units; sample considered not representative for MRL setting.

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

Additional trial information

TMU3311/B. GLP study. Weather, spray equipment and soil type were not reported. Spray volume not stated, but all spray applications were directed at the base of the trunk without contact to the trunk. Plots consisted of 4-8 trees, except MI-905R and CA-910R 3 trees/plot. Growth stage at harvest not stated. Sample sizes 5.0-13.0 lbs = >2.3 kg, except in OR not stated. Plums, not washed, but pitted and ground. Storage at -20 °C and analysed within 8 months. Samples were analysed for **total fluazifop using NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg.** Internal standard wer used (0.1 mg/kg), with a mean recovery of 124% ± 5.3%. Control samples fortified with 0.05 mg/kg resulted in a recovery of 124 % ± 10%. Samples were not corrected for indivual concurrent method recoveries. Control samples were < 0.02 mg/kg.

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.03 mg/kg.

AZ83558/91. GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. Plums, not washed, pitted. Storage at -20 °C (storage time not stated). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. A second HPLC clean-up was used and analysis without internal standard. Samples were not corrected for indivual concurrent method recoveries (111-117% at 0.02 and 0.20 mg/kg). Control samples were < 0.02 mg/kg.

Peaches

Three cGAP for peaches are available:

- cGAP from the USA with 3 ×0.42 kg ai/ha with PHI 14 days on cherries, plums, apricots, nectarines and peaches
- cGAP from the Netherlands or Belgium with 1 ×0.38 kg ai/ha with PHI 28 days on cherries, plums, peaches
- cGAP from France with 1 ×0.25 kg ai/ha with PHI 21 days on cherries, plums, apricots, nectarines and peaches

Trials that could be matched to these cGAPs were summarized.

Table 186 lists trials conducted in the USA (1986), Germany (1981, 1982) and Italy (1982). A weed directed spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer)

was conducted at the base of the trees under the conditions listed in Table 186. Results marked with "[QU]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62, 62/1 or 62/2.

As the peaches from the trials in the USA and Germany were pitted before analysis, the residue data do not represent the RAC and would generally not be considered suitable for MRL derivation. Though no information on the weight fractions of stones and flesh is given either, this is not considered to affect the result, since residues were below LOQ.

Table 186 Supervised field trials on peaches (flesh only), treated with fluazifop-butyl at the base of the trees

PEACHES Country; year; location; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Yakima, WA, USA, 1986 (variety ns)	EC 125 (P) +0.25% NIS	3; (15, 96)	0.42 0.42 0.42	0.11 0.11 0.11	2.5-3 inch fruit; 20 August	ns	MAT	9	< 0.03 [LOQ=0.05]	TMU3168/B 32WA86-912R [Watford and Francis, 1987, PP5/0476]
Haslet, MI, USA, 1986 (Red Haven)	EC 125 (P) + 1% COC	3; (14, 41)	0.42 0.42 0.42	0.13 0.13 0.13	nearly mature; 29 July	ns	MAT	14	< 0.03 [LOQ=0.05]	TMU3168/B 71MI86-906R [Watford and Francis, 1987, PP5/0476]
Fort Valley, GA, USA, 1986 (Coronet)	EC 125 (P) + 1% COC	3; (14, 25)	0.42 0.42 0.42	0.27 0.27 0.27	GS ns; 03 June	ns	MAT	14	< 0.03 [LOQ=0.05]	TMU3168/B 83GA86-906R [Watford and Francis, 1987, PP5/0476]
Farmersville, CA, USA, 1986 (variety ns)	EC 125 (P) + 1% COC	3; (14, 91)	0.42 0.42 0.42	0.18 0.18 0.18	2-3 inch fruit; 25 July	ns	MAT	14	< 0.03 [LOQ=0.05]	TMU3168/B US02-86-S08-H [Watford and Francis, 1987, PP5/0476]
6741 Winden, Germany. 1981 (Red Haven)	EC 250 (rac)	1	1.0	0.1	GS ns; 14 July	ns	ns	0 7 14 21 28	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 [QU]	PP009B132 8164-RS-V4; [Atreya *et al*., 1982, PP9/0644] and RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
6731 Edesheim; Germany, 1982 (South Haven)	EC 250 (rac)	1	1.0	0.1	fruit 40 mm diameter; 14 July	ns	ns	0 6 13 21 28	0.05 0.05 0.19 0.27 0.05 [QU]	PP009B159 RS 8278 E2; [Atreya *et al*., 1982, PP9/0710]
Doganella; Latina; Italy, 1982 (Red Haven)	EC 250 (rac)	1	1.5	0.12	GS ns; 15 May	ns	ns	35 51	< 0.04 < 0.04 [QU] [LOQ=0.05]	PP009B187 6/82 UT46E; [Atreya *et al*., 1983, PP9/0621]
idem	EC 250	1	2.0	0.19	GS ns; 15 May	ns	ns	35 51	< 0.04 < 0.04	idem

PEACHES Country; year; location; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	(rac)								[QU] [LOQ=0.05]	
idem	EC 250 (rac)	1	2.5	0.25	GS ns; 15 May	ns	ns	35 51	< 0.04 < 0.04 [QU] [LOQ=0.05]	idem

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

Additional trial information

TMU3168/B plus supplement. GLP study. Weather, spray equipment and soil type were not reported. Spray volume not stated, but all spray applications were directed at the base of the trunk without contact to the trunk. Plots consisted of 5-11 trees, except MI (3 trees/plot). Sample sizes 5.0-13.2 lbs, i.e. > 2 kg. Samples were pitted by hand. Storage at -20 °C and analysed within 4 months. **HPLC-UV method PPRAM 62/2 with minor modification with a valid LOQ of 0.05 mg/kg**. Fortification of samples with 0.5 mg/kg of internal standard showed a mean recovery of 80% ± 5%. Samples were not corrected for indivual concurrent method recoveries. Control samples were < 0.02 mg/kg.

PP009B132. Non-GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume 1000 L/ha. Storageconditions not stated. **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. No internal standard was used. Samples were not corrected for indivual concurrent method recoveries (93% at 0.1 mg/kg). Control samples were < 0.05 mg/kg.

PP009B159. Non-GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume 1000 L/ha. Storageconditions not stated. **HPLC-UV method PPRAM 62/1, using internal standard with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for average concurrent method recoveries (85% at 0.5 mg/kg internal std). Control samples were 0.05 mg/kg and therefore the LOQ needs to be increased to 0.05/0.3=0.2 mg/kg to meet the criterion control sample at <0.3LOQ

PP009B187. Non-GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume 800 L/ha. Storageconditions not stated. **HPLC-UV method PPRAM 62/1 using internal standard with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for average concurrent method recoveries (79% at 0.5 mg/kg int std). Control samples were < 0.04 mg/kg.

Grapes

Three possible cGAPs for grapes are available:

- cGAP from the USA with 3 ×0.42 kg ai/ha with PHI 50 days
- cGAP from Belgium with 1 ×0.38 kg ai/ha with PHI 28 days
- cGAP from France with 1 ×0.25 kg ai/ha with PHI 28 days

Trials that could be matched to these cGAPs were summarized.

Table 187 lists trials conducted in the USA (1984, 1986, 2000), Germany (1981, 1982), Greece (1997) and Spain (1997). A weed directed strip or banded interrow spray application at the base of the vines with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted at the base of the vines under the conditions listed in Table 187. Results marked with "[QU]" or "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62, 62/1 or 62/2 or NMR method PPRAM 83.

Grapes were sampled as bunches or as berries (without stalks). The RAC is defined as the grapes including stalks and therefore the residues analysed in the berries (without stalks) do not represent the RAC and would generally not be considered for MRL derivation. Though no information on the weight fractions of berries and stalks is given either, this is not considered to affect the result, since residues were below LOQ.

Additional trials from South Africa (1980) were available with 1 × 2.5–6.0 kg ai/ha and harvest at 118 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from Australia (1980-1981) were available with 1 × 2.0–4.0 kg ai/ha and harvest at 28–112 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. These trials were not summarized, because they would not assist in MRL setting.

Table 187 Supervised field trials on grapes (berries or bunches), treated with fluazifop-butyl at the base of the vines

GRAPES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Visalia, CA, USA, 1984 (Thompson)	EC 125 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.15 0.15	GS ns; 6 June	ns	ns	79	< 0.03 [LOQ=0.05]	TMU3330/B; US02-84-S08 [Watford and Francis, 1987, PP5/1113] (processing)
Sunnyside, WA, USA 1984 (Concord)	EC 125 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.14 0.13	GS ns; 30 June	ns	ns	91	< 0.03 [LOQ=0.05]	TMU3330/B; 32WA84-063 [Watford and Francis, 1987, PP5/1113]
Exeter, CA, USA, 1984 (French Colombard)	EC 125 (P) + 1% COC	2 (27)	0.42 0.42	0.26 0.26	GS ns, 15 May	ns	ns	94	< 0.02 [b], [LOQ=0.05]	TMU3330/B; 45CA84-031 [Watford and Francis, 1987, PP5/1113]
Phelps, NY, USA, 1984 (Dechaunac)	EC 125 (P) + 1% COC	2 (21)	0.42 0.42	0.13 0.13	GS ns, 22 June	ns	ns	98	< 0.03 [SS] [LOQ=0.05]	TMU3330/B; 34NY84-007 [Watford and Francis, 1987, PP5/1113]
Sunnyside, WA, USA, 1986 (Concord)	EC 125 (P) +0.25% NIS	2 (20)	0.42 0.42	0.11 0.11	1/8–1/4 inch berries; 10 June	ns	CH	101	< 0.03 [LOQ=0.05]	TMU3144/B; 32WA86-924R [Watford and Francis, 1987, PP5/0471]
Haslett, MI, USA, 1986 (Concord)	EC 125 (P) +1% COC	2 (21)	0.42 0.42	0.12 0.12	grapes present; 9 July	ns	CH	73	< 0.03 [LOQ=0.05]	TMU3144/B; 71MI86-903R [Watford and Francis, 1987, PP5/0471]
Center Hill, AR, USA, 1986 (Venus)	EC 125 (P) +0.25% NIS	2 (24)	0.42 0.42	0.15 0.15	early maturity; 26 June	ns	CH	12	< 0.03 [LOQ=0.05]	TMU3144/B; 06AR86-902R [Watford and Francis, 1987, PP5/0471]
Burgaw, NC, USA, 1986 (Carlos)	EC 125 (P) 0.25% NIS	2 (27)	0.42 0.42	ns	prebloom; 26 May	ns	CH	93	< 0.03 [SS] [LOQ=0.05]	TMU3144/B; 61NC86-903R [Watford and Francis, 1987, PP5/0471]

GRAPES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Tulare, CA, USA 1986 (Perlette)	EC 125 (P) +1% COC	2 (28)	0.42 0.42	0.22 0.22	80-90% calyptras; cane length 4-6 ft; 2 May;	ns	CH	59	< 0.03 [LOQ=0.05]	TMU3144/B; 45CA86-908R [Watford and Francis, 1987, PP5/0471]
idem	EC 125 (P) +1% COC	2 (28)	2.1 2.1	0.22 0.22	80-90% calyptras; cane length 4-6 ft; 2 May;	ns	CH	59	< 0.03 [LOQ=0.05]	idem
Sultana, CA, USA, 1986 (Thompson Seedless)	EC 125 (P) +1% COC	2 (28)	0.42 0.42	0.22 0.22	60-80% calyptras; cane length 4-6 ft; 2 May	ns	CH	87	< 0.03 [LOQ=0.05]	TMU3144/B; 45CA86-907R [Watford and Francis, 1987, PP5/0471]
idem	EC 125 (P) +1% COC	2 (28)	2.1 2.1	0.22 0.22	60-80% calyptras; cane length 4-6 ft; 2 May	ns	CH	87	< 0.03 [LOQ=0.05]	idem
Dundee, NY, USA, 2000 (juice grape: Concord)	EC 240 (P) + 0.5% NIS	3 (13, 14)	0.43 0.42 0.43	0.18 0.18 0.18	berries present; 28 July	SiL	CH	50	<u>≤ 0.01</u>	RR 00-062B; 182GRNY1 [Stewart, 2001, 406504]
Dundee, NY, USA, 2000 (wine grape: Aurora)	EC 240 (P) + 0.5% NIS	3 (12, 14)	0.43 0.44 0.43	0.18 0.18 0.18	berries present; 6 July	L	CH	50	< 0.01	RR 00-062B; 183GRNY2 [Stewart, 2001, 406504]
Poplar, CA, USA, 2000 (raisin-table-wine grape: Thompson Seedless)	EC 240 (P) + 0.5% NIS	3 (14, 14)	0.42 0.42 0.42	0.28 0.28 0.28	berries present; 24 July	SaL	CH	50	<u>≤ 0.01</u>	RR 00-062B; 184GRCA1 [Stewart, 2001, 406504]
Richgrove, CA, USA, 2000 (table grape: Emperor)	EC 240 (P) + 0.5% NIS	3 (14, 14)	0.43 0.42 0.42	0.29 0.28 0.28	berries present; 29 Aug	SaL	CH	50	<u>≤ 0.01</u>	RR 00-062B; 185GRCA2 [Stewart, 2001, 406504]
Fresno, CA, USA, 2000 (raisin-table-wine grape: Thompson Seedless)	EC 240 (P) + 0.5% NIS	3 (14, 14)	0.42 0.42 0.41	0.22 0.22 0.22	berries present; 14 July	SaL	CH	50	<u>≤ 0.01</u>	RR 00-062B; 186GRCA3 [Stewart, 2001, 406504]
Fresno, CA, USA, 2000 (table grape: Ruby Seedless)	EC 240 (P) + 0.5% NIS	3 (14, 14)	0.41 0.41 0.42	0.22 0.21 0.21	berries present; 7 July	SaL	CH	50	< 0.01	RR 00-062B; 187GRCA4 [Stewart, 2001, 406504]
Madera, CA, USA, 2000	EC 240 (P)	3 (14, 14)	0.40 0.41 0.43	0.22 0.22 0.22	berries present; 3 July	LSa	CH	49	<u>≤ 0.01</u>	RR 00-062B; 188CRCA5 [Stewart,

GRAPES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(raisin-table-wine grape: Thompson Seedless)	+ 1% COC									2001, 406504]
Rerolby, CA, USA, 2000 (wine grape: Colombard)	EC 240 (P) + 1% COC	3 (14, 14)	0.41 0.42 0.43	0.22 0.22 0.22	berries present; 5 July	SaL	CH	49	< 0.01	RR 00-062B; 189GRCA6 [Stewart, 2001, 406504]
Santa Maria, CA, USA, 2000 (wine grape: Sauvignon Blanc)	EC 240 (P) + 0.5% NIS	3 (14, 13)	0.41 0.42 0.42	0.22 0.22 0.22	berries present; 10 Aug	SaL	CH	49	< 0.01	RR 00-062B; 190CRCA7 [Stewart, 2001, 406504]
Woodlake, CA, USA, 2000 (table grape: Red Flame)	EC 240 (P) + 1% COC	3 (14, 14)	0.43 0.43 0.42	0.17 0.17 0.17	berries present; 5 July	SaL	CH	50	< 0.01	RR 00-062B; 191GRCA8 [Stewart, 2001, 406504]
Hood River, OR, USA, 2000 (wine grape: Chardonnay)	EC 240 (P) + 0.5% NIS	3 (14, 15)	0.41 0.42 0.41	0.16 0.17 0.16	berries present; 27 July	L	CH	49	< 0.01	RR 00-062B; 192GROR1 [Stewart, 2001, 406504]
Paterson, WA, USA, 2000 (wine grape: Cheninblanc)	EC 240 (P) + 0.5% NIS	3 (14, 14)	0.40 0.43 0.44	0.17 0.17 0.18	berries present; 31 July	Sa	CH	50	< 0.01	RR 00-062B; 193GRWA1 [Stewart, 2001, 406504]
Hanford; CA, USA, 2000 (Thompson Seedless)	EC 240 (P) + 0.5% NIS	3 (14, 14)	2.1 2.1 2.1	1.4 1.4 1.4	berries present; 24 July	SaL	NH	50	< 0.01	RR 00-067B; 197GRCA1; [Stewart, 2001, 406498] (processing)
6748 Bad Bergzabern; Germany, 1981 (Sylvaner)	EC 250 (rac)	1	1.0	0.1	GS ns; 2 Sept	ns	ns	0 8 14 21 29	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 [QU] [LOQ=0.05]	PP009B139 8165-RS-V1; [Atreya *et al*., 1982, PP9/0436]
6749 Klingenmünster; Germany, 1981 (Portogieser)	EC 250 (rac)	1	1.0	0.1	GS ns; 27 Aug	ns	ns	0 7 14 21 28	0.05 < 0.02 < 0.02 < 0.02 < 0.02 [QU] [LOQ=0.05]	PP009B139 8165-RS-V2; [Atreya *et al*., 1982, PP9/0436]
7580 Bühl-Eisental Germany, 1981 (Riesling)	EC 250 (rac)	1	1.0	0.1	GS ns; 16 Sept	ns	ns	0 7 14 22 28	< 0.02 0.14 < 0.02 0.03 < 0.02 [QU] [LOQ=0.05]	PP009B139 8165-RS-V3; [Atreya *et al*., 1982, PP9/0436]
7519	EC	1	1.0	0.1	GS ns;	ns	ns	0	< 0.02	PP009B139

GRAPES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Oberderdingen-Großvillars; Germany, 1981 (Müller-Thurgau)	250 (rac)				28 Aug			7 14 20 28	< 0.02 < 0.02 < 0.02 < 0.02 [QU] [LOQ=0.05]	8165-RS-V4; [Atreya *et al*., 1982, PP9/0436]
6749 Gleiszellen-Gleishorbarch; Germany, 1982 (Portogieser)	EC 250 (rac)	1	1.0	0.1	BBA 35; 24 Aug	ns	ns	0 7 14 21 28 28 [a]	0.06 < 0.04 < 0.04 < 0.04 < 0.04 < 0.04 [b] [QU] [LOQ=0.05]	PP009B180 RS 8279 E1; [Atreya *et al*., 1983, PP9/0437]
6740 Landau-Godramstein Germany, 1982 (Müller-Thurgau)	EC 250 (rac)	1	1.0	0.1	BBA 35; 27 Aug	ns	ns	0 6 14 21 28 28	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 [b] [QU] [LOQ=0.05]	PP009B180 RS 8279 E2; [Atreya *et al*., 1983, PP9/0437]
8945 Hirschberg-Großsachsen; Germany, 1982 (Spätburgunder)	EC 250 (rac)	1	1.0	0.1	BBA 35; 8 Sept	ns	ns	0 7 13 20 28 28	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 [b] [QU] [LOQ=0.05]	PP009B180 RS 8279 E3; [Atreya *et al*., 1983, PP9/0437]
7632 Friesenheim; Germany, 1982 (Ruländer)	EC 250 (rac)	1	1.0	0.1	BBA 35; 7 Sept	ns	ns	0 7 14 20 28 28	< 0.03 < 0.03 < 0.03 < 0.03 < 0.03 < 0.03 [b] [QU] [LOQ=0.05]	PP009B180 RS 8279 E4; [Atreya *et al*., 1983, PP9/0437]
Binefar, Aragón; Spain, 1997 (Garnacha)	EC 125 (P)	1	0.75	0.19	GS ns; 7 Aug	C	MAT	27	< 0.01	RJ2636B ES10-97-SH009; [Mason *et al*., 1999, PP5/0189]
Tamarite; Aragón; Spain, 1997 (Macabeo)	EC 125 (P)	1	0.75	0.19	GS ns; 7 Aug	C	MAT	27	< 0.01	RJ2636B ES10-97-SH109 [Mason *et al*., 1999, PP5/0189]
Zevgolatio; Korinthia; Greece, 1997 (Sultanina)	EC 125 (P)	1	0.75	0.15	Mature bunches; 31 July	L	CH	28	< 0.01	RJ2636B GR-97-H201 [Mason *et al*., 1999, PP5/0189]
idem	EC	1	0.75	0.15	Mature	LC	CH	28	< 0.01	RJ2636B

GRAPES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	125 (P)				bunches; 31 July					GR-97-H202 [Mason *et al*., 1999, PP5/0189]

[SS] Sample sizes not stated; results are considered not representative for MRL derivation.

QU Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[a] Grapes left lying on the ground for 6-8 days

[b] First analysis with HPLC-UV method PPRAM 62/2 showed residues as 0.04 mg/kg total fluazifop. The control sample also had a peak at the retention time of fluazifop. Both the treated sample and the control sample were reanalysed by 19F-NMR. No measurable residues (< 0.02 mg/kg) were found in either the treated sample or the control sample. Concurrent method recoveries for the 19F-NMR method were 96% at 0.05 mg/kg fluazifop acid (II). The NMR result is reported here.

Additional trial information

TMU3330/B. GLP. No unusual weather conditions. Plot sizes are not stated. Treatment of grass weeds under the grape vines via a directed weed spray (strip or band spray). Application equipment not indicated. Spray volume 17-34 GPA = 160-320 L/ha. An adjuvant was added. Sample sizes of grapes were 5.0 lbs = 2.3 kg, except NY where sample size is not stated. Samples were stored at -20 °C for a maximum of 16 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 at 230 nm with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent method recoveries (78-106% at 0.025, 0.05, 0.1 mg/kg (n = 1-4 per fortification level). Control samples were < 0.03 mg/kg. All samples from trial 45CA84-031,excluding the control, were fortified with 0.1 mg/kg of PP748 as an internal standard for reanalysis with **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**.

TMU3144B. GLP. Unusual weather conditions did not affect the results. Plots consisted of 2 vines (NC) or 7-11 vines (WA, MI, AR, CA, CA). Treatment of grass weeds under the grape vines via a directed spray to the rows between the vines. Application by CO2 knapsack/backpack sprayers with straight boom or single nozzle sprayer or tractor with side-mounted elbow boom sprayer. Spray volume 20.0- 40.6 GPA = 190-380 L/ha. An adjuvant was added. Sample sizes of grapes (AR, NC), whole grape bunches (WA) or berries (MI) were 5.0-8.0 lbs = 2.3-3.6 kg, except NC where samples size is not stated. Samples were stored at -20 °C for a maximum of 4 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 at 230 nm with a valid LOQ of 0.05 mg/kg**. Average internal std recoveries were 80 ± 8% at 0.5 mg/kg. Control samples were < 0.03 mg/kg.

RR 00-062B. GLP. No unusual weather conditions. Plot size >12 vines/plot except except trial 193 with 3 vines and trials 183, 190, 191 and 192 with 6-10 vines. Treatment of grass weeds on the vineyard floor beneath the vines. Application by backpack sprayers, handheld spreayers or tractor mounted sprayers Spray volume 16- 28 GPA = 150-260 L/ha. An adjuvant was added. Whole grape bunches were sampled with clippers, knives, pruners and shears and were taken from at least 12 areas within the plot Whole grape bunches were 2.8-19 lbs = 1.3-8.6 kg. Samples were stored at -5 °C or lower for 8- 45 days. Samples were analysed for total fluazifop using **GC-MS method RR 91-014B with a valid LOQ of 0.01 mg/kg**. Average concurrent recoveries were 88-90% at 0.01-1.0 mg/kg for fluazifop-butyl. Control samples were < 0.01 mg/kg.

RR 00-067B. GLP. No unusual weather conditions. Plot size >12 vines/plot Treatment of grass weeds on the vineyard floor beneath the vines. Application by backpack sprayers. Spray volume 16 GPA = 150 L/ha. An adjuvant was added. Whole grape bunches were sampled with clippers and were taken from at least 12 areas within the plot Whole grape bunches were 150 lbs = 68 kg. Samples were stored at 8 °C until processing (2 weeks) and were than stored at -15 °C or lower for 33 days. Samples were analysed for total fluazifop using **GC-MS Method RR91-014B with a valid LOQ of 0.01 mg/kg.** Average concurrent recoveries were 84-97% at 0.01-1.0 mg/kg for fluazifop-butyl. Control samples were < 0.01 mg/kg.

PP009B139. Non-GLP study. Poor quality of the study. Weather, soil type, plot size, sample size, growth stage at harvest were not reported.. Knapsack sprayer, spray volume 1000 L/ha. Growth stage at harvest not stated. Storage 309-364 days; storage temperature not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. An internal std was not used. Samples were not corrected for averageconcurrent method recoveries (86-88% at 0.05-0.5 mg/kg). Control samples were < 0.02 mg/kg.

PP009B180. Non-GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Spray volume 1000 L/ha. Storage 62-104 days; storage temperature not stated. Samples were analysed for total fluazifop using HPLC-UV method **PPRAM 62/1 using internal standard with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for average concurrent method recoveries (76% at 0.5 mg/kg internal standard). Control samples were < 0.03 or < 0.04 or < 0.05 mg/kg total fluazifop.

RJ2636B. GLP. No unusual weather conditions. Plot size 12 plants (on 5-6 m^2 in Greece and 11-14 m^2 in Spain). Soil directed spray (Greece) or inter-row banded spray (Spain); application by gas knapsack sprayers with a hand lance. Spray volume 400-500 L/ha. Grapes (2.5-3.7 kg; 8-12 bunches) were sampled by hand taking care to avoid plot boundaries;

samples were taken sytematically from across plots. Samples were stored at -13 °C or lower for 47-125 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for individual concurrent method recoveries (71-86% at 0.05-0.1 mg/kg). Control samples were < 0.01 mg/kg.

Olives

One cGAP for olives is available:

- cGAP from France with 1 ×0.25 kg ai/ha with PHI 21 days

Trials that could be matched to this cGAP were summarized.

Table 188 lists trials conducted in Italy (1986, 1997). A weed directed spray application with fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 188. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for NMR method PPRAM 83.

As the olives from the trials in Italy were pitted before analysis, the residue data do not represent the RAC and would generally not be considered suitable for MRL derivation. Though no information on the weight fractions of stones and flesh is given either, this is not considered to affect the result, since residues were below LOQ.

Table 188 Supervised field trials on olives (flesh only), treated with fluazifop-butyl at the base of the trees

OLIVES Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Manfredonia Italy, 1986; (Coratina)	EC 125 (P)	1	0.75	0.12	pre-ripening 23 Sept	ns	BR	20 40	< 0.03 < 0.03 [SS] [LOQ=0.05]	M4526B; 37/86/2; [O'Brien and Harradine; 1987; PP5/0485]
idem	EC 125 (P)	1	1.5	0.25	pre-ripening 23 Sept	ns	BR	20 40	< 0.03 < 0.03 [SS] [LOQ=0.05]	idem
Vernareccia-Manfredonia; Puglia; Italy; 1997 (Coratina)	EC 125 (P)	1	0.75	0.19	21 Oct	CSi	CH	27	< 0.01	RJ2634B; IT51-97-H346 [Mason *et al*, 1999, PP5/0188]
Montemurlo; Toscana; Italy; 1997; (Moraiolo)	EC 125 (P)	1	0.75	0.19	20 Oct	L	CH	28	< 0.01	RJ2634B; IT24-97-H347 [Mason *et al*, 1999, PP5/0188]

[SS] Sample sizes not stated or less than the required 1 kg; results are considered not representative for MRL derivation.

Additional trial information

M4526B. Non-GLP. No unusual weather conditions. Plot size not stated. Application at the base of the trees using a motor pump. Spray volume 600 L/ha. Stones were removed and only the olive flesh was analysed. Sample size not stated. Storage at -18 °C for

maximum 199 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Average internal standard recovery (92% at 0.5 mg/kg). Control samples were < 0.03 mg/kg.
RJ2634B. GLP. No unusual weather conditions. Plot size: 4 trees/plot. Application at the base of the trees using a motor knapsack sprayer with boom. Spray volume 400 L/ha. Suitable nets were laid on the soil below the trees and the olives were dropped on the nets by combing or beating. The olives were picked by hand from the nets laid on the ground (2.4-2.8 kg). Storage at -18 °C for maximum 129 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for individual concurrent method recoveryie (77-985 at 0.05-0.5 mg/kg). Control samples were < 0.01 mg/kg.

Banana

Two possible cGAPs for bananas are available:

- cGAP from the USA with 3 ×0.42 kg ai/ha with a PHI of 0 days
- cGAP from France (and its overseas areas) with 1 ×0.25 kg ai/ha with a PHI of 0 days

Trials that could be matched to these cGAPs were summarized.

Table 189 lists trials conducted in the USA (1999) and Australia (1985). A weed directed spray application with fluazifop-P-butyl (R-enantiomer) was conducted at the base of the banana plants under the conditions listed in Table 189. Results marked with "[RT]" or "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[RT] indicates that the fruit samples were rotten.

[SS] indicates that the sample sizes were not reported or less than 24 fruits (from 4 bunches).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62/2.

Additional trials from Honduras (1984-1986) were available on bananas with an application of 1–11 × 0.25 kg ai/ha and harvest at 14–41 DAT [Pay, 1987, PP5/0185; report M4388B]. Additional trials from Martinique (1984) were available on bananas with an application of 1 × 0.50–1.0 kg ai/ha and harvest at 2–30 DAT [Culoto and Mallmann, 1985, PP9/0130, report RIC1933]. Since the residues were only measured in the pulp, these studies are summarized in the section "residues in the edible portion of food commodities".

Table 189 Supervised field trials on bananas (bagged (bg) or unbagged (ub), whole fruit), treated with fluazifop-butyl at the base of the banana plants

BANANAS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Homestead, FL, USA, 1999 (Raja Puri)	EC 240 (P) + 0.125% NIS	3 (30, 30)	0.42 0.42 0.42	0.16 0.16 0.17	Maturing, 27 July	SaL	MAT	0	< 0.01 ub < 0.01 ub mean < 0.01 ub [RT] [a]	RR 00-043B 42-FL-99-131 [Miller, 2000, PP5/0454 and Kleinschmidt and Miller, 2000, 405683]
idem	EC 240 (P) + 0.125% NIS	3 (30, 30)	0.42 0.42 0.42	0.16 0.16 0.17	Maturing, 27 July	SaL	MAT	0	< 0.01 bg < 0.01 bg mean < 0.01 bg [RT] [a]	idem
Keaau, HI, USA, 1999 (Cavendish Williams	EC 240 (P) + 0.25% NIS or	3 (30, 30)	0.42 0.42 0.42	0.20 0.20 0.20	Normal harvest, 25 May	rocky muck	CH	0	< 0.01 ub < 0.01 ub mean < 0.01 ub	RR 00-043B 14-HI-99-132 [Miller, 2000, PP5/0454 and

BANANAS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Dwarf)	1% COC								[a]	Kleinschmidt and Miller, 2000, 405683]
idem	EC 240 (P) + 0.25% NIS or 1% COC	3 (30, 30)	0.42 0.42 0.42	0.20 0.20 0.20	Normal harvest, 25 May	rocky muck	CH	0	< 0.01 bg < 0.01 bg mean < 0.01 bg [a]	idem
Kurtistown, HI, USA, 1999 (Cavendish Williams Dwarf)	EC 240 (P) + 0.25% NIS or 1% COC	3 (30, 30)	0.42 0.42 0.42	0.22 0.22 0.22	Normal harvers, 25 May	SiCL	CH	0	< 0.01 ub < 0.01 ub mean <u>< 0.01</u> ub [a]	RR 00-043B 14-HI-99-133 [Miller, 2000, PP5/0454 and Kleinschmidt and Miller, 2000, 405683]
idem	EC 240 (P) + 0.25% NIS or 1% COC	3 (30, 30)	0.42 0.42 0.42	0.22 0.22 0.22	Normal harvers, 25 May	SiCL	CH	0	< 0.01 bg < 0.01 bg mean < 0.01 bg [a]	idem
Waialua, HI, USA, 1999 (Cavendish Williams Dwarf)	EC 240 (P) + 0.25% NIS or 1% COC	3 (30, 30)	0.42 0.42 0.42	0.19 0.19 0.19	Full maturity, green, 11 July	C	MAT green	0	< 0.01 ub < 0.01 ub mean <u>< 0.01</u> ub [a]	RR 00-043B 14-HI-99-134 [Miller, 2000, PP5/0454 and Kleinschmidt and Miller, 2000, 405683]
idem	EC 240 (P) + 0.25% NIS or 1% COC	3 (30, 30)	0.42 0.42 0.42	0.19 0.19 0.19	Full maturity, green, 11 July	C	MAT green	0	< 0.01 bg < 0.01 bg mean < 0.01 bg [a]	idem
Pepeekeo, HI, USA, 1999 (Cavendish Williams Dwarf)	EC 240 (P) + 0.25% NIS or 1% COC	3 (30, 30)	0.42 0.42 0.42	0.26 0.26 0.26	Green mature stage, 26 July	SiCL	MAT green	0	< 0.01 ub < 0.01 ub mean <u>< 0.01</u> ub [a]	RR 00-043B 14-HI-99-135 [Miller, 2000, PP5/0454 and Kleinschmidt and Miller, 2000, 405683]
idem	EC 240 (P) + 0.25% NIS or 1% COC	3 (30, 30)	0.42 0.42 0.42	0.26 0.26 0.26	Green mature stage, 26 July	SiCL	MAT green	0	< 0.01 bg < 0.01 bg mean < 0.01 bg [a]	idem
Tully, Queensland, Australia, 1985 (Williams)	EC, 212 (P)	1	1.1	ns	3-4 wks PCH, 10 Oct	CL	4 wks 2 wks PCH	1 14	< 0.02 ub < 0.02 ub [SS]	RIC1934 [Markus and Nguy, 1986, PP5/0184]
idem	EC 212 (P)	1	2.1	ns	3-4 wks PCH, 10 Oct	CL	4 wks 2 wks PCH	1 14	< 0.02 ub < 0.02 ub [SS]	idem

[SS] Sample sizes less than the required 24 fruits from 4 bunches; results are considered not representative for MRL derivation.

[RT] Fruit samples were rotten and are considered not representative for MRL derivation.

[a] Resuls came from two replicate field samples; the mean is take for MRL derivation, , if according to cGAP

Additional trial information

RR-00-043B GLP study. No unusual weather conditions reported. Plots sizes consisted of at least 8 trees per plot. Soil type not reported. The product was applied as broadcast spray to soil. Either a non-ionic surfactant was used as adjuvant or a crop oil concentrate. Duplicate samples per plot were taken (24 fruits, weighing at least 6.8 kg); samples from plot 42-FL-99-131 weighed only 1.4-1.8 kg due to fruit rot. The samples were stored frozen at -18 °C for 10 months (127-182 to extraction and a further 1-4 days to analysis). Storage stablity was demonstrated by reference to other studies. Samples were analysed for total fluazifop using **GC-MS method ABC 45820-M-1 (=GC-MS method RR91-014B) with a valid LOQ of 0.01 mg/kg**. The recovery rate ranged from. The samples were not corrected for concurrent revcoveries (77-110% at 0.01 and 0.5 mg/kg (n = 3/level)). Control samples were < 0.01 mg/kg.

RIC1934. GLP not reported. Weather conditions were not reported. Plot sizes 40 m2, with 15 plants per plot. Soil type clay loam. The product was applied with hand lance Tee jet to point of run off. Sample size (4 fingers/sample, i.e. 4 fruits per sample). The samples were stored at -20 °C for 293-307 days. Samples were analysed **for total fluazifop using HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Concurrent recovery was 80% at 0.5 mg/kg. Control samples were < 0.02 mg/kg.

Mango

A GAP for mango is not available. As the manufacturer was not seeking the establishment of maximum residue levels for mangoes, no further action was taken to consider the available studies on mango [Baron, 1989, report PR 2644, not referenced].

Almond nutmeat

One cGAP for almonds is available:

- cGAP from France with 1 ×0.25 kg ai/ha and a PHI of 21 days for almonds, chestnuts, hazelnuts, macadamia nuts and walnuts

Trials that could be matched to this cGAPs were summarized.

Table 190 lists trials conducted in the USA (1990). A weed directed spray application with fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 190.

Table 190 Supervised field trials on almonds (nutmeat), treated with fluazifop-butyl at the base of the trees

ALMOND NUTMEAT Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
College City, Colusa, CA, USA; 1990; (NonPareil)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	Hulls cracking; 03 Aug	SaL	NH	14	< 0.01 < 0.01; [a]	RR 92-041B; 17-CA-90-601 and 602; [Roper, 1992; PP5/0572]
Idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	Hulls cracking; 03 Aug	SaL	NH	14	< 0.01 < 0.01 [a]	idem
Durham, Butte, CA; USA; 1990 (Nonpareil)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	GS ns; 17 Aug	L	At hull split	14	< 0.01	RR 92-041B; 72-CA-90-603; [Roper, 1992; PP5/0572]

ALMOND NUTMEAT Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	GS ns; 17 Aug	L	At hull split	14	< 0.01	idem
Ord Bent, Glenn, CA, USA; 1990 (Texas)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	GS ns; 31 Aug	L	At hull split	14	< 0.01	RR 92-041B; 72-CA-90-604; [Roper, 1992; PP5/0572]
idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	GS ns; 31 Aug	L	At hull split	14	< 0.01	idem
Lost Hills, Kern, CA; USA; 1990; (Nonpareil)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	GS ns; 30 Aug	SaL	MAT	14	< 0.01	RR 92-041B; 81-CA-90-605; [Roper, 1992; PP5/0572]
idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	GS ns; 30 Aug	SaL	MAT	14	< 0.01	idem
Lost Hills, Kern, CA, USA; 1990 (Carmel)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	GS ns, 01 Oct	SiL	MAT	14	< 0.01	RR 92-041B; 81-CA-90-606; [Roper, 1992; PP5/0572]
idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	GS ns, 01 Oct	SiL	MAT	14	< 0.01	

[a] Results came from two replicate plots; the highest is taken for MRL derivation if according to cGAP

Additional trial information:

RR 92-041B; GLP study. No unsual weather conditions. Application by ground sprayer. Spray volume 93.46 L/ha. Plot size 13-310 m^2., with 4-11 trees/plot, except in trial 605 with 3 trees/plot. Nuts were shaken from the tree mechanically and picked by hand. One nutmeat (>1 kg) sample was taken per plot. Samples were stored at <-20 °C for max 411 days. Samples were analysed for total fluazifop using **GC-MS-MS method RR91-014B with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average method recoveries (86% at 0.01 mg/kg). Control samples < 0.01 mg/kg.

Hazelnuts

One cGAP for hazelnuts is available:

- cGAP from France with 1 ×0.25 kg ai/ha and a PHI 21 of days for almonds, chestnuts, hazelnuts, macadamia nuts and walnuts

  Trials that could be matched to this cGAP were summarized.

Table 191 lists trials conducted in the UK (1997) and Italy (1982). A weed directed spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 191.Results marked with "[QU]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

Table 191 Supervised field trials on hazelnuts (nutmeat), treated with fluazifop-butyl at the base of the trees

HAZELNUT NUTMEAT Location; Country; Year; (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Plaxtol; Kent; UK; 1997 (Filberts; Kent Cob)	EC 125 (P)	1	0.75	0.38	Mature; 5 Aug	L	MAT	28	0.01; 0.01 [a]	RJ2656B; GB52-97-S061andS062 [Jones and Hughes, 1999, PP5/0223]
Avella; Italy; 1982 (Martarella-Giafoni)	EC 250 (rac)	1	1.5	0.19	nuts visible; 6 July	ns	ns	49 73	< 0.05 < 0.05 [QU] [CT] [cntrl=0.05]	PP009B194; 6/82/UT 47E; [Atreya *et al.*; 1983; PP9/0628]
idem	EC 250 (rac)	1	2.0	0.25	nuts visible; 6 July	ns	ns	49 73	< 0.05 < 0.05 [QU] [CT] [cntrl=0.05]	idem
idem	EC 250 (rac)	1	2.5	0.31	nuts visible; 6 July	ns	ns	49 73	0.08 0.07 [QU] [CT] [cntrl=0.05]	idem

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

[a] Results came from two replicate field plots; highest result is taken for MRL derivation if according to cGAP

Additional trial information

RJ2656B. GLP study. No unusual weather conditions. Application as directed band spray to soil using a gas knapsack sprayer with lance with an off-set spray pattern with a spray volume of 200 L/ha. Whole nuts (2 kg from 7 trees) were taken systematically by hand from across the plots. Storage at -18°C for a maximum of 86days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 197/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (71-84% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg (n = 2).

PP009B194. Non-GLP study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray application around the base of the trees; volume of 800 L/ha. Storage time and conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/1 with avalid LOQ of 0.05 mg/kg**. Concurrent method recoveries not stated. Control samples < 0.05-0.05 mg/kg (n = 2).

Macadamia nuts

Two cGAPs for macadamia nuts are available:

- cGAP from the USA with 3 ×0.42 kg ai/ha and a PHI of 1 day for macadamia and pecans
- cGAP from France with 1 ×0.25 kg ai/ha and a PHI 21 of days for almonds, chestnuts, hazelnuts, macadamia nuts and walnuts

Trials that could be matched to these cGAPs were summarized.

Table 192 lists trials conducted in the USA (1986, 1987). A weed directed spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the base of the trees under the conditions listed in Table 192. Results marked with "[SS]"are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample size was less than the required 1 kg nutmeat.

Table 192 Supervised field trials on macadamia (nutmeat), treated with fluazifop-butyl at the base of the trees

MACADAMIA NUTMEAT; Location; Country; Year; (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Honomalino, Hawaii, USA; 1986 (variety ns)	EC 120 (P) + 0.25% NIS	3 (130, 48)	0.28 0.28 0.28	0.077 0.077 0.077	mature nuts; 28 Oct, 1986	ns	MAT	1 15	< 0.1[a] < 0.1 [a] [SS]	IR-4 PR 3431; Trial no ns; [Baron, 1989; 464386]
idem	EC 120 (P) + 0.25% NIS	3 (130, 48)	1.1 1.1 1.1	0.31 0.31 0.31	mature nuts; 28 Oct, 1986	ns	MAT	1 15	< 0.1 [a] < 0.1 [a] [SS]	IR-4 PR 3431; Trial no ns; [Baron, 1989; 464386]
Honomalino, Hawaii, USA, 1987, (variety ns) 2nd year application	EC 120 (P) + 0.25% NIS	3 (136, 87)	0.28 0.28 0.28	0.077 0.077 0.077	mature nuts; 19 Aug, 1987	ns	MAT	1 14	< 0.1 [a] < 0.1 [a] [SS]	IR-4 PR 3431; Trial no ns; [Baron, 1989; 464386]
idem	EC 120 (P) + 0.25% NIS	3 (136, 87)	1.1 1.1 1.1	0.31 0.31 0.31	mature nuts; 19 Aug, 1987	ns	MAT	1 14	< 0.1 [a] < 0.1 [a] [SS]	IR-4 PR 3431; Trial no ns; [Baron, 1989; 464386]

[SS] Sample size below the required 1 kg nutmeat(0.45-0.90 kg in all trials); samples are considered not representative for MRL derivation.

[a] Results are the average of 4 replicate field samples; the mean is taken for MRL derivation, if according to cGAP; all samples were < 0.1 mg/kg.

Additional trial information:

IR-4 PR 3431; GLP study. No unusual weather conditions. Ground application. Spray volume 364 L/ha. Plot size 80 m^2, with 8 trees/plot. Macadamia nuts were picked by hand from the ground (1.9-5.4 kg). Samples were stored at room temperature (23-26 °C) for 1-2 days until they were husked. After husk removal, the wet in-shell nuts were placed in a forced air dryer for 10 days at +43 °C. Nuts were cracked and shells were discarded. The raw nutmeats (1-2 lbs, 0.45-0.90 kg) were stored at -15 °C or lower for 7-9 months (1986 samples) or 4 months (1987 samples). Samples were analysed for total fluazifop using **HPLC-UV Method PPRAM, 62/2 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for average concurrent method recoveries (68% at 0.1–0.5 mg/kg). Control samples were < 0.1 mg/kg.

Pecans

One cGAP for pecans is available:

- cGAP from the USA with 3 ×0.42 kg ai/ha and a PHI of 1 day for macadamia and pecans
- Trials that could be matched to this cGAP were summarized.

Table 193 lists trials conducted in the USA (1985). A weed directed spray application with fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 193. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[SS] indicates that the sample size was less than the required 1 kg nutmeat.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method TMU3251.

Table 193 Supervised field trials on pecans (nutmeat), treated with fluazifop-butyl at the base of the trees

PECAN NUTMEAT; Location; Country; Year; (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Stephenville, TX; USA; 1985; (Mahan)	EC - 120 (P)	3 (15, 95)	0.42 0.42 0.42	0.23 0.23 0.23	GS ns; 14 Oct	ns	ns	31	< 0.03 [SS] [LOQ=0.05]	TMU3251/B; 60TX85-038R; [Watford and Francis, 1987, 434208]
College Station, TX, USA; 1985; (Success, Stuart, Desirable, Mahan)	EC - 120 (P)	3 (14, 124)	0.42 0.42 0.42	0.30 0.29 0.27	GS ns; 18 Sept	ns	ns	30	< 0.03 [LOQ=0.05]	TMU/3251/B; 60TX85-002R; [Watford and Francis, 1987, 434208]
Robson, LA; USA; 1985; (Moneymaker)	EC - 120 (P)	3 (14, 147)	0.42 0.42 0.42	0.20 0.20 0.20	GS ns; 03 Oct	ns	ns	34	< 0.03 [LOQ=0.05]	TMU/3251/B; 36LA85-048; [Watford and Francis, 1987, 434208]

[SS] Sample size less than the required 1 kg nutmeat (0.29 kg in trial 60TX85-038R); sample considered not representative for MRL derivation.

Additional trial information:

TMU3251/B; GLP study. No unusual weather conditions. Ground spray application. Spray volume 140-215 L/ha. Plots consisted of 1 to 3 trees/plot. Nutmeat samples of 0.29 kg (trial 60TX85-038R), 2.3 kg (trial 60TX85-002R) and 3.5 kg (trial 36LA85-048) were collected. Samples were stored frozen at <-20 °C for 9 months. Samples were analysed for total fluazifop using **HPLC-UV method, TMU3251 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for individual method recoveries (68% at 0.05-0.1 mg/kg). Control samples were < 0.03 mg/kg.

Walnuts

One cGAP for walnuts is available:

- cGAP from France with 1 ×0.25 kg ai/ha and a PHI 21 of days for almonds, chestnuts, hazelnuts, macadamia nuts and walnuts

Trials that could be matched to this cGAP were summarized.

Table 194 lists trials conducted in the USA (1990). A weed directed spray application with fluazifop-P-butyl (R-enantiomer) was conducted at the base of the trees under the conditions listed in Table 194.

Table 194 Supervised field trials on walnuts (nutmeat) treated with fluazifop-butyl at the base of the trees

WALNUT NUTMEAT; Location; Country; Year; (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Meridian, Suteter, CA; USA; 1990; (Serr)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	Full size, green; 28 Aug	CL	MAT	14	< 0.01	RR 92-009B; 17-CA-90-641; [Roper, 1992, PP5/0582]
idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42	0.45	Full size, green; 28 Aug	CL	MAT	14	< 0.01	idem
Gridley, Butte, CA; USA; 1990; (Chico)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	Mature; 27 Aug	CL	MAT	14	< 0.01	RR 92-009B; 72-CA-90-642; [Roper, 1992, PP5/0582]
Idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42	0.45	Mature; 27 Aug	CL	MAT	14	< 0.01	idem
Gridley, Butte, CA; USA; 1990; (Serr)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	Mature; 27 Aug	CL	MAT	14	< 0.01	RR 92-009B; 72-CA-90-643; [Roper, 1992, PP5/0582]
idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42	0.45	Mature; 27 Aug	CL	MAT	14	< 0.01	idem
Arvin, Kern, CA; USA; 1990; (variety ns)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	Mature; 08 Oct	SiL	MAT	14	< 0.01	RR 92-009B; 81-CA-990-644; [Roper, 1992, PP5/0582]
idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42	0.45	Mature; 08 Oct	SiL	MAT	14	< 0.01	idem

Additional trial information:

RR 92-009B; GLP study. No unusual weather conditions. Weed directed application by ground sprayer. Spray volume 93 L/ha. Plot size 36-341 m^2, with 3 trees/plot. Nuts were picked by hand from the ground. Nuts were cracked and shells discarded. Approximately 1 kg of nutmeats were collected. Ssamples were stored at <-20 °C for max 411 days. Samples were analysed for total fluazifop using GC-MS-MS method RR91-014B with a valid LOQ of 0.01 mg/kg. Samples were not corrected for individual method recoveries (107% at 0.01 mg/kg). Control samples were < 0.01 mg/kg.

Coffee beans

One cGAP for coffee beans is available:

- cGAP from the USA with 2 ×0.42 kg ai/ha with a PHI of 1 day

Trials that could be matched to this cGAPs were summarized.

Table 195 lists trials conducted in the USA (1986, 2008), Brazil (1981). A weed directed spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) at the base of the shrubs was conducted under the conditions listed in Table 195. Results marked with "[QU]" or "[SS]", are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample size was less than the required 1 kg green coffee beans.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2.

The Codex RAC is defined as green coffee beans, which are obtained by drying the fresh coffee berries until a moisture content of 5–6% and removing the hull to get the green coffee beans. Roasted coffee beans is a processed commodity.

- The study performed in Brazil in 1981 did not indicate the commodity type.
- For the studies performed in the USA in 1989, coffee "cherries" were harvested and pulped within 12 hrs. Pulped coffee was allowed to ferment for 12–24 hrs (to allow for removal of mucilage surround the seed). Seeds were then air dried for 2–4 days until the outer skin could be easily removed from the seeds. The clean dried seeds (parchment coffee) was analysed for total fluazifop.
- For the studies performed in the USA in 2011 ripe coffee bean "cherries" were harvested and processed the next day (normal rate) or the second day (exaggerated rate) into green beans. Coffee cherries were dried for approximately 7 hours (50 °C) to moisture content of 5–6% followed by hull removal to produce green beans.

Table 195 Supervised field trials on coffee (green beans), treated with fluazifop-butyl at the base of the shrubs

GREEN COFFEE BEANS Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Kona, HI, USA, 1986 (variety ns)	EC 120 (P) +0.25% NIS	3 (132, 48)	0.28	0.14	immature and ripe berries; 27 Oct	SiCL	MAT	1 14	< 0.1 < 0.1 [a], [SS]	IR-4 PR 03432 (1988); trial ns [Baron, 1988, 471695]
idem	EC 120 (P) +0.25% NIS	3 (132, 48)	1.1	0.57	immature and ripe berries; 27 Oct	SiCL	MAT	1 14	< 0.1 < 0.1 [a], [SS]	idem
Waimanola, HI, USA, 1986 (variety ns)	EC 120 (P) +0.25% NIS	3 (111, 78)	0.28	0.11–0.16	60% ripe berries; 3 Dec	C	MAT	1 15	< 0.1 < 0.1 [a], [SS]	IR-4 PR 03432 (1988); trial ns [Baron, 1988, 471695]
idem	EC 120 +0.25% NIS (P)	3 (111, 78)	1.1	0.44-0.63	60% ripe berries; 3 Dec	C	MAt	1 15	< 0.1 < 0.1 [a], [SS]	idem

GREEN COFFEE BEANS Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Kauai, HI, USA, 2008 (Red caturra)	EC 240 (P) +0.25% NIS	3 (13, 14)	0.28	0.15	Ripe berries; 19 Nov	SiCL	MAT	1	< 0.05 [a]	IR-4 PR 03432 (2011); 08-HI04; [Barney, 2011; PP5_50291] (processing)
idem	EC 240 (P) +0.25% NIS	3 (13, 14)	1.4	0.75	Ripe berries; 19 Nov	SiCL	MAT	1	< 0.05 [a]	IR-4 PR 03432 (2011); 08-HI04; [Barney, 2011; PP5_50291] (processing)
Faz Anelviario Riberto Preto, Brazil; 1981 (Robusta)	EC 250 (rac)	1	1.0	0.25	GS ns 28 Aug	ns	ns	4 7 14 21	< 0.03 < 0.03 < 0.03 < 0.03 [QU] [LOQ=0.05	PP009B122; [Atreya and Upton, 1982; PP9/0633] and RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	2.0	0.5	GS ns 28 Aug	ns	ns	4 7 14 21	< 0.03 < 0.03 < 0.03 < 0.03 [QU] [LOQ=0.05	idem

QU Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[SS] Sample size less than the required 1 kg green coffee beans (0.45-0.90 kg in PR 03432(1))

[a] Results are the mean of 2-3 field samples; individual samples were below LOQ (< 0.1 mg/kg in PR 03432(1988) and < 0.05 mg/kg in PR 03432 (2011))

Additional trial information:

PR 03432 (1988). GLP. No unusual weather conditions. Application by pressurized boom sprayer. Plot size: 8 plants/plot; 3 plots. Spray volume 21 GPA = 200 L/ha. Coffee "cherries" were harvested and pulped within 12 hrs. Pulped coffee was allowed to ferment for 12-24 hrs (to allow for removal of mucilage surround the seed). Seeds were then air dried for 2-4 days until the outer skin could be easily removed from the seeds. The clean dried seeds (parchment coffee; 1-2 lbs = 0.45-0.90 kg per plot) were stored for 175days at -15 °C or lower**.** Samples were analysed for total fluazifop using **HPLC-UV method PPRAM62/2 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent recoveries (62% at 0.1 mg/kg; 70% at 0.5 mg/kg). Control < 0.1 mg/kg.

PR 03432 (2011). GLP. No unusual weather conditions. Application by backpack CO2 sprayer to the ground. Plot size: 40-148 plants. Ripe coffee bean "cherries" were harvested and processed the next day (normal rate) or the second day (exaggerated rate) into green beans (2-3 lbs = 0.9-1.4 kg/sample; normal rate and 12 kg exaggerated rate for processing). Coffee cherries were dried for approximately 7 hours (50 °C) to moisture content of 5-6% followed by hull removal to produce green beans. Samples were stored for 473-934 days (31 months) at -20 °Cor lower. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM62/2 modification C with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent recoveries (81% at 0.05-2.0 mg/kg). Control not stated.

PP009B122. Non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Application at the base of the tree. Coffee berries were left drying on the ground for 10 days after harvest. No information provided on any further processing of the coffee berries (husk removal etc.). Coffee beans were not frozen (storage temperature not stated) for 194-211 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Average concurrent recovery 83% at 0.5-5.0 mg/kg. Control samples not stated.

Caneberries

Blackberries

Three possible cGAPs for blackberries and raspberries are available:

- cGAP from France with 1 ×0.25 kg ai/ha with PHI 42 days (unspecified spray)
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha with PHI 45 days (weed directed spray between bushes)
- cGAP from the UK or Belgium with 1 ×0.38 kg ai/ha before bloom or after harvest (UK:where possible weed directed spray; Belgium unspecified spray)

Trials that could be matched to these cGAPs were summarized.

Table 196 lists trials conducted in the USA (2010) and Germany (1987). An over-the-top spray or a weed directed interrow banded spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 196. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[SS] indicates that the sample sizes were not reported or less than 1 kg fruits.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62/2.

Table 196 Supervised field trials on blackberries (whole fruit), treated with an over-the-top or interrow banded fluazifop-butyl spray

BLACK BERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Over-the-top spray application										
6749 Gleiszellen-Gleishorbach; Germany; 1987; (ns)	EC 125 (P)	1	0.50	0.12	start of flowering; 6 June;	L	RP	62 82	< 0.01 < 0.01 [SS] [LOQ=0.05]	M4779B; Rs8718E3; [Mak and Scott, 1988, PP5/0462]
3119 Bornsen; Germany; 1987; (ns)	EC 125 (P)	1	0.50	0.12	before flowering; 29 May	L	RP	108 122	< 0.01 < 0.01 [SS] [LOQ=0.05]	M4779B; Rs8718B3 [Mak and Scott, 1988, PP5/0462]
Weed directed banded soil spray										
Jackson Springs, NC, USA, 2010 (Kiowa)	EC 250 (P) + Induce	2 (12)	0.41; 0.42	0.21 0.21	black fruit; 10 June	LSa	CH	1	< 0.02, < 0.02, mean < 0.02 [a] [SS]	IR-4 PR 03947; 10-NC10; [Arsenovic and Jolly, 2013, PP5_50556]
Aurora, OR, USA, 2010 (Marion)	EC 250 (P) + Prime Oil	2 (13)	0.42; 0.43	0.13; 0.13	fruiting; 7 July	L	CH	1	< 0.02, < 0.02, mean < 0.02 [a]	IR-4 PR 03947; 10-OR18; [Arsenovic and Jolly, 2013, PP5_50556]
Kingsburg, CA, USA 2010 (Ouachita)	EC 250 (P) + Activator 90	2 (14)	0.42; 0.40	0.15; 0.15	fruiting; 23 June	SaL	CH	1	< 0.02, < 0.02, mean < 0.02 [a]	IR-4 PR 03947; 10-CA57; [Arsenovic and Jolly, 2013,

BLACK BERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
									[SS]	PP5_50556]

[SS] Samples size smaller than the required 1 kg; samples are considered not representative for MRL derivation.
[a] Results came from two replicate field samples/plot; mean is taken for MRL derivation, , if according to cGAP

Additional trial information
M4779B. Non-GLP. Rain 3-4 hours after application when deposit was dry (blackberry E3). Plot size 50 m2, consisting of a hedge of 4 m wide and 12.5 m long along scarp; considered to consist of > 6 bushes/plot. **Spray over head application** using (motor) knapsack sprayer with boom. Spray volume 400 L/ha. Fruits (0.5-0.6 kg) were sampled by hand. Storage at -27 °C for a maximum of 267 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg.** Internal standard recovery (mean 78% at 0.5 mg/kg). Control sample < 0.01 mg/kg.
PR 03947. GLP. Unusual weather conditions did not affect the study.**Soil directed banded spray** using backpack sprayer (NC10). Plot size 6-60 shrubs/plot. Spray volume 21-36 GPA = 196-336 L/ha (blackberries); 20-30 GPA = 187-280 L/ha (raspberries). Fruits (> 1 kg from plot OR18, 0.9 kg from plot NC10, 0.93–0.95 kg from plot CA57) were taken from at least 12 areas within the plot, across or along rows and from high, low, sheltered and exposed positions on the shrubs. Storage at -38- -0.2 °C or lower for a maximum of 679days. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.01A modification B with a valid LOQ of 0.01 mg/kg.** Samples were not corrected for average concurrent method recoveries (86-95% at 0.02-5.0 mg/kg). Control sample < 0.02 mg/kg.

Raspberries

Three possible cGAPs for blackberries and raspberries are available:

- cGAP from France with 1 ×0.25 kg ai/ha with PHI 42 days (unspecified spray)
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha with PHI 45 days (weed directed spray between bushes)
- cGAP from the UK or Belgium with 1 ×0.38 kg ai/ha before bloom or after harvest (UK:where possible weed directed spray; Belgium unspecified spray)

Trials that could be matched to these cGAPs were summarized.

Table 197 lists trials conducted in the USA (2010), Germany (1981), UK (1981, 1984, 1989), Southern France (2000). An over-the-top spray or a weed directed interrow banded spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 197. Results marked with "[WC]", "[QU]", "[SS]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[WC] indicates that the weather affected growing conditions and fruit yield.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

[LOQ = nn] indicates that the results needs to be increased to the LOQ of 0.05 mg/kg valid for HPLC-UV method PPRAM 62, 62/1 or 62/2 or NMR method PPRAM 83.

Table 197 Supervised field trials on raspberries (whole fruit), treated with an over-the-top or interrow banded fluazifop-butyl spray

RASP BERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Over-the-top spray application										
Location ns; UK, 1981 (var ns)	EC 94 (rac)	1	1.5	ns	GS ns date ns	ns	ns	0 3 7	1.2 0.72 0.53 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; UK,1981 (var ns)	EC 250 (rac)	2	1.0	ns	GS ns date ns	ns	ns	7 14	1.3 0.53 [QU] [cntrl = 0.04] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	68 75	< 0.03 0.09 [QU] [CT] [cntrl = 0.04] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	3.0	ns	GS ns date ns	ns	ns	75	0.06 [QU] [CT] [cntrl = 0.04] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
7545 Hofen ad Enz (Nordscharzwald); Germany; 1987 (forest raspberries)	EC 125 (P)	1	0.50	0.25	flower buds developed; 12 June	L	RP	59 75	< 0.01 < 0.01 [SS] [LOQ=0.05]	M4779B; Rs8718E2; [Mak and Scott, 1988, PP5/0462]
2059 Büchen, Germany, 1987 (forest raspberries)	EC 125 (P)	1	0.50	0.25	before flowering; 11 May	Sa	RP	71 81	< 0.01 < 0.01 [SS] [LOQ=0.05]	M4779B; Rs8718B2; [Mak and Scott, 1988, PP5/0462]
Weed directed banded soil spray or spray at the base of the shrubs										
Holt, MI, USA, 2010 (Heritage)	EC 250 (P) + Activator 90	2 (15)	0.42 0.43	0.23; 0.23	fruiting; 8 Sept	LSa	CH	1	0.022, 0.070, mean 0.046 [a] [SS], [WC]	IR-4 PR 03947; 10-MI15; [Arsenovic and Jolly, 2013, PP5_50556]
Aurora, OR, USA, 2010 (Willamette)	EC 250 (P) + Prime oil	2 (16)	0.41 0.43	0.22; 0.22	ripe and green fruit; 17 June	L	MAT	1	< 0.02, < 0.02, mean < 0.02 [a] [SS]	IR-4 PR 03947; 10-OR16; [Arsenovic and Jolly, 2013, PP5_50556]
Aurora, OR, USA, 2010 (Willamette)	EC 250 (P) + Prime oil	2 (14)	0.43 0.42	0.15; 0.15	fruiting: red and green fruit; 22 June	L	MAT	0	< 0.02, < 0.02, mean < 0.02 [a]	IR-4 PR 03947; 10-OR17; [Arsenovic and Jolly,

RASP BERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
									[SS]	2013, PP5_50556]
idem	EC 250 (P) + Prime oil	2 (14)	0.43 0.42	0.15; 0.15	fruiting: red and green fruit; 22 June	L	MAT	1	< 0.02, < 0.02, mean < 0.02 [a]	idem
idem	EC 250 (P) + Prime oil	2 (14)	0.43 0.42	0.15; 0.15	fruiting: red and green fruit; 22 June	L	MAT	2	< 0.02, < 0.02, mean < 0.02 [a]	idem
idem	EC 250 (P) + Prime oil	2 (14)	0.43 0.42	0.15; 0.15	fruiting: red and green fruit; 22 June	L	MAT	4	< 0.02, < 0.02, mean < 0.02 [a]	idem
idem	EC 250 (P) + Prime oil	2 (14)	0.43 0.42	0.15; 0.15	fruiting: red and green fruit; 22 June	L	MAT	7	< 0.02, < 0.02, mean < 0.02 [a]	idem
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	17 25 31	< 0.03 < 0.03 < 0.03 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Alyth, Scotland; UK; 1984; (Malin Jewel)	EC 125 (P)	1	0.38	0.19	pre-flowering; 18 May	L	H1	61	0.03 [SS] [LOQ=0.05]	M3847B; 14R84SAI28; [Dick, 1984, PP5/0488]
Kirriemuir, Scotland; UK; 1984; (Glen Clova)	EC 125 (P)	1	0.38	0.19	pre-flowering; 18 May	L	H2 H2	61 62	< 0.03 < 0.03 [SS] [LOQ=0.05]	M3847B; 14R84SAI29 and 14R84SAI30 ; [Dick, 1984, PP5/0488]
Forfar, Scotland; UK; 1984; (Malin Jewel)	EC 125 (P)	1	0.38	0.19	pre-flowering; 18 May	L	H2	62	< 0.03 [SS] [LOQ=0.05]	M3847B; 14R84SAI31; [Dick, 1984, PP5/0488]
Forfar, Scotland; UK; 1984; (Glen Clova)	EC 125 (P)	1	0.38	0.19	pre-flowering; 18 May	L	H3	66	< 0.03 [SS] [LOQ=0.05]	M3847B; 14R84SAI32; [Dick, 1984, PP5/0488]
Alyth, Scotland; UK; 1989; (Moya)	EC 125 (P) + Agral	1	0.35	ns	pre-flowering; 10% crop cover; 19 May	ns	RP	56	< 0.05	M5320B; GB18-89-S131; [Jones, 1991, PP5/0193]
idem	EW 125 (P) + Agral	1	0.38	ns	pre-flowering; 10% crop cover; 19 May	ns	RP	56	<u>< 0.05</u>	idem
idem	EW 250 (P) +	1	0.38	ns	pre-flowering; 10% crop cover;	ns	RP	56	< 0.05	idem

RASP BERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	Agral				19 May					
Alyth, Scotland; UK; 1989; (Glen Glova)	EC 125 (P) + Agral	1	0.35	ns	pre-flowering; 10% crop cover; 19 May	ns	RP	56	< 0.05	M5320B; GB18-89-S132; [Jones, 1991, PP5/0193]
idem	EW 125 (P) + Agral	1	0.38	ns	pre-flowering; 10% crop cover; 19 May	ns	RP	56	< 0.05	idem
idem	EW 250 (P) + Agral	1	0.38	ns	pre-flowering; 10% crop cover; 19 May	ns	RP	56	< 0.05	idem
Dunières sur Eyrieux; South-East; S-France; 2000 (Meeker, hedge type)	EC 125 (P)	1	0.38	0.12	BBCH55; 2 May	Sa	85	45	< 0.01 [SS]	RJ3210B; FR53-00-S760; [Mason and Atger, 2001, PP5/1111]
Solferino; South-West; S-France; 2000 (Héritage, shrub type)	EC 125 (P)	1	0.38	0.12	BBCH55; 6 July	Sa	85	49	< 0.01 [SS]	RJ3210B; FR92-00-S756; [Mason and Atger, 2001, PP5/1111]

Growth Stage at Harvest (GSH): H1 = 1st pick harvest, H2 = 2nd pick harvest, H3 = 3rd pick harvest

[WC] Weather conditions affected growing conditions and fruit yield; samples not representative for MRL setting

[SS] Sample size not stated or smaller than the required 1 kg; samples not representative for MRL setting

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

[a] Results came from replicate field samples, average value is taken for MRL derivation, , if according to cGAP

Additional trial information

PR 03947. GLP. Unusual weather conditions did affect trial MI15. Due to the drip line not functioning and the resulting very dry soil reduced the fruit yield. Soil directed banded spray using backpack sprayer (NC10). Plot size 16-50 shrubs/plot. Spray volume 20-30 GPA = 187-280 L/ha (raspberries). Fruits (at least 1.0 kg at DAT 1, 2, 4, 7 in plot OR17; 0.58-0.64 kg in plot MI15; 0.36-0.52 kg in plot OR16; 0.82–0.84 kg at DAT 0 in plot OR17) were taken from at least 12 areas within the plot (along rows) and from high, low, sheltered and exposed positions on the shrubs. Storage at -38 °C for a maximum of 679days. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.01A modification B with a valid LOQ of 0.01 mg/kg.** Samples were not corrected for average concurrent method recoveries (86-95% at 0.02-5.0 mg/kg). Control sample < 0.02 mg/kg.

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. In Germany bushes were sprayed at the base of the trees, in the UK this is an over the top application. Growth stage at harvest not stated. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.05 mg/kg.

M3847B Non-GLP. No unusual weather conditions. Plot size 10-14 m row, considered to consist of at least 6 bushes. Application around the base of the canes by CO2 knapsack with boom. Spray volume 200 L/ha. Samples of ripe fruits were sampled by hand (1st, 2nd or 3rd pick). Sample sizes were not stated. Samples were stored at -20 °C (storage time not stated but less than 12 months). Sample sere analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Internal standard recoveries (mean 78%). Control samples < 0.03 mg/kg.

M5320B. Non-GLP No unusual weather conditions. Plot size 5 m, considered to consist of at least 6 bushes (2 plants/m). Application around the base of the cane using a boom with one nozzle. Spray volume not stated. Adjuvant AGRAL was added. Fruits were samples by hand from tip, middle and bottom of the canes. Field samples were 1.0 kg ripe fruit. Storage time 6 months at -18 °C. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Internal standard recovery (mean 77% at 0.5 mg/kg). Control sample < 0.05 mg/kg.

RJ3210B. GLP. No unusual weather conditions. Plot size: 10 m row, considered to consist of at least 6 bushes (2 plants/m). Soil spray on either side of a row of raspberries. Spray volume 300 L/ha. Berries (0.5 kg) were taken by hand systematically from across the plots. Storage at -18 °C for 88-157days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg** Samples were not corrected for individual concurrent method recoveries (101-102% at 0.01–0.05 mg/kg. Control samples < 0.01 mg/kg.

M4779B. Non-GLP. Rain shower 3 hrs after application (raspberry E2). Plot size 440-2400 m2, considered to consist of more than 6 bushes/plot. Spray over head application using (motor knapsack mist blower. Spray volume 200 L/ha. Fruits (0.50-0.65 kg) were sampled by hand. Storage at -27 °C for a maximum of 284 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg.** Internal standard recovery (mean 78% at 0.5 mg/kg). Control sample < 0.01 mg/kg.

Bush berries

Bilberries

One cGAPs for Vaccinium berries (i.e. bilberries, blueberries) is available:

- cGAP from France with 1 ×0.25 kg ai/ha with PHI 42 days (unspecified spray)

Trials that could be matched to this cGAP were summarized.

Table 198 lists trials conducted in Germany (1987). An over-the-top spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 198. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample sizes were not reported or less then 1 kg.

Table 198 Supervised field trials on bilberries (whole fruit), treated with an over-the-top fluazifop-butyl spray

BILBERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
6729 Wörth-Schaidt (Bienwald); Germany; 1987; (forest bilberries	EC 125 (P)	1	0.50	0.12	end of flowering; 8 May	Sa	R	49 64 75 97	2.2 1.1 0.65 0.48 [SS]	M4779B; Rs8718E1; [Mak and Scott, 1988, PP5/0462]
2124 Hohenesch, Amelinghausen, Germany, 1987 (forest bilberries)	EC 125 (P)	1	0.50	0.12	start of flowering; 8 May	SaL	R	67 81	0.14 0.18 [SS]	M4779B; Rs8718B1; [Mak and Scott, 1988, PP5/0462]

[SS] Sample size smaller than the required 1 kg; samples not representative for MRL setting

Additional trial information

M4779B. Non-GLP. No unusual weather conditions. Plot size 400-620 m2, considered to be > 6 bushes/plot. Spray over head application using (motor) knapsack sprayer with boom. Spray volume 400 L/ha. The use of an adjuvant was not reported. Fruits (0.5 kg berries) were sampled by hand. Storage at -27 °C for a maximum of 309 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg.** Internal standard recovery (mean 78% at 0.5 mg/kg). Control sample < 0.01 mg/kg.

Blueberries

One cGAP for Vaccinium berries (i.e. bilberries, blueberries) is available:

- cGAP from France with 1 ×0.25 kg ai/ha with PHI 42 days (unspecified spray)

No trials could be matched to this cGAP.

Trials from the USA (2010) were available on lowbush and highbush blueberries with 2 × 0.41–0.46 kg ai/ha with harvest at 0-6 DAT [Arsenovic and Jolly, PP5_50557, report IR-4 PR 02083]. These trials were not summarized, because they would not assist in MRL setting.

Currants

Three possible cGAPs for currants are available:

- cGAP from France with 1 ×0.25 kg ai/ha with PHI 42 days (unspecified spray)
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha with PHI 45 days (weed directed spray between bushes)
- cGAP from the UK and Belgium with 1 ×0.38 kg ai/ha and application before bloom or after harvest (UK: weed directed spray, Belgium unspecified spray)

Trials that could be matched to these cGAPs were summarized.

Table 199 lists trials conducted in Germany (1981) and the UK (1980, 1981, 1984-1985, 1989). An over-the-top spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 199. Results marked with "[QU]", "[SS]", or "[CT]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2.

Table 199 Supervised field trials on currants (whole fruit), treated with an over-the-top fluazifop-butyl spray

CURRANTS; Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Location ns; Germany, 1981, (var ns; red currants)	EC 125 (P)	1	1.5	ns	GS ns date ns	ns	ns	21 28 38	< 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; UK, 1980, (var ns; black currants)	EC 94 (rac)	1	1.0	ns	GS ns date ns	ns	ns	0 3 7	3.2 0.83 0.80 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; UK, 1981, (var ns; black currants)	EC 125 (P)	1	1.0	ns	GS ns date ns	ns	ns	7 14 24 38	0.46 0.20 0.05 [CT] 0.12 [CT]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]

CURRANTS; Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
									[QU], [cntrl = 0.04] [LOQ=0.05]	
Isle of Ely; Norfolk UK; 1984; (black: Wellington)	EC 125 (P)	1	0.38	0.15	leaves unfolding; 29 March	SaL	CH	104	< 0.03 [SS] [LOQ=0.05]	M3870B; 13R/84/EA68and 13R/84/EA69; [Dick, 1984, PP5/0458]
idem	EC 125 (P)	1	0.38	0.15	leaves unfolding; 29 March	SaL	10-14 days before CH	104	< 0.03 [SS] [LOQ=0.05]	M3870B; 13R/84/EA69; [Dick, 1984, PP5/0458]
Milton; Cambridgeshire; UK; 1984 (black: Ben Loman)	EC 125 (P)	1	0.38	0.15	leaves unfolding; 5 April	SaLC	CH	97	< 0.03 [SS] [LOQ=0.05]	M3870B; R13/84/EA70; [Dick, 1984, PP5/0458]
Alcester, Warwickshire; UK; 1984; (black: Jet)	EC 125 (P)	1	0.38	0.19	1st early bud; 15% crop cover; 19 April;	LC	MAT	97	< 0.03 [SS] [LOQ=0.05]	M3870B; 13R/84/WM1; [Dick, 1984, PP5/0458]
Isle of Ely; Norfolk; UK; 1984-1985; (black: Wellington)	EC 125 (P)	2 (349)	0.38 0.38	0.15; 0.19	leaves just unfolding; 13 March; 2-3% crop cover	SaL	7 days before CH	131	< 0.03; < 0.03 [a] [SS] [LOQ=0.05]	M4197B; 13R/84/EA68and 13R/84/EA69; [Harradine and Pay, 1986, PP5/0457]
Milton, Cambridgeshire; UK; 1984-1985; (black: Ben Loman)	EC 125 (P)	2 (342)	0.38 0.38	0.19 0.19	early bud burst; 13 March	SaLC	CH	127	< 0.03 [SS] [LOQ=0.05]	M4197B; R13/84/EA70; [Harradine and Pay, 1986, PP5/0457]
Alcester, Warwickshire; UK; 1984-1985; (black: Jet)	EC 125 (P)	2 (378)	0.38 0.38	0.19 0.19	15% flower set; 2 May	LC	CH	85	0.04 [SS] [LOQ=0.05]	M4197B; 13R/84/WM1; [Harradine and Pay, 1986, PP5/0457]
Cranbrook, Kent; UK; 1989; (black: Baldwin)	EC 125 (P) + Agral	1	0.38	0.12	bud burst 30 March	CL	90% ripe fruit	103	< 0.05	M5091B; GB52-89-S111; [Bunker and Jones, 1991, PP5/0460]
idem	EW 125 (P) + Agral	1	0.38	0.12	bud burst; 30 March	CL	90% ripe fruit	103	< 0.05	idem
idem	EW 250 (P) + Agral	1	0.38	0.12	bud burst; 30 March	CL	90% ripe fruit	103	< 0.05	idem
Cranbrook, Kent; UK; 1989; (black: Ben More)	EC 125 (P) + Agral	1	0.38	0.12	bud burst; 13 April	CL	75% ripe fruit	89	< 0.05	M5091B; GB52-89-S112; [Bunker and Jones, 1991, PP5/0460]
idem	EW 125	1	0.38	0.12	bud burst; 13 April	CL	75% ripe	89	< 0.05	idem

CURRANTS; Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	(P) + Agral						fruit			
idem	EW 250 (P) + Agral	1	0.43	0.14	bud burst; 13 April	CL	75% ripe fruit	89	< 0.05	idem

[SS] Samples sizes were not stated; results are not considered representative for MRL derivation.

QU Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

[a] Two replicate plots, the highest value is taken for MRL derivation if according to cGAP

Additional trial information

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. It's not clear how the bushes were sprayed. The growth stage at application is not stated, but total fluazifop residues are most likely caused by an over the top spray when bushes had flowers and/or fruits. The use of an adjuvant not reported. Storage conditions not stated. Samples were analysed for total fluazifop using HPLC-UV method **PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.05 mg/kg (Germany) or < 0.01–0.04 mg/kg (UK 1981).

M3870B Non-GLP. No unusual weather conditions. Plot size 5 bushes. Bushes were sprayed over the top by CO2 knapsack (EA 68/69/70) or handheld CO2 knapsack (WM1). Spray volume 200-250 L/ha. No adjuvant added. Fruits were sampled by hand. Sample sizes were not stated. Samples were stored at -20 °C (storage time not stated but less than 12 months). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Internal standard recoveries (mean 73%). Control < 0.03 mg/kg as fluazifop.

M4197B Non-GLP. The same bushes as in M3870B were treated again in the following year; so only 1 treatment in 1985. There was light rain 1 hr post application at trials EA68, EA69 and EA70. Plot size 5 bushes, except 13R/84/WM1 where 40 plants (2 rows of 20 m with 1plant/m) were sprayed. Bushes were sprayed over the top by handheld CO2 boom (EA 68/69), CO2 knapsack sprayer (EA 70) or CO2 single man knapsack with boom (WM1). Spray volume 200-250 L/ha. No adjuvant added. Fruits were sampled by hand (sample size not stated). Samples were stored at -20 °C (storage time not stated but less than 12 months). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Internal standard recoveries (mean 75%). Control < 0.03 mg/kg.

M5091B Non-GLP. No unusual weather conditions. Plot size 4 bushes (S111 = 3.3 m2/plant, plot size 12.5 m2; S112 = 2.7 m2/plant, plot size 10.8 m2) Bushes were sprayed with 1 pass over the top by CO2 knapsack plus boom. Spray volume 300 L/ha. Adjuvant (AGRAL) added. Samples (1 kg) were sampled at random by hand from all parts of the bushes. Samples were stored at -18 °C (storage time not stated but less than 12 months). Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Results were corrected for external standard recoveries (mean 98%);uncorrected results are not reported. Control < 0.05 mg/kg.

Gooseberries

Three possible cGAPs for gooseberries are available:

- cGAP from France with 1 ×0.25 kg ai/ha with PHI 42 days (unspecified spray)
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha with PHI 45 days (weed directed spray between bushes)
- cGAP from the UK and Belgium with 1 ×0.38 kg ai/ha and application before bloom or after harvest (UK: weed directed spray; Belgium unspecified spray).

Trials that could be matched to these cGAPs were summarized.

Table 200 lists trials conducted in the UK (1981, 1984-1985, 1989). An over-the-top spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under

the conditions listed in Table 200. Results marked with "[QU]", "[RF]" or "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[RF] indicates that rainfall occurred within 1 hr after application.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2.

Table 200 Supervised field trials on gooseberries (whole fruit), treated with an over-the-top fluazifop-butyl spray

GOOSE BERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Location ns; UK,1981 (var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	7 14 38	1.1, 1.8 [a] 0.85, 1.4 [a] 0.11 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; UK,1981 (var ns)	EC 94 (rac)	1	2.4	ns	GS ns date ns	ns	ns	0 3 8	2.2 3.1 1.5 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 94 (rac)	1	4.5	ns	GS ns date ns	ns	ns	0 3 8	2.6 2.0 2.7 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Isle of Ely, Norfolk; UK; 1984; (Carless)	EC 125 (P)	1	0.38	0.15	leaves unfolding; 2-3% crop cover; 29 March	SaL	CH	81	< 0.03; < 0.03 [SS] [a] [LOQ=0.05]	M3869B; 11R/84/EA66; 11R/84/EA67; [Dick, 1984, PP5/0473]
Tilehurst, East Sussex; UK; 1984; (Carless)	EC 125 (P)	1	0.38	0.13	pre-flowering; 30% crop cover; 17 April	L	MAT	58	< 0.03 [SS] [LOQ=0.05]	M3869B; KGP 1; [Dick, 1984, PP5/0473]
Maidstone, Kent; UK; 1984; (Carless)	EC 125 (P)	1	0.38	0.13	pre-flowering; 40% crop cover; 17 April	L	MAT	58	< 0.03 [SS] [LOQ=0.05]	M3869B; KGP 2; [Dick, 1984, PP5/0473]
Ashford, Kent; UK; 1984; (Carless)	EC 125 (P)	1	0.38	0.13	pre-flowering; 50% crop cover; 16 April	L	MAT	57	< 0.03 [SS] [LOQ=0.05]	M3869B; KGP 3; [Dick, 1984, PP5/0473]
Isle of Ely, Norfolk; UK; 1985; (Carless)	EC 125 (P)	1	0.38	0.19	early bud burst; 2-3% crop cover; 13 March	SaL	CH	97	< 0.02 [SS] [LOQ=0.05]	M4186B; 11R/84/EA66; [Harradine, 1986, PP5/0472]
idem	EC 125 (P)	1	0.38	0.19	leaves unfolding; 2-3% crop cover;	SaL	CH	88	< 0.02 [SS] [RF] [LOQ=0.05]	M4186B; 11R/84/EA67 ; [Harradine, 1986, PP5/0472]

GOOSE BERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					22 March					
Pluckley, Kent; UK; 1984-1985; (Carless)	EC 125 (P)	3 (163, 195)	0.38 0.38 0.38	0.13 0.13 0.13	pre-flowering; 45% crop cover; 9 Apr 1985	SaL	CH	62	< 0.02 [SS] [LOQ=0.05]	M4186B; R119/85 (ex 11R/84/KGP3); [Harradine, 1986, PP5/0472]
Peckham, Kent; UK; 1984-1985; (Carless)	EC 125 (P)	2 (195)	0.38 0.38	0.13 0.13	pre-flowering; 40% crop cover; 9 Apr 1985	L	CH	62	< 0.02 [SS] [LOQ=0.05]	M4186B; R119/85 (ex 11R/84); [Harradine, 1986, PP5/0472]
East Farleigh, Kent; UK; 1984-1985; (Carless)	EC 125 (P)	2 (183)	0.38 0.38	0.13 0.13	pre-flowering; 40% crop cover; 9 April	L	CH	62	< 0.02 [SS] [LOQ=0.05]	M4186B; R119/85 (ex 11R/84); [Harradine, 1986, PP5/0472]
Tilehurst, Sussex; UK; 1989; (Carless)	EC 125 (P) + Agral	1	0.25	0.084	flower buds just visible; 30 March	CL	MAT	63	< 0.05	M5092B; GB50-89-S121; [Bunker and Jones, 1991, PP5/0474]
idem	EW 125 (P) + Agral	1	0.27	0.090	flower buds just visible; 30 March	CL	MAT	63	< 0.05	idem
idem	EW 250 (P) + Agral	1	0.27	0.091	flower buds just visible; 30 March	CL	MAT	63	< 0.05	idem
Rochester, Kent; UK; 1989; (Leveller)	EC 125 (P) + Agral	1	0.38	0.12	flower buds just visible; 31 March	CL	MAT	87	< 0.05	M5092B; GB50-89-S122; [Bunker and Jones, 1991, PP5/0474]
idem	EW 125 (P) + Agral	1	0.38	0.12	flower buds just visible; 31 March	CL	MAT	87	< 0.05	idem
idem	EW 250 (P) + Agral	1	0.38	0.12	flower buds just visible; 31 March	CL	MAT	87	< 0.05	idem

[SS] Sample sizes were not stated; results are considered not representative for MRL derivation.

QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[RF] Rainfall within 1 hr after application; results are considered not representative for MRL derivation

[a] Results came from 2 replicate plots; the highest value is taken for MRL derivation if according to cGAP

Additional trial information

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Spray volume not stated.. It's not clear how the bushes were sprayed . The growth stage at application is not stated, but total fluazifop residues are most likely caused by an over the top spray when bushes had flowers and/or fruits. The use of an adjuvant not reported. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.05 mg/kg.

M3869B Non-GLP. No unusual weather conditions. Plot size 6 bushes (EA66 and EA67). Trial KGP1 with 80m2, trial KGP2 with 20 m2 and trial KGP3 with 60 m2 are considered to consist of at least 6 bushes (assuming 3 m2/plant). Spray over the top of the bushes by CO2 knapsack sprayer (EA 66/67), CO2 knapsack boom sprayer (KGP 1), CO2 knapsack sprayer with handheld boom (KGP 2/3). Spray volume 250-280 L/ha. No adjuvant added. Fruits were sampled by hand. Sample sizes were not stated. Samples were stored at -20 °C (storage time not stated but less than 12 months). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Internal standard recoveries (mean 77%). Control < 0.03 mg/kg.

M4186B Non-GLP. At trial EA67 rain fell within 1 hour after application and therefore results at DAT=88 are considered not reliable. Plot size 6 bushes (EA66andEA67). TrialsR119/85 with 80-120 m2 are considered to consist of at least 6 bushes with 2-4.5 m2/plant. Bushes were sprayed over the top by CO2 one man boom (EA 66/67) or CO2 knapsack boom sprayer (R119/85). Spray volume 250-280 L/ha. No adjuvant added. Fruits were sampled by hand. Sample sizes were not stated. Samples were stored at -20 °C (storage time not stated but less than 12 months). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Internal standard recoveries (mean 97%). Control < 0.02 mg/kg.

M5092B Non-GLP. No unusual weather conditions. Plot size 6 bushes for S121 (20m2) and plot size < 6bushes for S122 (10 m2), assuming 2 m2/plant. Spray by one pass over the top of the bushes by CO2 knapsack boom. Spray volume 300 L/ha. An adjuvant (AGRAL) was added Ripe berries (1 kg) were picked by hand at random from whole plot. Samples were stored at -18°C (storage time not stated but less than 12 months). Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Internal standard recoveries (mean 99%). Control samples < 0.05 mg/kg.

Low growing berries

Strawberries

Two possible cGAPs for strawberries are available:

- cGAP from the Netherlands and France with 1 ×0.38 kg ai/ha with PHI 42 days
- cGAP from the UK and Belgium with 1 ×0.38 kg ai/ha and application before bloom or after harvest

Trials that could be matched to these cGAPs were summarized.

Table 201 lists trials conducted in Germany (1981), Sweden (1988), UK (1989, 1994), Southern France (1999, 2004), Italy (1999, 2004, 2012, 2013), Spain (2012, 2013). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 201. Results marked with "[QU]" or "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2.

Additional trials from the UK (1980) were available on strawberries with 1 ×1.0–1.5–2.0 kg ai/ha and harvest at 0–8, 52 or 56–110 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. These trials were summarized in the metabolism section, because only the fluazifop-butyl and free fluazifop acid (II) residues were analysed.

Additional trials from the Netherlands (1981) were available with 1 × 0.62–0.75 kg ai/ha and harvest at 32 and 48 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Italy (1981) were available with 1 × 0.25–0.50–0.75 kg ai/ha and harvest 30 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from the UK (1981) were available with 1 × 1.0 kg ai/ha and harvest at 7–14, 34 or 46–47 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from the USA (2012) were available with 1 × 0.28–0.35 kg ai/ha and harvest at 0–21 DAT [Arsenovic, 2014, PP5_50553, report IR-4 PR A2085]. These trials were not summarized, because they would not assist in MRL setting.

Table 201 Supervised field trials on strawberries (whole fruit), treated with a broadcast foliar fluazifop-butyl spray

STRAWBERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	37 41 50	< 0.03, 0.05, 0.04, 0.04 < 0.03, < 0.03 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	28 42 56	0.13, 0.19 0.06, 0.06, < 0.03, < 0.03 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Sibbhult, Skåne; Sweden; 1988; (Senga Sengana)	EC 125 (P)	1	0.38	0.19	GS ns; 9 May	Sa	ns	41	0.05 [SS]	M4883B; SE10-88-H862; [Armstrong and Mak, 1989, PP5/0194]
Arkelstorp, Skåne; Sweden; 1988; (Zephyr)	EC 125 (P)	1	0.38	0.19	GS ns; 9 May	ns	ns	41	0.05 [SS]	M4883B; SE10-88-H861; [Armstrong and Mak, 1989, PP5/0194]
Abington, Cambridgeshire; UK; 1989; (Hapil)	EW 250 (P)	1	0.38	ns	3-4 leaves; 40% crop cover; 18 April	SaL	CH	61	< 0.05	M5319B; GB51-89-S101; [Cullen and Jones, 1991, PP5/0195]
idem	EW 125 (P)	1	0.38	ns	3-4 leaves; 40% crop cover; 18 April	SaL	CH	61	< 0.05	idem
idem	EC 125 (P)	1	0.38	ns	3-4 leaves; 40% crop cover; 18 April	SaL	CH	61	< 0.05	idem
Marden, Kent; UK; 1989; (Elsanta)	EW 250 (P)	1	0.38	ns	early flower; 4 May	SaL; PC	CH	53	< 0.05	M5319B; GB52-89-S101; [Cullen and Jones, 1991, PP5/0195]
idem	EW 125 (P)	1	0.38	ns	early flower; 4 May	SaL; PM	CH	53	< 0.05	idem
idem	EC 125 (P)	1	0.38	ns	early flower; 4 May	SaL; PC	CH	53	< 0.05	idem
Yalding, Kent; UK; 1989; (Hapil)	EW 250 (P)	1	0.38	ns	pre-flower; 4 May	L; PC	CH	41	0.05	M5319B; GB52-89-S102; [Cullen and Jones, 1991,

STRAWBERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
										PP5/0195]
idem	EW 125 (P)	1	0.38	ns	pre-flower; 4 May	L; PC	CH	41	0.07	idem
idem	EC 125 (P)	1	0.38	ns	pre-flower; 4 May	L; PC	CH	41	0.06	idem
Maidstone, Kent; K; 1989; (Domanil)	EW 250 (P)	1	0.42	ns	buds at plant base; 8 May	CL; SM	CH	52	< 0.05	M5319B; GB52-89-S103; [Cullen and Jones, 1991, PP5/0195]
idem	EW 125 (P)	1	0.42	ns	buds at plant base; 8 May	CL; SM	CH	52	< 0.05	idem
idem	EC 125 (P)	1	0.42	ns	buds at plant base; 8 May	CL; SM	CH	52	< 0.05	idem
Maidstone, Kent; UK; 1994; (Elsanta)	EC 125 (P) + 0.1% Agral	1	0.38	0.19	flowering just started; 45% crop cover; 9 May	SiL	CH	42	0.06	RJ1817B; GB52-94-S181; [Bolygo *et al*., 1995, PP5/0196]
idem	EC 125 (P) + 0.5% Out put	1	0.38	0.19	flowering just started; 45% crop cover 9 May	SiL	CH	42	0.09	idem
idem	EW 250 (P) + 0.1% Agral	1	0.38	0.19	flowering just started; 50% crop cover 9 May	SiL	CH	42	0.09	idem
idem	EW 250 (P) + 0.5% Out put	1	0.38	0.19	flowering just started; 50% crop cover; 9 May	SiL	CH	42	0.12	idem
idem	EW 250 (P) + 0.5% TF8035	1	0.38	0.19	flowering just started; 55% crop cover; 9 May	SiL	CH	42	0.10	idem
Marden, Kent; UK; 1994; (Evita)	EC 125 (P) + 0.1% Agral	1	0.38	0.19	flower buds removed; 30% crop cover; 31 May	SiCL	CH	45	< 0.05	RJ1817B; GB52-94-S182; [Bolygo *et al*., 1995, PP5/0196]
idem	EC 125 (P) + 0.5% Out put	1	0.38	0.19	flower buds removed; 30% crop	SiCL	CH	45	< 0.05	idem

STRAWBERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					cover; 31 May					
idem	EW 250 (P) + 0.1% Agral	1	0.38	0.19	flower buds removed; 30% crop cover; 31 May	SiCL	CH	45	< 0.05	idem
idem	EW 250 (P) + 0.5% Out put	1	0.38	0.19	flower buds removed; 30% crop cover; 31 May	SiCL	CH	45	< 0.05	idem
idem	EW 250 (P) + 0.5% TF8035	1	0.38	0.19	flower buds removed; 30% crop cover; 31 May	SiCL	CH	45	< 0.05	idem
Bridewater, Somerset; UK; 1994; (Pegasus)	EC 125 (P) + 0.1% Agral	1	0.38	0.19	flower buds visible; 40% crop cover; 26 April	SaCL	CH	55	0.05	RJ1817B; GB14-94-S181; [Bolygo *et al*., 1995, PP5/0196]
idem	EC 125 (P) + 0.5% Out put	1	0.38	0.19	flower buds visible; 40% crop cover; 26 April	SaCL	CH	55	0.07	idem
idem	EW 250 (P) + 0.1% Agral	1	0.38	0.19	flower buds visible; 40% crop cover; 26 April	SaCL	CH	55	0.07	idem
idem	EW 250 (P) + 0.5% Out put	1	0.38	0.19	flower buds visible; 40% crop cover; 26 April	SaCL	CH	55	0.11	idem
idem	EW 250 (P) + 0.5% TF8035	1	0.38	0.19	flower buds visible; 40% crop cover; 26 April	SaCL	CH	55	0.10	idem
Yeovil, Somerset; UK; 1994; (Elsanta)	EC 125 (P) + 0.1% Agral	1	0.38	0.19	flower buds visible; 45% crop cover; 28 April	LSa	CH	57	0.05	RJ1817B; GB14-94-S182; [Bolygo *et al*., 1995, PP5/0196]
idem	EC 125 (P) + 0.5% Out put	1	0.38	0.19	flower buds visible; 45% crop	LSa	CH	57	0.08	idem

STRAWBERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					cover; 28 April					
idem	EW 250 (P) + 0.1% Agral	1	0.38	0.19	flower buds visible; 45% crop cover; 28 April	LSa	CH	57	0.12	idem
idem	EW 250 (P) + 0.5% Out put	1	0.38	0.19	flower buds visible; 45% crop cover; 28 April	LSa	CH	57	0.09	idem
idem	EW 250 (P) + 0.5% TF8035	1	0.38	0.19	flower buds visible; 45% crop cover; 28 April	LSa	CH	57	0.11	idem
Cendrieux; Dordogne; S-France; 1999; (Sceascape)	EC 125 (P)	1	0.18	0.075	BBCH 41-56; 29 July	LSa	CH	42	0.01	RJ3074B; AF/4723/ZE/1 [McGill, 2000, PP5/0455]
82370 Reynies; S-France; 2004; (Darcelec)	EC 125 (P)	1	0.18	0.062	BBCH 55; 1 Apr	SaSiL	85	48	0.01	CEMR-2306; AF/7836/SY4; [Kang, 2005, PP5/1438]
82000 Montauban; S-France; 2004 (Darcelec)	EC 125 (P)	1	0.20	0.062	BBCH 55, 1 Apr	LSa	85	47	< 0.01	CEMR-2306; AF/7836/SY5; [Kang, 2005, PP5/1438]
822210 Puygaillard de Lomagne; S-France; 2004; (Naiad)	EC 125 (P)	1	0.19	0.063	BBCH 59, 3 May	C	87	42	0.03	CEMR-2306; AF/7836/SY6; [Kang, 2005, PP5/1438]
30000 Nîmes; S-France; 2012; (Charlotte)	EC 125 (P)	1	0.38	0.19	BBCH 49; 17 May	CL	89	39	0.02	CEMR-5448; SRFR12-010-37HR; [Langridge, 2013, A12791A_10077]
Buttapietra; Veneto; Italy; 1999 (Marmolada)	EC 125 (P)	1	0.18	0.075	BBCH 49; 30 Aug	SiL	CH	42	< 0.01	RJ3074B; AF/4723/ZE/2 [McGill, 2000, PP5/0455]
40050 Bologna; Italy; 2004; (Alba)	EC 125 (P)	1	0.18	0.063	BBCH 55; 8 Apr	CL	87	42	0.01	CEMR-2306; AF/7836/SY1; [Kang, 2005, PP5/1438]
44028 Poggio Renatico; Italy; 2004; (Aroza)	EC 125 (P)	1	0.19	0.062	BBCH 55; 8 Apr	CL	85	48	< 0.01	CEMR-2306; AF/7836/SY2; [Kang, 2005, PP5/1438]
40018 Bologna; Italy; 2004;	EC 125	1	0.19	0.062	BBCH 56;	CL	87	42	< 0.01	CEMR-2306; AF/7836/SY3;

STRAWBERRIES Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Roxana)	(P)				6 Apr					[Kang, 2005, PP5/1438]
12012 Boves; Piedmont; Italy; 2012; (Arosa)	EC 125 (P)	1	0.39	0.11	BBCH 19; 27 Apr	SaL	87	42	0.08	CEMR-5448; SRIT12-1033-37HR; [Langridge, 2013, A12791A_10077]
75020; Scanzano Jonico; Basilicata; Italy; 2012; (Candonga)	EC 125 (P)	1	0.38	0.12	BBCH 49; 3 May	C	87	42	0.11	CEMR-5448; SRIT12-1034-37HR; [Langridge, 2013, A12791A_10077]
23010 Albosaggia; Lombardia; Italy; 2013; (Monterey)	EC 125 (P)	1	0.36	0.093	BBCH19; 30 May	SaL; PC	87	43	0.06	CEMR-6043; DMC-13-14947-IT01; [Kennedy, 2014, A12791B_11992]
23010 Berbenno di Valtellina; Lombardia; Italy, 2013 (Selva)	EC 125 (P)	1	0.36	0.12	BBCH19;	LSa; PC	87	42	0.02	CEMR-6043; DMC-13-14947-IT02; [Kennedy, 2014, A12791B_11992]
Cartaya; Huelva; Spain; 2012; (Amiga)	EC 125 (P)	1	0.41	0.12	BBCH 59; 8 May	Sa	87	28	0.08	CEMR-5448; SRES12-213-37HR; [Langridge, 2013, A12791A_10077]
17441 Brunyola; Cataluna; Spain, 2013; (Albion)	EC 125 (P)	1	0.39	0.11	BBCH49; 5 June	SaCL; SM	87	39	0.02	CEMR-6043; DMC-13-14947-ES03; [Kennedy, 2014, A12791B_11992]
36680 Berres; Galicia; Spain, 2013 (Albion)	EC 125 (P)	1	0.38	0.11	BBCH49; 26 Aug	SaL	87	43	0.06	CEMR-6043; DMC-13-14947-ES04; [Kennedy, 2014, A12791B_11992]

Soil type: PC = black polythene or plastic spreadsheet covering the soil around the plants; SM = straw mulch around the plants

[SS] Sample sizes not stated; results are considered not representative for MRL derivation.

QU Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

Additional trial information

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.03 mg/kg.

M4883B. GLP. No unusual weather conditions. Plot size 36 m2 with at least 12 plants/plot assuming 3 plants/m2. Spray application using a field trial sprayer. Spray volume 200 L/ha. Berries were harvested by hand. Sample sizes not stated. Storage at -18 °C for a maximum of 134 days. Samples were analysed for total fluazifop using **HPLC-UV method**

PPRAM 62/2 with a valid LOQ of 0.05 mg/kg. Mean internal standard recovery 95% at 0.2 mg/kg. Control samples were < 0.01 mg/kg.

M5319B. GLP. No unusual weather conditions. Plot size not stated (GB51-89-S101, 0.9 m row) or 5.0-7.5 m2 with at least 12 plants/plot assuming 3 plants/m2 (GB52-89-S101, GB52-89-S102, GB-52-89-103). Spray application with one pass over the top of the crop by CO2 knapsack sprayer with boom; spray volume not stated. Samples (1 kg) were picked at random from over the whole plot. Storage at -18 °C for a maximum of 259 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery 89% at 0.5 mg/kg. Control samples were < 0.05 mg/kg.

RJ1817B. GLP. No unusual weather conditions. Plot size: at least 12 plants. Spray application by hand-held small plot boom sprayers, spray volume 200 L/ha. Berries (1.0 kg) were taken by hand systematically from across the plots. Calyx was removed in the field. Storage at -15 °C for a maximum of 133 days. Samples were analysed for total fluazifop using **NMR method RAM197/02 with a valid LOQ of 0.05 mg/kg**. Individual internal standard recoveries 71-109% at 0.5 mg/kg. Control samples were < 0.05 mg/kg.

RJ3074B. GLP. Unusual weather conditions had no effect on crop health. Spray application by hand-held boom sprayers, spray volume 250 L/ha. Berries (1.0 kg from 12 plants) were taken by hand systematically from across the plots. Storage at -18 °C for 94-126 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg.** Samples were not corrected for individual concurrent method recoveries (85-91% at 0.05-0.10 mg/kg. Control samples were < 0.01 mg/kg.

CEMR-2306. GLP. No unusual weather conditions. Plot size at least 12 plants (24-60 m2 with 3 plants/m2). Foliar spray application by plot sprayer, spray volume 280-320 L/ha. Fruit (1.0 kg) were taken by hand. Calyx was removed in the field. Storage at -10 °C for a maximum of 361 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg.** Samples were not corrected for individual concurrent method recoveries (64-81% at 0.01–0.10 mg/kg). Control samples were < 0.01 mg/kg.

CEMR 5448. GLP. No unusual weather conditions. Foliar spray application back pack sprayers, spray volume 200-360 L/ha. Berries (1.0-1.4 kg) were taken by hand using a suitable distributive pattern from at least 12-24 plants. Calyx and stalk were discarded. Storage at -18 °C for a maximum of 291 days. Samples reached temperatures of -12, -11, -9 and -1.5 ºC for peaks of less than 3 hrs. This is considered to have no effect on the residue results, since samples remained frozen at all times. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (66-71% at 0.01–0.5 mg/kg). Control samples were < 0.01 mg/kg.

CEMR-6043. GLP. No unusual weather conditions. Plot size 121-333 plants/plot. Broadcast foliar spray by back sprayers, spray volume 290-390 L/ha. Berries (1.0-1.1 kg) were taken by hand randomly from the plots (Italy) or from 12 different areas in the plots (Spain). Stalk and calyx was removed in the field. Storage at -10 °C for a maximum of 248 days. Samples reached temperatures of -14, -10, -8 ºC for peaks of less than 3 hrs. This is considered to have no effect on the residue results, since samples remained frozen at all time. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A, modification A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (95-104% at 0.01–0.5 mg/kg). Control samples were < 0.01 mg/kg.

Assorted tropical and sub-tropical fruits-inedible peel

Pineapple

A GAP for pineapple is not available. Trials from South Africa (1980) were available on pineapple with an application of 1 × 3.0–6.0 kg ai/ha and harvest at 300 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. As the manufacturer was not seeking to have maximum residue levels estimated on pineapple, the available studies on pineapple were not summarized.

Bulb vegetables

Dry harvested bulb onions

Four possible cGAPs for dry harvested bulb onions are available:

- cGAP from the USA with 2 ×0.42 kg ai/ha with PHI 45 days
- cGAP from Belgium with 2 ×0.38 kg ai/ha with PHI 28 days for onions, shallots and garlic
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha with PHI 28 days for onions and shallots
- cGAP from Brazil with 1 × 0.25 kg ai/ha with PHI 28 days

Trials that could be matched to these cGAPs were summarized.

Table 202 lists trials conducted in the USA (1984, 1986), Brazil (2011), UK (1984, 1989), Netherlands (1984, 1985) and Spain (1987, 1997, 1998). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 202. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[SS] indicates that the sample sizes were not reported or less than 12 plants or 2 kg.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2 or NMR method PPRAM 83.

Trials were conducted with various types of onions: bulb onions, Japanese onions and silverskin onions. Onions were sown and harvested in the same year (one-year varieties) or sown in the first year, planted again in the second year and harvested in the second year (two-year varieties). Onions were harvested either in a green stage or when leaves started to senesce. Leaves were removed and only the bulbs were analysed.

Additional trials from Canada (1980) were available with 1 × 0.50 kg ai/ha and harvest at 94–105 DAT, [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from Italy (1981) were available with 1 × 0.25–0.50–0.75 kg ai/ha and harvest at 47–48 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from the Netherlands (1981) were available with 1 × 0.50–0.75 kg ai/ha and harvest at 74–107 DAT or 1 ×0.38 kg ai/ha and harvest at 107 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Australia (1982) were available with 1 × 0.75-1.5 kg ai/ha and harvest at 19 or 83 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from South Africa (1982) were available with 1 × 0.50–1.0 kg ai/ha, 2 ×0.50–1.0 kg ai/ha or 0.50+1.0 kg ai/ha or 1.0+0.50 kg ai/ha and harvest at 38 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. These trials were not summarized, because they would not assist in MRL setting.

Additional trials were performed on 20 locations in the USA in 1982 and 1983 [Koubek, 1984, 406215, report TMU1257/B]. Ten trials were conducted with 2 ×1.1 kg ai/ha with harvest at 38, 39, 40, 70 DAT, 2 ×0.56 kg ai/ha with harvest at 32, 39, 49, 52, 53, 58, or 70 DAT. These trials were not summarized because they would not assist in MRL setting. Ten remaining trials were summarized in the table below.

Besides total fluazifop, also despyridinyl acid (III) was analysed in bulb onion samples from some trials conducted in the USA in 1981 and 1982 [Atreya, 1984, PP9/0728, report PP009B272]. These trials were summarized in the metabolism section.

Besides total fluazifop, also CF3-pyridone (X) was analysed in bulb onion samples from some trials conducted in the USA in 1981, 1982, 1984 and 1986 [Atreya, 1984, PP9/0731, report PP009B290; Morgan and Crook, 1986, PP5/0250, report M4266B; Hayward, 1987, PP5/0251, report M4545B]. These trials were summarized in the metabolism section.

Table 202 Supervised field trials on bulb onions (bulb only), treated with a broadcast foliar fluazifop-butyl spray

BULB ONIONS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Niland, CA, USA, 1982 (Creole)	EC (rac) no Adj	2 (76)	1.1	0.40	GS ns 31 March	ns	bulb	45	< 0.04 [LOQ=0.05]	TMU1257/B, 38CA81-038 [Koubek, 1984, 406215]
Brawley, CA, USA, 1982 (Creole)	EC 40 (rac)	2 (22)	1.1	0.39	GS ns, 5 March	ns	bulb	46	0.48	TMU1257/B, 38CA82-005 [Koubek, 1984, 406215]
Calipatria,	EC 480	2	1.1	0.45	GS ns,	ns	bulb	45	0.26	TMU1257/B,

BULB ONIONS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
CA, USA, 1982 (Collosal)	(rac) +COC	(17)			6 April					38CA82-007 [Koubek, 1984, 406215]
El Centro, CA, USA, 1982/1983 (Ringer Grano)	EC 480 (rac) +COC	2 (46)	1.1	0.45	GS ns, 14 January, 1983	ns	bulb	46	0.34	TMU1257/B, 38CA82-056 [Koubek, 1984, 406215]
Five Points, CA, USA, 1982 (South Port White Globe)	EC 240 (rac) +COC	2 (22)	1.1 1.1	0.32 0.36	GS ns, 18 April	ns	bulb	45	0.07 [SS]	TMU1257/B, 41CA82-001 [Koubek, 1984, 406215]
Five Points, CA, USA, 1982 (Basic White dehydration 613)	EC 240 (rac) +COC	2 (24)	1.1	0.32	GS ns, 10 June	ns	bulb	46	0.19 [SS]	TMU1257/B, 41CA82-009 [Koubek, 1984, 406215]
Tulelake, CA, USA, 1982 (South Port White Globe)	EC 480 (rac)	2 (33)	1.1	0.20	GS ns, 27 June	ns	bulb	45	< 0.06 [SS]	TMU1257/B, 41CA82-043 [Koubek, 1984, 406215]
Longmont, CO, USA, 1982 (White Sweet Spanish)	EC 480 (rac) +AL411F	2 (32)	1.1	0.50	GS ns, 29 June	ns	bulb bulb	45 87	< 0.06 < 0.06 [SS]	TMU1257/B, 37CO82-016 [Koubek, 1984, 406215]
Collins, CO, USA, 1982 (Brown Beauty)	EC 480 (rac) +AL411F	2 (65)	0.84 0.84	0.32 0.19	GS ns 20 August	ns	bulb	45	< 0.06 [SS]	TMU1257/B, 37CO82-033 [Koubek, 1984, 406215]
Uvalde, TX, USA, 1982 (New Mexico Grande)	EC 480 (rac) +COC	2 (13)	0.56 0.56	0.30 0.30	GS ns, 31 May	ns	bulb	29 45	0.06 < 0.06	TMU1257/B, 60TX82-004 [Koubek, 1984, 406215]
Fort Collins, Co, USA, 1984 (Brown Beauty)	EC, 120 (P) + 1 % COC	2; (54)	0.42 0.42	0.19	GS not reported; 14 August	ns	ns	45	0.06	TMU1815/B 37CO84-056 [Francis, 1985, 434142]
Hastings, FL, USA, 1984 (Texas Grano 429)	EC, 120 (P) + NIS	2; (19)	0.42 0.42	0.11	GS not reported; 03 April	ns	ns	44	0.06	TMU1815/B 75FL84-023 [Francis, 1985, 434142]
idem	EC 480 (rac) + NIS	2; (19)	0.42 0.42	0.11	GS not reported; 03 April	ns	ns	44	0.11	idem
Claxton, GA, USA, 1984 (Granex)	EC, 120 (P) + 1 % COC	2; (21)	0.42 0.42	0.20	GS not reported; 02 April	ns	ns	45	< 0.03 [SS] [LOQ=0.05]	TMU1815/B 83GA84-001 [Francis, 1985, 434142]
idem	EC, 480 (rac) + 1 % COC	2; (21)	0.42 0.42	0.20	GS not reported; 02 April	ns	ns	45	< 0.03 [SS] [LOQ=0.05]	idem

BULB ONIONS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Donna, TX, USA, 1984 (Granex 33)	EC, 120 (P) + 0.25 % NIS	2; (21)	0.42 0.42	0.11	GS not reported; 27 January	ns	ns	39	0.04 [SS] [LOQ=0.05]	TMU1815/B 71TX83-037 [Francis, 1985, 434142]
idem	EC, 480 (rac) + 0.25 % NIS	2; (21)	0.42 0.42	0.11	GS not reported; 27 January	ns	ns	39	< 0.02 [SS] [LOQ=0.05]	idem
Santa Rosa, TX, USA, 1984 (AandM 1015)	EC, 120 (P) + 1% COC	2; (12)	0.42 0.42	0.19	GS not reported; 28 February	ns	ns	45	0.03 [LOQ=0.05]	TMU1815/B 71TX83-046 [Francis, 1985, 434142]
idem	EC, 240 (P) + 1% COC	2; (12)	0.42 0.42	0.19	GS not reported; 28 February	ns	ns	45	0.03 [LOQ=0.05]	idem
idem	EC, 480 (rac) + 1% COC	2; (12)	0.42 0.42	0.19	GS not reported; 28 February	ns	ns	45	0.03 [LOQ=0.05]	idem
Mission, TX, USA, 1984 (Henry Special)	EC, 120 (P) + 1% COC	2; (15)	0.42 0.42	0.19	GS not reported; 29 March	ns	ns	46	0.04 [LOQ=0.05]	TMU1815/B 71TX83-056 [Francis, 1985, 434142]
idem	EC, 240 (P) + 1% COC	2; (15)	0.42 0.42	0.19	GS not reported; 29 March	ns	ns	46	0.04 [LOQ=0.05]	idem
idem	EC, 480 (rac) + 1% COC	2; (15)	0.42 0.42	0.19	GS not reported; 29 March	ns	ns	46	0.03 [LOQ=0.05]	idem
Visalia, CA, USA, 1984 (Red Weatherhead)	EC, 120 (P) + 1 % COC	2; (15)	0.42 0.42	0.15	GS not reported; 7 April	ns	ns	45	0.18	TMU1815/B US2-83-S14 [Francis, 1985, 434142]
idem	EC, 480 (rac) + 1.0% COC	2; (15)	0.42 0.42	0.15	GS not reported; 7 April	ns	ns	45	0.12	idem
Visalia, CA, USA, 1984 (Red Weatherhead)	EC, 120 (P) + 1 % COC	2; (15)	0.42 0.42	0.15	GS not reported; 7 April	ns	ns	45	0.16	TMU1815/B US2-83-S15 [Francis, 1985, 434142]
idem	EC, 480 (rac) + 1.0% COC	2; (15)	0.42 0.42	0.15	GS not reported; 7 April	ns	ns	45	0.07	idem
location ns; USA, 1986; (variety ns)	ns (P)	2; (15)	0.42 0.42	ns	ns	ns	ns	45	0.02 [SS] [LOQ=0.05]	M4545B; NCA/87/204 [Hayward, 1987, PP5/0251]
location ns; USA, 1986; (variety ns)	ns (P)	2; (15)	0.42 0.42	ns	ns	ns	ns	45	0.13 [SS]	M4545B; NCA/87/224 [Hayward, 1987,

BULB ONIONS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
										PP5/0251]
Piedade, SP, Brazil, 2011; (2nd yr onion: Optima)	EW 250 (P)	1	0.25	0.25	BBCH 43; 5 May	C	49	27	0.03	M11026, AMA [Draetta, 2012, A12530B_10016]
Planaltina, DF, Brazil, 2011; (1st yr onion: Andromeda F1)	EW 250 (P)	1	0.25	0.25	BBCH 41; 5 May	SiCL	47	28	< 0.01	M11026, MFG1 [Draetta, 2012, A12530B_10016]
Baraúna, RN, Brazil, 2011; (1st yr onion: Hibrido Gobi)	EW 250 (P)	1	0.25	0.25	BBCH 45; 14 Oct	SaCL	49	28	0.05	M11026, MFG2 [Draetta, 2012, A12530B_10016]
Baraúna, RN, Brazil, 2011; (2nd yr onion: IPA-11)	EW 250 (P)	1	0.25	0.25	BBCH 47; 14 Oct	SaCL	49	28	0.02	M11026, MFG3 [Draetta, 2012, A12530B_10016]
Bacton Suffolk; UK, 1984; (2nd year onion; Japan S-Yellow)	EC 125 (P) + Agral	1	0.38	0.15	2.5-6 cm Ø; 20% crop cover; at 14 June	SaLC	ns	22	0.07; 0.11 [SS], [a]	M3872B; 5R/84 EA60 and 5R/84 EA61 [Dick, 1984, PP5/0088]
Brothertoft, Lincolnshire; UK, 1984; (2nd year onion; Japan S-Gold)	EC 125 (P) + Agral	1	0.38	0.14	2-3 leaves; 60% crop cover; at 31 May	SaL	ns	28	0.05 [SS]	M3872B; 5R/84 LN36 [Dick, 1984, PP5/0088]
Anwyck; near Sleaford, UK, 1984 (2nd year onion; Japan S-Gold)	EC 125 (P) + Agral	1	0.38	0.14	2-3 leaves; 65% crop cover; at 31 May	L	ns	28	0.06 [SS]	M3872B; 5R/84 LN35 [Dick, 1984, PP5/0088]
Wyeboston; Beds UK, 1984 (2nd year onion: Japan S-Gold)	EC 125 (P) + Agral	1	0.38	0.17	5-10 leaves; 25% crop cover; at 24 May	LC	ns	32	0.06 [SS]	M3872B; 5R/84 NA84-R10 [Dick, 1984, PP5/0088]
Heckington; UK, 1984, (1st yr onion: Robusta)	EC 125 (P) + Agral	1	0.38	0.14	5-7 leaves; 40% crop cover; at 13 August	SaC	ns	28	0.11; 0.11 [SS], [a]	M3872B; 5R/84 LN 32 and 5R/84 LN 33 [Dick, 1984, PP5/0088]
Sleaford, Lincolnshire, UK, 1984, (1st yr onion: Robusta)	EC 125 (P) + Agral	1	0.38	0.14	5-7 leaves; 70% crop cover; at 13	SaC	ns	24	0.05 [SS]	M3872B; 5R/84 LN 91 [Dick, 1984, PP5/0088]

BULB ONIONS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					August					
Bacton Suffolk; UK, 1984, (1st yr onion: Hyper)	EC 125 (P) + Agral	1	0.38	0.15	2-5 leaves; 40% crop cover; at 25 July	LC	ns	27	0.19 [SS]	M3872B; 5R/84 EA 62 [Dick, 1984, PP5/0088]
Swineshead; UK; 1984; (1st yr onion: Tarzan)	EC 125 (P) + Agral	1	0.38	0.14	6-7 leaves; 40% crop cover; at 13 August	SaL	ns	28	0.07 [SS]	M3872B; 5R/84 LN 93 [Dick, 1984, PP5/0088]
Reculver, Kent; UK 1989; (1st yr onion: Balstora)	EW 125 (P) + Agral	1	0.38	0.19	5-6 leaves; 50% crop cover; at 3 July	CL	100% top tall	28	0.09	M5264B; GB52-89-S151; [Cullen and Jones, 1991, PP5/0091]
idem	EW 250 (P) + Agral	1	0.38	0.19	5-6 leaves; 50% crop cover; at 3 July	CL	100% top tall	28	0.06	M5264B; GB52-89-S151; [Cullen and Jones, 1991, PP5/0091]
idem	EC 125 (P) + Agral	1	0.38	0.19	5-6 leaves; 50% crop cover; at 3 July	CL	100% top tall	28	0.09	M5264B; GB52-89-S151; [Cullen and Jones, 1991, PP5/0091]
Holbeach, Lincolnshire; UK 1989; (Caribo)	EW 125 (P) + Agral	1	0.38	0.19	5-6 leaves; 55% crop cover; at 3 July	SaL	47	37	0.08	M5264B; GB12-89-S151; [Cullen and Jones, 1991, PP5/0091]
idem	EW 250 (P) + Agral	1	0.38	0.19	5-6 leaves; 55% crop cover; at 3 July	SaL	47	37	0.08	idem
idem	EC 125 (P) + Agral	1	0.38	0.19	5-6 leaves; 55% crop cover; at 3 July	SaL	47	37	0.06	idem
Hoofddorp; NL, 1984; (1st yr onion: Hyduro)	EC 125 (P) + Agral	1	0.38	0.075	12 cm length; 30% crop cover; at 18 June	C	ns	39	< 0.02 (4); mean < 0.02 [SS],b [LOQ=0.05]	M3975B; H84-229; [Dick and Rounds, 1985, PP5/0089]
Kruisland; NL, 1984;	EC 125 (P)	1	0.38	0.075	25-30 cm length;	C	ns	40	< 0.02 (4); mean < 0.02	M3975B; H84-329;

BULB ONIONS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(1st yr onion: Hyduro)	+ Agral				50% crop cover; at 6 July				[SS],[b] [LOQ=0.05]	[Dick and Rounds, 1985, PP5/0089]
Dinteloord; NL 1984; (1st yr silverskin onion; Barletta van der Plog)	EC 125 (P) + Agral	1	0.38	0.075	20-30 cm length; 80% crop cover; at 20 July	C	ns	28	0.02; 0.03 (2); 0.05; mean 0.03 [SS],[b] [LOQ=0.05]	M3975B; H84-334; [Dick and Rounds, 1985, PP5/0089]
idem	EC 125 (P) + Agral	1	0.38	0.075	20-30 cm length; 80% crop cover; at 20 July	C	ns	41	< 0.02 (4); mean < 0.02 [SS],[b] [LOQ=0.05]	idem
Flevopolder; NL 1985; (1st yr onion: Hyton)	EC 125 (P)	2 (30)	0.19	0.038	40-50 cm; 60% crop cover; at 17 July	ns	ns	28	< 0.03 (4); mean < 0.03 [SS],[b] [LOQ=0.05]	M4205B; H85/126; [Harradine and Crook, 1986, PP5/0090]
idem	EC 125 (P)	2 (30)	0.38	0.075	40-50 cm; 60% crop cover; at 17 July	ns	ns	28	0.04; 0.06; 0.09; 0.10; mean 0.07 [SS],[b] [LOQ=0.05]	idem
idem	EC 125 (P)	2 (30)	0.38	0.075	40-50 cm; 60% crop cover; at 17 July	ns	ns	42	< 0.03 (4); mean < 0.03 [SS],[b] [LOQ=0.05]	idem
Flevopolder; NL; 1985; (1st yr onion: Balstora)	EC 125 (P)	2 (30)	0.19	0.038	40 cm; 60% crop cover; at 17 July	ns	ns	28	< 0.03 (4); mean < 0.03 [SS],[b] [LOQ=0.05]	M4205B; H85/127; [Harradine and Crook, 1986, PP5/0090]
idem	EC 125 (P)	2 (30)	0.38	0.075	28 March 1985; (40 cm; 60% crop cover); at 17 July	ns	ns	28	< 0.03; 0.04 (2); 0.06; mean 0.04 [SS],[b] [LOQ=0.05]	idem
idem	EC 125 (P)	2 (30)	0.38	0.075	28 March 1985; (40 cm; 60% crop cover); at 17 July	ns	ns	42	< 0.03 (4); mean < 0.03 [SS],[b] [LOQ=0.05]	idem

BULB ONIONS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Almussafes; Valencia; Spain, 1987 (1st onion; variety ns)	EC 300 (P)	1	0.30	ns	10% crop cover; 2 June	ns	ns ns	24 80	0.03 < 0.01 [SS] [LOQ=0.05]	M4799B ES01-87-D003-H; [Crook, 1988, PP5/0380]
Sanlucar de Barrameda; Cadiz; Spain, 1997 (1st yr onion: Babosa)	EC 125 (P)	1	0.32	0.1	BBCH 41–43; 25 June	Sa	ns	28	0.02	RJ2728B AF/3704/ZE/1; [Jones and McGill, 1999, PP5/0295]
Triguerros; Huelva; Spain, 1997 (1st yr onion: Valenciana)	EC 125 (P)	1	0.32	0.10	BBCH 17–21 11 June	SaSiL	ns	28	< 0.01	RJ2728B AF/3704/ZE/2; [Jones and McGill, 1999, PP5/0295]
Las Cabezas; Sevilla; Spain, 1998 (1st yr onion: Grano de Oro)	EC 125 (P)	1	0.32	0.10	60 cm tall; BBCH 45; 23 June	SiC	ns	28	0.02 [SS]	RJ2827B AF/4146/ZE/3; [Ryan, 1999, PP5/0126]
Lora del Dio; Sevilla; Spain, 1998 (Persa)	EC 125 (P)	1	0.31	0.10	28 July 1998; 50 cm tall; BBCH 45; 7 Oct	SiCL	ns	28	< 0.01 [SS]	RJ2827B AF/4146/ZE/4 ; [Ryan, 1999, PP5/0126]
Bolognia; Emilia Romagna; Italy, 1997 (1st yr onion: Blando Duro)	EC 125 (P)	1	0.32	0.15	BBCH 48; 26 June;	CL	ns	28	0.05	RJ2728B AF/3704/ZE/3; [Jones and McGill, 1999, PP5/0295]
Piacenza; Emilia Romagna; Italy, 1997 (1st yr onion: Density)	EC 125 (P)	1	0.31	0.10	BBCH 48–49; 14 July	CL	ns	28	< 0.01	RJ2728B AF/3704/ZE/4; [Jones and McGill, 1999, PP5/0295]
Castelnau d'Arbieu; Tarn et Garonne; S-France, 1998 (1st yr onion: Sturon)	EC 125 (P)	1	0.31	0.10	40-50 cm tall; BBCH 45; 17 June	SaC	ns	28	0.03 [SS]	RJ2827B AF/4146/ZE/1; [Ryan, 1999, PP5/0126]
Cumont; Tarn et Garonne; S-France, 1998	EC 125 (P)	1	0.30	0.10	40-50 cm tall; BBCH 45-47; 20 July	SiC	ns	28	0.10 [SS]	RJ2827B AF/4146/ZE/2; [Ryan, 1999, PP5/0126]

BULB ONIONS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(1st yr onion: Spirit)										

[SS] Trials cannot be selected for MRL derivation because sample size is not stated (study M3872B, M3975B, M4205B, M4799B, RJ2728B, M4545B) and trial 37CO82-016, or sample size is below the minimum required 2 kg of onion bulbs (TMU 1815B and trials 41CA82-001, 41CA82-009, 41CA82-043, 37CO82-033 from TMU 1257B).

[a] Results came from replicate plots; the highest value is selected for MRL derivation if according to cGAP

[b] Results came from replicate field samples; the mean value is selected for MRL derivation, , if according to cGAP.

Additional trial information

TMU1257B. Non-GLP study. Weather conditions not reported. Soil type not stated. Post emergence broadcast applications. Spray volumes 20-60 GPA, ie 187-560 L/ha. Sample sizes 5-10 lb, i.e. 2.3-4.5 kg, except 41CA82-001,41CA82-009, 41CA82-043, 37CO82-016, 37CO82-033 with samples sized 0.9-1.4 kg. The growth stage of the onion bulbs when harvested was not reported. Storage at -23 °C; duration not stated. Samples were analysed for total fluazifop using HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg. Concurrent recoveries average 98% over the range of 0.05-1.0 mg/kg (n = 13, fluazifop). Raw data not provided. Control samples < 0.04 or,< 0.06 mg/kg. The results were corrected for concurrent recoveries of <100% and for HPLC clean-up recoveries.

TMU1815B. GLP study. No unusual weather conditions. Soil type not stated. Post emergence broadcast foliar applications. Spray volume not provided . Sample sizes >2 kg, except GA, 71TX83-037 with 0.9-1.4 kg. The growth stage of the onion bulbs when harvested was not reported. Any remaining leaves or roots were removed to leave only the bulb. Storage at -18 °C; for maximum of 9 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Internal standard recovery (86% at 0.03–0.8 mg/kg). Control samples < 0.03 mg/kg.

M4545B GLP study. Weather conditions, spray equipment, spray volumes, sampling, sample sizes are not stated. Leaves, roots or soil were removed and bulbs were analysed. Storage at -18°C; for a maximum of 302 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries or results in control samples were not recorded.

M11026 GLP study. No unusual weather conditions. Broadcast foliar spray using a pre-pressurized sprayer with CO2 gas. Spray volume 100 L/ha. Onion plants were sampled by hand and were taken systematically from across the plots. Foliage was removed with a knife, sample size at least 12 onions (> 2 kg). Storage at -20 °C; for 2.7-8.1 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET.138 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for individual concurrent method recoveries (81% at 0.01 mg/kg). Control samples < 0.01 mg/kg.

M3872B Non-GLP study. No unusual weather conditions. Applications by C02 knapsack or CO2 plot sprayers. Spray volume 225-260 L/ha. Sample sizes not stated. Onions were harvested green, but only the bulbs were analysed (tops or leaves were removed). The outer layer of dead skin was removed from the onions before analysis. Storage at -20 °C for maximum 208 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Average internal standard recovery (80% at 0.5-1.0 mg/kg). Control samples were < 0.03 mg/kg.

M5264B Non-GLP study. No unusual weather conditions. Applications by single man boom sprayers. AGRAL added as adjuvant. Spray volume 200 L/ha. Onions were harvested at maturity and 12 bulbs taken at random across the plot. **Parchment** and roots were removed prior to preparation. Storage 3-8 months at -18 °C. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Average internal standard recovery (90% at 0.5 mg/kg). Samples were not corrected for average concurrent recoveries (99% at 0.5 mg/kg). Control samples < 0.05 mg/kg.

M3975B Non-GLP study. No unusual weather conditions. AGRAL added as adjuvant. Applications by knapsack propane sprayer. Spray volume 500 L/ha. Sample sizes not stated. Growth stage at harvest not indicated, only bulbs were analysed. Four replicate samples from each trial were taken for analysis. Storage at -18 °C for maximum 321 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Internal standard recovery (83% at 0.5 mg/kg). Control samples < 0.02 mg/kg.

M4205B Non-GLP study. No unusual weather conditions. Soil type not stated. Applications by knapsack propane gas sprayer. Spray volume 500 L/ha. Sample sizes not stated. Onion bulbs were harvested in a green stage; any remaining leaves or roots were removed to leave only the bulb. Four replicate samples from each trial were taken for analysis. Storage at -18 °C; for maximum 296 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Internal standard recovery (79% at 0.1 mg/kg). Control samples < 0.03 mg/kg.

M4799B Non-GLP study. Weather conditions, soil type, application equipment, spray volume, growth stage at harvest not stated. Sample sizes not stated. Storage at -18 °C; for maximum 309 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Internal standard recovery (98% at 0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2728B GLP study. No unusual weather conditions. Applications by boom sprayer. Spray volume 300 L/ha. Onion bulbs (2.0 kg; 12 units) were harvested by hand taking care to avoid plot boundaries and were taken systematically from across the plots. Storage at -18 °C; for 246-279 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for individual concurrent method recoveries (101-106% for 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2827B GLP study. No unusual weather conditions. Overall foliar spray using a plot sprayer. Spray volume 200 L/ha. Sample sizes not stated. Mature onion bulbs were sampled by hand and were taken systematically from across the plots. Storage at -18 °C; for maximum 112-145 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for individual concurrent method recoveries (103-125% for 0.05-0.1 mg/kg). Control samples < 0.01 mg/kg.

Garlic

One cGAP for garlic is available:

- cGAP from Belgium with 2 ×0.38 kg ai/ha with a PHI of 28 days
- cGAP from France with 1 ×0.38 kg ai/ha with a PHI of 28 days

No studies to support these uses were submitted.

Green onions

One cGAP for green onions is available:

- cGAP from France with 1 ×0.38 kg ai/ha with PHI 42 days

No trials could be matched to this cGAP.

Trials from Spain (1987) on onion tops were available with 1 ×0.30 kg ai/ha with harvest at 24 DAT [Crook, 1988, PP5/0380, report M4799B]. Trials from the USA (1982, 2011–2012) were available on green onions with 2 ×0.41–0.50 kg ai/ha with harvest at 0–16 DAT [Arsenovic, 2014, PP5_50555, report IR-4, PR 03405] or 2 ×1.1 kg ai/ha with harvest at 0–46 DAT [Koubek, 1984, 406215, TMU1257/B]. These trials were not summarized, because they would not assist in MRL setting.

Besides total fluazifop, also despyridinyl acid (III) was analysed in green onion samples from some trials conducted in the USA in 1982 [Atreya and Dick, 1984, PP9/0728, PP009B272]. These trials were summarized in the metabolism section.

Besides total fluazifop, also CF3-pyridone (X) was analysed in green onion samples from some trials conducted in the USA in 1982 [Atreya and Upton, 1984, PP9/0731, PP009B290]. These trials were summarized in the metabolism section.

Leeks

One cGAP for leeks is available:

- cGAP from Belgium or France with 1 ×0.38 kg ai/ha with a PHI of 42 days

Trials that could be matched to this cGAP were summarized.

Table 203 lists trials conducted in the UK (2001, 2002), the Netherlands (1985, 2002, 2003) and Northern France (2001, 2002, 2005). A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 203.

Additional trials from the Netherlands (1981) were available with 1 × 0.38–0.75 kg ai/ha and harvest at 80 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. These trials were not summarized, because they would not assist in MRL setting.

Table 203 Supervised field trials on leeks (whole plant), treated with a broadcast foliar fluazifop-butyl spray

LEEKS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Bergen op Zoom; Netherlands; 1985; (Starlina)	EC 125 (P)	1	0.19	0.038	crop height 20 cm; 10% crop cover; 7 Aug	Sa	diam 1.5 cm	29 . . . . . 43	< 0.05 < 0.05; < 0.05; < 0.05; mean [a] < 0.05 < 0.05; < 0.05; < 0.05; < 0.05; mean [a] < 0.05	M4217B; 85/321 [Harradine and Crook, 1986, PP5/0087]
idem	EC 125 (P)	1	0.38	0.075	crop height 20 cm; 10% crop cover; 7 Aug	Sa	diam 1.5 cm	29 . . . . . 43	< 0.05, 0.06, 0.06, 0.10, mean [a] 0.068 < 0.05; < 0.05, < 0.05, < 0.05 mean [a] < 0.05	idem
Woensdrecht; Netherlands; 1985; (Starlina)	EC 125 (P)	1	0.19	0.038	crop height 20 cm; 10% crop cover	Sa	diam 2.5 cm	29 . . . . . 43	< 0.05 < 0.05; < 0.05; < 0.05; mean [a] < 0.05 < 0.05; < 0.05; < 0.05; < 0.05; mean [a] < 0.05	M4217B; 85/322 [Harradine and Crook, 1986, PP5/0087]
idem	EC 125 (P)	1	0.38	0.075	crop height 20 cm; 10% crop cover	Sa	diam 2.5 cm	28 . . . . . 42	< 0.05 < 0.05; < 0.05; < 0.05; mean [a] < 0.05 < 0.05; < 0.05; < 0.05; < 0.05; mean [a] < 0.05	idem
Oud Gastel; Netherlands; 2002; (Roxton)	EC 125 (P)	1	0.41	0.12	BBCH 16-17; crop height 20-30 cm; 10% crop	Sa	49	64	< 0.01 blb < 0.01 lvs < 0.01 RAC	02-7083; 02-7083; [Mason, 2004, PP5/1376]

LEEKS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					cover; 11 Jul					
Tienray; Netherlands; 2003; (Shelton)	EC 125 (P)	1	0.38	0.13	BBCH 14-15; crop height 20 cm; 10% crop cover; 23 July	Sa	49	103	< 0.01	03-7029; 03-7029; [Mason, 2004, PP5/1409]
Tattishall Bridge; Lincolnshire; UK; 2001; (Virzo)	EC 125 (P)	1	0.38	0.12	BBCH 14-16; crop height 15-30 cm; 10% crop cover; 24 Aug	SaL	CH	103 103	0.03 blb 0.02 lvs 0.03 RAC	RJ3278B; AF/6068/SY1; [Richards, 2002, PP5/1222]
Whittlesford, Cambridge, UK, 2002; (Porvite)	EC 125 (P)	1	0.38	0.12	BBCH 41; crop height 20 cm; 8-12% crop cover; 27 June	SaCL	48	76	0.02 blb; 0.03 lvs; 0.03 RAC; 0.02 blb; 0.03 lvs; 0.03 RAC; mean [a]; 0.03 RAC	02-7035; 02-7035 [Mason, 2004, PP5/1377]
St Hilaire St Mestmin; Loiret; N-France; 2001 (Siegried)	EC 125 (P)	1	0.38	0.12	crop height 20-25 cm; 10% crop cover; 25 Sept	Sa	CH	108 108	0.04 blb 0.07 lvs 0.06 RAC	RJ3278B; AF/6068/SY2; [Richards, 2002, PP5/1222]
L' Abergement de Cuisery; Normancy; N-France; 2002; (Tadorna)	EC 125 (P)	1	0.38	0.12	BBCH 19; crop height 30-40 cm; 10-15% crop cover; 5 Aug	LSa	407	70	0.01 blb; 0.02 lvs; 0.02 RAC	02-21401; 02-21401 [Mason, 2004, PP5/1405]
Ploubazlannec; N-France; 2005; (Allium ampeloprasum: Bleu Solaize)	EC 125 (P)	1	0.38	0.12	BBCH 19; crop height 20 cm; 13 Sept	SaL	51	189	< 0.01 RAC	CEMR-2687; FR-HR-05-477; [Bell, 2006, PP5/1489]
Auxonne; Burgundy; N-France; 2005 (Kenton)	EC 125 (P)	1	0.37	0.12	BBCH 16-41; crop height 20 cm; 17 Aug	Sa	49	112	< 0.01 RAC	CEMR-2687; SRF05SYN17; [Bell, 2006, PP5/1489]
La Chapell de Guinchay; Burgundy; N-France 2005 (Chelton)	EC 125 (P)	1	0.39	0.13	BBCH 13-14; Crop height 20-25 cm; 22 Sept	SaSi	49	196	< 0.01 blb < 0.01 lvs < 0.01 RAC	CEMR-2687; SRF05SYN18; [Bell, 2006, PP5/1489]

blb = bulbs; lvs = leaves; RAC = whole plant without roots. Residue in the RAC calculated from weight fractions and residue levels for leaves and bulbs (32-39% (w/w) is bulb; 61-68% (w/w) is remaining leaves). The bulb is the edible portion, equivalent to class 2 leeks: stems consisting of 25% white and 75% green

[a] Results came from replicate field samples; the mean value is taken for MRL derivation, , if according to cGAP

Additional trial information

M4217B. non-GLP. No unusual weather conditions. Spray using a propane gas sprayer. Spray volume 500 L/ha. Leeks with 20 cm leaves were sampled by hand (15 plants/field sample; 4 field samples per location). Also samples were taken at DAT56, but these were not analysed. Storage at -20 °C for a maximum of 279 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Average internal std recovery (86-89% at 0.1 mg/kg). Control samples < 0.05 mg/kg.

02-7083. GLP. No unusual weather conditions. Foliar backpack sprayer with 3 m boom. Spray volume 327 L/ha. Whole plants (> 4 kg, 12 items) was sampled manually and taken from across the plots. Whole plants were separated into leaves and stems/bulbs. Storage at -18 °C for 328 days. Temperature rose to -2.4 ºC for a 15 hr period, but this is considered to have no impact on the study results, since the samples remained frozen at all times. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (102-113% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

03-7029. GLP. No unusual weather conditions. Foliar spray using a hand-held compressed air sprayer with 3 m boom. Spray volume 303 L/ha. Whole plants (3.0-3.7 kg, 12 items) were sampled by hand. Storage at -18 °C for 77 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (82-93% at 0.01–0.05 mg/kg). Control samples < 0.01 mg/kg.

RJ3278B. GLP. No unusual weather conditions. Overall foliar spray using a precision boom sprayer. Spray volume 304-312 L/ha. Leek whole plants (> 2.0 kg, 12 items) were sampled manually and taken systematically from all areas of the plots. Storage at -18 °C for 41-78 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (81-97% at 0.1-1.0 mg/kg). Control samples < 0.01 mg/kg.

02-7035. GLP. No unusual weather conditions. Foliar spray using a one-man hand-held backpack sprayer with 2.5 m boom. Spray volume 300 L/ha. Whole plants (> 5 kg, 15 items) was sampled manually in a W transect from the centre of the plots. Whole plants were separated into leaves and stems/bulbs. Storage at -18 °C for 330 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (102-113% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

02-21401. GLP. No unusual weather conditions. Foliar spray using a hand-held boom sprayer. Spray volume 300 L/ha. Whole plants (3.85-4.75kg, 12 items) was sampled by hand. Whole plants were separated into leaves and stems/bulbs (equivalent to class 2 leeks). Storage at -18 °C for 297 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (102-113% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

CEMR-2687. GLP. No unusual weather conditions. Broadcast foliar spray using a knapsack sprayer with boom. Spray volume 296-315 L/ha. Whole plants (3.3-4.0 kg, 12 items) were sampled by hand. Whole plants were separated into leaves and stems/bulbs, except trials FR-HR-05-477 and SRF05SYN17 where the whole plant was prepared. Storage at -18 °C for 64-184 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recovery (74-85% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

Brassica (cole or cabbage) vegetables

Broccoli

Three cGAPs for broccoli are available:

- cGAP from Brazil with 1 ×0.25 kg ai/ha with PHI 28 days for broccoli and cauliflower
- cGAP from France with 1 ×0.19 kg ai/ha with PHI 42 days for broccoli and cauliflower

Trials from the UK (1980) were available on broccoli with an application of 1 ×1.0–2.0 kg ai/ha and harvest at 27 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Since the manufacturer did not intend to have MRLs on broccoli, the available studies on broccoli were not summarized.

Some available supervised trials on broccoli were not submitted: [Baron, 1988, MRID 40566801, report IR-4 PR 2074; not referenced]. No further efforts were taken to retrieve these studies.

Brussels sprouts

A GAP for Brussels sprouts is not available.

Trials from Germany (1980) were available on Brussels sprouts with an application of 1 × 0.19 kg ai/ha and harvest at 42 DAT [Gardyan, 1992, PP5/0129, report AZ83592/91]. Since the manufacturer did not intend to have MRLs on Brussels sprouts, the available studies on Brussels sprouts were not summarized.

Cauliflower

Three cGAPs for cauliflower are available:

- cGAP from Brazil with 1 ×0.25 kg ai/ha with PHI 28 days for broccoli and cauliflower
- cGAP from France with 1 ×0.19 kg ai/ha with PHI 42 days for broccoli and cauliflower

Trials from Canada (1980) were available on cauliflower with an application of 1 ×0.30 kg ai/ha and harvest at 75 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Trials from Germany (1980, 1981) were available on cauliflower with an application of 1 ×0.38 kg ai/ha and harvest at 0, 10, 21–22, 35, 49 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B] or 1 ×0.19 kg ai/ha and harvest at 42 DAT [Gardyan, 1992, PP5/0129, report AZ83592/91]. Trials from the Netherlands (1981) were available on cauliflower with an application of 1 × 0.50–0.75 kg ai/ha and harvest at 40 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Since the manufacturer did not intend to have MRLs on cauliflower, the available studies on cauliflower were not summarized.

Head cabbages

Three cGAPs for head cabbages are available:

- cGAP from Brazil with 1 ×0.25 kg ai/ha with PHI 28 days for cabbage (as nn)
- cGAP from FR with 1 ×0.19 kg ai/ha with PHI 42 days for head cabbage (as nn)
- cGAP from BE with 1 ×0.19 kg ai/ha with application until BBCH 18

Trials that could be matched to these cGAPs were summarized.

Table 204 lists trials conducted in the Brazil (2012), Germany (1983, 1991, 1993, 1996, 2002), Northern France (1996, 1997, 1998, 1999), Greece (2000), Spain (1987). A broadcast or banded foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 204. Results marked with "[SS]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample sizes were not reported or less than 12 plants.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

Additional trials from Canada (1980) were available on unspecified cabbage with 1 ×0.30 kg ai/ha and harvest at 56 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from the UK (1981) were available on unspecified cabbage with 1 × 1.0 kg ai/ha and harvest at 64-65 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from Germany (1981) were available on red, white and Savoy cabbage with 1 × 0.38 kg ai/ha and harvest at 0, 10, 20-22, 35, 49 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from the Netherlands (1981) were available on red cabbage with 1 ×0.50-1.0 kg ai/ha and harvest at 51 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. These trials were not summarized, because they would not assist in MRL setting.

Table 204 Supervised field trials on head cabbages (heads only), treated with a broadcast or banded foliar fluazifop-butyl spray

HEAD CABBAGE Location, Country; year; (variety)	For-mu-lation	no. of appl (inter-val)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT	Total fluazifop (mg/kg)	Report no, Trial no. [ref]
Broadcast foliar spray										
Planaltina, DF, Brazil, 2012 (Asteca)	EW 250 (P)	1	0.19	0.063	BBCH 41; 27 Jul	SaC	47	28	0.29	M12060 MFG [Lopes, 2013, A12530D_10013]
Piedade, SP, Brazil, 2012 (Shinsei)	EW 250 (P)	1	0.19	0.063	BBCH 41; 6 Jul	LS	48	28	0.51	M12060 RWC1 [Lopes, 2013, A12530D_10013]
Engenheiro Coelho, SP, Brazil, 2012 (Astrus Plus)	EW 250 (P)	1	0.19	0.063	BBCH 22-24; 25 Jul	SaC	47-49	28	0.27	M12060 RWC2 [Lopes, 2013, A12530D_10013]
Bandeirantes, PR, Brazil, 2012 (Astrus Plus)	EW 250 (P)	1	0.19	0.063	BBCH 45; 30 June	C	49	28	0.29	M12060 RWC3 [Lopes, 2013, A12530D_10013]
6742 Herxheim Hayna; Germany, 1983, (Savoy cabbage: Wirosa)	EC 250 (rac)	1	0.38	0.094	20 cm high; 70% crop cover; 4 Aug	SaL	IMM IMM HD MAT MAT	0 14 28 42 54	9.8 2.1 1.7 0.43 0.46 [SS]	M3681B RS 8376 E2 [Harradine, 1984, PP9/0057]
idem	EC 125 (P)	1	0.12	0.030	20 cm high; 70% crop cover; 4 Aug	SaL	IMM IMM HD MAT MAT	0 14 28 42 54	5.3 0.84 0.94 0.28 0.19 [SS]	idem
6742 Herxheim Hayna; Germany, 1983, (Red cabbage: Marner Lagerrot)	EC 250 (rac)	1	0.38	0.094	25 cm high; 40% crop cover; 4 Aug	SaL	IMM IMM HD MAT MAT	0 14 28 42 54	6.7 1.1 1.4 0.82 0.50 [SS]	M3681B RS 8376 E1 [Harradine, 1984, PP9/0057]
idem	EC 125 (P)	1	0.12	0.030	25 cm high; 40% crop cover; 4 Aug	SaL	IMM IMM HD MAT MAT	0 14 28 42 54	3.9 0.81 0.88 0.57 0.23 [SS]	idem
Location ns; Germany, 1991; (variety ns)	EC 125 (P)	1	0.19	ns	ns	ns	ns	41	0.18 [CT] [Cntrl=0.06]	AZ83592/91; 91HJ068Ext 1, [Gardyan, 1992, PP5/0129] (processing)
85354 Freising; Bavaria; Germany; 1993	ME 125 (P)	1	0.19	0.094	BBCH 19 (10 leaves); 80% crop cover;	L	10L 13L HD HD CH	0 11 26 38 49	13 3.6 2.2 1.4 1.1	RJ1583B; RS-9310-G1; [Patel and Robinson, 1994, [PP5/0130]

HEAD CABBAGE Location, Country; year; (variety)	For-mu-lation	no. of appl (inter-val)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT	Total fluazifop (mg/kg)	Report no, Trial no. [ref]
(Savoy: Wirosa)					13 Aug					
idem	EC 125 (P)	1	0.19	0.094	BBCH 19; (10-leaves); 80% crop cover; 13 Aug	L	10L 13L HD HD CH	0 11 26 38 49	13 3.6 2.1 <u>1.7</u> 1.2	idem
D-94550; Künzing; Bavaria; Germany, 1996 (Savoy: Wirosa)	EC 125 (P)	1	0.19	0.075	BBCH 17-18; 15-20 cm tall; 85% crop cover; 29 July	LSa	43 45 47-49	31 49 60	0.70 <u>0.56</u> 0.51 [Cntrl=0.01]	RJ2306B; RS 9618 G1; [Jones *et al*, 1997, PP5/0135]
D-03096; Burg; Brandenburg; Germany, 1996 (Savoy: Midvoy)	EC 125 (P)	1	0.19	0.094	BBCH 17; 12-16 cm tall; 15% crop cover; 20 Aug	Sa	40 43 45	31 45 59	0.12 <u>0.06</u> 0.06	RJ2306B; RS 9618 K1; [Jones *et al*, 1997, PP5/0135]
Bonn, Germany, 2002 (Savoy Cabbage: Polasa)	EC 125 (P)	1	0.19	0.047	BBCH 14-15; 22 July	ns	ns	49 231	0.02 < 0.01 [GS]	03-7068; 02/037; [Mason, 2004, PP5/1394]
idem	EC 125 (P)	1	0.19	0.047	BBCH 17-18; 2 Aug	ns	ns	49	<u>0.16</u>	idem
Bonn; Germany, 2002 (Savoy; Siberia)	EC 125 (P)	1	0.19	0.047	BBCH 14-15; 4 Sept	ns	ns	49 233	0.03 < 0.01 [GS]	03-7076; 02-038; [Mason, 2004, PP5/1395]
idem	EC 125 (P)	1	0.19	0.047	BBCH 17-18; 16 Sept	ns	ns	49 221	<u>0.15</u> 0.03 [GS]	idem
St Pierre les Elbeuf; Seine-Maritime; N-France, 1996 (Vivoy)	EC 125 (P)	1	0.19	0.062	BBCH 16; 14 June	Sa	ns	30 45 60	0.04 < 0.01 < 0.01 [CT] [Cntrl=0.01]	RJ2312B AP/3220/ZE/1; [Miles and Hill, 1997; PP5/0133]
Gometz le Chatel; Ile-de France; N-France, 1996 (Reglo)	EC 125 (P)	1	0.19	0.062	BBCH 17; 5 Aug	SaCL	ns	30 45 60	0.07 < 0.01 [CT] 0.01 [CT] [Cntrl=0.01]	RJ2312B AP/3220/ZE/2; [Miles and Hill, 1997; PP5/0133]
Renneville; Champagne; N-France, 1997 (Bartolo)	EC 125 (P)	1	0.19	0.062	BBCH 18; 3 July	Ca	ns	42 56 71	<u>0.12</u> 0.07 0.04	RJ2645B; S401.98; [Mason *et al*., 1999, PP5/0137]
Martot, Normandy, N-France,	EC 125 (P)	1	0.19	0.062	BBCH 17; 10 cm	Si	IMM IMM	42 56	0.01 < 0.01	RJ2794B; S201.99; [Ryan and

HEAD CABBAGE Location, Country; year; (variety)	For-mu-lation	no. of appl (inter-val)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT	Total fluazifop (mg/kg)	Report no, Trial no. [ref]
1998 (Virosa)					tall; 8 July				[SS]	Renard; 1999, PP5/0146]
Renneville, North-East, N-France, 1998 (Harathon)	EC 125 (P)	1	0.19	0.062	BBCH 19; 18-21 cm; 29 June	Ca	IMM IMM	42 56	0.12 0.06 [SS]	RJ2794B; S402.99; [Ryan and Renard; 1999, PP5/0146]
Renneville; North-East; N-France; 1998; (Savoy: Saga)	EC 125 (P)	1	0.38	0.12	BBCH 41-43; crop height 25 cm; 13 Aug	Ca	49	49	0.85	RJ2834B; S401.99 [Ryan and Sutra, 1999, PP5/0147]
St Genouph; Loire; N-France; 1998; (Savoy: Midwoy)	EC 125 (P)	1	0.38	0.12	BBCH 42; crop height 30 cm; 10 Sept	SaSi	49	50	3.1	RJ2834B; S641.99 [Ryan and Sutra, 1999, PP5/0147]
Gomez le Chatel; Essonne; N-France, 1999 (Ice Prince)	EC 125 (P)	1	0.19	0.062	BBCH 16-17; 8 cm tall; 21 July	CL	MAT MAT	42 56	0.01 [SS] < 0.01	RJ2992B AF/4718/ZE/1; [Mason, 2000, PP5/0356]
Almussafes; Valencia; Spain, 1987 (ns)	EC 300 (P)	1	0.30	ns	GS not reported 70% crop cover; 25 May	ns	ns	56	1.1 [SS] [cntrl=0.02]	M4799B ES01-87-D004-E; [Crook, 1988, PP5/0380]
Banded foliar spray over rows										
Inoi, Marathonas; Attica; Greece, 2000 (Baner)	EC 125 (P)	1	0.19	0.062	BBCH 15-17; 10 cm tall; 28 Aug	CL	MAT	57	0.09	RJ3232B; GR-00-H201; [Mason and Alevra, 2001, PP5/0006]
Ipsoma; Marathonas; Attica; Greece, 2000 (Sakata)	EC 125 (P)	1	0.19	0.062	BBCH 13-14; 10 cm tall; 11 Sept	C	MAT	56	< 0.01	RJ3232B; GR-00-H202; [Mason and Alevra, 2001, PP5/0006]

Soil type: Ca = calcareous

GSH: 10 L = 10 leaves developed (immature plant); 13L = 13 leaves developed (immature plant); HD = head development (early harvest); BBCH 40 = head begins to form, BBCH 41-48 = 10-80% of expected head size reached; BBCH 49 = typical size, form and firmness of heads reached.

[SS] Sample is not considered representative for MRL setting, since sample size is not stated (M4799B) or sample size is less than the required amount of 12 items or 2 kg. In report RJ2992B only 6 items were taken at DAT 42. In report RJ2794B the number of items per sample were not stated and growing conditions resulted in crops smaller than normal.

[GS] Indicates that the cabbages were not of commercial standards. The cabbages in report 03-7068, trial 02/037 with DAT 231 were sown/planted on 11 June 2002 and harvested on 10 March 2003. The cabbages in report 03-7076 trial 02/038 with DAT 221 and 223 were sown on 23 July 2002 and were harvested on 25 April 2003. This means these cabbages had been on the land during the winter period and they must be nearly rotten.

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

Additional trial information

M12060 GLP . No unusual weather conditions. Broadcast foliar spray, spray volume 300 L/ha. Cabbages (12 heads, >8.7 kg) were sampled by hand and taken systematically from across the plots. Storage at -20°C; for 2.4-3.1 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET138 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recoveries (84% at 0.01 mg/kg). Control samples < 0.01 mg/kg.

M3681B GLP study. No unusual weather conditions. Knapsack sprayer with boom, spray volume 400 L/ha. At DAT 0-14 the whole plant was harvested; at DAT 28-54 the outer leaves were removed and the head was sampled. Sample size not stated. Storage at -18 °C or lower; for 91 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Samples were corrected for average internal standard recoveries (81% at 0.5-5.0 mg/kg). Control samples < 0.02 mg/kg.

AZ83592/91. GLP. Weather, spray equipment and soil type were not reported. Spray volume not stated. Samples contained 2-3 kg brassica. Growth stage at harvest not stated. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with additional clean-up with column chromatography with a valid LOQ of 0.05 mg/kg**. Residues not corrected for concurrent method recoveries (71-112% at 0.05-1.0 mg/kg). Control samples contained 0.06 mg/kg,.

RJ1583B GLP. No unusual weather conditions. Spray application by air pressurised knapsack sprayer with boom, spray volume 200 L/ha. Cabbage plants (DAT 0, 11, 26, 38) or heads (DAT 49) were sampled by hand (12 items, each >4 kg) systematically from across the plot. At normal commerical harvest heads were trimmed to market requirements. Samples were sub-sampled to yield laboratory samples of 0.9-1.1 kg, by taking vertical segments of each plant. Storage at -15 °C; for a maximum of 109 days. Samples were analysed for total fluazifop using **NMR method RAM 197/01 with a valid LOQ of 0.05 mg/kg**. Mean internal std recovery (90% at 0.2 mg/kg). Control samples < 0.05 mg/kg

RJ2306B GLP study. No unusual weather conditions. Air pressurised knapsack sprayer with handheld boom, spray volume 200-250 L/ha., Heads (12 items, 14-13 kg, trial G1; 14 items, 2.4-5.4 kg trial K1) were sample by hand sytematically from across the plots. Heads were trimmed to market requirements. Field samples were reduced to 1 kg laboratory samples by cutting plants into segments. Storage at -18 °C; for 139-189days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were corrected for average concurrent method recoveries (95% at 0.1 mg/kg); uncorrected results are not stated in the report. Control samples < 0.01–0.01 mg/kg.

03-7068 GLP study. Weather conditions not stated. Wheeled plot sprayer, spray volume 400 L/ha. Samples of 12 heads (reduced to 1 kg laboratory samples) were taken from each plot in a wave-like order. Storage at -18 °C for 207-389 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (100-111% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

03-7076 GLP study. Weather conditions not stated. Wheeled plot sprayer, spray volume 400 L/ha. Samples of 12 heads (reduced to 1 kg laboratory samples) were taken from each plot in a wave-like order. Storage at -18 °C for 174-358 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (107-110% at 0.01–0.2 mg/kg). Control samples < 0.01 mg/kg.

RJ2312B GLP study. No unusual weather conditions. Hand held precision boom sprayer, spray volume 300 L/ha. Heads (12 items) were sample by hand sytematically from across the plots. Storage at -18 °C; for 109-176 days Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recoveries (107-111% at 0.1-3.0 mg/kg). Control samples < 0.01–0.01 mg/kg..

R2645B GLP study. No unusual weather conditions. Foliar spray with a hand-held sprayer, spray volume 300 L/ha cabbage heads were sampled by hand (12 items) systematically from across the plot. Storage at -18 °C; for 223-252 days Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (99-103% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2794B GLP study. Temperatures were fresh and unstable during the trial periods; crops in trial S201.99 were smaller than normal at harvest. Broadcast foliar spray using a hand held boom sprayer, spray volume 300 L/ha. Immature head cabbages were sampled by hand from across the plots; 1.3-9.2 kg; number of items not stated. Storage at -17 °C; for 106-157 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for mean concurrent method recoveries (108-112% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2834B GLP . No unusual weather conditions. Broadcast foliar spray using an hand held boom sprayer, spray volume 300 L/ha. Cabbages (12 units) were sampled by hand and taken systematically from across the plots. Storage at -18 °C; for 125-154 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (107-111% at 0.5-1.0 mg/kg). Control samples < 0.01 mg/kg.

RJ2992B GLP study. No unusual weather conditions. Overall foliar spray, precision boom sprayer, spray volume 300 L/ha. Mature cabbage heads were sampled by hand (6 items at DAT 42; 12 items at DAT 56, each 3.2-4.8 kg) systematically from across the plot. Decayed or inedible outer leaves were removed from the plant heads. DAT 56

samples were subsampled in the field, although no information was provided how this was done. Storage at -18 °C; for 71-85 days. Samples were analysed for total fluazifop using H**PLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (102-104% at 0.2–0.5 mg/kg). Control samples < 0.01 mg/kg

M4799B Non-GLP study. Weather conditions, soil type, application equipment, spray volume and growth stage at harvest not stated. Sample sizes not stated. Storage at -18 °C; for maximum 285 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Internal standard recovery (98% at 0.1 mg/kg). Control samples 0.02 mg/kg .

RJ3232B GLP study. No unusual weather conditions. **Banded spray over rows**, CO2 knapsack sprayer with hand lance, spray volume 300 L/ha. Mature cabbage heads were sampled by hand (12 items) systematically from across the plot. Damaged or decaying outer leaves were removed from the plant heads. Heads were cut longitudinally in the field and one half was discarded. Storage at -18 °C; for 185-198 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (92-105% at 0.01–0.05 mg/kg). Control samples < 0.01 mg/kg

Fruiting vegetables -cucurbits

Cucumbers

One cGAP for cucumber is available:

- cGAP from France with 1 ×0.19 kg ai/ha with PHI 28 days for cucumber, summer squash and gherkin.

Trials that could be matched to this cGAP were summarized.

Table 205 and Table 206 list trials conducted in Italy (1996, 1997) and Spain (1996, 1997, 1999) on indoor and field cucumbers. A broadcast foliar or a weed directed interrow banded spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 205 and Table 206. Results marked with "[SS]" and "[QU]" are not selected for derivation of the MRL, if according to cGAP.

Additional trials from Canada (1980) were available on cucumber with 1 × 0.50 kg ai/ha and harvest at 55 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. These trials were not summarized, because they would not assist in MRL setting.

[SS] indicates that the sample sizes were not reported.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) were not reported.

Additional trials from Canada (1980 and 1984) were available on cucumber with 1 × 0.50 kg ai/ha and harvest at 55 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B] or with 0.50 kg ai/ha [Harradine, 1985, PP5/0421, report M4106B] or 1.0 kg ai/ha and harvest at 16 DAT [Harradine, 1985, PP5/0122, report M4097B]. Additional residue trials in cucumbers and pickling cucumbers from the USA (1982, 1983) were available with 1 ×0.25 kg ai/ha (18, 20 or 25 DAT), 1 ×0.28 kg ai/ha (36, 39 or 43 DAT), 1 × 0.50 kg ai/ha (18 or 20 DAT), 1 × 0.56 (25, 36 or 39 DAT), 2 ×0.25 kg ai/ha (7 or 8 DAT), 2 ×0.28 kg ai/ha (20, 21, 26 or 29 DAT), 2 × 0.56 kg ai/ha (8, 15, 20, 21, 26 or 29 DAT) [Yates and Monaco, 1984, no code, no report no]. Additional trials were performed in the USA in 1984. Cucumbers were treated with 1 x 0.28 or 0.56 kg ai/ha and harvest at 21 DAT [IR-4, 1984, PR 1878 (DE), also summarized in Baron, 1986, IR-4 PR1878]. These trials were not summarized, because they would not assist in MRL setting.

Besides total fluazifop, also despyridinyl acid (III) was analysed in cucumber samples from some trials conducted in the USA in 1981 and 1982 [Atreya and Dick, 1984, PP9/0728, report PP009B272]. These trials were summarized in the metabolism section.

Table 205 Supervised indoor trials on cucumbers (whole fruit), treated with a broadcast foliar or weed directed interrow fluazifop-butyl spray

INDOOR CUCUMBERS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Sabaudia; Lazio; Italy, 1997 (Jazzer)	EC 125 (P)	1	0.31	0.078	< BBCH 51; 20-25 cm tall; 28 Apr [a]	Sa	CR	22 29 43	0.04 0.02 < 0.01	RJ2507B IT41-97-H344; [Mason *et al*., 1998; PP5/0173]
Weed directed band application										
Los Palacios; Sevilla; Spain, 1999 (Barinas)	EC 125 (P)	1	0.31	0.096	BBCH 51; 85 cm tall; 16 June [a]	Sa	CH	28	< 0.01	RJ3058B AF/4720/ZE/1; [Ryan, 2000, PP5/0447]

[a] Cucumber plants were covered with a polythene tunnel from application to Mid May and were grown in the open field thereafter

Additional trial information

RJ2507B GLP study. No unusual weather conditions. Broadcast foliar spray using a gas knapsack sprayer with boom; spray volume 400 L/ha. Cucumbers were sampled by hand systematicallyfrom across the plots: 12 items of 2.5-4.8 kg. Storage at -18 °C; for 99 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01mg/kg**. Samples were not corrected for individual concurrent method recoveries (86-113% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ3058B GLP study. Crops were grown in a greenhouse under normal conditions and agricultural practices for protected cucumber in Spain. **Weed directed band application** using a hydraulic knapsack sprayer, spray volume 327 L/ha. Cucumbers (12 items, > 2 kg) were sampled by hand from 12 plants. Storage at -18 °C; for 134 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (110-116% at 0.05-0.1 mg/kg). Control samples < 0.01 mg/kg.

Table 206 Supervised field trials on cucumbers (whole fruit), treated with a broadcast foliar or weed directed interrow fluazifop-butyl spray

FIELD CUCUMBERS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Location ns, NC, USA; 1984; (variety ns)	ns (rac)	1	0.56	ns	ns, 6 June	ns	ns	8	0.24 [a], [SS], [QU]	IR-4 PR 1878 Trial ns [Baron, 1986, no code] and [IR-4, 1984 (NC), no code]
idem	ns (rac)	1	0.56	ns	ns, 30 May	ns	ns	15	0.19 [a], [SS], [QU]	idem
idem	ns (rac)	1	0.56	ns	ns, 23 May	ns	ns	22	< 0.05 [a], [SS], [QU]	idem
idem	ns (rac)	1	0.56	ns	16 May	ns	ns	29	< 0.05 [a], [SS], [QU]	idem
idem	ns	1	0.28	ns	ns,	ns	ns	8	0.19	idem

FIELD CUCUMBERS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	(P)				6 June				[a], [SS], [QU]	
idem	ns (P)	1	0.28	ns	ns, 30 May	ns	ns	15	0.19 [a], [SS], [QU]	idem
idem	ns (P)	1	0.28	ns	ns, 23 May	ns	ns	22	< 0.05 [a], [SS], [QU]	idem
idem	ns (P)	1	0.28	ns	16 May	ns	ns	29	< 0.05 [a], [SS], [QU]	idem
Sabaudia; Latina-Lazio; Italy, 1996 (Jazzer)	EC 125 (P)	1	0.31	0.078	BBCH 51; 29 April	Sa	85 87	30 45	0.01 < 0.01	RJ2265B IT10-96-R342; [Jones and Volpi, 1998, PP5/0168]
Versentino; Puglia; Italy, 1997 (Marketmore 76)	EC 125 (P)	1	0.31	0.10	< BBCH 51; 35 cm tall; 13 June	L	CH	27 42	<u>< 0.01</u> < 0.01	RJ2507B IT51-97-H345; [Mason *et al*., 1998; PP5/0173]
Sanlucar de Barrameda; Cadiz; Spain, 1996 (Dasher II)	EC 125 (P)	1	0.31	0.099	BBCH 102–104; 20 Aug	ns	701	30 45	< 0.01 < 0.01	RJ2380B AP/3222/ZE-3; [Jones *et al*., 1997, PP5/0171]
Weed directed interrow banded spray application										
Tamarite; Aragon; Spain, 1997 (Bellondo)	EC 125 (P)	1	0.30	0.074	< BBCH 51; 60-70 cm tall; 6 June	C	CH	21 28 42	< 0.01 < 0.01 < 0.01	RJ2507B ES10-97-SH007; [Mason *et al*., 1998; PP5/0173]
Tamarite; Aragon; Spain, 1997 (Bellondo)	EC 125 (P)	1	0.28	0.075	< BBCH 51; 60-70 cm tall; 20 June	C	CH	21 28 42	< 0.01 < 0.01 < 0.01	RJ2507B ES10-97-SH107; [Mason *et al*., 1998; PP5/0173]
Tamarite; Aragon; Spain, 1997 (Bellondo)	EC 125 (P)	1	0.32	0.078	< BBCH 51; 60-70 cm tall 17 July	C	CH	21 28 42	< 0.01 < 0.01 < 0.01	RJ2507B ES10-97-SH207; [Mason *et al*., 1998; PP5/0173]

[a] Mean of 4 replicate analyses.

[SS] Sample size not stated; trial cannto be selected for MRL deriveation.

[QU] Quality of the study insufficient for MRL derivation since the field conditions were not reported

Additional trial information

IR-4 PR1878 and IR-4 PR1878 (NC) Non-GLP study. Poor study quality since field conditions were not reported (weather conditions, application details, growth stages at application and harvest). Sample size not stated. Storage at -17°C for a maximum of 6 months (14 June-December, 1984). Samples were analysed for total fluazifop residues using **HPLC-UV method PR1878, with a valid LOQ of 0.05 mg/kg**.Samples were not corrected for concurrent recoveries (mean 82%, n = 6). Control samples were < 0.05 mg/kg.

RJ2265B GLP study. No unusual weather conditions. Broadcast foliar spray using a motor knapsack sprayer with boom, spray volume 400 L/ha. Mature cucumbers (12 items) were sampled by hand systematically from across the plots. Storage

at -18 °C; for 123-138days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (75-92% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2380B GLP study. No unusual weather conditions. Overall spray using a precision boom sprayer, spray volume 316 L/ha. Cucumbers (12 plants) were sampled by hand from across the plots. Storage at -18 °C; for 181-196 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recoveries (100% at 0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2507B GLP study. No unusual weather conditions. **Interrow banded spray** using a gas knapsack sprayer in Spain; Broadcast foliar spray using a motor knapsack sprayer in Italy, spray volume 300-400 L/ha. Cucumbers were sampled by hand systematicallyfrom across the plots: 2-3 kg in Spain; 12 items of 2.5-4.8 kg in Italy. Storage at -18 °C; for 140-202 days. For a short period there was an increase in temperature to -5 ºC (Spain); this is considered to have no effect on the results, since the samples remained frozen. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (86-113% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

Summer squash

One cGAP for summer squash is available:

- cGAP from France with 1 ×0.19 kg ai/ha with PHI 28 days for cucumber, summer squash and gherkin.

Trials that could be matched to this cGAP were summarized.

Table 207 lists trials conducted in Italy (1996) and South Africa (1991). A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 207. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample sizes were not reported or less than 12 fruits (at least 2 kg).

Additional residue trials in summer squash and Zucchini squash from USA (1982, 1983) were available with 1 ×0.28 (18 or 28 DAT), 1 × 0.56 (18 or 28 DAT), 2 ×0.28 (14 DAT), 2 × 0.56 kg ai/ha (14 DAT) and 1 ×0.31 kg ai/ha with DAT 22 or 44 [Yates and Monaco, 1984, no code, no report no]. Although one trial could be matched to the cGAP through proportionality, these trials were not summarized because they would not assist in MRL setting.

Table 207 Supervised field trials on summer squash (whole fruit), treated with a broadcast foliar fluazifop-butyl spray

SUMMER SQUASH Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Carobbio degli Angeli; Bergamo-Lombardia; Italy, 1996 (Afrodite)	EC 125 (P)	1	0.31	0.078	BBCH 33; 11 June	C	CH	29 44	≤ 0.01 < 0.01	RJ2265B IT10-96-R343 [Jones and Volpi, 1998, PP5/0168]
Komatipoort, East Transvaal South Africa, 1991; (Gem squash: Rolet)	EC 125 (P)	1	0.50	ns	BBCH 16 (6 leaves); 15 cm; 15 April	ns	MAT MAT MAT MAT	21 28 35 42	0.13 0.07 < 0.05 < 0.05 [SS]	RJ1085B; ZA19-91-H014 [Bolygo, 1992, PP5/0422]

[SS] Sample size was below the minimum requirement and therefore this trial cannot be selected for MRL derivation.

Additional trial information

RJ2265B GLP study. No unusual weather conditions. Broadcast foliar spray using a motor knapsack sprayer with boom, spray volume 400 L/ha. Mature courgettes (26-28 items) were sampled by hand systematically from across the plots. Storage at -18 °C; for 74-89 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01**

with a valid LOQ of 0.01 mg/kg. Samples were not corrected for individual concurrent method recoveries (75-92% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ1085B GLP study. No unusual weather conditions. Broadcast foliar spray, spray volume not stated. Mature courgettes (7 items, 1 kg) were sampled. Storage at -18 °C; for a maximum of 147 days. Samples were analysed for total fluazifop using **NMR method ARAM 197 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for individual concurrent method recoveries (109-112% at 0.05-0.5 mg/kg). Control samples < 0.05 mg/kg.

Melons

A GAP for melons is not available.

Trials from Italy (1981) and South Africa (1991) were available on melons with an application of 1 × 0.25-0.75 kg ai/ha and harvest at 0-57 DAT [Bolygo, 1992, PP5/0425, report RJ1082B] and [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from the USA (1982 and 1983) were available with, 1 ×0.28 (63, or 69 DAT), 1 × 0.56 (63, or 69 DAT), 2 ×0.28 (46, or 52 DAT), 2 × 0.56 kg ai/ha (29, 33, 37, 43, 46, or 52 DAT) [Yates and Monaco, 1984, no code, no report no]. As the manufacturer did not seek to have maximum residue levels estimated for melons, the available studies on melons were not summarized.

Fruiting vegetables other than cucurbits

Chili peppers

Two cGAPs for chili peppers are available:

- cGAP from the USA with 2 ×0.42 kg ai/ha with PHI 45 days (on tabasco peppers)
- cGAP from France (and its overseas areas) with 0.38 kg ai/ha with PHI 35 days

None of the available studies on tobasco peppers, cubanelle peppers, chili peppers or Jalapeno peppers were submitted: [IR-4, 1985, PR 2947, MRID 157191; Baron, 1987, PR 3385, MRID 40224901, Baron, 1987, PR 2997, MRID 40225001, Baron, 1987, PR 3531, MRID 40448901, each not referenced]. As the manufacturer did not seek to have maximum residue levels estimated on chili peppers, no further action was taken in relation to the studies.

Tomato

One cGAP for tomatoes is available:

- cGAP from France with 1 ×0.38 kg ai/ha with a PHI of 35 days.

Trials that could be matched to this GAP were summarized.

Table 208 lists trials conducted in Brazil (2011), Southern France (1982, 1996), Italy (1997) and Spain (1996, 1997). A broadcast or banded foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 208. Results marked with "[AM]" are not selected for derivation of the MRL, if according to cGAP.

[AM] indicates that the analytical method did not contain a hydrolysis step and therefore fluazifop acid (II) conjugates are not included.

Additional trials from Canada (1980) were available on tomatoes with 1 × 0.40 kg ai/ha and harvest at 77 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Italy (1981) were available on tomatoes with 1 × 0.25–0.50–0.75 kg ai/ha and harvest at 89 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. These trials were not summarized, because they would not assist in MRL setting.

Table 208 Supervised field trials on tomatoes (whole fruit), treated with a broadcast or banded foliar fluazifop-butyl spray

TOMATO Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Piedade, SP, Brazil, 2011 (Rio Grande)	EW 250 (P)	1	0.25	0.25	BBCH 81; 7 June	SaC	85	30	< 0.01	M11033; AMA [Draetta, 2012, A12530B_10020]
Uberlandia, MG, Brazil, 2011; (Caryna)	EW 250 (P)	1	0.25	0.25	BBCH 73; 17 Mar	C	81	30	< 0.01	M11033; JJB [Draetta, 2012, A12530B_10020]
Formosa, GO, Brazil, 2011 (Helen)	EW 250 (P)	1	0.25	0.25	BBCH 64; 18 May	C	76	30	< 0.01	M11033; MFG [Draetta, 2012, A12530B_10020]
Palmeira, PR, Brazil, 2011 (Santa Clara)	EW 250 (P)	1	0.19	0.25	BBCH 72; 11 Mar	SaC	74	30	< 0.01	M11033; RWC [Draetta, 2012, A12530B_10020]
Lavras, MG, Brazil, 2011; (Santa Clara)	EW 250 (P)	1	0.25	0.25	BBCH 83; 9 Apr	C	88	30	< 0.01	M11033; RWC1 [Draetta, 2012, A12530B_10020]
Sonito; France, 1982; (Anita)	EC 250 (rac)	1	1.0	ns	30 June;	ns	ns	61	0.20 [AM]	RIC2816; Invuflec; [Culoto and Mallmann, 1983, PP5/0280]
idem	EC 250 (rac) + actiplus	1	0.38	0.044	23 June	ns	ns	61	< 0.02; < 0.02; < 0.02; 0.04 [d] [AM]	RIC2816; Invuflec; [Culoto and Mallmann, 1983, PP5/0280]
Firorenzuola; Emilia Romagna; Italy, 1997 (Red River)	EC 125 (P)	1	0.31	0.078	BBCH 51; 10-12 cm tall; 16 May	LC	ns	28 35 41	< 0.01 < 0.01 < 0.01	RJ2657B IT33-97-H350; [Jones *et al*., 1999, PP5/0175]
Borgo Sabotino; Lazio; Italy, 1997 (Joi)	EC 125 (P)	1	0.31	0.089	BBCH 51; 15-18 cm tall; 28 May	Sa	ns	28 35 42	< 0.01 < 0.01 < 0.01	RJ2657B IT42-97-H351; [Jones *et al*., 1999, PP5/0175]
Tamarite de Litera; Huesca; Spain, 1997 (Royesta)	EC 125 (P)	1	0.31	0.078	Flower Buds Visible; 13 June	C	ns	28 35 42	0.04 < 0.01 < 0.01	RJ2657B ES10-97-SH00; [Jones *et al*., 1999, PP5/0175]
idem	EC 125 (P)	1	0.31	0.078	Flower Buds Visible; 13 June	C	ns	28 42	0.10 < 0.01	RJ2657B ES10-97-SH108; [Jones *et al*., 1999, PP5/0175]
Fiorenzuola d'Arda; Emilia Romagna; Italy,	EC 125 (P)	1	0.31	0.078	all flowers set and first fruits pale	LC	CH	28	0.20	RJ2780B; IT30-98-H322 [Masonand Volpi, 1999, PP5/0177]

TOMATO Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
1998 (Ideal Peel)					6 July					
idem	idem	1	0.31	0.078	presence of flowers and fruits 22 June	idem	BBCH 89	42	0.25	idem
idem	idem	1	0.31	0.078	presence of flowers and fruits 8 June	idem	ns	56	0.16	idem
Pontinia; Lazio; Italy, 1998 (Snob)	EC 125 (P)	1	0.31	0.078	all flowers set and first fruits pale 7 July	Peaty	ns	28	0.04	RJ2780B; IT40-98-H323 [Masonand Volpi, 1999, PP5/0177]
idem	idem	1	0.31	0.078	presence of flowers and fruits 23 June	idem	ns	42	0.12	idem
idem	idem	1	0.31	0.078	presence of flowers and fruits 9 June	idem	ns	56	< 0.01	idem
Valdezorras; Sevilla; Spain, 1996 (Figaro)	EC 125 (P)	1	0.31	0.051	before flower buds visible; 18 June	CL	[a] [b] [c] MAT	21 28 35 45	0.03 0.02 0.01 0.03	RJ2268B ES10-96-SH002; [Jones *et al*., 1997, PP5/0169]
idem	EC 125 (P)	1	0.31	0.050	before flower buds visible; 18 June	CL	[a] [b] [c] MAT	21 28 35 45	0.01 < 0.01 0.06 0.06	RJ2268B ES10-96-SH102; [Jones *et al*., 1997, PP5/0169]
Banded foliar application over rows										
Velleron; Vaucluse; S-France, 1996 (Lerika)	EC 125 (P)	1	0.31	0.12	BBCH 52; 7 June	SiCSa	64 65 71 81	21 28 35 52	0.06 < 0.05 < 0.05 < 0.05	RJ2370B 96HCLSAP01; [Miles and Nassoy; 1997, PP5/0170]
Goult; Vaucluse; S-France, 1996 (Lerika)	EC 125 (P)	1	0.31	0.10	BBCH 52; 24 June	SiSaC	64 65 71 81	21 28 35 46	0.13 0.08 < 0.05 0.10	RJ2370B 96HCLSAP02; [Miles and Nassoy; 1997, PP5/0170]

GSH; growth stage at harvest:

[a] = hazelnut to nutsize fruit;

[b]= nut size fruit;

[c] = almost mature

[AM] Results are for free fluazifop acid (II); fluazifop-butyl < 0.01 mg/kg for each plot; conjugates are not included. Results are therefore underestimated and cannot be used for MRL-derivation.

[d] Results came from replicate plots; the highest value is taken for MRL derivation if according to cGAP

Additional trial information

M11033. GLP. No unusual weather conditions. Broadcast foliar application by pre-pressurized sprayer with CO2 gas with spray boom, spray volume 75-100 L/ha. Tomatoes (at least 12 items, at least 2 kg) were sampled by hand systematically from across the plots. Storage at -20 °C for 0.3-9.2 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET.138 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recoveries (795-103% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

RIC2816 Non-GLP. Weather conditions not reported. Foliar spray with spray volume 500 L/ha. Sample sizes not stated. Samples were stored at unknown conditions for 144 days. Samples were analysed for free fluazifop acid (II) using **HPLC-UV PPRAM 52 with a valid LOQ of 0.02 mg/kg.** Fluazifop acid (II) conjugates are not determined with this method. Samples were corrected for mean concurrent recovery (80% at 0.02–0.08 mg/kg for fluazifop acid); uncorrected results were not reported. Control samples < 0.02 mg/kg fluazifop acid; < 0.01 mg/kg fluazifop-butyl.

RJ2657B GLP study. No unusual weather conditions. Broadcast foliar spray using a motor knapsack sprayer with boom (Italy) or CO2 sprayer (Spain), spray volume 350-410 L/ha. Tomatoes (1.1-3.6 kg; up to 60 items, not indicated which trial corresponds to which sample size) were sampled by hand systematically from across the plots. Storage at -18 °C; for 68-398 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (75-100% at 0.05-0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2268B GLP study. No unusual weather conditions. Spray using a knapsack sprayer, spray volume 620-630 L/ha. Tomatoes (24 items from 12 plants at DAT 21-28; > 2 kg at DAT 35-45) were sampled by hand systematically from across the plots. Storage at -18 °C; for 90-188 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recoveries (89-92% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2780B GLP study. No unusual weather conditions. Spray using a knapsack sprayer, spray volume 400 L/ha. Tomatoes (24-40 items, 2.2-2.8 kg) were sampled by hand systematically from across the plots. Storage at -18 °C; for 100-101 days until extraction. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recovery (97% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2370B GLP study. No unusual weather conditions. **Banded spray over rows** using a handheld boom sprayer, spray volume 252-302 L/ha. Tomatoes (12 items) were sampled by hand systematically from across the plots. Storage at -14 °C; for 174-216 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recoveries (103% at 0.1 mg/kg). Control samples < 0.05 mg/kg.

Leafy vegetables

Endive

One cGAP for endive is available:

- cGAP from Belgium with 1 ×0.19 kg ai/ha with PHI 42 days

None of the available studies on endive were submitted: [Baron, 1987, report PR 2337, MRID 40341601; Baron, 1989, report PR 3921, MRID 41148301, each not referenced]. As the manufacturer did not seek to have maximum residue levels estimated on endive, no further efforts were taken to retrieve these studies.

Kale

Two cGAPs for kale are available:

- cGAP from France with 1 ×0.19 kg ai/ha and harvest at 42 days (underlining as nn)
- cGAP from the United Kingdom with 1 ×0.38 kg ai/ha and harvest at 56 days (kale for animal fodder, underlining as nn)

Trials that could be matched to these cGAPs were summarized.

Table 209 lists trials conducted in Germany (1981, 1991) and the UK (1979-1980, 1997, 1998). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 209. Results marked with "[QU]" are not selected for derivation of the MRL, if according to cGAP.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

Additional trials from the UK (1979-1980) were available on kale with 2 ×1.0, 1.0+1.5 or 1.0+2.0 kg ai/ha and harvest at 35 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. These trials were not summarized, because they would not assist in MRL setting.

Table 209 Supervised field trials on kale (whole plant), treated with a broadcast foliar fluazifop-butyl spray

KALE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 7 13 20 27 34 41	13 4.7 2.6 2.0 1.9 1.5 0.6 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 6 13 20 27 34	10 4.5 1.3 1.2 1.7 0.84 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany 1991 (var ns)	EC 125 (P)	1	0.19	ns	GS ns; date ns	ns	ns	47	0.95	AZ/83592/91; 91HJ068B1, [Gardyan, 1992, PP5/0129] (processing)
Location ns; UK, 1979-1980 (var ns)	EC 250 (rac)	1	0.50	ns	GS ns date ns	ns	ns	44 70	0.16 0.11 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	44 70	0.07 0.07 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Chard, Somerset; UK, 1997 (curly kale, Maris Kestrel)	EC 125 (P)	1	0.38	0.12	BBCH 7-9; crop height 25-35 cm; 14 July	CL	ns	56	0.16	RJ2654B; GB14-97-S031; [Jones *et al*., 1999, PP5/0140]
Bath; Somerset; UK; 1997 (curly kale, Keeper)	EC 125 (P)	1	0.38	0.12	BBCH 8-11; crop height 15-35 cm; 14 July	L	ns	56	0.22	RJ2654B; GB14-97-S032; [Jones *et al*., 1999, PP5/0140]
Bracknell, Berkshire, UK, 1998; (curly kale: Aris)	EC 125 (P) (YF1)	1	0.38	0.19	13-16 leaves; crop height 30 cm; 5 Aug	L	MAT	49	0.24	RJ2759B; GB14-98-S131; [Mason *et al*., 1999, PP5/0143]
idem	EC 125 (P) (YF2)	1	0.38	0.19	13-16 leaves; crop height 30 cm; 5 Aug	L	MAT	49	0.97	idem
Shere, Surrey UK, 1998; (curly kale: Winter Bore)	EC 125 (P) (YF1)	1	0.38	0.19	12 leaves; crop height 28 cm; 5 Aug	Sa	MAT	49	0.97	RJ2759B; GB14-98-S132; [Mason *et al*., 1999, PP5/0143]
idem	EC	1	0.43	0.22	12 leaves;	Sa	MAt	49	1.4	idem

KALE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	125 (P) (YF2)				crop height 28 cm; 5 Aug					
Bretforton; Worcestershire; UK, 1998; (curly kale: Winter Bore)	EC 125 (P))	1	0.38	0.19	BBCH 14-17; crop height 10-16 cm; 6 Jul	SaL	MAT	49	0.06	RJ2759B; GB14-98-S133; [Mason *et al*., 1999, PP5/0143]
idem	EC 125 (P)	1	0.38	0.19	BBCH 14-17; crop height 10-16 cm; 6 Jul	SaL	MAT	49	0.10	idem
Chipping Campden; Gloucestershire; UK, 1998; (curly kale: Winter Bore)	EC 125 (P)	1	0.38	0.19	BBCH 16-18; crop height 20-23 cm; 28 Jul	SiL	MAT	49	0.28	RJ2759B; GB14-98-S134; [Mason *et al*., 1999, PP5/0143]
idem	EC 125 (P)	1	0.38	0.19	BBCH 16-18; crop height 20-23 cm; 28 Jul	SiL	MAT	49	0.33	idem

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

Additional trial information

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.05 mg/kg (Germany 1981)

AZ83592/91. GLP. Weather, spray equipment and soil type were not reported. Spray volume not stated. Samples contained 2-3 kg brassica. Growth stage at harvest not stated. Storage conditions not stated. Samples were analysed for total fluazifop using HPLC-UV method **PPRAM 62/2 with additional clean-up with column chromatography**. Residues not corrected for concurrent method recoveries (70-114% at 0.05-1.0 mg/kg). Control samples were < 0.05 mg/kg.

RJ2654B GLP . Unusual weather conditions did not affect crop health. Varieties for use as game bird cover crop. Maris Kestrel is a low yielding short plant (70 cm full height) often grown as fodder crop. Keeper has a high leaf to stem ratio. Foliar spray using an CO2 pressurised knapsack sprayer, spray volume 300 L/ha. Kale plants were sampled by hand and taken systematically from across the plots. Leaves and petiole (2.0 kg) were taken from the top, middle and lower levels of at least 12 plants. Storage at -18 °C; for 66 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (77-82% at 0.05-0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2759B GLP . Unusual weather conditions did not affect crop health. Varieties commonly grown for human consumption. Winter bore grows to a height of 1.5 m as a bushy plant with finely curled leaves. Aris is medium tall with finely curled leaves. Broadcast foliar spray using an hand held boom sprayer, spray volume 200 L/ha. Kale plants were sampled by hand and taken systematically from across the plots. Leaves and petiole (2.0 kg) were taken from at least 12 plants. Storage at -18 °C; for 25-59 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (95-106% at 0.01-1.0 mg/kg). Control samples < 0.01 mg/kg.

Lettuce

Two cGAPs for lettuce are available:

- cGAP from Brazil with 1 ×0.25 kg ai/ha with harvest at 28 days
- cGAP from Belgium or France with 1 ×0.19 kg ai/ha with harvest at 42 days

Trials that could be matched to these cGAPs were summarized.

Table 210, Table 211 and Table 212 list trials on head lettuce, leaf lettuce and Cos lettuce conducted in Brazil (2011), France (1996, 1998, 2012), Greece (1997), Italy (1981, 1998, 2012) and Spain (1996, 2012). A broadcast or banded foliar spray or banded soil application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 210, Table 211 and Table 212. Results marked with "[QU]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62.

Additional trials from the UK (1980) were available on lettuce with 1 × 1.0–1.5–2.0 kg ai/ha and harvest at 55 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from the USA (1984, 2010, 2011) were available with 2 × 0.40–0.46 kg ai/ha with harvest at 7–16 DAT or 2 ×0.28 kg ai/ha with harvest at 29–30 or 44–45 DAT [Plyler and Francis, 1987, PP9/0107, report TMU3005/B revised; Arsenovic, 2013, PP5_50561, report IR-4 PR 02072]. These trials were not summarized, because they would not assist in MRL setting.

Besides total fluazifop, also despyridinyl acid (III) was analysed in lettuce samples from some trials conducted in the USA in 1981 and 1982 [Atreya, 1984, PP9/0728, PP009B272]. These trials were summarized in the metabolism section.

Table 210 Supervised field trials on head lettuce (whole plant), treated with a broadcast foliar fluazifop-butyl spray

HEAD LETTUCE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Martot; Normandy; N-France; 1996 (head lettuce Boston type: Opera)	EC 125 (P)	1	0.31	0.10	BBCH 19; 28 May	Sa	FL FL FL	21 29 41	0.01 ≤ 0.01 < 0.01	RJ2302B; S216.96 [Jones *et al*., 1997, PP5/0138]
Le Mesnil le Roi; Ile de France; N-France, 1996 (butterhead: Domino)	EC 125 (P)	1	0.31	0.10	BBCH 15; 9 Sept	SaL	43 47 49	21 30 42	0.04 < 0.01 < 0.01	RJ2363B AP/3221/ZE/2; [Miles and Cowley; 1997; PP5/0134]
La Moutonne; South-East; S-France, 1998 (Butterhead: Nadine)	EC 125 (P) (a)	1	0.31	0.10	10 leaves = BBCH 19; 15 cm tall; 70% soil cover; 20 Oct	C	49	31	0.22 0.66 [a]	RJ2782B S340.99; [Ryan and Atger, 1999, PP5/0145]
Grisolles; South-West; S-France, 1998 (butterhead: Locness)	EC 125 (P) (a)	1	0.31	0.10	6 leaves = BBCH 16; 8 cm tall; 10% soil cover; 29 Sept	SaL	49	41	< 0.01 < 0.01 [a]	RJ2782B S560.99; [Ryan and Atger, 1999, PP5/0145]
Location ns; Italy,	EC 250	1	0.25	ns	GS ns date ns	ns	ns	26	< 0.03	RJ0291B summary

HEAD LETTUCE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
1981 (var ns)	(rac)								[QU] [LOQ=0.05]	[Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.50	ns	GS ns date ns	ns	ns	26	< 0.03 [QU] [LOQ=0.05]	idem
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	26	< 0.03 [QU] [LOQ=0.05]	idem
Manfredonia; Puglia; Italy, 1998 (Closed Head: Classic)	EC 125 (P)	1	0.31	0.10	6-8 leaves =BBCH 16-18; 5-8 cm tall; 20 May	L	CH CH	30 40	< 0.01 < 0.01	RJ2786B IT50-98-H312; [Mason and Volpi, 1999; PP5/0351]

GSH: FL = formation of lettuce (early harvest of immature plants);
QU Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported
[a] Results came from 2 replicate plots, the highest value is taken for MRL derivation, , if according to cGAP.

Additional trial information

RJ2302B GLP No unusual weather conditions. Broadcast foliar spray using a hand held boom connected to a gas pressurised knapsack sprayer, spray volume 300 L/ha. Lettuces (12 plants) were sampled by hand systematically from across the plots. Storage at -18 °C; for 81-119 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (83-85% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2363B GLP study. No unusual weather conditions. Broadcast foliar spray using a hand held boom sprayer, spray volume 298-328 L/ha. Lettuce heads (12 heads, > 1 kg) were sampled by hand systematically from across the plots. Samples were trimmed to a marketable condition by trimming diseased and inedible leaves, but leaving the leaves which covered the plant head. Storage at -18 °C; for 85-217 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for averageconcurrent method recoveries (80-83% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2782B GLP study. No unusual weather conditions. Broadcast foliar spray using a handheld boom sprayer, spray volume 300 L/ha. Lettuce heads (12 heads, > 1 kg) were sampled by hand systematically from across the plots. Storage at -18 °C; for 69-80 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01mg/kg**. Samples were not corrected for individual concurrent method recoveries (80-107% at 0.01–0.05 mg/kg). Control samples < 0.01 mg/kg.

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported Spray volume not stated. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.03 mg/kg.

RJ2786B GLP study. No unusual weather conditions. Broadcast foliar spray using a gas knapsack sprayer with boom, spray volume 300 L/ha. Lettuce heads (12 heads) were sampled by hand systematically from across the plots. Samples were trimmed to a marketable condition by removing damaged and decaying leaves. Lettuce heads were put upside down in clean crates, rinsed with pouring water and drained. Storage at -18 °C; for 15-129 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (79-93% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

Table 211 Supervised field trials on leaf lettuce (whole plant), treated with a broadcast or banded foliar fluazifop-butyl spray

LEAF LETTUCE Location, Country; year; (variety)	For-mu-lation	no. of appl (inter-val)	kg ai/ha	kg ai/hL	GS; at last treatment day	Soil type	GSH	DALT	Total fluazifop (mg/kg)	Report no, Trial no. [ref]
Broadcast foliar sprays										
Piedade,	EW	1	0.25	0.25	BBCH	C	49	28	< 0.01	M11028;

LEAF LETTUCE Location, Country; year; (variety)	For-mu-lation	no. of appl (inter-val)	kg ai/ha	kg ai/hL	GS; at last treatment day	Soil type	GSH	DALT	Total fluazifop (mg/kg)	Report no, Trial no. [ref]
SP, Brazil, 2011; (leafy: alface crespa: Veronica)	250 (P)				16; 1 Apr					AMA1; [Draetta, 2012, A12530B_10013]
Engenheiro Coelho, SP, Brazil, 2011; (leafy: alface crespa: Bruna)	EW 250 (P)	1	0.25	0.25	BBCH 13-14; 1 Aug	SaC	47	28	< 0.01	M11028; AMA2; [Draetta, 2012, A12530B_10013]
Uberlandia, MG, Brazil, 2011; (leafy: alface crespa Veronica)	EW 250 (P)	1	0.25	0.25	BBCH 15; 25 Mar	C	45	28	< 0.01	M11028; JJB; [Draetta, 2012, A12530B_10013]
Planaltina, DF, Brazil, 2011; (leafy: alface crespa Vanda)	EW 250 (P)	1	0.25	0.25	BBCH 13; 25 May	SiCL	48	28	< 0.01	M11028; MFG; [Draetta, 2012, A12530B_10013]
Prunay; Champagne; N-France; 1996 (leafy, open head: Daphne)	EC 125 (P)	1	0.31	0.10	BBCH 22 10 May	Ch	30L 20 cm 25 cm	21 32 41	0.16 < 0.01 < 0.01	RJ2302B; S405.96 [Jones *et al*., 1997, PP5/0138]
St Pierre les Elbeuf; Seine Maritime; N-France, 1996 (Batavia open head: Vanity)	EC 125 (P)	1	0.31	0.10	BBCH 14; 14 June	Sa	45 49 51	21 30 42	0.04 < 0.01 < 0.01	RJ2363B AP/3221/ZE/1; [Miles and Cowley; 1997; PP5/0134]
31440; Merville; S-France, 2012 (leafy: Pitice)	EC 125 (P)	1	0.30	0.10	BBCH 14; 5 cm tall; 11 July	SiCL	49	41	< 0.01	CEMR-5451; SRFR12-006-37HR; [Langridge, 2013; A12791B_11028]
30000; Nimes; S-France, 2012 (leafy: Feuille de chêne)	EC 125 (P)	1	0.30	0.12	BBCH 12-15; 5-8 cm tall; 13 July	SiCL	48	42	< 0.01	CEMR-5451; SRFR12-007-37HR; [Langridge, 2013; A12791B_11028]
El Viso; Sevilla; Spain, 1996 (leafy, open head: Rubia Malagueña)	EC 125 (P)	1	0.31	0.10	BBCH 17-18; 29 May	LSa	36 49 49	21 30 42	< 0.01 < 0.01 < 0.01	RJ2363B AP/3221/ZE/3; [Miles and Cowley; 1997; PP5/0134]
Manfredonia; Puglia; Italy, 1998 (leafy, open head: PS 920)	EC 125 (P)	1	0.31	0.10	8-10 leaves; BBCH 18-19 28 Sept	L	CH CH	28 42	< 0.01 < 0.01	RJ2786B IT50-98-H313; [Mason and Volpi, 1999; PP5/0351]
74013;	EC	1	0.31	0.078	BBCH	C	19	0	21	CEMR-5451;

LEAF LETTUCE Location, Country; year; (variety)	For-mu-lation	no. of appl (inter-val)	kg ai/ha	kg ai/hL	GS; at last treatment day	Soil type	GSH	DALT	Total fluazifop (mg/kg)	Report no, Trial no. [ref]
Ginosa; Italy, 2012 (leafy, open head: Trocadero)	125 (P)				19; 5 cm tall; 5 July		41 43 44 46 49	7 14 21 28 39	2.8 0.19 < 0.01 < 0.01 < 0.01	SRIT12-1029-37HR; [Langridge, 2013; A12791B_11028]
70022; Altamura; Italy, 2012 (leafy, alface crespa, open head: Paola)	EC 125 (P)	1	0.31	0.078	BBCH 41; 18 June	L	41 42 43 45 47 49	0 7 14 21 28 39	16 3.1 0.23 0.02 < 0.01 < 0.01	CEMR-5451; SRIT12-1030-37HR; [Langridge, 2013; A12791B_11028]
Banded foliar spray over rows										
Marathonas; Attica Greece, 1997 (leafy, open head:Gramsi)	EC 125 (P)	1	0.31	0.062	3-4 leaves; 5-10 cm tall; 19 Sept	L	MAT	40	< 0.01	RJ2631B GR-97-H204; [Mason *et al*., 1999, PP5/0340]

Soil type: Ch = chalk;

GSH, 30L = 30 leaves (immature plants); 20 and 25 cm = diameter of the plant

Additional trial information

M11028 GLP No unusual weather conditions. Broadcast foliar spray using a pre-pressurised CO2 sprayer with boom, spray volume 100 L/ha. Lettuces (12 plants, > 1 kg) were sampled by hand systematically from across the plots. Storage at -20 °C; for 4.5-8.5 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT 138 rev 01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (104% at 0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2302B GLP No unusual weather conditions. Broadcast foliar spray using a hand held boom connected to a gas pressurised knapsack sprayer, spray volume 300 L/ha. Lettuces (12 plants) were sampled by hand systematically from across the plots. Storage at -18 °C; for 81-119 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (83-85% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2363B GLP study. No unusual weather conditions. Broadcast foliar spray using a hand held boom sprayer, spray volume 298-328 L/ha. Lettuce heads (12 heads, > 1 kg) were sampled by hand systematically from across the plots. Samples were trimmed to a marketable condition by trimming diseased and inedible leaves, but leaving the leaves which covered the plant head. Storage at -18 °C; for 85-217 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for averageconcurrent method recoveries (80-83% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

CEMR-5451 GLP study. No unusual weather conditions. Broadcast foliar spray using a knapsack sprayer (France) or boom sprayer with compressed air pump (Italy), spray volume 245-402 L/ha. Lettuce (>12 plants) were sampled by hand using a distributive pattern. Storage at -18 °C for a maximum of 271 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (71-103% at 0.01-30 mg/kg). Control samples < 0.01 mg/kg.

RJ2786B GLP study. No unusual weather conditions. Broadcast foliar spray using a gas knapsack sprayer with boom, spray volume 300 L/ha. Lettuce heads (12 heads) were sampled by hand systematically from across the plots. Samples were trimmed to a marketable condition by removing damaged and decaying leaves. Lettuce heads were put upside down in clean crates, rinsed with pouring water and drained. Storage at -18 °C; for 15-129 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (79-93% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2631B GLP study. No unusual weather conditions. **Banded spray over rows** using a knapsack sprayer with hand lance, spray volume 500 L/ha. Lettuce plants (12 plants) were sampled by hand systematically from across the plots. Samples were cleaned and trimmed to a marketable condition. Storage at -17 °C; for 85-90 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (106-114% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

Table 212 Supervised field trials on cos lettuce (whole plant), treated with a broadcast foliar or banded soil fluazifop-butyl spray

COS LETTUCE Location, Country; year; (variety)	For-mu-lation	no. of appl (inter-val)	kg ai/ha	kg ai/hL	GS; at last treatment day	Soil type	GSH	DALT	Total fluazifop (mg/kg)	Report no, Trial no. [ref]
Broadcast foliar sprays										
Trigueros; Huelva; Spain, 1996 (Cos: Inverna)	EC 125 (P)	1	0.31	0.10	BBCH 41; 9 May	SaSiL	49 49	21 30	< 0.01 < 0.01	RJ2363B AP/3221/ZE/4; [Miles and Cowley; 1997; PP5/0134]
Banded soil application										
Vilena; Alicante; Spain, 2012 (Cos: Larga verde)	EC 125 (P)	1	0.30	0.10	BBCH 13-14; 6 cm tall; 8 June	C	13 14 14 16 42 48	0 7 14 21 28 42	17 2.4 0.38 0.03 < 0.01 < 0.01	CEMR-5451; SRES12-209-37HR; [Langridge, 2013; A12791B_11028]
Vinarós; Castellón, Spain, 2012 (Cos: Cervantes)	EC 125 (P)	1	0.32	0.10	BBCH 14-16; 12 cm tall; 7 June	L	14 18 19 42 45 48	0 7 14 20 27 42	29 2.7 0.10 < 0.01 < 0.01 < 0.01	CEMR-5451; SRES12-210-37HR; [Langridge, 2013; A12791B_11028]

Additional trial information

RJ2363B GLP study. No unusual weather conditions. Broadcast foliar spray using a hand held boom sprayer, spray volume 298-328 L/ha. Lettuce heads (12 heads, > 1 kg) were sampled by hand systematically from across the plots. Samples were trimmed to a marketable condition by trimming diseased and inedible leaves, but leaving the leaves which covered the plant head. Storage at -18 °C; for 85-217 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for averageconcurrent method recoveries (80-83% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

CEMR-5451 GLP study. No unusual weather conditions. **Banded soil application** using a knapsack sprayer (Spain), spray volume 245-402 L/ha. Lettuce (>12 plants) were sampled by hand using a distributive pattern. Storage at -18 °C for a maximum of 271 days. Spanish samples reached a maximum of -0.6 ºC for 1 hr during transport. Since the samples remained frozen, this is considered to have no impact on the residue levels. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (71-103% at 0.01-30 mg/kg). Control samples < 0.01 mg/kg.

Spinach

Two cGAPs for spinach are available:

- cGAP from Belgium with 1 ×0.38 kg ai/ha with a PHI of 42days
- cGAP from France with 1 ×0.28 kg ai/ha with a PHI of 28 days

Trials from Italy (1981) were available on spinach with an application of 1 × 0.25–0.38–0.75 kg ai/ha and harvest at 9–20 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Trials from Germany (1997) were available on spinach with an application of 1 × 0.38 kg ai/ha and harvest at 21–29 DAT [Mason *et al*., 1999, PP5/0139, report RJ2632B]. Since the manufacturer did not intend to have MRLs on spinach, the available studies on spinach were not summarized.

Some available supervised trials on spinach were not submitted: [Baron, 1987, MRID 40341101, report PR-2073, not referenced]. No further efforts were taken to retrieve these studies.

Turnip greens

Three cGAPs for turnips are available:

- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 42 days

- cGAP from Belgium with 1 ×0.38kg ai/ha and a PHI of 56 days
- cGAP from the UK with 1 x 0.38 kg ai/ha and a PHI of 56 days (stock feed only)

Turnip tops are harvested at the same time as the roots. Trials that could be matched to these cGAPs were summarized.

Table 213 lists trials conducted in the UK (1990). A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 213.

Additional trials from the United Kingdom (1980) were available on turnip tops with 1 ×0.5–1.0 kg ai/ha, DAT 23 days [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. These trials were not summarized, because they would not assist in MRL setting.

Table 213 Supervised field trials on turnip (tops), treated with a broadcast foliar fluazifop-butyl spray

TURNIP TOPS Location, Country, year (Variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Balerno, Mid Lothian; UK; 1990; (Wallace)	EW 125 (P) + Agral	1	0.38	0.19	10-12 leaves; 50% crop cover; 5 July	SiL	CH	68	0.94	RJ0997B; GB18-90-S481; [Jones, 1992, PP5/0099]
idem	EW 250 (P) +ajd	1	0.38	0.19	10-12 leaves; 50% crop cover; 5 July	SiL	CH	68	1.6	idem
idem	EC 125 (P) + Agral	1	0.38	0.19	10-12 leaves; 50% crop cover; 5 July	SiL	CH	68	1.1	idem
Gorebridge, Mid Lothian; UK; 1990; (Wallace)	EW 125 (P) + Agral	1	0.38	0.19	12 leaves; 30-50% crop cover; 2 Aug	CL	CH	62	1.2	RJ0997B; GB18-90-S482; [Jones, 1992, PP5/0099]
idem	EW 250 (P) + Agral	1	0.38	0.19	12 leaves; 30-50% crop cover; 2 Aug	CL	CH	62	0.91	idem
idem	EC 125 (P) + Agral	1	0.38	0.19	12 leaves; 30-50% crop cover; 2 Aug	CL	CH	62	1.3	idem

Additional trial information:

RJ0997B. GLP. No unusual weather conditions. Spray application using a hand-held CO_2 pressurised knapsack sprayer. Spray volume 200 L/ha. Samples of 12 plants. Roots and leaves were separated. Roots were subsampled in the field: opposite quarters of the roots were retained for analysis. Storage at -18 °C; storage time not stated but less than 24 months. Samples were analysed for total fluazifop using **NMR method PPRAM 83 using internal standard with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (99% at 0.5 mg/kg). Control samples < 0.05 mg/kg.

Legume vegetables

Green beans with pods (Phaseolus spp)

Two cGAPs for green Phaseolus beans are available:

- cGAP from the Netherlands with 1 × 0.38 kg ai/ha with a PHI of 60 days (beans with pods)
- cGAP from Belgium with 1 ×0.38 kg ai/ha with a PHI of 28 days (green *Phaseolus* beans)

Trials that could be matched to these cGAPs were summarized.

Table 214 lists trials conducted in Canada (1980), Germany (1996, 1997), the UK (1980, 2001), the Netherlands (1996, 1997), France (2001, 2006, 2008, 2009), Spain (1987, 1999, 2008, 2009). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 214. Results marked with "[QU]" or "[SS]" or "GS" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[GS] indicates that the growth stage at harvest does not represent the commercial product

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for NMR method PPRAM 83.

Additional trials from the Netherlands (1981) were available on green beans with 1 × 0.38-0.75 kg ai/ha with harvest at 13 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Italy (1981) were available on green beans with 1 × 0.25–0.50–0.75 kg ai/ha with harvest at 41 or 55 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Spain (1997) on green Phaseolus beans with pods were available with 1 × 0.31 kg ai/ha with harvest at 19 DAT [Mason *et al*., 1998, PP5/0365, report RJ2493B]. These trials were not summarized, because they would not assist in MRL setting.

Table 214 Supervised field trials on Phaseolus beans (green beans with pods), treated with a broadcast foliar fluazifop-butyl spray

GREEN PHASEOLUS BEANS WITH PODS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Location ns; Canada, 1980 (snap beans; var ns)	EC 250 (rac)	1	0.12	ns	GS ns date ns	ns	ns	58	0.05 [QU]	RJ0226B summary [Atreya and Froggatt, 1981, PP9/0384]
idem	EC 250 (rac)	1	0.25	ns	GS ns date ns	ns	ns	58	0.06 [QU]	idem
idem	EC 250 (rac)	1	0.50	ns	GS ns date ns	ns	ns	58	0.29 [QU]	idem
D-94574 Wallerfing-Oberviehhausen Germany; 1996; (Scuba)	EC 125 (P)	1	0.38	0.15	BBCH 22-29; 40% crop cover; 28 June	SaL	75	43	0.07	RJ2290B; RS-9619-G1 [Jones *et al*., 1997, PP5/0151]
D-04749 Pulsitz; Germany; 1996; (Maradona)	EC 125 (P)	1	0.38	0.19	BBCH 33; 20% crop cover; 10 July	L	77	34	0.08	RJ2290B; RS-9619-K1 [Jones *et al*., 1997, PP5/0151]
D-94574 Wallerfing-	EC 125	1	0.38	0.12	BBCH 51;	SiL	73	27	0.17	RJ2629B; RS-9730-G1

GREEN PHASEOLUS BEANS WITH PODS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Oberviehhausen; Germany; 1997; (Scuba)	(P)				35% crop cover; 01 July					[Mason *et al*., 1999, PP5/0156]
D-53919 Mulheim-Ottenheim; Germany; 1997; (Forum)	EC 125 (P)	1	0.38	0.094	BBCH 51; 70% crop cover; 10 July	L	77	28	0.38	RJ2629B; RS-9730-H1 [Mason *et al*., 1999, PP5/0156]
Location ns; UK, 1980 (green beans; var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	58	1.0, 1.2, 1.5 [QU]	RJ0226B summary [Atreya and Froggatt, 1981, PP9/0384]
Hemington; Leicestershire; UK; 2001 (Forum)	EC 125 (P)	1	0.38	0.12	BBCH 22-24; 15 Jul	CL	CH	39	0.05	RJ3299B; AF/6070/SY1 [McGill and Crawford, 2002, PP5/1232]
Stourport on Seven; Worcestershire; UK; 2001 (Tasman)	EC 125 (P)	1	0.38	0.12	BBCH 37; 21 June	SaL	CH	34	0.25	RJ3299B; AF/6070/SY2 [McGill and Crawford, 2002, PP5/1232]
Lelystad; Netherlands; 1996; (ns)	EC 125 (P)	1	0.38	0.075	BBCH 55; 8 Aug	C	75	28	0.29	RJ2287B; NL10-96-H209 [Jones *et al*., 1997, PP5/0152]
Dronten; Netherlands; 1996; (ns)	EC 125 (P)	1	0.38	0.075	BBCH 51; 13 Aug	C	75	29	0.23	RJ2287B; NL10-96-H210; [Jones *et al*., 1997, PP5/0152]
Biddinghuizen; Netherlands; 1997; (Odessa)	EC 125 (P)	1	0.38	0.075	BBCH 51; 13 Aug	C	720	27	0.35	RJ2611B; NL10-97-H125; [Mason and Bouwman, 19998, PP5/0154]
Dronten; Netherlands; 1997; (Odessa)	EC 125 (P)	1	0.41	0.083	BBCH 51; 13 Aug	C	710	27	0.48	RJ2611B; NL10-97-H126 [Mason and Bouwman, 19998, PP5/0154]
Goury; Eure et Loir; N-France; 2001 (NU9699)	EC 125 (P)	1	0.38	0.12	BBCH 37-38; 25 July	CL	CH	36	0.02	RJ3299B; AF/6070/SY3 [McGill and Crawford, 2002, PP5/1232]
Montauban; Tarn-et-Garonne; S-France, 2001; (Adana)	EC 125 (P)	1	0.31	0.10	10 cm height; BBCH 15; 7 June	SiL	CH	33	0.20	RJ3294B; AF/5939/SY/1 [Mason and Clark, 2003, PP5/1333]
St Jory, Haute-Garonne; S-France; 2001 (Bouster)	EC 125 (P)	1	0.31	0.11	20 cm height; BBCH 14; 13 July	SaL	CH	38	0.08	RJ3294B; AF/5939/SY/2 [Mason and Clark, 2003, PP5/1333]
13210 St Remy; Provence;	EC 125 (P)	1	0.34	0.11	BBCH 23; 7 Jul	C	74	34	0.05	CEMR-3014; FR-HR-06-0225 [Bell, 2008,

GREEN PHASEOLUS BEANS WITH PODS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
S-France, 2006 (Booster)										A12791B_10430]
47180 Meilhan S-France, 2006 (Denver)	EC 125 (P)	1	0.33	0.11	BBCH 14; 7 July	L	75	34	0.02	CEMR-3014; FR-HR-06-0226 [Bell, 2008, A12791B_10430]
34590 Marsillargues; S-France, 2006; (Booster)	EC 125 (P)	1	0.30	0.10	BBCH 39; 9 June	CL	79	33	0.02	CEMR-3014; FR-HR-06-0227 [Bell, 2008, A12791B_10430]
82170 Grisolles; S-France, 2006 (Callisto)	EC 125 (P)	1	0.30	0.10	BBCH 29; 30 June	SiC	79	27	0.27	CEMR-3014; FR-HR-06-0228 [Bell, 2008, A12791B_10430]
40800 Duhort Bachen; S-France; 2006; (Angers)	EC 125 (P)	1	0.31	0.099	BBCH 39 31 July	L	89 dry!	30	0.60 [GS]	CEMR-3014; FR-HR-06-0229 [Bell, 2008, A12791B_10430]
Pexiora, Languedoc-Roussilon; S-France, 2008 (Linex)	EC 125 (P)	1	0.32	0.078	BBCH 71; 7 July	CL	71	28	4.6	T009248-07-REG; S08-01602-01 [Marshall, 2009, A12791B_10788]
Pexiora, Aude, 11150, S-France, 2009; (Livex)	EC 125 (P)	1	0.31	0.052	BBCH 51; 7 July	CL	75	28	1.6	CEMR-4384-REG; S09-00354-01 [Jutsum, 2011, A12791B_10829]
Montauban, 82000, S-France, 2009; (Rigoleto)	EC 125 (P)	1	0.32	0.078	BBCH 25, 20 July	SL	78-79	28	0.06	CEMR-4384-REG; S09-00354-02 [Jutsum, 2011, A12791B_10829]
Boncellino, Emilia Romagna; Italy, 2001 (Masai)	EC 125 (P)	1	0.31	0.11	15 cm height; BBCH 37-38; 28 Aug	Sa SiL	CH	41	0.06	RJ3294B; AF/5939/SY/3 [Mason and Clark, 2003, PP5/1333]
Almussafes; Valencia; Spain, 1987 (ns)	EC 300 (P)	1	0.30	ns	20% crop cover; 2 June	ns	ns	55	0.03 [SS] [LOQ=0.05]	M4799B ES01-87-D005-H; [Crook, 1988, PP5/0380]
Santo Domingo de la Calzada; Rioja; Spain, 1999 (Vina)	EC 125 (P)	1	0.31	0.080	<35 cm high; before BBCH 50; 28 July	C	CH	29	0.32	RJ2993B; ES30-99-S026 [Mason *et al.*, 2000, PP5/0372]
Funes, 31360 Navarra; Spain; 2008;	EC 125 (P)	1	0.31	0.078	BBCH 49; 27 Aug	SaCL	49	28	0.84	TK009248-07-REG; S08-01602-02 [Marshall, 2009,

GREEN PHASEOLUS BEANS WITH PODS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Moncayo)										A12791B_10788]
Bolbaite, Canal Navarres, Valencia, Spain, 2009; (Cleo)	EC 125 (P)	1	0.35	0.078	BBCH 29; 24 Aug	CL	81 dry!	38	0.15 [GS]	CEMR-4384-REG; S09-00354-03; [Jutsum, 2011, A12791B_10829]
Xativa, La Costera, Valencia Spain, 2009; (Cardeno)	EC 125 (P)	1	0.34	0.097	BBCH 29 24 Aug	CL	82 dry!	39	< 0.01 [GS]	CEMR-4384-REG; S09-00354-04 [Jutsum, 2011, A12791B_10829]

BBCH 20-29: Formation of side shoots. BBCH 30-49: Growing (leaves only). BBCH 50-59: inflorescence emergence (BBCH 51 = First flower buds visible. BBCH 55 = First flower buds enlarged). BBCH 60-69: flowering. BBCH 70-79: development of fruit (BBCH 70-71: Beginning of pod development or 10% of pods have reached typical length).

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[GS] beans with pods at BBCH >80 are considered not representative for commercial green beans with pods and trials are not selected for MRL derivation

Additional trial information

RJ0226B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries not indicated. Control samples were < 0.05 mg/kg.

RJ2290B. GLP. French beans. No unusual weather conditions. Air pressurised knapsack sprayer with hand-held 2.5 m boom. Spray volume 200-250 L/ha. Sample sizes 30 plants or 1 kg. Storage at -18 °C for 114-117 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were corrected for average concurrent method recoveries (86% at 0.1 mg/kg); uncorrected results are not reported. Control samples < 0.01–0.01 mg/kg. LOQ needs to be increased to 0.01/0.3 = 0.04 mg/kg. However, since the residue results were >0.04 mg/kg, this has no impact on the study.

RJ2629B. GLP. Dwarf French beans. No unusual weather conditions. Air pressurized knapsack sprayer with hand-held 2.5 mg boom. Spray volume 300-400 L/ha. Sample sizes 30 plants or 1 kg. Storage at -18 °C for 154-164 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent external recovery (100-101% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ3299B. GLP. French dwarf beans (Phaseolus vulgaris). Unusual weather conditions did not effect crop health. Overall foliar spray using a precision boom sprayer. Spray volume 302-307 L/ha. Samples (> 12 plants) were taken by hand systematically from all areas of the plot. Sample sizes > 24 whole pods . Storage at -18 °C for 138-174 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent external recovery (102-104% at 0.1–0.2 mg/kg). Control samples < 0.01 mg/kg.

RJ2287B. GLP. Green Beans with edible pods (Phaseolus vulgaris). No unusual weather conditions. Compressed air knapsack sprayer. Spray volume 500 L/ha. Samples were taken by hand sytematically from across the plots. Sample sizes 1.0-1.4 kg. Storage at -18 °C for 85-91 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recoveries (99% at 0.3 mg/kg). Control samples < 0.01–0.01 mg/kg. LOQ needs to be increased to 0.01/0.3=0.04 mg/kg. However, since the residue results were >0.04 mg/kg, this has no impact on the study.

RJ2611B. GLP. Fresh beans (Phaseolus vulgaris). No unusual weather conditions. Air pressurised knapsack sprayer. Spray volume 500 L/ha. Samples were taken by hand systematically from across the plots. Sample sizes 1.0-1.4 kg. Storage at -18 °C at 94 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (84-90% at 0.05-0.25 mg/kg). Control samples < 0.01 mg/kg.

RJ3294B. GLP. Fresh beans (Phaseolus spp). Weather conditions had no effect on crop health. Overall foliar spray using a precision boom sprayer. Spray volume 289-314 L/ha. Samples were taken by hand from across the plot from at least 12 plants. Sample sizes 24 whole pods with 1.0 kg minimum. Storage at -18 °C or lower for 105-195 days. Samples were

analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (108-111% at 0.1-1.0 mg/kg). Control samples < 0.01 mg/kg.

CEMR-3014. GLP. Fresh beans with pods. No unusual weather conditions. Overall foliar spray using a knapsack sprayer. Spray volume 300-316 L/ha. Samples were taken by hand. Sample sizes 1.0-1.5 kg beans with pods. Storage at -18 °C or lower for 469 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (88-104% at 0.01-1.0 mg/kg pods). Control samples < 0.01 mg/kg.

T009248-07-REG. GLP. Fresh beans with pods (Phaseolus spp). No unusual weather conditions. Broadcast foliar spray using a boom sprayer. Spray volume 400 L/ha. Samples were taken by hand. Sample sizes 2.0-2.4 kg beans with pods (at least 24 units). Storage at -12 °C or lower for 176-227 days (7.5 months). Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (80-106% at 0.01-4.0 mg/kg pods). Control samples < 0.01 mg/kg.

CEMR-4384-REG. GLP. Field beans (Phaseolus spp). No unusual weather conditions. Broadcast foliar spray using a boom sprayer. Spray volume 595 L/ha. Samples were taken by hand from 12-16 areas of the plot. Sample sizes 1.0-1.6 kg beans with pods. Storage at -12 °C or lower for 371 days. Samples were left at +1 °C for 2.5 hrs. Since the samples remained frozen, this is considered to have no impact on the study results. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent external recovery (64-80% (beans with pods) at 0.01-2.0 mg/kg). Control samples < 0.01 mg/kg.

M4799B Non-GLP study. Weather conditions, soil type, application equipment, spray volume, sample sizes and growth stage at harvest not stated. Storage at -18 °C; for maximum 277 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Internal standard recovery (98% at 0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2993B. GLP. Fresh beans (Phaseolus spp). No unusual weather conditions. Application by gas knapsack sprayer with a lance. Spray volume 393 L/ha. Samples were taken by hand from across the plot. Sample sizes >2.1 kg beans with pods. Storage at -18 °C or lower for 81 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (105-118% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg.

Green peas with pods (Pisum spp, Vigna spp)

One cGAP for green peas with pods is available:

- cGAP from Belgium with 1 ×0.38 kg ai/ha with a PHI of 28 days for peas with pods

Trials that could be matched to this GAP were summarized.

Table 215 lists trials conducted in the Canada (1983, 1990), the UK (1981, 2006, 2010), Netherlands (1981, 1984), Northern France (2006) and Spain (2006). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 215. Results marked with "[QU]"" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2.

Additional trials from the Netherlands (1985) were available on green peas with pods with 1 ×0.19 or 0.38 kg ai/ha with harvest at 42 or 56 DAT [Crook and Harradine, 1986, PP5/0161, M4261B, trial 85-216]. Additional trials from Denmark (1989) were available on green peas with pods with 1 × 0.38 kg ai/ha with harvest at 62 DAT [Jones, 1991, PP5/0150, report M5347B]. These trials were not summarized, because they would not assist in MRL setting.

Table 215 Supervised field trials on Pisum peas (green peas with pods), treated with a broadcast foliar fluazifop-butyl spray

GREEN PEA PODS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Location ns; Canada, 1983, (vining pea: Little Marvel)	EC 250 (rac)	1	0.50	ns	5-10 cm high	ns	green	34	0.08, 0.20, mean 0.14 [a]	M3754B; CA/QU/HE/83/410/C; [Harradine, 1984, PP9/0116]
Dalmeny; Saskatchewan, Canada, 1990 (field peas)	WG 250 (rac)	1	0.12	ns	8-18 cm; vegetative; stage BJ 104-105; 50% soil cover; 14 June	SaL	BJ 207	45	< 0.05	RJ1059B; CA-50-90-S912; [Jones, 1992, PP5/0405]
Location ns; UK, 1981 (pods var ns)	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	38 49 56	0.04 < 0.02 0.29 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; UK, 1981 (pods, var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	38 49 56	0.18, 0.22, 0.09 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Holbeach Hurn; UK, 2006; (Geisha)	EC 125 (P)	1	0.38	0.075	BBCH 16-17; 10 Jul	SaSiL	79	35	0.90	CEMR-3009; AF/10375/SY1 [Bell, 2008, PP5/1552]
Thwing; UK, 2006; (Ibis)	EC 125 (P)	1	0.38	0.075	BBCH 15-21; 5 Jul	SaL	77	35	0.08	CEMR-3009; AF/10375/SY2 [Bell, 2008, PP5/1552]
Luddington; Warwickshire; CV37 9SJ UK; 2010; (Samish)	EC 125 (P)	1	0.37	0.083	BBCH 38-39; 3 June	SiL	79	34	0.85	CEMR-4658-REG; CEMS-4658-02; [Jutsum and Allen, 2011, A1279B_10837]
Location ns; Netherlands, 1981 (pods; var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	53	< 0.02, 0.03, 0.03, 0.03 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Netherlands, 1981 (pods; var ns)	EC 250 (rac)	1	1.25	ns	GS ns date ns	ns	ns	53	0.05, 0.05, 0.05, 0.09 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Beerta; Netherlands	EC 125	1	0.38	0.075	pre-flower;	C	16d PCH	63	< 0.03; < 0.03;	M3976B; 84-132;

GREEN PEA PODS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
; 1984; (canning pea: Joff)	(P) +Agral				6-8 leaves; 60% crop cover; 1 June		green		< 0.03; < 0.03; mean < 0.03 [a] [SS]	[Dick and Rounds, 1985, PP5/0412]
28140 Loigny la Bataille; N-France; 2006; (Piano)	EC 125 (P)	1	0.38	0.075	BBCH 51; 12 May	CL	79	35	0.23	CEMR-3009; AF/10375/SY3 [Bell, 2008, PP5/1552]
71530 Viney le Grand; N-France; 2006; (Atlanta)	EC 125 (P)	1	0.39	0.075	BBCH 59; 1 Jun	SaL	79	35	0.42	CEMR-3009; AF/10375/SY4 [Bell, 2008, PP5/1552]
22280; Gurrea de Gallego; Spain, 2006; (Valverde)	EC 125 (P)	1	0.32	0.062	BBCH 57; 5 May	CL	77	35	0.16	CEMR-3012; AF/10376/SY1 [Bell, 2007, PP5/1550]
22196; Huesca; Spain, 2006; (Meteor)	EC 125 (P)	1	0.32	0.062	BBCH 57; 5 May	CL	77	35	0.55	CEMR-3012; AF/10376/SY2 [Bell, 2007, PP5/1550]

GSH: 16dPCH = 16 days before commercial harvest;

BJ = Bjorkman scale; Code 105-107 is vegetative; 203 is 10% flowering, 207 is podding, 303 is mature

QU Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

Additional trial information

M3754B. Non-GLP. Weather conditions, spray equipment, spray volumes not stated. Sample sizes pea seeds (> 1 kg) , pods (> 0.5 kg). Storage at -18 °C or lower for a maximum of 977days (1981 trials), 612 days (1982 trials), 247 days (1983 trials) (harvet to report date). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with internal standard with a valid LOQ of 0.05 mg/kg**. Samples were corrected for internal std recovery (58-86% at 1.0 mg/kg). Control samples < 0.02 mg/kg (1983 trials) or < 0.05 mg/kg (1981 and 1982 trials).

RJ1059B. GLP. No unusual weather conditions. Spray equipment andspray volumes not stated. Sampling from 10 areas within a plot by cutting with scissors at ground level. Sample sizes pods (> 1.0 kg). Storage at -18 °C or lower for a maximum of 412 days. Samples were analysed for total fluazifop using **NMR methods PPRAM 83 and RAM 197 with internal standard, each with a valid LOQ of 0.05 mg/kg**. Samples were corrected for external std recovery (91% at 0.1 mg/kg), uncorrected results are not reported. Control samples < 0.05 mg/kg.

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.02mg/kg (Netherlands, 1981; UK, 1981)

CEMR-3009. GLP Fresh Peas. No unusual weather conditions. Foliar spray using a plot sprayer. Spray volume 502-515 L/ha. Fresh pea plants were sampled by hand (9-14 Aug in the UK; 16 June, 6 July in France) and threshed using a mini pea viner (UK) or by hand (N-France) resulting in 0.9-1.2 kg pea pods. Storage at -15 °C for a maximum of 495 days. The temperature reached a maximum of -9 ºC for a peak of 3 days. This is considered to have no effect on the study, since the samples remained frozen at all times. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg.** Samples were not corrected for individual concurrent method recovery (71-76% at 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg.

CEMR-4658. GLP Fresh peas. No unusual weather conditions. Broadcast foliar spray using a backpack sprayer. Spray volume 379-448 L/ha. Fresh pea plants (12 units) were taken by hand (7 July UK) using a suitable distributive pattern.

Sample sizes >1 kg pea pods. Storage at -18 °C for a maximum of 317 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (70-92% at 0.01–0.1mg/kg for seeds, 71-76% at 0.01–0.5 mg/kg for pods. Control samples < 0.01 mg/kg (seeds, pods).

M3976B. Non-GLP. Vining peas. No unusual weather conditions. Broadcast foliar spray using a knapsack propane gas sprayer. Spray volume 500L/ha. Pods were sampled by hand (3 August). Seeds and pods were separated. Sample sizes were not stated. Storage at -20°C. Storage time not stated but no longer than 314 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (64% at 0.5 mg/kg). Control samples < 0.03 mg/kg.

CEMR-3012. GLP. Fresh Peas. No unusual weather conditions. Foliar spray using a plot sprayer. Spray volume 508-509 L/ha. Fresh pea pods (> 1 kg) were sampled by hand. Storage at -15 °C for a a maximum of 436 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (85-99% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

Green pea seeds (Pisum sativum)

Two possible cGAPs for green pea seeds are available

- cGAP from Belgium is 1 ×0.38 kg ai/ha with PHI 28 days for peas without pods
- cGAP from the Netherlands is 1 ×0.38 kg ai/ha with PHI 56 days for peas without pods (underling as nn)

Trials that could be matched to these cGAPs were summarized.

Table 216 lists trials conducted in Canada (1981, 1982, 1983), Germany (1985, 2010), UK (1980, 1981, 1984, 2001, 2006, 2010), Netherlands (1980, 1984, 1985), France (2006, 2012), Italy (1997) and Spain (2003, 2006, 2012). A broadcast or banded foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 216. Results marked with "[QU]", "[SS]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (> 25% of residue value).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2 or NMR method PPRAM 83.

Additional trials from Italy (1981) were available on peas with 1 × 0.25-0.50-1.0 kg ai/ha with harvest at 36 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Sweden (1980) were available on peas with 2 × 0.25 kg ai/ha with harvest at 27 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from the Netherlands were available on green pea seeds with 1 × 0.19 kg ai/ha with harvest at 42 days [Crook and Harradine, 1986, PP5/0161, report M4261B, trials 85–210, 85–211, 85–215]. These trials were not summarized, because they would not assist in MRL setting.

Table 216 Supervised field trials on Pisum peas (green pea seeds), treated with a broadcast or banded foliar fluazifop-butyl spray

GREEN PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Location ns; Canada,	EC 250	1	0.40	ns	June	ns	green	25	< 0.05	M3754B; CA/BC/HE/81/760/

GREEN PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
1981, (vining pea: Improved Laxtons Progress)	(rac)									X; RDS 3568 [Harradine, 1984, PP9/0116]
idem	EC 250 (rac)	1	1.0	ns	June	ns	green	25	< 0.05	idem
Location ns; Canada, 1982, (vining pea: Improved Laxtons Progress)	EC 250 (rac)	1	0.40	ns	June	ns	green	42	< 0.05	M3754B; CA/BC/HE/82/760/X; RDS 5069 [Harradine, 1984, PP9/0116]
Location ns; Canada, 1983, (vining pea: Little Marvle)	EC 250 (rac)	1	0.50	ns	3 leaves	ns	green	69	0.053 [b], 0.10 [b], mean 0.076	M3754B; CA/ON/HE/83/508/C [Harradine, 1984, PP9/0116]
idem	EC 125 (P)	1	0.25	ns	3 leaves	ns	green	69	0.030 [b]	idem
6749 Dierbach; Germany; 1985 (fodder pea; Stehgold)	EC 125 (P)	1	0.38	0.094	BBA07; 8-12 cm high; 40% soil cover; 15 May	SaL	green seeds	65	0.22	M4234B; RS8528E1 [Pay, 1986, PP5/0396]
idem	EC 125 (P)	1	0.38	0.094	BBA09; 30 cm high; 95% soil cover; 30 May	SaL	green seeds	50	3.8	idem
idem	EC 125 (P)	1	0.38	0.094	BBA11; 40 cm high 100% soil cover; in bloom 11 June	SaL	green seeds	38	8.6	idem
2409 Neustadt-Holstein; Germany; 1985; (fodder pea; Birte)	EC 125 (P)	1	0.38	0.094	BBA07; 50% soil cover; 22 May	SaL	green seeds	86	0.10	M4234B; RS8528B1 [Pay, 1986, PP5/0396]
idem	EC 125 (P)	1	0.38	0.094	BBA09; 80% soil cover; 11 June	SaL	green seeds	66	0.44	idem
idem	EC 125 (P)	1	0.38	0.094	BBA11; 100% soil	SaL	green seeds	46	7.6	idem

GREEN PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					cover; in bloom 1 Jul					
2059 Brötchen-Büchen; Germany; 1985; (fodder pea; Columba)	EC 125 (P)	1	0.38	0.094	BBA07; 70% soil cover; 18 May	Sa	green seeds	79	0.13	M4234B; RS8528B2 [Pay, 1986, PP5/0396]
idem	EC 125 (P)	1	0.38	0.094	BBA09; 90% soil cover; 31 May	Sa	green seeds	66	0.27	idem
idem	EC 125 (P)	1	0.38	0.094	BBA11; 90% soil cover; in bloom 13 June	Sa	green seeds	53	NA	idem
Bardowick, Lueneburg, Germany, 2010; (Maxigold)	EC 125 (P)	1	0.76	0.19	BBCH 35-36; 10 August, 2010	Sa	75-79	37	0.86 [b] [CT] [cntrl=0.29]	CEMR-4751-REG; CEMS-4751-03; [Langridge, 2013, A12791B_11029] (processing)
Idem	EC 125 (P)	1	1.9	0.45	BBCH 34-35; 10 August, 2010	Sa	75-79	37	Not reported	idem
Location ns; UK, 1980 (peas, var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	35 56 63	0.88 0.03 0.01 [QU] [LOQ=0.05]	RJ0226B summary [Atreya and Froggatt, 1981, PP9/0384]
Location ns; UK, 1981 (peas, var ns)	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	38 49 56	< 0.02 < 0.02 < 0.02 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; UK, 1981 (peas, var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	38 49 56	0.22, < 0.02 0.05 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Kettlestone; UK; 1984; (vining pea: Sprite)	EC 125 (P)	1	0.19	0.085	flower buds visible; 100% crop	LSa	CH green	42	0.23 [SS]	M4008B; trial ns; [Harradine, 1985, PP5/0397]

GREEN PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					cover; 2 June					
idem	EC 125 (P)	1	0.38	0.17	flower buds visible; 100% crop cover; 2 June	LSa	CH green	42	< 0.03 [SS] [LOQ=0.05]	idem
Thornhaugh, Peterborough UK; 1984; (vining pea: Dark skinned perfection)	EC 125 (P)	1	0.19	0.085	no buds; 6 leaves; 100% crop cover; 4 June	L	CH green	46	< 0.03; 0.05; mean 0.04 [a], [SS], [LOQ=0.05]	M4008B; trial ns; [Harradine, 1985, PP5/0397] M4209B; trial ns; [Harradine, 1986, PP5/0398]
idem	EC 125 (P)	1	0.38	0.17	no buds; 6 leaves; 100% crop cover; 4 June	L	CH green	46	< 0.03, 0.04 mean 0.035 [[a], [SS], [LOQ=0.05]	idem
Canwick, Lincolnshire; UK; 1984; (vining pea: Scout)	EC 125 (P)	1	0.19	0.085	no buds; 5-6 leaves; 100% crop cover; 4 June	L	CH green	39	< 0.03 [SS] [LOQ=0.05]	M4008B; trial ns; [Harradine, 1985, PP5/0397] M4209B; trial ns; [Harradine, 1986, PP5/0398]
idem	EC 125 (P)	1	0.38	0.17	no buds; 5-6 leaves; 100% crop cover; 4 June	L	CH green	39	0.41 [SS]	idem
Coldham; UK; 1984; (vining pea: Tristar)	EC 125 (P)	1	0.38	0.17	3 leaves; 100% crop cover; 4 May	pL	CH green	76	< 0.03 [SS] [LOQ=0.05]	M4008B; trial ns; [Harradine, 1985, PP5/0397]
Crowland; UK; 1984; (vining pea: Sprite)	EC 125 (P)	1	0.38	0.17	(ns); 100% crop cover; 8 May	L	CH green	64	< 0.03 [SS] [LOQ=0.05]	M4008B; trial ns; [Harradine, 1985, PP5/0397]
Epworth; Lincolnshire; UK, 2001 (Waierex)	EC 125 (P)	1	0.38	0.13	BBCH 16/36; 22 June	SaL	CH green	39	0.19	RJ3336B; AF/6067/SY1 [Mason, 2002, PP5/1260]
Holbeach Hurn; UK, 2006; (Geisha)	EC 125 (P)	1	0.38	0.075	BBCH 16-17; 10 Jul	SaSiL	79	35	0.80	CEMR-3009; AF/10375/SY1 [Bell, 2008, PP5/1552]
Thwing; UK, 2006; (Ibis)	EC 125 (P)	1	0.38	0.075	BBCH 15-21; 5 Jul	SaL	77	35	0.48	CEMR-3009; AF/10375/SY2 [Bell, 2008,

GREEN PEA SEEDS Location, Country; year; (variety)	For m (g ai/L)	No (inter val in days)	kg ai/h a	kg ai/h L	GS and last treatmen t day	Soil type	GSH	DAL T (days)	Total fluazifop mg/kg	Report; Trial no [ref]
										PP5/1552]
Luddington; Warwickshir e; CV37 9SJ UK; 2010; (Samish)	EC 125 (P)	1	0.37	0.08 3	BBCH 38-39; 3 June	SiL	79	34	0.47	CEMR-4658-REG; CEMS-4658-02; [Jutsum and Allen, 2011, A1279B_10837]
Stratford upon Avon, Warwickshir e, United Kingdom; 2010; (Samish) Plot 2	EC 125 (P)	1	0.74	0.17	BBCH 38-39; 03 June, 2010	SiL	78-79	32	Not reported	CEMR-4751-REG; CEMS-4751-02; [Langridge, 2013, A12791B_11029]
Idem	EC 125 (P)	1	1.8	0.42	BBCH 38-39; 03 June, 2010	SiL	78-79	32	2.3 [b]	idem (processing)
Location ns; Netherlands, 1980 (peas, var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	53	< 0.02, < 0.02, < 0.02, < 0.02 [QU] [LOQ=0.0 5]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Netherlands, 1980 (peas, var ns)	EC 250 (rac)	1	1.2	ns	GS ns date ns	ns	ns	53	0.03, 0.06, 0.06, 0.10 [QU] [LOQ=0.0 5]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Beerta; Netherlands; 1984; (canning pea: Joff)	EC 125 (P) + Agra l	1	0.38	0.07 5	pre-flower; 6-8 leaves; 60% crop cover; 1 June	C	16 d PCH green	63	< 0.03; < 0.03; < 0.03; < 0.03; mean < 0.03 [a] [SS] [LOQ=0.0 5]	M3976B; 84-132; [Dick and Rounds, 1985, PP5/0412]
Zuidlaren; Netherlands; 1985; (pea, Finale)	EC 125 (P)	1	0.19	0.03 8	buds formed; 100% crop cover; 1 June	Sa	14 d PCH (greenseed s)	56	0.24, 0.39, 0.41, 0.41; mean 0.36 [a]	M4261B; 85-210; [Crook and Harradine, 1986, PP5/0161]
idem	EC 125 (P)	1	0.38	0.07 5	buds formed; 100% soil cover; 1 June	Sa	14 d PCH (greenseed s)	56	0.41, 0.48, 0.55, 0.68 mean 0.53 [a]	idem
Kielwinde weer;	EC 125	1	0.19	0.03 8	buds formed;	Sa	14d PCH (greenseed	56	0.08; 0.08;	M4261B; 85-211 ;

GREEN PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Netherlands; 1985; (pea, Finale)	(P)				100% soil cover; 31 May		s)		0.10, 0.12 mean 0.11 [a]	[Crook and Harradine, 1986, PP5/0161]
idem	EC 125 (P)	1	0.38	0.075	bud formed; 100% soil cover; 31 May	Sa	14 d PCH (greenseeds)	56	0.13, 0.14, 0.17, 0.20; mean 0.16 [a]	idem
Assen; Netherlands; 1985; (pea, Minarette)	EC 125 (P)	1	0.38	0.075	no buds; 70% soil cover; 5 June	Sa	green seeds	56	< 0.05; < 0.05; < 0.05; < 0.05; mean < 0.05 [a] [SS]	M4261B; 85-215 ; [Crook and Harradine, 1986, PP5/0161]
28140 Loigny la Bataille; N-France; 2006; (Piano)	EC 125 (P)	1	0.38	0.075	BBCH 51; 12 May	CL	79	35	0.26	CEMR-3009; AF/10375/SY3 [Bell, 2008, PP5/1552]
71530 Viney le Grand; N-France; 2006; (Atlanta)	EC 125 (P)	1	0.39	0.075	BBCH 59; 1 Jun	SaL	79	35	0.27	CEMR-3009; AF/10375/SY4 [Bell, 2008, PP5/1552]
32490 Monferran Saves Gers; S-France; 2012; (Numerica)	EC 125 (P)	1	0.34	0.10	BBCH 37; 27 June	CL	79	35	0.03	CEMR-5453; SRFR12-008-37HR [Langridge, 2013, A12791B_11035]
San Bonifacio; Verona; Italy, 1997, (Abador)	EC 125 (P)	1	0.38	0.12	BBCH 38; 29 Apr	L	CH green	35	0.06	RJ2254B; IT10-96-R344 [Jones *et al*., 1997, PP5/0162]
Filetto; Ravenna, Italy, 1997 (Linx)	EC 125 (P)	1	0.38	0.12	BBCH 38; 21 May	L	CH green	29	0.08	RJ2254B; IT10-96-R345 [Jones *et al*, 1997, PP5/0162]
31360 Funes; Spain; 2003 (Remu)	EC 125 (P)	1	0.31	0.10	BBCH 50; 25 Apr	SaCL	79	35	0.36	03-7031; AF/7290/SY1 [Mason, 2004; PP5/1412]
50100; La Almunia de Dona Godina; Spain; 2003; (NZ)	EC 125 (P)	1	0.31	0.10	BBCH 50; 29 Apr	LSa	79	34	1.0	03-7032; AF/7291/SY1 [Mason, 2004; PP5/1413]
22280; Gurrea de	EC 125	1	0.32	0.062	BBCH 57;	CL	77	35	0.22	CEMR-3012; AF/10376/SY1

GREEN PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Gallego; Spain, 2006; (Valverde)	(P)				5 May					[Bell, 2007, PP5/1550]
22196; Huesca; Spain, 2006; (Meteor)	EC 125 (P)	1	0.32	0.062	BBCH 57; 5 May	CL	77	35	0.77	CEMR-3012; AF/10376/SY2 [Bell, 2007, PP5/1550]
Banded foliar spray over rows										
03400 Villena; Spain; 2012; (Aston)	EC 125 (P)	1	0.31	0.10	BBCH 36-37; 15 May	C	77	35	0.20	CEMR-5453; SRFR12-211-37HR; [Langridge, 2013, A12791B_11035]
02007 Albacete; Spain; 2012 (Resal)	EC 125 (P)	1	0.32	0.10	BBCH 36-37	LSa	77	34	0.37	CEMR-5453; SRES12-212-37HR; [Langridge, 2013, A12791B_11035]

Soil type: pL = peaty Loam;

GSH: 16dPCH = 16 days before commercial harvest;

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

[a] Results came from replicate field samples taken from the same plot; the mean is used for MRL deriviation if according to cGAP.

[b] Results are the mean of 3-8 replicate analytical samples.

Additional trial information

M3754B. Non-GLP. Weather conditions, spray equipment, spray volumes not stated. Sample sizes pea seeds (> 1 kg) , pods (> 0.5 kg). Storage at -18 °C or lower for a maximum of 977days (1981 trials), 612 days (1982 trials), 247 days (1983 trials) (harvet to report date). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with internal standard with a valid LOQ of 0.05 mg/kg**. Samples were corrected for internal std recovery (58-86% at 1.0 mg/kg). Control samples were < 0.05 mg/kg .

M4234B Non-GLP. Fodder peas. Considering application timing in combination with harvest times, these peas are considered fresh peas [Syngenta, 2016, response to questions 15]. No unusual weather conditions. Spray using a knapsack sprayer. Spray volume 400 L/ha.. Samples of 1 kg seeds were sampled by hand. Samples were stored at -20°C or lower. Storage period was not stated but was at maximum of 320 days (Harvest to final report date). Samples were analysed for total fluazifop using **NMR method PPRAM83 with a valid LOQ of 0.05 mg/kg**. Average internal standard recovery was 77% at 0.25 mg/kg. Control samples < 0.02 mg/kg

CEMR-4751-REG. GLP. No unusual weather conditions. Broadcast foliar spray by hand-held boom sprayer. Spray volume 406-447 L/ha. Samples were harvested by hand using a suitable distributive pattern. Sample sizes pea seeds (17.5 -34.6 kg). Samples were stored chilled for 2-4 days before processing. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (75-107% at 0.01–0.5 mg/kg). Control samples were < 0.01 mg/kg (trial 02-UK) or 0.21–0.29 mg/kg (trial 03-Germany).

RJ0226B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries not indicated. Control samples were < 0.01 mg/kg (UK, 1980).

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. Storage conditions not stated. Samples were analysed for total

fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.02 mg/kg.

M4008B andM4209B Non-GLP. Vining peas. No unusual weather conditions. Application by plot sprayer; spray volume 220 L/ha. Samples were hand cut and pea seeds were harvested using a plot viner (11-20 July). Sample sizes were not stated. Samples were stored at -20 °C but no longer than 328 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recoveries (78%). Control samples < 0.03 mg/kg.

RJ3336B. GLP. Vining Peas. No unusual weather conditions. Overall foliar spray using a precision boom sprayer. Spray volume 297L/ha. Whole plants (24 plants) were sampled by hand and taken systematically from across the plots (31 July). Samples were threshed using a static viner to produce samples of seed (> 1 kg). Storage at -18 °C for 170-174 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recovery (104-107% at 0.1-1.0 mg/kg). Control samples < 0.01 mg/kg.

CEMR-3009. GLP Fresh Peas. No unusual weather conditions. Foliar spray using a plot sprayer. Spray volume 502-515 L/ha. Fresh pea plants were sampled by hand (9-14 Aug in the UK; 16 June, 6 July in France) and threshed using a mini pea viner (UK) or by hand (N-France). resulting in seeds 1.0-1.1 kg fresh pea seeds. Storage at -15 °C for a maximum of 495 days. The temperature reached a maximum of -9 ºC for a peak of 3 days. This is considered to have no effect on the study, since the samples remained frozen at all times. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg.** Samples were not corrected for individual concurrent method recovery (103% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

CEMR-4658. GLP Fresh peas. No unusual weather conditions. Broadcast foliar spray using a backpack sprayer. Spray volume 379-448 L/ha. Fresh pea plants (12 units) were taken by hand (7 July UK) using a suitable distributive pattern. Sample sizes > 1 kg fresh pea seeds. Storage at -18 °C for a maximum of 317 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (70-92% at 0.01–0.1mg/kg). Control samples < 0.01 mg/kg.

M3976B. Non-GLP. Vining peas. No unusual weather conditions. Broadcast foliar spray using a knapsack propane gas sprayer. Spray volume 500L/ha. Pods were sampled by hand (3 August). Seeds and pods were separated. Sample sizes were not stated. Storage at -20°C. Storage time not stated but no longer than 314 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (64% at 0.5 mg/kg). Control samples < 0.03 mg/kg.

M4261B Non-GLP. This trial reports fresh peas [Syngenta, 2016, Response to questions 15]. No unusual weather conditions. Application by propane sprayer; spray volume 500 L/ha. Four replicate samples per plot were harvested by hand. Peas from trial 85-216 were too small to separate seeds from pods and were assumed to be green peas with pods (addressed in the section on green peas with pods). Samples from trials 85-210 and 85-211 were harvested 4 and 2 weeks before commercial harvest (DAT = 42 and 56). Sample sizes were not stated. Samples were stored at -20 °C (storage time not stated but less than 12 months). Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recoveries 95%. Control samples < 0.05 mg/kg.

CEMR-5453. GLP Fresh peas. No unusual weather conditions. Broadcast foliar spray (S-France) or **banded foliar spray (Spain)** using a backpack sprayer. Spray volume 297-331 L/ha. Fresh pea plants (12 units) were taken by hand (1 Aug S-France, 18-19 June Spain) using a suitable distributive pattern. Sample sizes seeds (> 1 kg). Storage at -18 °C for a maximum of 190days. Freezer reached peak temperatures of -9, -10 ºC for 2 hours. This is considered to have no impact on the results, since samples remained frozen at all times. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (95-113% at 0.01–0.5 mg/kg. Control samples < 0.01 mg/kg.

RJ2254B Fresh Peas. GLP. No unusual weather conditions. Foliar spray using a motor knapsack sprayer with a boom. Spray volume 300 L/ha. Fresh pea pods (2.1-3.3 kg) were sampled by hand and taken systematically from across the plots. Storage at -18 °C for a 118-135 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recovery (80-82% at 0.1–0.5 mg/kg Control samples < 0.01 mg/kg.

03-7031. GLP. Fresh vining peas. No unusual weather conditions. Overal spray using a plot sprayer. Spray volume 295 L/ha. Fresh pea plants (24 units) were sampled by hand and seed fractions were separated: >1 kg seeds. Storage at -18 °C for a maximum of 283 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (109-112% at 0.1–0.5 mg/kg. Control samples < 0.01 mg/kg.

03-7032. GLP. Fresh vining peas. No unusual weather conditions. Overal spray using a plot sprayer. Spray volume 299 L/ha. Fresh pea plants (24 units) were sampled by hand and seed fractions were separated off: >1 kg seeds. Storage at -18 °C for a maximum of 255 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (106-109% at 0.05-0.1 mg/kg for seeds. Control samples < 0.01 mg/kg.

CEMR-3012. GLP. Fresh Peas. No unusual weather conditions. Foliar spray using a plot sprayer. Spray volume 508-509 L/ha. Fresh pea seeds (0.60-1.2 kg) were sampled by hand. Storage at -15 °C for a a maximum of 436 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (95-107% at 0.01–0.1 mg/kg. Control samples < 0.01 mg/kg.

Green soya bean seeds (Glycine spp)

A GAP for green soya bean seeds is not available.

Trials from Brazil (2005) were available on green soya bean seeds with an application of 1 × 0.25-0.50 kg ai/ha and harvest at 60 DAT [Baptista and Bahia, 2006, A13680A_10002, report M04064]. As the manufacturer did not seek to have maximum residue levels on green soya bean seeds estimated the available studies on green soya bean seeds were not summarized.

Pulses

Beans, dry (Phaseolus spp)

Three cGAPs for dry Phaseolus beans are available:

- cGAP from the USA with 2 × 0.42 kg ai/ha and PHI 60 days (as nn)
- cGAP from the Netherlands with 1 × 0.38 kg ai/ha and PHI 90 days
- cGAP from Brazil with 1 × 0.25 kg ai/ha and PHI 60 days

Trials that could be matched to these cGAPs were summarized.

Table 217 lists trials conducted in the USA (1984, 1986, 1987, 2000), Canada (1985, 2007, 2008), Brazil (1989, 1990, 1991, 2011) and Spain (1987, 1997, 1998, 1999). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 217. Results marked with "[RT]", "[SS]", "[SK]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[RT] indicates that samples were rotten.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[SK] no soaking step was used for dry pulses before extraction (or it was unclear whether soaking was used (SK*), whereby fluazifop (II) conjugates are not quantitatively extracted.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62/2.

In the trials conducted in the USA (RR 00-061B) there was a clear relationship between the timing of application with respect to flowering and the amount of residue detected. Pre-flowering applications resulted in the lowest residues (0.5–0.7 mg/kg), mid-flowering applications resulted in moderate residues (1.1–3.9 mg/kg) and late flowering applications resulted in the highest residues (5.2–21 mg/kg). The two sets of decline samples indicated a residue half-life of 15–20 days.

Additional trials from Canada (1980, 1981, 1990) on white or dry beans were available with 1 × 0.12–0.20–0.25–0.30–0.50–1.0–2.0 kg ai/ha with harvest at 70, 76, 77, 82 or 84 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B; Atreya and Harradine, 1982, PP9/0062, report RJ0291B; Jones, 1991, PP5/0386, report M5386B]. These trials were not summarized, because they would not assist in MRL setting.

Table 217 Supervised field trials on Phaseolus beans (dry seeds), treated with a broadcast foliar fluazifop-butyl spray

DRY BEANS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Casselton ND, USA, 1984 (navy bean; Fleetwood)	EC 120 (P) + COC 1%	2 (10)	0.42	0.53	just before bloom; 5 May	ns	ns	62	1.5 [SS][SK*]	TMU3094/B; 64ND84-088 [Watford and Francis, 1986, PP5/0378] *
Kingsville TX, USA, 1984 (bean; Purple Hull)	EC 120 (P) + NIS 0.25%	2 (7)	0.42	0.37	just before bloom; 14 Sept	ns	ns	68	< 0.03 [SS][SK*] [LOQ=0.05]	TMU3094/B; 71TX84-057 [Watford and Francis, 1986, PP5/0378] *
Scottsbluff NE, USA, 1984; (bean, Spinel)	EC 120 (P) + COC 1.0%	2 (7)	0.42	0.23	just before bloom; 17 Jul	ns	MAT	77	0.30 [SS][SK*]	TMU3094/B; 52NB84-069 [Watford and Francis, 1986, PP5/0378] *
Euclid, MN, USA, 1984; (bean, Sanilac)	EC 120 (P) + COC 1.0%	2 (15)	0.42	0.28	just before bloom; 13 Jul	ns	ns	83	< 0.03 [SS][SK*] [LOQ=0.05]	TMU3094/B; 64MN84-089 [Watford and Francis, 1986, PP5/0378] *
Kimberly, ID USA, 1986 (bean; Small Red)	EC 120 (P) + NIS 0.25%	2 (18)	0.42	0.14	just before bloom: buds forming; 1 July	ns	MAT	70 (sw 3)	0.32	RR 89-046B; 32ID86-907 [McKay, 1989, 405660]
Haslett, MI USA, 1986 (bean; Seafarer)	EC 120 (P) + COC 1%	2 (16)	0.42	0.12	just before bloom: bud stage; 4 July	ns	MAT	92	< 0.03 [RT] [LOQ=0.05]	RR 89-046B; 71MI86-902R [McKay, 1989, 405660]
Ft Collins, CO, USA, 1987 (bean; Idaho III)	EC 120 (P) + COC 1%	2 (17)	0.42	0.18	just before bloom; 13 July	ns	MAT	75	0.47;0.48[c] mean 0.48 [SS] [b]	RR 89-046B; 92CO87-446 [McKay, 1989, 405660]
Trimble, MO USA, 1987 (pinto bean; Fiesta)	EC 120 (P) + COC 1%	2 (22)	0.42	ns	just before bloom: budding; 17 July	ns	MAT	98	< 0.05(2); mean < 0.05 [b] [SS] [RT] [LOQ=0.05]	RR 89-046B; 48MO87-444 [McKay, 1989, 405660]
Manteca, CA USA, 1987; (kidney bean; Light Red)	EC 120 (P) + COC 1%	2 (14)	0.42	0.24	just before bloom: 8-10 trifoliates; 17 July	ns	MAT	75	0.87;0.78[c] mean 0.82 [b]	RR 89-046B; 45CA87-443 [McKay, 1989, 405660]
North Rose, NY, USA, 2000; (kidney bean, California Light Red)	EC 240 (P) + NIS 0.5%	2 (14)	0.42	ns	early bloom; 10 Jul	Sa	NH	60 (sw 0)	1.1; 1.3 mean 1.2 [b]	RR 00-061B; 167-DBNY1; [Stewart, 2001, PP5/1069]
East Grand Forks, MN, USA, 2000; (navy bean,	EC 240 (P) + NIS 0.5%	2 (14)	0.42	ns	mid-flower; BBCH 65; 17 Jul	SiCL	NH	59 (sw 2)	19; 21 mean 20 [b]	RR 00-061B; 168-DBMN1; [Stewart, 2001, PP5/1069]

DRY BEANS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Snow Bunting)										
Geneva, MN, USA, 2000; (navy bean, Great Northern)	EC 240 (P) + COC 1%	2 (13)	0.42	ns	flowering; 3 Aug	CL	NH	60 (sw 0)	4.7; 5.2 mean 5.0 [b]	RR 00-061B; 169-DBMN2; [Stewart, 2001, PP5/1069]
Conklin, MI, USA, 2000; (navy bean, Avanti)	EC 240 (P) + NIS 0.5%	2 (14)	0.42	ns	early bloom with pods up to 2.5 cm; 25 Jul	L	NH	60 (sw 0)	3.3; 3.9 mean 3.6 [b]	RR 00-061B; 170-DBMI1; [Stewart, 2001, PP5/1069]
Larimore, ND, USA, 2000; (pinto bean, Remington)	EC 240 (P) + COC 1%	2 (14)	0.42	ns	initial bloom; 11 Jul	SaL	NH	59 (sw 3)	1.1; 1.1 mean 1.1 [b]	RR 00-061B; 171-DBND1; [Stewart, 2001, PP5/1069]
Velva, ND, USA, 2000; (pinto bean; Maverick NCR317)	EC 240 (P) + NIS 0.5%	2 (12)	0.42	ns	first bloom; 10 Jul	L	NH	60 (sw 0)	3.1; 3.6 mean 3.4 [b]	RR 00-061B; 174-DBND2; [Stewart, 2001, PP5/1069]
Delavan, WI, USA, 2000; (pinto bean; Field Bean)	EC 240 (P) + NIS 0.5%	2 (14)	0.42	ns	blooming, with pods up to 13 cm; 4 Aug	SiL	NH	60 (sw 0)	16; 16 mean 16 [b]	RR 00-061B; 172-DBWI1; [Stewart, 2001, PP5/1069]
Grand Island, NE, USA, 2000; (navy bean; Great Northern)	EC 240 (P) + NIS 0.5%	2 (14)	0.42	ns	blooming and pods forming; 11 Jul	SiL	NH	59 (sw 3)	9.4; 9.5 mean 9.4 [b]	RR 00-061B; 173-DBNE1; [Stewart, 2001, PP5/1069]
Levelland, TX, USA, 2000; (pinto bean; Taylor Horticulture Improved)	EC 240 (P) + COC 1%	2 (14)	0.42	ns	flowering, setting pods; 7 Aug	SaL	NH	60 (sw 4)	2.8; 3.9 mean 3.4 [SS] [b]	RR 00-061B; 175-DBTX1; [Stewart, 2001, PP5/1069]
Edgar, MT, USA, 2000; (pinto bean; Orthello)	EC 240 (P) + NIS 0.5%	2 (12)	0.42	ns	3rd trifoliate; 1 Jul	L	- - NH - -	50 (sw 4) 55 (sw 3) 60 (sw 2) 65 (sw 3) 68 (sw 1)	0.58; 0.86 mean 0.72 0.32; 0.33 mean 0.32 0.42; 0.50 mean 0.46 0.19; 0.28 mean 0.24 0.27; 0.32 mean 0.30 [b]	RR 00-061B; 176-DBMT1; [Stewart, 2001, PP5/1069]
Meridian, ID, USA, 2000; (pinto bean; Orthello)	EC 240 (P) + NIS 0.5%	2 (14)	0.42	ns	7-14 trifoliate; 5 Jul	SiL	- - NH -	50 (sw 5) 55 (sw 5) 60 (sw 5) 65	1.0; 1.3; mean 1.2 0.67; 1.3 mean 0.98 0.59; 0.70 mean 0.64 0.74; 0.79;	RR 00-061B; 178-DBID1; [Stewart, 2001, PP5/1069]

DRY BEANS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
							-	(sw 5) 68 (sw 5)	mean 0.76 0.52; 0.57; mean 0.54 [b]	
50 Road; Canada, 1985; kidney beans: Dark Red	EC 125 (P)	1	0.25	0.11	3 trifoliate; 25% soil cover; 8 July	SaL	MAT dry	65	0.09, 0.11, 0.25, 4.0, mean 1.1 [b] [SS] [CT] [SK*] [Cntrl=0.06]	M4130B; CA/ON/R/85/300/C [Harradine, 1986, PP5/0376]
Burlington; Canada, 1985; (dry green beans: Improved Tender Green)	EC 125 (P)	1	0.25	0.22	3 trifoliate; 25% soil cover; 4 July	LC	MAT dry	70	3.6, 3.7, 4.0, 4.0, mean 3.8 [b] [SS] [SK*] [cntrl=0.26]	M4130B; CA/ON/R/85/301/C [Harradine, 1986, PP5/0376]
idem	EC 125 (P)	1	0.50	0.22	3 trifoliate; 25% soil cover 4 July	LC	MAT dry	70	5.3, 5.3, 6.0, mean 5.5 [b] [SS] [SK] [cntrl=0.26]	idem
Paris, ON Canada, 2007; (cranberry bean; Etna)	EC 125 (P) Venture	1	0.23	0.15	1st trifoliate leaf stage; BBCH 11; 12-15 cm high; 28 June	L	MAT dry	74	0.75, 0.91, mean 0.83 [b]	CER 02607/07; T235 [Sagan, 2008, A12791B_50001]
idem	EC 125 (P) Venture	1	0.23	0.16	1st trifoliate leaf stage; BBCH 11; 28 June	L	MAT dry	74	1.1, 1.1, mean 1.1 [b]	idem
idem	EC 125 (P) Venture	1	0.081	0.054	post-bloom; BBH 68-69; 60-65 cm high; 2 Aug	L	MAT dry	39	4.0, 4.4, mean 4.2 [b]	idem
Elm Creek, MB, Canada, 2007; (navy bean: Envoy)	EC 125 (P) Vent L	1	0.25	0.17	1st trifoliate leaf stage; BBCH 13; 14 June	LSa	89	75	0.016, 0.024, mean 0.020 [b]	CER 02607/07; T236 [Sagan, 2008, A12791B_50001]
idem	EC 125 (P) Fus MAX X	1	0.25	0.16	1st trifoliate leaf stage; BBCH 13; 14 June	LSa	89	75	0.025, 0.028, mean 0.026 [b]	idem
idem	EC 125 (P) Vent L	1	0.080	0.053	post-bloom; BBH 67-	LSa	89	28	1.6, 2.2, mean 1.9 [b]	idem

DRY BEANS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					69; 30-40 cm high; 31 Jul					
Taber, AB, Canada, 2007; (black bean: AC Black Diamond)	EC 125 (P) Vent L	1	0.25	0.17	1st trifoliate leaf stage; BBCH 13; 5-10 cm high 14 June	SaL	99	105	< 0.01, < 0.01, mean < 0.01 [b]	CER 02607/07; T237 [Sagan, 2008, A12791B_50001]
idem	EC 125 (P) Fus MAX X	1	0.25	0.17	1st trifoliate leaf stage; BBCH 13; 5-10 cm high 14 June	SaL	99	105	0.015, 0.020, mean 0.018 [b]	idem
idem	EC 125 (P) Vent L	1	0.076	0.050	post-bloom; BBH 67-69; 50-60 cm high; 2 Aug	SaL	99	56	3.0, 3.8, mean 3.4 [b]	idem
Branchton, ON, Canada, 2008; (adzuki bean)	EC 125 (P)	1	0.26	0.17	1st trifoliate leaf stage; BBCH 12; 6-10 cm high; 9 July	SiL	89	92	< 0.01, < 0.01, mean < 0.01 [b]	CER 02609/08; T453 [Sagan, 2009, A12791N_50001]
idem	EC 125 (P)	1	0.080	0.053	post bloom; BBCH 67; 40-45 cm high; 22 Aug	SiL	89	48	0.24, 0.27, mean 0.26 [b]	idem
Branchton, ON, Canada, 2008; (cranberry bean: common #1)	EC 125 (P)	1	0.25	0.17	1st trifoliate leaf stage; BBCH 12; 10-15 cm high; 9 July	SiL	86-88 89 89 89	70 79 84 92	0.059; 0.039, 0.036; mean 0.038; [b] 0.043; 0.059	CER 02609/08; T454 [Sagan, 2009, A12791N_50001]
idem	EC 125 (P)	1	0.075	0.050	post bloom; BBCH 67-69; 40-45 cm high 5 Aug	SiL	86-88 89 . . 89 89	43 52 . . 57 65	0.20; 0.17, 0.20, mean 0.18; [b] 0.16; 0.16	idem
S Sebastião da Amoreira, PR, Brazil, 1989; (Carioca)	EC 125 (P) + 0.2% adj	1	0.19	0.078	30 days post-emergence; 22 Nov	ns	NH (dry)	60 (ad 0)	< 0.01 [SK*]	TECPAR 81981/92; BR10-90-S005H; [Bill and Kamienski, 1992, PP5/0390]
idem	EC 125 (P) +	1	0.38	0.16	30 days post-	ns	NH (dry)	60 (ad 0)	< 0.01	idem

DRY BEANS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	0.2% adj				emergence; 22 Nov					
S Sebastião da Amoreira, PR, Brazil, 1990; (Carioca)	EC 125 (P)	1	0.19	0.095	30 days post-emergence; 15 cm tall; 27 May	ns	NH (dry)	62 (ad 0)	< 0.01 [SK*]	TECPAR 81980/92; BR10-90-S006H; [Bill and Kamienski, 1992, PP5/0389]
idem	EC 125 (P)	1	0.38	0.19	30 days post-emergence; 14 cm tall; 26 June	ns	NH (dry)	62 (ad 0)	< 0.01	idem
Urai, PR, Brazil, 1991; (Carioquinha)	SL 125 (P)	1	0.25	0.083	47 days post-emergence; 29 May	SaCL	NH (dry)	60 (ad 1)	< 0.01 [SK]	TECPAR 81975/92; BR10-91-S007H; [Bill and Kamienski, 1992, PP5/1028]
idem	SL 125 (P)	1	0.50	0.17	47 days post-emergence; 29 May	SaCL	NH (dry)	60 (ad 1)	< 0.01	idem
idem	SL 200 (P)	1	0.20	0.067	47 days post-emergence; 29 May	SaCL	NH (dry)	60 (ad 1)	< 0.01	idem
idem	SL 200 (P)	1	0.40	0.13	47 days post-emergence; 29 May	SaCL	NH (dry)	60 (ad 1)	< 0.01	idem
idem	SL 125 (P)	1	0.25	0.083	32 days post-emergence; 13 June	SaCL	NH (dry)	45 (ad 1)	< 0.01	idem
idem	SL 125 (P)	1	0.50	0.17	32 days post-emergence; 13 June	SaCL	NH (dry)	45 (ad 1)	< 0.01	idem
idem	SL 200 (P)	1	0.20	0.067	32 days post-emergence; 13 June	SaCL	NH (dry)	45 (ad 1)	< 0.01	idem
idem	SL 200 (P)	1	0.40	0.13	32 days post-emergence; 13 June	SaCL	NH (dry)	45 (ad 1)	< 0.01	idem
Guaira, SP, Brazil, 1991; (IAC Carioca)	SL 125 (P)	1	0.25	0.083	vegetative; 26 June	C	NH (dry)	60 (ad 11)	< 0.01 [SK]	TECPAR 83030/92; BR14-91-S008H; [Bill and

DRY BEANS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
										Kamienski, 1992, PP5/1029]
idem	SL 125 (P)	1	0.50	0.17	vegetative; 26 June	C	NH (dry)	60 (ad 11)	< 0.01	idem
idem	ME 200 (P)	1	0.20	0.067	vegetative; 26 June	C	NH (dry)	60 (ad 11)	< 0.01	idem
idem	ME 200 (P)	1	0.40	0.13	vegetative; 26 June	C	NH (dry)	60 (ad 11)	< 0.01	idem
idem	SL 125 (P)	1	0.25	0.083	flowering; 12 July	C	NH (dry)	45 (ad 11)	< 0.01	idem
idem	SL 125 (P)	1	0.50	0.17	flowering; 12 July	C	NH (dry)	45 (ad 11)	< 0.01	idem
idem	ME 200 (P)	1	0.20	0.067	flowering; 12 July	C	NH (dry)	45 (ad 11)	< 0.01	idem
idem	ME 200 (P)	1	0.40	0.13	flowering; 12 July	C	NH (dry)	45 (ad 11)	< 0.01	idem
Engenheiro Coelho, SP, Brazil, 2011; (Perola)	EW 250 (P)	1	0.25	0.25	BBCH 60-61; 20 Jan	C	BBCH 87-89	60 (ad 0)	< 0.01	M11034; AMA; [Draetta, 2012, A12530B_10018]
Palmeira, PR, Brazil, 2011; (Tuiuiu)	EW 250 (P)	1	0.25	0.25	BBCH 47-49; 21 Febr	C	BBCH 87-89	60 (ad 0)	1.0	M11034; DMO; [Draetta, 2012, A12530B_10018]
Uberlandia, MG, Brazil, 2011; (Alvorada)	EW 250 (P)	1	0.25	0.25	BBCH 61; 22 Mar	C	BBCH 89	60 (ad 0)	< 0.01	M11034; JJB; [Draetta, 2012, A12530B_10018]
Lavras, MG, Brazil, 2011; (Carioquinha)	EW 250 (P)	1	0.25	0.25	BBCH 59, 3 Mar	Medio	BBCH 89	60 (ad 0)	0.46	M11034; RWC; [Draetta, 2012, A12530B_10018]
Almussafes; Valencia; Spain,1987 (ns)	EC 300 (P)	1	0.30	ns	20% crop cover; 2 June	ns	ns	99	0.22 [SS]	M4799B ES01-87-D005-H; [Crook, 1988, PP5/0380]
Talavera de la Reina;Toledo; Spain; 1997 (Alpine)	EC 125 (P)	1	0.32 or 0.34	0.10 or 0.10	Before BBCH 50; rows; 30-40 cm height; 27 May	LC	MAT (dry)	66	0.89, 1.6 [a]	RJ2610B; ES10-97-SH010 and SH110; [Mason and Gallardo, 1998, PP5/0153
Talavera de la Reina;Toledo; Spain; 1998 (Alpiner)	EC 125 (P)	1	0.31	0.13	BBCH 50; rows; 25 cm height; 19 May	LC	MAT (dry)	80	0.09 [CT] [Cntrl=0.03]	RJ2826B; ES10-98-SH001 [Ryan and Gallardo, 1999; PP5/0160]
Caballar; Segovia; Spain;1998 (La Granja)	EC 125 (P)	1	0.34	0.13	BBCH 50; rows 20 cm height; 11 June	ns	MAT (dry)	111	< 0.01	RJ2826B; ES10-98-SH001 [Ryan and Gallardo, 1999;

DRY BEANS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
										PP5/0160]
Granollers; Barcelona; Spain; 1999 (Ganchet de caño)	EC 125 (P)	1	0.35	0.079	before BBCH 50; staked; 70 cm height; 3 Aug	L	89-97	84	< 0.01	RJ2994B; ES60-99-S023 [Mason *et al*, 2000, PP5/0373]

Sw = number of swath days or cure days after harvest

* These studies were listed again in [McKay, 1989, 405660, report RR 89-046B]

[SS] Sample size was below 1 kg (0.6-1.3 kg in trial 175-DBTX1, 0.9 kg in 1984 US trials, 0.9 kg in trial 92CO87-446 and 48MO87-444, 0.5 kg in 1985 Canadian trials) or sample size was not stated in the report (M4799B]; samples are considered not representative for MRL setting

[RT] Due to heavy rainfall at the time of harvest in trial 71MI86-902R and 48 MO87-444, rotting may have affected residue levels. In trial 71MI86-92B bean plants were dried off in a greenhouse for 3 days to avoid rotting. In trial 48MO87-444, beans did not senesce properly and many of the beans appeared rotted. Samples are considered not representative for MRL setting.

[SK] The soaking step was omitted in the analytical method, whereby fluazifop (II) acid conjugates are incompletely extracted.

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

[a] Results represent samples from two replicate plots; the highest residue is taken for MRL derivation if according to cGAP

[b] Results represent replicate samples from a single plot; the average residue is taken for MRL derivation if according to cGAP

[c] Result represents the average of 2 replicate analyses of the same sample

Additional trial information

TMU3094B: **non-GLP study**. No unusual weather conditions. Information on spray equipment is not available. Spray volume 8.5-20 GPA = 80-190 L/ha. Dry bean seeds (2 lbs = 0.9 kg/sample). Information on harvesting practices is not available. Storage at -20°C for 18 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM62/2 with a valid LOQ of 0.05 mg/kg**. Average concurrent recovery (69-77% at 0.05-0.2 mg/kg). Control samples were < 0.03 mg/kg (n = 4).

RR89-046B: non-GLP study. Weather conditions had no impact on the crop growth and development, except where indicated. Broadcast foliar application by backpack or tractor mounted sprayer. Spray volume 18-30 GPA = 170-280 L/ha. Dry bean seeds (>2.5 lbs = 1.1 kg/sample) were taken, except where indicated. Information on harvesting practices is not available, except in trial 45CA87-443, where dry bean seeds were hand-harvested and then shelled by hand. Where information is available, swath days after harvest is indicated. Storage at -10°C or lower for up to 395 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM62/2 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent recovery (73-85% at 0.05-3 mg/kg). Control samples were < 0.05 mg/kg.

RR 00-061B: GLP study. Weather conditions had no impact on the crop growth and development, except in trial 175, where the high temperature and low rainfall resulted in a lower seed yield. Broadcast over the crop canopy application by backpack or tractor mounted sprayer. Spray volume 8-30 GPA = 75-280 L/ha. Dry bean seeds (>2.5 lbs = >1 kg/sample) were taken from 12 random areas from across the plots, except where indicated. Dry beans were swathed 0-5 days before threshing for desiccation. Dry beans were hand or machine treshed. Storage at -6°C for 40-83 days. Samples were analysed for total fluazifop using **GC-MS method RR91-014B modification A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery (76-129% at 0.01-25 mg/kg). Control samples were < 0.01 mg/kg.

M4130B: non-GLP study. No unusual weather conditions. CO2 hand held sprayer. Spray volume 225 L/ha. Mature dry bean seeds (0.5 kg/sample) were sampled by hand. Storage at -20°C for a maximum of 80days (harvest to final report). Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Samples were corrected for internal std recovery (74% at 0.5 mg/kg). Control samples were 0.06 mg/kg in trial 300C and 0.26 mg/kg in trial 301C.

CER02607/07: GLP study. No unusual weather conditions. spray, spray volume 150 L/ha. Plants were removed by hand [T235, T236] from 12 areas within the plot and then threshed by hand [T235] or by combine [T236] or seeds were harvested using a small plot combine [T 237]. Samples size >1 kg dry bean seeds. Storage at -10°C for 183 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method CER 2605 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery (86% -109% at 0.01-5.5 mg/kg). Control samples were <0.3LOQ.

CER02609/08: GLP study. Trial T454 (0.075 kg ai/ha) had 73 mm rain within 24 hrs after application. Boom sprayer. Spray volume 150 L/ha. Plants were removed by hand from 12 areas of the middle two rows of the plot and were run through a threshing machine to obtain >1 kg dry bean seeds. Storage at -10°C for a 89-121 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method CER 02609 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery (83% -100% at 0.01–0.5 mg/kg). Control samples were <0.3LOQ (LOQ=0.01 mg/kg).

TECPAR reports Non-GLP. No unusual wather conditions. Broadcast foliar spray using CO2 sprayer with boom, spray volume 200-300 L/ha. Soya plants were sampled by hand and seeds were threshed mechanically 0-11 days after harvest. Seed samples > 1 kg. Samples were kept below -18 °C for 171-198 days in the 1991 trials, 580 days in the 1990 trials and 767 days in the 1989 trials (harvest to final report date). Samples were analysed for total fluazifop using **HPLC-UV Method Yokomizo and Cavalho with a valid LOQ of 0.08 mg/kg**. Average concurrent method recoveries were 75 %. Control samples were < 0.01 mg/kg.

M11034:GLP study. Weather conditions had no impact on the crop growth and development. Broadcast foliar application by CO2 pressurized sprayer with boom. Spray volume 100 L/ha. Dry bean seeds (>1 kg/sample) were taken from 12 random areas from across the plots. Dry beans were threshed at the day of harvest by hitting the plants with a wooden stick. Storage at -20°C for up to 34 days (harvest to final laboratory results). Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET 138, rev 04 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery (76-93% at 0.01-2 mg/kg). Control samples were < 0.01 mg/kg (n = 4).

M4799B Non-GLP study. Weather conditions, soil type, application equipment, spray volume and growth stage at harvest not stated. Sample sizes not stated. Storage at -18 °C; for maximum 234 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Internal standard recovery (98% at 0.1 mg/kg). Control samples < 0.02 mg/kg.

RJ2610B: GLP study. No unusual weather conditions. Broadcast foliar spray using a motor knapsack sprayer. Spray volume 312-327 L/ha. Mature dry bean seeds (1.0-1.1 kg/sample) were sampled by hand and were taken systematically from across plots. Storage at -18°C for a maximum of 121 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery (90-94% at 0.05-2.0 mg/kg). Control samples were < 0.01 mg/kg (n = 2).

RJ2826B: GLP study. No unusual weather conditions. Application by gas knapsack sprayer. Spray volume 247-271 L/ha. Mature dry bean seeds (1.0 kg/sample) were sampled by hand and were taken systematically from across plots. Storage at -18°C for a maximum of 276 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery (100-129% at 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg, except in trial ES10-98-SH001 (0.03 mg/kg).

RJ2994B: GLP study. No unusual weather conditions. Application equipment not stated. Spray volume 442 L/ha. Mature dry bean seeds (1.2-1.3 kg/sample) were sampled by hand. Storage at -18°C for a maximum of 142 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recovery (108-113% at 0.1-1 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

Broad beans, dry (Vicia faba)

Three cGAPs for dry Vicia beans are available:

- cGAP from France with 1 × 0.38 kg ai/ha with a PHI of 56 days
- cGAP from the Netherlands with 1 × 0.38 kg ai/ha with a PHI of 90 days

Trials that could be matched to these cGAPs were summarized.

Table 218 lists trials conducted in the UK (1988, 1989, 1994), Southern France (2006), Italy (2006) and Spain (2006). A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted at under the conditions listed in Table 218. Results marked with "[SS]", "[ST]", "[SK]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[ST] indicates that samples were defrosted during freezer storage.

[SK] no soaking step was used for dry pulses before extraction (or it was unclear whether soaking was used (SK*), whereby fluazifop (II) conjugates are not quantitatively extracted.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (> 25% of residue value).

Vicia faba is also known as broad bean, fava bean, faba bean, field bean, bell bean, English bean, horse bean, Windsor bean, pigeon bean and tick bean. In this JMPR evaluation all trials indicated as field bean, fodder bean or tick bean have been interpreted as being *Vicia spp* unless the

study report indicated that the beans were *Phaseolus spp*. This may be in contradiction with the Codex classification, where field beans are interpreted as *Phaseolus spp*.

Additional trials from the UK (1981) on winter field beans were available with 1 × 0.38–0.50–0.75–1.0–1.5 kg ai/ha with harvest at 119-138 days [Atreya *et al*., 1981, PP9/0552, PP009B068; Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Canada (1980) on broad beans (fava beans) were available with 2 ×0.50 kg ai/ha with harvest at 54 DAT or 1 ×1.0 kg ai/ha with harvest at 76 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. These trials were not summarized, because they would not assist in MRL setting.

Table 218 Supervised field trials on Vicia beans (dry seeds), treated with a broadcast foliar fluazifop-butyl spray

DRY BROAD BEANS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
6749 Kapellen-Drusweiler; Germany; 1985 (fava bean; variety ns; spring sown)	EC 125 (P)	1	0.38	0.094	BBA 53; 30-40 cm high; 60% soil cover; 24 May	SaL	BBA 92	75	0.64 [SS] [SK*]	M4233B; RS8528E2; [Pay and Atreya, 1986, PP5/0374]
idem	EC 125 (P)	1	0.38	0.094	BBA 64; 40-60 cm high; 80% soil cover; 31 May	SaL	BBA 92	68	1.7 [SS] [SK*]	idem
idem	EC 125 (P)	1	0.38	0.094	BBA 66; 50-65 cm high; 90% soil cover; 5 June	SaL	BBA 92	63	1.8 [SS] [SK*]	idem
2321 Depenau-Wankendorf; Germany; 1985 (fava bean; Hara; spring sown)	EC 125 (P)	1	0.38	0.094	BBA 53; 40% soil cover; 11 June	SaL	BBA 92	97	0.08 [SK*]	M4233B; RS8528B3; [Pay and Atreya, 1986, PP5/0374]
idem	EC 125 (P)	1	0.38	0.094	BBA 57; 50% soil cover; 20 June	SaL	BBA 92	88	0.79 [SK*]	idem
idem	EC 125 (P)	1	0.38	0.094	BBA 66; 80% soil cover; 1 July	SaL	BBA 92	77	3.1 [SK*]	idem
Brietlingen, Lüneburg; Germany; 1985 (fava bean; Hara; spring sown)	EC 125 (P)	1	0.38	0.094	BBA 53; 50% soil cover; 5 June	Sa	BBA 92	99	0.06 [SK*]	M4233B; RS8528B4; [Pay and Atreya, 1986, PP5/0374]
idem	EC 125 (P)	1	0.38	0.094	BBA 57; 70% soil cover; 14 June	Sa	BBA 92	90	0.72 [SK*]	idem
idem	EC 125 (P)	1	0.38	0.094	BBA 66; 90% soil cover; 21 June	Sa	BBA 92	83	2.4 [SK*]	idem
Humby;	EC	1	0.38	0.19	flower	LC	MAT	156	< 0.05	M5002B;

DRY BROAD BEANS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Lincolnshire; UK; 1988; (Bourdon, autumn sown)	125 (P)				buds present; 15 cm high; 13 April				[SK*]	GB11-88-S061 [Freeman and Mak, 1989; PP5/0382]; M5002B addendum [Bolygo, 1992, PP5/0381]
Branston; Lincolnshire; UK; 1988; (Bourdon, autumn sown)	EC 125 (P)	1	0.38	0.19	flower buds enclosed, just visible; 8-12 bifoliate leaves; 15-23 cm high; 25 April	SaL	CH	120	< 0.05 [SK*]	M5002B; GB12-88-S061 [Freeman and Mak, 1989; PP5/0382]; M5002B addendum [Bolygo, 1992, PP5/0381]
Hambridge, Somerset; UK; 1988; (Banner, autumn sown)	EC 125 (P)	1	0.38	0.19	4-6 leaves; flower buds not visible; 31 March	LC	MAT	138	0.12 [SK*]	M5002B; GB14-88-S060 [Freeman and Mak, 1989; PP5/0382]; M5002B addendum [Bolygo, 1992, PP5/0381]
Butlers Marston; Warwickshire; UK, 1988; (fava bean; Alfred, spring sown)	EC 125 (P)	1	0.38	0.19	25 cm high; 17 May	C	CH	125	0.13 [SK*]	M5002B; GB15-88-S061 [Freeman and Mak, 1989; PP5/0382]; M5002B addendum [Bolygo, 1992, PP5/0381]
Butlers Marston, Warwickshire; UK; 1988; (fava bean; Alfred, spring sown)	EW 250 (P)	1	0.38	0.19	25 cm high 17 May	C	CH	125	0.21 [SK*]	M4994B; GB15-88-S381 [Freeman 1990; PP5/0384]
idem	EW 250 (P) +0.1% Agral 90	1	0.38	0.19	25 cm high; 17 May	C	CH	125	0.29 [SK*]	M4994B; GB15-88-S382 [Freeman, 1990; PP5/0384]
Dullingham, Newmarket; UK; 1988; (Tick beans, spring sown)	EC 125 (P)	1	0.38	0.19	4-6 leaves; 15 cm high; 17 May	C	CH	113	0.05 [SK*]	M5002B; GB16-88-S061 [Freeman and Mak, 1989; PP5/0382]; [Bolygo, 1992, PP5/0381]
Cockfield; Suffolk; UK; 1988; (fava bean; Alfred, spring sown)	EC 125 (P)	1	0.38	0.19	3-4 leaves; 15 cm high; 20 May	C	CH	115	0.05 [SK*]	M5002B; GB17-88-S061 [Freeman and Mak, 1989; PP5/0382];

DRY BROAD BEANS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
										M5002B addendum [Bolygo, 1992, PP5/0381]
Cockfield; Suffolk; UK; 1988; (fava bean; Alfred, spring sown)	EW 250 (P)	1	0.38	0.19	25 cm high; 20 May	LC	CH	115	0.19 [SK*]	M4994B; GB17-88-S381 [Freeman, 1990; PP5/0384]
Somerton; Somerset; UK; 1988; (fava bean; Alfred, spring sown)	EW 250 (P) +0.1% Agral 90	1	0.38	0.19	flower buds just formed 7 June	SaL	MAT	104	0.97 [SK*]	M4994B; GB14-88-S380 [Freeman 1990; PP5/0384];
Drayton; Somerset; UK; 1989; (Bourdon; autumn sown)	EW 125 (P) +0.1% Agral 90	1	0.44	ns	flower buds not visible; 70% crop cover; 31 March	L	MAT	137	< 0.05 (pods+ seeds) [SK*]	M5316B; GB15-89-S080; [Cullen and Jones, 1991, PP5/0387]; M5316B addendum [Bolygo, 1992, PP5/0388]
idem	EW 250 (P) +0.1% Agral 90	1	0.38	ns	flower buds not visible; 70% crop cover; 31 March	L	MAT	137	< 0.05 (pods+ seeds) [SK*]	idem
idem	EC 125 (P) +0.1% Agral 90	1	0.38	ns	flower buds not visible; 70% crop cover; 31 March	L	MAT	137	< 0.05 (pods+ seeds) [SK*]	idem
Kineton, Warwickshire; UK; 1989; (fava bean; Alfred; spring sown)	EW 125 (P)	1	0.38	ns	flower buds not visible; 25% crop cover; 19 May	ns	CH	94	0.25 [SK*]	M5316B; GB15-89-S080; [Cullen and Jones, 1991, PP5/0387]; M5316B addendum [Bolygo, 1992, PP5/0388]
idem	EW 250 (P)	1	0.38	ns	flower buds not visible; 25% crop cover; 19 May	ns	CH	94	0.15 [SK*]	idem
idem	EC 125 (P)	1	0.38	ns	flower buds not visible; 25% crop cover; 19	ns	CH	94	0.61 [SK*]	idem

DRY BROAD BEANS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					May					
Abington, Cambridgeshire; UK; 1989; (fava bean; Alfred; spring sown)	EW 125 (P)	1	0.38	ns	5-6 leaves; 48% crop cover; 17 May	CL	EH	78	0.29 [SK*]	M5316B; GB15-89-S080; [Cullen and Jones, 1991, PP5/0387]; M5316B addendum [Bolygo, 1992, PP5/0388]
idem	EW 250 (P)	1	0.38	ns	5-6 leaves; 48% crop cover; 17 May	CL	EH	78	0.37 [SK*]	idem
idem	EC 125 (P)	1	0.38	ns	5-6 leaves; 48% crop cover; 17 May	CL	EH	78	0.27 [SK*]	idem
Lawshall, Suffolk; UK; 1989; (Banner; autumn sown)	EW 125 (P) +0.1% Agral 90	1	0.38	0.19	3-4 leaves, 40% crop cover; 22 March	CL	fully senesced	147	< 0.05 [SK*]	M5316B; GB15-89-S080; [Cullen and Jones, 1991, PP5/0387]; M5316B addendum [Bolygo, 1992, PP5/0388]
idem	EW 250 (P) +0.1% Agral 90	1	0.38	0.19	3-4 leaves, 40% crop cover; 22 March	CL	fully senesced	147	< 0.05 [SK*]	idem
idem	EC 125 (P) +0.1% Agral 90	1	0.38	0.19	3-4 leaves, 40% crop cover; 22 March	CL	fully senesced	147	< 0.05 [SK*]	idem
Banbury, Oxon; UK; 1994; (fava bean; Victor, spring sown)	EC 125 (P) +0.1% Agral 90	1	0.38	0.19	3-4 nodes, no flower buds visible; 13 May	SaCL	PGRO 410	98	0.09	RJ1894B; GB15-94-S161 [Patel and Elliott, 1996; PP5/0416]
Bugbrooke, Warwicksh; UK; 1994; (fava bean; Victor, spring sown)	EC 125 (P) +0.1% Agral 90	1	0.38	0.19	3-4 nodes, no flower buds visible; 13 May	SaCL	PGRO 410	97	0.08	RJ1894B; GB15-94-S162 [Patel and Elliott, 1996; PP5/0416]
Grainsborough, Lincolnshire; UK; 1994; (fava bean;	EC 125 (P) +0.1%	1	0.38	0.19	2-4 nodes; 4-14 cm high, no	CL	KNOTT 310	103	0.10 [ST]	RJ1894B; GB11-94-S161 [Patel and Elliott, 1996;

DRY BROAD BEANS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Alfred, spring sown)	Agral 90				flower buds visible; 13 May					PP5/0416]
Grantham, Lincolnshire; UK; 1994; (fava bean; Punch, autumn sown)	EC 125 (P) + 0.1% Agral 90	1	0.38	0.19	1-4 nodes; 6.5-7.0 cm high, no flower buds visible; 29 April	SaL brash	KNOTT 310	116	0.07 [ST] [CT] [Cntrl=0.09]	RJ1894B; GB11-94-S162 [Patel and Elliott, 1996; PP5/0416]
82200, L'Homor de Cos, Southern France, 2006 (fava bean: Irena, winter sown)	EC 125 (P)	1	0.31	0.078	BBCH 24; 14 Apr	CL	BBCH 89	90	0.23 [SK]	CEMR-3008; AF/10374/SY/1; [Bell, 2007, PP5/1545]
82100, Escatalens, Castel sarrasin, Southern France, 2006 (fava bean: Melodie; winter sown)	EC 125 (P)	1	0.32	0.078	BBCH 22-23; 20 Apr	SaC	BBCH 89	91	0.19 [SS] [SK]	CEMR-3008; AF/10374/SY/2; [Bell, 2007, PP5/1545]
Pegola Italy, 2006; (fava bean: Polo; spring sown)	EC 125 (P)	1	0.31	0.078	BBCH 39; 18 Apr	SaCL	89	93	< 0.05 [SK]	CEMR-3008; AF/10374/SY/5 [Bell, 2007, PP5/1545]
Cortes, Spain, 2006 (fava bean: Reina Blanca, autumn sown)	EC 125 (P)	1	0.31	0.078	BBCH 39; 7 Apr	CL	BBCH 89	90	3.1 [SK]	CEMR-3008; AF/10374/SY/3 [Bell, 2007, PP5/1545]
Barboles, Spain, 2006 (fava bean: Reina Mora; autumn sown)	EC 125 (P)	1	0.31	0.078	BBCH 39; 4 Apr	SaCL	89	92	1.8 [SK]	CEMR-3008; AF/10374/SY/4 [Bell, 2007, PP5/1545]

GSH: EH = ripe; early harvest

[ST] GB11 samples were stored at -18 C, except for a 12 hr period, when the freezer temperature rose to +15 °C and the samples defrosted. These trials cannot be selected for MRL derivation.

[SS] Sample size was < 1 kg (0.8 kg in 1985 DE trial RS8528E2; 0.9 kg in 2006 FR trial AF/10374/SY/2). These trials cannot be selected for MRL derivation.

[SK] The soaking step was omitted in the analytical method, whereby fluazifop (II) acid conjugates are incompletely extracted.

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

Additional trial information

M4233B Non-GLP. Reverse decline trials. No unusual weather conditions. Application by knapsack sprayer with spray boom. Spray volume 400 L/ha.. Pods were harvested by hand. Seed samples were at least 1 kg, unless specified otherwise.

Samples were stored at -20 °C or lower. Storage period is not indicated, but is maximally 297 days (harvest to final report). Samples were analysed for total fluazifop using **NMR method PPRAM 83 using internal standard with a valid LOQ of 0.05 mg/kg**. Internal standard recoveries were 71%. Control samples < 0.02 mg/kg (n = 3).

M5002B and addendum. GLP. No unusual weather conditions. Plot size 30-72 m^2. Application one man hand held knapsack CO2 3 m sprayer (Cockfield), CO2 knapsack (Dullingham), CO2 plot sprayer (Hambridge), 2 m single man CO2 sprayer (Marston), CO2 single man knapsack spray (Branston) or 2 man boom (Humby). Spray volume 200 L/ha, formulation JF8908 (EC 125). Samples were harvested randomly by hand (Branston/Humby/Hambridge/Dullingham) or mechanical by a combine (Marston/Cockfield). Sample consisted of 400 g seeds. Samples were stored at -18 °C for a maximum period of 12 months. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recoveries were 79%. Controls samples were < 0.05 mg/kg as fluazifop**.**

M4994B Non-GLP. No unusual weather conditions. Plot size 18-36 m^2. Application by CO2 plot sprayer (Somerton, 3 m swatch, 2 bar pressure), or 2 m single man CO2 sprayer (Marston/Kineton) or one man held CO2 knapsack (Cockfield, 3.0 m). Spray volume 200 L/ha, formulation FD4282 (EW 250), PP005BX108T (EW 250), or PP005 (EW 250/EW 375). In some cases 0.1% AGRAL was added. Samples were harvested by hand, randomly within the plot (Somerton) or mechanical by combine (other locations). Samples consisted of 400 g seeds. Samples were stored at -18 °for a maximum period of 12. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recoveries were 78%. Control samples were < 0.05 mg/kg as fluazifop.

M5316B and **M5316B addendum**. GLP. Winter beans (Bourdon, Banner) or spring beans (Alfred). No unusual weather conditions. Application not stated, except for Lawshall: one man hand-held CO2 knapsack sprayer. Spray volume not stated or 200 L/ha (Lawshall). Samples were harvested by hand by walking diagonally across each plot (Drayton), by combine (Lawshall) or not stated (other locations). Field samples: 1 kg mature pods (Drayton), 1 kg mature seeds (Abington, Lawshall) or not stated (others). Samples were stored at -18 °C (storage time not stated but less than 12 months). Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Results were not corrected for recoveries (mean 76-86% for seeds, 88% for seeds+pods at 0.5 mg/kg). Control samples < 0.05 mg/kg for seeds or seeds+pods.

RJ1894B Non-GLP. Weather was drier than normal. Plot size at least 68 m^2. Application 2 man held CO2 plot sprayer (2 booms). Spray volume 200 L/ha, 0.1% AGRAL is added as adjuvant. All treatments were before flower buds were visible (PGRO 201). Pods were harvested by hand along the length of the plot (Banbury), on at least 10 points (Bugbrook), using a diagonal sampling pattern (Grainsborough/Grantham). Seeds were removed by hand thrashing. Seed samples were at least 1.0 kg. Samples were stored at -18 °C for 3 months. Samples were analysed for total fluazifop using **NMR method RAM 197/02 with a valid LOQ of 0.05 mg/kg**. Internal standard recoveries were 92%. Control samples had residues up to **0.09 mg/kg**.

CEMR-3008-GLP. No unusual weather conditions. Application by plot spraywer. Spray volume 400 L/ha. Plants were harvested by hand and then threshed into seed by hand (Spain) or using a static combine (France) or samples were collected by plot combine (Italy). Seed samples were at least 1 kg, except in trial SY/2 (0.9 kg). Samples were stored at -9 °C or lower. Storage period is not indicated, but is maximally 489 days (harvest to final report). Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent method recoveries were 105%. Control samples < 0.05 mg/kg.

Cowpea, dry (Vigna spp)

A GAP for dry cowpeas is not available (the USA GAP on dry beans excludes cowpeas).

Trials from the USA (1986) were available on blackeyed peas (cow peas) with an application of 2 × 0.42 kg ai/ha and harvest at 36–83 DAT [McKay, 1989, 405660, report RR 89-046B]. As the manufacturer did not seek to have maximum residue levels estimated on dry cowpeas, the available studies on cowpeas were not summarized.

Field peas, dry (Pisum sativum)

Four cGAPs for dry peas are available:

- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 56 days for dry peas (as nn with PHI)
- cGAP from Belgium with 1 ×0.38 kg ai/ha before bloom for dry peas (as nn with GS)
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha with a PHI 90 days for pulses
- cGAP from the UK with 1 x0.19 kg ai/ha before flower buds are visible for dry peas

Trials that could be matched to this cGAP were summarized.

Table 219 lists trials conducted in Germany (2000, 2010, 2011), UK (1981, 1983, 1998, 2000, 2001), Netherlands (1983), France (1997, 2001, 2003, , 2006, 2008-2009, 2011), Italy (2001, 2006,

2012) and Spain (2006, 2008, 2009)). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 219. Results marked with "[WC]", "[SS]", "[SK]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[WC] indicates that the weather conditions affected growth and yield of the crop

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[SK] no soaking step was used for dry pulses before extraction (or it was unclear whether soaking was used (SK*), whereby fluazifop (II) conjugates are not quantitatively extracted.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

Additional trials from the UK (1981, 1983)were available with 1 ×0.19–0.25–0.75-1.0 kg ai/ha with harvest at 77–84 days or 2 × 0.75 kg ai/ha with harvest at 84 days [Atreya *et al*., 1981, PP9/0554, report PP009B070; Atreya and Harradine, 1982, PP9/0062, report RJ0291B, Harradine, 1986, PP5/0398, report M4209B]. Additional trials from Canada (1982, 1990) were available with 1 × 0.12–0.25–0.50 kg ai/ha with harvest at DAT 66-82 days [Harradine, 1984, PP9/0116, report M3754B; Jones, 1992, PP5/0405, report RJ1059B]. Additional trials from Australia (1979-1980) were available with 1 × 0.25–0.50–1.0 kg ai/ha with harvest at DAT 134 days [Atreya *et al*., 1981, PP9/0525, report PP009B038; Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. These trials were not summarized, because they would not assist in MRL setting.

Table 219 Supervised field trials on Pisum peas (dry seeds), treated with a broadcast foliar fluazifop-butyl spray

DRY PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
D-23858 Klein Barnitz; Germany; 2000; (Miami)	EC 125 (P) YF2 (b)	1	0.38	0.12	BBCH 36; 40% crop cover; 15 May	SaL	89	86	0.17	RJ3209B; DE11-00-S161; [Mason, 2001, PP5/1112]
idem	EC 125 (P) YF1 (b)	1	0.38	0.12	BBCH 36; 40% crop cover; 15 May	SaL	89	86	0.10	idem
D-19089 Badegow; Germany; 2000; (Eiffel)	EC 125 (P) YF2 (b)	1	0.38	0.12	BBCH 35; 34% crop cover; 5 May	LSa	89	95	0.24	RJ3209B; DE12-00-S161; [Mason, 2001, PP5/1112]
idem	EC 125 (P) YF1 (b)	1	0.38	0.12	BBCH 35; 34% crop cover 5 May	LSa	89	95	0.16	idem
68623 Lampertheim; Germany; 2010; (Maxigold)	EC 125 (P)	1	0.35	0.093	BBCH 15-35; 30 July	Sa	87 dry	35	0.18 [SS]	CEMR-4658-REG; CEMS-4658-01; [Jutsum and Allen, 2011, A12791B_

DRY PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
										10837]
Baden-Württemberg, Germany, 2011 (Crackerjack)	EC 125	1	1.9	0.47	BBCH 33-34; 31 May, 2011;	CL	89	79	0.68, 0.63, 0.67 mean 0.66	CEMR-5037-REG; CEMS-5037-02; [Devine, 2013, A12791B_11068] (processing)
Thornhaugh UK, 1981, (pea, Meno)	EC 250 (rac)	1	0.75	0.28	5 leaves; 10 June	ns	dry	56	0.71 [SS] [SK*]	PP009B070; PGRD [Atreya *et al*., 1981, PP9/0554]
idem	EC 250 (rac)	1	1.0	0.37	5 leaves; 10 June	ns	dry	56	1.7 [SS] [SK*]	idem
Rauceby, Lincolnshire; UK, 1983 (pea, Progretta)	EC 125 (P)	1	0.19	0.072	30-40 cm high; in bud; 21 June	Sa	NH (dry)	41	1.1 [SS] [SK*]	M3724B; 8R/83 [Harradine, 1984, PP9/0119]
idem	EC 250 (rac)	1	0.38	0.14	30-40 cm high; in bud; 21 June	Sa	NH (dry)	41	2.4 [SS] [SK*]	idem
Rauceby, Lincolnshire; UK, 1983 (pea, Progretta)	EC 125 (P)	1	0.19	0.072	13 cm high; no buds; 23 May	Sa	NH (dry)	77	0.06 [SS] [CT] [SK*] [Cntrl=0.05]	M3724B; 30B/93/4/NE18 [Harradine, 1984, PP9/0119]
idem	EC 250 (rac)	1	0.38	0.14	13 cm high; no buds; 23 May	Sa	NH (dry)	77	0.05 [SS], [CT] [SK*] [Cntrl=0.05]	idem
Wincanton; UK; 1998; (combining field pea, Eiffel)	EC 125	1	0.38	0.19	BBCH 36; 12 May	SaSiL	97	87	0.10	RJ2785B; GB14-98-S121 [Mason and Myles, 1999; PP5/0158]
Steventon; UK; 1998 (combining field pea, Maro)	EC 125	1	0.38	0.19	BBCH 35-36; 15 May	CL	97	87	0.02	RJ2785B; GB14-98-S122 [Mason and Myles, 1999; PP5/0158]
Warwick; Warwickshire; UK; 2000 (Espace)	EC 125 (P)	1	0.38	0.19	BBCH 34-37; 31 May	ISB	89	82	0.51	RJ3211B; GB05-00-S074; [Mason and Bailey, 2001, PP5/1090]
idem	EC 125 (P)	1	0.38	0.19	BBCH 34-37; 31 May	ISB	89	82	0.54	idem
Ashorne; Warwickshire; UK; 2000; (Croma)	EC 125 (P)	1	0.38	0.19	BBCH 38-39; 30 May	CL	89	78	0.67	RJ3211B; GB05-00-S075 ; [Mason and Bailey, 2001, PP5/1090]
idem	EC 125 (P)	1	0.38	0.19	BBCH 38-39; 30 May	CL	89	78	1.0	idem
Lough	EC	1	0.38	0.13	BBCH	SaCL	CH	65	0.59	RJ3266B;

DRY PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
borough; Leicestersh; UK, 2001; (Nitouche)	125 (P)				37-38; 18 June		(dry)			AF/5815/SY1 [McGill and Richards, 2002, PP5/1227]
Vedskoele, Koege; Denmark; 1989; (Bodil)	EW 125 (P) + Lissafol	1	0.38	ns	BJ4 = 4 leaves; 15% crop cover; 18 May	L	BJ10 dry seeds	71	0.12	M5347B; DK10-89-5063; [Jones, 1991, PP5/0150]
Netherlands, 1983; (pea; Finale)	EC 250 (rac)	1	0.25	0.050	20-22 cm high; 70% soil cover; no flowers; 10 June	C	peas almost ripe (dry)	54	0.16, 0.19, 0.21, 0.24, mean 0.20 [a] [SK*]	M3759B; ICI H 83/120 [Harradine, 1984, PP9/0117]
idem	EC 250 (rac)	1	0.38	0.075	20-22 cm high; 70% soil cover; no flowers; 10 June	C	peas almost ripe (dry)	54	0.22, 0.25, 0.27, 0.29, mean 0.26 [a] [SK*]	idem
idem	EC 125 (P)	1	0.12	0.025	20-22 cm high; 70% soil cover; no flowers; 10 June	C	peas almost ripe (dry)	54	< 0.05, 0.10, 0.11, 0.12, mean 0.10 [a] [SK*]	idem
idem	EC 125 (P)	1	0.19	0.038	20-22 cm high; 70% soil cover; no flowers; 10 June	C	peas almost ripe (dry)	54	0.20, 0.20, 0.21, 0.21, mean 0.21 [a] [SK*]	idem
27190; Faverolle la Campagne; Normandy, N-France; 1997; (field pea, spring sown, Baccara)	EC 125	1	0.38	0.12	BBCH 39; 07 May	C	907	86	0.27	RJ2510B; S207.97; [Mason *et al*., 1999; PP5/0157]
80250 Grivesnes; Picardy; N-France; 1997; (field pea, spring sown, Baccara)	EC 125	1	0.38	0.12	BBCH 38; 23 May	SiC	89	68	2.0	RJ2510B; S106.97 [Mason *et al*., 1999; PP5/0157]
Castel sarrasin; Tarn et Garonne; S-France, 2001 (Solara)	EC 125 (P)	1	0.31	0.11	BBCH 38; 30-35 cm tall; 2 May	SiC	CH	56	0.91	RJ3300B; AF/5835/SY/2; [McGill, 2002, PP5/1233]

DRY PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
86120; Leger de Montbrillais; S-France; 2003; (Bingo)	EC 125 (P)	1	0.31	0.10	BBCH 50; 23 May	C	89	36	3.4 [SK]	03-7059; trial no ns; [Mason, 2004, PP5/1426]
82100 Castel sarrasin; S-France 2003; (Austin)	EC 125 (P)	1	0.31	0.10	BBCH 55; 5 May	CL	CH	46	5.9	03-7058; 03-7058; [Mason, 2004, PP5/1425]
82220 Vazerac; S-France; 2006 (Austin)	EC 125 (P)	1	0.32	0.063	BBCH 21-23; 13 Apr	CL	89	90	0.20 [SK]	CEMR-3373; AF/10377/SY2 [Bell, 2008, PP5/1544]
Cordes Tolosane; Tarn et Garonne; S-France; 2008 (Panache)	EC 125 (P)	1	0.31	0.062	BBCH 39; 25 Apr	CL	89 89	74 90	NA 1.1	T009247-07-REG; S08-00863-01 [Jutsum; 2001; A12791B_10830]
Castelsarrasin, Tarn et Garonne; S-France; 2008-09; (field pea: Enduro)	EC 125 (P)	1	0.34	0.062	BBCH 36; 10 Apr 2009	CL	89 89	76 90	NA 0.49	CEMR-4385-REG; S09-00355-02 [Jutsum, 2011, A12791B_10831]
62214 Beaumetz les Cambrai, Nord Pas-de-Calais, N-France; 2011 (Pactole)	EC 125	1	2.0	0.42	BBCH 39; 15 March, 2011;	SiL	89	63	3.6, 3.6 mean 3.6 [a] [WC]	CEMR-5037-REG; CEMS-5037-01; [Devine, 2013, A12791B_11068] (processing)
Ponticelli; Emilia Romagna; Italy, 2001 (Resal)	EC 125 (P)	1	0.31	0.11	BBCH 37; 10 cm tall; 14 May	CL	CH (dry)	52	0.10	RJ3300B; AF/5835/SY/3; [McGill, 2002, PP5/1233]
Crevalcore, Emilia Romagna; Italy; 2001 (Lambado)	EC 125 (P)	1	0.31	0.11	BBCH 35-37; 8-10 cm tall; 15 May	CL	CH (dry)	49	0.18	RJ3300B; AF/5835/SY/4 [McGill, 2002, PP5/1233]
40016 San Giorgio di Piano; Italy; 2006; (Dakota)	EC 125 (P)	1	0.30	0.062	BBCH 31-33; 24 Apr	SaCL	89	81	0.08 [SK]	CEMR-3373; AF/10377/SY5 [Bell, 2008, PP5/1544]
40058; Pegola di Malalbergo; Italy; 2006 (Coral)	EC 125 (P)	1	0.30	0.062	BBCH 31-33; 24 Apr	SaCL	89	87	0.06 [SK]	CEMR-3373; AF/10377/SY6 [Bell, 2008, PP5/1544]
29010;	EC	1	0.32	0.10	BBCH	SaL	89	38	0.04	CEMR-5453;

DRY PEA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Cascina Marazzo Gragnago Trebbiense; Italy; 2012; (Heidi)	125 (P)				30-33; 4 May		dry			SRIT12-1031-37HR; [Langridge, 2013, A12791B_11035]
50561 Bisimbre; Spain; 2006; (Ideal)	EC 125 (P)	1	0.31	0.062	BBCH 50; 7 Apr	SaL	89	63	2.0 [SK]	CEMR-3373; AF/10377/SY3 [Bell, 2008, PP5/1544]
50490 Villareal de Huelva; Spain; 2006 (Gracia)	EC 125 (P)	1	0.31	0.063	BBCH 32; 27 Apr	CL	89	88	0.54 [SK]	CEMR-3373; AF/10377/SY4 [Bell, 2008, PP5/1544]
Almansa; Albacete; Spain; 2008 (field pea, Messire)	EC 125 (P)	1	0.33	0.052	BBCH 39; 25 Apr	CL	89	90	1.4 [SS]	T009247-07-REG; S08-00863-04; [Jutsum; 2001; A12791B_10830]
Barrax; Albacete; 46160; Spain; 2009; (field pea: Messire)	EC 125 (P)	1	0.31	0.052	BBCH 34; 13 May	CL	88 88	56 92	NA 0.04	CEMR-4385-REG; S09-00355-03 [Jutsum, 2011, A12791B_10831]

BBCH 10-19 leaf development; BBCH 30-39: stem elongation of the main shoots, number indicates visible internodes, leaves only. BBCH 50-59 inflorescence emergence; BBCH 60-69 flowering; BBCH 70-79 = pod development; BBCH 80-89 = ripening of pods and seeds

Bjorkman Scale BJ 4 = 4 leaves; BJ10 equivalent to BBCH 89 [Syngenta, 2016, Response to questions 15]

[SS] Sample size not stated (report PP009B070) or less than 1 kg seeds (0.5 kg whole plants in 1983 UK trials, 0.7 kg in 2008 Spanish trials, 0.36 kg seeds in trial CEMS-4658-01. Samples are considered not representative for MRL setting.

[WC] In trial CEMS 5037-01 plants were fallen on the ground, were only 10-15 cm high and crop yield was lower than in the control plot, due to phytotoxicity. Samples did not comply with commercial standards.

[SK] The soaking step was omitted in the analytical method, whereby fluazifop (II) conjugates are incompletely extracted.

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

[a] Three to four replicate samples were taken from one plot. The mean is taken for MRL-derivation if according to cGAP.

Additional trial information

RJ3209B GLP. Dry peas. No unusual weather conditions. Application by air pressured knapsack plot sprayer with 2.5 m boom. Spray volume 300 L/ha. Samples were taken by hand from at least 20 spots per plot in a systematic way. Samples were threshed by hand to obtain 1 kg dry seeds. Samples were stored at -18 °C for 89-90 days. Samples were analysed for total fluazifop using HPLC-MS/MS method **RAM 287/02**. Results were not corrected for individual concurrent method recoveries (85-97% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

CEMR-4658. GLP Dry peas. No unusual weather conditions. Broadcast foliar spray using a backpack sprayer. Spray volume 379-448 L/ha. Pea plants (12 units) were taken by hand (3 Sept Germany at BBCH 87-89, indicating dry plants) using a suitable distributive pattern. Due to bad growth of the crop, the sample sizes for pea seeds (0.36 kg) were reduced. Storage at -18 °C for a maximum of 317 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (70-92% at 0.01–0.1mg/kg). Control samples < 0.01 mg/kg.

CEMR-5037 GLP Dry field peas. No unusual weather conditions, except in trial CEMS-5037-02, where 2 mm rain fell in the first 9 hrs after application. Broadcast foliar spray using a backpack sprayer. Spray volume 410-470 L/ha. Dry pea seeds were harvested mechanacilly using a small plot combine (10 kg for processing). In trial CEMS 5037-01 plants were fallen on the ground, were only 10-15 cm high and crop yield was lower than in the control plot, due to phytotoxicity. Storage at -18 °C for a maximum of 24 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (77-85% at 0.01–0.5mg/kg). Control samples < 0.01 mg/kg.

PP009B070 Non-GLP. Weather conditions not reported. Spray equipment not reported; spray volume 270 L/ha. Sampling conditions and sample size not stated. Storage temperature not stated; storage time 55 days. Samples were analysed for total fluazifop using **HPLC/UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Samples were corrected for average concurrent recoveries (77% for seeds); uncorrected results are not available. Control samples < 0.02 mg/kg.

M3724B Non-GLP. Dried peas (*Pisum sativum*). No unusual weather conditions. Spray using a hand-held sprayer. Spray volume 260 L/ha.. Samples of 0.5 kg whole dried plants were sampled by hand and seeds were separated off. Samples were stored at -18°C or lower. Storage period was not stated bus was at maximum of 200 days (Harvest to final report date). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM62/2 with a valid LOQ of 0.05 mg/kg**. Results were not corrected for average concurrent method recoveries (76-81% at 0. 1–0.5 mg/kg). Control samples < 0.02, except 0.05 mg/kg in trial 30B/93/4/NE18.

RJ2785B GLP. Weather was reported as normal temperatures, but unusal rainfall pattern. Plot size 54 m^2. Application by CO2 pressurised hand held sprayers. Application volume 200 L/ha. Pods were sampled by hand than shelled. Care was taken to avoid plot boundaries. Samples of 1 kg seeds were taken systematically across the plot. Storage time 3 months at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent method recoveries (79-105% at 0.01-1.0 mg/kg), Control samples were < 0.01 mg/kg as fluazifop.

RJ3211B GLP. Dried combining peas (*Pisum sativum*). No unusual weather conditions. Broadcast foliar spray using a one-man offset sprayer. Spray volume 200 L/ha.. Samples were taken systematically across the plots, 1 kg each. Samples were stored at -18 °C for 50-55 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for individual concurrent method recoveries (91-97% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ3266B GLP. Dried peas (*Pisum sativum*). Unusual weather conditions had no effect on crop health. Overall foliar spray using a precision boom sprayer. Spray volume 292 L/ha. Whole plants (> 4.0 kg) were taken by hand systematically from all areas of at least 12 plants and seeds were separated off (> 1.0 kg) by threshing using a small plot combine. Samples were stored at -18 °C for 149 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for individual concurrent method recoveries (94-97% at 0.2–0.5 mg/kg). Control samples < 0.01 mg/kg.

M5347B. GLP. Peas. No unusual weather conditions. Application equipment and spray volume not stated. Sample sizes: 0.5-0.6 kg seeds (DAT 71). At DAT 71, shells were removed, crushed and cleaned by blowing in the wind. Storage at -18 °C for a maximum of 619 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with internal std with a valid LOQ of 0.05 mg/kg**. Average concurrent external std recovery 80% at 0.5 mg/kg. Control samples < 0.05 mg/kg.

M3759B Non-GLP. No unusual weather conditions. Application by propane sprayer; spray volume 500 L/ha. Four replicate samples per plot were harvested by hand or machine. Sample size 1.5 kg dried pea seeds. Samples were stored at -20 °C (storage time not stated but less than 252 days). Samples were analysed for total fluazifop using **HPLC/UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Samples were corrected for internal standard recoveries (mean 61-74% for seeds). Control samples < 0.05 mg/kg.

RJ2510B GLP. No unusual weather conditions. Application by hand held sprayers, spray volume 300 L/ha. Mature field peas were sampled by hand or combine, taking care to avoid the plot boundaries. Samples of 1250-1600 g seeds were taken systematically from across the plots. Storage time 4 months at -18 °C. Samples were analysed for total fluazifop using HPLC-MS/MS method **RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (72-78% at 0.05-0.1 mg/kg). Control samples were < 0.01 mg/kg.

RJ3300B GLP. No unusual weather conditions. Overall foliar spray using a precision boom sprayer, spray volume 300 L/ha. Whole plants were sampled by hand and were taken systematically from all areas of a least 12 points in the plot. Seeds were separated using a small plot combine harvester. Samples consited of > 1 kg seeds. Storage time 145-153 days at -13 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (106-107% at 0.2–0.5 mg/kg). Control samples were < 0.01 mg/kg.

03-7059 GLP. Rainfall (1 mm) within 24 hrs of application. Overall foliar spray using a plot sprayer, spray volume 300 L/ha. Pea seeds (> 1 kg) were sampled by hand. Storage at -18 °C for a maximum of 103 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (100-104% at 1.0-5.0 mg/kg for seeds). Control samples were < 0.01 mg/kg.

03-7058 GLP. Dried peas (*Pisum sativum*). No unusual weather conditions. Overall spray with plot sprayer. Spray volume 299 L/ha.. Samples were taken by hand and threshed into seed (1.0 kg) using a small plot combine harvester. Samples were stored at -18 °C for 117 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02**

with a valid LOQ of 0.01 mg/kg. Results were not corrected for individual concurrent method recoveries (100-104% at 1.0-5.0 mg/kg). Control samples < 0.01 mg/kg.

CEMR-3373 GLP. Dried peas (*Pisum sativum*). No unusual weather conditions. Foliar spray using a plot sprayer. Spray volume 480-507 L/ha.. Samples were taken by hand and were threshed into seed (1.0-1.8 kg) using a combine harvester. Samples were stored at -9°C or lower for a maximum of 502 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 modification C with a valid LOQ of 0.01 mg/kg**. The soaking step was omitted. Results were not corrected for individual concurrent method recoveries (87-106% at 0.01–0.5 mg/kg). Control samples < 0.05 mg/kg.

T009247-07-REG. GLP study. No unusual weather conditions. Broadcast foliar spray by boom sprayer, spray volume 490-630 L/ha. At least 12 pea plants were sampled by hand. Seeds were separated off using a combine (France) or by hand (Spain). Pea seeds (> 1 kg in French trial; 0.7 kg seeds in Spanish trial. Storage at -12 ºC or lower for 145-161 days. Freezer temperature reached -3 ºC at 2 different days during storage (France). This is considered to have no impact on the results as the samples remained frozen. Samples were analysed for total fluazifop using HPLC-MS/MS method **RAM 287/02, modification B with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (85-90% at 0.01–0.1 mg/kg in seeds). Control samples were < 0.01 mg/kg.

CEMS-4385. GLP study. No unusual weather conditions. Broadcast foliar spray by boom sprayer, spray volume 540-600 L/ha. Pea seeds (> 1 kg) were collected from at least 12 plants. Sampling by hand using a suitably distributive pattern. Pea seeds were threshed at the test site using a plot combine. Storage time 398 days at -7 °C with 2.5 hrs at +1 ºC (samples remained frozen at all times) This is considered to have no impact on the results. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (68-89% at 0.01–0.1 mg/kg. Control samples were < 0.01 mg/kg.

CEMR-5453. GLP Dry field peas. No unusual weather conditions. Broadcast foliar spray (Italy) using a backpack sprayer. Spray volume 297-331 L/ha. Pea plants (12 units) were taken by hand (11 June Italy) using a suitable distributive pattern. Since plants were harvested at BBCH 89, these are considered as dry peas. Sample sizes pea seeds (> 1 kg). Storage at -18 °C for a maximum of 190days. Freezer reached peak temperatures of -9, -10 ºC for 2 hours. This is considered to have no impact on the results, since samples remained frozen at all times. Samples were analysed for total fluazifop using H**PLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (95-113% at 0.01–0.5 mg/kg for seeds). Control samples < 0.01 mg/kg.

Lupins

One cGAP for lupins is available:

- cGAP from France with 1 ×0.38 kg ai/ha with a PHI of 56 days for lupins

Trials from Australia (1980) were available on lupins with an application of 1 × 0.25-0.38-0.75 kg ai/ha and harvest at 163 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. As the manufacturer did not seek to have maximum residue levels estimated on lupins, the available studies on lupins were not summarized.

Soya beans, dry (Glycine spp)

Three cGAPs for dry soya beans are available:

- cGAP from the USA with 1 ×0.42 kg ai/ha pre-blooming (before BBCH 60, up to V5) plus 1 × 0.10 kg ai/ha at blooming or later (from BBCH 60 or R1) with a PHI of 60 days.
- cGAP from Brazil with 1 ×0.25 kg ai/ha with a PHI of 60 days
- cGAP from France wit 1 ×0.19 kg ai/ha with a PHI of 90 days.

Trials that could be matched to these cGAPs were summarized.

Table 220 lists trials conducted in the USA (1998, 2000, 2010), Canada (1981, 1983, 1987, 2007), Brazil (1985, 1991, 2005, 2011, 2013), Switzerland (2004), France (1996, 2003), Italy (1996, 1997, 1998, 1999, 2000). A broadcast or banded foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted at under the conditions listed in Table 220. Results marked with "[SS]", "[ST]", "[SK]", "[AM]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP.

[AM] indicates that the analytical method did not contain a hydrolysis step and therefore fluazifop (II) conjugates are not included

[SK] no soaking step was used for dry pulses before extraction (or it was unclear whether soaking was used (SK*), whereby fluazifop (II) conjugates are not quantitatively extracted.

[SS] indicates that the sample sizes were not reported or less than 1 kg.

[ST] indicates that the sample was at room temperature prior to freezer storage.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

Treated soya beans were determinate or indeterminate varieties. The determinate variety first finishes its growing cycle before producing flowers and pods and is generally taller than the indeterminate variety. The indeterminate variety starts flowering and pod formation during the growing cycle. The indeterminate variety may have pods at the base of the plant, flowers in the middle part, while the top part still developes new leaves. The indeterminate variety is generally taller than the determinate variety.

Additional trials from the USA (1978, 1979, 1980, 1981, 1982, 1983) were available with 1 ×0.56–1.2 kg ai/ha with harvest at 83 and 95–217 DAT or 2 × 0.28–0.42–0.56 kg ai/ha with harvest at 61–183 DALT [Atreya *et al*, 1981, PP9/0736, report PP009B036, Atreya and Froggatt, 1981, PP9/0384, report RJ0226B, Ussary, 1981, 406278, report TMU0678/B, Koubek, 1982, 406227, report TMU0922/B, Koubek, 1983, 406276, report TMU1037/B, Koubek, 1983, 432235, report TMU1172/B, Koubek, 1984, PP5/0406, TMU1403/B]. Additional trials from Canada (1979, 1980, 1981, 1982, 1985, 1987, 2006) were available with 1 × 0.12–0.25–0.50–0.75–1.0–2.0 kg ai/ha with harvest at 77–83 and 95–144 DAT or 1 × 0.50 kg ai/ha with harvest at 69-73 DAT [Atreya *et al*., 1980, PP9/0498, report PP009B004; Atreya *et al*., 1981, PP9/0510, report PP009B016; Atreya and Froggatt, 1981, PP9/0384, report RJ0226B; Atreya *et al*., 1983, PP9/0669, report PP009B229, Atreya and Harradine, 1983, PP9/0722, report PP009B261, Pay, 1987, PP5/0409, report M4322B, Dick, 1988, PP5/0410, report M4644B; Sagan, 2010, A12791B_50006, report CER 02401/06]. Additional trials from South Africa (1982, 1991) were available with 1 ×0.25–0.50–1.0 kg ai/ha with harvest at 118 DAT or 1 ×1.0–2.0 kg ai/ha with harvest at 69 DAT [Atreya and Collis, 1983, PP9/0606, report PP009B176, Johnson *et al*., 1993, PP5/1031, report TMJ3065B]. Additional trials from Hungary (1988) were available with 1 ×0.25 kg ai/ha with harvest at 98 DAT [Agnes, 1988, PP9/0121, report RIC1927]. Additional trials from Italy (2000) were available with 1 ×0.25 kg ai/ha with harvest at 98–113 DAT [Mason and Giacomelli, 2001, PP5/1068; report RJ3206B]. These trials were not summarized, because they would not assist in MRL setting.

Besides total fluazifop, also CF3-pyridone (X) was analysed in soya bean samples from some 1979 and 1980 trials conducted in the USA [Atreya *et al*., 1981, PP9/0733, PP009B061]. These trials were summarized in the metabolism section.

Table 220 Supervised residue field trials on soya (dry seeds), treated with a broadcast or banded foliar fluazifop-butyl spray

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray application										
Whitakers, NC, USA, 1998 (indeterminate Pioneer 9362); plot 2	EC 240 (P) +0.25% v/v NIS	2 (1)	0.42 0.21	0.22 0.11	1–V5; 2–V5; 20 June	SaL	MAT	87	0.17, 0.28 (mean: 0.23) [SK*]	RR 99-021B 01-NC-98-450 [Miller, 1999, PP5/0368]
Idem, plot 3	EC 240 (P) +0.25% v/v NIS	2 (5)	0.42 0.21	0.22 0.11	1–V5; 2–R1; 24 June	SaL	MAT	83	0.35, 0.35 (mean: 0.35) [SK*]	Idem

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Idem, plot 4	EC 240 (P) +0.25% v/v NIS	2 (21)	0.42 0.10	0.22 0.05 6	1–V5; 2–R3; 10 July	SaL	MAT	67	1.2, 1.0 (mean: 1.1) [SK*]	Idem
Idem, Plot 5	EC 240 (P) +0.25% v/v NIS A12460	2 (21)	0.42 0.21	0.22 0.11	1–V5; 2–R3; 10 July	SaL	MAT	67	1.3, 1.3 (mean: 1.3) [SK*]	Idem
Idem, plot 6	EC 240 (P) +0.25% v/v NIS	2 (26)	0.42 0.21	0.22 0.11	1–V5; 2-R4; 15 July	SaL	MAT	62	2.1, 1.9 (mean: 2.0) [SK*]	Idem
Idem, plot 7	EC 240 (P) +0.25% v/v NIS	2 (56)	0.42 0.21	0.22 0.11	1–V5; 2–R6; 14 August	SaL	MAT	32	1.2, 1.2 (mean: 1.2) [SK*]	Idem
Whitakers, NC, USA, 1998 (determinate Pioneer 9594[3]) Plot 9	EC 240 (P) +0.25% v/v NIS	2 (1)	0.42 0.21	0.22 0.11	1–V5; 2–V5; 20 June	SaL	MAT	128	0.03, 0.02 (mean: 0.03) [SK*]	RR 99-021B 01-NC-98-450 [Miller, 1999, PP5/0368]
Idem Plot 10	EC 240 (P) +0.25% v/v NIS	2 (26)	0.42 0.21	0.22 0.11	1–V5; 2-R1; 15 July	SaL	MAT	103	0.15, 0.14 (mean: 0.15) [SK*]	Idem
Idem Plot 11	EC 240 (P) +0.25% v/v NIS	2 (46)	0.42 0.10	0.22 5 0.05 6	1–V5; 2–R3; 4 August	SaL	MAT	83	0.30, 0.34 (mean: 0.32) [SK*]	Idem
Idem Plot 12	EC 240 (P) +0.25% v/v NIS A12460	2 (46)	0.42 0.21	0.22 0.11	1–V5; 2-R3; 4 August	SaL	MAT	83	0.53, 0.42 (mean: 0.48) [SK*]	Idem
Idem Plot 13	EC 240 (P) +0.25% v/v NIS	2 (54)	0.42 0.21	0.22 0.11	1–V5; 2-R4; 12 August	SaL	MAT	75	0.75, 0.81 (mean: 0.78) [SK*]	Idem
Idem Plot 14	EC 240 (P) +0.25% v/v NIS	2 (73)	0.42 0.21	0.22 0.11	1–V5; 2-R6; 31 August	SaL	MAT	56	1.30, 1.30 (mean: 1.3) [SK*]	Idem
Leland, MS, USA, 1998 (determinate Pioneer 9592[3]) Plot 2	EC 240 (P) +0.25% v/v NIS	2 (1)	0.42 0.21	0.24 0.12	1–V5; 2–V5; 18 June	SaL	MAT	98	< 0.01, < 0.01 (mean: < 0.01) [SK*]	RR 99-021B 05-MS-98-451 [Miller, 1999, PP5/0368]
Idem, Plot 3	EC 240 (P) +0.25% v/v NIS	2 (20)	0.42 0.21	0.24 0.08 6	1–V5; 2–R1; 7 July	SaL	MAT	79	0.01, 0.04 (mean: 0.02) [SK*]	Idem
Idem, Plot 4	EC 240 (P) +0.25% v/v NIS	2 (41)	0.42 0.10	0.24 0.05 1	1–V5; 2–R3; 28 July	SaL	MAT	58	0.39, 0.39 (mean: 0.39) [SK*]	Idem

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Idem, Plot 5	EC 240 (P) +0.25% v/v NIS A12460	2 (41)	0.42 0.21	0.24 0.10	1- V5; 2–R3; 28 July	SaL	MAT	58	0.98, 0.98 (mean: 0.98) [SK*]	Idem
Idem, Plot 6	EC 240 (P) +0.25% v/v NIS	2 (56)	0.42 0.21	0.24 0.098	1–V5; 2–R4; 12 August	SaL	MAT	43	0.18, 0.11 (mean: 0.15) [SK*]	Idem
Idem, Plot 7	EC 240 (P) +0.25% v/v NIS	2 (68)	0.42 0.21	0.24 0.102	1–V5; 2–R6; 24 August	SaL	MAT	31	0.99, 0.72 (mean: 0.84) [SK*]	Idem
Champaign, IL, USA, 1998 (indeterminate; Pioneer 9362) Plot 2	EC 240 (P) +0.25% v/v NIS	2 (9)	0.42 0.21	0.41 0.17	1–V5; 2–R1; 29 June	SaL	MAT	88	0.05, 0.08 (mean: 0.06) [SK*]	RR 99-021B 04-IL-98-452 [Miller, 1999, PP5/0368]
Idem, Plot 3	EC 240 (P) +0.25% v/v NIS	2 (13)	0.42 0.21	0.41 0.17	1–V5; 2–R1; 3 July	SaL	MAT	84	0.14, 0.16 (mean: 0.15) [SK*]	Idem
Idem, Plot 4	EC 240 (P +0.25% v/v NIS	2 (27)	0.42 0.10	0.41 0.087	1–V5; 2–R3; 17 July	SaL	MAT	70	0.45, 0.83 (mean: 0.64) [SK*]	Idem
Idem, Plot 5	EC 240 (P) +0.25% v/v NIS A12460	2 (27)	0.42 0.21	0.41 0.17	1- V5; 2–R3; 17 July	SaL	MAT	70	1.7, 1.9 (mean: 1.8) [SK*]	Idem
Idem, Plot 6	EC 240 (P) +0.25% v/v NIS	2 (32)	0.42 0.21	0.41 0.16	1- V5; 2–R4; 22 July	SaL	MAT	65	2.3, 2.2 (mean: 2.2) [SK*]	Idem
Idem, Plot 7	EC 240 (P) +0.25% v/v NIS	2 (51)	0.42 0.21	0.41 0.12	1–V5; 2–R6; 10 August	SaL	MAT	46	4.6, 5.1 (mean: 4.8) [SK*]	Idem
Champaign, IL, USA, 1998 (indeterminate soya bean; Pioneer 9362) Plot 2	EC 240 (P) +0.25% v/v NIS	2 (1)	0.42 0.21	0.35 0.17	1–V5; 2–V5; 2 July	SiCL	MAT	102	0.09, 0.06 (mean: 0.08) [SK*]	RR 99-021B 04-IL-98-453 [Miller, 1999, PP5/0368]
Idem, Plot 3	EC 240 (P) +0.25% v/v NIS	2 (13)	0.42 0.21	0.35 0.17	1–V5; 2-R1; 14 July	SiCL	MAT	90	0.37, 0.36 (mean: 0.36) [SK*]	Idem
Idem, Plot 4	EC 240 (P) +0.25% v/v NIS	2 (23)	0.42 0.10	0.35 0.075	1–V5; 2–R3; 24 July	SiCL	MAT	80	0.55, 0.50 (mean: 0.52) [SK*]	Idem
Idem, Plot 5	EC 240 (P)	2 (23)	0.42 0.21	0.35 0.15	1–V5; 2–R3;	SiCL	MAT	80	1.10, 1.10 (mean:	Idem

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	+0.25% v/v NIS A12460				24 July				1.1) [SK*]	
Idem, Plot 6	EC 240 (P) +0.25% v/v NIS	2 (30)	0.42 0.21	0.35 0.16	1–V5; 2–R4; 31 July	SiCL	MAT	73	1.80, 2.70 (mean: 2.3) [SK*]	Idem
Idem, Plot 7	EC 240 (P) +0.25% v/v NIS	2 (47)	0.42 0.21	0.35 0.19	1–V5; 2–R6; 17 August	SiCL	MAT	61	1.1, 1.5 (mean: 1.3) [SK*]	Idem
Eelko, SC, USA, 2000 (determinate soya bean; S73-Z5[3])	EC 240 (P) +COC1 %	2; (33)	0.43 0.11	0.24 0.060	1–V5; 2–R3; 27 July	LSa	MAT	104	0.06, 0.10 (mean: 0.08) [SK*]	RR 00-065B 231 (SYSC1) [Stewart, 2001, 406507]
West Memphis, AR, USA, 2000 (determinant soya bean; Pioneer 9492[3])	EC 240 (P) +NIS 0.5%	2; (39)	0.43 0.10	0.30 0.073	1–V5; 2–R3; 31 July	SiL	MAT	61	1.5, 1.7 (mean: 1.6) [SK*]	RR 00-065B 232 (SYAR1) [Stewart, 2001, 406507]
Walls, MS, USA, 2000 (determinant soya bean Pioneer 9541[3])	EC 240 (P) +NIS 0.5%	2; (44)	0.43 0.10	0.30 0.074	1–V5; 2–R3; 2 August	SaL	MAT	62	0.67, 0.58 (mean: 0.63) [SK*]	RR 00-065B 233 (SYMS1) [Stewart, 2001, 406507]
Webster City, IA, USA, 2000 (indeterminate soya bean; RC 2323[3])	EC 240 (P) +NIS 0.5%	2; (18)	0.42 0.10	0.50 0.12	1–V5; 2–R3; 11 July	SiCL	MAT	77	0.29, 0.56 (mean: 0.43) [SK*]	RR 00-065B 234 (SYIA1) [Stewart, 2001, 406507]
Wyoming, IL, USA, 2000 (indertermina te soya bean Asgrow A2833)	EC 240 (P) +NIS 0.5%	2; (27)	0.42 0.10	0.21 0.042	1–V5; 2–R3; 24 July	SiL	MAT	81	0.91, 1.00 (mean: 0.96) [SK*]	RR 00-065B 235 (SYIL1) [Stewart, 2001, 406507]
Carlyle, IL, USA, 2000 (indeterminate soya bean AG3901)	EC 240 (P) +NIS 0.5%	2; (27)	0.43 0.10	0.22 0.056	1–V5; 2–R3; 08 August	SiL	MAT	62	1.5, 1.4 (mean: 1.5) [SK*]	RR 00-065B 236 (SYIL2) [Stewart, 2001, 406507]
Noblesville, IN, USA, 2000 (indertermina te soya bean Pioneer 9363-Roundup Ready)	EC 240 (P) +NIS 0.5%	2; (20)	0.43 0.11	0.22 0.057	1–V5; 2–R3; 19 July	SiL	MAT	85	1.2, 1.2 (mean: 1.2) [SK*]	RR 00-065B 237 (SYIN1) [Stewart, 2001, 406507]

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
East Grand Forks, MN, USA, 2000 (indeterminate soya bean McCall)	EC 240 (P) +COC 1%	2; (18)	0.43 0.10	0.22 0.057	1–V5; 2–R3; 20 July	SiCL	MAT	61	1.4, 1.7 (mean: 1.6) [SK*]	RR 00-065B 238 (SYMN1) [Stewart, 2001, 406507]
Geneva, MN, USA, 2000 (indeterminate soya bean Asgrow 1901)	EC 240 (P) +NIS 0.5%	2; (13)	0.42 0.10	0.50 0.13	1–V5; 2–R3; 12 July	L	MAT	70	0.62, 0.64 (mean: 0.63) [SK*]	RR 00-065B 239SYMN2 [Stewart, 2001, 406507]
Campbell, MN, USA, 2000 (indeterminate soya bean Asgrow A0868)	EC 240 (P) +NIS 0.5%	2; (22)	0.42 0.11	0.22 0.057	1–V5; 2–R3; 25 July	SiL	MAT	56	0.59, 0.54 (mean: 0.57) [SK*]	RR 00-065B 240SYMN3 [Stewart, 2001, 406507]
Leonard, MO, USA, 2000 (indeterminate soya bean NK/3911)	EC 240 (P) +NIS 0.5%	2; (34)	0.42 0.11	0.25 0.055	1–V5; 2–R3; 10 July	SiL	MAT	79	1.0, 1.2 (mean: 1.1) [SK*]	RR 00-065B 241SYMO1 [Stewart, 2001, 406507]
Macon, MO, USA, 2000 (indeterminate soya bean Pioneer 94B01)	EC 240 (P) +NIS 0.5%	2; (25)	0.42 0.11	0.25 0.062	1–V5; 2–R3; 23 July	SiL	MAT	77	0.58, 0.62 (mean: 0.60) [SK*]	RR 00-065B 242SYMO2 [Stewart, 2001, 406507]
York, NE, USA, 2000 (indeterminate soya bean Dunbar)	EC 240 (P) +NIS 0.5%	2; (41)	0.42 0.10	0.23 0.057	1–V5; 2–R3; 25 July	SiL	MAT	57 57	1.8, 1.7 (mean: 1.8) [SK*]	RR 00-065B 243SYNE1 [Stewart, 2001, 406507]
Osceola, NE, USA, 2000 (indeterminate soya bean Midland 9A280RR)SaL	EC 240 (P) +NIS 0.5%	2; (40)	0.42 0.10	0.23 0.057	1–V5; 2–R3; 25 July	SaL	MAT	70	1.2, 1.2 (mean: 1.2) [SK*]	RR 00-065B 244SYNE2 [Stewart, 2001, 406507]
Northwood, ND, USA, 2000 (indeterminate soya bean Jims)	EC 240 (P) + COC 1%	2; (22)	0.44 0.11	0.23 0.057	1–V5; 2–R3; 25 July	SaL	MAT	64	1.6, 1.4 (mean: 1.5) [SK*]	RR 00-065B 245(SYND1 [Stewart, 2001, 406507]
New Holland, OH, USA, 2000 (indeterminate soya bean	EC 240 (P) +NIS 0.5%	2; (28)	0.41 0.11	0.51 0.12	1–V5; 2–R3; 21 July	SiL	MAT	75	1.3, 1.1 (mean: 1.2) [SK*]	RR 00-065B 246SYOH1 [Stewart, 2001, 406507]

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
SC9388RR)										
Delvan, WI, USA, 2000 (indeterminate soya bean Dyna-Gro 3256RR)	EC 240 (P) +NIS 0.5%	2; (28)	0.42 0.11	0.47 0.12	1–V5; 2–R3; 02 August	SiL	MAT	69	1.3, 1.5 (mean: 1.4) [SK*]	RR 00-065B 247SYWI1 [Stewart, 2001, 406507]
Leonard, MO, USA, 2000 (NK 3911)	EC 240 (P) +NIS 0.5%	2 (26)	0.86 0.21	0.51 0.11	1–V5; 2–R3 11 July	SiL	MAT	79	2.2 [b] [SK*]	RR 00-069B 251SYMO1 [Stewart, 2001, 406508] (processing)
Geneva, MN, USA, 2010 (Pioneer)	EC 240 (P) +NIS 0.5%	2 (24)	0.42 0.10	0.26 0.066	1–V4-V5; 2–R3 30 July	SaCL	IMM IMM MAT MAT MAT	60 64 70 77 84	1.7, 2.1 [b] (mean: 1.9) 2.0 2.1, 2.3 (mean: 2.2) 2.1 2.5 [SK*]	TK0016832 C09-0221 [Hampton and Mazlo, 2013, A12460_50026]
Northwood, ND, USA, 2010 (PFS 0905)	EC 240 (P) +COC 1.0%	2 (27)	0.42 0.11	0.13 0.032	1–V4-V5; 2–R3 21 July	SaCL	IMM IMM MAT MAT MAT MAT	60 75 82 82 89 94	0.57, 0.54 (mean: 0.55) 0.88; 0.87, 0.81; (mean: 0.84) 0.79, 0.84, 0.84 (mean: 0.82) 0.97; 0.98; [SK*]	TK0016832 C13-0222 [Hampton and Mazlo, 2013, A12460_50026] (processing)
Mordel, Canada, 1981, (Maple Presto)	EC 250 (rac) + adj 5863	1	0.30	0.27	growth stage not indicated; 21 June	ns	ns	91	< 0.01 [SS] [SK*]	PP009B229 Mordon-U4; [Atreya *et al*., 1983, PP9/0669]
Dundas, Canada, 1983; (Maple Amber)	EC 250 (rac)	1	0.50	0.23	2nd trifoliate stage; 25 July	ns	ns	73	0.43, 0.44 [c] [SS] [SK*]	PP009B265; CA/ON/HE/83/535/C [Atreya and Harradine; 1983, PP9/0726
idem	EC 250 (rac)	1	0.50	0.23	1st trifoliate; 14 July	ns	ns	87	0.10, 0.13 [c] [SS] [SK*]	idem
idem	EC 250 (rac)	1	0.50	0.23	1st trifoliate stage; 14 July	ns	ns	87	0.06, 0.10, 0.12 [c]	PP009B265; CA/ON/HE/83/536/C [Atreya and

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
									[SS], [CT] [Cntrl = 0.14]	Harradine; 1983, PP9/0726
Elova; Canada, 1983; (Maple Arrow)	EC 250 (rac)	1	0.50	0.22	2nd trifoliate; 7 July	ns	ns	91	0.12, 0.17 [SK*] [SS] [c]	PP009B265; CA/ON/HE/83/506/X [Atreya and Harradine; 1983, PP9/0726
idem	EC 250 (rac)	1	0.50	0.22	1st trifoliate; 28 June	ns	ns	100	0.09 [SK*] [SS] [c]	idem
Location unknown; Canada, 1983; (Maple Arrow)	EC 250 (rac)	1	0.50	0.22	2nd trifoliate; 25 June	ns	ns	108	< 0.05, < 0.05, 0.07 [SK*] [SS] [c]	PP009B265; 6050; [Atreya and Harradine; 1983, PP9/0726
Dawson; Canada, 1987; (Maple Amber)	EC 125 (P)	1	0.25	0.11	3 trifoliate; 18 cm high; 1 July	ns	ns	87	0.07; 0.08; 0.09; 0.15; mean 0.10 [a] [SK*] [SS]	M4010B; CA/ON/HE/84/514 C [Harradine, 1985; PP5/0408]
Branchton, ON; Canada, 2007; (RC18 Mirra)	EC 125 (P)	1	0.26	0.17	BBCH 13-14; 10-15 cm high; 6 June	SiL	CH	120	0.10, 0.11; mean 0.10 [SK*]	CER 02605/07; T229; [Sagan, 2008, A12791B_50003]
idem	EC 125 (P)	1	0.27	0.18	BBCH 13-14; 10-15 cm high; 6 June	SiL	CH	120	0.12, 0.18; mean 0.15 [SK*]	idem
idem	EC 125 (P)	1	0.073	0.049	BBCH 68-69; 55-60 cm high; 27 Jul	SiL	CH	69	1.4, 1.7; mean 1.6 [SK*]	idem
Branchton, ON, Canada, 2007; (RC 18 Mirra)	EC 125 (P)	1	0.25	0.17	BBCH 13-14; 7-10 cm high; 6 June	SiL	CH	119	< 0.01; 0.012 mean 0.010 [SK*]	CER 02605/07; T230 [Sagan, 2008, A12791B_50003]
idem	EC 125 (P)	1	0.26	0.17	BBCH 13-14; 7-10 cm high; 6 June	SiL	CH	119	0.017; 0.019 mean 0.018 [SK*]	idem
idem	EC 125 (P)	1	0.078	0.052	BBCH 67-68; 65-70 cm high; 27 Jul	SiL	CH	68	1.0, 1.2 mean 1.1 [SK*]	idem
St Marc sur Richelieu, QC, Canada, 2007;	EC 125 (P)	1	0.27	0.18	BBCH 13-14; 7-10 cm high; 15 June	CL	BBCH 99	108	< 0.01 (2) mean < 0.01 [SK*]	CER 02605/07; [Sagan, 2008, A12791B_50003]

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(PS 46 RR)										
idem	EC 125 (P)	1	0.27	0.18	BBCH 10-11; 5-10 cm high; 15 June	CL	BBCH 99	108	< 0.01, 0.010 mean < 0.01 [SK*]	idem
idem	EC 125 (P)	1	0.080	0.053	BBCH 67-69; 40-50 cm high; 23 July	CL	BBCH 99	70	0.97, 0.99 mean 0.98 [SK*]	idem
Ponto Grosso, Brazil, 1985; (Castolino)	EC 250 (rac)	1	0.38	0.15	12 trifoliates; 19 March	ns	MAT	73 ad 6	0.16 [SS] [SK*]	M4140B; PRS(84)R002 [Harradine, 1985, PP9/0120]
idem	EC 250 (rac)	1	0.75	0.30	12 trifoliates; 19 March	ns	MAT	73 ad 6	0.78 [SS] [SK*]	idem
idem	EC 250 (rac)	1	0.38	0.15	7 trifoliates; 26 Febr	ns	MAT	93 ad 6	0.07 [SS] [SK*]	idem
idem	EC 250 (rac)	1	0.75	0.30	7 trifoliates; 26 Febr	ns	MAT	93 ad 6	0.05 [SS] [SK*]	idem
Rio Grande, Brazil, 1985 (BC-4)	EC 250 (rac)	1	0.38	0.12	flowering; 22 Febr	ns	MAT	73 ad 10	0.15 [SS] [SK*]	M4140B; RS84R1 [Harradine, 1985, PP9/0120]
idem	EC 250 (rac)	1	0.75	0.25	flowering; 22 Febr	ns	MAT	73 ad 10	0.46 [SS] [SK*]	idem
idem	EC 250 (rac)	1	0.38	0.12	4-6 trifoliates; 28 Jan	ns	MAT	98 ad 10	< 0.03 [SS] [SK*]	idem
idem	EC 250 (rac)	1	0.75	0.25	4-6 trifoliates; 28 Jan	ns	MAT	98 ad 10	0.56 [SS] [SK*]	idem
Rib Preto, SP, Brazil, 1985; (IAC-8)	EC 250 (rac)	1	0.38	0.12	30% crop cover; 10 Febr	ns	MAT	60	1.4 [SK*]	M4140B SP(85)RSJ-01 [Harradine, 1985, PP9/0120
idem	EC 250 (rac)	1	0.75	0.25	30% crop cover; 10 Febr	ns	MAT	60	2.4 [SK*]	idem
idem	EC 250 (rac)	1	0.38	0.12	30% crop cover; 20 Jan	ns	MAT	80	NA	idem
idem	EC 250 (rac)	1	0.75	0.25	30% crop cover; 20 Jan	ns	MAT	80	0.15 [SK*]	idem
Rib Preto, SP, Brazil, 1985; (IAC-8)	EC 125 (P)	1	0.19	0.063	30% crop cover; 10 Febr	ns	MAT	60	0.56 [SK*]	M4141B SP(85)RSJ-01 [Harradine, 1985, PP5/0407
idem	EC 125 (P)	1	0.22	0.073	30% crop cover; 10 Febr	ns	MAT	60	1.7 [SK*]	idem
idem	EC 125 (P)	1	0.45	0.15	30% crop cover;	ns	MAT	60	1.8 [SK*]	idem

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					10 Febr					
idem	EC 125 (P)	1	0.19	0.063	30% crop cover 20 Jan	ns	MAT	80	0.16 [SK*]	idem
idem	EC 125 (P)	1	0.22	0.073	30% crop cover; 20 Jan	ns	MAT	80	0.12 [SK*]	idem
idem	EC 125 (P)	1	0.45	0.15	30% crop cover; 20 Jan	ns	MAT	80	0.19 [SK*]	idem
Gubird, SP, Brazil, 1985 (IAC-8)	EC 250 (rac)	1	0.38	0.12	30% crop cover; 4 Febr	ns	MAT	60	0.59 [SK*]	M4140B; SP(85)RSJ-03 [Harradine, 1985, PP9/0120]
idem	EC 250 (rac)	1	0.75	0.25	30% crop cover; 4 Febr	ns	MAT	60	1.0 [SK*]	idem
idem	EC 250 (rac)	1	0.38	0.12	30% crop cover; 15 Jan	ns	MAT	80	0.06 [SK*]	idem
idem	EC 250 (rac)	1	0.75	0.25	30% crop cover; 15 Jan	ns	MAT	80	0.10 [SK*]	idem
Gubird, SP, Brazil, 1985 (IAC-8)	EC 125 (P)	1	0.19	0.063	30% crop cover; 4 Febr	ns	MAT	60	0.25 [SK*]	M4141B; SP(85)RSJ-03 [Harradine, 1985, PP5/0407]
idem	EC 125 (P)	1	0.22	0.073	30% crop cover; 4 Febr	ns	MAT	60	0.75 [SK*]	idem
idem	EC 125 (P)	1	0.45	0.15	30% crop cover; 4 Febr	ns	MAT	60	0.95 [SK*]	idem
idem	EC 125 (P)	1	0.19	0.063	30% crop cover; 15 Jan	ns	MAT	80	0.09 [SK*]	idem
idem	EC 125 (P)	1	0.22	0.073	30% crop cover; 15 Jan	ns	MAT	80	0.06 [SK*]	idem
idem	EC 125 (P)	1	0.45	0.15	30% crop cover; 15 Jan	ns	MAT	80	0.10 [SK*]	idem
Grossa, PR, Brazil, 1985 (Bossier)	EC 250 (rac)	1	0.38	0.15	11 trifoliates; 19 March	ns	MAT	60 ad 3	0.68 [CT] [SK*] [cntrl=0.20]	M4140B; PRS(84)H001 [Harradine, 1985, PP9/0120;]
idem	EC 250 (rac)	1	0.75	0.30	11 trifoliates; 19 March	ns	MAT	60 ad 3	1.8 [SK*]	idem
Grossa, PR, Brazil, 1985 (Bossier)	EC 125 (P)	1	0.22	0.088	11 trifoliates 26 Febr	ns	MAT	60 ad 3	1.5 [SK*]	M4141B; PRS(84)H001 [Harradine, 1985, PP5/0407;]
idem	EC 125 (P)	1	0.45	0.18	11 trifoliates; 26 Febr	ns	MAT	60 ad 3	2.7 [SK*]	idem
Dourados,	EW 250	1	0.19	0.06	blooming;	C	ns	60	< 0.01	TECPAR

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
MS, Brazil; 1991; (Cobb)	(P)			3	22 Jan				[SK*]	81976/92; BR-12-91-S002-H [Bill and Kamienski, 1992, PP5/0411]
idem	EW 250 (P)	1	0.38	0.12	blooming; 22 Jan	C	ns	60	< 0.01 [SK*]	idem
Valinhos; Brazil; 1991; (Davis)	EC 200 (P)	1	0.20	0.067	stage R2; 14 Febr	ns	NH	60	< 0.01 [SK*]	TECPAR 81978/92; BR-12-91-S004-H [Bill and Kamienski, 1992, PP5/1027]
idem	EC 200 (P)	1	0.40	0.13	stage R2; 14 Febr	ns	NH	60	< 0.01 [SK*]	idem
idem	EC 200 (P) + energetic 0.2%	1	0.25	0.083	stage R2; 14 Febr	ns	NH	60	< 0.01 [SK*]	idem
idem	EC 200 (P) + energetic 0.2%	1	0.50	0.17	stage R2; 14 Febr	ns	NH	60	< 0.01 [SK*]	idem
Rolandia, PR, Brazil, 1991; (F1-5)	ME 200 (P)	1	0.20	0.067	68 days post-emergence; 23 Jan	C	MAT	60	< 0.01 [SK*]	TECPAR 81979/92; BR-10-91-S005-H [Bill and Kamienski, 1992, PP5/1072]
idem	ME 200 (P)	1	0.40	0.13	68 days post-emergence; 23 Jan	C	MAT	60	< 0.01 [SK*]	idem
idem	SL 125 (P)	1	0.25	0.083	68 days post-emergence; 23 Jan	C	MAT	60	< 0.01 [SK*]	idem
idem	SL 125 (P)	1	0.50	0.17	68 days post-emergence; 23 Jan	C	MAT	60	< 0.01 [SK*]	idem
Santa Amelia; PR; Brazil, 2005; (CD 206)	EC 128 (P)	1	0.25	0.12	BBCH 67; 24 Febr	C	BBCH 89	60	< 0.05 [AM] [SK*]	M04064; M04064-LZF [Baptista and Bahia, 2006, A13680A-10002]
idem	EC 128 (P)	1	0.50	0.25	BBCH 67; 24 Febr	C	BBCH 89	60	< 0.05 [AM] [SK*]	idem
Engenheiro Coelho; SP Brazil, 2011 (BRS Valiaosa PR)	EW 250 (P)	1	0.25	0.25	BBCH 69-71; 24 Febr	C	BBCH 89	60	0.49	M11032; M11032-AMA [Draetta, 2012, A12530B_10015]

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Bandeirantes, PR, Brazil, 2011 (NK 7059 PR)	EW 250 (P)	1	0.25	0.25	BBCH 78; 15 Febr	C	BBCH 97	60	1.7	M11032; M11032-DMO [Draetta, 2012, A12530B_10015]
Cabeceiras, GO, Brazil, 2011; (9070 RR)	EW 250 (P)	1	0.25	0.25	BBCH 70; 1 Febr	C	BBCH 97	60	1.2	M11032; M11032-MFG [Draetta, 2012, A12530B_10015]
Uberlandia, MG, Brazil, 2011; (Syn 9070-RR)	EW 250 (P)	1	0.25	0.25	BBCH 69; 3 Febr	C	BBCH 98	60	0.93	M11032; M11032-JJB [Draetta, 2012, A12530B_10015]
Minas Gerais, Brazil, 2013 (BRS Valiosa RR)	EC 125 (P) +0.2% NIS	1	0.25	0.12	BBCH 63 (R2-R4), 18 Febr	ns	BBCH 86-88	60	0.88 [SK*]	M13030; M13030-FSB [Matarazzo, 2013, A13680D_10051]
idem	EC 125 (P) +0.2% NIS	2 (10)	0.15 0.15	0.075 0.075	BBCH 63 (R2-R4), 18 Febr	ns	BBCH 86-88	60	0.39 [SK*]	Idem
Goias, Brazil, 2013 (Potência)	EC 125 (P) +0.2% NIS	1	0.25	0.12	BBCH 67 (R2-R4), 10 February	ns	BBCH 97	60	3.0 [SK*]	M13030; M13030-GBE [Matarazzo, 2013, A13680D_10051]
idem	EC 125 (P) +0.2% NIS	2 (10)	0.15 0.15	0.075 0.075	BBCH 67 (R2-R4), 10 February	ns	BBCH 97	60	2.2 [SK*]	idem
Parana, Brazil, 2013 (P98Y12)	EC 125 (P) +0.2% NIS	1	0.25	0.12	BBCH 74 (R2-R4), 18 February,	ns	BBCH 99	60	2.0 [SK*]	M13030; M13030-JJB1 [Matarazzo, 2013, A13680D_10051]
idem	EC 125 (P) +0.2% NIS	2 (1)	0.15 0.15	0.075 0.075	BBCH 74 (R2-R4), 18 February,	ns	BBCH 99	60	1.6 [SK*]	Idem
Sao Paulo, Brazil, 2013 (SD 820)	EC 125 (P) +0.2% NIS	1	0.25	0.125	BBCH 69 (R2-R4), 15 February,	ns	BBCH 96	60	0.96 [SK*]	M13030; M13030-JJB2 [Matarazzo, 2013, A13680D_10051]
idem	EC 125 (P) +0.2% NIS	2 (10)	0.15 0.15	0.075 0.075	BBCH 69 (R2-R4), February,	ns	BBCH 96	60	0.89 [SK*]	idem
Roche, VD Switzerland; 2004; (Amphor)	EC 125 (P)	1	0.24	0.083	BBCH 15; V5; 12 June	L	BBCH 89	96	0.04	03-7026; 03-7026; [Mason, 2004, PP5/1398]
idem	EC 125 (P)	1	0.24	0.083	BBCH 17-60; V7-R1; 17 June	L	BBCH 89	91	0.14	idem
71240 St Cyr; France	EC 125 (P)	1	0.25	0.084	BBCH 15; 16 June	L	BBCH 89	65	1.4 [b]	03-7072; AF/7297/SY/1 [Mason, 2004,

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North; 2003 (Prunto)										PP5/1396]
idem	EC 125 (P)	1	0.25	0.083	BBCH 59; 25 June	L	BBCH 89	56	2.1 [b]	idem
71590 Verjuy; France North; 2003; (Essor)	EC 125 (P)	1	0.24	0.083	BBCH 15; 6 June	SaC	BBCH 89	76	0.90 [b]	03-7073; AF/7298/SY/1 [Mason, 2004, PP5/1397]
idem	EC 125 (P)	1	0.24	0.083	BBCH 17; 19 June	SaC	BBCH 89	63	3.2 [b]	idem
71500 Nontcony; France North; 2003; (Sepia)	EC 125 (P)	1	0.24	0.083	BBCH 15; 11 June	L	BBCH 89	77	0.79 [b]	03-7074; AF/7299/SY/1; [Mason, 2004, PP5/1399]
idem	EC 125 (P)	1	0.26	0.083	BBCH 59; 20 June	L	BBCH 89	68	2.4 [b]	idem
Caldiero (VR), Veneto; Italy, 1996; (Pati)	EC 125 (P)	1	0.38	0.12	flowers at the top; 2 cm pods at the base; 10 tripeltate leaves; 15 July	L	Ripening	73	2.8	RJ2405B; IT10-96-R346 [Jones *et al*., 1997, PP5/0164]
Costanzana (VC); Piemonte; Italy, 1996; (Sapporo)	EC 125 (P)	1	0.38	0.12	pods at all plant levels; 29 July	L	CH	60	9.8	RJ2405B; IT10-96-R347 [Jones *et al*., 1997, PP5/0164]
Bondeno (FE); Emilia Romagna; Italy, 1996; (Sapporo)	EC 125 (P)	1	0.31	0.10	30-40 pods per plant; flowers at the top; 80-90 cm high; 25 July	C	CH	62	5.5 [SK*]	RJ2442B; IT10-96-R395 [Jones and Bonfanti, 1998, PP5/1024]
idem	EC 125 (P) + TF8035	1	0.31	0.10	30-40 pods per plant; flowers at the top; 80-90 cm high; 25 July	C	CH	62	8.3 [SK*]	idem
Caldiero (VR); Veneto; Italy, 1996; (Pati)	EC 125 (P)	1	0.31	0.10	flowers at the top; pods 2 cm long at the bottom; 10th trefoil	L	CH	73	2.8 [SK*]	RJ2442B; IT10-96-R396 [Jones and Bonfanti, 1998, PP5/1024]

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					leaf 100 cm high; 15 July					
idem	EC 125 (P) +TF 8035	1	0.31	0.10	flowers at the top; pods 2 cm long at the bottom; 10th trefoil leaf 100 cm high; 15 July	L	CH	73	4.0 [SK*]	idem
Filetto (RA); Emilia Romagna Italy, 1996; (Combir)	EC 125 (P)	1	0.31	0.10	flowering; no pods present; 50 cm high; 9 July	L	CH	73	1.0 [SK*]	RJ2442B; IT10-96-R397; [Jones and Bonfanti, 1998, PP5/1024]
idem	EC 125 (P) + TF8035	1	0.31	0.10	flowering; no pods present; 50 cm high; 9 July	L	CH	73	0.74 [SK*]	idem
Costanzana (VC); Piemonte Italy, 1996; (Sapporo)	EC 125 (P)	1	0.31	0.10	pods present; pods 1-2 cm at the top; pods 4-5 cm at the bottom 100 cm high; 29 July	L	CH	60	7.1 [SK*]	RJ2442B; IT10-96-398; [Jones and Bonfanti, 1998, PP5/1024]
idem	EC 125 (P) +TF8035	1	0.31	0.10	pods present; pods 1-2 cm at the top; pods 4-5 cm at the bottom 100 cm high; 29 July	L	CH	60	6.7 [SK*]	idem
Sette polesini; Emilia Romagna Italy, 1997; (indeterminate soya bean; Sapporo)	SL 125 (P) +0.5% TF8035	1	0.28	0.093	V4; 7 June	L	CH	102	< 0.01 [SK*]	RJ2720B; IT22-97-H352 [Jones *et al*., 1998, PP5/1026]
S Agata Bolognese,	SL 125 (P)	1	0.28	0.093	V3; 10-12 cm	L	CH	107	0.01 [SK*]	RJ2720B; IT22-97-H353

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Emilia Romagna; Italy, 1997; (intederminate soya bean; Sapporo)	+0.5% TF8035				high; 27 May					[Jones *et al*., 1998, PP5/1026]
Belfiore, Veneto; Italy, 1997; (indeterminate soya bean; Ardir)	SL 125 (P) +0.5% TF8035	1	0.28	0.093	V4; 25-30 cm high 9 June	LC	CH	109	0.02 [SK*]	RJ2720B; IT22-97-H354 [Jones *et al*., 1998, PP5/1026]
Filetto, Emilia Romagna; Italy, 1997; (indeterminate soya bean; Dawson)	SL 125 (P) +0.5% TF8035	1	0.28	0.093	V3; 15-20 cm; 16 June	L	CH	94	0.02 [SK*]	RJ2720B; IT22-97-H355 [Jones *et al*., 1998, PP5/1026]
Settepolesini; Emilia Romagna Italy, 1997; (Lory)	EC 125 (P)	1	0.31	0.078	V7-R1; 20 June	CSi	R8	89	1.1	RJ2481B; IT22-97-H340 [Mason and Volpi; 1998; PP5/0165]
idem	EC 125 (P)	1	0.31	0.078	R3; 60-70 cm high; 4 July	CSi	R8	75	4.0	idem
idem	EC 125 (P)	1	0.31	0.078	R4-R5; 70-80 cm high; 18 July	CSi	R8	61	4.7	idem
S Agata Bolognese; Emilia Romagna Italy, 1997; (Sapporo)	EC 125 (P)	1	0.31	0.078	R1; 35 cm high; 16 June	L	R8	87	0.99	RJ2481B; IT21-97-H341 [Mason and Volpi; 1998; PP5/0165]
idem	EC 125 (P)	1	0.31	0.078	R3; 60-70 cm high; 1 July	L	R8	72	4.2	idem
idem	EC 125 (P)	1	0.31	0.078	R4; 90 cm high; 16 July	L	R8	57	5.4	idem
Arcole, Veneto; Italy, 1997; (Queen)	EC 125 (P)	1	0.31	0.10	R1; 35-40 cm high; 16 June	L	CH	102	0.19	RJ2481B; IT29-97-H342 [Mason and Volpi; 1998; PP5/0165]
idem	EC 125 (P)	1	0.31	0.10	R2; 60 cm high; 30 June	L	CH	88	0.83	idem
idem	EC 125 (P)	1	0.31	0.10	R4; 110-115 cm high	L	CH	72	2.9	idem

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					16 July					
Filetto (RA); Emilia Romagna; Italy, 1997; (Dawson)	EC 125 (P)	1	0.31	010	R1; 25-30 cm high; 23 June	L	R8	87	0.43 [cntrl=0.10]	RJ2481B; IT23-97-H343 [Mason and Volpi; 1998; PP5/0165]
idem	EC 125 (P)	1	0.31	0.10	R2; 40 cm high; 7 July	L	R8	73	3.5 [cntrl=0.10]	idem
idem	EC 125 (P)	1	0.31	0.10	R4; 80 cm high; 22 July	L	R8	58	11 [cntrl=0.10]	idem
Diamantina; Emilia Romagna; Italy, 1998; (Lory)	EC 125 (P)	1	0.31	0.078	V6-V7; 24 June	L	MAT	90	0.14	RJ2781B; IT20-98-H314 [Mason *et al*., 1999, PP5/0159]
idem	EC 125 (P)	1	0.31	0.078	R2; 47 cm high; 11 July	L	MAT	73	2.7	idem
Lavezzola; Emilia Romagna; Italy, 1998 (Albir)	EC 125 (P)	1	0.31	0.089	R2; 45-50 cm high; 23 June	L	MAT	90	3.7	RJ2781B; IT20-98-H315 [Mason *et al*., 1999, PP5/0159]
idem	EC 125 (P)	1	0.31	0.089	R4; 60 cm high; 9 July	L	MAT	74	8.9	idem
San Bonifacio; Veneto; Italy, 1998; (Lory)	EC 125 (P)	1	0.31	0.10	R1; 50-60 cm high; 24 June	L	MAT	90	1.7	RJ2781B; IT20-98-H316 [Mason *et al*., 1999, PP5/0159]
idem	EC 125 (P)	1	0.31	0.10	R4; 90-100 cm high; 9 July	L	MAT	75	6.8	idem
Filetto; Emilia Romagna; Italy, 1998; (Nikir)	EC 125 (P)	1	0.31	0.089	R1; 35 cm high; 24 June	L	MAT	89	0.60	RJ2781B; IT20-98-H317 [Mason *et al*., 1999, PP5/0159]
idem	EC 125 (P)	1	0.31	0.089	R3; 50 cm high; 9 July	L	MAT	74	3.1	idem
idem	EC 125 (P)	1	0.31	0.089	R3; 50 cm high; 9 July	L	MAT	61 74	2.9 2.7 [ST], [SS]	RJ2914B; IT20-98-H319; [Mason and Volpi, 1998, PP5_50435] processing
Lavezzola,	EC 125	1	0.31	0.08	R4;	L	MAT	61	7.3	RJ2914B;

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Emilia Romagna, Italy, 1998, (Albir)	(P)			9	60 cm high; 9 July				[SS]	IT20-98-H318; [Mason and Volpi, 1998, PP5_50435]
Torri, Italy, 1999 (Nankino), plot 2	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	V4, 10 cm, 01 June	L	MAT	133	< 0.01	RJ3149B; IT20-99-H385 [Mason and Volpi, 2002, PP5/1144]
Idem, plot 3	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	R1, 30-35 cm, 15 June,	L	MAT	119	0.08	idem (processing)
Ferrera Erbogone, Italy, 1999 (Adel), plot 2	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	V3, 20-25 cm, 09 June,	SiSa	MAT	118	0.02	RJ3149B; IT20-99-H386 [Mason and Volpi, 2002, PP5/1144]
Idem, plot 3	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	R1, 45-50 cm, 29 June	SiSa	MAT	98	0.46	idem (processing)
Arcole, Italy, 1999 (Nikir), plot 2	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	V4, 14-15 cm, 01 June	L	MAT	135	< 0.01	RJ3149B; IT20-99-H387 [Mason and Volpi, 2002, PP5/1144]
Idem, plot3	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	R1, 30-35 cm, 16 June	L	MAT	120	0.08	idem (processing)
Filetto, Italy, 1999 (Nikir) plot 2	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	V3, 8-10 cm, 28 May	L	MAT	119	0.01	RJ3149B; IT20-99-H388 [Mason and Volpi, 2002, PP5/1144]
idem plot 3	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	R1, 30-40 cm, 15 June,	L	MAT	101	0.20	idem (processing)
Glorie di Mezzano (RA), Italy, 2000 (Mixer)	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	R1, 30 cm, 07 June,	ns	MAT	96 104 104	0.11 [b] 0.20 [bd], 0.21 [be]	RJ3208B; IT20-00-S356 [Mason and Giacomelli, 2001, PP5/1122] (processing)
Lavezzola (RA), Italy, 2000 (Albir)	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	R1, 40 cm, 07 June,	CL	MAT	96 103 103	0.79 [b] 0.62 [bd] 0.68 [be]	RJ3208B; IT20-00-S357 [Mason and Giacomelli, 2001, PP5/1122] (processing)

DRY SOYA SEEDS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Bando di Argenta (FE), Italy, 2000 (Lynda)	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	R1, 30 cm, 13 June,	L	MAT	90 98 98	0.24 [b] 0.56 [bd] 0.56 [be]	RJ3208B; IT20-00-S358 [Mason and Giacomelli, 2001, PP5/1122] (processing)
Filetto (RA), Italy , 2000 (Nikir)	EW 250 (P) + TF8035 mineral oil	1	0.25	0.083	R1, 30 cm, 09 June,	C	MAT	94	0.12	RJ3208B; IT20-00-S359 [Mason and Giacomelli, 2001, PP5/1122]
Banded foliar spray over rows										
Puymaurin, Gironde; S-France, 1996 (Imari)	EC 125 (P)	1	0.38	0.13	BBCH 75; 13 Aug	C	MAT	57	6.3	RJ2368B; 96H-SO-SA-P03 [Miles and Nassoy, 1997, PP5/0163]
St Pierre de Mons, Haut-Garonne, S-France, 1996 (Tiziana)	EC 125 (P)	1	0.38	0.13	BBCH 69; 14 Aug	SaC	MAT	56	6.7	RJ2368B; 96H-SO-SA-P04 [Miles and Nassoy, 1997, PP5/0163]

Soil type: F = Friable

Ad = air dried for indicated number of days after harvest

Fehr Caviness Growth Stages for soya beans: R1= one open flower at any node; R2=one open flower at one of the 2 upper nodes on the stem; R3 = beginning of pod growth; at least one pod 5 mm long at one of the four uppermost fully developed leaf nodes on the main stem; V5 = plants with 4 nodes (counting the unifoliate node) with fully developed trifoliate leaves. No blooms present a V5 stage. V7 = 7 nodes present

[ST] Sample was stored 16 days at ambient temperature before being frozen; result cannot be used for MRL derivation.

[SS] Sample size less than the required 1 kg seeds (RJ2914; 0.62–0.66 kg); samples are considered not representative for MRL derivation; Samples size was less than 1 kg (0.58-0.82 kg; report M4140B; 0.3–0.8 kg in report PP009B265) or sample size was not indicated in the report (trial Mordon-U4; report M4010B); samples are considered not representative for MRL setting.

[SK] The soaking step was omitted in the analytical method, whereby fluazifop (II) conjugates are incompletely extracted.

[AM] Samples were only analysed for free fluazifop acid (II) or extraction and hydrolysis conditions insufficient to determine total fluazifop residues; results cannot be selected for MRL setting

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

[a] Results came from 3-4 replicate samples taken from one plot. The mean is taken for MRL-derivation if according to cGAP.

[b] Results are the mean of two-three replicate analyses on the same sample.

[c] Samples are from replicate plots; highest value is taken for MRL derivation if according to cGAP.

[d] Whole soya bean analysed just before soya oil processing (storage for 2 months at ambient temperature)

[e] Whole soya bean analysed just before soya bean milk processing (storage for 2 months at ambient temperature)

[f] Residue in original subsample was 2.82 mg/kg. Re-analysis in duplicate afforded 2.47 and 2.24 mg/kg.

Additional trial information

RR 99-021B GLP. No unusual weather conditions. Tractor mounted or backpack broadcast over the crop canopy at the prebloom (V5) and post bloom (R3) growth stage; spray volume 20-280 L/ha. V5 = plants with 4 nodes (counting the unifoliate node) with fully developed trifoliate leaves. No blooms present a V5 stage. R3 = beginning of pod growth; at least one pod 5 mm long at one of the four uppermost fully developed leaf nodes on the main stem. For trial 04-IL-98-452, the second application to plot 2 at the V5 growth stage was missed. The make-up application occurred at ta later growth

stage than that required by protocol. Also the rate of the second application to plot 7 was on average 8% greater than than that required by protocol. Duplicate samples per plot were harvested at normal maturity (PHI 56 to 104 days). Sample sizes at least 1.13 kg from 12 areas from across the plot. Samples were stored at -20 °C 4.6 months before analysis. Samples were analysed for total fluazifop using **GC-MS method RR 91-014B with a valid LOQ of 0.01 mg/kg**. Internal standard recoveries (mean 95% for seeds). Control samples < 0.05 mg/kg

RR 00-065B GLP. No unusual weather conditions. Tractor mounted or backpack broadcast over the crop canopy at the prebloom (V5) and post bloom (R3) growth stage; spray volume 500 L/ha. V5 = plants with 4 nodes (counting the unifoliate node) with fully developed trifoliate leaves. No blooms present a V5 stage. R3 = beginning of pod growth; at least one pod 5 mm long at one of the four uppermost fully developed leaf nodes on the main stem. Duplicate samples per plot were harvested at normal maturity (PHI 56 to 104 days). Sample sizes at least 1.13 kg from 12 areas from across the plot. Duplicate samples were taken . Samples were stored at -20 °C 4.6 months before analysis. Samples were analysed for total fluazifop using **GC-MS method RR 91-014B with a valid LOQ of 0.01 mg/kg** Internal standard recoveries (mean 86.8% for seeds). Control samples < 0.01 mg/kg.

RR 00-069B GLP. No unusual wather conditions. Backpack sprayer. Spray volume 170-192 L/ha. Sample size 34-39 kg. Samples were kept <-12.2 °C until processed and below -11.7 °C until shipment. Samples wer stored frozen for max of 4 months. Samples were analysed for total fluazifop using **GC-MS method RR 91-014B with a valid LOQ of 0.01 mg/kg**. Concurrent recoveries ranged from 74.1-100%, mean 85%, SD 7.10% over various fortification levels (0.01-50 mg/kg). Results were not corrected for concurrent method recoveries. Control samples were < 0.01 mg/kg.

TK0016832 GLP. No unusual wather conditions. Backpack sprayer. Spray volume 159-327 L/ha. Sample sizes were 1.4-2.3 kg for the decline trials and 317 kg for the processing study. Samples were kept below -102 °C until shipment and below -20 °C for further storage for a maximum of 12.9 months. Samples were analysed for total fluazifop using **HPLC-MS/MS Method GRM044.01A with a valid LOQ of 0.01 mg/kg** was used. Concurrent recoveries ranged from 94.7-121%, mean 107% (seed), 121% (seed), 112% (aspirated grain), over various fortification levels (0.01-10). Results were not corrected for concurrent method recoveries. Control samples were < 0.01 mg/kg.

PP009B229, non-GLP. Weather conditions not indicated. Spray volume 112 L/ha. Information on sampling and sample size not indicated in the report. Samples were kept for a maximum of 411 days at unknown temperature. Samples were analysed for total fluazifop using **HPLC-UV Method PPRAM62/1 with a valid LOQ of 0.05 mg/kg**.Samples were corrected for internal std recoveries (92% seeds) Control samples were < 0.05 mg/kg.

PP009B265, non-GLP. Weather conditions not indicated. Spray volume 220-225 L/ha. Information on sampling not indicated in the report. Samples of seeds were less than 1 kg (300-800 g). Samples were kept for a maximum of 24 days at -20 °C. Samples were analysed for total fluazifop using **HPLC-UV Method PPRAM62/1 with a valid LOQ of 0.05 mg/kg**.Samples were corrected for internal std recoveries (78% seeds). Control samples were < 0.05 mg/kg, except 0.14 mg/kg in trial CA/ON/HE/83/536/C.

M4010B, non-GLP. Weather conditions not indicated. Spray volume 225 L/ha. Soybean plants were sampled by hand; sample size not indicated. Samples were kept frozen for a maximum of 239 days at -20 °C. Samples were analysed for total fluazifop using **HPLC-UV Method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Samples were corrected for internal std recoveries (77% seeds). Control samples were < 0.04 mg/kg.

CER2605-07 GLP. Weather conditions did not have an effect on the results. Boom sprayer with spray volume 150 L/ha. Soybean plants were collected from 12 separate areas in the plot. Soybean seeds were threshed by hand or bya portable threshing machine. Sample sizes were >1 kg for seeds, except where indicated. Samples T229-15 (seed), were 0.925 kg, respectively, which were below the 1 kg required. Samples were kept below -10 °C for a maximum of 236 days. Samples were analysed for total fluazifop using **HPLC-MS/MS Method CER2605 with a valid LOQ of 0.01 mg/kg**.Samples were not corrected for concurrent recoveries (84-106% seeds) Control samples were < 0.01 mg/kg.

M4140B and M4141B Non-GLP. No unusual wather conditions. Broadcast foliar spray using hand held boom, spray volume 250-300 L/ha. Soya bean seeds were sampled by hand or by machine. Sample sizes not stated, except in trial RS84R1 (0.50-0.86 kg seeds). Where indicated samples were air dried after harvest. Samples were kept below -20 °C. Storage period not stated but maximum 250 days (harvest to final report date). Samples were analysed for total fluazifop using **NMR Method PPRAM 83 with internal standard with a valid LOQ of 0.05 mg/kg**. Average internal standard recoveries were 75 % at 0.05 mg/kg. Control samples were < 0.03 mg/kg, except in trial PRS(84)H001 where 0.20 mg/kg was found.

TECPAR reports Non-GLP. No unusual wather conditions. Broadcast foliar spray using CO2 sprayer with boom, spray volume 300 L/ha. Soya plants were sampled by hand and seeds were threshed mechanically at the day of harvest. Seed samples > 1 kg. Samples were kept below -18 °C. Storage period not stated but maximum 328-344 days (harvest to final report date). Samples were analysed for total fluazifop using **HPLC-UV Method Yokomizo and Cavalho with a valid LOQ of 0.08 mg/kg**. Average internal standard recoveries were 78 %. Control samples were < 0.01 mg/kg.

M04064-GLP. No unusual wather conditions. Pressurized CO2 boom sprayer. Spray volume 200 L/ha. Sample sizes were >1 kg seeds. Samples were kept at -18 °C for a maximum of 333 days. Samples were analysed for free fluazifop acid using **GC-MS Method IT125 with a valid LOQ of 0.05 mg/kg** (Fluazifop conjugates not taken into account). Samples were not corrected for concurrent method recoveries (77-805). Control samples were < 0.05 mg/kg.

M11032 GLP. No unusual wather conditions. Pressurised CO2 sprayers with spray volume 100 L/ha. Samples were collected from at least 12 representative points in the plot and the collected plants were threshed by hitting with a wooden stick. Sample sizes were >1 kg seeds. Samples were kept below -20 °C for a maximum of 332 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET.138.Rev04 with a valid LOQ of 0.01 mg/kg**.

Results were not corrected for average concurrent method recoveries (85-106 % at 0.01, 0.1 and 2.0 mg/kg). Control samples were < 0.01 mg/kg.

M13030 GLP. No unusual wather conditions. Plot size 30-75 m^2.Teejet spray. Spray volume 200 L/ha. Sample sizes were 1- 24 kg. Samples were kept below -20 °C during storage for a maximum of 5.3 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET.138.Rev08 with a valid LOQ of 0.01 mg/kg**. Concurrent recoveries ranged from 71-110 % (n = 5-7/fortification level) over fortification levels 0.01, 0.1 and 2.0 mg/kg. Results were not corrected for concurrent method recoveries. Control samples were < 0.01 mg/kg.

03-7026 GLP. No unusual weather conditions. Broadcast foliar application with knapsack sprayers with boom, spray volume 280-290 L/ha.Samples were collected using a plot combine. Samples of >1 kg seeds were taken. Storage time 140 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recoveries (76-85% at 1.0-4.0 mg/kg). Control samples were < 0.01 mg/kg.

03-7072; 0.3-7073, 03-7074 GLP. No unusual weather conditions. Application by small plot sprayers, spray volume 300 L/ha.Whole plants were cut 15 cm above ground level and threshed into seed using a static ear thresher. Samples of >1 kg seeds were taken. Storage time 141-175 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recoveries (95-108% at 1.0-4.0 mg/kg). Control samples were < 0.01 mg/kg.

RJ2405B GLP. Weather conditions had no effect on the growth of the crop. Application by knapsack motor sprayers with boom, spray volume 300 L/ha. Mature soya bean pods were sampled by hand taking care to avoid the plot boundaries. Pods were shelled by hand and > 1 kg) seeds were obtained. Storage time 346-347 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (83-91% at 0.5-0.5 mg/kg). Control samples were < 0.01 mg/kg.

RJ2442B GLP. Weather conditions caused a delay in maturity. The PHI was lengthened to 75 days in trials R396 and R397, while the application in trials R395 and R398 was at a later stage (pods up to 5 cm long) to be able to obtain the PHI of 60 days as intended in the protocol. Application by knapsack motor sprayers with boom, spray volume 300 L/ha. Mature soya bean pods were sampled by hand taking care to avoid the plot boundaries. Pods were shelled by hand and > 1 kg) seeds were obtained. Storage time 5 months at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Results were corrected for concurrent method recoveries (98% at 0.1 mg/kg); Uncorrected results were not stated. Control samples were < 0.05 mg/kg.

RJ2720B GLP. Weather conditions had no effect on the growth of the crop. Application equipment was not stated; Spray volume 300 L/ha. Mature soya bean pods were sampled by hand taking care to avoid the plot boundaries. Pods were shelled mechanically using a threshing machine and > 1 kg) seeds were obtained. Storage time up 45-60 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for average concurrent method recoveries (91-115% at 0.05-0.5 mg/kg). Control samples were < 0.01 mg/kg.

RJ2481B GLP. No unusual weather conditions. Application by knapsack motor sprayers with boom, spray volume 300-400 L/ha. Mature soya bean pods were sampled by hand taking care to avoid the plot boundaries. Pods were shelled using a threshing machine and > 1 kg) seeds were obtained. Storage time 37-111 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. The extraction solvent was enriched with acetonitrile (67:33 v/v) in the analysis of samples from trials H342 and H343 as a co-extractant was preventing the acetonitrile and acid solutions from mixing fully. Results were not corrected for average concurrent method recoveries (107-119% at 0.05-0.5 mg/kg; 130% at 2.0 mg/kg). The high recovery is caused by the presence of a co-extractant in samples from trials H342 and H343. Control samples were < 0.05 mg/kg, except in trial IT23-97-H343 (0.10 mg/kg).

RJ2781B-GLP Weather caused early ripening of the crop in trial H315. Broadcast foliar spray. Application by motor knapsack sprayers with boom, spray volume 300-400 L/ha. Mature soya bean pods were sampled by hand taking care to avoid the plot boundaries. The pods were threshed by hand or by threshing machine. Samples were > 1 kg seeds. Storage time 43-44 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (108-116% at 0.01-5.0 mg/kg. Control samples were < 0.01 mg/kg.

RJ2914B-GLP Weather caused early ripening of the crop in trial H318 and gave a low yield. Broadcast foliar spray. Application by motor knapsack sprayers with boom, spray volume 300 L/ha. Mature soya bean pods were sampled from across the plots by hand taking care to avoid the plot boundaries. The pods were shelled by hand. Samples were 0.62–0.66 kg seeds. Storage time 2 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (100% at 0.01-5.0 mg/kg. Control samples were < 0.01 mg/kg.

RJ3149B GLP. No unusual weather conditions. Application by knapsack sprayers, spray volume 300 L/ha. Mature soya bean pods (for hand shelling) and mature soybean plants (for mechanical threshing) were sampled by hand, taking care to avoid the plot boundaries. Samples of 1-1.4 kg seeds (analysis) and 12-18 kg (processing) were taken systematically from across the plots. Storage time 16 months at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 a with a valid LOQ of 0.01 mg/kg**. The uncorrected results are tabulated above. Concurrent method recoveries (74-118% at 0.01-1.0 mg/kg for different foritication levels/matrices, n = 1-4). Mean method validation recoveries ranged from 72-107%, RSD 6-17%, n = 5/fortification level Control samples were < 0.01 mg/kg (=LOQ).

RJ3208B GLP. No unusual weather conditions. One application of 250 g ai/ha, by knapsack sprayers, spray volume 300 L/ha at growth stage R1. Mature soya bean pods (for hand shelling) and mature soybean plants (for mechanical threshing) were sampled by hand, taking care to avoid the plot boundaries. Samples were taken at PHI 90-96 to confirm presence of residues. Samples for processing were harvested at PHI 98-104 days. Samples of >35.6-38.8 kg seeds (processing) were taken systematically from across the plots. Storage time 16 months at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Corrected and uncorrected results were reported. The uncorrected results are tabulated above. Concurrent method recoveries (74-118% at 0.01-1.0 mg/kg for different foritication levels/matrices, n = 1-4). Mean method validation recoveries ranged from 72-107%, RSD 6-17%, n = 5/fortification level Control samples were < 0.01 mg/kg (=LOQ).

RJ2368B. GLP. No unusual weather conditions. **Banded spray over rows** using a hand-held boom sprayer, spray volume 284-293 L/ha. Mature samples were collected manually. Samples of >1 kg dry seeds were taken. Storage time 105 days at -18 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average oncurrent recoveries (99% at 0.1 mg/kg). Control samples were < 0.05 mg/kg.

Root and tuber vegetables

Carrots

Four cGAPs for carrots are available:

- cGAP from the USA with 2 ×0.42 kg ai/ha with a PHI 45 days (underlining nn)
- cGAP from Brazil with 1 ×0.25 kg ai/ha with a PHI of 30 days (underlining nn)
- cGAP from France with 1 ×0.38 kg ai/ha with a PHI of 42 days (underlining nn or nn with PHI)
- cGAP from Belgium, the UK and the Netherlands with 1 ×0.38 kg ai/ha a PHI of 56 days (underlining nn, one value overlaps with French GAP)

Study reports with trials that could be matched to these cGAPs were summarized.

Table 221 lists trials conducted in the USA (1983-1984, 1986-1987, 2008), Brazil (2011), the UK (1984, 1989, 1994), France (1983, 1997, 1999), Italy (1998) and Spain (1997, 1998). A broadcast or banded foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 221. Results marked with "[SS]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[SS] indicates that the sample sizes were not reported or less then 12 roots (or less than 2 kg).

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62/2.

Additional trials from the Netherlands (1981) were available on carrots with 1 ×0.50 or 0.75 kg ai/ha and harvest at 51 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Sweden (1981) were available with 2 × 0.50 kg ai/ha and harvest at 26 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from the UK (1980) were available with 1 ×1.0 kg ai/ha and harvest at 98 and 105 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B, PP9/0384]. Additional trials from Canada (1980) were available with 1 ×0.25 kg ai/ha or 1 ×0.50 kg ai/ha and harvest at 51 and 121 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B, PP9/0384]. Additional trials from the USA (1981, 1982) were available with 2-3 × 0.28 kg ai/ha or 2–3 × 0.56 kg ai/ha or 2 × 1.1 kg ai/ha with harvest at 19–36 or 52–72 DAT [Koubek, 1982, 06305, TMU0902/B; Koubek, 1983, 406307, TMU1182/B]. These trials were not summarized, because they would not assist in MRL setting.

Besides total fluazifop, also despyridinyl acid (III) was analysed in carrot root samples from some 1981 and 1982 trials conducted in the USA [Atreya, 1984, PP9/0728, PP009B272; Atreya *et al*., 1984, PP9/0065, PP009B300]. These trials were summarized in the metabolism section.

Besides total fluazifop, also CF3-pyridone (X) was analysed in carrot roots from some 1981, 1982 and 1984 trials conducted in the USA [Atreya, 1984, PP9/0731, PP009B290; Dick and Rounds, 1985, PP5/0238, M4041B; Atreya *et al*., 1984, PP9/0065, PP009B300]. These trials were summarized in the metabolism section.

Table 221 Supervised field trials on carrots (roots), treated with a broadcast or banded foliar fluazifop-butyl spray

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray application										
Belle Glade, FL, USA, 1982 (Scarlet Nantes)	EC120 (rac) + COC	2 (17)	0.56	ns	GS ns; 12 April	muck	ns	24 44	< 0.05 ≤ 0.05	TMU0902/B; 53FL82-029 [Koubek, 1982, 406305] and PP009B300; 53FL82-029; [Atreya *et al*., 1984, PP9/0065]
Zellwood, FL, USA, 1983 (PAK-MOR)	480 EC (rac) + COC	2 (11)	0.56	0.17	GS ns; 25 March	ns	ns	45	≤ 0.04 [LOQ=0.05]	TMU1231/B; 53FL83-067 [Koubek, 1983, 406309]
Lake Jem, Florida, USA, 1983 (Hi-color 9)	480 EC (rac) + COC	2 (28)	0.28	0.083	GS ns; 11 April	ns	ns	45	< 0.04 [LOQ=0.05]	TMU1231/B; 53FL83-070 [Koubek, 1983, 406309]
Belle Glade, Florida, USA, 1983 (Scarlet Nantes)	480 EC (rac) + COC	2 (7)	0.28	0.1	GS ns; 04 April, 1983	ns	ns	45	< 0.04 [LOQ=0.05]	TMU1231/B; 53FL83-083 [Koubek, 1983, 406309]
idem	idem	2 (7)	0.56	0.2	GS ns; 4 April	ns	ns	45	≤ 0.04 [LOQ=0.05]	idem
Center Point, TX, USA, 1983 (Chanteney Red Core)	480 EC (rac) + COC	2 (71)	0.28	0.2	GS ns; 30 March	ns	ns 20 April, 1983	21	0.15	TMU1231/B; 60TX82-099 [Koubek, 1983, 406309]
idem	idem	2 (71)	0.56	0.4	GS ns; 30 March	ns	ns	21	0.27	idem
Portage, WI, USA, 1983 (Gold King)	480 EC (rac) + COC	2 (ns)	0.56	0.3	GS ns; 22 July	ns	ns	38	0.05	TMU1231/B; 49WI83-068 [Koubek, 1983, 406309]
Santa Rosa, TX, USA, 1983-1984 (Long imperator 58)	EC 240 (P) + 1% Agridex COC	2 (53) [c]	0.42	0.19	GS ns, 27 Jan 1984	ns	ns	31 45	0.09 0.05 [SS] [CT] [cntrl=0.07]	TMU1812B; 71TX83-044 [Francis, 1985, 406311]
idem	120 EC (P) + 1% Agridex COC	2 (53) [c]	0.42	0.19	GS ns, 27 Jan	ns	ns	31 45	0.06 0.04 [SS] [CT] [cntrl=0.07]	idem
idem	EC 240	2	0.56	0.25	GS ns,	ns	ns	31	0.16	idem

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	(P) + 1% Agridex COC	(53) [c]			27 Jan			45	0.05 [SS] [CT] [cntrl=0.07]	
idem	120 EC (P) + 1% Agridex COC	2 (53) [c]	0.56	0.25	GS ns, 27 Jan	ns	ns	31 45	0.12 0.05 [SS] [CT] [cntrl=0.07]	idem
idem	480 EC (rac) + 1% Agridex COC	2 (53) [c]	0.56	0.25	GS ns, 27 Jan	ns	ns	31 45	0.12 0.05 [SS] [CT] [cntrl=0.07]	idem
Mission, TX, USA, 1984 (Long imperator 58)	120 EC (P) + 1% Agridex COC	2 (15) [c]	0.42	0.19	GS ns, 14 March	ns	ns	30 45	0.12 0.04 [LOQ=0.05]	TMU1812B; 71TX83-055; [Francis, 1985, 406311]
idem	EC 240 (P) + 1% Agridex COC	2 (15) [c]	0.42	0.19	GS ns, 14 March	ns	ns	30 45	0.13 0.04 [LOQ=0.05]	idem
idem	120 EC (P) + 1% Agridex COC	2 (15) [c]	0.56	0.25	GS ns, 14 March	ns	ns	30 45	0.16 0.06	idem
idem	EC 240 (P) + 1% Agridex COC	2 (15) [c]	0.56	0.25	GS ns, 14 March	ns	ns	30 45	0.18 0.06	idem
idem	480 EC (rac) + 1% Agridex COC	2 (15) [c]	0.56	0.25	GS ns, 14 March	ns	ns	30 45	0.09 0.06	idem
South Bay, FL, USA, 1984 (Nantes)	120 EC (P) + 1% Nalcotrol	2 (15) [c]	0.42	0.11	GS ns, 23 February	ns	ns	29 44	0.08 ≤ 0.03 [LOQ=0.05]	TMU1812B; 75FL84-004; [Francis, 1985, 406311]
idem	120 EC (P) + 1% Nalcotrol	2 (15) [c]	0.56	0.15	GS ns, 23 February	ns	ns	29 44	0.07 < 0.03 [LOQ=0.05]	idem
idem	480 EC (rac) + 1% Nalcotrol	2 (15) [c]	0.56	0.15	GS ns, 23 February	ns	ns	29 44	< 0.03 < 0.03 [LOQ=0.05]	idem
Zellwood, FL, USA, 1984 (variety not specified)	120 EC (P) + 0.25% Induce	2 (17)	0.42	0.11	GS ns, 31 March	ns	ns	31 48	0.08 ≤ 0.03 [LOQ=0.05]	TMU1812B; 75FL84-032; [Francis, 1985, 406311]
idem	120 EC (P) + 0.25%	2 (17)	0.56	0.13	GS ns, 31 March	ns	ns	31 48	0.11 < 0.03	idem

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	Induce								[LOQ=0.05]	
idem	480 EC (rac) + 0.25% Induce	2 (17)	0.56	0.13	GS ns, 31 March	ns	ns	31 48	0.06 < 0.03 [LOQ=0.05]	idem
Scottsdale, AZ, USA, 1983-84 (Dominator)	120 EC (P) + 1% Moract COC	2 (75)	0.84	0.37	GS ns; 16 Jan 1984	ns	ns	30	< 0.03 [LOQ=0.05]	TMU1812B; 38AZ83-047; [Francis, 1985, 406311]
idem	EC 240 (P) + 1% Moract COC	2 (75)	0.84	0.37	GS ns; 16 Jan 1984	ns	ns	30	< 0.03 [LOQ=0.05]	idem
idem	120 EC (P) + 1% Moract COC	2 (75)	1.1	0.49	GS ns; 16 Jan 1984	ns	ns	30	< 0.03 [LOQ=0.05]	idem
idem	EC 240 (P) + 1% Moract COC	2 (75)	1.1	0.49	GS ns; 16 Jan 1984	ns	ns	30	< 0.03 [LOQ=0.05]	idem
idem	480 EC (rac) + 1% Moract COC	2 (75)	1.1	0.49	GS ns; 16 Jan 1984	ns	ns	30	< 0.03 [LOQ=0.05]	idem
Uvalde, TX, USA, 1983-84 (Imperator 58)	EC 240 (P) + 0.25% AG98 NIS	2 (44)	0.42	0.22	GS ns; 8 Dec 1983	ns	ns	30	1.0 [SS]	TMU1812B; 71TX83-041; [Francis, 1985, 406311]
idem	EC 240 (P) + 0.25% AG98 NIS	2 (44)	0.56	0.45	GS ns; 8 Dec 1983	ns	ns	30	0.71 [SS]	idem
idem	480 EC (rac) + 0.25% AG98 NIS	2 (44)	0.56	0.45	GS ns; 8 Dec 1983	ns	ns	30	1.5 [SS]	idem
Uvalde, TX, USA, 1986 (Imperator 58)	120 EC (P) + COC	2 (15)	0.42	0.26	GS ns; 22 Nov. 1986	ns	ns 22 Dec, 1986	31	0.5	RSR-027-87/C; 6OTX86-431R; [Roper and Francis, 1987, 430705]
Uvalde, TX, USA, 1986-87 (PAK-MOR)	120 EC (P) + COC	2 (15)	0.42	0.26	GS ns; 02 Dec, 1986	ns	ns 01 Jan, 1987	31	0.6	RSR-027-87/C; 6OTX86-432R [Roper and Francis, 1987, 430705]
Uvalde, Texas, USA, 1986-87 (PAK-MOR)	120 EC (P) + COC	2 (15)	0.42	0.26	GS ns; 07 Dec. 1986	ns	ns 06 Jan, 1987	31	0.5	RSR-027-87/C; 6OTX86-433R [Roper and Francis, 1987, 430705]
Uvalde, TX,	120 EC	2	0.42	0.26	GS ns;	ns	ns	31	1.0	RSR-027-87/C;

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
USA, 1986-87 (Imperator 58)	(P) + COC	(15)			22 Dec, 1986		21 Jan, 1987			6OTX86-434R [Roper and Francis, 1987, 430705]
Uvalde, TX, USA, 1987 (Six pence)	120 EC (P) + COC	2 (15)	0.42	0.26	GS ns; 28 Feb, 1987	ns	ns 30 March, 1987	31	0.7	RSR-027-87/C; 6OTX86-435R [Roper and Francis, 1987, 430705]
Fresno, CA, USA, 2008 (Vitana)	EC 240 (P) + COC	2 (14)	0.42	0.11 – 0.90	BBCH43; 8 July	SL	ns	45	0.027, 0.019 [a]	T002222-07; W30CA 081301; [Mazlo, 2009, PP5_50071]
Madera, CA, USA, 2008 (Danvers Half Long 12 G)	EC 240 (P) + NIS	2 (14)	0.42	0.11 – 0.90	13BBCH; 15 May [b]	LS	ns	46	< 0.01, < 0.01 [a]	T002222-07; W29CA 081302; [Mazlo, 2009, PP5_50071]
Madera, CA, USA, 2008 (Vitana F1)	EC 240 (P) + COC	2 (14)	0.42	0.11 – 0.90	BBCH43; 14 July	SL	ns	45	0.017, 0.019 [a]	T002222-07; W30CA081303; [Mazlo, 2009, PP5_50071]
Ephrata, WA, USA, 2008 (Danvers 126)	EC 240 (P) + NIS	2 (14)	0.42	0.11 – 0.90	BBCH43; 25 July	SL	ns	45	0.072, 0.072 [a]	T002222-07; W18WA081304; [Mazlo, 2009, PP5_50071]
Engenheiro Coehlo, Sao Paulo, Brazil, 2011 (Brasília)	EW 250, (P)	1	0.25	0.25	BBCH 41-42; 17 August	C	BBCH 47-48	30	0.05	M11030; AMA1; [Draetta, 2012, A12530B_10012]
Piedade, Sao Paulo, Brazil, 2011 (Juliana)	EW 250 (P)	1	0.25	0.25	BBCH46; 1 April	C	BBCH 49	30	0.04	M11030; AMA2; [Draetta, 2012, A12530B_10012]
Espírito Santo do Dourado, Minas Gerais, Brazil, 2011 (Nayarit)	EW 250 (P)	1	0.25	0.25	BBCH42; 29 April	C	BBCH 49	30	0.17	M11030; JJB; [Draetta, 2012, A12530B_10012]
Iraí de Minas, Minas Gerais, Brazil, 2011 (Nanci)	EW 250 (P)	1	0.25	0.25	BBCH40; 24 May	C	BBCH 48	30	0.04	M11030; RWC; [Draetta, 2012, A12530B_10012]
Tatershall, Lincolnshire; UK; 1984; (Nandor)	EC 125 (P)	1	0.38	0.14	10-18 cm roots; 21 June	Sa	ns	56	< 0.03 [LOQ=0.05]	M3954B; 6R/84; [Harradine, 1985, PP5/0084]
Chattens, Cambridgeshire; UK; 1984; (Chantency)	EC 125 (P)	1	0.38	0.19	40% crop cover; 15 June	LC	CH	60	< 0.03 [LOQ=0.05]	M3954B; 6R/84; [Harradine, 1985, PP5/0084]
Mance, Cambridgeshire UK; 1984; (Chantency)	EC 125 (P)	1	0.38	0.19	40% crop cover; 20 July	LC	CH	56	< 0.03 [LOQ=0.05]	M3954B; 6R/84; [Harradine, 1985, PP5/0084]

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
ns; UK; 1984; ns	EC 125 (P)	1	0.38	0.19	50% crop cover; 11 July	peat	plant 46 cm tall	64	< 0.03 [LOQ=0.05]	M3954B; 6R/84; [Harradine, 1985, PP5/0084]
Hockwold; UK; 1984; (Chantonay Long)	EC 125 (P)	1	0.38	0.15	no roots; 20% crop cover; 19 June	Sa	ns	57	< 0.03 [LOQ=0.05]	M3954B; 6R/84E/A63; [Harradine, 1985, PP5/0084]
idem	EC 125 (P)	1	0.38	0.15	roots just forming; 25% crop cover; 26 June	Sa	roots 20 cm long	56	< 0.03 [LOQ=0.05]	M3954B; 6R/84E/A64; [Harradine, 1985, PP5/0084]
idem	EC 125 (P)	1	0.38	0.19	roots pencil thick; 50% crop cover; 6 July	Sa	roots 5 cm thick	56	< 0.03 [LOQ=0.05]	M3954B; 6R/84E/A65; [Harradine, 1985, PP5/0084]
Misterton, Lincolnshire; UK; 1984; (Toudo)	EC 125 (P)	1	0.38	0.19	2-7 cm roots; 26 June	Sa	ns	56	< 0.03 [LOQ=0.05]	M3954B; 6R/84/6R; [Harradine, 1985, PP5/0084]
Thorney, Lincolnshire; UK; 1989; (Nelson)	EW 125 (P) + Agral	1	0.38	ns	4-6 leaves; 8-12 cm roots; 65% crop cover; 12 June	LSa	CH	53	0.21	M5317B; GB12-89-S141; [Cullen, 1991, PP5/0085]
idem	EW 250 (P) + Agral	1	0.38	ns	4-6 leaves; 8-12 cm roots; 65% crop cover; 12 June	LSa	CH	53	0.13	idem
idem	EC 125 (P) + Agral	1	0.38	ns	4-6 leaves; 8-12 cm roots; 65% crop cover; 12 June	LSa	CH	53	0.15	idem
Wigsley, Lincolnshire; UK; 1989; (Nelson)	EW 125 (P) + Agral	1	0.38	ns	4-6 leaves; 12-16 cm roots; 55% crop cover; 12 June	LSa	CH	42	0.26	M5317B; GB12-89-S142; [Cullen, 1991, PP5/0085]
idem	EW 250 (P) + Agral	1	0.38	ns	4-6 leaves; 12-16 cm roots; 55% crop cover; 12 June	LSa	CH	42	0.29	idem
idem	EC 125 (P)	1	0.38	ns	4-6 leaves;	LSa	CH	42	0.23	idem

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	+ Agral				12-16 cm roots; 55% crop cover; 12 June					
Carlton-LE-Moorland, Lincolnshire; UK; 1994; (Nirobi)	EC 125 (P) + Agral	1	0.38	ns	crop height 21-38 cm; 85% crop cover; 5 July	SaL	roots 11-12 cm long	59	< 0.05	RJ1884B; GB11-94-S171; [Patel *et al.*, 1995, PP5/0103]
idem	EC 125 (P) + Output	1	0.38	ns	crop height 21-38 cm; 85% crop cover; 5 July	SaL	roots 11-12 cm long	59	0.05	idem
idem	EW 250 (P) + Agral	1	0.38	ns	crop height 21-38 cm; 85% crop cover; 5 July	SaL	roots 11-12 cm long	59	0.05	idem
idem	EW 250 (P) + Output	1	0.38	ns	crop height 21-38 cm; 85% crop cover; 5 July	SaL	roots 11-12 cm long	59	0.08	idem
idem	EW 250 (P) +TF8035	1	0.38	ns	crop height 21-38 cm; 85% crop cover; 5 July	SaL	roots 11-12 cm long	59	0.09	idem
Upton, Lincolnshire; UK; 1994; (Primo)	EC 125 (P) + Agral	1	0.38	ns	crop height 33-43 cm; 65% crop cover; 10 June	SaL	roots 12-21 cm	56	0.08	RJ1884B; GB11-94-S172; [Patel *et al.*, 1995, PP5/0103]
idem	EC 125 (P) + Output	1	0.38	ns	crop height 33-43 cm; 65% crop cover; 10 June	SaL	roots 12-21 cm	56	0.15	idem
idem	EW 250 (P) + Agral	1	0.38	ns	crop height 33-43 cm; 65% crop cover;	SaL	roots 20-21 cm	56	0.14	idem

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					10 June					
idem	EW 250 (P) + Output	1	0.38	ns	crop height 33-43 cm; 65% crop cover; 10 June	SaL	roots 20-21 cm	56	0.23	idem
idem	EW 250 (P) +TF8035	1	0.38	ns	crop height 33-43 cm; 65% crop cover; 10 June	SaL	roots 20-21 cm	56	0.22	idem
Lacenheath, Suffolk; UK; 1994; (Nirobi)	EC 125 (P) + Agral	1	0.38	ns	crop height 25-30 cm; 80% crop cover; 25 July	LSa	CH	64	0.09	RJ1884B; GB51-94-S171; [Patel *et al*., 1995, PP5/0103]
idem	EC 125 (P) + Output	1	0.38	ns	crop height 25-30 cm; 80% crop cover; 25 July	LSa	CH	64	0.09	idem
idem	EW 250 (P) + Agral	1	0.38	ns	crop height 25-30 cm; 80% crop cover; 25 July	LSa	CH	64	< 0.05	idem
idem	EW 250 (P) + Output	1	0.38	ns	crop height 25-30 cm; 80% crop cover; 25 July	LSa	CH	64	< 0.05	idem
idem	EW 250 (P) +TF8035	1	0.38	ns	crop height 25-30 cm; 80% crop cover; 25 July	LSa	CH	64	0.06	idem
Peterborough, Cambridgeshire; UK; 1994; (Navarre)	EC 125 (P) + Agral	1	0.38	ns	crop height 30 cm; 50% crop cover; 25 July	peat	CH	64	≤ 0.05	RJ1884B; GB51-94-S172; [Patel *et al*., 1995, PP5/0103]
idem	EC 125 (P) + Output	1	0.38	ns	crop height 30 cm; 50% crop	peat	CH	64	< 0.05	idem

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					cover; 25 July					
idem	EW 250 (P) + Agral	1	0.38	ns	crop height 30 cm; 50% crop cover; 25 July	peat	CH	64	< 0.05	idem
idem	EW 250 (P) + Output	1	0.38	ns	crop height 30 cm; 50% crop cover; 25 July	peat	CH	64	< 0.05	idem
idem	EW 250 (P) +TF8035	1	0.38	ns	crop height 30 cm; 50% crop cover; 25 July	peat	CH	64	< 0.05	idem
Carpentras; S-France; 1983; (Touchon)	EC 125 (P)	1	0.19	0.038	GS ns; 23 Aug	ns	ns	34	< 0.05	RIC1913; Ca 301.84 [Culoto and Mallmann, 1984, PP9/0050]
idem	EC 125 (P)	1	0.38	0.075	GS ns; 23 Aug	ns	ns	34	< 0.05	idem
idem	EC 250 (rac) + Agral	1	0.75	0.15	GS ns; 23 Aug	ns	ns	34	< 0.05	idem
Carpentras; S-France; 1983; (Touchon)	EC 125 (P)	1	0.19	0.038	GS ns; 11 July	ns	ns	77	< 0.05	RIC1913; Ca 305.84 [Culoto and Mallmann, 1984, PP9/0050]
idem	EC 125 (P)	1	0.38	0.075	GS ns; 11 July	ns	ns	77	< 0.05	idem
idem	EC 250 (rac) + Actiplus	1	0.38	0.075	GS ns; 11 July	ns	ns	77	< 0.05	idem
Reims; N-France; 1983; (Condor)	EC 125 (P)	1	0.19	0.062	GS ns; 25 July	ns	ns	134	< 0.05	RIC1913; R 25.83 [Culoto and Mallmann, 1984, PP9/0050]
idem	EC 125 (P)	1	0.38	0.12	GS ns; 25 July	ns	ns	134	< 0.05	idem
idem	EC 250 (rac) + Actiplus	1	0.75	0.25	GS ns; 25 July	ns	ns	134	< 0.05	idem
Villefranche;	EC	1	0.38	0.07	GS ns;	ns	ns	57	< 0.05	RIC1913;

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
S-France; 1983; (Ringo Lefevre)	250 (rac) + Agral			5	9 Aug					VF 203.84 [Culoto and Mallmann, 1984, PP9/0050]
Villefranche; S-France; 1983; (Tito)	EC 125 (P)	1	0.12	0.025	GS ns; 29 July	ns	ns	56	< 0.05	RIC1913; VF 101.83 [Culoto and Mallmann, 1984, PP9/0050]
idem	EC 125 (P)	1	0.19	0.038	GS ns; 29 July	ns	ns	56	< 0.05	idem
idem	EC 250 (rac) + Actiplus	1	0.38	0.075	GS ns; 29 July	ns	ns	56	< 0.05	idem
Grisolles; South West; S-France; 1997; (Presto)	EC 125 (P)	1	0.31	0.10	BBCH43; crop height 40 cm; 3 Sept	Sa	MAT	28	< 0.04 [CT] [cntrl=0.03]	RJ2638B; S557.97; [Mason *et al*., 1999, PP5/0112]
Ondes; South West; S-France; 1997; (Bolerot)	EC 125 (P)	1	0.31	0.10	BBCH43; crop height 45-50 cm; 9 Sept	Sa	MAT	24	0.03	RJ2638B; S558.97; [Mason *et al*., 1999, PP5/0112]
Grisolles; SouthWest; S-France; 1999 (Presto Fa)	EC 125 (P)	1	0.36	0.12	BBCH 47 crop height 40 cm; 27 Aug	Sa	BBCH 47	26	0.02	RJ3065B; FR12-99-S760; [McGill and Sutra, 2000, PP5/0309]
Borgo Sabotino; Lazio, Italy, 1998; (Turbo)	EC 125 (P)	1	0.31	0.078	BBCH 47; crop height 30-35 cm; 19 Mar	Sa	BBCH 47	29	0.19	RJ2659B; IT40-98-H310; [Mason and Volpi, 1999, PP5/0125]
Manfredonia; Puglia; Italy, 1998; (Efeso Hybrid)	EC 125 (P)	1	0.31	0.10	BBCH 42; crop height 15-20 cm; 23 Mar	Sa	BBCH 42	28	0.05	RJ2659B; IT50-98-H311; [Mason and Volpi, 1999, PP5/0125]
Silla; Valencia; Spain; 1997; (Nantesa)	EC 125 (P)	1	0.31	0.10	crop height 50 cm 14 Mar	LiC	MAT	28	0.07	RJ2638B; ES10-97-SH001; [Mason *et al*., 1999, PP5/0112]
Silla; Valencia; Spain; 1997; (Nantesa)	EC 125 (P)	1	0.31	0.10	crop height 50 cm; 14 Mar	LiC	MAT	28	0.07	RJ2638B; ES10-97-SH101; [Mason *et al*., 1999, PP5/0112]

CARROTS Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Banded foliar spray over rows										
Talavera de la Reina; Toledo; Spain; 1998; (Nantesa)	EC 125 (P)	1	0.33	0.10	BBCH45; crop height 20 cm; 15 July	LC	49	28	0.02	RJ2772B; ES10-98-SH003; [Ryan and Gallardo; 1999; PP5/0118]
L'Alcudia; Valencia; Spain; 1998; (Nantesa)	EC 125 (P)	1	0.32	0.16	BBCH 43; crop height 32 cm; 21 Apr	LC	CH	28	0.03	RJ2772B; ES10-98-SH103; [Ryan and Gallardo; 1999; PP5/0118]

Soil type: LiC = lime clay;

GSH: CH = Commercial harvest (roots 20-25 cm long; 2-4 cm diameter);

[SS] Sample size less than the required 2 kg (0.9 kg or 31-35 roots in trial 71TX83-044 Santa Rosa; 0.9 kg or 30 roots in trial 71TX83-041 Uvalde); sample considered not representative for MRL setting.

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample.

[a] Results of duplicate field samples [report T002222-07]

[b] W29CA081302: BBCH growth stage reported to be 13 BBCH. Considering the application dates and harvest dates, the BBCH stage is estimated to be between 40-43 and as such considered suitable.

[c] 71TX83-044 and 71TX83-055 and 75FL84-004: It is unclear whether 2 or 3 applications were performed as 3 dates were mentioned. However, in none of the other parts of the reports a third application is mentioned. If there was a third (the first) application was performed it was performed with an interval of 50 days and 412 days, respectively for the trials in Santa Rosa and Mission, Texas. Considering the decline data in the various studies, this application is not considerd to influence the residue level in the end. The trials perfomed in South bay Florida (interval 14 days) and Zellwood Florida (interval 30 days) residue levels were below LOQ and as such the influence of a possible third application is not relevant.

Additional trial information

TMU0902/B and PP009B300. Non-GLP. Only trial 53FL82-029 was summarized; other trials did not comply with cGAP. Field conditions described in TMU0902/B; analysis described in PP009B300. Backpack sprayer, spray volume of 160-930 L/ha. Samples (2.3 kg) were collected. Samples were stored frozen at -23 °C for a maximum of 670 days (harvest to report date). Samples were analysed for total fluazifop using **HPLC-UV and GC-MS method ref III modificationB (i.e. PPRAM 62) with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries were not reported. Control samples were < 0.05 mg/kg

TMU1231/B Non-GLP. Spray volume of 140-336 L/ha. Samples (at least 2.27 kg, containing 21-45 carrots) were collected. Samples were stored frozen within 24 hours after sampling and kept frozen until analysis (<-20 °C). No further data on storage time reported. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62**, with minor modifications **with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for mean recovery (82% at 0.04-0.8 mg/kg for fluazifop acid (II); 91% for fluazifop-butyl). Control samples were < 0.04 mg/kg.

TMU1812/B Non-GLP. No unusal weather conditions. Spray volume of 187-229 L/ha. Samples (0.9- 2.27 kg, 30-45 carrots) were collected at PHI 29-48 days. Samples were stored frozen until analysis (<-20 °C) and analysed within 9 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2**, with minor modifications **with a valid LOQ of 0.05 mg/kg**. The recovery at fortification levels 0.04, 0.05, 0.12, 0.16, 0.6 (n = 1-3) ranged from 70-113%, with a mean recovery of 89 ± 13%. Control samples were < 0.03 mg/kg.

RSR-027-87/C Non-GLP. The trials were performed to confirm the high residue levels that were found in trials performed in 1984 in that region [Francis, 1985, 406311, report TMU1182/B]. Sspray volume of 160 L /ha. Samples (at least 2.27 kg) were collected at PHI 31 days. Samples were stored frozen within 24 hours after sampling and kept frozen until analysis (<-20 °C). No further data on storage time reported. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2**, with minor modifications **with a valid LOQ of 0.05 mg/kg**. Samples **were corrected** for recovery of internal standard (average recovery 73% ± 6% at 0.5 mg/kg, n=ns), mean recovery of internal standard was 66 ± 5%. Control samples were < 0.03 mg/kg.

T002222-07. GLP study. No unusual weather condition. Spray volume of 47-374 L /ha. Samples consisted of at least 12 large carrots or 24 small carrots weighing in total at least 2.27 kg. Samples were stored frozen within 8 hours after sampling and kept frozen until analysis (<-20 °C) for a maximum of 10.4 months (less then 24 months of demonstrated storage stability). Samples were analysed for total fluazifop using **HPLC-MS/MS Method GRM044.01A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for mean concurrent recovery (88-96% at 0.01–0.10 mg/kg). Control samples were < 0.01 mg/kg.

M11030. GLP study. No unusual weather conditions. Prepressurized spraying with a spray volume of 100 L/ha. Samples consisted of at least 12 carrots with a minimum of 2 kg roots and were collected systematically from across the plots. Samples were stored at <-20 °C for less than 9 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET.138 with a valid LOQ of 0.01 mg/kg**. Procedural recovery (n = 1) = 98% at 0.1 mg/kg. Mean method validation (n = 5-7/fortification level) 92-103% perfortification level. Control samples < 0.01 mg/kg.

M3954B. Non-GLP. No unusual weather conditions. Broadcast foliar spray application using a CO2 knapsack sprayer or CO2 hand held boom. Spray volume 200-260 L/ha. Samples were taken by hand and tops were trimmed. Sample sizes not stated. Samples were stored at -20 °C, storage time not stated but less than 12 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (77% at 0.5 mg/kg). Control samples < 0.03 mg/kg.

M5317B. Non-GLP. No unusual weather conditions. Broadcast foliar spray using a one man boom sprayer. Spray volume not stated. Sample of 24 items taken at random from the central beds. Soil and tops were removed. Storage time 6 months at -16°C. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg.** Mean internal standard recovery (95% at 0.5 mg/kg). Control samples < 0.05 mg/kg.

RJ1884B. GLP. No unusual weather conditions. Broadcast foliar spray using a boom sprayer. Samples of 12 roots were taken by hand from across the plots. Tops were trimmed and excess soil was brushed off or wiped off. Storage time 1-4 months at -15 °C. Samples were analysed for total fluazifop using **NMR method RAM 197/02 with a valid LOQ of 0.01 mg/kg**. Mean internal standard recovery (93% at 0.50 mg/kg). Control samples < 0.05 mg/kg.

RIC1913. non-GLP. Weather conditions, treatment type and spray equipment ns. Spray volume 300-500 L/ha. Sample sizes not stated. Samples were stored at -18 °C for 50-68 days. Samples were analysed for total fluazifop using **HPLC-UV method 62/1 with a valid LOQ of 0.05 mg/kg.** Samples were corrected for mean concurrent recovery (90% at 0.2–0.5 mg/kg); uncorrected results were ns. Control samples < 0.05 mg/kg.

RJ2638B. GLP. No unusual weather conditions. Broadcast foliar spray using a CO2 sprayer. Spray volume 300-310 L/ha. Mature roots (2.0-2.5 kg) were sampled by hand and were taken systematically from across the plots. Samples were stored at -18 °C for 159-204 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (105-115% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg, except 0.03 mg/kg in trial S557.97.

RJ3065B. GLP. No unusual weather conditions. Broadcast foliar spray using a handheld boom sprayer. Spray volume 300 L/ha. Roots (1.0-1.3 kg; 15 units) were sampled by hand and were taken systematically from across the plots. Samples were cleaned and trimmed. Samples were stored at -18 °C for 224 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (101-105% at 0.05-0.2 mg/kg). Control samples < 0.01 mg/kg.

RJ2659B. GLP. No unusual weather conditions. Broadcast foliar spray using a motor knapsack sprayer with boom. Spray volume 300-400 L/ha. Plants (2.1-2.6 kg; 24-32 roots) were sampled by hand and were taken systematically from across the plots. Adhering soil was removed and roots were trimmed. Samples were stored at -18 °C for 38-41 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (110-110% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2772B. GLP. No unusual weather conditions. **Banded spray over rows** using a gas knapsack sprayer with a lance. Spray volume 200-320 L/ha. Mature roots (1.2-2.5 kg) were sampled by hand and were taken systematically from across the plots. Samples were cleaned and trimmed. Samples were stored at -18 °C for 56-106 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (82-98% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

Celeriac

One cGAPs for celeriac is available:

- cGAP from Belgium and the Netherlands with 1 ×0.38 kg ai/ha with a PHI of 56 days

  Trials that could be matched to this cGAP were summarized.

Table 222 lists trials conducted in the Northern France (1997, 1998). A broadcast or banded foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 222.

Table 222 Supervised field trials on celeriac (roots), treated with a broadcast or banded foliar fluazifop-butyl spray

CELERIAC Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray application										
27800 Brionne; N-France; 1998; (Monarque)	EC 125 (P)	1	0.38	0.12	BBCH 41, crop height 20-25 cm; 26 Aug	SiC	49	50	0.11	RJ2804B; S207.98 [Ryan and Renard, 1999, PP5/0120]
27340 Criquebeuf /Seine; N-France; 1998; (Monarch)	EC 125 (P)	1	0.38	0.12	BBCH 41; crop height 20-25 cm; 25 Aug	C	49	50	0.17	RJ2804B; S208.98 [Ryan and Renard, 1999, PP5/0120]
Banded foliar spray application over rows										
37510 Berthenay; N-France; 1997; (Monarch)	EC 125 (P)	1	0.38	0.11	BBCH 41; crop height 30 cm; 11 July	LSa	MAT	56	< 0.01	RJ2630B; 97HCLSAP03; [Mason, 1999, PP5/0116]
37270 St Martin le Beau; N-France; 1997; (Monarch)	EC 125 (P)	1	0.38	0.11	BBCH 43; crop height 30 cm; 1 July	Sa	MAT	56	< 0.01	RJ2630B; 97HCLSAP04; [Mason, 1999, PP5/0116]

BBCH 41 = roots beginning to expand (diameter >0.5 cm), BBCH 43 = 30% of expected root diameter reached.

Additional trial information:

RJ2804B. GLP. No unusual weather conditions. Broadcast foliar spray using a hand held boom. Spray volume 300 L/ha. Samples (12 roots or 9.5-10.5 kg) were taken by hand systematically from across the plots. Storage at -17 °C for 143-144 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02**. Samples were not corrected for concurrent method recovery (98-102% at 0.05-0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2630B. GLP. Weather conditions were unusual (more rainfall, higher temperatures, drier), but no major effect on the crops was noticed. **Banded spray(2 m wide) over the rows** using a gas knapsack sprayer with side boom. Spray volume 327-343 L/ha. Samples (12 roots, > 5 kg) were taken systematically from across the plots. Samples were cleaned and trimmed by removing excess soil and cutting radicles and leaves. Storage at -17 °C for 81 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01**. Samples were not corrected for concurrent method recovery (69-97% at 0.025-0.25 mg/kg). Control samples < 0.01 mg/kg.

Manioc (cassava)

A GAP for manioc is not available. As the manufacturer did not seek to have maximum residue levels estimated on manioc, no further action was taken to retrieve the available studies on manioc [report M12002, not referenced].

Potatoes

Two cGAPs for potatoes are available:

- cGAP from Brazil with 1 ×0.25 kg ai/ha with a PHI of 28 days
- cGAP from the Netherlands with 1 ×0.25 kg ai/ha and a PHI of 75 days.

Trials that could be matched to these cGAPs were summarized.

Table 224 lists trials conducted in the Canada (1980, 2007, 2008), Brazil (2010-2011), Germany (1980, 1981, 1983, 1992, 2001, 2002, 2003), the UK (1980, 1982, 1983, 2000, 2002, 2005),

the Netherlands (1980, 1984), France (2001, 2003, 2004, 2005, 2006), Greece (2003,), Italy (2003,) and Spain (2000, 2002, 2003, 2005, 2006). A broadcast or banded foliar spray over rows with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 224. Results marked with "[QU]", "[SS]" or "[AM]" are not selected for derivation of the MRL, if according to cGAP.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[AM] indicates that the analytical method did not contain a hydrolysis step and therefore fluazifop (II) conjugates are not included.

[SS] indicates that the sample size was less than the required 12 tubers or 2 kg potatoes.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.02 mg/kg for HPLC-UV methods PPRAM 51 and PPRAM 52 or to 0.05 mg/kg for HPLC-UV method PPRAM 62, 62/1 or 62/2.

Additional trials from Canada (1979) were available with 1 ×0.25–0.50–1.0 kg ai/ha with harvest at 85–95 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from the Netherlands (1979, 1980) were available with 1 × 0.50–1.0–2.0 kg ai/ha with harvest at 118–141 DAT [Atreya *et al*., 1980, report PP009B010; Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from Germany (1980) were available with 1 ×1.0 kg ai/ha with harvest at 33, 35, 39–54, 56, 66, 68, 71, 78, 80, 102–105 DAT [Atreya *et al*., 1980, PP9/0507, report PP009B013; Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. These trials were addressed in the metabolism section, because only the fluazifop-butyl and free fluazifop residues were analysed.

Additional residue trials from Canada (1979, 1980) were available with 1 ×0.20–0.25–0.40–0.50–1.0–2.0 kg ai/ha, DAT44–68 or 88–114 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional residue trials from the UK (1980, 1981) were available with 1 ×0.50–0.75–1.0–1.5 kg ai/ha with harvest at 63–70 or 91–124 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B; Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Germany (1981, 1993) were available with 1 ×0.31 kg ai/ha or 1 ×0.75 kg ai/ha and harvest at 26, 31–69, 93–94 DAT or 1 × 0.38 kg ai/ha with harvest at 42 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B, Weeren, 1994, PP5/0102, report AZ13403/93]. Additional trials from Sweden (1981) were available with 1 ×0.50 or 1 ×1.0 kg ai/ha with harvest at 31-52 DAT and 2 ×0.5 kg ai/ha with harvest at 49 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. Additional trials from Italy (1981 and 1998) were available with 1 ×0.25–0.50–0.75 kg ai/ha and harvest at 39 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B or with 1 ×0.38 kg ai/ha and harvest at 41–42 and 45–57 DAT [Mason and Volpi, 1999, PP9/0119, report RJ2757B]. These trials were not summarized, as they do not assist in MRL setting.

Variability factor for potatoes

A study was performed to determine the magnitude of residues of total fluazifop in 120 individual potato tubers from a study conducted in the UK in 2001 to establish a variability factor for use in the estimation of acute dietary exposure [McGill, 2002, PP5/1150, report 01HJ128/01].Two separate plots were treated with a single application of fluazifop-P-butyl (EC 125 g ai/L) at an exaggerated dose of 0.55 kg ai/ha. Plot 1 was treated at BBCH 41-43 with harvest at 72 DAT and plot 2 was treated at BBCH 40 with harvest at 86 DAT. Batches of 120 individual tubers were sampled from these two treated plots. Samples were also taken from untreated plots. Only the individual tubers from plot 2 were weighed, but individual weights were not reported.

Upon receiptat the analytical facility, the fresh samples were stored frozen at -18 °C and kept for a maximum of 4 months. Samples were analysed for total fluazifop using HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg. Concurrent recoveries and residue levels in control samples were not reported.

Residues were determined in the range < 0.01 to 1.66 mg/kg and < 0.01 to 0.95 mg/kg, respectively from the two plots. Residues were shown corrected for the mean concurrent recovery, where this was < 100%. Uncorrected results were not reported. In those cases, where the residue was reported as below the LOQ, the concentration was taken as 0.005 mg/kg in the statistical analysis. The average of each plot was calculated without consideration of the weight of the individual tubers. The variability factor was calculated as the ratio between the 97.5th percentile and the mean of residues in individual units and results in a variability factor of 2.91 and 5.55 for plot 1 and 2 respectively (Table 223).

For plot 2, a scatterplot of tuber weight versus total fluazifop residue showed that there was no discernable relationship between tuber mass and total fluazifop residue (see Figure 5). Nevertheless, a P95/mean ratio was calculation for plot 2, where the mean was calculated with and without consideration of the weight of the individual tubers (Table 223), using the formula:

$Mean\ corrected\ for\ weight = \frac{\sum_{i=1}^{i=120}(ri_i w_i)}{\sum_{i=1}^{i=120} w_i}$, where r is the residue and w is the weight of the tuber.

Table 223 Distribution of total fluazifop residues in single potato tubers (variability factor)

	Residue distriution (mg/kg)	P5 (mg/kg)	P50 (mg/kg)	P95 (mg/kg)	P97.5 (mg/kg)	Mean (mg/kg)	St. dev.	P95/mean	P97.5/mean (variability factor)
plot 1 (n = 120)	0.005-1.66 (< 0.01, n = 1)	0.05	0.3	0.869	1.17	0.401 not corrected for tuber weight	0.29	2.17	2.91
plot 2 (n = 120)	0.005-0.95 (< 0.01, n = 16)	0.005	0.080	0.442	0.704	0.127 not corrected for tuber weight	0.16	3.48	5.55
plot 2 (n = 120)	0.005-0.95 (< 0.01, n = 16)	0.005	0.080	0.442	0.704	0.123 corrected for tuber weight	nc	3.59	5.72

Distribution p = [rank/(n+1)]*100 is the percentile of the individual ranked value using a distribution free method.

nc = not calculated

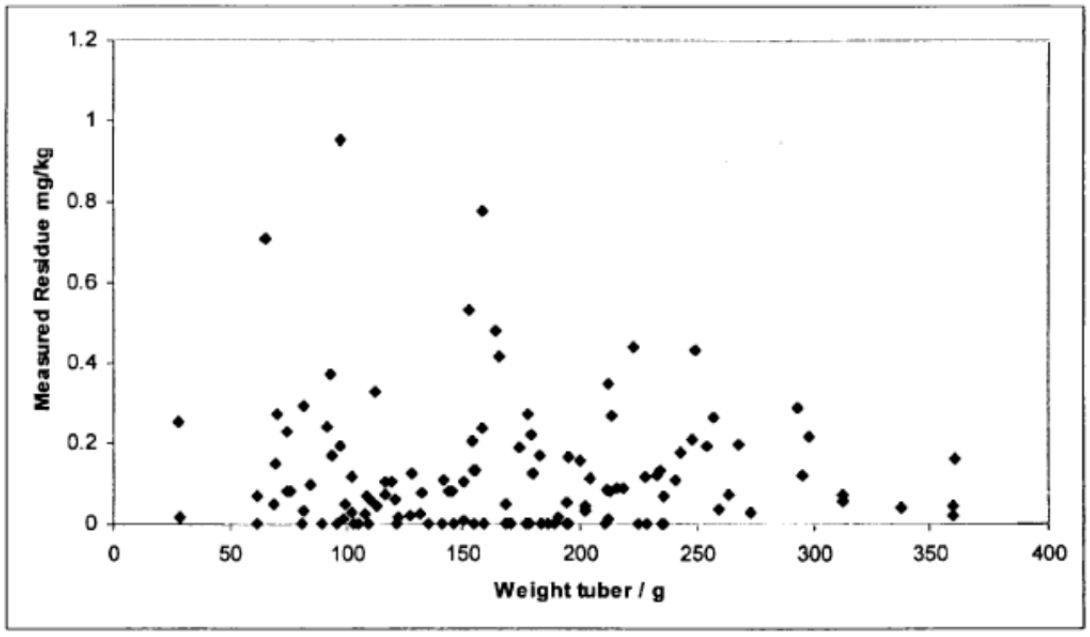


Figure 5 Tuber weight versus total fluazifop measured (plot 2)

Table 224 Supervised field trials on potato (tubers), treated with a broadcast or banded foliar fluazifop-butyl spray

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Location ns; Canada, 1980 (var ns)	EC 250 (rac)	1	0.30	ns	GS ns date ns	ns	ns	73	< 0.02 [QU] [LOQ=0.05]	RJ0226B summary [Atreya and Froggatt, 1981, PP9/0384]
idem	EC 250 (rac)	1	0.50	ns	GS ns date ns	ns	ns	73	< 0.02 [QU] [LOQ=0.05]	idem
idem	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	73 82	< 0.02 < 0.02 [QU] [LOQ=0.05]	idem
Hunter River, PEI, Canada; 2007 (Russet Burbank)	EC 125 (P)	1	0.24	0.16	BBCH 21-25 (plants 10-20 cm high) 17 July	SaL	49	90	< 0.01, < 0.01 mean < 0.01 [a]	CER 02606/07; T232; [Sagan, 2008, A12791B_50005]
idem	EC 125 (P)	1	0.24	0.16	BBCH 21-25 (plants 10-20 cm high) 17 July	SaL	49	90	< 0.01, < 0.01 mean < 0.01 [a]	idem
idem	EC 125 (P)	1	0.26	0.16	BBCH 41-43 (plants 45-55 cm high) 16 August	SaL	49	60	0.042, 0.038 mean 0.040 [a]	idem
idem	EC 125 (P)	1	0.25	0.16	BBCH 44-46 (plants 50-60 cm high) 30 August	SaL	49	46	0.065, 0.072 mean 0.068 [a]	idem
Hunter River, PEI, Canada; 2007 (Yukon Gold)	EC 125 (P)	1	0.24	0.16	BBCH 21-25 (plants 10-20 cm high) 17 July, 2007	SaL	49	90	< 0.01, < 0.01 mean < 0.01 [a]	CER 02606/07; T233 [Sagan, 2008, A12791B_50005]
idem	EC 125 (P)	1	0.24	0.16	BBCH 21-25 (plants	SaL	49	90	< 0.01, < 0.01 mean	idem

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					10-20 cm high) 17 July				< 0.01 [a]	
idem	EC 125 (P)	1	0.26	0.16	BBCH 41-43 (plants 50-60 cm high) 16 August, 2007	SaL	49	46	0.13, 0.11 mean 0.12 [a]	idem
idem	EC 125 (P)	1	0.25	0.16	BBCH 44-46 (plants 50-60 cm high) 30 August	SaL	49	46	0.093, 0.085 mean 0.089 [a]	idem
Portage la Prairie, MB, Canada, 2007 (Russet Burbank)	EC 125 (P)	1	0.24	0.16	BBCH 13-19 (plants 5-10 cm high) 4 July	CL	95 (1 Oct)	89	< 0.01, < 0.01 mean < 0.01 [a]	CER 02606/07; T234; [Sagan, 2008, A12791B_50005]
idem	EC 125 (P)	1	0.24	0.16	BBCH 13-19 (plants 5-10 cm high) 4 July	CL	95	89	< 0.01, < 0.01 mean < 0.01 [a]	idem
idem	EC 125 (P)	1	0.26	0.16	BBCH 38-39 (plants 60-72 cm high) 2 August	CL	95	61	0.024, 0.024 mean 0.024 [a]	idem
idem	EC 125 (P)	1	0.25	0.16	BBCH 42-43 (plants 59-71 cm high) 17 August	CL	95	46	0.17, 0.14 mean 0.16 [a]	idem
New Glasgow, PEI, Canada, 2008 (Shepody)	EC 125 (P)	1	0.24	0.13	BBCH 41-42 (plants 40-50 cm high), 07 Aug	SaL	48	46	0.16 [c], 0.080 [c] mean 0.12 [a]	CER 02608/08; T455; [Sagan, 2009, A12791N_50004]
Albany, PEI, Canada, 2008 (Carlingford)	EC 125 (P)	1	0.22	0.12	BBCH 41-42 (plants 40-50 cm high), 21 Aug	SaL	49	46	< 0.01, < 0.01 mean < 0.01 [a]	CER 02608/08; T456; [Sagan, 2009, A12791N_50004]
New Glasgow, PEI, Canada, 2008 (Goldrush)	EC 125 (P)	1	0.23	0.13	BBCH 38-39 (plants 40-50 cm high), 07	SaL	48 48 49 49 49	39 46 53 60 68	0.28; 0.61 [c], 0.49; 0.32; 0.37;	CER 02608/08; T457; [Sagan, 2009, A12791N_50004]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					Aug				0.34 [c]	
Malden, NB, Canada, 2008 (Red Norland)	EC 125 (P)	1	0.24	0.13	BBCH 43-45 (plants 40-50 cm high), 07 Aug	L	49	46	0.035, 0.036 mean 0.036 [a]	CER 02608/08; T458; [Sagan, 2009, A12791N_50004]
Malden, NB, Canada, 2008 (Goldrush)	EC 125 (P)	1	0.24	0.13	BBCH 45-47 (plants 50-60 cm high), 07 Aug	SaL	49	46	0.27, 0.28 mean 0.28 [a]	CER 02608/08; T459; [Sagan, 2009, A12791N_50004]
Branchton , ON, Canada, 2008 (Goldrush)	EC 125 (P)	1	0.24	0.16	BBCH 43-45 (plants 25-30 cm high), 05 Aug	SiL	49	45	< 0.01, 0.016 mean 0.08 [a]	CER 02608/08; T460; [Sagan, 2009, A12791N_50004]
Elm Creek, MB, Canada, 2008 (Shepody)	EC 125 (P)	1	0.24	0.16	BBCH 67-68 (plants 48-55 cm high), 23 July	SaL	79 91 93 95 97	37 44 51 58 65	0.020; 0.016, 0.012 0.024; < 0.01; 0.020 [a]	CER 02608/08; T461 [Sagan, 2009, A12791N_50004]
Elm Creek, MB, Canada, 2008 (Russet Burbank)	EC 125 (P)	1	0.25	0.16	BBCH 67-68 (plants 48-55 cm high), 23 July	SaL	91	44	< 0.01, 0.036 mean 0.018 [a]	CER 02608/08; T462; [Sagan, 2009, A12791N_50004]
St Marc-sur-Richelieu, QC, Canada, 2008 (Kennebec)	EC 125 (P)	1	0.23	0.11	BBCH 68-69 (plants 60-70 cm high), 29 July	SiCL	97	45	0.044, 0.037 mean 0.040 [a]	CER 02608/08; T463; [Sagan, 2009, A12791N_50004]
Taber, AB, Canada, 2008 (Russet Burbank)	EC 125 (P)	1	0.24	0.16	BBCH 47-48 (plants 45-50 cm high), 11 Aug	L	48-49	46	0.14, 0.20 mean 0.17 [a]	CER 02608/08; T464; [Sagan, 2009, A12791N_50004]
Abbotsford, BC, Canada, 2008 (WanBa)	EC 125 (P)	1	0.23	0.16	BBCH 62-63 (plants 30-50 cm high), 2 Sept	SiL	48-49	44	0.043, 0.035 mean 0.039 [a]	CER 02608/08; T465; [Sagan, 2009, A12791N_50004]
Minto, MB, Canada, 2008 (Norland)	EC 125 (P)	1	0.24	0.16	BBCH 68 (plants 45-65 cm high), 24 July	L	49-95	46	0.14, 0.16 mean 0.15 [a]	CER 02608/08; T466; [Sagan, 2009, A12791N_50004]
Innisfail, AB, Canada, 2008	EC 125 (P)	1	0.23	0.15	BBCH 43-44 (plants	CL	46-48	45	0.065, 0.050 mean 0.058	CER 02608/08; T467; [Sagan, 2009,

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					50-60 cm high), 25 July				[a]	A12791N_50004]
Piedade, SP, Brazil; 2010/11; (Asterix)	EW 250 (P)	1	0.25	0.25	BBCH 45; 5 May, 2011	C	48	27	0.07	M11031; AMA; [Draetta, 2012, A12530B_10019]
Uberlândia, MG, Brazil; 2010/11; (Agata)	EW 250 (P)	1	0.25	0.25	BBCH 42; 26 May	C	47	28	< 0.01	M11031; JJB; [Draetta, 2012, A12530B_10019]
Cachoeira de Minas, MG, Brazil; 2010/11; (Atlantic)	EW 250 (P)	1	0.25	0.25	BBCH 47 – 48; 11 March	CL	49	28	< 0.01	M11031; RWC1; [Draetta, 2012, A12530B_10019]
Palmeira, PR, Brazil; 2010/11; (Agata)	EW 250 (P)	1	0.25	0.25	BBCH 43; 16 May	C	49	28	< 0.01	M11031; RWC2 ; [Draetta, 2012, A12530B_10019]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.31	ns	GS ns date ns	ns	ns	76	0.03 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.31	ns	GS ns date ns	ns	ns	78	< 0.03 [QU] [LOQ=0.05]	idem
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	76	0.03 [QU] [LOQ=0.05]	idem
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	78	0.23 [QU] [LOQ=0.05]	idem
Steinweiler, Germany, 1983, (Saskia)	EC 125 (P)	1	0.38	0.094	BBCH ns; (30-35 cm; 40% ground cover); 13 June	L	- 75	11 29	0.34 < 0.04 [LOQ=0.05]	M3676B; RS 8368 E2 (A) [Harradine, 1984, PP9/0052]
idem	EC 250 (rac)	1	0.75	0.19	BBCH ns; (30-35 cm; 40% ground cover); 13 June	L	- 75	11 29	0.63 0.09	M3676B; RS 8368 E2 (B) [Harradine, 1984, PP9/0052]
Steinweiler, Germany, 1983	EC 125 (P)	1	0.38	0.094	BBCH ns; (20 cm; 25%	L	- 81	29 87	< 0.04 < 0.04	M3676B; RS 8368 E3 (A) [Harradine,

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Aula)					ground cover); 13 June				[LOQ=0.05]	1984, PP9/0052]
idem	EC 250 (rac)	1	0.75	0.188	BBCH ns; (20 cm; 25% ground cover); 13 June	L	- 81	29 87	< 0.04 < 0.04 [LOQ=0.05]	M3676B; RS 8368 E3 (B) [Harradine, 1984, PP9/0052]
Luneburg, Germany, 1983 (Hollander Erstling)	EC 125 (P)	1	0.38	0.094	BBCH 45; (80% ground cover), 26 May	Sa	80 90	27 41	<u>< 0.04</u> 0.04 [LOQ=0.05]	M3676B; RS 8368 B1 (A) [Harradine, 1984, PP9/0052]
idem	EC 250 (rac)	1	0.75	0.19	BBCH 45; (80% ground cover), 26 May	Sa	80 90	27 41	0.05 < 0.04 [LOQ=0.05]	M3676B; RS 8368 B1 (B) [Harradine, 1984, PP9/0052]
Buchendorf, Germany, 1983 (Hela)	EC 125 (P)	1	0.38	0.094	BBCH 24; (90% ground cover), 21 June	Sa	80-92	29 44	<u>0.06</u> 0.06	M3676B; RS 8368 B2 (A) [Harradine, 1984, PP9/0052]
idem	EC 250 (rac)	1	0.75	0.19	BBCH 24; (90% ground cover), 21 June	Sa	80-92	29 44	< 0.04 0.05 [LOQ=0.05]	M3676B; RS 8368 B2 (B) [Harradine, 1984, PP9/0052]
Vanendorf, Germany, 1983 (Fortuna)	EC 125 (P)	1	0.38	0.094	BBCH 25; (80% ground cover), 4 July	SaL	85 99	52 67	0.04 < 0.04 [LOQ=0.05]	M3676B; RS 8368 B3 (A) [Harradine, 1984, PP9/0052]
idem	EC 250 (rac)	1	0.75	0.19	BBCH 25; (80% ground cover), 4 July	SaL	85 99	52 67	0.13 0.05	M3676B; RS 8368 B3 (B) [Harradine, 1984, PP9/0052]
Steinweiler, Germany, 1983, (Jetta)	EC 125 (P)	1	0.38	0.094	BBCH ns; (20-30 cm; 30% ground cover); 13 June	L	- 81	21 44	0.22 0.06	M3676B; RS 8368 E1 (A) [Harradine, 1984, PP9/0052]
idem	EC 250 (rac)	1	0.75	0.19	BBCH ns; (20-30 cm; 30% ground cover); 13 June	L	- 81	21 44	0.52 0.35	M3676B; RS 8368 E1 (B) [Harradine, 1984, PP9/0052]
8601, Coblenz, Germany, 1992, (Adretta)	EC 125 (P)	1	0.38	0.125	BBCH 41 (40 cm; BBCH 41); 17 June	L	95	13 42 83	0.50 0.21 0.14	RJ1405B, RF 11/92-CO, plot 2 [Bolygo, 1993, PP5/0095]
idem	ME 125	1	0.38	0.12	BBCH	L	95	13	0.86	RJ1405B,

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	(P)			5	41 (40 cm; BBCH 41); 17 June			42 83	0.26 0.21	RF 11/92-CO, plot 4 [Bolygo, 1993, PP5/0095]
2021, Rosenow, Germany, 1992, (Likaria)	EC 125 (P)	1	0.38	0.125	BBCH 41 (30 cm; BBCH 41); 23 June	SaL	99	14 42 86	0.11 0.07 0.06	RJ1405B, RF 11/92-RO, plot 2 [Bolygo, 1993, PP5/0095]
idem	ME 125 (P)	1	0.38	0.125	BBCH 41 (30 cm; BBCH 41); at 23 June	SaL	99	14 42 86	0.17 0.10 0.08	RJ1405B, RF 11/92-RO, plot 4 [Bolygo, 1993, PP5/0095]
7101 Cunnersdorf; Germany; 1992; (Liu)	EC 125 (P)	1	0.38	0.125	BBCH 41-49 (35 cm; BBCH 41-49); 22 June	SaL	95	11 42 81	0.81 0.26 0.11	RJ1405B, RF 11/92-CU, plot 2 [Bolygo, 1993, PP5/0095]
idem	ME 125 (P)	1	0.38	0.125	BBCH 41-49 (35 cm; BBCH 41-49); 22 June	SaL	95	11 42 81	0.62 0.16 0.20	RJ1405B, RF 11/92-CU, plot 4 [Bolygo, 1993, PP5/0095]
5301, Kötschau, Germany, 1992, (Solina)	EC 125 (P)	1	0.38	0.125	BBCH 41 (35 cm; BBCH 41); 18 June	SaL	95	13 42 84	0.65 0.07 < 0.05	RJ1405B, RF 11/92-KÖ, plot 2 [Bolygo, 1993, PP5/0095]
idem	ME 125 (P)	1	0.38	0.125	BBCH 41 (35 cm; BBCH 41); 18 June	SaL	95	13 42 84	0.54 < 0.05 < 0.05	RJ1405B, RF 11/92-KÖ, plot 4 [Bolygo, 1993, PP5/0095]
D-29553 Bienbüttel-Varendorf, Germany, 1993 (not specified)	ME 125 (P)	1	0.38	ns	ns	ns	ns	42	0.17 [QU]	AZ13430/93;RS-9307-B1 plot 1 [Weeren, 1994, PP5/0102 = Pelz, 1994, PP5_50062] (processing)
idem	SC 125 (P)	1	0.38	ns	ns	ns	ns	42	0.17 [QU]	idem, plot 2
D-21514 Büchen, Germany, 1993 (not specified)	ME 125 (P)	1	0.38	ns	ns	ns	ns	42	0.22 [QU]	AZ13430/93;RS-9307-B2 plot 1 [Weeren, 1994, PP5/0102 = Pelz, 1994, PP5_50062]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
										(processing)
idem	SC 125 (P)	1	0.38	ns	ns	ns	ns	42	0.23 [QU]	idem, plot 2
D-86565 Gachenbach-Etzelberg, Germany, 1993 (not specified)	ME 125 (P)	1	0.38	ns	ns	ns	ns	42	< 0.01 [QU]	AZ13430/93;RS-9307-G1 plot 1 [Weeren, 1994, PP5/0102 = Pelz, 1994, PP5_50062]
idem	SC 125 (P)	1	0.38	ns	ns	ns	n	42	0.01 [QU]	idem, plot 2
D-86565 Gachenbach-Weilach, Germany, 1993 (not specified)	ME 125 (P)	1	0.38	ns	ns	ns	ns	42	< 0.01 [QU]	AZ13430/93;RS-9307-G2, plot 1 [Weeren, 1994, PP5/0102 = Pelz, 1994, PP5_50062]
idem	SC 125 (P)	1	0.38	ns	ns	ns	nr	42	< 0.01 [QU]	idem, plot 2
D-04886 Grosstreben, Germany, 1993 (not specified)	ME 125 (P)	1	0.38	ns	ns	ns	ns	42	0.04 [QU]	AZ13430/93;RS-9307-K1, plot 1 [Weeren, 1994, PP5/0102 = Pelz, 1994, PP5_50062]
idem	SC 125 (P)	1	0.38	ns	ns	ns	ns	42	0.13 [QU]	idem, plot 2
D-06925 Löben, Germany, 1993 (not specified)	ME 125 (P)	1	0.38	ns	ns	ns	ns	42	< 0.01 [QU]	AZ13430/93;RS-9307-K2, plot 1 [Weeren, 1994, PP5/0102 = Pelz, 1994, PP5_50062]
idem	SC 125 (P)	1	0.38	ns	ns	ns	ns	42	< 0.01 [QU]	idem, plot 2
D-21279, Appel, Germany; 2001; (Ponto)	EC 125 (P)	1	0.13	0.042	BBCH leaf 22–23; 10-12% soil cover; 25 May	LSa	49	110	< 0.01	gpo11501; plot 2; [Simon, 2002, PP5/1148]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 22–23; 10-12% soil cover; 25 May	LSa	49	110	< 0.01	gpo11501; plot 3; [Simon, 2002, PP5/1148]
idem	EC 125 (P)	1	0.13	0.042	BBCH leaf 26-28; 45-60%	LSa	49	99	< 0.01	gpo11501; plot 4; [Simon, 2002, PP5/1148]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					soil cover; 5 June;					
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 26-28; 45-60% soil cover; 5 June	LSa	49	99	0.03	gpo11501; plot 5; [Simon, 2002, PP5/1148]
idem	EC 125 (P)	1	0.13	0.042	BBCH leaf 29-31; 80-85% soil cover; 14 June	LSa	49	90	0.02	gpo11501; plot 6; [Simon, 2002, PP5/1148],
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 29-31; 80-85% soil cover; 14 June	LSa	49	90	0.06	gpo11501; plot 7; [Simon, 2002, PP5/1148]
D-04886, Großtreben; Germany; 2001; (Agria)	EC 125 (P)	1	0.13	0.042	BBCH leaf 22–25; 25% soil cover; 6 June	SaL	49	111	< 0.01	gpo31501; plot 2 [Simon, 2002, PP5/1147]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 22–25; 25% soil cover; 6 June	SaL	49	111	< 0.01	gpo31501; plot 3 [Simon, 2002, PP5/1147]
idem	EC 125 (P)	1	0.13	0.042	BBCH leaf 22-26; 45% soil cover; 15 June	SaL	49	102	< 0.01	gpo31501; plot 4 [Simon, 2002, PP5/1147]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 22-26; 45% soil cover; 15 June	SaL	49	102	< 0.01	gpo31501; plot 5 [Simon, 2002, PP5/1147]
idem	EC 125 (P)	1	0.13	0.042	BBCH leaf 22-26; 60% soil cover 25 June	SaL	49	92	< 0.01	gpo31501; plot 6 [Simon, 2002, PP5/1147]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 22-26; 60% soil cover; 25 June	SaL	49	92	< 0.01	gpo31501; plot 7 [Simon, 2002, PP5/1147]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
D-94522; See; Germany; 2001; (Agria)	EC 125 (P)	1	0.13	0.042	BBCH leaf 13-16; 10% soil cover; 1 June	SiL	49	101	< 0.01	gpo41501; plot 2; [Simon, 2002, PP5/1145]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 13-16; 10% soil cover; 1 June	SiL	49	101	< 0.01	gpo41501; plot 3; [Simon, 2002, PP5/1145]
idem	EC 125 (P)	1	0.13	0.042	BBCH leaf 16-19; 25% soil cover; 12 June	SiL	49	90	< 0.01	gpo41501; plot 4; [Simon, 2002, PP5/1145]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 16-19; 27% soil cover; 12 June	SiL	49	90	< 0.01	gpo41501; plot 5; [Simon, 2002, PP5/1145]
idem	EC 125 (P)	1	0.13	0.042	BBCH leaf 31-33; 60% soil cover; 21 June	SiL	49	81	< 0.01	gpo41501; plot 6; [Simon, 2002, PP5/1145]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 32-33; 65% soil cover; 21 June	SiL	49	81	< 0.01	gpo41501; plot 6; [Simon, 2002, PP5/1145]
D-19089; Wessin; Germany; 2001; (Ponto)	EC 125 (P)	1	0.13	0.042	BBCH leaf 23-30; 40-50% soil cover; 8 June	LSa	47-48	95	< 0.01	gpo91501 plot 2; [Simon, 2002, PP5/1146]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 23-30; 40-50% soil cover; 8 June	LSa	47-48	95	< 0.01	gpo91501 plot 3; [Simon, 2002, PP5/1146]
idem	EC 125 (P)	1	0.13	0.042	BBCH leaf 30; 70% soil cover; 19 June	LSa	47-48	84	< 0.01	gpo91501 plot 4; [Simon, 2002, PP5/1146]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 30; 70% soil cover; 19 June	LSa	47-48	84	0.10	gpo91501 plot 5; [Simon, 2002, PP5/1146]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
idem	EC 125 (P)	1	0.13	0.042	BBCH leaf 38-39; 95-98% soil cover; 28 June	LSa	47-48	75	0.02	gpo91501 plot 6; [Simon, 2002, PP5/1146]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 38-39; 95-98% soil cover; 28 June	LSa	47-48	75	0.07	gpo91501 plot 7; [Simon, 2002, PP5/1146]
D-19089; Wessin; Germany; 2002; (Sommer gold)	EC 125 (P)	1	0.13	0.042	BBCH leaf 23-25; 30% soil cover; 30 May	LSa	49	106	< 0.01, < 0.01 mean < 0.01 [a]	gpo079002; plot 1 [Simon, 2003, PP5/1342]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 23-25; 30% soil cover; 30 May	LSa	49	106	0.02, 0.07 mean 0.05 [a]	gpo079002; plot 2; [Simon, 2003, PP5/1342]
D-04720; Döbeln-Bormitz; Germany; 2003; (Power)	EC 125 (P)	1	0.13	0.042	BBCH leaf 23–26; 30-35% soil cover; 6 June	SiL	49	77	< 0.01	gpo023103; plot 1; [Simon, 2004, PP5/1427]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 23–26; 30-35% soil cover; 6 June	SiL	49	77	< 0.01	gpo023103; plot 2; [Simon, 2004, PP5/1427]
Location ns; UK, 1980 (var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	77	0.03 [QU] [LOQ=0.05]	RJ0226B summary [Atreya and Froggatt, 1981, PP9/0384]
idem	EC 250 (rac)	1	0.50	ns	GS ns date ns	ns	ns	77	< 0.02 [QU] [LOQ=0.05]	idem
Manthrope, Linc, UK, 1982 (Estima)	Formulation ns (P)	1	0.75	ns	BBCH ns, post flowering, ns	ns	ns	21	1.2, 1.2 [b] [QU]	PP009B153; trial ns [Atreya *et al*., 1982, PP9/0702] (processing)
Swillington Common, UK, 1982 (Wilja)	Formulation ns (P)	1	0.75	ns	BBCH ns, crop 25 cm high, ns	ns	ns	35	1.2 [b] [QU]	PP009B153; trial ns [Atreya *et al*., 1982, PP9/0702] (processing)

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Cornwall, UK, 1982 (Mari Peer)	Formulation ns (P)	1	0.75	ns	BBCH ns, before meeting rows, ns	ns	ns	70	0.64 [b] [QU]	PP009B153; trial ns [Atreya *et al*., 1982, PP9/0702] (processing)
Suffolk, UK, 1982 (Desira)	Formulation ns (P)	1	0.75	ns	BBCH ns, before meeting rows, ns	ns	ns	21	1.3 [b] [QU]	PP009B153; trial ns [Atreya *et al*., 1982, PP9/0702] (processing)
Grantham, Lincolnshire, UK, 1983, (Record)	EC 125 (P)	1	0.38	0.14	BBCH ns (50% ground cover); 27 June	Sa	ns	70	< 0.03 [SS] [LOQ=0.05]	M3694B; NE41, [Dick and Atreya, 1984, PP5/0092]
Doddington, Lincolnshire, UK, 1983, (Record)	EC 125 (P)	1	0.38	0.15	BBCH ns (50% ground cover); 14 July	Sa	ns	70	0.05 [SS] [LOQ=0.05]	M3694B; NE42, [Dick and Atreya, 1984, PP5/0092]
St. Nicolas, Lincolnshire, UK, 1983, (Record)	EC 125 (P)	1	0.38	0.15	BBCH ns (50% ground cover); 14 July	ns	ns	70	< 0.03 [SS] [LOQ=0.05]	M3694B; NE43, [Dick and Atreya, 1984, PP5/0092]
Southery, Norfolk, UK, 1983, (Record)	EC 125 (P)	1	0.38	0.15	BBCH ns (50% ground cover); 22 June	peat	ns	70	< 0.03 [SS] [LOQ=0.05]	M3694B; EA1, [Dick and Atreya, 1984, PP5/0092]
Hockwold, Norfolk, UK, 1983, (Record)	EC 125 (P)	1	0.38	0.15	BBCH ns (50% ground cover); 22 June	peat	ns	70	< 0.03 [SS] [LOQ=0.05]	M3694B; EA2 [Dick and Atreya, 1984, PP5/0092]
Maidstone, Kent, UK, 1983, (Pentland Crown)	EC 125 (P)	1	0.38	0.13	BBCH ns (50% ground cover); 4 July	ns	ns	77	< 0.03 [SS] [LOQ=0.05]	M3694B; SE1 [Dick and Atreya, 1984, PP5/0092]
Ashford, Kent, UK, 1983, (Pentland Crown)	EC 125 (P)	1	0.38	0.13	BBCH ns (50% ground cover); 30 June	ns	ns	70	0.04 [SS] [LOQ=0.05]	M3694B; SE2 [Dick and Atreya, 1984, PP5/0092]
Cuckney, Nottinghamshire, UK, 1983, (Desiree)	EC 125 (P)	1	0.38	0.14	BBCH ns (50% ground cover); 27 June	ns	ns	67	0.20 [SS] [cntrl=0.05]	M3694B; NE52 [Dick and Atreya, 1984, PP5/0092]
St. Nicholas, Lincolnshire, UK, 1983, (Desiree)	EC 125 (P)	1	0.38	0.14	BBCH ns (50% ground cover); 27 June	ns	ns	64	< 0.03 [SS] [LOQ=0.05]	M3694B; NE53 [Dick and Atreya, 1984, PP5/0092]
Cullross, Fife, UK, 1983, (Desiree)	EC 125 (P)	1	0.38	0.19	BBCH ns (50% ground cover); 12 July	L	ns	70	0.03 [SS] [LOQ=0.05]	M3694B; SAI21 [Dick and Atreya, 1984, PP5/0092]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Ballinluig, Perthshire, UK, 1983, (Desiree)	EC 125 (P)	1	0.38	0.19	BBCH ns (50% ground cover); 7 July	Sa	ns	67	0.06 [SS]	M3694B; SAI22 [Dick and Atreya, 1984, PP5/0092]
South Powrie, Dundee, UK, 1983 (Desiree)	EC 125 (P)	1	0.38	0.19	BBCH ns (50% ground cover); 18 July	L	ns	70	0.08 [SS]	M3694B; SAI23 [Dick and Atreya, 1984, PP5/0092]
Ely, Cambridgeshire, UK, 1983, (Maris Piper)	EC 125 (P)	1	0.38	0.19	BBCH ns (50% ground cover); 16 June	peat	ns	70	0.10 [SS]	M3694B; NA12 [Dick and Atreya, 1984, PP5/0092]
Soham, Cambridgeshire, UK, 1983, (Maris Piper)	EC 125 (P)	1	0.38	0.19	BBCH ns (50% ground cover); 13 June	ns	ns	70	0.07 [SS]	M3694B; NA13 [Dick and Atreya, 1984, PP5/0092]
ns, UK, 1983 (Maris Piper)	EC 125 (P)	1	0.38	0.19	BBCH ns (50% ground cover); 15 July	ns	ns	70	0.07 [SS]	M3694B; NA14 [Dick and Atreya, 1984, PP5/0092]
March, Cambridgeshire, UK, 1983, (Maris Piper)	EC 125 (P)	1	0.38	0.19	BBCH ns (50% ground cover); 10 June	peat	ns	70	0.04 [SS] [LOQ=0.05]	M3694B; EM2 [Dick and Atreya, 1984, PP5/0092]
Christchurch, Cambridgeshire, UK, 1983 (Maris Piper)	EC 125 (P)	1	0.38	0.19	BBCH ns (50% ground cover); 24 June	L	ns	70	0.20 [SS]	M3694B; EM3 [Dick and Atreya, 1984, PP5/0092]
Woodbridge, Suffolk, UK, 2000 (Maris)	EC 125 (P)	1	0.38	0.19	BBCH ns; (20 cm; BBCH 34-35), 9 June	SaCL	81	54	0.03	RJ3200B; GB06-00-S070; [Mason and Henson, 2001, PP5/1091]
idem	EW 250 (P) + 0.5% TF8035	1	0.38	0.19	BBCH ns; (20 cm; BBCH 34-35), 9 June	SaCL	81	54	0.06	RJ3200B; GB06-00-S070; [Mason and Henson, 2001, PP5/1091]
Ely, Cambridgeshire, UK, 2000 (Maris)	EC 125 (P)	1	0.38	0.19	BBCH ns; (30 cm; BBCH 35-36), 16 June	Black fen peat	69 89	55 84	0.34 0.11	RJ3200B; GB06-00-S071; [Mason and Henson, 2001, PP5/1091]
idem	EW 250 (P) + 0.5% TF8035	1	0.38	0.19	BBCH ns; (30 cm; BBCH 35-36), 16 June	Black fen peat	69 89	55 84	0.35 0.07	RJ3200B; GB06-00-S071; [Mason and Henson, 2001, PP5/1091]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Whittles ford; Cambridge shire; UK; 2002; (Cara)	EC 125 (P)	1	0.13	0.042	pre BBCH leaf 30; plants not meeting between rows; 12 June;	SaL	CH	105	< 0.01	02-7068; plot 1; [Gill, 2003, PP5/1357]
idem	EC 125 (P)	1	0.25	0.083	pre BBCH leaf 30; plants not meeting between rows; 12 June;	SaL	CH	105	0.01	02-7068; plot 2; [Gill, 2003, PP5/1357]
Bracknell; Berkshire; UK; 2002; (King Edward)	EC 125 (P)	1	0.13	0.042	BBCH leaf 25-30; plants not meeting between rows; 20 June	SaL	99	89	< 0.01	02-7069; plot 1; [Gill, 2003, PP5/1359]
idem	EC 125 (P)	1	0.25	0.083	BBCH leaf 25-30; plants not meeting between rows; 20 June	SaL	99	89	< 0.01	02-7069; plot 2; [Gill, 2003, PP5/1359]
Eaton, Nottingham shire; UK; 2005; (Marfona)	EC 125 (P)	1	0.29	0.083	BBCH leaf 26; coverage ns; 7 June	SaL	49	121	< 0.01	CEMR-2688; AF/8653/ SY/1; [Bell, 2006, PP5/1487]
Nieuw Vennep, NL, 1984, (Bintje)	EC 125 (P) + Agral	1	0.38	0.075	BBCH ns (25 cm; 60% ground cover); 18 June	C	ns	56	0.06, 0.07, 0.09, 0.11; mean 0.08 [a] [SS] [LOQ=0.05]	M3977B; 84-227; [Dick and Rounds, 1985, PP5/0094]
Weteringbrug, Netherlands, 1984, (Bintje)	EC 125 (P) + Agral	1	0.38	0.075	BBCH ns (25 cm; 60% ground cover; at 18 June	C	ns	56	0.07, 0.08, 0.11, 0.66; mean 0.23 [a] [SS]	M3977B; 84-228; [Dick and Rounds, 1985, PP5/0094]
Balkbrug, Netherlands, 1984, (Prominent)	EC 125 (P) + Agral	1	0.38 + Agral	0.075	BBCH ns (35 cm; 60% ground cover);	Sa	green	58	0.03, 0.05 (2), 0.08; mean 0.05 [a] [SS]	M3977B; 84-143; [Dick and Rounds, 1985, PP5/0094]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
					16 June				[LOQ=0.05]	
Borgercompagnie, Netherlands, 1984, (Elkana)	EC 125 (P) + Agral	1	0.38	0.075	BBCH ns (40 cm; 40% ground cover); 8 June	Sa	green	60	< 0.03 (4), mean < 0.03 [a] [SS] [LOQ=0.05]	M3977B; 84-142; [Dick and Rounds, 1985, PP5/0094]
Klundert, Netherlands, 1984, (Bintje)	EC 125 (P) + Agral	1	0.38	0.075	BBCH ns (30 cm; 40% ground cover; 15 June	C	[d]	60	< 0.03 (4), mean < 0.03 [a] [SS] [LOQ=0.05]	M3977B; 84-327; [Dick and Rounds, 1985, PP5/0094]
Kruisland, Netherlands, 1984, (Bintje)	EC 125 (P) + Agral	1	0.38	0.075	BBCH ns (30 cm; 60% ground cover); 15 June	C	[d]	60	< 0.03 (3); 0.04; mean 0.03 [a] [SS] [LOQ=0.05]	M3977B; 84-328; [Dick and Rounds, 1985, PP5/0094]
Saint Sardos; Tarn et Garonne; S-France; 2001; (Lisetta)	EC 125 (P)	1	0.12	0.040	BBCH 31; 14 June	L	CH	29	< 0.01	RJ3295B; AF/5814/SY1 [Mason, 2002, PP5/1241]
idem	EC 125 (P)	1	0.12	0.040	BBCH 33 20 Jun	L	CH	23	0.04	idem
idem	EC 125 (P)	1	0.12	0.040	BBCH 36 27 Jun	L	CH	16	< 0.01	idem
idem	EC 125 (P)	1	0.19	0.061	BBCH 31 14 June	L	CH	29	0.03	idem
idem	EC 125 (P)	1	0.19	0.061	BBCH 33 20 Jun	L	CH	23	0.02	idem
idem	EC 125 (P)	1	0.19	0.060	BBCH 36 27 Jun	L	CH	16	< 0.01	idem
idem	EC 125 (P)	1	0.25	0.080	BBCH 31 14 June	L	CH	29	0.11	idem
idem	EC 125 (P)	1	0.25	0.080	BBCH 33 20 Jun	L	CH	23	0.19	idem
idem	EC 125 (P)	1	0.25	0.083	BBCH 36 27 Jun	L	CH	16	< 0.01	idem
St Martial; Tarn et Garonne; S-France; 2001 (Mona Lisa)	EC 125 (P)	1	0.12	0.041	BBCH 23 5 Sept	C	CH	44	0.03	RJ3295B; AF/5814/SY2 [Mason, 2002, PP5/1241]
idem	EC 125	1	0.12	0.042	BBCH 32	C	CH	37	0.11	idem

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	(P)				12 Sept					
idem	EC 125 (P)	1	0.12	0.065	BBCH 35 21 Sept	C	CH	28	0.10	idem
idem	EC 125 (P)	1	0.19	0.065	BBCH 23 5 Sept	C	CH	44	0.31	idem
idem	EC 125 (P)	1	0.19	0.062	BBCH 32 12 Sept	C	CH	37	0.23	idem
idem	EC 125 (P)	1	0.19	0.062	BBCH 35 21 Sept	C	CH	28	0.57	idem
idem	EC 125 (P)	1	0.25	0.080	BBCH 23 5 Sept	C	CH	44	0.31	idem
idem	EC 125 (P)	1	0.25	0.080	BBCH 32 12 Sept	C	CH	37	0.62	idem
idem	EC 125 (P)	1	0.25	0.083	BBCH 35 21 Sept	C	CH	28	0.44	idem
31790; St Jory; S-France; 2003; (Charlotte)	EC 125 (P)	1	0.25	0.083	BBCH leaf 24; before rows closing; 23 May	SaCL	89	90	< 0.01	03-7056; AF/7294/SY/1; [Mason, 2004, PP5/1424]
69830 St George de Remains; S-France; 2003; (Delicatess)	EC 125 (P)	1	0.25	0.083	BBCH 17; soil cover ns; 19 June	Sa	49	77	< 0.01	03-7057; AF/7295/SY1; [Mason, 2004, PP5/1411]
82440 Cayrac; S-France; 2003; (Mona Lisa)	EC 125 (P)	1	0.25	0.083	BBCH 29; 19 May	LC	93	67	< 0.01	03-7047; 03-7047; [Mason, 2004, PP5/1419]
34590; Marsillar gues; S-France; 2003; (Agata)	EC 125 (P)	1	0.25	0.085	BBCH 29; 20 May	SaCL	99	57	0.02	03-7048; 03-7048; [Mason, 2004, PP5/1420]
31790; St Jory, S-France; 2004; (Charlotte)	EC 125 (P)	1	0.19	0.063	BBCH leaf 19; soil cover 20%; 14 June	SaCL	49	77	< 0.01	CEMR-2309; AF/7839/SY/1; [Kang, 2005; PP5/1440]
82000, St Martial, S-France; 2004; (Mona Lisa)	EC 125 (P)	1	0.19	0.063	BBCH leaf 24; soil cover 30%; 16 July	C	49	74	< 0.01	CEMR-2309; AF/7839/SY/2; [Kang, 2005; PP5/1440]
Vinzelles; 71680; N-France; 2005;	EC 125 (P)	1	0.25	0.083	BBCH leaf 22; crop cover ns;	CL	49	90	< 0.01	CEMR-2688; AF/8653/ SY/2; [Bell, 2006, PP5/1487]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Anais)					3 June					
84170, Monteux; S-France; 2006; (Monalisa)	EC 125 (P)	1	0.25	0.083	BBCH 40; soil cover ns; 2 June	CaC	97	90	< 0.01	CEMR-3374; FR-HR-06-0127; [Bell, 2008, A12791B_10432]
34590, Marsillargues; S-France; 2006; (Liseta)	EC 125 (P)	1	0.25	0.083	BBCH 38; soil cover ns; 24 May	SiC	97	90	< 0.01	CEMR-3374; FR-HR-06-0128 [Bell, 2008, A12791B_10432]
Livanates Lokridos; Greece; 2003; (Spunta)	EC 125 (P)	1	0.25	0.083	BBCH 30; 29 Sept	SaL	89	58	0.07	03-7079; 03-7079; [Mason, 2004, PP5/1414]
idem	EC 125 (P)	1	0.25	0.083	BBCH 30; 29 Sept	SaCL	89	67	0.30	03-7080; 03-7080; [Mason, 2004; PP5/1415]
40061 Tintoria Minerbo; Bologna; Italy; 2003; (Agata)	EC 125 (P)	1	0.25	0.083	BBCH leaf 15-19; soil cover ns; 5 May	ns	49	77	< 0.01	03-7037; AF/7070/SY/1; [Mason, 2004, PP5/1410]
40052 Baricella; Emilia Romagna; Italy; 2003; (Kuroda)	EC 125 (P)	1	0.25	0.083	BBCH leaf 15 – 19; soil cover ns; 5 May	ns	49	92	< 0.01	03-7038; AF/7071/SY/1; [Mason, 2004; PP5/1408]
Trigueros; 21620 Huelva; Spain; 2002; (Espunta)	EC 125 (P)	1	0.20	0.065	BBCH 19; soil cover ns; 22 April	L	49	71	0.03	02-7044; plot 2; [Gill, 2003, PP5/1355]
Gurendes; 01427 Álava; Spain; 2002; (Hermes)	EC 125 (P)	1	0.19	0.063	BBCH leaf 33 – 34; soil cover ns; 21 June	L	99	94	< 0.01	02-7045; plot 2; [Gill, 2003, PP5/1353]
Tamarite de Litera; 22550 Huesca; Spain; 2003; (Kennebec)	EC 125 (P)	1	0.19	0.063	BBCH 30; soil cover ns; 14 May	LC	49	69	< 0.01	03-7027; Trial 2/0; [Mason, 2004; PP5/1418]
Villanañe; Álava Spain; 2003; (Hermes)	EC 125 (P)	1	0.19	0.075	BBCH 30; soil cover ns; 27 June	L	CH	80	< 0.01	03-7028; plot 3; [Mason, 2004; PP5/1416]
Corio del Rio; Sevilla; Spain; 2003;	EC 125 (P)	1	0.25	0.083	BBCH 30; 3 Apr	SaCL	47	49	< 0.01	03-7030; 03-7030; [Mason, 2004; PP5/1417]

POTATO Location; Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Agria)										
Salillas; 50290 Zaragoza; Spain; 2005; (Red Pontiak)	EC 125 (P)	1	0.25	0.083	BBCH 27; soil cover ns; 19 May	SaL	99	56	< 0.01	CEMR-2689; AF/8654/SY/1; [Bell, 2006, PP5/1486]
Villareal; 50490; Spain; 2005; (Agria)	EC 125 (P)	1	0.25	0.083	BBCH 27 – 29; soil cover ns; 14 June	SaCL	49	98	< 0.01	CEMR-2689; AF/8654/SY/2; [Bell, 2006, PP5/1486]
Soto de Cerrato; Palencia, 34208, Spain; 2006; (Hermes)	EC 125 (P)	1	0.24	0.083	BBCH 41 – 51; soil cover ns; 14 June	L	95-97	89	< 0.01	CEMR-3375; ES-HR-06-0017; [Bell, 2007, PP5/1555]
Petrajas de San Esteban, Valladolid 47430, Spain; 2006; (Monalisa)	EC 125 (P)	1	0.26	0.13	BBCH 41– 51; soil cover ns; 14 June	Sa	97-98	84	< 0.01	CEMR-3375; ES-HR-06-0018; [Bell, 2007, PP5/1555]
Banded foliar spray over rows										
Brenes; Seville; Spain; 2000; (Frisia)	EC 125 (P)	1	0.29	0.10	BBCH 19; soil cover ns; 3 March	LC	45 49	63 91	0.11 0.05	RJ3222B; ES50-00-S016; plot 2 [Ryan and Iniesta, 2002, PP5/1149]
idem	EC 125 (P)	1	0.39	0.13	BBCH 19; soil cover ns; 3 March	LC	45 49	63 91	0.20 0.17	RJ3222B; ES50-00-S016; plot 3 [Ryan and Iniesta, 2002, PP5/1149]
Alcalá del Río; Seville; Spain; 2000; (Hermes)	EC 125 (P)	1	0.34	0.10	BBCH 35; soil cover ns; 29 March	L	CH	63 64	< 0.01 < 0.01	RJ3222B; ES51-00-S116; plot 2 [Ryan and Iniesta, 2002, PP5/1149]
idem	EC 125 (P)	1	0.38	0.13	BBCH 35; soil cover ns; 29 March	L	CH	63 64	< 0.01 < 0.01	RJ3222B; ES51-00-S116; prot 3 [Ryan and Iniesta, 2002, PP5/1149]

BBCH 30-39: main stem elongation (10-90% of plants meet between rows = 10-90% crop cover);

BBCH 40-49: tuber formation (10-90% of total final tuber mass reached).

[QU] Quality of the study insufficient for MRL derivation

[SS] Samples size not stated (M3694B, M3977B).

[cntrl] Residue level found in the untreated control sample

[a] Mean from two replicate field samples is taken for MRL derivation if according to cGAP.

[b] PP009B153: Results calculated from flesh and peel fractions (total residues/total weight).

[c] CER 02808/08 T455 and T457: mean of 2-3 replicate analyses (original analysis and 1-2 re-analyses of the sample)

[d] M3977B 84-327 and 84-328: harvest about 4 weeks before desiccation of the the potatoes

Additional trial information:

RJ0226B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 or 62/1 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries not stated. Control samples were < 0.02 mg/kg.

CER02606/07. GLP study. No unsusual weather conditions. An old and a new formulation of fluazifop-P-butyl were applied to side-by-side plots by broadcast foliar spraying. Two samples of potato tubers (2.7-5.1 kg) were collected at earliest normal commercial harvest at each treated plot. Storage at -10 °C for 94-142 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method CER 2605 with a valid LOQ of 0.01 mg/kg**. Average concurrent recoveries were 94 - 107 % at 0.01–0.25 mg/kg. Control samples < 0.0033 mg/kg

CER02608/08. GLP study. No unusual weather conditions. An old and a new formulation of fluazifop-P-butyl were applied at nominal rates of 1 x 250 g ai/ha to side-by-side plots by broadcast foliar spraying. Two samples (1.68-5.2 kg) of potato tubers were collected at earliest normal commercial harvest at each treated plot, with preharvest intervals (PHI) of 44-46 days and 37-68 days for decline trials. Storage at -10 °C for 63-143 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method CER 2608 with a valid LOQ of 0.01 mg/kg**. Hydrolysis with 2 M HCl. Although no radiovalidation is conducted, highest residue values were obtained with this method, indicating that hydrolysis is sufficient for fluazifop conjugates in potatoes. Average concurrent recoveries were 84-107% at 0.01-1.0 mg/kg. Control samples < 0.0033 mg/kg.

M11031; GLP study. No unsual weather conditions. Foliar application by knapsack sprayer, spray volume 100 L/ha. Plot size 30 m^2.Tubers (at least 2 kg) were sampled at twelve points by hand, one sample per plot. Potatoes were washed in running water to remove excess soil and then dried with paper towels. Samples were stored at -20 °C for 3.8-6.6 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET 138 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual method recoveries (82-83%, n = 2, 0.1 mg/kg). Control samples were < 0.01 mg/kg. Method validation was performed at fortification levels 0.01 and 0.1 mg/kg, with mean recoveries of 83 and 78%, respectively, n = 7 and 5, respectively.

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Spray volume not stated. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.03 mg/kg.

M3676B GLP. No unusual weather conditions. Spray knapsack sprayer with 2.5 m boom. Spray volume 400 L/ha. Field samples (5 kg). Storage at -18 °C; storage time not stated but less than 12 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg.** Samples were corrected for mean internal standard recovery (57-106% at 0.5 mg/kg, n = 24). Control samples < 0.05 mg/kg.

RJ1405B GLP No unusual weather conditions. Hand held small plot sprayers. Spray volume 300 L/ha. Field samples 12 plants (>2 kg). Storage at -18 °C for up to 6 months. Samples were analysed for total fluazifop using NMR method ARAM 197 **with a valid LOQ of 0.05 mg/kg**. Average concurrent method recoveries 76-97% at 0.05-0.5 mg/kg. Control samples were < 0.05 mg/kg.

AZ13430/93. GLP. Processing study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volume not stated. **GC-MS Method P-14.077 with a valid** LOQ of 0.01 mg/kg. Recoveries from fortified control samples (0.01 and 0.1 mg/kg) ranged from 69-114% (mean 91% (n = 19). Control samples were < 0.01 mg/kg in all matrices.

GPO11501; GLP study. No unusual weather conditions. Foliar application by mobile plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled randomly from 12 plants when the crop was ripe for harvest. Samples were stored at -18 ºC for 120 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (99-110%, n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

GPO31501; GLP study. No unusual weather conditions. Foliar application by mobile plot sprayer, spray volume 300 L/ha. Tubers were sampled randomly from 12 plants when the crop was ripe for harvest. Tubers (>2 kg; >12 tubers/sample), except in gpo31501 plot 2 (1.8 kg; 12 tubers); gpo41505 plot 3 (1.9 kg; 12 tubers). Since a sufficient number of tubers is taken, results can be used for MRL derivation if according to cGAP. Samples were stored at -18 ºC for 85 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (104-105% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

GPO41501; GLP study. No unusual weather conditions. Foliar application by mobile plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample, except where indicated) were sampled randomly from 12 plants when the crop was ripe for harvest. Samples were stored at -18 ºC for 100 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (103-104% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

GPO91501; GLP study. No unusual weather conditions. Application by mobile plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; 36 tubers/sample) were sampled randomly from 12 plants when the crop was ripe for harvest. Samples were stored at -18 ºC for 104 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02**

with a valid LOQ of 0.01 mg/kg. Samples were not corrected for individual concurrent method recoveries (95-99% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

GPO079002; GLP study. No unusual weather conditions. Application by plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled at harvest. Samples were stored at -18 ºC for 137 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (90-99% n = 1/level, 0.02–0.05 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

GPO023103; GLP study. No unusual weather conditions. Application by plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled at harvest. Samples were stored at -18 ºC for 172 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (90-103% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

PP009B153 non-GLP study. Processing study. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Tubers were harvested at 3-10 weeks interval. Storage conditions were not reported. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM62 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recoveries at 1 mg/kg were 80% (n = 7) in uncooked flesh. Control samples were not reported.

M3694B. Non-GLP No unusual weather conditions. CO2 plot sprayer or CO2 knapsack sprayer with handheld spray boom Spray volume 200-280 L/ha. Sample size not stated (laboratory samples 500 g). Storage at -18 °C for up to 1.5 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Samples were corrected for mean internal standard recoveries (83-97% at 1.0-2.0 mg/kg). Control samples < 0.03 mg/kg, except 0.03 mg/kg in trial EM2 and 0.05 mg/kg in trial NE52. The LOQ for trial NE52 needs to be increased to 0.05/0.3=0.2 mg/kg.

RJ3200B GLP. No unusual weather conditions. Broadcast foliar spray. Application equipment not stated. Spray volume 200 L/ha. Sampling by hand, systematically across plots. Field samples 24 tubers or 2 kg. Samples were stored at -18 °C for 39-58 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for mean concurrent recoveries (85-102% at 0.05-0.5 mg/kg). Control samples were < 0.01 mg/kg

02-7068; GLP study. No unusual weather conditions. Foliar application by boom sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled randomly from five points across the middle rows at normal harvest. Samples were stored at -18 ºC for 138 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (86-98% n = 1/level, 0.02–0.05 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

02-7069; GLP study. No unusual weather conditions. Foliar application by one man hand-held boom, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled in zig-zag pattern from five points across the middle rows at normal harvest. Samples were stored at -18 ºC for 36 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (100-105% n = 1/level, 0.1–0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

CEMR-2688; GLP study. No unusual weather conditions. Foliar application by plot sprayer, spray volume 300-342 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand at normal harvest. Samples were stored at -9ºC or lower for a maximum of 281 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (89-102% n = 1/level, 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg (n = 2).

M3977B. GLP. No unusual weather condtions. Gas knapsack sprayer. Spray volume 500 L/ha. Sample size not stated. Four replicate field samples were taken. Storage at -18 °C; storage time not stated but less than 12 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Samples were corrected for mean internal standard recovery (83% at 0.5 mg/kg) . Control samples < 0.03 mg/kg.

RJ3295B; GLP study. Unusual weather conditions did not affect crop health. Overall foliar spray by precision boom sprayer, spray volume 290-311 L/ha. Tubers (>2 kg; 24 tubers) were sampled by hand and taken systematically from at least 12 different plants. Samples were stored at -16ºC or lower for 83-181 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (97-104% at 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg.

03-7056; GLP study. No unusual weather conditions. Foliar application by plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand at normal harvest. Samples were stored at -18ºC or lower for a maximum of 152 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (81-104% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

03-7057; GLP study. No unusual weather conditions. Foliar application by plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand at normal harvest. Samples were stored at -18ºC or lower for a maximum of 61 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (87-94% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

03-7047; GLP study. No unusual weather conditions. Foliar application by knapsack sprayer, spray volume 300 L/ha. Tubers (4.3 kg; no of tubers/sample not stated) were sampled by hand. Samples were stored at -18ºC or lower for 97days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (100-109% at 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg.

03-7048; GLP study. No unusual weather conditions. Foliar application by knapsack sprayer, spray volume 300 L/ha. Tubers (3.45 kg; no of tubers/sample not stated) were sampled by hand. Samples were stored at -18ºC or lower for 106 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (93-104% at 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg.

CEMS-2309; GLP study. No unusual weather conditions; plot SY1 had rain at application. Foliar application by plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand at normal harvest. Samples were stored at -11ºC or lower for a maximum of 235 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (90-92% n = 1/level, 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg (n = 2).

CEMR-3374; GLP study. No unusual weather conditions. Foliar application by knapsack sprayer, spray volume 300 L/ha. Tubers (>3.3 kg; number of tubers/sample not stated) were sampled by hand at normal harvest. Samples were stored at -16ºC or lower for a maximum of 381days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (72-81% n = 1/level, 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg (n = 2).

03-7079; GLP study. No unusual weather conditions. Foliar application by knapsack sprayer, spray volume 300 L/ha. Tubers (3.7 kg; 27 tubers) were sampled by hand in an S pattern; 4-5 tubers from 6 plants. Samples were stored at -18ºC or lower for 61 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287**/02 with a valid LOQ of 0.01 mg/kg. Samples were not corrected for individual concurrent method recoveries (76-107% at 0.05-2.0 mg/kg). Control samples were < 0.01 mg/kg.

03-7080; GLP study. No unusual weather conditions. Foliar application by knapsack sprayer, spray volume 300 L/ha. Tubers (4.0 kg; 28 tubers) were sampled by hand in an S pattern; 4-5 tubers from 6 plants. Samples were stored at -18ºC or lower for 52 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (88-91% at 0.05-2.0 mg/kg). Control samples were < 0.01 mg/kg.

03-7037; GLP study. No unusual weather conditions. Foliar application plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand at normal harvest. Samples were stored at -18ºC or lower for a maximum of 106 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (92-95% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

03-7038; GLP study. No unusual weather conditions. Foliar application plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand at normal harvest. Samples were stored at -18ºC or lower for a maximum of 91 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (91-92% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

02-7044; GLP study. No unusual weather conditions. Foliar application by knapsack sprayer, spray volume 305 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand avoiding plot edges. Samples were stored at -18ºC or lower for a maximum of 113 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (102-106% n = 1/level, 0.1–0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

02-7045; GLP study. No unusual weather conditions. Foliar application by knapsack sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand. Samples were stored at -18ºC or lower for a maximum of 140 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (109-111% n = 1/level, 0.02–0.5 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

03-7027; GLP study. No unusual weather conditions. Foliar application by knapsack sprayer, spray volume 307 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand. Samples were stored at -18ºC or lower for a maximum of 185 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (91-106% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

03-7028; GLP study. No unusual weather conditions. Foliar application by knapsack sprayer, spray volume 250 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand from 8 plants in the middle rows. Samples were stored at -16ºC or lower for a maximum of 120 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (86-103% n = 1/level, 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg (n = 1).

03-7030; GLP study. No unusual weather conditions. Overallapplication by plot sprayer, spray volume 295 L/ha. Tubers (2.0 kg; 24 tubers) were sampled by hand. Samples were stored at -18ºC or lower for 264 days. Samples were analysed for

total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (73-94% at 0.05-0.2 mg/kg). Control samples were < 0.01 mg/kg.

CEMS-2689; GLP study. No unusual weather conditions. Foliar application by plot sprayer, spray volume 300 L/ha. Tubers (>2 kg; >12 tubers/sample) were sampled by hand. Samples were stored at -9ºC or lower for a maximum of 330 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (86-103% n = 1/level, 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg (n = 2).

CEMR-3375; GLP study. Rain (10 mm) within 24 hrs of application (both trials). Foliar application by knapsack sprayer, spray volume 211-294 L/ha. Tubers (3.5-5.0 kg; no of tubers/sample not stated) were sampled by hand. Samples were stored at -16ºC or lower for a maximum of 366 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (72-81% n = 1/level, 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg (n = 2).

RJ3222B; GLP study. No unusual weather conditions. **Banded spray over rows,** spray volume 281-322 L/ha. Tubers (kg not stated; 24-29 tubers/sample) were sampled by hand and were taken systematically from acros the plots. Samples were stored at -18ºC or lower for a maximum of 104 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recoveries (98-101% n = 1/level, 0.05-0.1 mg/kg). Control samples were < 0.01 mg/kg (n = 2).

Radish

Two cGAPs for radish are available:

- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 42 days
- cGAP from Belgium with 1 ×0.38 kg ai/ha and PHI of 56 days

Trials that could be matched to these cGAPs were summarized.

Table 225 lists trials conducted in the the UK (1980). A broadcast foliar spray application with fluazifop-butyl (racemate) was conducted under the conditions listed in Table 225. Results marked with "[QU]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62, 62/1.

Table 225 Supervised field trials on radish (roots), treated with a broadcast foliar fluazifop-butyl spray

RADISH ROOTS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Location ns; UK, 1980 (radish; var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	55	< 0.02 (3 ×) [QU] [LOQ=0.05]	RJ0226B summary [Atreya and Froggatt, 1981, PP9/0384]
idem	idem	1	1.5	ns	GS ns date ns	ns	ns	55	< 0.02 (3 ×) [QU] [LOQ=0.05]	idem
idem	idem	1	2.0	ns	GS ns date ns	ns	ns	55	< 0.02 (3 ×) [QU] [LOQ=0.05]	idem

Additional trial information:

RJ0226B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Storage conditions not stated. Samples were analysed for total fluazifop using HPLC-UV method PPRAM 62 or 62/1. Concurrent method recoveries not stated. Control samples were < 0.02 mg/kg.

Sugar beet and fodder beet roots

Two cGAPs for sugar beets are available:

- cGAP from the USA with 2 × 0.42 kg ai/ha and a PHI of 90 days (underlining nn)
- cGAP from the Netherlands, Belgium and the UK with 1 ×0.38 kg ai/ha and a PHI of 56 days (underlining nn)

Trials that could be matched to these cGAPs were summarized.

Table 226 and Table 227 list trials on sugar beet roots and fodder beet roots conducted in the USA (2000), Germany (1981, 1983, 1992, 2002), the UK (1981), Denmark (1988), Sweden (1981), France (1985, 2004), Greece (1997), Italy (1998) and Spain (1997, 1998, 1999). A broadcast or banded foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 226 and Table 227. Results marked with "[QU]", or "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 12 plants (i.e. at least 4 kg).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2.

[Atreya and Froggatt, 1981, PP9/0384, report RJ0226B] summarized the results of several residue trials carried out with fluazifop-butyl in 1979 and 1980 in the UK (1979: 1 ×1.0 kg ai/ha, PHI 0-21 days or 115-119 and 134-137 days for roots[FB+F]; 1980: 1 ×1.0-2.0-4.0 kg ai/ha, PHI 77-118 days, roots and tops[FP+F] and two decline studies 1 ×1.0 kg ai/ha, PHI 0, 21/20 , 42, 75/81, 106/112 days[FP+F]). Additional trials were carried out in 1980 in the Netherlands with 1 ×0.25-1.0 kg ai/ha, PHI 140 days, root and top. Four decline trails were performed in Germany in 1980 with 1 ×1.0 kg ai/ha and PHI 0, 19-23, 41-43, 62-64, 85-86, 112-113 days, root and tops[FP+F]). Additional trials were carried out in Canada in 1979 with 1 ×0.125-0.5-1.0 kg ai/ha, PHI 123 days, roots and tops[FB+F] and in 1980 with 1 ×0.25-0.5 kg ai/ha, PHI 111 days, for root and tops. The summary report contains data on a broad variety of crops, but no trial location details, sampling details or storage information. HPLC-UV methods PPRAM 51 (determines fluazifop-butyl) and PPRAM52 (determines free fluazifop acid (II)) were used in the majority of these residue trials, indicated with [FP+F] and these were addressed in the metabolism section. For the remaining trials HPLC-UV method PPRAM62 (total fluazifop) was applied. Some of the residue trials reported in [RJ0226B] were also reported in individual study reports; Canada 1979 [Atreya *et al*, 1980, PP9/0499, report 369/PP009B005], UK 1979 [Atreya *et al*, 1980, PP9/0502, report PP009B008], Germany [Atreya *et al*, 1981, PP9/0508, report PP009B014]. The original study report of the trials performed in the UK in 1980 were submitted [Atreya, 1981, PP9/0512, report PP009B019].These trials were not summarized because they would not assist in MRL setting

Additional trials were performed on 15 locations in the USA in 1981 and 1982 [Koubek, 1983, 405726, report TMU 1211/B]. The application rates could not be matched to the GAPs; 1 ×0.56 kg ai/ha and harvest at 112 DAT, 1 ×0.84 or 1.12 kg ai/ha and harvest at 108 DAT, 2 × 0.56 kg ai/ha and harvest at 42, 72, 120 DAT, 2 ×1.12 kg ai/ha and harvest at 33, 56, 57, 58, 59, 60, 129, 133, 134 DAT, or 2 × 0.84 kg ai/ha and harvest at 56 DAT. The trials were not summarized because they would not assist in MRL setting

[Atreya and Harradine, 1982, PP9/0062, report RJ0291B] summarized the results of several residue trials carried out with fluazifop-butyl on sugar beets and fodder beets in the UK (1981: 2 ×0.38 kg ai/ha, PHI 29-78 days, 1 ×0.19 kg ai/ha, PHI 63-77 days or 1 × 0.75-1.5 kg ai/ha, PHI 43-46 or 63-84 days in foliage and roots), Canada (1981: 1 ×0.25-0.35-0.40 kg ai/ha, PHI 74-87 days),

Sweden (1981: 1 × 0.50 kg ai/ha, PHI 86-103 days). These trials were not summarized because they would not assist in MRL setting.

CF3-pyridone (X)

Besides total fluazifop, also CF3-pyridone (X) was analysed in sugar beet roots from some 1981, 1982, 1983 and 1984 trials conducted in the USA [Atreya, 1984, PP9/0731, report PP009B290; Dick and Rounds, 1985, PP5/0238, report M4041B]. These trials were summarized in the metabolism section.

Table 226 Supervised field trials on sugar beet (roots), treated with a broadcast or banded foliar fluazifop-butyl spray

SUGAR BEET ROOTS Location; country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Campbell, MN, USA, 2000 (Resist)	EC 240 (P) +0.5% NIS	2 (12)	0.42 0.42	0.22 0.22	post emergence; 30 June	CL	28 Sept, 2000	90	0.06, 0.05 mean: 0.06	RR 00-066B 255 (SBMN1) [Stewart, 2001, PP5/1070]
Geneva, MN, USA, 2000 (Crystal 205)	EC 240 (P) +1% COC	2 (14)	0.42 0.42	0.52 0.50	post emergence; 18 July	CL	16 Oct, 2000	90	0.09, 0.10 mean: 0.10	RR 00-066B 256 (SBMN2) [Stewart, 2001, PP5/1070]
Brampton, ND, USA, 2000 (Beta 6104)	EC 240 (P) +0.5% NIS	2 (14)	0.42 0.42	0.36 0.36	post emergence; 7 July	SiL	04 Oct, 2000	89	0.08, 0.07 mean: 0.08	RR 00-066B 257 (SBND1) [Stewart, 2001, PP5/1070]
Northwood, ND, USA, 2000 (Beta 6600)	EC 240 (P) +1% COC	2 (14)	0.42 0.42	0.23 0.23	post emergence; 11 July	SiL	09 Oct, 2000	90	0.05, 0.07 mean: 0.06	RR 00-066B 258 (SBND2) [Stewart, 2001, PP5/1070]
Delavan, WI, USA, 2000 (American Crystal 196)	EC 240 (P) +0.5% NIS	2 (13)	0.42 0.42	0.46 0.47	post emergence; 27 July	SiL	25 Oct, 2000	90	0.06, 0.07 mean: 0.06	RR 00-066B 259 (SBWI1) [Stewart, 2001, PP5/1070]
Grand Island, NE, USA, 2000 (HM1605)	EC 240 (P) +0.5% NIS	2 (14)	0.42 0.42	0.45 0.45	post emergence; 11 July	SiL	09 Oct, 2000	90	0.10, 0.11 mean:.0.10	RR 00-066B 260 (SBNE1) [Stewart, 2001, PP5/1070]
Larned, KS, USA, 2000 (Crystal 203)	EC 240 (P) +0.5% NIS	2 (14)	0.42 0.42	0.23 0.22	post emergence; 19 June	LSa	17 Sept, 2000	90	< 0.01, < 0.01 mean: < 0.01	RR 00-066B 261 (SBKS1) [Stewart, 2001, PP5/1070]
Edgar, MT, USA, 2000 (Mono-Hy)	EC 240 (P) +0.5% NIS	2 (12)	0.42 0.42	0.30 0.24	post emergence; 1 July	L	29 Sept, 2000	90	0.08, 0.08 mean: 0.08	RR 00-066B 262 (SBMT1) [Stewart, 2001, PP5/1070]
Porterville, CA, 2000, USA (Encrusted 8 1/2-9 1/2)	EC 240 (P) +0.5% NIS	2 (14)	0.42 0.42	0.22 0.22	post emergence; 25 July	SaL	23 Oct, 2000	90	0.02, 0.03 mean: 0.02	RR-00-066B 263 (SBCA1) [Stewart, 2001, PP5/1070]
Visalia, CA, USA, 2000 (SB-SS-NBSR)	EC 240 (P) +1% COC	2 (14)	0.42 0.42	0.23 0.23	post emergence; 15 August	fine SaL	13 Nov, 2000	90	0.05, 0.07 man: 0.06	RR-00-066B 264 (SBCA2) [Stewart, 2001, PP5/1070]
American Falls, ID, USA, 2000 (Beta 4490R)	EC 240 (P) +0.5% NIS	2 (13)	0.42 0.42	0.24 0.26	post emergence; 29 June	SiL	27 Sept, 2000	90	0.11, 0.11 mean: 0.11	RR-00-066B 265 (SBID1) [Stewart, 2001, PP5/1070]
Ephrata, WA,	EC 240	2	0.42	0.18	post	SaL	11	90	0.20, 0.23	RR-00-066B

SUGAR BEET ROOTS Location; country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
USA, 2000 (Canyon)	(P) +0.5% NIS	(14)	0.42	0.18	emergence; 13 July		Oct, 2000		mean: 0.22	266 (SBWA1) [Stewart, 2001, PP5/1070]
idem	EC 240 (P) +0.5% NIS	2 (14)	2.1 2.1	0.91; 0.91	post emergence; 13 July	SaL	11 Oct, 2000	90	0.85, 0.86 mean: 0.86	RR-00-070B 270 (SBWA1) RS-9307-B1 [Stewart, 2001, 406493] (processing)
Location ns; Sweden, 1981 (var ns)	EC 250 (rac)	2	0.50	ns	GS ns date ns	ns	ns	84 91	0.06 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	NA 0.09 < 0.05 < 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 19 42 61 85 111	NA 0.08 < 0.05 < 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 97	NA 0.13 0.08 < 0.05 < 0.05 < 0.05 [QU]	idem
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 95	NA 0.07 < 0.05 < 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	NA 0.12 < 0.05 < 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 19 42 61 85 111	NA 0.15 0.06 0.05 < 0.05 < 0.05	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]

SUGAR BEET ROOTS Location; country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
									[QU]	
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 21 42 63 84 97	NA 0.21 0.18 < 0.05 < 0.05 < 0.05 [QU]	idem
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 21 42 63 84 95	NA 0.17 0.08 < 0.05 < 0.05 < 0.05 [QU]	idem
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	NA < 0.05 < 0.05 < 0.05 NA < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 97	NA 0.33 0.05 < 0.05 < 0.05 < 0.05 [QU]	idem
idem	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 95	NA 0.21 0.06 < 0.05 < 0.05 < 0.05 [QU]	idem
6748 Bad Bergzabern; Germany; 1983; (Monopur)	EC 125 (P)	1	0.38	0.094	10-14 leaves; 20 cm tall; 23 June	ns	ns	0 21 42 62 93	0.05 0.21 0.07 0.06 < 0.02 [SS] [LOQ=0.05]	M3701B; RS 8369 E1(A); [Upton and Atreya, 1984; PP9/0054]
idem	EC 250 (rac)	1	0.75	0.19	10-14 leaves; 20 cm tall; 23 June	ns	ns	0 21 42/43 62/64 77 93/96	0.10 0.35 0.14 0.08 NA 0.30 [SS]	M3701B; RS 8369 E1[b]; [Upton and Atreya, 1984; PP9/0054]
6745 Offenbach; Germany;	EC 125 (P)	1	0.38	0.094	10-14 leaves; 20 cm tall;	ns	ns	0 21 42	0.04 0.21 0.06	M3701B; RS 8369 E2(A);

SUGAR BEET ROOTS Location; country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
1983; (Kawemono)					21 June			62 93	0.04 0.04 [SS] [LOQ=0.05]	[Upton and Atreya, 1984; PP9/0054]
idem	EC 250 (rac)	1	0.75	0.19	10-14 leaves; 20 cm tall; 21 June	ns	ns	0 21 42/43 62/64 77 93/96	0.10 0.23 0.06 0.07 NA < 0.02 [SS] [LOQ=0.05]	M3701B; RS 8369 E2[b]; [Upton and Atreya, 1984; PP9/0054]
2057 Schwarzenbek; Germany; 1983; (Nova Gema)	EC 125 (P)	1	0.38	0.094	12-14 leaves; 7 July	ns	ns	0 21 42 62 77	NA 0.56 0.19 0.12 0.08 [SS]	M3701B; RS 8369 B1(A); [Upton and Atreya, 1984; PP9/0054]
idem	EC 250 (rac)	1	0.75	0.19	12-14 leaves; 7 July	ns	ns	0 21 42/43 62/64 77 93/96	NA 0.46 0.39 0.20 0.15 NA [SS]	M3701B; RS 8369 B1[b]; [Upton and Atreya, 1984; PP9/0054]
7101 Cunnersdorf; Germany; 1992; (Hilma)	EC 125 (P)	1	0.38	0.12	BBA 45; at 3 July	SaL	43 49 90	0 47 90	< 0.05 0.07 0.05	RJ1424B; RF 12/92 CU [Bolygo, 1993; PP5/0098]
idem	ME 125 (P)	1	0.38	0.12	BBA 45; 3 July	SaL	43 49 90	0 47 90	< 0.05 <u>0.10</u> < 0.05	RJ1424B; RF 12/92 CU [Bolygo, 1993; PP5/0098]
2021 Rosenow; Germany; 1992; (Kawetina)	EC 125 (P)	1	0.38	0.12	BBA 27-41; 20 July	L	27 43 90	0 31 91	< 0.05 0.20 < 0.05	RJ1424B; RF 12/92 RO [Bolygo, 1993; PP5/0098]
idem	ME 125 (P)	1	0.38	0.12	BBA 27-4); 20 July	L	27 43 90	0 31 91	< 0.05 0.18 0.05	RJ1424B; RF 12/92 RO [Bolygo, 1993, PP5/0098]
Coblenz; Germany, 1992; (Kawetina)	EC 125 (P)	1	0.38	0.12	BBA 4); 30 June	C	43 49 90	0 49 90	< 0.05 <u>0.09</u> < 0.05	RJ1424B; RF 12/92 CO [Bolygo, 1993; PP5/0098]
idem	ME 125 (P)	1	0.38	0.12	BBA 4); 30 June	C	43 49 90	0 49 90	< 0.05 0.07 < 0.05	RJ1424B; RF 12/92 CO [Bolygo, 1993; PP5/0098]
5301 Kötschau; Germany, 1992; (Dunja)	EC 125 (P)	1	0.38	0.12	BBA 43-45; 1 July	L	43 49 90	0 49 90	< 0.05 <u>0.09</u> 0.05	RJ1424B; RF 12/92 KO [Bolygo, 1993; PP5/0098]
idem	ME	1	0.38	0.12	BBA 43-	L	43	0	< 0.05	RJ1424B;

SUGAR BEET ROOTS Location; country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	125 (P)				45; 1 July		49 90	49 90	0.09 0.06	RF 12/92 KO [Bolygo, 1993; PP5/0098]
Wallersdorf-See Germany, 2002 (Cyntia)	EC 125 (P)	1	0.38	0.12	BBCH 39-49; 65 cm tall; 90% crop cover; 29 July	CL	49	56	0.32; 0.32; Mean: 0.32 [a]	gsb064002 trial no ns; [Simon, 2003, PP5/1337]
Wallersdorf-See Germany, 2002 (Corinna)	EC 125 (P)	1	0.38	0.12	BBCH 49; 60-65 cm tall; 90% crop cover; 27 August	CL	MAT	52	0.25 0.27 Mean: 0.26	gsb064202 trial no ns [Simon, 2003, PP5/1336]
Location ns; UK, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	63	< 0.02 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; UK, 1982 (var ns)	EC 250 (rac)	2	1.5 1.0	ns ns	GS ns date ns	ns	ns	73	0.46	PP009B089 981-SBB-EA7 [Atreya, 1982, PP9/0366] (processing)
Reims; N-France, 1985 (Monostar)	EC 250 (P)	1	0.38	0.12	8-10 leaves; 17 June	Si	MAT	115	< 0.05 [SS]	D 26-EP; R145.85; [Massenot and Culoto, 1986, PP5/0096]
idem	EC 300 (P)	1	0.38	30	8-10 leaves; 17 June	Si	MAT	115	0.07 [SS]	idem
Tours; N-France, 1985 (Tosca)	EC 250 (P)	1	0.38	0.12	12 leaves; 11 June	Si	IMM IMM MAT	30 45 108	0.32 0.23 0.14 [SS]	D 26-EP TRS 211.85; [Massenot and Culoto, 1986, PP5/0096]
idem	EC 300 (P)	1	0.38	30	12 leaves; 11 June	Si	IMM IMM MAT	30 45 108	0.37 0.18 0.15 [SS]	idem
42110; Feurs; S-France, 2004 (Laetitia)	EC 125 (P)	1	0.37	0.12	BBCH 38; 21 July	LC	49	56	0.20 [SS]	CEMR-2310 AF/7840/SY/1; [Kang, 2005, PP5/1441]
42450; Sury le Comtal; S-France, 2004 (Laetitia)	EC 125 (P)	1	0.36	0.12	BBCH 39; 21 July	LC	49	56	0.13 [SS]	CEMR-2310 AF/7840/SY/2 [Kang, 2005, PP5/1441]
Guillena; Sevilla; Spain, 1997 (Lola)	EC 125 (P)	1	0.42	0.13	50-60 cm tall; 8 May	LC	MAT	60	0.05	RJ2553B ES10-97-SH003; [Mason *et al.*, 1999, PP5/0115]
idem	EC 125	1	0.41	0.14	50-60 cm tall;	LC	MAT	60	0.05	RJ2553B ES10-97-

SUGAR BEET ROOTS Location; country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	(P)				8 May					SH103 [Mason *et al*., 1999, PP5/0115]
S Agata Bolognese; Emilia Romagna; Italy, 1998 (Nubia)	EC 125 (P)	1	0.38	0.11	BBCH 39; 40-50 cm tall; 6 July	L	ns	56	0.08	RJ2779B IT20-98-H321; [Mason and Volpi; 1999, PP5/0124]
Voghera; Lombardia; Italy, 1998 (Asso)	EC 125 (P)	1	0.38	0.094	BBCH 39 – 41; 40-50 cm tall; 5 June	L	ns	56	0.09	RJ2779B IT30-98-H320; [Mason and Volpi; 1999, PP5/0124]
40016; Mascarino Venezzano; Italy, 2004 (Flavia)	EC 125 (P)	1	0.37	0.12	BBCH 39-49; 13 July	CL	49	57	0.14 [SS]	CEMR-2310 AF/7840/SY/3 [Kang, 2005, PP5/1441]
Banded foliar spray over rows										
Litohoro; Pieria; Greece, 1997 (Rizor)	EC 125 (P)	1	0.38	0.076	14-16 leaves; 45 cm tall; 16 July	LC	MAT	60	0.12	RJ2553B GR-97-H101 [Mason *et al*., 1999, PP5/0115]
Korinos; Pieria; Greece, 1997 (Rizor)	EC 125 (P)	1	0.37	0.075	12-14 leaves; 40 cm tall; 16 July	LC	MAT	61	0.08	RJ2553B GR-97-H102 [Mason *et al*., 1999, PP5/0115]
Biota; Zaragoza; Aragón; Spain, 1998 (Oryx)	EC 125 (P)	1	0.38	0.094	BBCH 85; 50 cm tall; 7 Oct	C	93	54	0.21 [b] [SS]	RJ2833B ES10-98-SH007; [Ryan and Gallardo, 1999, PP5/0121]
Biota; Zaragoza; Aragón; Spain, 1998 (Korif)	EC 125 (P)	1	0.37	0.094	BBCH 85; 50 cm tall; 7 Oct	C	93	54	0.13 [b] [SS]	RJ2833B ES10-98-SH107; [Ryan and Gallardo, 1999, PP5/0121]
Sevilla; Andalucia; Spain, 1999 (Lola)	EC 125 (P)	1	0.43	0.093	BBCH 49; 60 cm tall; 24 May	L	MAT	56	0.14	RJ2995B ES51-99-S021; [Mason and Gallardo, 2000, PP5/0308]

BBCH 27-29: leaf development; BBCH 30-39: rosette growth (leaves cover 10-90% of ground); BBCH 40-49: development of roots (beets have 10-90% of harvestable size).

GSH = IMM = immature (trial TRS 211.85, DAT 30-45 harvested at 12-16 leaves)

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[SS] Sample size not stated (M3701B, D26-EP) or less than the required 12 plants (1 kg in CEMR 2310, 1.4-3.4 kg in RJ2833).

[a] Replicate samples were taken from the same plot; the mean is taken for MRL derivation, if according to cGAP.

[b] Each field sample was analysed in triplicate; the mean result is reported in the table

Additional trial information:

RR-00-066B GLP study. Apart from early frost (no impact on study) no unusual weather conditions. Spray application bytractor mounted, equipment or backpack sprayer. Spray volume 75-280 L/ha. Sugar beet plants (at least 12 items) were sampled by hand. Roots and tops were separated. Duplicate samples consisted of at least 12 roots (>1.1 kg). They were stored frozen for up to 2.5 months. Samples were analysed for total fluazifop using **GC-MS method RR91-014B with a valid LOQ of 0.01 mg/kg**. Concurrent recoveries of total fluazifop ranged from 67-118% (0.01 and 1.0 mg/kg). Control samples were < 0.01 mg/kg.

RR-00-070B GLP study. Field trial performed for processing study. No unusual weather conditions. Spray application with tractor mounted, wasit mounted or backpack sprayer. Spray volume 93-280 L/ha. Sugar beet root samples were harvested for processing (167-170 kg). Tops were not collected They were stored frozen for up to 2.5 months prior to analysis. Samples were analysed for total fluazifop using **GC-MS method RR91-014B with a valid LOQ of 0.01 mg/kg**. Concurrent recoveries of total fluazifop ranged from 69-108%. Control samples were < 0.01 mg.kg.

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.03 mg/kg.

M3701B. Non GLP study. No unusual weather conditions. Application by knapsack sprayer with 1.5-2.5 boom. Spray volume 400 L/ha. Sample size not stated. Dry leaves were removed and roots were washed before analysis. Storage at -30°C, maximum 231 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (83% for roots at 0.5 mg/kg). Control samples < 0.02 mg/kg.

RJ1424B. GLP study. Weather conditions did not affect growth except at Rosenow, where the growth of the sugar beets was slower due to dry weather conditions. Application by mobile small plot sprayers. Spray volume 300 L/ha. Sample size 12 plants at each sampling interval > 2 kg roots except 0.9-1.9 kg roots at DAT 0 each location, Rosenow 1.8-2.0 kg roots at DAT31. Roots were briefly rinsed under running water. Storage at -18 °C for maximum 7 months. Samples were analysed for total fluazifop using **NMR method ARAM 197 with a valid LOQ of 0.05 mg/kg**. Concurrent mean internal standard recovery (90-91% at 0.5 mg/kg for roots). Control samples < 0.05 mg/kg.

gsb064002. GLP study. No unusual weather conditions. Broadcast foliar application by plot sprayer. Spray volume 300 L/ha. Sugar beet plants (12 plants, roots > 3 kg) were sampled by hand. Roots and tops were separated. Storage at -18°C, maximum 169 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recoveries (112-114% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

gsb064202. GLP study. No unusual weather conditions. Application by knapsack sprayer using a spray volum of 300 L/ha. 12 plants were sampled . Roots and tops were separated. Storage at -18°C, maximum 144 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Mean internal standard recovery (109-115% at 0.01-1.0 mg/kg). Control samples < 0.01 mg/kg.

PP009B089 Non GLP study. Poor quality of the study and aimed at processing. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (82, 91, and 87% at 0.1, 0.5, and 1.0 mg/kg in sugar beet). Control samples were not reported. Selected samples were methylated to the methyl ester of fluazifop and residues were confirmed by GC-MS.

D26-EP. Non GLP study. Weather conditions not stated. Application by electrodyne spray with ultra low volume (1.25 L/ha) or classic spray (300 L/ha). Sample size not stated. Tops were not collected. Storage at -20°C for 223-313 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/1 modification B with a valid LOQ of 0.05 mg/kg.**. Samples were not corrected for average concurrent recovery (76% at 0.1–0.2 mg/kg). Control samples < 0.05 mg/kg.

CEMR-2310. GLP study. No unusual weather conditions. Broadcast foliar spray using a plot sprayer. Spray volume 290-300 L/ha. Sugar beet roots (1 kg, no of items not stated, but weight too low) were harvested by hand. Storage at -17°C, maximum 285 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recoveries (103-113% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg

RJ2779B. GLP study. No unusual weather conditions. Broadcast foliar spray with motor knapsack sprayer with boom. Spray volume 350-400 L/ha. Sugar beet plants (12 items; 2.1-2.8 kg) were sampled by hand and taken systematically from across the plots. Roots were cleaned by rinsing in running water and light brushing (trial H320) or by hand (trial H31). The roots with tops and leaves were subsampled by dividing the roots, longitudinally, into four parts and retaining one quarter of each.. Roots and tops were separated thereafter. Storage at -18°C for 74-105 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for averageconcurrent recoveries (104% at 0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2553B. GLP study. No unusual weather conditions. **Banded spray over rows** using a CO2 knapsack sprayer with 2 m boom (Greece). Broadcast foliar spray using a knapsack sprayer with lance (Spain). Spray volume 330-500 L/ha Sugar beet roots (12 items) were sampled by hand and taken systematically from across the plots. Tops were not collected. The

roots from Greece were sub-sampled by dividing the roots, longitudinally, into four parts and retaining one quarter of each.. Storage at -6°C, or lower for 51-179 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recoveries (110-124% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

RJ2833B. GLP study. No unusual weather conditions. **Banded spray over rows** using a gas knapsack sprayer. Spray volume 400 L/ha. Sugar beet plants (1.45-3.45 kg; no of items not stated, but weight too low) were sampled by hand and taken systematically from across the plots. Roots and leaves were separated. Thereafter, the roots were sub-sampled by dividing the roots, longitudinally, into four parts and retaining one quarter of each. Storage at -18°C for 206-234 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were corrected for individual concurrent recoveries (95-112% at 0.05-0.5 mg/kg). Uncorrected results were not reported. Control samples < 0.01 mg/kg.

RJ2995B. GLP study. No unusual weather conditions. **Banded spray over rows** using a gas knapsack sprayer. Spray volume 464 L/ha. Sugar beet whole plants (3-6 kg, no of items not stated, but weight sufficient) were sampled by hand and taken systematically from across the plots. Roots and leaves were separated. Thereafter, the roots were sub-sampled by dividing the roots, longitudinally, into four parts and retaining one quarter of each. Storage at -18°C for 122-129 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recoveries (104-104% at 0.05-0.1 mg/kg). Control samples < 0.01 mg/kg.

Table 227 Supervised field trials on fodder beet (roots), treated with a broadcast foliar fluazifop-butyl spray

FODDER BEET ROOTS Location; country; year; (variety)	For-mu-lation	no. of appl	kg ai/ha	kg ai/hL	GS; at last treatment day	Soil type	GSH	DAT days	Total fluazifop[1] (mg/kg)	Code no; Report no; Trial no. [ref]
Almind; Denmark, 1988 (Magna-Mono)	EW 125 (P)	2; (30)	0.19 0.19	0.075 0.075	6-8 leaves; 50% crop cover; 19 June	L	ns	46 61 75	0.04 0.03 0.02 [SS]	M4870B DK10-88-H070; [Hayward and Harradine; 1989; PP5/0519]
Almind; Denmark, 1988; (Magna-Mono)	EW 125 (P)	1	0.38	0.19	4-5 leaves; 40% crop cover; 2 June	L	ns	44 63 77	0.04 0.01 < 0.01 [SS]	M4870B DK10-88-HI41 [Hayward and Harradine; 1989; PP5/0519]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	- 0.06 < 0.05 < 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 91	- 0.17 0.07 < 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	- 0.24 0.05 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 21 42	- 0.38 0.09	RJ0291B summary [Atreya and Harradine, 1982,

FODDER BEET ROOTS Location; country; year; (variety)	For-mu-lation	no. of appl	kg ai/ha	kg ai/hL	GS; at last treatment day	Soil type	GSH	DAT days	Total fluazifop[1] (mg/kg)	Code no; Report no; Trial no. [ref]
(var ns)								63 84 91	0.06 < 0.05 < 0.05 [QU]	PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	- 0.07 < 0.05 < 0.05 na < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
2057 Schwarzenbek; Germany; 1983; (Rote Eckern-dorfer)	EC 125 (P)	1	0.38	0.094	12-14 leaves; 7 July	ns	ns	21 42 62 77	0.71 0.21 0.15 0.07 [SS]	M3701B; RS 8369 B2A; [Upton and Atreya, 1984, PP9/0054]
idem	EC 250 (rac)	1	0.75	0.19	12-14 leaves; 7 July	ns	ns	21 42 63 77	1.4 0.38 0.04 0.11 [SS]	M3701B; RS 8369 B2B; [Upton and Atreya, 1984, PP9/0054]

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[SS] Sample size not stated (M3701B, M4870B) or less than the required 12 plants. Not suitable for MRL derivation.

Additional trial information:

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.03 mg/kg.

M3701B. Non GLP study. No unusual weather conditions. Application by knapsack sprayer with 1.5-2.5 boom. Spray volume 400 L/ha. Sample size not stated. Roots were washed before analysis. Storage at -30°C, maximum 231 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (83% at 0.5 mg/kg). Control samples < 0.02 mg/kg.

M4870B Non GLP study. No unusual weather conditions. Broadcast foliar spray application by plot sprayer. Spray volume 200-250 L/ha. Fodder beets were sampled by hand and roots and tops were separated. Sample size not stated.. Storage -18 ºC for a maximum of 138days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (91% at unknown level). Control samples < 0.01mg/kg.

Swede roots

Two cGAPs for swedes are available:

- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 42 days
- cGAP from the Netherlands, Belgium and the UK with 1 ×0.38 kg ai/ha and a PHI 56 days (underlining nn)

Trials that could be matched to these cGAPs were summarized.

Table 228 lists trials conducted in Canada (1984) and the UK (1981, 1984, 1989). The trials performed in Canada in 1984 were performed on rutabaga, a swede belonging to the mustard family *cruciferae* and genus *brassica*. A broadcast foliar spray application with fluazifop-butyl (racemate) or

fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 228. Results marked with "[QU]"or "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less than 12 plants (or 2 kg).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2.

One report with supervised trials on swedes could not be retrieved [Atreya, 1983, PP009B216, not referenced]. This is considered to have no impact on the trials selected for MRL derivation, since in such old studies the description of the field conditions is very limited.

Table 228 Supervised field trials on swede (roots), treated with a broadcast foliar fluazifop-butyl spray

SWEDE ROOTS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
St. Amable, Quebec, Canada, 1984 (Rutabaga, variety Laurentian)	EC 125 (rac)	1	0.25	0.10	2-5 leaves; 19 July	SaL	MAT	42	0.64, 0.48 mean = 0.56 [SS] [QU]	M4052B CA/QU/HE/84/413C [Harradine, 1985, PP5-0273]
Site undecipherable, Canada, 1984 (Rutabaga, variety York)	EC 125 (P)	1	0.25	0.11	7-8 leaves; 23 July	CSi	MAT	44	0.33, 0.38 mean = 0.36 [SS][QU]	M4052B trial code not given [Harradine, 1985, PP5-0273]
Location not reported, UK 1981 (variety not reported)	EC 250 (rac)	1	0.75	0.29	5-6 leaves; 02 July	ns	ns	47	< 0.02 [QU] [LOQ=0.05]	PP009B169; NE1 [Atreya *et al*., 1982, ASF64_10000]
Location not decipherable, UK, 1981 (Ruta Otofte)	EC 250 (rac)	1	0.75	0.29	5-6 leaves; 02 July	ns	ns	47	< 0.02 [QU] [LOQ=0.05]	PP009B169 NE2 [Atreya *et al*., 1982, ASF64_10000]
Newcastle, UK, 1981 (Ruta Otofte)	EC 250 (rac)	1	0.75	0.29	15-23 cm diameter 02 July	ns	ns	47	0.11 [QU]	PP009B169 NE3 [Atreya *et al*., 1982, ASF64_10000]
idem	EC 250 (rac)	1	1.5	0.29	idem	ns	ns	47	0.13 [QU]	idem
Invergawrie, UK, 1981 (variety not decipherable)	EC 250 (rac)	1	1.0	0.18	10-15 cm tall; 18 June	ns	ns	146	0.07 [QU]	PP009B169 Invergawrie [Atreya *et al*., 1982, ASF64_10000]
idem	EC 250 (rac)	1	2.0	0.36	10-15 cm tall; 18 June	ns	ns	146	0.18 [QU]	idem
Balerno, Mid Lothian; UK; 1984; (Ruta Otofte)	EC 125 (P)	1	0.38	0.19	4 leaves; 25 June	L	MAT	56	< 0.03 [SS] [LOQ=0.05]	M4001B; 9R/84 SAI37; [Harradine, 1985, PP5/0100]
Winchburgh,	EC	1	0.38	0.19	4 leaves;	L	MAT	56	0.24	M4001B;

SWEDE ROOTS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
West Lothian; UK; 1984; (Ruta Otofte)	125 (P)				25 June				[SS]	9R/84 SAI38; [Harradine, 1985, PP5/0100]
Kirknewton, West Lothian; UK; 1984; (Ruta Otofte)	EC 125 (P)	1	0.38	0.19	4-6 leaves; 25 June	L	MAT	56	0.17 [SS]	M4001B; 9R/84 SAI39; [Harradine, 1985, PP5/0100]
Upper Largo, Fife; UK; 1984; (Ruta Otofte)	EC 125 (P)	1	0.38	0.19	4-6 leaves; 25 June	ns	MAT	56	0.05 [SS]	M4001B; 9R/84 SAI41; [Harradine, 1985, PP5/0100]
South Queensferry; UK; 1984; (Marian Greentop)	EC 125 (P)	1	0.38	0.19	4 leaves; 25 June	L	MAT	56	0.17 [SS]	M4001B; 9R/84 SAI42; [Harradine, 1985, PP5/0100]
Horbling, Lincs, UK; 1985 (Best of all)	EC 125 (P)	1	0.38	0.19	50% crop cover; 01 July	Sa/L	MAT	92	0.11	M4204B 3R85LN50; [Harradine, 1986, PP5/0272]
Linlithgow, West Lothian; UK; 1989; (Ruta Otofte)	EW 125 (P) + Agral	1	0.42	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.84	M5318B; GB18-89-S421; [Cullen, 1991, PP5/0101]
idem	EW 250 (P) + Agral	1	0.42	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.39	idem
idem	EC 125 (P) + Agral	1	0.38	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.55	idem
Linlithgow, West Lothian; UK; 1989; (Doon Major)	EW 125 (P) + Agral	1	0.38	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.43	M5318B; GB18-89-S422 [Cullen, 1991, PP5/0101]
idem	EW 250 (P) + Agral	1	0.42	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.48	idem
idem	EC 125 (P)+ Agral	1	0.42	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.60	idem

GSH: MAT = mature (roots 7.6-15 cm diameter);

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[SS] Samples not stated (M4001B, M4204B) or less than the required 12 plants (or 2 kg)

Additional trial information:

M4052B. non-GLP. Poor quality of the study. Weather (except "hot dry summer for CA/QU/HE/84/413C"), equipment, plot size, sample size (except "sample size in trial CA/QU/HE/84/413C was smaller due to poor growth", remark was not quantified), growth stage at harvest were not reported. Samples were handpicked Storage at -20 °C. Storage time not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2** with internal standard **with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery 85% at 0.5 mg/kg. Control samples < 0.02 mg/kg.

PP009B169 Non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Spray volumes range between 260 and 570 L/ha. No data on storage. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/1** was used for analyses of the samples. Samples were corrected for recoveries; uncorrected results not reported. Mean recoveries 76% at 1.0-5.0 mg/kg. Control samples were < 0.02 mg/kg.

M4001B. Non-GLP. No unusual weather conditions. Spray application using a CO_2 knapsack sprayer. Spray volume 200 L/ha. Sample sizes not stated. Storage at -20 °C, storage time not stated but less than 12 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with internal standard with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (76% at 0.5 mg/kg). Control samples < 0.03 mg/kg.

M4204B Non-GLP. No unusual weather conditions reported. Spray application using CO_2 knapsack sprayer. Spray volume 200 L/ha. Sampling method and size not stated. Storage at -20 °C, but time not stated, but less than 1 year. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2** with internal standard **with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery 86% at 0.2 mg/kg. Control samples < 0.02 mg/kg.

M5318B. GLP. No unusual weather conditions. Samples of 12 plants. Roots and leaves were separated. Roots were sub-sampled in the field: cut longitudinally in 4 quarters and 1 quarter was retained for analysis. Storage at -18 °C; storage time not stated but less than 12 months. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg.** Mean internal standard recovery (108% at 0.5 mg/kg). Control samples < 0.05 mg/kg.

Sweet potatoes

One cGAP for sweet potatoes (and yam) is available:

- cGAP from the USA is 4×0.21 kg ai/ha and a PHI of 14 days (underlining nn).

Trials that could be matched to this cGAP were summarized.

Table 229 lists trials conducted in the USA (2008). A broadcast foliar application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 229.

Additional residue trials from USA (1982 -1985) were available with 1 ×0.28 kg ai/ha and harvest at 45, 83, 84, 117, 118 DAT, 1 ×0.56 kg ai/ha and harvest at 83, 84, 117, 118 DAT, 2 ×0.28 kg ai/ha with harvest at 54, 55, 83, 110 DAT, 2 × 0.42 kg ai/ha with harvest at 46 DAT, 2 × 0.56 kg ai/ha with harvest at 45, 46, 54, 55, 61, 62, 83, 110 DAT, 2 ×1.2 kg ai/ha with harvest at 61, 62 DAT [Yates and Monaco, 1984, no code, report no code; Baron, 1990, no code, report IR-4 PR 2328 (1990)]. These trials were not summarized, because they would not assist in MRL setting.

Table 229 Supervised field trials on sweet potato (tubers), treated with a broadcast foliar fluazifop-butyl spray

SWEET POTATO TUBERS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Salisbury, Maryland, USA, 2008 (Beauregard)	EC 240 (P) + Induce	4 (14-14-14)	0.21 0.21 0.21 0.21	0.11 0.11 0.11 0.11	nearly mature roots; 25-56 cm crop height 26 Aug	LSa	MAT	13	0.88, 0.82 mean = 0.85	IR-4 PR 02328 (2011); 08-MD01 [Barney, 2011, PP5_50290]
Clinton, NC, USA, 2008	EC 240 (P)	4 (14-	0.20 0.21	0.088 0.088	vegetative; 25-56 cm	LSa	MAT	13	0.38, 0.37	IR-4 PR 02328

SWEET POTATO TUBERS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Beauregard)	+ Induce	13-15)	0.20 0.21	0.089 0.088	crop height 1 Sept				mean = 0.38	(2011); 08-NC01 [Barney, 2011, PP5_50290]
Clinton, NC, USA, 2008 (Covington)	EC 240 (P) + Induce	4 (13-14-14)	0.21 0.21 0.20 0.21	0.086 0.085 0.086 0.087	vegetative; 25-56 cm crop height 4 Sept	LSa	MAT	16	0.44, 0.48 mean = 0.46	IR-4 PR 02328 (2011); 08-NC25 [Barney, 2011, PP5_50290]
Clinton, NC, USA, 2008 (NC99-573)	EC 240 (P) + Induce	4 (14-13-15)	0.21 0.21 0.21 0.20	0.088 0.088 0.088 0.088	vegetative; 25-56 cm crop height 1 Sept	LSa	MAT	13	0.59, 0.55 mean = <u>0.57</u>	IR-4 PR 02328 (2011); 08-NC26 [Barney, 2011, PP5_50290]
Citra, FL, USA, 2008 (Covington)	EC 240 (P) + 4.7% Chemnut NIS	4 (14-12-16)	0.21 0.22 0.22 0.22	0.11 0.11 0.11 0.11	vegetative; 25-56 cm crop height 3 Sept	Sa	MAT	14	0.54, 0.51 mean = <u>0.52</u>	IR-4 PR 02328 (2011); 08-FL02 [Barney, 2011, PP5_50290]
Crossville, TN, USA, 2008 (Beauregard)	EC 240 (P) + COOP	4 (14-14-14)	0.21 0.21 0.21 0.21	0.095 0.097 0.091 0.090	vegetative; 25-56 cm crop height 5 Sept	SaL-SiL	MAT	14	0.097, 0.13 mean = <u>0.11</u>	IR-4 PR 02328 (2011); 08-TN03 [Barney, 2011, PP5_50290]
Weslaco, TX, USA, 2008 (Beauregard)	EC 240 (P) + Dyne-amic	4 (12-15-15)	0.21 0.21 0.21 0.21	0.12 0.12 0.12 0.096	vining; 25-56 cm crop height 9 July	SaL	MAT	12	0.11, 0.12 mean = <u>0.12</u>	IR-4 PR 02328 (2011); 08-TX37 [Barney, 2011, PP5_50290]
Parlier, CA, USA, 2008 (Beauregard)	EC 240 (P) + Induce	4 (14-14-14)	0.21 0.21 0.22 0.21	0.074 0.074 0.075 0.075	vegetative; 25-56 cm crop height 31 July	SaL	MAT	14	0.54, 0.48 mean = <u>0.51</u>	IR-4 PR 02328 (2011); 08-CA110 [Barney, 2011, PP5_50290]

Additional trial information:

PR 02328 GLP. No unusual weather conditions. Application by Tee jet, spray volume 200- 300 L/ha. Field samples of commercially mature tubers (at least 12 roots from 12 separate plants) were dugged out with a shovel and removed from the plant by hand. Samples weighed at least 2 kg. Storage time max 795 days (sampling to extraction) at appr. -20 °C.Samples were analysed for total fluazifop using **HPLC-MS/MS method MRID 40831305 with a valid LOQ of 0.02 mg/kg**. Results were not corrected for concurrent method recoveries (84-96% at 0.02-5.0 mg/kg). Control samples were < 0.02 mg/kg.

Turnip roots

Three cGAPs for turnips are available:

- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 42 days
- cGAP from Belgium with 1 ×0.38kg ai/ha and a PHI of 56 days (underlining nn)
- cGAP from the UK with 1 x 0.38 kg ai/ha and a PHI of 56 days (stock feed only)

Trials that could be matched to these cGAPs were summarized.

Table 230 lists trials conducted in Canada (1980), the UK (1990). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 230. Results marked with "[QU]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62.

Additional trials from the UK (1981) were available with 1 ×0.50-1.0 kg ai/ha and harvest at 23 and 26 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. These trials were not summarized, because they would not assist in MRL setting.

Table 230 Supervised field trials on turnip (roots), treated with a broadcast foliar fluazifop-butyl spray

TURNIP ROOTS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Location, ns, Canada, 1980 (variety ns)	EC 250 (rac)	1	0.25	ns	GS ns date ns	ns	ns	41	< 0.05 [QU] [LOQ=0.05]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.50	ns	GS ns date ns	ns	ns	41	< 0.05 [QU] [LOQ=0.05]	idem
Balerno, Mid Lothian; UK; 1990; (Wallace)	EW 125 (P) + Agral	1	0.38	0.19	10-12 leaves; 50% crop cover; 5 July	SiL	CH	68	0.63	RJ0997B; GB18-90-S481; [Jones, 1992, PP5/0099]
idem	EW 250 (P) + Agral	1	0.38	0.19	10-12 leaves; 50% crop cover; 5 July	SiL	CH	68	0.74	idem
idem	EC 125 (P) + Agral	1	0.38	0.19	10-12 leaves; 50% crop cover; 5 July	SiL	CH	68	0.59	idem
Gorebridge, Mid Lothian; UK; 1990; (Wallace)	EW 125 (P) + Agral	1	0.38	0.19	12 leaves; 30-50% crop cover; 2 Aug	CL	CH	62	1.3	RJ0997B; GB18-90-S482; [Jones, 1992, PP5/0099]
idem	EW 250	1	0.38	0.19	12 leaves; 30-50%	CL	CH	62	2.0	idem

TURNIP ROOTS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	(P) + Agral				crop cover; 2 Aug					
idem	EC 125 (P) + Agral	1	0.38	0.19	12 leaves; 30-50% crop cover; 2 Aug	CL	CH	62	1.8	idem

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

Additional trial information

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.03 mg/kg.

RJ0997B. GLP. No unusual weather conditions. Spray application using a hand-held CO2 pressurised knapsack sprayer. Spray volume 200 L/ha. Samples of 12 plants. Roots and leaves were separated. Roots were subsampled in the field: opposite quarters of the roots were retained for analysis. . Storage at -18 °C; storage time not stated but less than 24 months**. NMR method PPRAM 83 using internal standard with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (99% at 0.5 mg/kg). Control samples < 0.05 mg/kg.

Stem vegetables

Artichokes

A GAP for artichokes is not available. Trials from Italy (1981) were available on artichokes with an application of 1 × 0.25–0.50–0.75 kg ai/ha and harvest at 30 DAT [Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. As the manufacturer did not seek to have maximum residue levels estimated on artichokes, the available studies on artichokes were not summarized.

Asparagus

Four cGAPs for asparagus are available:

- cGAP from the USA with 2 ×0.42 kg ai/ha with a PHI of 1 day
- cGAP from France with 1 ×0.38 kg ai/ha with a PHI of 42 days
- cGAP from Belgium with 1 ×0.19 kg ai/ha with a PHI of 42 days
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha after harvest of the spears

Trials that could be matched to these cGAPs were summarized.

Table 231 lists trials conducted in the USA (1984, 1991) and Northern France (1997) and Spain (1996, 1997). A broadcast or banded foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 231. Results marked with "[WC]", "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[WC] indicates that the weather affected growing conditions and yield.

[SS] indicates that the sample size was less than the required 2 kg.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.2 mg/kg for HPLC-UV method PCY 86-1.

Table 231 Supervised field trials on asparagus (spears) treated with a broadcast or banded foliar fluazifop-butyl spray

ASPARAGUS SPEARS; Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Salisbury, MD, USA, 1984 (Mary Washington)	EC 120 (rac) +1% COC	1	0.42	0.19	mowed to 5 cm height; 3 May	SaL	CH	8	0.14, 0.15, 0.16, 0.16, mean 0.15 [a], [SS] [LOQ=0.2]	IR-4 PR 2201; MD [Baron, 1987, 464389]
idem	EC 120 (rac) +1% COC	2 (13)	0.42 0.42	0.19 0.19	spears 13-15 cm; 16 May	SaL	CH	1	0.74, 0.82, 0.90, 0.90, mean 0.84 [a], [SS]	IR-4 PR 2201; MD [Baron, 1987, 464389]
idem	EC 120 (rac) +1% COC	1	0.84	0.37	mowed to 5 cm height; 3 May	SaL	CH	8	0.18, 0.22, 0.31, 0.32, mean 0.26 [a], [SS] [LOQ=0.2]	IR-4 PR 2201; MD [Baron, 1987, 464389]
idem	EC 120 (rac) +1% COC	2 (13)	0.84 0.84	0.37 0.37	spears 13-15 cm; 16 May	SaL	CH	1	1.6, 1.8, 2.2, 1.9, mean 1.9 [a], [SS]	IR-4 PR 2201; MD [Baron, 1987, 464389]
Prosser, WA, USA, 1984 (500W)	EC 120 (rac) + 1% COC	2 (29)	0.42 0.42	0.16 0.16	spears 18 cm tall; 5 June	SaL	MAT	1	0.20, 0.27, 0.37, 0.37, mean 0.30 [a], [SS]	IR-4 PR 2201; WA [Baron, 1987, 464389]
	EC 120 (rac) + 1% COC	2 (29)	0.84 0.84	0.31 0.31	spears 18 cm tall; 5 June	SaL	MAT	1	0.88, 1.0, 1.1, 1.1, mean 1.0 [a], [SS]	IR-4 PR 2201; WA [Baron, 1987, 464389]
East Lansing, MI, USA, 1988 (Mary Washington)	EC 120 (rac) + 1% COC	2 (6)	0.42 0.42	0.22 0.22	spear 2.5-20 cm tall; 11 May	L	MAT	1	1.7, 1.6, 2.2, 1.3 mean 1.7 [a]	IR-4 PR3944 88-MI013 [Baron, 1989, no code]
Stockton, San Joaquin, CA USA, 1991 (UC157)	EC 120 (P) +0.5% NIS	2 (21)	0.21 0.21	0.45 0.45	crop height 50 cm; 06 March	CL	NH	1	1.4, 2.7, 1.5 mean:1.9 [a]	RR 92-057B; 17-CA-91-321 [Roper and Graham, 1992, PP5/0584]
Idem	EC 120 (P) +0.5% NIS	2 (21)	0.42 0.42	0.90 0.90	crop height 50 cm; 06 March	CL	NH	1	4.7, 2.9, 4.1, mean: 3.9 [a]	Idem (processing)
Coachella, Riverside, CA, USA, 1991 (UC157)	EC 120 (P) + 0.5% NIS	2 (21)	0.19 0.22	0.069 0.076	crop height 61 cm; 25 March	SaL	NH	1	0.4, 0.3, 0.4, mean: 0.4 [a], [SS]	RR 92-057B; 14-CA-91-322 [Roper and Graham, 1992, PP5/0584]
idem	EC 120 (P)	2 (21)	0.41 0.42	0.146 0.152	crop height 61 cm; 25	SaL	NH	1	0.4, 0.8, 0.6 mean: 0.6	Idem

ASPARAGUS SPEARS; Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	+0.5% NIS				March				[a], [SS]	
Lincoln, Kent, DE, USA, 1991 (Jersey giant)	EC 120 (P) +0.5% NIS	2 (21)	0.21 0.21	0.060 0.060	crop height 30-38 cm; 1 May	SaL	NH	1	0.4, 0.2, 0.2 mean: 0.3 [a], [SS]	RR 92-057B; 54-DE-91-323 [Roper and Graham, 1992, PP5/0584]
idem	EC 120 (P) +0.5% NIS	2 (21)	0.42 0.42	0.120 0.120	crop height 30-38 cm; 1 May	SaL	NH	1	0.4, 0.5, 0.5 mean: 0.5 [a], [SS]	Idem
Belvidere, Warren, NJ, USA, 1991 (Jersey giant)	EC 120 (P) + 0.5% NIS	2 (21)	0.21 0.21	0.16 0.18	crop height 30 cm; 17 May	L	NH	1	0.7, 0.5, 0.6 mean: 0.6 [a], [SS]	RR 92-057B; 57-NJ-91-326 [Roper and Graham, 1992, PP5/0584]
idem	EC 120 (P) + 0.5% NIS	2 (21)	0.42 0.42	0.31 0.36	crop height 30 cm; 17 May	L	NH	1	2.7, 2.4, 2.5 mean: 2.5 [a], [SS]	Idem
Ephrata, Grant, WA, USA, 1991 (Comman)	EC 120 (P) + 0.5% X-77	2 (21)	0.21 0.21	0.10 0.10	crop height 23 cm; 21 April	SaL	NH	1	0.3, 0.3, 0.3 mean: 0.3 [a] [SS]	RR 92-057B; 15-WA-91-327 [Roper and Graham, 1992, PP5/0584]
idem	EC 120 (P) + 0.5% X-77	2 (21)	0.42 0.42	0.20 0.20	crop height 23 cm; 21 April	SaL	NH	1	0.4, 0.4, 0.5 mean: 0.4 [a] [SS]	Idem (processing)
George, Grant, WA, USA, 1991 (Comman)	EC 120 (P) + 0.5% NIS	2 (21)	0.21 0.21	0.10 0.09	crop height 25 cm; 20 April	SaL	NH	1	0.2, 0.4, 0.3 mean: 0.3 [a], [SS]	RR 92-057B; 15-WA-91-328 [Roper and Graham, 1992, PP5/0584]
idem	EC 120 (P) + 0.5% NIS	2 (21)	0.42 0.42	0.20 0.18	crop height 25 cm; 20 April	SaL	NH	1	0.7, 0.9, 1.0 mean: 0.9 [a], [SS]	Idem
Benton Harbor, Berrie, MI, USA, 1991 (var ns)	EC 125 (P)	2 (21)	0.42 0.42	ns ns	ns	SaL	NH	1	1.8	RR 92-057B; 28-MI-91-325 [Roper and Graham, 1992, PP5/0584] (processing)
El Rocio, Huelva; Spain; 1997 (white asparagus;	EC 125 (P)	1	0.40	0.094	2 Apr	Sa	CH	28	< 0.01 [SS]	RJ2673B; ES10-97-SH002; [Jones *et al*, 1998, PP5/0110]

ASPARAGUS SPEARS; Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
JACMA 2002)										
El Rocio, Huelva; Spain; 1997 (white asparagus; JACMA 2002)	EC 125 (P)	1	0.40	0.094	2 Apr	Sa	CH	28	< 0.01 [SS]	RJ2673B; ES10-97-SH102; [Jones *et al*, 1998, PP5/0110]
El Rocio, Huelva; Spain; 1997 (white asparagus; JACMA 2002)	EC 125 (P)	1	0.39	0.094	2 Apr	Sa	CH	28	< 0.01 [SS]	RJ2673B; ES10-97-SH202; [Jones *et al*, 1998, PP5/0110]
Banded foliar spray over rows										
F-37120 Verneuil le Chateau; N-France; 1997; (Jacquema 2000)	EC 125 (P)	1	0.19	0.067	8 cm tall; 22 April	CSa	MAT	42	< 0.01 [SS]	RJ2701B; 97HCLSAP01; [Jones and Kenny, 1999, PP5/0113]
F-37120 Courcoue; N-France; 1997; (Argenteuil)	EC 125 (P)	1	0.19	0.064	8 cm tall; 30 April	Sa	MAT	42	< 0.01 [SS]	RJ2701B; 97HCLSAP02; [Jones and Kenny, 1999, PP5/0113]
Lebrija; Sevilla; Spain; 1996; (UC-157)	EC 125 (P)	1	0.38	0.13	BBCH 23; 15 Apr	LSa	MAT	21	< 0.01 [WC],[SS]	RJ2281B; AP/3225/ZE/1; [Miles and Cowley, 1997, PP5/0105]

[SS] Samples size less than the required 2 kg; results not considered representative for MRL derivation.

[WC] Weather conditions affected crop yield and samples are considered not representative for MRL derivation.

[a] Results are the mean of 3-4 replicate field samples per plot (individual results and mean are listed). The mean is taken for MRL derivation if according to cGAP.

Additional trial information

PR-2201. GLP. No unusual weather conditions. Plot size 20-200 plants/plot (100-1000 ft2 with 5 ft2/plant). MD: First application immediately after harvest by mowing to 5 cm height; second application 13 days later with spears 13-15 cm present. Broadcast foliar spray using a CO2 boom sprayer or backpack sprayer with boom. Spray volume 24-29 GPA = 220-270 L/ha. Asparagus spears (2 lbs = 0.90 kg). Storage at -17 °C for 658 days (analysis in Febr 1986). Samples were analysed for total fluazifop using **HPLC-UV method PCY 86-1 with a valid LOQ of 0.2 mg/kg** . Samples were not corrected for mean concurrent method recovery (78-98% at 0.2-1.0 mg/kg). Control samples (< 0.01 mg/kg).

IR-4 PR3944. GLP. No unsual weather conditions. Plot size 330 ft, with 1 ro/plot). First application at emerging spears, but after all spears were removed and second application (6 days later) when spears were 2.5-20 cm. Broadcast foliar spray using a CO_2 boom sprayer or backpack sprayer with boom. Spray volume 20 GPA = 187 L/ha. All spears 6 inches (15 cm) or taller were harvested. 33-58 spears (weight of sample not stated). Storage at -20 °C. Duration not stated, but less than 1 year. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Average concurrent recovery (fluazifop-butyl: 62%, n = 4, spike level 0.085 mg/kg; fluazifop acid (II): 77%, n = 5, spike level 0.10 mg/kg)). Control samples < 0.10 mg/kg.

RR92-057B. GLP. No unusual weather conditions. Plot size 90 plants/plot (450 ft2 assuming 5 ft2/plant). Broadcast foliar spray using a CO2 backpack sprayer or tractor mounted sprayer. Spray volume 5-30 GPA = 50-280 L/ha. Mature spears are cut with a knife at the base of the stem. Samples contained, 24 spears (trial 321), 0.45-0.70 kg (trials 322, 323, 326, 327, 328), 23 kg for processing (trial 325, 327). Storage at -23 °C for 221 days Samples were analysed for total fluazifop using **GC-MS method RR 91-014B with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for mean concurrent method recovery (90-102% at 0.01-5.0 mg/kg). Control samples (< 0.01 mg/kg).

RJ2673B. GLP. No unusual weather conditions. Plot size not stated. Broadcast foliar spray using a CO2 knapsack sprayer. Spray volume 420-430 L/ha. Asparagus (1 kg) was sampled manually and taken systematically from across the plots. Storage at -18 °C for 89 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for mean concurrent method recovery (99% at 0.1 mg/kg). Control samples (< 0.01 mg/kg).

RJ2701B. GLP. No unusual weather conditions. Plot size 98 m2 (i.e. 98 plants assuming 1 m2/plant). **Banded spray (2 m wide) over rows** using a gas knapsack sprayer with side boom. Spray volume 280L/ha. Asparagus (1.8 kg, 12 items) was sampled manually and taken randomly from across the plots. Storage at -18 °C for 64 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (95-95% at 0.1–0.2 mg/kg). Control samples (< 0.01 mg/kg).

RJ2281B. GLP. Weather conditions were unusual and contributed to a lower than expected crop yield. Plot size 30 m2 (i.e. 30 plants assuming 1m2/plant); **Banded spray over beds** using a hand held boom sprayer. Spray volume 300 L/ha. Asparagus (1 kg, 12 items) was sampled manually and taken systematically from across the plots. Storage at -18 °C for 354 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for mean concurrent method recovery (108% at 0.1 mg/kg). Control samples (< 0.01 mg/kg).

Celery

Two cGAPs for celery are available:

- cGAP from Belgium with 1 ×0.25 kg ai/ha with a PHI of 42 days
- cGAP from France with 1 ×0.19 kg ai/ha with a PHI of 42 days

Trials that could be matched to these cGAPs were summarized.

Trials from USA (1986) were available on celery with an application of 2 × 0.42 kg ai/ha and harvest at 28-30 DAT [Watford and Francis, 1988, PP5/0323, report TMU3418/B]. Since the manufacturer did not intend to have MRLs on celery, the available studies on celery were not summarized.

Some supervised trials on celery were not submitted: [Trumbo and Francis, 1986, MRID 40693103, report TMU3077/B, not referenced]. No further efforts were taken to retrieve these studies.

Besides total fluazifop, also CF3-pyridone (X) was analysed in celery from some 1986 trials conducted in the USA [Watford and Francis, 1988, MRID 40693104, report TMU3418/B]. These trials were summarized in the metabolism section.

Rhubarb

One cGAP for rhubarb is available:

- cGAP from France with 1 ×0.19 kg ai/ha with a PHI of 42 days

Trials that could be matched to this cGAP were summarized.

Table 232 lists trials conducted in the USA (1984–1985, 2010). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 232. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample size was not stated or less than the required 2 kg.

Table 232 Supervised field trials on rhubarb (stalks) treated with a broadcast foliar fluazifop-butyl spray

RHUBARB Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Frederick, MD, USA,	EC 250	3 (51, 280)	0.45 0.45	0.16 0.16	Mature plants;	SiCL	MAT	14	0.13, 0.17, 0.20, 0.26,	IR-4 PR 2404 (1987);

RHUBARB Location, Country; year; (variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
1984-1985 (Victoria Red)	(rac)		0.45	0.16	8 May				mean 0.19 [a], [SS]	MD [Baron, 1987, 464387] and summary IR-4 PR 2073 [Baron, 1990, 463130]
idem	EC 250 (rac)	3 (51, 280)	0.45 0.45 0.45	0.16 0.16 0.16	Mature plants; 8 May	SiCL	MAT	29	< 0.1, < 0.1, < 0.1, < 0.1, mean < 0.1 [a], [SS]	idem
idem	EC 250 (rac)	3 (51, 280)	0.90 0.90 0.90	0.32	Mature plants; 8 May	SiCL	MAT	14	0.24, 0.29, 0.65, 0.89, mean 0.35 [a]	idem
idem	EC 250 (rac)	3 (51, 280)	0.90	0.32	Mature plants; 8 May	SiCL	MAT	29	< 0.1, < 0.1, < 0.1, 0.12, mean 0.10 [a], [SS]	idem
Holt, MI, USA, 2010 (German Wine)	EC 240 (P) + 0.25% NIS	2 (14)	0.28 0.28	0.15 0.15	flowering; 1 June	SaL	CH	14	0.11, 0.14, mean 0.13 [a]	IR-4 PR A2404; 10-MI13 [Arsenovic, 2013, PP5_50552
Clarksville, MI, USA, 2010, (McDonald)	EC 240 (P) + 0.25% NIS	2 (14)	0.28 0.29	0.15	flowering; 1 June	SaL	CH	14	0.096, 0.078, mean 0.087	IR-4 PR A2404; 10-MI14 [Arsenovic, 2013, PP5_50552

[SS] Sample size not stated; results are considered not representative for MRL derivation.

[a] Results came from replicate field samples, the mean residue is used for MRL derivation if according to cGAP.

Additional trial information

PR-2404. GLP. Weather conditions not stated. Plot size: 24 plants/plot. Broadcast foliar spray using a biccycle sprayer. Plants were sprayed over 2 seasons in 1984 and 1985. Spray volume 30 GPA = 280 L/ha. Whole stalks ((sample size not stated) were sampled. Storage at -10 °C for 343 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for average concurrent method recovery (73% at 0.4 mg/kg). Control samples < 0.1 mg/kg.

PR-A2404. GLP. No unusual weather conditions. Plot size: 25 plants/plot. Broadcast foliar spray using a CO2 backpack sprayer. Spray volume 20 GPA = 190 L/ha. Stalks were cut with a kitchen style knife from 20 plants in the plot; leaf portions were removed. Storage at -18 °C for 744 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.01A modification A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recovery (87-96% at 0.02-5.0 mg/kg). Control samples < 0.02 mg/kg.

Witloof chicory (roots and sprouts)

One cGAP for witloof roots is available:

- cGAP from Belgium, France or the Netherlands with 1 ×0.38 kg ai/ha with a PHI of 56 days for the roots used for sprout production (underlining nn)

  Trials that could be matched to this cGAP were summarized.

Table 233 lists trials conducted in the Netherlands (1983) and Northern France (1982, 1997). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 233. In report RJ2646B roots were harvested 55-57 days after treatment and they were analysed for residues. In addition roots were taken from the field and stored in the cold room (-0.6 to + 4 ºC) for 25-47 days and then forced during 21-23 days in hydroponic solution at 20 ºC to form the witloof sprouts (endives), which were sampled 101 - 126 days after treatment. Results marked with "[SS]" or "[AM]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[SS] indicates that the sample size was not stated or was less than the required 12 plants or 2 kg.

[AM] indicates that the analytical method did not contain a hydrolysis step and therefore fluazifop (II) conjugates are not included.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62/2.

Table 233 Supervised field trials on witloof (roots and sprouts), treated with a broadcast foliar fluazifop-butyl spray

WITLOOF ROOTS AND SPROUTS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Crop part	Total fluazifop mg/kg	Report; Trial no [ref]
Medemblik; Netherlands; 1983 (ns)	EC 250 (rac)	1	0.31	0.062	12 leaves; 40% crop cover; 19 July	SaC		101	root	< 0.02; < 0.02; < 0.02; < 0.02 [a]	M3690B; 83.124; [Harradine, 1984, PP9/0071]
idem	EC 250 (rac)	1	0.38	0.075	12 leaves; 40% crop cover; 19 July	SaC		101 . . . 274	root . . . sprouts	< 0.02; < 0.02; < 0.02; < 0.02 [a] < 0.02 < 0.02; 0.02; 0.02 [a]	M3690B; 83.124; [Harradine, 1984, PP9/0071] and M4058B; [Harradine, 1985, PP9/0089]
idem	EC 250 (rac)	1	0.75	0.15	12 leaves; 40% crop cover; 19 July	SaC		101 . . . 274	root . . . sprouts	< 0.02; < 0.02; < 0.02; < 0.02 [a] < 0.02 0.02 0.03 0.03 [a]	M3690B; 83.124; [Harradine, 1984, PP9/0071] M4058B; and [Harradine, 1985, PP9/0089]
Reims; N-France; 1982; (Zoom)	EC 250 (rac) + ActiPlus	1	0.75	0.15	6-7 leaves; 26 June	ns	ns	157	root	< 0.02 [SS], [AM]	RIC2816; Invuflec [Culoto and Mallmann, 1984, PP5/0280]
idem	EC 250 (rac)	1	0.75	0.15	6-7 leaves; 6 July	ns	ns	142	root	< 0.02 [SS],	RIC2816; Invuflec [Culoto and

WITLOOF ROOTS AND SPROUTS Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Crop part	Total fluazifop mg/kg	Report; Trial no [ref]
	+ Actiplus									[AM]	Mallmann, 1984, PP5/0280]
80110 Le Plessier, Rozainvillers; N-France; 1997; (Turbo)	EC 125 (P)	1	0.38	0.12	BBCH 43; crop height 25 cm; 30 July	Si	49 CH	57 126	root sprouts	< 0.01 <u>< 0.01</u>	RJ2646B; S102.97 [Mason and Picard, 1999, PP5/0111]
80250 Sourdon; N-France; 1997; (Atlas)	EC 125 (P)	1	0.38	0.12	BBCH 45; crop height 25 cm; 26 Aug	Si	49 89	55 101	root sprouts	< 0.01 <u>< 0.01</u>	RJ2646B; S104.97; [Mason and Picard, 1999, PP5/0111]

BBCH 43-45 = 30-50% of expected root diameter reached.

AM Results are for fluazifop free acid (F); fluazifop-butyl or conjugates are not included. Results are therefore underestimated and cannot be used for MRL-derivation.

[SS] Sample size not stated (report RIC2816) or less than the required 12 plants (or 2 kg)

[a] Results came from 4 replicate plots; the highest value is taken for MRL-derivation if according to cGAP.

Additional trial information

M3690B and M4058B. Non-GLP. No unusual weather conditions. Spray application using a knapsack gas sprayer. Spray volume 500 L/ha. Residues in pin roots are reported in M3690B; Residues in endives grown from these roots are reported in M4058B. Conditions for endive growth are not stated. Roots (4 kg, i.e. > 12 roots) were taken by hand from replicate plots and they were cut in pieces prior to freezing. Roots and endives were washed with cold water to remove soil. [Sample size for endive not stated, but derived from 4 kg roots i.e. > 12 plants] Storage at -20 °C for 32 days (roots) or . Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (81% for roots, 85% for endives, each at 0.5 mg/kg). Control samples < 0.02 mg/kg for roots and endives.

RIC2816 Non-GLP. Weather conditions not reported. Foliar spray with spray volume 500 L/ha. Sample sizes not stated. Samples were stored at unknown conditions for 55 days.Samples were analysed for free fluazifop acid (II) using **HPLC-UV PRAM 52 with a valid LOQ of 0.02 mg/kg.** Fluazifop-butyl and fluazifop conjugates are not included. Samples were corrected for mean concurrent recovery (80% at 0.08 mg/kg); uncorrected results were not reported. Control samples < 0.02 mg/kg.

RJ2646B. GLP. No unusual weather conditions. Foliar spray application using a hand-held boom. Spray volume 300 L/ha. Witloof endives were obtained (101-126 days after treatment) from roots harvested 55-57 days after treatment. Roots (> 2 kg) were sampled by hand systematically from across the plots. Endives were obtained from at least 12 roots. Storage at -18 °C for 118-189 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recoveries (101% at 0.1 mg/kg). Control samples < 0.01 mg/kg.

Grasses for sugar or syrup production

Sugar cane

One cGAP for sugar cane is available:

- cGAP from Brazil with 1 ×0.075 kg ai/ha with a PHI 42 days (underlining <u>nn</u>)

Trials that could be matched to this cGAP were summarized.

Table 234 lists trials conducted in the USA (1986) and Brazil (2011). A broadcast foliar application or weed directed spot application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 234.

Table 234 Supervised field trials on sugarcane (stalks), treated with a broadcast foliar or weed directed spot application of fluazifop-butyl

SUGAR CANE STALKS Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Weed directed spot application										
Pahokee, FL, USA, 1986 (CP 70-1133)	EC 120 (P) + 1% COC	1	NR	0.67	GS ns; 21 April	ns	MAT	169	< 0.02, < 0.02	TMU3310/B; 75FL86-904R; [Roper and Francis, 1988, 405720]
Clewiston, FL, USA, 1986 (CP 65357)	EC 120 (P) + 1% COC	1	NR	0.67	GS ns; 22 April	ns	MAT	168	< 0.02, < 0.02	TMU3310/B; 75FL86-905R; [Roper and Francis, 1988, 405720]
Houma, LA, USA, 1986 (CP 65-357 and CP 72-356)	EC 120 (P) + 1% COC	1	NR	0.67	GS ns 9 July	ns	MAT	111	< 0.02, < 0.02	TMU3310/B; 36LA86-900R; [Roper and Francis, 1988, 405720]
Broadcast foliar application										
Jaboticabal, SP, Brazil, 2011 (RB 5536)	EW 250 (P)	1	0.075	0.025	BBCH 47; 31 March	CL	BBCH 49	35	< 0.01	M11029; AMA; [Draetta, 2012, A12530B_10011]
Rio das Pedras, SP, Brazil, 2011 (RB 85 7515)	EW 250 (P)	1	0.075	0.025	BBCH 47-48; 11 March	SaCL	BBCH 48-49	35	< 0.01	M11029; RWC1; [Draetta, 2012, A12530B_10011]
Bandeirantes, PR, Brazil, 2011 (RB 72454)	EW 250 (P)	1	0.075	0.025	BBCH 39; 11 April	C	BBCH 39	35	< 0.01	M11029; RWC2; [Draetta, 2012, A12530B_10011]
Tupaciguara, MG, Brazil, 2011 (SP80 1816)	EW 250 (P)	1	0.075	0.025	BBCH 39; 28 March 2011	SaC	BBCH 49	35	< 0.01	M11029; JJB; [Draetta, 2012, A12530B_10011]

NR = not relevant, because it is a directed spot application;

Additional trial information

TMU3310/B: GLP study. No unusual weather conditions. Weed directed spot application on a field size of 100-1040 m2. Samples of 5 kg mature cane internodes were harvested. Samples were stored at -17 °C for a maximum period of 6 months (harvest date to last study date). Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg.** Concurrent recovery at 0.1 mg/kg was 108%. Control samples were < 0.02 mg/kg.

M11029; GLP study. No unsual weather conditions. Foliar broadcast application by ground sprayer, spray volume 300 L/ha. Plot size 30 m^2. Stalks (minimum of 12 sugar canes to get at least 2 kg) were sampled, one cane at every step, one sample per plot. After the collection, the sugarcanes were divided in 3 (three) groups with the same number of canes and from each group stalks with approximately 20 cm were cut once from the under, once from the middle and once from the upper part of the cane. Samples were stored at -20 °C for 5.5-6.4 months. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET 138 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual method recoveries (83% at 0.1 mg/kg). Control samples were < 0.01 mg/kg.

Oilseeds

Cotton seed

Two cGAPs for cotton are available:

- cGAP from the USA with 2 ×0.42 kg ai/ha and a PHI of 90 days (underlining nn)
- cGAP from Brazil with 1 ×0.25 kg ai/ha and a PHI of 60 days (underlining nn)

Trials that could be matched to these cGAPs were summarized.

Table 235 lists trials conducted in the USA (1979, 1980, 1982, 1983, 2008,), Brazil (2011), Spain (1987) and South Africa (1991). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 235. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[SS] indicates that the sample size was less than the required 1 kg cottonseeds without lints.

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/1 or NMR method PPRAM 83.

Besides total fluazifop, also despyridinyl acid (III) was analysed in cottonseed samples from the 1979 and 1980 trials conducted in the USA [Ussary, 1981, 405793, report TMU0680/B; Francis and Kennedy, 1981, 407582, report PP009B042]. The results of these trials were summarized in the metabolism section.

Besides total fluazifop, also CF3-pyridone (X) was analysed in cottonseed samples from some 1979 and 1980 trials conducted in the USA [Atreya *et al*., 1981, PP9/0733, report PP009B061]. These trials were summarized in the metabolism section.

Table 235 Supervised field trials on cotton (undelinted seeds), treated with a broadcast foliar fluazifop-butyl spray

COTTON SEED Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Vicksburg MS, USA, 1979 (variety ns)	EC 250 (rac)	2 (7)	0.28	0.10	15-20 cm height; 22 June	ns	MAT	133	< 0.02 [SS] [LOQ=0.05]	TMU0679/B; HU5-79-04; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
Visalia, CA, USA, 1979 (variety ns)	EC 250 (rac)	1	1.1	0.40	15-18 cm height; 13 June	ns	MAT	147	0.02 [SS] [LOQ=0.05]	TMU0679/B; HU2-79-10; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
Center Point, TX, USA, 1980 (ns)	EC 240 (rac)	2 (5)	0.28	0.083	18-23 cm height; 4 June	ns	MAT	110	0.03 [LOQ=0.05]	TMU0679/B 20TX80-007; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
idem	EC 480 (rac)	2 (5)	0.28	0.083	18-23 cm height; 4 June	ns	MAT	110	0.02 [LOQ=0.05]	idem

COTTON SEED Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
idem	EC 240 (rac)	1	1.1	0.33	18-23 cm height; 4 June	ns	MAT	110	< 0.03 [LOQ=0.05]	TMU 0987/B 20TX80-007; [Koubek, 1982, 405794
idem	EC 240 (rac)	2 (5)	0.56	0.17	18-23 cm height; 4 June	ns	MAT	110	< 0.03 [LOQ=0.05]	idem
Tolleson, AZ, USA, 1980 (variety ns)	EC 240 (rac)	2 (20)	0.56	0.15	5-10 cm height; 14 May	ns	MAT	154	0.03 [SS] [LOQ=0.05]	TMU0679/B 42AZ80-001; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
Idalou, TX, USA, 1980 (Cascott L7)	EC 240 (rac)	2 (21)	0.28	0.14	18-20 cm height; 28 June	ns	MAT	103	< 0.02 [LOQ=0.05]	TMU0679/B; 33TX80-001; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
idem	EC 480 (rac)	2 (21)	0.28	0.14	18-20 cm height; 28 June	ns	MAT	103	0.02 [LOQ=0.05]	idem
idem	EC 240 (rac)	1	1.1	0.56	18-20 cm height; 28 June	ns	MAT	124	< 0.03 [LOQ=0.05]	TMU 0987/B 33TX80-001; [Koubek, 1982, 405794
idem	EC 240 (rac)	2 (21)	0.56	0.29	18-20 cm height; 28 June	ns	MAT	103	< 0.03 [LOQ=0.05]	idem
Sunnyside, MS, USA, 1980 (DPL 61)	EC 240 (rac)	2 (5)	0.28	0.097	10-25 cm height; 3 July	ns	MAT	83	< 0.02 [LOQ=0.05]	TMU0679/B; 29MS80-017; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
idem	EC 480 (rac)	2 (5)	0.28	0.097	10-25 cm height; 3 July	ns	MAT	83	0.02 [LOQ=0.05]	idem
idem	EC 240 (rac)	1	1.1	0.38	10-25 cm height; 3 July	ns	MAT	83	< 0.03 [LOQ=0.05]	TMU 0987/B 29MS80-017; [Koubek, 1982, 405794
idem	EC 240 (rac)	2 (5)	0.56	0.19	10-25 cm height; 3 July	ns	MAT	83	< 0.03 [LOQ=0.05]	idem
Rosedale, CA, USA, 1980 (variety ns)	EC 240 (rac)	2 (21)	0.56	0.15	13-15 cm height; 29 May	ns	MAT	179	< 0.02 [SS] [LOQ=0.05]	TMU0679/B; 41CA80-003; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
idem	EC 480 (rac)	2 (21)	0.56	0.15	13-15 cm height; 29 May	ns	MAT	179	< 0.02 [SS] [LOQ=0.05]	idem
Tipton,	EC	2	0.56	0.15	20-25 cm	ns	MAT	136	0.04	TMU0679/B;

COTTON SEED Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
CA, USA, 1980 (variety ns)	240 (rac)	(18)			height; 17 June				[SS] [LOQ=0.05]	41CA80-004; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
idem	EC 480 (rac)	2 (18)	0.56	0.15	20-25 cm height; 17 June	ns	MAT	136	< 0.02 [SS] [LOQ=0.05]	idem
Goldsboro, NC, USA, 1980 (variety ns)	EC 240 (rac)	1	1.1	0.50	GS ns; 24 April	ns	MAT	193	< 0.02 [SS] [LOQ=0.05]	TMU0679/B; RU1-80-01; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
Cochran, GA, USA, 1980 (Coker 310)	EC 240 (rac)	2 (9)	0.28	0.12	GS ns; 9 June	ns	MAT	105	0.03 [LOQ=0.05]	TMU0679/B; 28GA80-001; [Ussary, 1981, 405792] and PP009B035; [Atreya *et al*, 1981, PP9/0734]
idem	EC 480 (rac)	2 (9)	0.28	0.12	GS ns; 9 June	ns	MAT	105	< 0.02 [LOQ=0.05]	idem
idem	EC 240 (rac)	1	1.1	0.49	GS ns; 9 June	ns	MAT	105	< 0.03 [LOQ=0.05]	TMU 0987/B 28GA80-001; [Koubek, 1982, 405794
idem	EC 240 (rac)	2 (9)	0.56	0.25	GS ns; 9 June	ns	MAT	105	< 0.03 [LOQ=0.05]	idem
Grady, AL, USA, 1982 (DPL 61)	EC 480 (rac) +COC	2 (20)	0.56	0.31	GS ns; 24 June	ns	ns	83	< 0.04 [LOQ=0.05]	TMU1027/B; 45AL82-031; [Koubek, 1982, 407595]
Somerton, AZ, USA, 1982 (DPL 61)	EC 480 (rac) +COC	2 (54)	1.1	0.39	GS ns; 19 June	ns	ns	104	0.08	TMU1027/B; 38AZ82-010; [Koubek, 1982, 405795]
Roll, AZ, USA, 1982 (DPL 61)	EC 480 (rac) +COC	2 (23)	1.1	0.41	GS ns; 28 May	ns	ns	129	< 0.04 [LOQ=0.05]	TMU1027/B; 38AZ82-011; [Koubek, 1982, 405795]
Florence, AZ, USA, 1982 (DPL 61)	EC 480 (rac) +COC	2 (39)	1.1	0.34 0.36	GS ns; 14 June	ns	ns	99	< 0.04 [SS] [LOQ=0.05]	TMU1027/B; 38AZ82-006; [Koubek, 1982, 405795]
Maricopa, AZ, USA, 1982 (DPL 61)	EC 480 (rac) +COC	2 (19)	1.1	0.32 0.33	GS ns; 16 June	ns	ns	97	< 0.04 [SS] [LOQ=0.05]	TMU1027/B; 42AZ82-016; [Koubek, 1982, 405795]
Bakersfield, CA, USA, 1982 (Acala SJ-2)	EC 480 (rac) +COC	2 (24)	1.1	0.31	GS ns; 18 June	ns	ns	116	< 0.04 [LOQ=0.05]	TMU1027/B; 41CA82-014; [Koubek, 1982, 405795]
Poplar,	EC	2	1.1	0.31	GS ns;	ns	ns	112	< 0.04	TMU1027/B;

COTTON SEED Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
CA, USA, 1982 (Acala SJ-2)	480 (rac) +COC	(18)			21 June				[LOQ=0.05]	41CA82-015; [Koubek, 1982, 405795]
idem	EC 480 (rac) +NIS	2 (18)	1.1	0.31	GS ns; 21 June	ns	ns	112	< 0.04 [LOQ=0.05]	TMU1027/B; 41CA82-015; [Koubek, 1982, 405795]
Rosedale CA, USA, 1982 (Acala SJ-2, Acala SJ-5)	EC 480 (rac) +COC	2 (37)	0.56	0.16	GS ns; 15 July	ns	ns	89	0.05	TMU1027/B; 41CA82-016; [Koubek, 1982, 405795]
idem	EC 480 (rac) +NIS	2 (37)	0.56	0.16	GS ns; 15 July	ns	ns	89	< 0.04 [LOQ=0.05]	TMU1027/B; 41CA82-016; [Koubek, 1982, 405795]
Waynesboro CA, USA, 1982 (variety ns)	EC 480 (rac) +COC	2 (40)	0.56	0.30 0.19	GS ns; 24 May	ns	ns	81	< 0.04 [LOQ=0.05]	TMU1027/B; 62GA82-021; [Koubek, 1982, 405795]
idem	EC 480 (rac) +NIS	2 (40)	0.56	0.30 0.19	GS ns; 24 May	ns	ns	81	< 0.04 [LOQ=0.05]	TMU1027/B; 62GA82-021; [Koubek, 1982, 405795]
Bastrop, TX, USA, 1982, (Stoneville 825)	EC 480 (rac) +COC	2 (18)	0.56	1.2 aerial	GS ns; 18 June	ns	ns	115	< 0.04 [LOQ=0.05]	TMU1027/B; 36LA82-018; [Koubek, 1982, 405795]
Oak Ridge, LA, USA, 1982 (DPL 55)	EC 480 (rac) +COC	2 (29)	0.56	1.2 aerial	GS ns; 6 July	ns	ns	97	< 0.04 [LOQ=0.05]	TMU1027/B; 36LA82-021; [Koubek, 1982, 405795]
St Joseph, LA, USA, 1982 (Stoneville 506)	EC 480 (rac) +COC	2 (17)	0.56	0.30	GS ns; 12 July	ns	ns	85	< 0.04 [LOQ=0.05]	TMU1027/B; 36LA82-045; [Koubek, 1982, 405795]
Clayton, NC, USA, 1982 (Coker 310)	EC 480 (rac) +COC	2 (14)	0.56	ns	GS ns; 19 July	ns	ns	85	< 0.04 [c], [SS] [LOQ=0.05]	TMU1027/B; 61NC82-012; [Koubek, 1982, 405795]
idem	EC 480 (rac) +NIS	2 (14)	0.56	ns	GS ns; 19 July	ns	ns	85	< 0.04 [c], [SS] [LOQ=0.05]	TMU1027/B; 61NC82-012; [Koubek, 1982, 405795]
Edmondson, TX, USA, 1982, (Paymaster 303)	EC 480 (rac) +COC	2 (38)	0.56	1.5 aerial	GS ns; 10 July	ns	ns	95	< 0.04 [LOQ=0.05]	TMU1027/B; 33TX82-029; [Koubek, 1982, 405795]
Bryan, TX, USA, 1982 (Stoneville 213)	EC 480 (rac) +COC	2 (24)	0.56	0.24 0.29	GS ns; 28 May	ns	ns	91	< 0.04 [LOQ=0.05]	TMU1027/B; 60TX82-013; [Koubek, 1982, 405795]
Bryan, TX, USA, 1982 (Stoneville 825)	EC 480 (rac) +COC	2 (36)	0.56	1.2 aerial	GS ns; 9 June	ns	ns	72	< 0.04 [LOQ=0.05]	TMU1027/B; 60TX82-014; [Koubek, 1982, 405795]
Bryan, TX, USA,	EC 480	2 (24)	0.56	0.29	GS ns; 28 May	ns	ns	70	0.12	TMU1027/B; 60TX82-015;

COTTON SEED Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
1982 (Stoneville 213)	(rac) +COC									[Koubek, 1982, 405795]
Ramer, AL, USA, 1983 (variety ns)	EC 480 (rac) + primeoil	2 (64)	0.28	0.19	Early bloom; 02 Aug	ns	MAT	89	0.05 [SS]	TMU1401/B; 45AL83-055; [Koubek, 1984, 405796]
idem	EC 240 (P) + primeoil	2 (64)	0.28	0.19	Early bloom; 02 Aug	ns	MAT	89	< 0.04 [SS] [LOQ=0.05]	idem
idem	EC 240 (P) + primeoil	2 (64)	0.42	0.28	Early bloom; 2 Aug	ns	MAT	89	< 0.04 [SS] [LOQ=0.05]	idem
Scott, AR, USA, 1983 (variety ns)	EC 480 (rac) + 1% COC	2 (58)	0.28	0.22	Blooming; 29 July	ns	MAT	88	< 0.04 [LOQ=0.05]	TMU1401/B; 06AR83-027; [Koubek, 1984, 405796]
idem	EC 240 (P) + 1% COC	2 (58)	0.28	0.22	Blooming; 29 July	ns	MAT	88	0.05	idem
idem	EC 240 (P) + 1% COC	2 (58)	0.42	0.33	Blooming; 29 July	ns	MAT	88	≤ 0.04 [LOQ=0.05]	idem
Avondale, AZ, USA, 1983 (variety ns)	EC 480 (rac) + crop oil	2 (33)	0.28	0.13	Pre-bloom; 22 June	ns	MAT	90	< 0.04 [LOQ=0.05]	TMU1401/B; 38AZ83-028; [Koubek, 1984, 405796]
idem	EC 240 (P) + crop oil	2 (33)	0.28	0.13	Pre-bloom; 22 June	ns	MAT	90	< 0.04 [LOQ=0.05]	idem
idem	EC 240 (P) + crop oil	2 (33)	0.42	0.19	Pre-bloom; 22 June	ns	MAT	90	≤ 0.04 [LOQ=0.05]	idem
Maricopa, AZ, USA, 1983 (variety ns)	EC 480 (rac) + crop oil	2 (35)	0.28	0.13	Early bloom; 7 July	ns	MAT	97	< 0.04 [LOQ=0.05]	TMU1401/B; 38AZ83-029; [Koubek, 1984, 405796]
idem	EC 240 (P) + crop oil	2 (35)	0.28	0.13	Early bloom; 7 July	ns	MAT	97	0.05	idem
idem	EC 240 (P) + crop oil	2 (35)	0.42	0.19	Early bloom; 7 July	ns	MAT	97	≤ 0.04 [LOQ=0.05]	idem
Poplar, CA, USA, 1983 (Acala SJ-5)	EC 480 (rac) + 1% Mor-Act	2 (32)	0.28	0.08	Squares, 21 June	ns	MAT	91	< 0.04 [LOQ=0.05]	TMU1401/B; 41CA83-013; [Koubek, 1984, 405796]
idem	EC 240 (P) + 1% Mor-Act	2 (32)	0.28	0.08	Squares; 21 June	ns	MAT	91	< 0.04 [LOQ=0.05]	idem
idem	EC 240 (P) + 1% Mor-Act	2 (32)	0.43	0.12	Squares; 21 June	ns	MAT	91	≤ 0.04 [LOQ=0.05]	idem
Rosedale, CA, USA, 1983	EC 480	2 (20)	0.28 0.28	0.15 0.10	Squares, 23 June	ns	MAT	90	< 0.04	TMU1401/B; 41CA83-019;

COTTON SEED Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Acala SJ-2 or SJ-5)	(rac) + 0.25% Spred Stik								[LOQ=0.05]	[Koubek, 1984, 405796]
idem	EC 240 (P) + 0.25% Spred Stik	2 (20)	0.28 0.28	0.15 0.10	Squares; 23 June	ns	MAT	90	< 0.04 [LOQ=0.05]	idem
idem	EC 240 (P) + 0.25% Spred Stik	2 (20)	0.43 0.43	0.23 0.15	Squares; 23 June	ns	MAT	90	<u>≤ 0.04</u> [LOQ=0.05]	idem
Cochran, GA, USA, 1983 (Stoneville 825)	EC 480 (rac) + 0.25% NIS	2 (19)	0.28	0.15	Early bloom; 15 July	ns	MAT	92	< 0.04 [LOQ=0.05]	TMU1401/B; 62GA83-013; [Koubek, 1984, 405796]
idem	EC 240 (P) + 0.25% NIS	2 (19)	0.28	0.15	Early bloom; 15 July	ns	MAT	92	< 0.04 [LOQ=0.05]	idem
idem	EC 240 (P) + 0.25% NIS	2 (19)	0.42	0.23	Early bloom; 15 July	ns	MAT	92	<u>≤ 0.04</u> [LOQ=0.05]	idem
Bosco, LA, USA, 1983 (DPL 55)	EC 240 (P) + 1% COC	2 (ns)	0.42	0.30	Early mid-square; 22 July	ns	MAT	90	<u>≤ 0.04</u> [LOQ=0.05]	TMU1401/B; 36LA83-035; [Koubek, 1984, 405796]
Bouina, TX, USA, 1983 (CAMD-E)	EC 480 (rac) + 0.25% NIS	2 (22)	0.28	0.19	Early bloom; 18 Aug,	ns	MAT	90	0.12 [b]	TMU1401/B; 72TX83-016; [Koubek, 1984, 405796]
idem	EC 240 (P) + 0.25% NIS	2 (22)	0.28	0.19	Early bloom; 18 Aug	ns	MAT	90	< 0.04 [LOQ=0.05]	idem
idem	EC 240 (P) + X77	2 (22)	0.42	0.29	Early bloom; 18 Aug	ns	MAT	90	<u>0.08</u>	idem
Goldsboro, NC, USA, 1983 (McNair 220)	EC 480 (rac) + 0.25% NIS	2 (32)	0.28	0.10	Blooming; 29 July	ns	MAT	89	< 0.04 [LOQ=0.05]	TMU1401/B; US1-83-S101; [Koubek, 1984, 405796]
idem	EC 240 (P) + 0.25% NIS	2 (32)	0.28	0.10	Blooming; 29 July	ns	MAT	89	< 0.04 [LOQ=0.05]	idem
idem	EC 240 (P) + 0.25% NIS	2 (32)	0.42	0.15	Blooming; 29 July	ns	MAT	89	<u>≤ 0.04</u> [LOQ=0.05]	idem
Elco, SC, USA, 2008	EC 240 (P) +0.25%	2 (14)	0.42 0.42	0.90 0.90	BBCH 71 – mid bloom;	LSa	MAT	90	0.015, 0.016, mean	T002224-07, E11SC 081311; [Mazlo, 2009;

COTTON SEED Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(DP 555/ BG/RR)	NIS				30 July				0.016 [a],	PP5_50076]
Fisk, MO, USA, 2008 (DP 445/ BG/RR)	EC 240 (P) + +0.25% NIS	2 (14)	0.41 0.42	0.22 0.22	BBCH 60 - first flowers open; 9 Aug	SiL	MAT	90	< 0.01, < 0.01 mean ≤ 0.01 [a],	T002224-07, C23MO 081312; [Mazlo, 2009; PP5_50076]
Proctor, AR, USA, 2008 (DG2215B2RF)	EC 240 (P) + +0.25% NIS	2 (14)	0.42 0.42	0.47 0.46	Squaring 29 June	SiCL	MAT	90	< 0.01, < 0.01 mean ≤ 0.01 [a]	T002224-07, C24AR 081313; [Mazlo, 2009; PP5_50076]
Cotton Plant, AR, USA, 2008 (DP 445/ BG/RR)	EC 240 (P) + +0.25% NIS	2 (14)	0.43 0.41	0.34 0.32	BBCH 36; 30 July	L	MAT	90	< 0.01, < 0.01 mean ≤ 0.01 [a],	T002224-07, E13GA 081314; [Mazlo, 2009; PP5_50076]
Wharton, TX, USA, 2008 (DP 445/ BG/RR)	EC 240 (P) + +0.25% NIS	2 (14)	0.42 0.42	0.21 0.22	BBCH 59-63; 0 June	SiL	MAT	90	< 0.01, < 0.01 mean ≤ 0.01 [a]	T002224-07, W05TX 081315; [Mazlo, 2009; PP5_50076]
Uvalde, TX, USA, 2008 (DP434)	EC 240 (P) + +0.5% NIS	2 (14)	0.42 0.42	0.39 0.18	BBCH 59; June 6	CL	MAT	90	< 0.01, < 0.01 mean ≤ 0.01 [a]	T002224-07, W07TX 081316; [Mazlo, 2009; PP5_50076]
Claude, TX, USA, 2008 (ST4554RF)	EC 240 (P) + +0.5% NIS	2 (14)	0.43 0.42	0.18 0.17	BBCH 66; 15 Aug	CL	MAT	90	0.67 [b], 0.75 [b], mean 0.71 [a]	T002224-07, E13TX 081317; [Mazlo, 2009; PP5_50076]
Levelland, TX, USA, 2008 (FM9063 B2F)	EC 240 (P) + +0.3–0.4% NIS	2 (14)	0.41 0.41	0.22 0.22	Bloom; 5 Aug	L	MAT	90	0.045, 0.047 mean 0.046 [a]	T002224-07, W39TX 081318; [Mazlo, 2009; PP5_50076]
Sanger, CA, USA, 2008 (PHY725RF Acala)	EC 240 (P) + +0.3% NIS	2 (14)	0.42 0.42	0.16 0.16	BBCH 65; 07 Aug	SaL	MAT	90	0.042, 0.045 mean 0.044 [a]	T002224-07, W31CA 081319; [Mazlo, 2009; PP5_50076]
Firebaugh, CA, USA, 2008 (YDO2-5)	EC 240 (P) + +0.5% NIS	2 (14)	0.42 0.41	0.34 0.31	BBCH 43; 03 July	CL	MAT	90	< 0.01, < 0.01 mean ≤ 0.01 [a],	T002224-07, W27CA 081320; [Mazlo, 2009; PP5_50076]
Kettleman City, CA, USA, 2008 (Deltapine 340 Pima)	EC 240 (P) + +0.5% NIS	2 (14)	0.41 0.42	0.30 0.27	BBCH 61; 13 July	SaL	MAT	90	0.080, 0.098 mean 0.089 [a],	T002224-07, W27CA 081321; [Mazlo, 2009; PP5_50076]
Jaboticabal, SP, Brazil, 2011 (Delta Opal)	EW 250 (P)	1	0.25	0.083	BBCH 72-73; 25 Febr	C	90	60	≤ 0.01	M11027; AMA; [Draetta, 2012, A12530B_10014]
Uberlândia, MG, Brazil, 2011 (Delta Opal)	EW 250 (P)	1	0.25	0.083	BBCH78; 19 May	C	92	60	≤ 0.01	M11027; JJB; [Draetta, 2012, A12530B_10014]
Bandei rantes,	EW 250	1	0.25	0.083	BBCH65;	C	89	60	≤ 0.01	M11027;

COTTON SEED Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
PR, Brazil, 2011 (Delta Opal)	(P)				21 Jan					DMO; [Draetta, 2012, A12530B_10014]
Cabeceiras, GO, Brazil 2011 (Delta Opal)	EW 250 (P)	1	0.25	0.083	BBCH75; 28 March	C	94	60	< 0.01	M11027; MFG; [Draetta, 2012, A12530B_10014]
Almussafes; Valencia; Spain, 1987 (variety ns)	EC 300 (P)	1	0.3	ns	20% crop cover; 25 June	ns	ns	127	< 0.03 [SS] [LOQ=0.05]	M4799B ES01-87-D006-E; [Crook, 1988, PP5/0380]
Groblers daal, South Africa, 1991 (Acala 151788)	EC 125 (P)	1	0.25	ns	6-8 leaves; 15 Jan, 1991	SaL	MAT	148	< 0.05 [SS]	RJ1131B ZA10-91-H022 [Bolygo, 1992, PP5/0576]
idem	EC 125 (P)	1	0.50	ns	6-8 leaves; 15 Jan, 1991	SaL	MAT	148	< 0.05 [SS]	idem
idem	EC 125 (P)	1	1.0	ns	6-8 leaves; 15 Jan, 1991	SaL	MAT	148	< 0.05 [SS]	idem

[SS] Sample sizes not stated (PP009B0035andTMU0679/B, RR 90-075B, RJ1131B ,M4799B,) or less than the required 1 kg cotton seeds without lints (0.9 kg for HU2-79-10 and 45AL83-055); samples considered not representative for MRL derivation

[a] Results came from replicate field samples; the mean is taken for MRL derivation if according to cGAP

[b] Results came from 3-4 replicate analyses per sample for confirmation of the results; the mean per sample is reported.

[c] Raw field data not included in the report.

Additional trial information:

TMU0679/B and PP009B035 Non-GLP study. Weather conditions, soil type not stated. Over the top application with tractor mounted equipment or hand-held boom sprayer, spray volume 21-41 GPA i.e. 200-380 L/ha. Sample sizes not stated, except for HU2-79-10 (2 lbs = 0.90 kg and 33 lbs= 15 kg for oil processing). Sample sizes (3-5 lbs=1.4-2.3 kg) for 30TX80-001, 20TX80-007, 28GA80-001, and 29MS80-017 were stated in TMU 0987/B. Samples of mature cottonseed were ginned up to 6 days after harvest, ground and then stored at -23 °C or lower for a maximum of 238 days (1980 trials) or 556 days (1979 trials). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 53/1 (i.e. PPRAM 62) with a valid LOQ of 0.05 mg/kg**. Samples were corrected for average concurrent recovery (99% at 0.1 mg/kg); uncorrected results were not available. Control samples < 0.02 mg/kg.

TMU0987/B. Non-GLP study. Weather conditions, soil type not stated. Ground spray equipment, spray volume 21-36 GPA i.e. 196-336 L/ha. Sample sizes 3-5 lbs = 1.4-2.3 kg. Storage at -23 °C or lower. Duration not stated, but less than a year. Samples were then ginned, ground and analysed. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Average concurrent recovery is105.3% for fluazifop and 99.9% for fluazifop-butyl; uncorrected results were not available. Control samples < 0.04 mg/kg.

TMU1027/B. Non-GLP study. Weather conditions, soil type not stated. Ground spray equipment, spray volume 13-38 GPA i.e. 120-360 L/ha. Areal treatment spray volume 4-5 GPA i.e. 37-47 L/ha. Sample sizes 3-5 lbs = 1.4-2.3 kg, except 2 lbs = 0.90 kg in trial 42AZ82-006. Storage at -23 °C or lower . Duration not stated, but less than a year. Samples were then ginned, ground and analysed. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Average concurrent recovery (85.5%); uncorrected results were not available. Control samples < 0.04 mg/kg.

TMU1401/B. Non-GLP study. Weather conditions, soil type not stated. Ground spray equipment, spray volume 13-38 GPA i.e. 120-36 L/ha. Sample sizes 5 lbs = 2.3 kg, except 2 lbs = 0.90 kg in trial 45AL83-055. Storage at -23 °C or lower for a maximum of 149 days Samples were then ginned, ground and analysed. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/1 with a valid LOQ of 0.05 mg/kg**. Average concurrent recovery (94%); uncorrected results were not available. Control samples < 0.04 mg/kg.

T002224-01; GLP study. No unsual weather conditions. Tractor mounted or backpack ground sprayer. Spray volume 47.7-246 L/ha. Replicate samples of seed cotton were picked by hand (3 lbs >1.4 kg, 81311, 81312, 81314, 81320, 81321) or by mechanical picking (100 lbs, >45 kg) or by mechanical stripper (75 lbs, > 34 kg). Seed cotton samples were processed into commercially representative undelinted cottonseed. Samples were stored frozen at <-23 °C for max 228 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.01A with a valid LOQ of**

0.01 mg/kg. Samples were not corrected for average concurrent recoveries (90-102% 0.01-1.0 mg/kg). Control samples were < 0.01 mg/kg.

M11027: GLP sudy: No unusual weather conditions. Tractor mounted ground sprayer. Spray volume 300 l/ha. Seed cotton was obtained from at least 12 representative points. Cotton bolls were delinted mechanically to produce 1 kg of cotton seeds. After processing samples were stored frozen for less than 12 months before analysis. Samples were analysed for total fluazifop using **HPLC-MS/MS method POPIT MET.138.Rev.04 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for method recovery (78-86%, 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg

M4799B Non-GLP study. Weather conditions, soil type, application equipment, spray volume, growth stage at harvest not stated. Sample sizes not stated. Storage at -18 °C; for maximum 183 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Internal standard recovery (98% at 0.1 mg/kg). Control samples < 0.03 mg/kg.

RJ1131B GLP study. No unusual on weather conditions. Equipment not stated. Spray volume not stated. Samples of cotton balls (1 kg) were picked by hand (1 kg). Seed cotton were separated from the lints to produce cotton seeds (sample size not stated). Samples were stored frozen at <-18 °C for max 233 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for average concurrent recoveries (87% 0.05-0.5 mg/kg). Control samples were < 0.05 mg/kg.

Linseed

Two cGAPs are available:

- cGAP from France with 1 ×0.38 kg ai/ha with a PHI of 90 days
- cGAP from the UK with 1 ×0.19 kg ai/ha before visible flower bud stage

Trials from Canada (1979, 1980) were available on linseed (flax) with an application of 1 × 0.25–0.40–0.50–0.60–1.0 kg ai/ha and harvest at 98–134 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Since the manufacturer did not intend to have MRLs on linseed, the available studies on linseed were not summarized.

Some supervised trials on linseed were not submitted: [reports RJ1485B and RJ1099B, not referenced]. No further efforts were taken to retrieve these studies.

Oilseed rape seed

Four cGAPs for oilseed rape are available:

- cGAP from Brazil and the UK with 1 ×0.19 kg ai/ha and a PHI of 14 days
- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 90 days (underlining nn)
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha before winter (i.e. end of December) and post-emergence of the crop
- cGAP from Belgium with 1 × 0.19 kg ai/ha before winter (i.e. end of December) and up to 15 cm crop height

Trials that could be matched to these cGAPs were summarized.

Table 236 lists trials conducted in the Germany (1982–1983, 1992–1993, 1997–1998), the UK (1992, 1998), Sweden (1981), Southern France (2001–2002, 2002–2003, 2012–2013), Italy (2011–2012) and Spain (1997–1998, 2012). A broadcast or banded foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 236. Results marked with "[QU]", "[SS]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample size was less than the required 1 kg seeds.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

Additional trials from the UK (1979–1980) were available with 1 × 0.5–1.0–2.0 kg ai/ha with harvest at 3.0–3.5 and 8.0–11 months after treatment or 2 × 0.50 kg ai/ha with harvest at 3 months after treatment [Atreya, 1981, PP9/0509report PP009B015; Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from Germany (1979–1980) were available on oilseed rape seeds with 2 × 0.50 kg ai/ha with harvest at 253–256 DAT or 1 × 1.0 kg ai/ha with harvest at 256–276 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. Additional trials from Canada (1979, 1980) were available with 1 ×0.12–0.20–0.25–0.40–0.50–0.75–1.0 kg ai/ha with harvest at 60–67, 69–83, 88–93, 95–112 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. These trials were summarized in the metabolism section, because only the fluazifop-butyl and free fluazifop residues were analysed.

Additional trials from the UK (1981, 1993–1994) were available on winter oilseed rape with 2 applications of 0.19+0.12 kg ai/ha or 0.50+1.0 kg ai/ha with harvest at 90 or 115–216 DAT or 1 × 0.50–1.0 kg ai/ha with harvest at 240–300 days [Atreya and Harradine, 1982, PP9/0062, report RJ0291B; Bolygo and Thornton, 1995, report RJ1837B, not submitted]. Additional trials from Germany (1981, 1993–1994) were available on spring or winter oilseed rape with 1 × 0.31–0.75 kg ai/ha and harvest at 98–112 DAT or 1 ×0.38 kg ai/ha with harvest at 259–266 days [Atreya and Harradine, 1982, PP9/0062, report RJ0291B; Bolygo, 1995, PP5/0220, report RJ1846B].These trials were not summarized, because they would not assist in MRL setting.

Table 236 Supervised field trials on oilseed rape (seeds), treated with a broadcast or banded foliar fluazifop-butyl spray

OILSEED RAPE SEEDS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
2411 Klein-Zecher/Mollin Germany; 1982-83; (autumn sown: Belinda)	EC 125 (P)	1	0.38	0.094	30 cm high; 7- 9 leaves; crop cover ns; 10 Apr 1983	ns	ns	116	2.1 [SS]	M3685B; RS 8370 B1; [Upton, 1984, PP9/0399]
idem	EC 250 (rac)	1	0.75	0.19	30 cm high; 7- 9 leaves; crop cover ns; 10 Apr 1983	ns	ns	116	1.7 [SS]	M3685B; RS 8370 B1; [Upton, 1984, PP9/0399]
2059 Krukow-Lavenberg E; Germany; 1983 (spring sown: Ergula)	EC 125 (P)	1	0.38	0.094	30 cm high; 7-9 leaves; crop cover ns; 10 May	ns	ns	102	1.5 [SS]	M3685B; RS 8370 B2; [Upton, 1984, PP9/0399]
idem	EC 250 (rac)	1	0.75	0.19	30 cm high; 7-9 leaves; crop cover ns; 10 May	ns	ns	102	2.5 [SS]	M3685B; RS 8370 B2; [Upton, 1984, PP9/0399]
3141 Brakeded-Bleckede Germany; 1982-83; (autumn sown: Jet neuf)	EC 125 (P)	1	0.38	0.094	30 cm high; 7-9 leaves; crop cover ns; 18 Apr 1983	ns	ns	97	2.6 [SS]	M3685B; RS 8370 B3; [Upton, 1984, PP9/0399]
idem	EC	1	0.75	0.19	30 cm high;	ns	ns	97	0.4	M3685B;

OILSEED RAPE SEEDS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	250 (rac)				7-9 leaves; crop cover ns; 18 Apr 1983				[SS]	RS 8370 B3; [Upton, 1984, PP9/0399]
Pirmasens-Windberg; Germany; 1982-83; (autumn sown: Belinda)	EC 125 (P)	1	0.38	0.094	40 cm high; Flower buds developing; crop cover 75%; 19 Apr 1983	ns	ns	90	3.2 [SS]	M3685B; RS 8370 E1; [Upton, 1984, PP9/0399]
idem	EC 250 (rac)	1	0.75	0.19	40 cm high; Flower buds developing; crop cover 75%; 19 Apr1983	ns	ns	90	4.4 [SS]	M3685B; RS 8370 E1; [Upton, 1984, PP9/0399]
23919 Berkenthin; Germany; 1992-93; (autumn sown: Lirajet)	EC 125 (P)	1	0.38	0.12	30 cm high; BBA 39; 90% crop cover; 8 Apr 1993	SaL	BBA 92	116	2.9	RJ1660B; RS-9304-B1; [Bolygo, 1994; PP5/0217]
idem	ME 125 (P)	1	0.38	0.12	30 cm high; BBA 39; 90% crop cover; 8 Apr 1993	SaL	BBA 92	116	3.2; 3.3 mean 3.2 [b]	RJ1660B; RS-9304- B1; [Bolygo, 1994, PP5/0217] and RJ1684B; RS-9306-B1; [Bolygo, 1994b, PP5/1105] (processing)
85375 Neufarn; Germany; 1992-93; (autumn sown: Lirabon)	EC 125 (P)	1	0.38	0.19	20 cm high; BBA 37; 80% crop cover; 14 Apr 1993	L	BBA 92	101	1.7	RJ1660B; RS-9304-G1; [Bolygo, 1994, PP5/0217]
idem	ME 125 (P)	1	0.38	0.19	30 cm high; BBA 37; 80% crop cover; 14 Apr 1993	L	BBA 92	101	1.5; 2.4 mean <u>2.0</u> [b]	RJ1660B; RS-9304-G1; [Bolygo, 1994, PP5/0217] and RJ1684B; RS-9306-B1; [Bolygo, 1994b, PP5/1105] (processing)
D-85395 Wolfers-dorf-Billingsdorf; Germany; 1997-98; (autumn sown: Lirajet)	EC 125 (P))	1	0.38	0.19	10-15 cm high; 50% crop cover; 1 Apr 1998	SaL	ns	109	<u>1.5</u>	RJ2766B; RS-9812-G1; [Mason and Chamier, 1999, PP5/0210]
idem	EC	1	0.38	0.19	10-15 cm	SaL	ns	109	1.1	RJ2766B;

OILSEED RAPE SEEDS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	125 (P)				high; 50% crop cover; 1 Apr 1998					RS-9812-G1; [Mason and Chamier, 1999, PP5/0210]
D-85405 Nandlstadt-Wadensdorf; Germany; 1997-98; (autumn sown: Express)	EC 125 (P)	1	0.38	0.19	15 cm high; 60% crop cover; 1 Apr 1998	SaL	ns	110	2.2	RJ2766B; RS-9812-G2; [Mason and Chamier, 1999; PP5/0210]
idem	EC 125 (P)	1	0.38	0.19	15 cm high; 60% crop cover; 1 Apr 1998	SaL	ns	110	1.7	RJ2766B; RS-9812-G2; Mason and Chamier, 1999; PP5/0210]
Büchen; Schleswig-Holstein; Germany; 1998; (spring sown:: Lambada)	EC 125 (P)	1	0.38	0.19	BBCH 50; 15-25 cm high; 45% crop cover; 25 May 1998	LSa	CH	85	1.7 [SS]	RJ2806B; RS-9814-B1; [Mason and Chamier, 1999, PP5/0211]
idem	EC 125 (P)	1	0.38	0.19	BBCH 50; 20-25 cm high; 65% crop cover; 25 May 1998	LSa	CH	85	2.2 [SS]	RJ2765B; RS-9816-B1; [Mason and Chamier, 1999, PP5/0209]
idem	EC 125 (P)	1	0.38	0.19	BBCH 50; 20-25 cm high; 65% crop cover; 25 May 1998	LSa	CH	85	2.5 [SS]	idem
Kladow; Mecklenburg-Vorpommern; Germany; 1998; (spring sown:: Optima)	EC 125 (P)	1	0.38	0.19	BBCH 50; 10-15 cm high; 70% crop cover; 29 May 1998	LSa	CH	102	0.71 [SS]	RJ2806B; RS-9814-R1; [Mason and Chamier, 1999, PP5/0211]
idem	EC 125 (P)	1	0.38	0.19	BBCH 50; 10-15 cm high; 70% crop cover; 29 May 1998	LSa	CH	102	1.3 [SS]	RJ2765B; RS-9816-R1; [Mason and Chamier, 1999, PP5/0209]
idem	EC 125 (P)	1	0.38	0.19	BBCH 50 10-15 cm high; 70% crop cover; 29 May 1998	LSa	CH	102	1.8 [SS]	idem
Lighthorne, Warkshire; UK, 1992	EC 125 (P) +	1	0.19	0.094	3-4 leaves; no flower buds	SaL	MAT	114	0.27 [SS]	RJ1456B; GB15-92-S111 [Patel *et al*.,

OILSEED RAPE SEEDS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(spring sown: Bingo)	0.1% NIS				visible; 50% crop cover; 19 May					1993, PP5/0564]
idem	EW 125 (P) + 0.1% NIS	1	0.19	0.094	3-4 leaves; no flower buds visible; 50% crop cover 19 May	SaL	MAT	114	0.25 [SS]	idem
idem	EW 250 (P) + 0.1% NIS	1	0.19	0.094	3-4 leaves; no flower buds visible; 50% crop cover 19 May	SaL	MAT	114	0.22 [SS]	idem
Ufton Fields Warkshire, UK, 1992, (spring sown: Puma)	EC 125 (P) + 0.1% NIS	1	0.19	0.094	2-3 leaves; no flower buds visible; 40% crop cover; 20 May	SaCL	MAT	107	0.06, 0.23 [a], [SS]	RJ1456B; GB15-92-S112 and S113 [Patel *et al*., 1993, PP5/0564]
idem	EW 125 (P) + 0.1% NIS	1	0.19	0.094	2-3 leaves; no flower buds visible; 40% crop cover; 20 May	SaCL	MAT	107	0.06, 0.19 [a], [SS]	idem
idem	EW 250 (P) + 0.1% NIS	1	0.19	0.094	2-3 leaves; no flower buds visible; 40% crop cover; 20 May	SaCL	MAT	107	0.20, 0.20 [a], [SS]	idem
Upper Billesley, Stratford, UK, 1992, (spring sown: Bingo)	EC 125 (P) + 0.1% NIS	1	0.19	0.094	4-5 leaves; no flower buds visible; 60% crop cover; 20 May	CL	MAT	110	0.80 [SS] [CT] [Cntrl=0.23]	RJ1456B; GB15-92-S114 [Patel *et al*., 1993, PP5/0564]
idem	EW 125 (P) + 0.1% NIS	1	0.19	0.094	4-5 leaves; no flower buds visible; 60% crop cover; 20 May	CL	MAT	110	0.50 [SS] [CT] [Cntrl=0.23]	idem
idem	EW 250 (P) + 0.1% NIS	1	0.19	0.094	4-5 leaves; no flower buds visible; 60% crop cover; 20 May	CL	MAT	110	0.71 [SS] [CT] [Cntrl=0.23]	idem
Berkswell;	EC	1	0.38	0.19	BBCH 50;	SaCL	MAT	112	0.28	RJ2758B;

OILSEED RAPE SEEDS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Warwickshire; UK; 1998; (spring sown: Sprinter)	125 (P)				5-7 cm high; 18 May 1998				[SS]	GB15-98-S111; [Mason and Codd, 1999; PP5/0212]
Leamington Spa; Warwickshire; UK; 1998; (spring sown:: Liason)	EC 125 (P)	1	0.38	0.19	BBCH 50; 6-9 cm high; 19 May 1998	CL	MAT	107	0.83 [SS]	RJ2758B; B15-98-S112; [Mason and Codd, 1999; PP5/0212]
Location ns; Sweden, 1981 (summer rape; var ns)	EC 250 (rac)	1	0.5	ns	GS ns date ns	ns	ns	78 88 90	2.5 1.6 4.2 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	84 95 96	1.4 0.81 4.7 [QU]	idem
Location ns; Sweden, 1981 (winter rape; var ns)	EC 250 (rac)	1	0.5	ns	GS ns date ns	ns	ns	84 104 110	2.0; 0.61, 0.83; 1.1 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
F-82200; Moissac; S-France; 2001-02; (autumn sown: Lutin)	EC 125 (P)	1	0.38	0.12	BBCH 50; crop cover ns; 11 March 2002	CL	ns	105	4.7, 4.6; mean 4.6 [b] [SS]	02-7015; plot 1; [Mason, 2003, PP5/1256]
82170 Pompignan; S-France; 2002-03; (autumn sown: Constant)	EC 125 (P)	1	0.38	0.13	BBCH 39; crop cover ns; 7 March 2003	SiC	89	101	2.3	03-7004; plot 1; [Mason, 2003, PP5/1365]
35590 Marsillargues; S-France; 2002-03; (autumn sown: Spirit)	EC 125 (P)	1	0.39	0.13	BBCH 39; crop cover ns; 11 March 2003	SiC	99	100	2.2	03-7005; plot 1; [Mason, 2003, PP5/1367]
Nîmes; Languedoc-Roussillon; S-France; 2012-2013; (autumn sown: ES Mercure)	EC 125 (P)	1	0.38	0.12	BBCH 15-17; height 25.5 cm; crop cover ns; 12 Mar 2013	SiCL	89	108	6.3 [SS]	CEMR-5449; SRFR12-015-37HR; [Langridge, 2013; A12791B_11249]
Costigliole d'Asti; Piedmont; Italy; 2011-12; (autumn sown: Hybristar)	EC 125 (P)	1	0.37	0.11	BBCH 39; crop cover ns; 5 Apr 2012	ns	89	81	4.2 [SS]	CEMR-5449; SRIT12-1032-37HR; [Langridge, 2013; A12791B_11249]
Casseres del Castillo; Huesca, Spain,	EC 125 (P)	1	0.38	0.13	BBCH 50; height 50 cm; crop cover ns;	C	89	86	0.35 [SS]	RJ2771B; ES10-98-SH006; [Ryan and Gallardo, 1999,

OILSEED RAPE SEEDS Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
1997-98; (autumn sown: Kreta)					1 Apr 1998					PP5/0208]
Saganta; Huesca; Spain; 1997-98; (autumn sown: Kreta)	EC 125 (P)	1	0.39	0.12	BBCH 50; height 40 cm; crop cover ns; 1 Apr 1998	LC	89	86	0.47 [SS]	RJ2771B; ES10-98-SH106; [Ryan and Gallardo, 1999, PP5/0208)
Banded foliar spray over rows										
47100 Tordesillas; Vallodolid; Spain; 2012; (spring sown: Belinda)	EC 125 (P)	1	0.38	0.12	BBCH 18-34; height 20 cm; crop cover ns; 9 May 2012	SaL	88-89	65	3.5 [SS]	CEMR-5449; SRES12-204-37HR; [Langridge, 2013; A12791B_11249]

BBCH 20-29: Formation of side shoots. BBCH 30-39: stem elongation. BBCH 50: flower buds present, still enclosed by leaves.
[cntrl] Residue level found in the untreated control sample
[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample
[SS] Sample size not stated (M3865B) or less than the required 1 kg seeds (0.5 kg in RJ1456B, RJ2758B, 02-7015, 0.5-0.6 kg in CEMR-5449, 0.8-0.9 kg in RJ2765B, RJ2806B); results considered not representative for MRL derivation.
[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported
[a] Results came from replicate plots; the highest value is taken for MRL derivation if according to cGAP
[b] Results came from replicate field samples (from the same plot); mean is taken for MRL derivation if according to cGAP

Additional trial information

M3685B, GLP study. Winter oilseed rape, sown on 18-28 August 1982, treated in April 1983. Spring oilseed rape. Weather conditions not stated. Boom sprayer or knapsack boom sprayer with spray volume 400 L/ha. Seeds were harvested by hand or by combine harvester; foliage was harvested by hand. Sample sizes were not stated. Storage at -18 °C or lower for maximum 127 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Average internal standard recoveries were 72-75%. Control samples were < 0.05 mg/kg.

RJ1660B, GLP study. Winter oilseed rape. No unusual weather conditions. Application by air powered knapsack boom sprayer with spray volume 200-300 L/ha. Sampling were harvested by hand (B1) or with a small plot harvester (G1). Sample size 1.0-1.5 kg seeds. Storage at -18 °C for maximum 11 months. Samples were analysed for total fluazifop using **NMR method RAM 197/01 with a valid LOQ of 0.05 mg/kg**. Seed samples were not corrected for concurrent individual method recoveries (80-109% at 0.05-1.0 mg/kg). Control samples were < 0.05 mg/kg.

RJ1684B, GLP Study, used for processing. Identical to study RJ1660b. Seeds were harvested by a small plot harvester. Except: Sample size for processing 8-10 kg seeds. Storage at -18 °C for 1-3 months. Samples were analysed for total fluazifop using **NMR method RAM 197/02 with a valid LOQ of 0.05 mg/kg**. Average concurrent recovery 90-117% at 0.1-1.0 mg/kg. Control samples < 0.05 mg/kg.

RJ2766B, GLP Study. Winter oilseed rape. No unusual weather conditions. Formulations used: YF7660A or YF10880 (EC 125). Application by air pressurised knapsack plot sprayer. Spray volume 200 L/ha. Sample size 1 kg seeds. Storage at -18 °C for 87-88 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Seed samples were not corrected for concurrent method recoveries (97-102% at 0.01-1.0 mg/kg) Control samples < 0.01 mg/kg.

RJ2806B. GLP study. Field conditions identical toYF7660A in report RJ2765B. Spring oilseed rape. No unusual weather conditions. Air pressurised knapsack sprayer with hand held boom. Spray volume 200 L/ha. Seed samples were taken by hand from at least 30-40 spots across the plot. Sample size 0.8-0.9 kg seeds. Storage at -18 °C for 90-111 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent method recoveries (78-91% at 1-5 mg/kg) Control samples < 0.01 mg/kg.

RJ2765B. GLP study. Field conditions for YF7660A identical to report RJ2806B. Spring oilseed rape. No unusual weather conditions. Air pressurised knapsack sprayer with hand held boom. Spray volume 200 L/ha. Seed samples were taken by hand from at least 30-40 spots across the plot. Sample size 0.8-0.9 kg seeds. Storage at -18 °C for 66-87 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent method recoveries (90-99% at 0.1-1 mg/kg) Control samples < 0.01 mg/kg.

RJ1456B. GLP study. Spring oilseed rape. No unusual weather conditions. Hand held small plot sprayers. Spray volume 200 L/ha. Seeds were sampled using a small plot combine harvester. Sample size 0.5 kg seeds. Storage at -20 °C for 177 days. Samples were analysed for total fluazifop using **NMR method ARAM 197 with a valid LOQ of 0.05 mg/kg**. Samples were not

corrected for individual concurrent method recoveries (76-94% at 0.1–0.5 mg/kg) Control samples < 0.05 mg/kg, except in trial 114, where 0.23 mg/kg was measured.
RJ2758B. GLP study. Spring oilseed rape. Unusual weather conditions did not affect crop health. Broadcast foliar spray using a hand held boom sprayer. Spray volume 200 L/ha. Seeds were sampled using a small plot combine harvester. A minimum of 12 grab samples were taken from the grain elevator as the plot was harvested. Sample size 0.5 kg seeds. Storage at -18 °C for 91-95 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent method recoveries (81-86% at 1-5 mg/kg) Control samples < 0.01 mg/kg, except in trial GB15-98-S112, where 0.05 mg/kg was measured.
RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.05 mg/kg.
02-7015, GLP study. Winter oilseed rape. No unusual weather conditions. Plot sprayer with spray volume 304 L/ha. Mature rape seed plants were sampled by hand; whole plants were threshed by hand to produce seed (0.5 kg). Storage at -14 °C or lower for maximum of 147 days (seed). Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Seed samples were not corrected for concurrent method recoveries (98-100% at 0.5-2.0 mg/kg) Control samples < 0.05 mg/kg (1).
03-7004, GLP study. Winter oilseed rape. No unusual weather conditions. Knapsack sprayer with spray volume 300 L/ha. Mature rape seeds were sampled with a combine harvester to produce 1.4 kg seeds. Storage at -12 °C or lower for maximum of 140 days (seed). Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Seed samples were not corrected for concurrent method recoveries (99-102% at 0.1–0.5 mg/kg) Control samples were 0.02 mg/kg (1).
03-7005, GLP study. Winter oilseed rape. No unusual weather conditions. Knapsack sprayer with spray volume 300 L/ha. Mature rape seeds were sampled with a combine harvester to produce 1.1 kg seeds. Storage at -12 °C or lower for maximum of 148 days (seed). Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Seed samples were not corrected for concurrent method recoveries (92-98% at 0.1–0.5 mg/kg) Control samples < 0.01 mg/kg (1).
RJ2771B, GLP study. Winter oilseed rape. No unusual weather conditions. Motor knapsack sprayer with spray volume 300-310 L/ha. Mature rape seed with pods (1-2 kg) were sampled by hand avoiding plot boundaries. Seeds were separated from the pods by hand. Seed sample sizes were not stated. Storage at -18 °C or lower for maximum of 174 days (seeds). Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Seed samples were not corrected for concurrent method recoveries (94-101% at 0.1–0.5 mg/kg). Control samples < 0.01 mg/kg (2).
CEMR-5449, GLP study. Winter and Spring oilseed rape. No unusual weather conditions. Backpack broadcast foliar (015-37HR and 1032-37HR) or banded foliar spray (204-37HR) with spray volume 308-345 L/ha. Mature plants were collected by hand from at least 12 places in the plot. Plants were threshed by hand to produce 0.52–0.66 kg seeds . Storage at -6°C or lower for maximum of 220 days (seed). Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Seed samples were not corrected for concurrent method recoveries (73-104% at 0.01, 0.1, 7.5 mg/kg; 68% at 0.5 mg/kg). Control samples < 0.01 mg/kg (3).

Peanuts

One cGAPs for peanuts is available:

- cGAP from the USA with 2 ×0.42 kg ai/ha with a PHI of 40 days (and restriction for feeding immature peanut plants or peanut seeds)

None of the available supervised trials on peanuts were submitted: Koubek, 1984, TMU1196/B, MRID 40831302; Francis and Plyler, 1986, TMU1978, MRID 40831306; Hayward, 1988, M4600B, MRID 40831308; Francis, 1989, TMU1196/B supplement, MRID 41165101; Stewart, 2001, RR/00/064B, MRID 47285503. As the manufacturer did not seek to have maximum residue levels estimated on peanuts, no further efforts were taken to retrieve these studies.

Sunflower seed

Two cGAPs for sunflowers are available:

- cGAP from Brazil with 1 ×0.25 kg ai/ha with a PHI of 59 days (underlining nn)
- cGAP from France with 1 ×0.38 kg ai/ha with a PHI of 90 days (underlining nn)

Trials that could be matched to these cGAPs were summarized.

Table 237 lists trials conducted in the USA (1989), Brazil (2007), Germany (1993, 2000), France (1984, 1999, 2005), Hungary (2002), Italy (1996, 1997) and Spain (1996, 1997, 2000). A broadcast or banded foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted

under the conditions listed in Table 237. Results marked with "[RT]", "[SS]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP.

[RT] indicates that the samples were heavily infested with Botrytis.

[SS] indicates that the sample size was not stated or less than the required 12 heads or 1 kg seeds.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

Additional trials from Canada (1979) were available on sunflowers with 1 ×0.50 kg ai/ha and harvest at 130–137 DAT [Atreya *et al*., 1980, PP9/0501, report PP009B007; Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. These trials were summarized in the metabolism section, because only the fluazifop-butyl and free fluazifop residues were analysed.

Additional trials from Canada (1980, 1981) were available on sunflowers with 1 ×0.15–0.20–0.25–0.27–0.40–0.50 kg ai/ha and harvest at 118–134 DAT or 1 ×0.60–1.0 kg ai/ha and harvest at 106 DAT [Atreya *et al*., 1981, PP9/0523, report PP009B034; Atreya and Froggatt, 1981, PP9/0384, report RJ0226B; Atreya and Harradine, 1982, PP9/0062, report RJ0291B]. These trials were not summarized because they would not assist in MRL setting.

Besides total fluazifop, also CF3-pyridone (X) was analysed in sunflower seed samples from the 1989 trials conducted in the USA [Alferness and Kleinschmidt, 1991, PP5/0233, report RR 91-010B]. These trials were summarized in the metabolism section.

Table 237 Supervised field trials on sunflower (seeds), treated with a broadcast or banded foliar spray fluazifop-butyl spray

SUNFLOWER SEEDS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Petersburg, Hale, TX, USA, 1989 (Large Grey Stripe)	EC 120 (P) + AG98	2 (14)	0.56 0.56	0.41 0.38	12-13 leaves; 19 June	ns	MAT	66	0.37	RR 91-010B 13-TX-89-851; [Alferness and Kleinschmidt, 1991, PP5/0233] (processing)
Mooreton, Richland, ND, USA, 1989 (Sigco 468)	EC 120 (P) + 1% COC	2 (14)	0.56 0.56	0.26 0.26	10-12 leaves; 22 June	ns	MAT	99	< 0.01	RR 91-010B; 33-ND-89-852; [Alferness and Kleinschmidt, 1991, PP5/0233] (processing)
Iracemapolis, SP, Brazil, 2007 (Iac Iarama)	EW 250 (P)	1	0.25	0.12	30 days after planting; 13 Oct	ns	MAT	67	≤ 0.02	T06030; no trial number [Tomaz, 2008, A12530B_10010]
Santa Mariana, PR, Brazil, 2007 (Catissol)	EW 250 (P)	1	0.25	0.12	30 days after planting; 14 Oct	ns	MAT	59	≤ 0.02	T06030; no trial number [Tomaz, 2008, A12530B_10010]
Araras, SP, Brazil, 2007 (Iac Iarama)	EW 250 (P)	1	0.25	0.12	30 days after planting; 19 Oct	ns	MAT	62	≤ 0.02	T06030; no trial number [Tomaz, 2008, A12530B_10010]
Morrinhos, GO, Brazil, 2007	EW 250 (P)	1	0.25	0.12	30 days after planting;	ns	MAT	66	≤ 0.02	T06030; no trial number [Tomaz, 2008,

SUNFLOWER SEEDS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(M 734)					26 Nov					A12530B_10010]
06922 Axien; Germany, 1993, (spring: Frankasol)	EC 125 (P)	1	0.38	ns	28 cm high; BBA 27; 21 May	SaL	93	108	< 0.05	RJ1656B; RS-9308-K1; [Robinson and Patel, 1994, PP5/0218]
idem	ME 125 (P)	1	0.38	ns	28 cm high; BBA 27; 21 May	SaL	93	108	< 0.05	idem
06922 Gehmen; Germany, 1993, (spring; Fleuron)	EC 125 (P)	1	0.38	ns	30 cm high; BBA 27; 25 May	SaL	93	107	< 0.05	RJ1656B; RS-9308-K2; [Robinson and Patel, 1994, PP5/0218]
idem	ME 125 (P)	1	0.38	ns	30 cm high; BBA 27; 25 May	SaL	93	107	< 0.05	idem
29378 Wittingen-Gannerwinkel; Germany, 1993, (spring: Frankasol)	EC 125 (P)	1	0.38	ns	18-20 cm height; BBA 25-27; 19 May	LSa	87-93	109	< 0.05 [RT]	RJ1656B; RS-9308-B1; [Robinson and Patel, 1994, PP5/0218]
idem	ME 125 (P)	1	0.38	ns	18-20 cm height; BBA 25-27; 19 May	LSa	87-93	109	< 0.05 [RT]	idem
85410 Haag-Inkofen; Germany, 1993, (spring: Sonsisa)	EC 125 (P)	1	0.38	ns	30-40 cm height; BBA 27-29; 26 May	SaL	93	117	< 0.05	RJ1656B; RS-9308-G1 [Robinson and Patel, 1994, no code]
idem	ME 125 (P)	1	0.38	ns	30-40 cm height; BBA 27-29; 26 May	SaL	93	117	< 0.05	idem
D-06922 Axien; Germany, 2000 (spring: Anika)	EC 125 (P)	1	0.38	0.19	BBCH 33; 20-30 cm high; 23 May	LSa	92	106	0.02	RJ3234B; DE16-00-S162; [Mason, 2001, PP5/0005]
D-55546 Volxheim; Germany, 2000 (spring: San Luca)	EC 125 (P)	1	0.38	0.19	BBCH 19; 30 cm high; 17 May	L	89	106	< 0.01	RJ3234B; DE17-00-S162; [Mason, 2001, PP5/0005]
Tours, N-France, 1984 (Veraflor)	EC 250 (P)+ 0.1% NIS	1	0.18	0.062	8 leaves; 15 May	ns	MAT	134	< 0.05 [SS]	H19/834-P; 211-84 [Culoto, 1984, PP5/0540]
idem	EC 250 (P)+	1	0.18	0.062	6 leaves; 15 May	ns	MAT	134	< 0.05 [SS]	H19/834-P; 235-84 [Culoto, 1984,

SUNFLOWER SEEDS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	0.1% NIS									PP5/0540]
Carpentras, S-France, 1984 (Minassol)	EC 250 (P)+ 0.1% NIS	1	0.18	0.062	6-8 leaves; 23 May	ns	MAT	111	< 0.05 [SS]	H19/834-P; 67-84 [Culoto, 1984, PP5/0540]
Villefranche sur Saone, S- France, 1984 (Rodeo)	EC 250 (P)+ 0.1% NIS	1	0.18	0.062	4 leaves; 11 May	ns	MAT	117	< 0.05 [SS]	H19/834-P; 110-84 [Culoto, 1984, PP5/0540]
idem	EC 250 (P)+ 0.2% NIS	1	0.38	0.12	6 leaves; 24 May	ns	MAT	104	< 0.05 [SS]	H19/834-P; 113-84 [Culoto, 1984, PP5/0540]
La Chapelle de Guinchay; Bourgogne N-France, 1999 (spring: Alinka)	EC 125 (P)	1	0.38	0.19	BBCH 15; 10-20 cm high; 1 June	SiSaC	MAT	104	<u>< 0.01</u>	RJ2940B; FR33-99-S751 [Ryan and Siourd, 2000, PP5/0542]
idem	EC 125 (P)	1	0.38	0.19	BBCH 15; 10-20 cm high; 1 June	SiSaC	MAT	104	< 0.01	RJ2940B; FR33-99-S751; [Ryan and Siourd, 2000, PP5/0542]
Chambray les Tours; Loire Valley N-France, 1999 (spring: Flores)	EC 125 (P)	1	0.38	0.12	BBCH 19; 30 cm high; 14 June	Si	MAT	113	<u>0.06</u>	RJ2940B; FR72-99-S751; [Ryan and Siourd, 2000, PP5/0542]
idem	EC 125 (P)	1	0.38	0.12	BBCH 19; 30 cm high; 14 June	Si	MAT	113	0.06	RJ2940B; FR72-99-S751; [Ryan and Siourd, 2000, PP5/0542]
Cordes Tolosannes, 82700 S-France, 2005 (spring: Orasol)	EC 125 (P)	1	0.37	0.12	BBCH 18; 25 May	SaCL	89	89	<u>< 0.05</u>	CEMR-2690; AF/8655/SY/1 [Bell, 2006, PP5/1488]
Sajópálfala, BAZ, Hungary, 2002 (spring: Arena)	EC 150 (P)	1	0.41	0.14	6-8 leaf – stage of sprouts 5 June	brown forest soil	MAT	96	0.11, 0.17, 0.17, mean 0.15 [a] [x0.85]	02SYNAA0505; SPC-6 HRSZ 047/1 [Suszter, 2003, PP5/1497]
Curiano, Siena, Italy, 1996 (spring; Odil Pioneer)	EC 125 (P)	1	0.38	0.13	BBCH 18-19; 24 May	LC	89	101	0.01 [CT] [Cntrl=0.01]	RJ2284B; IT10-96-R348; [Jones and Volpi, 1997, PP5/0221]
Pozzo Alto, Pesaro, Italy, 1996	EC 125 (P)	1	0.38	0.094	BBCH 18-19; 20 May	C	89	94	<u>0.04</u>	RJ2284B; IT10-96-R-349; [Jones and Volpi, 1997, PP5/0221]

SUNFLOWER SEEDS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(spring: Sarah)										
Piacenza, Emilia Romagna Italy, 1997 (spring: Vidoc)	EC 125 (P)	1	0.38	0.12	BBCH 65-69; 11 July	CL	CH	60	5.6	RJ2726B; AF/3702/ZE/3; [Mason and Hill; 1999, PP5/0534]
Ravenna; Emilia Romagna Italy, 1997 (spring Solbel)	EC 125 (P)	1	0.40	0.12	BBCH 60-65; 4 July	CL	CH	60	0.90	RJ2726B; AF/3702/ZE/4; [Mason and Hill; 1999, PP5/0534]
Conselice, 48021, Italy; 2005 (spring: Challenger)	EC 125 (P)	1	0.38	0.12	BBCH 18-19; 31 May	CL	89	83	< 0.05	CEMR-2690; AF/8655/SY/2 [Bell, 2006, PP5/1488]
El Cuervo, Sevilla, Spain, 1996 (spring: Florida 2000)	EC 125 (P)	1	0.38	0.13	BBCH 16-17; 18 Apr	SaL	95	110	< 0.01 [SS]	RJ2303B; AP/3223/ZE/1; [Miles *et al*., 1997, PP5/0207]
Villalaba del Alcor; Huelva Spain 1996 (spring: Doblon)	EC 125 (P)	1	0.38	0.13	BBCH 14 24 Apr	SiL	95	98	< 0.01 [SS]	RJ2303B; AP/3223/ZE/2; [Miles *et al*., 1997, PP5/0207]
El Cuervo, Sevilla, Spain, 1997 (spring: Tesoro)	EC 125 (P)	1	0.38	0.12	BBCH 79 11 June	CL	CH	60	2.2	RJ2726B; AF/3702/ZE/1; [Mason and Hill; 1999, PP5/0534]
Villalaba del Alcor; Huelva Spain 1997 (spring: Doblon)	EC 125 (P)	1	0.34	0.12	BBCH 80 9 June	SaCL	CH	60	4.0	RJ2726B; AF/3702/ZE/2; [Mason and Hill; 1999, PP5/0534]
Banded foliar spray over rows										
La Puebla del Rio, Sevilla, Spain, 2000 (spring: Cortes Hibrido 3L)	EC 125 (P)	1	0.34	0.15	BBCH 15; 20-30 cm high; 27 Mar	LSa	89	121	< 0.01	RJ3252B; ES50-00-S015; [Mason and Iniesta, 2001, PP5/1118]
Arahal, Sevilla, Spain, 2000 (spring: Sunko)	EC 125 (P)	1	0.37	0.15	BBCH 14; 20 cm high; 29 Mar	C	99	107	< 0.01	RJ3252B; ES51-00-S115. [Mason and Iniesta, 2001, PP5/1118]

BBA 26-27 (shortly before inflorescence heads became visible at the tip of the crop shoots)

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample

[RT] Seeds from trial B1 were heavily infested with Botrytis; sample considered not representative for MRL derivation

[SS] Sample size not stated (RJ2303B, H19/834-P, RJ2303B); sample considered not representative for MRL derivation

[a] Results are the mean of replicate field samples; the mean is taken for MRL derivation if according to cGAP

[x0.85] Residues need to be multiplied by a factor 0.85 to get the total fluazifop residues, expressed as fluazifop.

Additional trial information:

RR 91-010B, GLP. No unusual weather conditions. Hand-held CO2 backpack sprayer with boom. with spray volume 16-23 GPA = 150-215 L/ha. Sunflowers were picked randomly from each plot and mature marketable seeds (5 lbs = 2.3 kg as well as bulk samples > 9 kg for processing) were collected. Storage at -10°C or lower for maximum of 6 months Samples were analysed for total fluazifop using Samples were analysed for total fluazifop using **GC-MS method RR 89-073B with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (76-104% at 0.01–0.1 mg/kg) Control samples < 0.01 mg/kg.

T06030;, GLP study. No unusual weather conditions. CO2 pressurized backpack sprayer, spray volume 200 L/ha. Heads were collected from across the plots and seeds (1.0-1.6 kg) were removed manually. Storage at -20 °C or lower for maximum of 273 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method PLMV-027-C with a valid LOQ of 0.02 mg/kg**. Seed samples were not corrected for average concurrent method recoveries (94-98% at 0.02–0.2 mg/kg) Control samples < 0.02 mg/kg.

RJ1656B. GLP. Trial G1: Slight crop damage by thunder storm and hail the day after application; Trial K1 rainfall within 1 hr after application. Air pressurized knapsack plot sprayer with boom. Spray volume 200-400 L/ha. Seeds were sampled by hand from 30 heads (trial B1, K1, K2) or 15 heads (trial G1) and seeds were collected: 1 kg trial B1, 1.2-1.3 kg trial G1, 1.7-2.0 kg trial K1, >2 kg trial K2. Seeds from trial B1 were heavily infested with Botrytis. Storage at -18 °C or lower for maximum of 176 days (seed). Samples were analysed for total fluazifop using **NMR- method RAM 197/01 with a valid LOQ of 0.05 mg/kg**. Results were corrected for internal standard recoveries (average 88-107% at 1.0 mg/kg). Control samples < 0.05 mg/kg.

RJ3234B, GLP. No unusual weather conditions. Air pressurized knapsack plot sprayer with spray volume 200 L/ha. Seeds (1 kg Germany16; 2.0-2.1 kg Germany17) were collected from 20-30 plants/plot. Storage at -18 °C or lower for maximum of 251 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (89-91% at 0.01–0.05 mg/kg) Control samples < 0.01 mg/kg.

H19/834-P, nonGLP. No unusual weather conditions. Spray equipment not reported. Spray volume 300 L/ha. Mature sunflower heads were sampled by hand; seed sample size not stated. Storage at -18 °C or lower for maximum of 86 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/1 modification B with a valid LOQ of 0.05 mg/kg**. Result were not corrected for concurrent method recoveries (70% at 0.2 mg/kg) Control samples < 0.05 mg/kg.

RJ2940B, GLP. No unusual weather conditions. Hand held sprayer with spray volume 200-300 L/ha. Mature sunflower heads were sampled by hand and 1.0-1.1 kg seeds were collected. Storage at -18 °C or lower for maximum of 57 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (108-116% at 0.1-2.0 mg/kg) Control samples < 0.01 mg/kg.

CEMR-2690, GLP. No unusual weather conditions. AUK plot sprayer with spray volume 300 L/ha. Plants were cut by hand approximately 15 cm above ground and than threshed into seed (1 kg) using a combine harvester. Storage at -9 °C or lower for maximum of 219 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (75-78% at 0.05-0.5 mg/kg) Control samples < 0.05 mg/kg.

02SYNAA0505, GLP. No unusual weather conditions. Spray equipment not stated. Spray volume 300 L/ha. Sunflower heads were collected from 12 sampling positions and 2 kg seeds were collected. Storage at -18 °C or lower for maximum of 91 days. Samples were analysed for total fluazifop using **GC-NPD method 606/BAZ/1 with a valid LOQ of 0.05 mg/kg**. Results were not corrected for average concurrent method recoveries (95% at 0.05-0.25 mg/kg) Control samples < 0.05 mg/kg. Since residues are expressed as fluazifop-butyl, a conversion factor of 0.85 is needed to get total fluazifop resides as fluazifop [Syngenta, 2016, Response to questions 14].

RJ2284B, GLP. Unusual weather conditions did not affect crop condition. Broadcast foliar spray using a motor knapsack sprayer. Spray volume 300-400 L/ha. Mature sunflower heads (13-33 per plot) were sampled by hand. The seeds were extracted y rubbing two heads against each other and 1.1-1.2 kg seed was collected. Storage at -18 °C or lower for maximum of 105 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (84% at 0.1 mg/kg) Control samples < 0.01 mg/kg, except trial R348 where the control contained 0.01 mg/kg.

RJ2726B, GLP. Unusual weather conditions did not affect crop conditions. Precision boom sprayer with spray volume 274-324 L/ha. Mature sunflower heads were sampled by hand and threshed and seeds (1 kg) were collected. Storage at -18 °C or lower for maximum of 259 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (88-104% at 0.05-2.0 mg/kg) Control samples < 0.01 mg/kg.

RJ2303B, GLP. Unusual weather conditions did not affect crop condition. Boom sprayer with spray volume 300 L/ha. Mature sunflower heads (>12 per plot) were sampled by hand and threshed (seed sample size not stated). Storage at -18 °C or lower for maximum of 176 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM**

287/01 with a valid LOQ of 0.01 mg/kg. Results were not corrected for average concurrent method recoveries (71% at 0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ3552B GLP. No unusual weather conditions. **Banded spray** with spray volume 227-246 L/ha. Mature sunflower seeds were sampled by hand (1.2-1.6 kg). Storage at -18 °C or lower for maximum of 103 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (93-109% at 0.01–0.05 mg/kg) Control samples < 0.01 mg/kg.

Herbs

Parsley

One cGAP for parsley is available:

- cGAP from Belgium with 2 ×0.38 kg ai/ha with PHI 21 days

None of the available supervised trials on parsley were submitted: Baron, 1987, PR 2330, MRID 40566701; Baron, 1987, PR 2330 supplement, MRID 41016001; Baron, 1987, PR 2330 addendum, MRID 41016002. As the manufacturer did not seek to have maximum residue levels estimated on parsley, no further efforts were taken to retrieve these studies.

Legume animal feeds

Bean forage (green)

Two cGAPs for green Phaseolus beans are available:

- cGAP from the Netherlands is 1 × 0.38 kg ai/ha with a PHI of 60 days (green beans with pods)
- cGAP from Belgium with 1 ×0.38 kg ai/ha with a PHI of 28 days (green *Phaseolus* beans)

Three cGAPs for dry Vicia beans are available:

- cGAP from France with 1 × 0.38 kg ai/ha with a PHI of 56 days
- cGAP from the Netherlands with 1 × 0.38 kg ai/ha with a PHI of 90 days

Phaseolus bean forage is not grazed and is harvested at the same time as the green beans with or without pods as a by-product. Since the GAP does not have grazing restrictions, Vicia bean forage can be harvested at any time after treatment (PHI = 0 days). Vicia bean forage is generally ensiled at the stage when bottom bean pods start to turn black. Trials that could be matched to these cGAPs were summarized.

Table 238 lists trials conducted in Southern France (2006, 2008, 2009) and Spain (2008) on green Phaseolus beans. No trials were submitted for Vicia bean forage. A broadcast spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 238. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample size was less than the required 1 kg green forage.

Table 238 Supervised field trials on Phaseolus beans (green haulms), treated with a broadcast foliar fluazifop-butyl spray

PHASEOLUS BEAN FORAGE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
13210 St Remy; Provence; S-France, 2006;	EC 125 (P)	1	0.34	0.11	BBCH 23; 7 Jul	C	74	34	0.57	CEMR-3014; FR-HR-06-0225 [Bell, 2008, A1279B_10430]

PHASEOLUS BEAN FORAGE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Booster)										
47180 Meilhan S-France, 2006 (Denver)	EC 125 (P)	1	0.34	0.11	BBCH 14; 7 July	L	75	34	0.12	CEMR-3014; [Bell, 2008, A1279B_10430]
34590 Marsillargues; S-France, 2006; (Booster)	EC 125 (P)	1	0.34	0.10	BBCH 39; 9 June	CL	79	33	0.07	CEMR-3014; FR-HR-06-0227 [Bell, 2008, A1279B_10430]
82170 Grisolles; S-France, 2006 (Callisto)	EC 125 (P)	1	0.30	0.10	BBCH 29; 30 June	SiC	79	27	1.0	CEMR-3014; FR-HR-06-0228 [Bell, 2008, A12791B_10430]
Pexiora, Languedoc-Roussilon; S-France, 2008 (Linex)	EC 125 (P)	1	0.32	0.078	BBCH 71; 7 July	CL	71	28	2.3	TK009248-07-REG; S08-01602-01 [Marshall, 2009, A12791B_10788]
Pexiora, Aude, 11150, S-France, 2009; (Livex)	EC 125 (P)	1	0.31	0.052	BBCH 51; 7 July	CL	75	28	0.63 [SS]	CEMR-4384-REG; S09-00354-01 [Jutsum, 2011, A12791B_10829]
Montauban, 82000, S-France, 2009; (Rigoleto)	EC 125 (P)	1	0.32	0.078	BBCH 25, 20 July	SL	78-79	28	0.19	CEMR-4384-REG; S09-00354-02 [Jutsum, 2011, A12791B_10829]
Funes, 31360 Navarra; Spain; 2008; (Moncayo)	EC 125 (P)	1	0.31	0.078	BBCH 49; 27 Aug	SaCL	49	28	2.1	TK009248-07-REG; S08-01602-02 [Marshall, 2009, A12791B_10788]

BBCH 50-59 inflorescence emergence; BBCH 60-69 = flowering; BBCH 70-79 = development of pods.
[SS] Sample size less than the required 1 kg green forage: trial S09-00354-01

Additional trial information:
CEMR-3014. GLP. Fresh beans (Phaseolus spp). No unusual weather conditions. Overall foliar spray using a knapsack sprayer. Spray volume 300-316 L/ha. Samples were taken by hand. Sample sizes 1.0-1.7 kg haulms. Storage at -18 °C or lower for 469 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (94-98% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.
T009248-07-REG. GLP. Fresh beans (Phaseolus spp). No unusual weather conditions. Broadcast foliar spray using a boom sprayer. Spray volume 400 L/ha. Samples were taken by hand. Sample sizes 1.1-1.5 kg haulms. Storage at -12 °C or lower for 176-227 days (7.5 months). Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (91-99% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.
CEMR-4384-REG. GLP. Field beans (Phaseolus spp). No unusual weather conditions. Broadcast foliar spray using a boom sprayer. Spray volume 595 L/ha. Samples were taken by hand from 12-16 areas of the plot. Sample sizes 0.66-0.71 kg green haulms (trial 01) or 1.5-1.6 kg green haulms (trial 02). Storage at -12 °C or lower for 371 days. Samples were left at +1 °C for 2.5 hrs. Since the samples remained frozen, this is considered to have no impact on the study results. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg.** Samples were not corrected for individual concurrent external recovery (74-87% at 0.01-2.0 mg/kg). Control samples < 0.01 mg/kg.

Bean fodder (straw)

Three cGAPs for dry Phaseolus beans are available:

- cGAP from the USA with 2 × 0.42 kg ai/ha and PHI 60 days
- cGAP from the Netherlands with 1 × 0.38 kg ai/ha and PHI 90 days
- cGAP from Brazil with 1 × 0.25 kg ai/ha and PHI 60 days

Three cGAPs for dry Vicia beans are available:

- cGAP from France with 1 × 0.38 kg ai/ha with a PHI of 56 days
- cGAP from the Netherlands with 1 × 0.38 kg ai/ha with a PHI of 90 days

Bean fodder is harvested at the same time as the dry Phaseolus or Vicia beans. Trials that could be matched to these cGAPs were summarized.

Table 239 lists trials conducted in Southern France (2006), Italy (2006) and Spain (2006). A broadcast spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 239.

Table 239 Supervised field trials on Phaseolus beans (dry fodder, straw), treated with a broadcast foliar fluazifop-butyl spray

PHASEOLUS BEAN STRAW Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
40800 Duhort Bachen; S-France; 2006; (Angers)	EC 125 (P)	1	0.31	0.099	BBCH 39 31 July	L	89 dry	30	2.1	CEMR-3014; FR-HR-06-0229 [Bell, 2008, A12791B_10430]
Bolbaite, Canal Navarres, Valencia, Spain, 2009; (Cleo)	EC 125 (P)	1	0.35	0.078	BBCH 29; 24 Aug	CL	81 dry	38	0.63	CEMR-4384-REG; S09-00354-03; [Jutsum, 2011, A12791B_10829]
Xativa, La Costera, Valencia Spain, 2009; (Cardeno)	EC 125 (P)	1	0.34	0.097	BBCh 29 24 Aug	CL	82 dry	39	< 0.01	CEMR-4384-REG; S09-00354-04 [Jutsum, 2011, A12791B_10829]

BBCH 80-89 = ripening of pods and hardening seeds; BBCH 90-99 = senescence

Additional trial information:

CEMR-3014. GLP. Fresh beans (Phaseolus spp). No unusual weather conditions. Overall foliar spray using a knapsack sprayer. Spray volume 300-316 L/ha. Samples were taken by hand at BBCH 89 (i.e. dry straw). Sample sizes 1.0-1.7 kg haulms. Storage at -18 °C or lower for 469 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (94-98% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg.

CEMR-4384-REG. GLP. Field beans (Phaseolus spp). No unusual weather conditions. Broadcast foliar spray using a boom sprayer. Spray volume 595 L/ha. Samples were taken by hand from 12-16 areas of the plot at BBCH 81-82 (i.e. dry straw). Sample sizes, 0.64-0.68 kg dry haulms (trial 04). Storage at -12 °C or lower for 371 days. Samples were left at +1 °C for 2.5 hrs. Since the samples remained frozen, this is considered to have no impact on the study results. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recovery (74-87% at 0.01-2.0 mg/kg). Control samples < 0.01 mg/kg.

Table 240 Supervised field trials on Vicia beans (straw), treated with a broadcast foliar fluazifop-butyl spray

FAVA BEAN STRAW Location, Country; year; (variety)	For-mu-lation	no. of appl (inter-val)	kg ai/ha	kg ai/hL	GS; last treatment day	Soil type	GSH	DALT	Total fluazifop[1] (mg/kg)	Report no, Trial no. [ref]
82200, L'Homor de Cos, S-France, 2006 (fava bean: Irena, winter sown)	EC 125 (P)	1	0.31	0.078	BBCH 24; 14 Apr	CL	BBCH 89	90	0.38	CEMR-3008; AF/10374/SY/1; [Bell, 2007, PP5/1545]
82100, Escatalens, Castel sarrasin, S-France, 2006 (fava bean: Melodie; winter sown)	EC 125 (P)	1	0.32	0.078	BBCH 22-23; 20 Apr	SaC	BBCH 89	91	0.37	CEMR-3008; AF/10374/SY/2; [Bell, 2007, PP5/1545]
Pegola Italy, 2006; (fava bean: Polo; spring sown)	EC 125 (P)	1	0.31	0.078	BBCH 39; 18 Apr	SaCL	89	93	0.05	CEMR-3008; AF/10374/SY/5 [Bell, 2007, PP5/1545]
Cortes, Spain, 2006 (fava bean: Reina Blanca, autumn sown)	EC 125 (P)	1	0.31	0.078	BBCH 39; 7 Apr	CL	BBCH 89	90	3.1	CEMR-3008; AF/10374/SY/3 [Bell, 2007, PP5/1545]
Barboles, Spain, 2006 (fava bean: Reina Mora; autumn sown)	EC 125 (P)	1	0.31	0.078	BBCH 39; 4 Apr	SaCL	89	92	1.6	CEMR-3008; AF/10374/SY/4 [Bell, 2007, PP5/1545]

BBCH 80-89 = ripening of pods and hardening seeds; BBCH 90-99 = senescence

Additional trial information:

CEMR-3008-GLP. Dry fava beans. No unusual weather conditions. Application by plot sprayer. Spray volume 400 L/ha. Plants were harvested by hand and then threshed into seed and haulm by hand (Spain) or using a static combine (France) or samples were collected by plot combine (Italy). Haulm samples were at least 0.5 kg. Samples were stored at -9 °C or lower. Storage period is not indicated, but is maximally 489 days (harvest to final report). **HPLC-MS/MS method RAM 287/02**. **with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent method recoveries (105%). Control samples < 0.01 mg/kg.

Pea forage (green)

Three cGAPs for green peas are available:

- cGAP from Belgium with 1 ×0.38 kg ai/ha with a PHI of 28 days for peas with or without pods
- cGAP from France with 1 ×0.38 kg ai/ha with a PHI of 42 days for peas for ensilage

- cGAP from the Netherlands with 1 ×0.38 kg ai/ha with a PHI of 56 days for peas without pods

Four cGAPs for dry peas are available:

- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 56 days for dry peas
- cGAP from Belgium with 1 ×0.38 kg ai/ha before bloom for dry peas
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha with a PHI 90 days for pulses
- cGAP from the UK with 1 x0.19 kg ai/ha before flower buds are visible for dry peas

As the GAP does not have grazing restrictions, pea forage can be harvested at any time after treatment (PHI = 0 days). In practice forage peas are generally harvested at the flowering or flat pod stage (i.e., before commercial harvest of green peas with pods or green pea seeds). Trials that could be matched to these cGAPs were summarized.

Table 241 lists trials conducted in Canada (1990), the UK (1980, 1984, 2001, 2006, 2008, 2010), Denmark (1989), France (2006, 2012) and Spain (2003, 2006, 2012). A broadcast or banded foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 241. Results marked with "[QU]", "[SS]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample size was less than the required 2 kg green forage.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2.

Table 241 Supervised field trials on Pisum peas (green pea forage), treated with a broadcast or banded foliar fluazifop-butyl spray

GREEN PEA FORAGE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Dalmeny; Saskatchewan, Canada, 1990 (field peas)	WG 250 (rac)	1	0.12	ns	8-18 cm; vegetative; stage 104-105; 50% soil cover; 14 June	SaL	105; 107; 203 207	0 7 25 45	9.5 2.6 0.20 < 0.05	RJ1059B; CA-50-90-S912; [Jones, 1992, PP5/0405]
Location ns; UK, 1980 (peas, var ns)	EC 250 (rac)	1	1.0	ns	GS ns date ns	ns	ns	35 56 63	< 0.01 0.07 0.01 [QU] [LOQ=0.05]	RJ0226B summary [Atreya and Froggatt, 1981, PP9/0384]
Kettlestone; UK; 1984; (Sprite)	EC 125 (P)	1	0.19	0.085	flower buds visible; 100% crop cover; 2 June	LSa	CH green	42	0.79	M4008B; trial ns; [Harradine, 1985, PP5/0397]

GREEN PEA FORAGE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
idem	EC 125 (P)	1	0.38	0.17	flower buds visible; 100% crop cover; 2 June	LSa	CH green	42	1.8	idem
Thornhaugh, Peterborough UK; 1984; (Dark skinned perfection)	EC 125 (P)	1	0.19	0.085	no buds; 6 leaves; 100% crop cover; 4 June	L	CH green	46	0.11, 0.19 mean 0.15 [a] [CT] [cntrl=0.08]	M4008B; trial ns; [Harradine, 1985, PP5/0397]; and M4209B [Harradine, 1986, PP5/0398]
idem	EC 125 (P)	1	0.38	0.17	no buds; 6 leaves; 100% crop cover; 4 June	L	CH green	46	0.22, 0.33 mean 0.28 [a] [CT] [cntrl=0.08]	idem
Canwick, Lincolnshire; UK; 1984; (Scout)	EC 125 (P)	1	0.19	0.085	no buds; 5-6 leaves; 100% crop cover; 4 June	L	CH green	39	0.06 [CT] [cntrl=0.08]	M4008B; trial ns; [Harradine, 1985, PP5/0397]; and M4209B [Harradine, 1986, PP5/0398]
idem	EC 125 (P)	1	0.38	0.17	no buds; 5-6 leaves; 100% crop cover; 4 June	L	CH green	39	0.65 [cntrl=0.08]	idem
Coldham; (Tristar) UK; 1984;	EC 125 (P)	1	0.38	0.17	3 leaves; 100% crop cover; 4 May	pL	CH green	76	0.04 [CT] [cntrl=0.08]	M4008B; trial ns; [Harradine, 1985, PP5/0397]
Crowland; UK; 1984; (Sprite)	EC 125 (P)	1	0.38	0.17	(ns); 100% crop cover; 8 May	L	CH green	64	0.06 [CT] [cntrl=0.08]	M4008B; trial ns; [Harradine, 1985, PP5/0397]
Epworth; Lincolnshire; UK, 2001 (Waierex)	EC 125 (P)	1	0.38	0.13	BBCH 16/36; 22 June	SaL	CH green	39	0.92	RJ3336B; AF/6067/SY1 [Mason, 2002, PP5/1260]
Holbeach Hurn; UK, 2006; (Geisha)	EC 125 (P)	1	0.38	0.075	BBCH 16-17; 10 Jul	SaSiL	79	35	2.3	CEMR-3009; AF/10375/SY1 [Bell, 2008, PP5/1552]
Thwing; UK, 2006; (Ibis)	EC 125 (P)	1	0.38	0.075	BBCH 15-21; 5 Jul	SaL	77	35	0.68	CEMR-3009; AF/10375/SY2 [Bell, 2008, PP5/1552]
Luddington; Warwickshire; CV37 9SJ UK; 2010;	EC 125 (P)	1	0.37	0.083	BBCH 38-39; 3 June	SiL	79	34	2.2	CEMR-4658-REG; CEMS-4658-02; [Jutsum and

GREEN PEA FORAGE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Samish)										Allen, 2011, A1279B_10837]
Vedskoele, Koege; Denmark; 1989; (Bodil)	EW 125 (P) +adjL	1	0.38	ns	BJ4 = 4 leaves; 15% crop cover; 18 May	L	BJ9 green peas	62	0.93 [SS]	M5347B; DK10-89-5063; [Jones, 1991, PP5/0150]
Zuidlaren; Netherlands; 1985; (Finale)	EC 125 (P)	1	0.38	0.075	bud formation; 100% crop cover; 1 June	Sa	14d PCH (green pea forage)	56	0.83, 1.1, 1.2, 1.6 mean 1.2 [SS]	M4261B; 85-210; [Crook and Harradine, 1986, PP5/0161]
Kielwinde weer; Netherlands; 1985; (Finale)	EC 125 (P)	1	0.38	0.075	bud formation; 100% soil cover; 31 May	Sa	14d PCH (green pea forage)	56	0.58, 0.64, 0.90; mean 0.72 [SS]	M4261B; 85-211 ; [Crook and Harradine, 1986, PP5/0161]
28140 Loigny la Bataille; N-France; 2006; (Piano)	EC 125 (P)	1	0.38	0.075	BBCH 51; 12 May	CL	79	35	1.3	CEMR-3009; AF/10375/SY3 [Bell, 2008, PP5/1552]
71530 Viney le Grand; N-France; 2006; (Atlanta)	EC 125 (P)	1	0.39	0.075	BBCH 59; 1 Jun	SaL	79	35	1.8	CEMR-3009; AF/10375/SY4 [Bell, 2008, PP5/1552]
32490 Monferran Saves Gers; S-France; 2012; (Numerica)	EC 125 (P)	1	0.34	0.10	BBCH 37; 27 June	CL	79	35	0.06	CEMR-5453; SRFR12-008-37HR [Langridge, 2013, A12791B_11035]
31360 Funes; Spain; 2003 (Remu)	EC 125 (P)	1	0.31	0.10	BBCH 50; 25 Apr	SaCL	79	35	0.18 [SS]	03-7031; AF/7290/SY1 [Mason, 2004; PP5/1412]
50100; La Almunia de Dona Godina; Spain; 2003; (NZ)	EC 125 (P)	1	0.31	0.10	BBCH 50; 29 Apr	LSa	79	34	1.2 [SS]	03-7032; AF/7291/SY1 [Mason, 2004; PP5/1413]
22280; Gurrea de Gallego; Spain, 2006; (Valverde)	EC 125 (P)	1	0.32	0.062	BBCH 57; 5 May	CL	77	35	0.31	CEMR-3012; AF/10376/SY1 [Bell, 2007, PP5/1550]
22196; Huesca; Spain, 2006; (Meteor)	EC 125 (P)	1	0.32	0.062	BBCH 57; 5 May	CL	77	35	0.49	CEMR-3012; AF/10376/SY2 [Bell, 2007, PP5/1550]
Banded foliar spray application over rows										
03400 Villena;	EC 125	1	0.31	0.10	BBCH 36-37;	C	77	35	0.18	CEMR-5453; SRFR12-211-

GREEN PEA FORAGE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Spain; 2012; (Aston)	(P)				15 May					37HR; [Langridge, 2013, A12791B_11035]
02007 Albacete; Spain; 2012 (Resal)	EC 125 (P)	1	0.32	0.10	BBCH 36-37	LSa	77	34	1.0	CEMR-5453; SRES12-212-37HR; [Langridge, 2013, A12791B_11035]

GSH: 16dPCH = 16 days before commercial harvest; CHfc = commercial harvest for green freezing or canning peas;

Code 105-107 is vegetative; 203 is 10% flowering, 207 is podding, 303 is mature

Bjorkman Scale BJ 4 = 4 leaves; BJ9 just before maturity; BJ10 equivalent to BBCH 89 [Syngenta, 2016, Response to questions 15]

BBCH 50-59 = inflorescence emergence; BBCH 60-69 = flowering; BBCH 70-79 = development of pods

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[SS] Sample sizes less then the required 1 kg forage and thereby not representative for MRL derivation.

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample [a] Replicate samples were taken from the trial; the mean is used for MRL deriviation if according to cGAP.

Additional trial information:

RJ1059B. GLP. No unusual weather conditions. Spray equipment andspray volumes not stated. Sampling from 10 areas within a plot by cutting with scissors at ground level. Sample sizes forage/haulms (>1.0 kg). Storage at -18 °C or lower for a maximum of 412 days. Samples were analysed for total fluazifop using **NMR methods PPRAM 83 and RAM 197 with internal standard, each with a valid LOQ of 0.05 mg/kg**. Samples were corrected for external std recovery (91% at 0.1 mg/kg), uncorrected results are not reported. Control samples < 0.05 mg/kg.

RJ0226B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries not available. Control samples were 0.02 mg/kg.

M4008B Non-GLP. Vining peas. No unusual weather conditions. Application by plot sprayer; spray volume 220 L/ha. Samples were hand cut and were separated in haulms and peas using a plot viner (11-20 July). Samples were stored at -20 °C but no longer than 328 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recoveries (72%). Control samples were 0.08 mg/kg.

RJ3336B. GLP. Vining Peas. No unusual weather conditions. Overall foliar spray using a precision boom sprayer. Spray volume 297L/ha. Whole plants (24 plants) were sampled by hand and taken systematically from across the plots (31 July). Samples were threshed using a static viner to produce residual plant (> 1 kg). Storage at -18 °C for 170-174 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recovery (107-112% at 0.10-1.0 mg/kg). Control samples < 0.01 mg/kg.

M4261B Non-GLP. Peas. No unusual weather conditions. Application by propane sprayer; spray volume 500 L/ha. Four replicate samples per plot were harvested by hand (26 July DAT56 trial 85-210 and 85-211 (green pea forage). Sample sizes were not stated. Samples were stored at -20 °C (storage time not stated but less than 12 months). Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Internal standard recoveries (mean 75%. Control samples < 0.05 mg/kg.

CEMR-3009. GLP Fresh Peas. No unusual weather conditions. Foliar spray using a plot sprayer. Spray volume 502-515 L/ha. Fresh pea plants were sampled by hand) and threshed using a mini pea viner (UK) or by hand (N-France), resulting in remaining plants (1.0-1.2 kg). Storage at -15 °C for a maximum of 495 days. The temperature reached a maximum of -9 ºC for a peak of 3 days. This is considered to have no effect on the study, since the samples remained frozen at all times. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 modification B with a valid LOQ of 0.01 mg/kg.** Samples were not corrected for individual concurrent method recovery (98-100% at 0.01–0.10 mg/kg); Control samples < 0.01 mg/kg.

CEMR-4658. GLP Fresh peas. No unusual weather conditions. Broadcast foliar spray using a backpack sprayer. Spray volume 379-448 L/ha. Fresh pea plants (12 units) were taken by hand using a suitable distributive pattern and pods were removed to obtain haulms (> 1 kg). Storage at -18 °C for a maximum of 317 days. Samples were analysed for total

fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (90-93% at 0.01–0.5 mg/kg). Control samples < 0.01 mg/kg.

M5347B. GLP. Peas. No unusual weather conditions. Application equipment and spray volume not stated. Sample sizes: 0.4 kg green haulms (DAT62) Storage at -18 °C for a maximum of 619 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with internal std with a valid LOQ of 0.05 mg/kg**. Average concurrent external std recovery 80% at 0.5 mg/kg. Control samples were 0.10 mg/kg.

CEMR-5453. GLP Field peas. No unusual weather conditions. Broadcast foliar spray (S-France, Italy) or **banded foliar spray** (Spain) using a backpack sprayer. Spray volume 297-331 L/ha. Fresh pea plants (12 units) were taken by hand using a suitable distributive pattern. Seeds were removed to obtain > 1 kg pea podsandhaulms . Storage at -18 °C for a maximum of 190days. Freezer reached peak temperatures of -9, -10 ºC for 2 hours. This is considered to have no impact on the results, since samples remained frozen at all times. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (69-110% at 0.01-2.0 mg/kg). Control samples < 0.01 mg/kg.

03-7031. GLP. Fresh vining peas. No unusual weather conditions. Overal spray using a plot sprayer. Spray volume 295 L/ha. Fresh pea plants (24 units) were sampled by hand and seeds were removed to obtain > 0.5 kg haulms + empty pods. Storage at -18 °C for a maximum of 283 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (105-110% at 0.05-0.10 mg/kg for haulms). Control samples < 0.01 mg/kg.

03-7032. GLP. Fresh vining peas. No unusual weather conditions. Overal spray using a plot sprayer. Spray volume 299 L/ha. Fresh pea plants (24 units) were sampled by hand and seeds were removed to obtainn: > 0.5 kg haulms + empty pods. Storage at -18 °C for a maximum of 255 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (70-100% at 0.05-0.10 mg/kg for haulms. Control samples < 0.01 mg/kg.

CEMR-3012. GLP. Fresh Peas. No unusual weather conditions. Foliar spray using a plot sprayer. Spray volume 508-509 L/ha. Fresh pea plants were sampled by hand and seeds and pods were removed to obtain > 1 kg remaining plants (forage). Storage at -15 °C for a a maximum of 436 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (79-90% at 0.01–0.10 mg/kg); Control samples < 0.01 mg/kg.

Pea fodder (straw)

Four cGAPs for dry peas are available:

- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 56 days for dry peas
- cGAP from Belgium with 1 ×0.38 kg ai/ha before bloom for dry peas
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha with a PHI 90 days for pulses
- cGAP from the UK with 1 ×0.19 kg ai/ha before flower buds are visible for dry peas

Pea fodder is harvested at the same time as the dry pea seeds. Trials that could be matched to these cGAPs were summarized..

Table 242 lists trials conducted in Germany (2010), the UK (1983, 2001), Denmark (1989), the Netherlands (1983, 1985), Southern France (2001, 2006, 2008–2009), Italy (2001, 2006, 2012) and Spain (2006, 2008, 2009). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 242. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample size was not stated or less than the required 0.5 kg straw.

Table 242 Supervised field trials on Pisum peas (dry fodder, straw), treated with a broadcast foliar fluazifop-butyl spray

DRY PEA STRAW Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
68623 Lampertheim; Germany; 2010;	EC 125 (P)	1	0.35	0.093	BBCH 15-35; 30 July	Sa	87 dry	35	0.14 [b] [SS]	CEMR-4658-REG; CEMS-4658-01; [Jutsum and

DRY PEA STRAW Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Maxigold)										Allen, 2011, A12791B_10837]
Rauceby, Lincolnshire; UK, 1983 (Progretta)	EC 125 (P)	1	0.19	0.072	30-40 cm high; in bud; 21 June	Sa	NH dry	41	1.9	M3724B; 8R/83 [Harradine, 1984, PP9/0119]
idem	EC 250 (rac)	1	0.38	0.14	30-40 cm high; in bud; 21 June	Sa	NH dry	41	7.8	idem
idem	EC 125 (P)	1	0.19	0.072	13 cm high; no buds; 23 May	Sa	NH dry	77	0.42	M3724B; 30B/93/4/NE18 [Harradine, 1984, PP9/0119]
idem	EC 250 (rac)	1	0.38	0.14	13 cm high; no buds; 23 May	Sa	NH dry	77	0.20	idem
Lough borough; Leicestersh; UK, 2001; (Nitouche)	EC 125 (P)	1	0.38	0.13	BBCH 37-38; 18 June	SaCL	CH	65	1.2	RJ3266B; AF/5815/SY1 [McGill and Richards, 2002, PP5/1227]
Vedskoele, Koege; Denmark; 1989; (Bodil)	EW 125 (P) +adjL	1	0.38	ns	BJ4 = 4 leaves; 15% crop cover; 18 May	L	BJ10 dry	71	2.2	M5347B; DK10-89-5063; [Jones, 1991, PP5/0150]
Netherlands, 1983; (Finale)	EC 250 (rac)	1	0.25	0.050	20-22 cm high; 70% soil cover; 10 June	C	peas almost ripe (dry)	54	0.52, 0.64, 0.72, 0.78 mean 0.67	M3759B; ICI H 83/120 [Harradine, 1984, PP9/0117]
idem	EC 250 (rac)	1	0.38	0.075	20-22 cm high; 70% soil cover; 10 June	C	peas almost ripe (dry)	54	0.80, 0.84, 1.3, 1.5, mean 1.1	idem
idem	EC 125 (P)	1	0.12	0.025	20-22 cm high; 70% soil cover; 10 June	C	peas almost ripe (dry)	54	0.85, 0.97, 1.2, 1.3, mean 1.2	idem
idem	EC 125 (P)	1	0.19	0.038	20-22 cm high; 70% soil cover; 10 June	C	peas almost ripe (dry)	54	0.92, 1.0, 1.4, 1.6, mean 1.2	idem
Castel sarrasin; Tarn et Garonne; S-France, 2001 (Solara)	EC 125 (P)	1	0.31	0.11	BBCH 38; 30-35 cm tall; 2 May	SiC	CH	56	6.1	RJ3300B; AF/5835/SY/2; [McGill, 2002, PP5/1233]
82220 Vazerac; S-France; 2006 (Austin)	EC 125 (P)	1	0.32	0.063	BBCH 21-23; 13 Apr	CL	89	90	3.1	CEMR-3373; AF/10377/SY2 [Bell, 2008, PP5/1544]

DRY PEA STRAW Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Cordes Tolosane; Tarn et Garonne; S-France; 2008 (Panache)	EC 125 (P)	1	0.31	0.062	BBCH 39; 25 Apr	CL	89 89	74 90	NA 0.84	T009247-07-REG; S08-00863-01 [Jutsum; 2001; A12791B_10830]
Castelsarrasin, Tarn et Garonne; S-France; 2008-09; (field pea: Enduro)	EC 125 (P)	1	0.34	0.062	BBCH 36; 10 Apr 2009	CL	89 89	76 90	NA 0.56	CEMR-4385-REG; S09-00355-02 [Jutsum, 2011, A12791B_10831]
Ponticelli; Emilia Romagna; Italy, 2001 (Resal)	EC 125 (P)	1	0.31	0.11	BBCH 37; 10 cm tall; 14 May	CL	CH	52	0.75	RJ3300B; AF/5835/SY/3; [McGill, 2002, PP5/1233]
Crevalcore, Emilia Romagna; Italy; 2001 (Lambado)	EC 125 (P)	1	0.31	0.11	BBCH 35-37; 8-10 cm tall; 15 May	CL	CH	49	2.4	RJ3300B; AF/5835/SY/4 [McGill, 2002, PP5/1233]
40016 San Giorgio di Piano; Italy; 2006; (Dakota)	EC 125 (P)	1	0.30	0.062	BBCH 31-33; 24 Apr	SaCL	89	81	0.63	CEMR-3373; AF/10377/SY5 [Bell, 2008, PP5/1544]
40058; Pegola di Malalbergo; Italy; 2006 (Coral)	EC 125 (P)	1	0.30	0.062	BBCH 31-33; 24 Apr	SaCL	89	87	0.58	CEMR-3373; AF/10377/SY6 [Bell, 2008, PP5/1544]
29010; Cascina Marazzo Gragnago Trebbiense; Italy; 2012; (Heidi)	EC 125 (P)	1	0.32	0.10	BBCH 30-33; 4 May	SaL	89	38	0.03	CEMR-5453; SRIT12-1031-37HR; [Langridge, 2013, A12791B_11035]
50561 Bisimbre; Spain; 2006; (Ideal)	EC 125 (P)	1	0.31	0.062	BBCH 50; 7 Apr	SaL	89	63	4.5	CEMR-3373; AF/10377/SY3 [Bell, 2008, PP5/1544]
50490 Villareal de Huelva; Spain; 2006 (Gracia)	EC 125 (P)	1	0.31	0.063	BBCH 32; 27 Apr	CL	89	88	1.6	CEMR-3373; AF/10377/SY4 [Bell, 2008, PP5/1544]
Almansa; Albacete; Spain; 2008	EC 125 (P)	1	0.33	0.052	BBCH 39; 25 Apr	CL	89	90	5.0	T009247-07-REG; S08-00863-04; [Jutsum; 2001;

DRY PEA STRAW Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(Messire)										A12791B_10830]
Barrax; Albacete; 46160; Spain; 2009; (field pea: Messire)	EC 125 (P)	1	0.31	0.052	BBCH 34; 13 May	CL	97 97	56 92	NA 0.23	CEMR-4385-REG; S09-00355-03 [Jutsum, 2011, A12791B_10831]

GSH: 16dPCH = 16 days before commercial harvest; CHfc = commercial harvest for green freezing or canning peas; BJ = Bjorkman scale; Code 105-107 is vegetative; 203 is 10% flowering, 207 is podding, 303 is mature

[SS] Sample size less than the required 0.5 kg straw: CEMS-4658-01 (0.3–0.4 kg dry haulms)

[a] Replicate samples were taken from the trial; the mean is used for MRL deriviation if according to cGAP.

Additional trial information:

CEMR-4658. GLP Fresh peas. No unusual weather conditions. Broadcast foliar spray using a backpack sprayer. Spray volume 379-448 L/ha. Pea plants (12 units) were taken by hand at BBCH 89 (i.e. dry plants) using a suitable distributive pattern. Sample sizes haulms (> 1 kg). Storage at -18 °C for a maximum of 317 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (90-93% at 0.01–0.5 mg/kg). Control samples < 0.01 mg/kg.

M3724B Non-GLP. Dried peas (*Pisum sativum*). No unusual weather conditions. Spray using a hand-held sprayer. Spray volume 260 L/ha.. Samples of 0.5 kg whole dried plants were sampled by hand and seeds and pods were removed to obtain dry haulms. Samples were stored at -18°C or lower. Storage period was not stated bus was at maximum of 200 days (Harvest to final report date). Samples were analysed for total fluazifop using **HPLC-UV method PPRAM62/2 with a valid LOQ of 0.05 mg/kg**. Results were not corrected for average concurrent method recoveries (76-81% at 0. 1–0.5 mg/kg). Control samples < 0.04 mg/kg.

RJ3266B GLP. Dried peas (*Pisum sativum*). Unusual weather conditions had no effect on crop health. Overall foliar spray using a precision boom sprayer. Spray volume 292 L/ha. Whole plants (> 4.0 kg) were taken by hand systematically from all areas of at least 12 plants. Samples were separated into haulms (> 0.5 kg straw) by threshing using a small plot combine. Samples were stored at -18 °C for 149 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for individual concurrent method recoveries (85-91% at 0.2–0.5 mg/kg). Control samples < 0.01 mg/kg.

M5347B. GLP. Peas. No unusual weather conditions. Application equipment and spray volume not stated. Sample sizes: 0.3–0.4 kg dry haulms (DAT71). At DAT 71, shells were removed, crushed and cleaned by blowing in the wind. Storage at -18 °C for a maximum of 619 days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with internal std with a valid LOQ of 0.05 mg/kg.** Average concurrent external std recovery 80% at 0.5 mg/kg. Control samples 0.24 mg/kg (DAT 71). LOQ needs to be increased to 0.24/0.3=0.8 mg/kg. Since residues in the trials were higher, this has no impact on the study results.

M3759B Non-GLP. No unusual weather conditions. Application by propane sprayer; spray volume 500 L/ha. Four replicate samples per plot were harvested by hand or machine. Sample size 0.8 kg pea straw. Samples were stored at -20 °C (storage time not stated but less than 252 days). Samples were analysed for total fluazifop using **HPLC/UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Samples were corrected for internal standard recoveries (mean 80-84%). Control samples < 0.05 mg/kg.

RJ3300B GLP. No unusual weather conditions. Overall foliar spray using a precision boom sprayer, spray volume 300 L/ha. Whole plants were sampled by hand and were taken systematically from all areas of a least 12 points in the plot. Samples consisted of >0.5 kg straw (haulms). Storage time 145-153 days at -13 °C. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (104-108%). Control samples were < 0.01 mg/kg.

CEMR-3373 GLP. Dried peas (*Pisum sativum*). No unusual weather conditions. Foliar spray using a plot sprayer. Spray volume 480-507 L/ha.. Samples were taken by hand and were threshed into haulms (0.6-1.4 kg) using a combine harvester. Samples were stored at -9°C or lower for a maximum of 502 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02, modification A with a valid LOQ of 0.01 mg/kg**. Results were not corrected for individual concurrent method recoveries (95-99% at 0.05-0.5 mg/kg). Control samples < 0.01 mg/kg.

T009247-07-REG. GLP study. No unusual weather conditions. Broadcast foliar spray by boom sprayer, spray volume 490-630 L/ha. Pea straw (haulms, >0.5 kg were collected from at least 12 plants. Sampling by hand. Samples were separated into seed and haulm using a combine (France) or by hand (Spain). Storage at -12 ºC or lower for 145-161 days. Freezer temperature reached -3 ºC at 2 different days during storage (France). This is considered to have no impact on the results as the samples remained frozen. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM**

287/02 with a valid LOQ of 0.01 mg/kg. Results were not corrected for concurrent method recoveries (82-83% at 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg.

CEMS-4385. GLP study. No unusual weather conditions. Broadcast foliar spray by boom sprayer, spray volume 540-600 L/ha. Pea straw (haulms + empty pods, >0.5 kg) were collected from at least 12 plants. Sampling by hand using a suitably distributive pattern. Storage time 398 days at -7 °C with 2.5 hrs at +1 ºC (samples remained frozen at all times) This is considered to have no impact on the results. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Results were not corrected for concurrent method recoveries (68-89% at 0.01–0.1 mg/kg). Control samples were < 0.01 mg/kg.

CEMR-5453. GLP Dry Fresh peas. No unusual weather conditions. Broadcast foliar spray (Italy) using a backpack sprayer. Spray volume 297-331 L/ha. Pea plants (12 units) were taken by hand at BBCH 89 (i.e. dry plants) using a suitable distributive pattern. Seeds were removed to obtain > 1 kg pea podsandhaulms , Storage at -18 °C for a maximum of 190days. Freezer reached peak temperatures of -9, -10 ºC for 2 hours. This is considered to have no impact on the results, since samples remained frozen at all times. Samples were analysed for total fluazifop using **HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent method recovery (69-110% at 0.01-2.0 mg/kg). Control samples < 0.01 mg/kg.

Soya bean forage

Three cGAPs for dry soya beans are available:

- cGAP from Brazil with 1 ×0.25 kg ai/ha with a PHI of 60 days
- cGAP from the USA with 1 ×0.42 kg ai/ha pre-blooming (before BBCH 60, up to V5) plus 1 × 0.10 kg ai/ha at blooming or later (from BBCH 60 or R1) with harvest at a PHI of 60 days
- cGAP from France with 1 ×0.19 kg ai/ha with a PHI of 90 days

Soya bean forage can be grazed. As the GAP does not have grazing restrictions, soya bean forage can be harvested at any time after treatment (PHI = 0 days). Trials that could be matched to these cGAPs were summarized.

Table 243 lists trials conducted in the Canada (2006, 2007) and South Africa (1991). A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 243. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample size was less than the required 1 kg forage.

Table 243 Supervised residue field trials on soya (green forage), treated with a broadcast foliar fluazifop-butyl spray

SOYA FORAGE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Valens, ON, Canada, 2006; (QAC Raptor)	EC 125 (P) + Turbo charge	1	0.25, 0.26 [a]	0.13	BBCH 10-12; 20 June	L	BBCH 62	24	1.3, 1.4 [a]	CER 02401/06; T112; [Sagan, 2010, A12791B_50006]
idem	EC 125 (P) + Turbo charge	1	0.25, 0.26 [a]	0.13	BBCH 10-12; 20 June	L	BBCH 62	24	1.4, 1.4 [a]	idem
idem	EC 125 (P) + Turbo charge	1	0.25, 0.26 [a]	0.13	BBCH 10-12; 20 June	L	BBCH 62	24	0.79, 0.96 [a]	idem
idem	EC	1	0.26,	0.13	BBCH 10-	L	BBCH	24	0.94, 1.2	idem

SOYA FORAGE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	125 (P)		0.26 [a]		12; 20 June		62		[a]	
Thorndale, ON, Canada, 2006; (PS56RR)	EC 125 (P) + Turbo charge	1	0.24, 0.25 [a]	0.12	2nd trifoliate; 11 July	SiL	R2	21	0.40, 0.53 [a]	CER 02401/06; T113 [Sagan, 2010, A12791B_50006]
idem	EC 125 (P) + Turbo charge	1	0.24, 0.25 [a]	0.12	2nd trifoliate; 11 July	SiL	R2	21	0.42, 0.51 [a]	idem
idem	EC 125 (P) + Turbo charge	1	0.24, 0.25 [a]	0.12	2nd trifoliate; 11 July	SiL	R2	21	0.14, 0.17 [a]	idem
idem	EC 125 (P)	1	0.24, 0.24 [a]	0.12	2nd trifoliate; 11 July	SiL	R2	21	0.20, 0.24 [a]	idem
St-Pie-de Bagot, PQ, Canada, 2006; (DKB 06-52)	EC 125 (P) + Turbo charge	1	0.24, 0.24 [a]	0.12	BBCH 12-13; 29 June	LSa	BBCH 59-60	20	1.2, 1.3 [a]	CER 02401/06; T114 [Sagan, 2010, A12791B_50006]
idem	EC 125 (P) + Turbo charge	1	0.24, 0.25 [a]	0.12	BBCH 12-13; 29 June	LSa	BBCH 59-60	20	1.8, 1.9 [a]	idem
idem	EC 125 (P) + Turbo charge	1	0.24, 0.24 [a]	0.12	BBCH 12-13; 29 June	LSa	BBCH 59-60	20	1.2, 1.0 [a]	idem
idem	EC 125 (P)	1	0.24, 0.24 [a]	0.12	BBCH 12-13; 29 June	LSa	BBCH 59-60	20	0.68, 0.94 [a]	idem
Branchton, ON; Canada, 2007; (RC18 Mirra)	EC 125 (P)	1	0.26	0.17	BBCH 13-14; 10-15 cm high; 6 June	SiL	BBCH 68-69	50	1.1, 1.1; mean 1.1 35% dm	CER 02605/07; T229; [Sagan, 2008, A12791B_50003]
idem	EC 125 (P)	1	0.27	0.18	BBCH 13-14; 10-15 cm high; 6 June	SiL	BBCH 68-69	50	1.5, 1.6; mean 1.6 35% dm	idem
idem	EC 125 (P)	1	0.25	0.17	BBCH 13-14; 7-10 cm high; 6 June	SiL	BBCH 67	50	0.079, 0.10 mean 0.090 35% dm	CER 02605/07; T230; [Sagan, 2008, A12791B_50003]
idem	EC 125 (P)	1	0.26	0.17	BBCH 13-14; 7-10 cm high; 6 June	SiL	BBCH 67	50	0.24, 0.28 mean 0.26 35% dm	idem

SOYA FORAGE Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
									[SS]	
St Marc sur Richelieu, QC, Canada, 2007; (PS 46 RR)	EC 125 (P)	1	0.27	0.18	BBCH 13-14; 7-10 cm high; 15 June	CL	BBCH 67-69	40	0.12, 0.15 mean 0.14 35% dm	CER 02605/07; T231; [Sagan, 2008, A12791B_50003]
idem	EC 125 (P)	1	0.27	0.18	BBCH 10-11; 5-10 cm high; 15 June	CL	BBCH 67-69	40	0.16, 0.26 mean <u>0.21</u> 35% dm	idem
Plaston, White River, South Africa, 1991 (Pioneer 855)	EC 125 (P)	1	0.25	0.099	3.8 = 6-8 trifoliate; 35 cm high; 35% soil cover; 22 Jan	25% clay	3.8 3.8 3.8 3.8 ns	3 7 14 28 56	<u>4.0</u> 3.7 1.9 0.87 0.28	TMJ3065B; ZA13-91-H118 [Johnson *et al*., 1993, PP5/1031]
idem	EC 125 (P)	1	0.50	0.20	3.8 = 6-8 trifoliate; 35 cm high; 35% soil cover; 22 Jan	25% clay	3.8 3.8 3.8 3.8 ns	3 7 14 28 56	9.0 7.0 4.0 2.0 0.44	idem
idem	EC 125 (P)	1	1.0	0.40	3.8 = 6-8 trifoliate; 35 cm high; 35% soil cover; 22 Jan	25% clay	3.8 3.8 3.8 3.8 ns	3 7 14 28 56	18 13 9.6 3.3 0.48	idem

BBCH 50-59 or 500-509 = inflorescence emergence; BBCH 60-69 or 600-609 = flowering; BBCH 70-79 or 700-709 = development of pods and seeds; Code 3.8 in the South African trials = 6-8 trifoliate stage

[SS] Sample size less than the required 1 kg green forage.

[a] Two replicate plots. The two dose rates reflect the doses used in each of these plots. The highest value is taken for MRL derivation if according to cGAP.

Additional trial information:

CER2401-06 GLP. Weather conditions did not have an effect on the results. Broadcast foliar spray, spray volume 200 L/ha. Soybean plants were collected from 12 separate areas in the plot. Sample sizes were >1 kg forage. Samples were kept below -10 °C for a maximum of 444 days. Samples were analysed for total fluazifop using **HPLC-MS/MS Method CER 2605 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recoveries (78-91%) Control samples were < 0.01 mg/kg.

CER2605-07 GLP. Weather conditions did not have an effect on the results. Boom sprayer with spray volume 150 L/ha. Soybean plants were collected from 12 separate areas in the plot. Sample sizes were >1 kg forage, except 0.90-0.97 kg for T230 (treatment B) Samples were kept below -10 °C for a maximum of 236 days. Samples were analysed for total fluazifop using **HPLC-MS/MS Method CER2605 with a valid LOQ of 0.01 mg/kg**.Samples were not corrected for concurrent recoveries (102-110%) Control samples were < 0.01 mg/kg.

TMJ3065B, GLP. Weather conditions did not have an effect on the results. Constant pressure sprayer with boom; spray volume 283 L/ha. Sampling conditions not indicated Sample sizes were >1 kg forage. Samples were kept below -18 °C for a maximum of 645 days. Samples were analysed for total fluazifop using **NMR Method 197 with internal standard with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent recoveries (107-124% at 0.05-0.5 mg/kg) . Control sample results were not stated.

Soya bean hay

Three cGAPs for dry soya beans are available:

- cGAP from Brazil with 1 ×0.25 kg ai/ha with a PHI of 60 days
- cGAP from the USA with 1 ×0.42 kg ai/ha pre-blooming (before BBCH 60, up to V5) plus 1 × 0.10 kg ai/ha at blooming or later (from BBCH 60 or R1) with harvest at a PHI of 60 days
- cGAP from France with 1 ×0.19 kg ai/ha with a PHI of 90 days

As the GAP does not have grazing restrictions, soya bean forage for hay can be harvested at any time after treatment (PHI = 0 days). This option is often considered either when forage is short or when the soya bean crop is damaged prior to harvest as a grain crop (for example following hail damage or an early frost). Trials that could be matched to these cGAPs were summarized.

Table 244 lists trials conducted in Canada (2006, 2007). A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 244. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample size was less than the required 0.5 kg soya bean hay.

Table 244 Supervised residue field trials on soya (dried forage, hay), treated with a broadcast foliar fluazifop-butyl spray

SOYA HAY Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Valens, ON, Canada, 2006; (QAC Raptor)	EC 125 (P) + adj 1	1	0.25; 0.26 [a]	0.13	BBCH 10-12; 20 June	L	BBCH 67-69	43 ad 10-12	0.23, 0.28	CER 02401/06; T112 [Sagan, 2010, A12791B_50006]
idem	EC 125 (P) + adj2	1	0.25; 0.26 [a]	0.13	BBCH 10-12; 20 June	L	BBCH 67-69	43 ad 10-12	0.21, 0.24	idem
idem	EC 125 (P) + adj 2 + TM	1	0.25; 0.26 [a]	0.13	BBCH 10-12; 20 June	L	BBCH 67-69	43 ad 10-12	0.045, 0.049	idem
idem	EC 125 (P) + TM	1	0.26	0.13	BBCH 10-12; 20 June	L	BBCH 67-69	43 ad 10-12	0.11, 0.14	idem
Thorndale, ON, Canada, 2006; (PS56RR)	EC 125 (P) + adj 1	1	0.24; 0.25 [a]	0.12	2nd trifoliate; 11 July	SiL	Stage 7 code 23	35 ad 10-12	0.42, 0.56	CER 02401/06; T113 [Sagan, 2010, A12791B_50006]
idem	EC 125 (P) + adj2	1	0.24; 0.25 [a]	0.12	2nd trifoliate; 11 July	SiL	Stage 7 code 23	35 ad 10-12	0.57, 0.58	idem
idem	EC 125 (P) + adj 2 + TM	1	0.24; 0.25 [a]	0.12	2nd trifoliate; 11 July	SiL	Stage 7 code 23	35 ad 10-12	0.25, 0.55 [SS]	idem
idem	EC 125 (P) + TM	1	0.24	0.12	2nd trifoliate; 11 July	SiL	Stage 7 code 23	35 ad 10-12	0.19, 0.21	idem
St-Pie-de Bagot, PQ, Canada, 2006; (DKB 06-	EC 125 (P) + adj 1	1	0.24	0.12	BBCH 12-13; 29 June	LSa	BBCH 67-70	36 ad 10-12	0.23, 0.26	CER 02401/06; T114 [Sagan, 2010, A12791B_50006]

SOYA HAY Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
52)										
idem	EC 125 (P) + adj2	1	0.24; 0.25 [a]	0.12	BBCH 12-13; 29 June	LSa	BBCH 67-70	36 ad 10-12	0.25, 0.27	idem
idem	EC 125 (P) + adj 2 + TM	1	0.24	0.12	BBCH 12-13; 29 June	LSa	BBCH 67-70	36 ad 10-12	0.14, 0.15	idem
idem	EC 125 (P) + TM	1	0.24	0.12	BBCH 12-13; 29 June	LSa	BBCH 67-70	36 ad 10-12	0.11, 0.11	idem
Branchton, ON; Canada, 2007; (RC18 Mirra)	EC 125 (P) Venture L	1	0.26	0.17	BBCH 13-14; 10-15 cm high; 6 June	SiL	BBCH 74-75	69 ad 16	0.52, 0.68; mean 0.60 82% dm	CER 02605/07; T229; [Sagan, 2008, A12791B_50003]
idem	EC 125 (P)	1	0.27	0.18	BBCH 13-14; 10-15 cm high; 6 June	SiL	BBCH 74-75	69 ad 16	1.3, 2.1; mean 1.7 82% dm	idem
idem	EC 125 (P)	1	0.073	0.049	BBCH 68-69; 55-60 cm high; 27 Jul	SiL	BBCH 74-75	18 ad 16	3.5, 3.7; mean 3.6 82% dm	idem
idem	EC 125 (P)	1	0.25	0.17	BBCH 13-14; 7-10 cm high; 6 June	SiL	BBCH 74-75	69 ad 16	0.048, 0.052 mean 0.050 84% dm	CER 02605/07; T230 [Sagan, 2008, A12791B_50003]
idem	EC 125 (P)	1	0.26	0.17	BBCH 13-14; 7-10 cm high; 6 June	SiL	BBCH 74-75	69 ad 16	0.044, 0.16 mean 0.10 84% dm	idem
idem	EC 125 (P)	1	0.078	0.052	BBCH 67-68; 65-70 cm high; 27 Jul	SiL	BBCH 74-75	18 ad 16	3.2, 4.4 mean 3.8 84% dm	idem
St Marc sur Richelieu, QC, Canada, 2007; (PS 46 RR)	EC 125 (P)	1	0.27	0.18	BBCH 13-14; 7-10 cm high; 15 June	CL	BBCH 74-75	54 ad 4	0.045, 0.065 mean 0.055 85% dm	CER 02605/07; [Sagan, 2008, A12791B_50003]
idem	EC 125 (P)	1	0.27	0.18	BBCH 10-11; 5-10 cm high; 15 June	CL	BBCH 74-75	54 ad 4	0.072, 0.073 mean 0.072 85% dm	idem
idem	EC 125 (P)	1	0.080	0.053	BBCH 67-69; 40-50 cm high; 23 July	CL	BBCH 74-75	16 ad 4	1.9, 2.2; mean 2.0 85% dm	idem

ad = air dried for the specified number of days

[SS] Sample size less than the required 0.5 kg soya hay

[a] Two application rates, corresponding to two replicate plots

Additional trial inforation:

CER2401-06 GLP. Weather conditions did not have an effect on the results. Broadcast foliar spray, spray volume 200 L/ha. Soybean plants were collected from 12 separate areas in the plot. Samples for hay were dried in mesh bags (T112 and T113) or on dry racks (T114) for 10-12 days. Sample sizes were >0.5 kg hay, except 0.44-0.48 kg in samples T113-15 and T113-16 (treatment C). Samples were kept below -10 °C for a maximum of 444 days. Samples were analysed for total fluazifop using **HPLC-MS/MS Method CER 2605 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for concurrent recoveries (78-114%). Control samples were < 0.01 mg/kg.

CER2605-07 GLP. Weather conditions did not have an effect on the results. Boom sprayer with spray volume 150 L/ha. Soybean plants were collected from 12 separate areas in the plot. Samples for hay were dried outside until moisture content was 15-18%. Sample sizes were >0.5 kg hay.. Samples were kept below -10 °C for a maximum of 236 days. Samples were analysed for total fluazifop using **HPLC-MS/MS Method CER2605 with a valid LOQ of 0.01 mg/kg**.Samples were not corrected for concurrent recoveries (86-103% hay) Control samples were < 0.01 mg/kg.

Soya bean fodder (straw)

Three cGAPs for dry soya beans are available:

- cGAP from Brazil with 1 ×0.25 kg ai/ha with a PHI of 60 days
- cGAP from the USA with 1 ×0.42 kg ai/ha pre-blooming (before BBCH 60, up to V5) plus 1 × 0.10 kg ai/ha at blooming or later (from BBCH 60 or R1) with harvest at a PHI of 60 days
- cGAP from France with 1 ×0.19 kg ai/ha with a PHI of 90 days

Soya bean fodder is harvested at the same time as the dry bean seeds. Trials that could be matched to these cGAPs were summarized.

Table 245 lists trials conducted in the South Africa (1982, 1991). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 245.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

Table 245 Supervised residue field trials on soya (dry fodder, straw), treated with a broadcast foliar fluazifop-butyl spray

SOYA STRAW Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Thabazimbi; Transvaal; South Africa, 1982	EC 250 (rac)	1	1.0	ns	3 Febr	ns	dry	69	1.3 [QU]	PP009B176 CR/SB/3 [Atreya and Collis, 1983, PP9/0606]
idem	EC 250 (rac)	1	2.0	ns	3 Febr	ns	dry	69	1.5 [QU]	idem
Plaston, White River, South Africa, 1991 (Pioneer 855)	EC 125 (P)	1	0.25	0.099	6-8 trifoliate; 35 cm high; code 3.8; 35% soil cover; 22 Jan	25% clay	dry	118	0.23	TMJ3065B; ZA13-91-H118 [Johnson *et al*., 1993, PP5/1031]

SOYA STRAW Location, Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
idem	EC 125 (P)	1	0.50	0.20	6-8 trifoliate; 35 cm high; code 3.8; 35% soil cover; 22 Jan	25% clay	dry	118	0.23	idem
idem	EC 125 (P)	1	1.0	0.40	6-8 trifoliate; 35 cm high; code 3.8; 35% soil cover; 22 Jan	25% clay	dry	118	0.22	idem

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

Additional trial information:

PP009B176, non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Samples were indicated as "plants". It is assumed they were harvested dry. Storage conditions were not stated. Samples were analysed for total fluazifop using **HPLC-UV Method PPRAM 62/1 with a valid LOQ of 0.05 mg/kg**. Concurrent recoveries were not stated. Control samples contained 0.14 mg/kg fluazifop. LOQ needs to be increased to 0.14/0.3=0.5 mg/kg. Since residue levels were higher, this is considered to have no effect on the results.

TMJ3065B. non-GLP. No unusual weather conditions. Constant pressure CO2 driven sprayer with boom. Spray volumes 253 L/ha. Sample sizes whole plants (> 1.0 kg). Storage frozen at -18 °C or lower for a maximum of 530 days (harvest to last analysis date). Samples were analysed for total fluazifop using **NMR method RAM 197 with internal standard with a valid LOQ of 0.05 mg/kg.** Samples were not corrected for recovery (75-92% at 0.1–0.5 mg/kg). Control samples were not indicated.

Clover, trefoil and alfalfa

One cGAP for alfalfa (lucerne) and clover is available:

- cGAP from Belgium with 1 ×0.38 kg ai/ha with a PHI of 28 days.

Trials that could be matched to this cGAP were summarized.

Table 246 lists trials conducted in the South Africa (1990) on medic pasture. Medic pastures are the *Medicago* species, commonly known as medick of burclover. This family covers over 87 species. *Medicago sativa* (alfalfa) is the best known member, which grows to 1 meter height. Most members are low, creeping herbs, resembling clover, but with burs (seed or dry fruit). The creeping members are often used as forage crops (e.g. *M. lupulina* and *M. trunculata*). Only alfalfa (*M sativa*) is in the Codex Classification.

A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 246. Results marked with "[SS]" or "[CT]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample sizes were not reported or less then 1 kg.

[CT] indicates the analytical result is considered not reliable because of the high level of residues in the control sample (>25% of residue value).

Additional trials from Canada (1980) were available on clover and trefoil with 1 x 0.50 or 1.0 kg ai/ha with harvest at 77 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. These trials were not summarized, because they would not assist in MRL setting.

Table 246 Supervised residue field trials on alfalfa (forage), treated with a broadcast foliar fluazifop-butyl spray

ALFALFA FORAGE Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Al Kharj, Saudi Arabia 1991/1992 (Alfalfa: Forager)	EC 125 (P)	1	0.25	0.10	3-4 trifoliate (5 cm), 70% crop cover 19 Dec 1991	Sa	5-7; 7-12 cm	28 56	5.1 0.25 [CT] [Cntr= 0.10]	RJ1338B; SA06-92-H026 [Aver *et al*, 1993, no code]
idem	EC 125 (P)	1	0.50	0.20	3-4 trifoliate (5 cm), 70% crop cover 19 Dec 1991	Sa	ns	28 56	5.2 0.35	idem
Al Kharj, Saudi Arabia 1991/1992 (Alfalfa: Green Devil)	EC 125 (P)	1	0.25	0.10	6 trifoliate (10 cm), 90% crop cover 19 Dec 1991	Sa	10-15; 15-20 cm	28 56	5.3 1.1	RJ1338B; SA06-92-H025 [Aver *et al*, 1993, no code]
idem	EC 125 (P)	1	0.50	0.20	6 trifoliate (10 cm), 90% crop cover 19 Dec 1991	Sa	idem	28 56	9.7 2.7	idem
Buraidah, Saudi Arabia 1991/1992 (Alfalfa: Shiver)	EC 125 (P)	1	0.25	0.10	well established (10-15 cm), 100% crop cover 22 Jan 1992	Sa	15-25; 25-40 cm	28 57	3.7 0.73	RJ1338B; SA06-92-H027 [Aver *et al*, 1993, no code]
idem	EC 125 (P)	1	0.50	0.20	well established (10-15 cm), 100% crop cover 22 Jan 1992	Sa	idem	28 57	6.8 1.4	idem
Slaapkraal, CERES, South Africa, 1990 (Medic Pasture; Paragio, SA Standard)	12.5% EC (P)	1	0.25	ns	Crop height 5 cm, 70% crop cover, 5% weed cover 1 October	CL	ns	0 2 4 8 16 32 64	25 9.2 7.6 7.5 5.9 4.0 3.5	RJ1068B; ZA18-90-H016, plot 1 [Bolygo, 1992, PP5/0521]
idem	12.5% EC (P)	1	0.50	ns	Crop height 5 cm, 70% crop cover, 5% weed cover 1 October	CL	ns	0 2 4 8 16 32 64	31 20 22 23 11 6.0 8.3	idem, plot 2

[cntrl] Residue level found in the untreated control sample

[CT] Residue value of the treated samples not considered for MRL derivation because of high residue levels in the untreated control sample.

Additional trial information:

RJ1338B. GLP. Unusual cold winter, which may have inhibited the growth. Plot size 21 m^2. Handheld sprayer. Spray volume 250 L/ha. Samples were cut off at ground level (minimum 1 kg). After mixing the samples, the weights were reduced to 0.6-0.7 kg laboratory samples. Storage within 6 hours at -15 °C. Duration not stated, but within 6 months. Samples were analysed for total fluazifop using **NMR method ARAM 197 with a valid LOQ of 0.05 mg/kg.** Mean recoveries reported to be 84-121% for fluazifop fortified samples at 0.05-0.1 mg/kg, respectively. Control samples were < 0.05 mg/kg, except in trial SA06-92-H026 (0.10 mg/kg) at 28 DAT.. Since residue levels are higher this is considered to have no impact on the residue levels reported.

RJ1068B. GLP. No unusual weather conditions. Plot size 60 m2. Foliar spray. Spray volume not reported. Samples were cut off at ground level (2 kg). Storage within 2 hours at -20 °C for maximum of 155 days to sample preparation and 364 days to analysis. Samples were analysed for total fluazifop using NMR method ARAM 197 with a valid LOQ of 0.05 mg/kg. Mean recoveries for internal standard report to be 83%, SD 10% (n = 22) and for Fluazifop fortified samples 88%, SD 4% (n = 4) at 0.5 mg/kg. Control samples were < 0.05 mg/kg, except DAT 0 (2.2 mg/kg), DAT 2 (0.27 mg/kg), DAT 4 (0.4 mg/kg) and DAT 64 (0.24 mg/kg). LOQ needs to be increased to 2.2/0.3=8 mg/kg (DAT0), 0.27/0.3=0.9 mg/kg (DAT 2), 0.40/0.3=1.5 mg/kg (DAT4) and 0.24/0.3=0.80 mg/kg. Since residue levels are higher this is considered to have no impact on the residue levels reported.

Miscellaneous fodder and forage crops

Sugar beet and fodder beet tops

Two cGAPs for sugar beets and fodderbeets are available:

- cGAP from the USA with 2 × 0.42 kg ai/ha and a PHI of 90 days
- cGAP from the Netherlands, Belgium and the UK with 1 ×0.38 kg ai/ha and a PHI of 56 days

Tops are harvested at the same time as the roots. Trials that could be matched to these cGAPs were summarized.

Table 247 lists trials conducted in USA (2000), Germany (1981, 1983, 1992, 2002), the UK (1981), Denmark (1988), Sweden (1981), Southern France (2004), Italy (1998, 2004) and Spain (1998, 1999). A broadcast or banded foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 247. Results marked with "[QU]", or "[SS]" are not selected for derivation of the MRL, if according to cGAP. Results marked with "LOQ = nn", need to be increased to the LOQ indicated.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample sizes were not reported or less then 12 plants (i.e. at least 4 kg).

[LOQ = nn] indicates that the results needs to be increased to the valid LOQ of 0.05 mg/kg for HPLC-UV method PPRAM 62 or 62/2

[Atreya and Froggatt, 1981, PP9/0384, report RJ0226B] summarized the results of several residue trials carried out with fluazifop-butyl in 1979 and 1980 in the UK (1979: 1 ×1.0 kg ai/ha, PHI 0–21 days or 115–119 and 134–137 days for roots[FB+F]; 1980: 1 ×1.0–2.0–4.0 kg ai/ha, PHI 77–118 days, roots and tops[FP+F] and two decline studies 1 ×1.0 kg ai/ha, PHI 0, 21/20 , 42, 75/81, 106/112 days[FP+F]). Additional trials were carried out in 1980 in the Netherlands with 1 ×0.25–1.0 kg ai/ha, PHI 140 days, root and top. Four decline trails were performed in Germany in 1980 with 1 ×1.0 kg ai/ha and PHI 0, 19–23, 41–43, 62–64, 85–86, 112–113 days, root and tops[FP+F]). Additional trials were carried out in Canada in 1979 with 1 ×0.125–0.5–1.0 kg ai/ha, PHI 123 days, roots and tops[FB+F] and in 1980 with 1 ×0.25–0.5 kg ai/ha, PHI 111 days, for root and tops. The summary report contains data on a broad variety of crops, but no trial location details, sampling details or storage information. HPLC-UV methods PPRAM 51 (determines fluazifop-butyl) and PPRAM52 (determines free fluazifop acid (II)) were used in the majority of these residue trials, indicated with [FP+F] and these were addressed in the metabolism section. For the remaining trials HPLC-UV method PPRAM62 (total fluazifop) was applied. Some of the residue trials reported in [RJ0226B] were also reported in individual study reports; Canada 1979 [Atreya, 1980, PP9/0499, report PP009B005], UK 1979 [Atreya, 1980, PP9/0502, report PP009B008], Germany [Atreya, 1981, PP9/0508, report PP009B014]. These trials were not summarized, because they would not assist in MRL setting.

[Atreya and Harradine, 1982, PP9/0062, report RJ0291B] summarized the results of several residue trials carried out with fluazifop-butyl on sugar beets and fodder beets in the UK (1981: 2 ×0.38 kg ai/ha, PHI 29–78 days, 1 ×0.19 kg ai/ha, PHI 63–77 days or 1 × 0.75–1.5 kg ai/ha, PHI 43–46 or 63–84 days in foliage and roots), Canada (1981: 1 ×0.25–0.35–0.40 kg ai/ha, PHI 74–87 days), Sweden (1981: 1 × 0.50 kg ai/ha, PHI 86–103 days). These trials were not summarized, because they would not assist in MRL setting.

Table 247 Supervised field trials on sugar beet (tops, foliage), treated with a broadcast or banded foliar fluazifop-butyl spray

SUGAR BEET TOPS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Broadcast foliar spray										
Campbell, Minnesota, USA, 2000 (Resist)	EC 240 (P) +0.5% NIS	2 (12)	0.42 0.42	0.22 0.22	post emergence; 30 June	CL	28 Sept, 2000	90	0.37, 0.52 mean: 0.44	RR 00-066B 255 (SBMN1); [Stewart, 2011, PP5/1070]
Geneva, Minnesota, USA, 2000 (Crystal 205)	EC 240 (P) +1% COC	2 (14)	0.42 0.42	0.52 0.50	post emergence; 18 July	CL	16 Oct, 2000	90	0.35, 0.35 mean: 0.35	RR 00-066B 256 (SBMN2); [Stewart, 2011, PP5/1070]
Brampton, North Dakota, USA, 2000 (Beta 6104)	EC 240 (P) +0.5% NIS	2 (14)	0.42 0.42	0.36 0.36	post emergence; 7 July	SiL	04 Oct, 2000	89	0.33, 0.42 mean: 0.38	RR 00-066B 257 (SBND1); [Stewart, 2011, PP5/1070]
Northwood, North Dakota, USA, 2000 (Beta 6600)	EC 240 (P) +1% COC	2 (14)	0.42 0.42	0.23 0.23	post emergence; 11 July	SiL	09 Oct, 2000	90	0.33, 0.33 mean: 0.33	RR 00-066B 258 (SBND2); [Stewart, 2011, PP5/1070]
Delavan, Wisconsin, USA, 2000 (American Crystal 196)	EC 240 (P) +0.5% NIS	2 (13)	0.42 0.42	0.46 0.47	post emergence; 27 July	SiL	25 Oct, 2000	90	0.44, 0.32 mean: 0.38	RR 00-066B 259 (SBWI1); [Stewart, 2011, PP5/1070]
Grand Island, Nebraska, USA, 2000 (HM1605)	EC 240 (P) +0.5% NIS	2 (14)	0.42 0.42	0.45 0.45	post emergence; 11 July	SiL	09 Oct, 2000	90	0.55, 0.22 mean: 0.38	RR 00-066B 260 (SBNE1); [Stewart, 2011, PP5/1070]
Larned, Kansas, USA, 2000 (Crystal 203)	EC 240 (P) +0.5% NIS	2 (14)	0.42 0.42	0.23 0.22	post emergence; 19 June	LSa	17 Sept, 2000	90	0.02, 0.06 mean: 0.04	RR 00-066B 261 (SBKS1); [Stewart, 2011, PP5/1070]
Edgar, Montana, USA, 2000 (Mono-Hy)	EC 240 (P) +0.5% NIS	2 (12)	0.42 0.42	0.30 0.24	post emergence; 1 July	L	29 Sept, 2000	90	0.60, 0.45 mean: 0.52	RR 00-066B 262 (SBMT1); [Stewart, 2011, PP5/1070]
Porterville, California 2000, USA (Encrusted 8 ½-9 1/2)	EC 240 (P) +0.5% NIS	2 (14)	0.42 0.42	0.22 0.22	post emergence; 25 July	SaL	23 Oct, 2000	90	0.63, 0.61 mean: 0.62	RR 00-066B 263 (SBCA1); [Stewart, 2011, PP5/1070]
Visalia, California, USA, 2000	EC 240 (P)	2 (14)	0.42 0.42	0.23 0.23	post emergence; 15 August	fine SaL	13 Nov, 2000	90	0.20, 0.10 mean:	RR 00-066B 264 (SBCA2); [Stewart, 2011,

SUGAR BEET TOPS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(SB-SS-NBSR)	+1% COC								0.15	PP5/1070]
American Falls, Idaho, USA, 2000 (Beta 4490R)	EC 240 (P) +0.5% NIS	2 (13)	0.42 0.42	0.24 0.26	post emergence; 29 June	SiL	27 Sept, 2000	90	0.56, 0.41 mean: 0.48	RR-00-066B 265 (SBID1); [Stewart, 2011, PP5/1070]
Ephrata, Washington, USA, 2000 (Canyon)	EC 240 (P) +0.5% NIS	2 (14)	0.42 0.42	0.18 0.18	post emergence; 13 July	SaL	11 Oct, 2000	90	0.60 (0.54), 1.5 (1.4) [c] mean: 1.0	RR-00-066B 266 (SBWA1); [Stewart, 2011, PP5/1070]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	13 0.17 0.05 < 0.05 n.a. < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 19 42 61 85 111	17 0.20 0.08 < 0.05 n.a. < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 97	12 0.51 0.33 0.09 0.11 0.07 [QU]	idem
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 95	8.3 0.20 0.10 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	12 0.25 < 0.05 < 0.05 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 19 42 61 85 111	24 0.46 0.40 0.14 0.08 0.09	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]

SUGAR BEET TOPS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg [QU]	Report; Trial no [ref]
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 21 42 63 84 97	25 0.67 0.050 < 0.05 0.14 < 0.05 [QU]	idem
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 21 42 63 84 95	25 3.6 0.10 < 0.05 < 0.05 0.07 [QU]	idem
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	9.6 < 0.05 0.06 < 0.05 na < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 97	13 0.97 0.29 0.18 0.14 0.05 [QU]	idem
idem	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 95	8.8 1.3 0.40 0.10 0.17 0.23 [QU]	idem
6748 Bad Bergzabern; Germany; 1983; (Monopur)	EC 125 (P)	1	0.38	0.094	10-14 leaves; 20 cm tall; 23 June	ns	ns	0 21 42 62 93	20 1.5 0.49 0.26 0.11 [SS]	M3701B; RS 8369 E1(A); [Upton, 1984, PP9/0054]
idem	EC 250 (rac)	1	0.75	0.19	10-14 leaves; 20 cm tall; 23 June	ns	ns	0 21 42/43 62/64 77 93/96	38 1.7 0.31 0.59 NA 0.25 [SS]	M3701B; RS 8369 E1[b]; [Upton, 1984, PP9/0054]
6745 Offenbach; Germany;	EC 125 (P)	1	0.38	0.094	10-14 leaves; 20 cm tall;	ns	ns	0 21 42	17 0.80 0.24	M3701B; RS 8369 E2(A); [Upton, 1984,

SUGAR BEET TOPS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
1983; (Kawemono)					21 June			62 93	0.27 0.16 [SS]	PP9/0054]
idem	EC 250 (rac)	1	0.75	0.188	10-14 leaves; 20 cm tall; 21 June	ns	ns	0 21 42/43 62/64 77 93/96	31 1.1 0.48 0.72 NA 0.06 [SS]	M3701B; RS 8369 E2[b]; [Upton, 1984, PP9/0054]
2057 Schwarzenbek; Germany; 1983; (Nova Gema)	EC 125 (P)	1	0.38	0.094	12-14 leaves; 7 July	ns	ns	0 21 42 62 77	20 2.1 <u>1.3</u> 0.73 0.40 [SS]	M3701B; RS 8369 B1(A); [Upton, 1984, PP9/0054]
idem	EC 250 (rac)	1	0.75	0.188	12-14 leaves; 7 July	ns	ns	0 21 42/43 62/64 77 93/96	26 3.0 1.8 1.0 0.57 NA [SS]	M3701B; RS 8369 B1[b]; [Upton, 1984, PP9/0054]
7101 Cunnersdorf; Germany; 1992; (Hilma)	EC 125 (P)	1	0.38	0.125	BBA 45; at 3 July	SaL	43 49 90	0 47 90	13 <u>0.36</u> 0.24	RJ1424B; RF 12/92 CU; [Bolygo, 1993, PP5/0098]
idem	ME 125 (P)	1	0.38	0.125	BBA 45; 3 July	SaL	43 49 90	0 47 90	13 0.32 0.13	RJ1424B; RF 12/92 CU; [Bolygo, 1993, PP5/0098]
2021 Rosenow; Germany; 1992; (Kawetina)	EC 125 (P)	1	0.38	0.125	BBA 27-41; 20 July	L	27 43 90	0 31 91	18 1.0 0.09	RJ1424B; RF 12/92 RO; [Bolygo, 1993; PP5/0098]
idem	ME 125 (P)	1	0.38	0.125	BBA 27-41; 20 July	L	27 43 90	0 31 91	15 0.63 0.13	RJ1424B; RF 12/92 RO; [Bolygo, 1993; PP5/0098]
Coblenz; Germany 1992; (Kawetina)	EC 125 (P)	1	0.38	0.125	BBA 43; 30 June	C	43 49 90	0 49 90	18 <u>0.37</u> 0.21	RJ1424B; RF 12/92 CO; [Bolygo, 1993; PP5/0098]
idem	ME 125 (P)	1	0.38	0.125	BBA 43; 30 June	C	43 49 90	0 49 90	24 0.29 0.15	RJ1424B; RF 12/92 CO; [Bolygo, 1993; PP5/0098]
5301 Kötschau; Germany; 1992; (Dunja)	EC 125 (P)	1	0.38	0.125	BBA 43-45; 1 July	L	43 49 90	0 49 90	19 <u>0.47</u> 0.21	RJ1424B; RF 12/92 KO; [Bolygo, 1993; PP5/0098]
idem	ME 125	1	0.38	0.125	BBA 43-45;	L	43 49	0 49	22 0.47	RJ1424B; RF 12/92 KO;

SUGAR BEET TOPS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
	(P)				1 July		90	90	0.26	[Bolygo, 1993; PP5/0098]
D94522 Wallersdorf-See Germany, 2002 (Cyntia)	EC 125 (P)	1	0.38	0.12	BBCH 39-49; 65 cm tall; 90% crop cover; 29 July	CL	49	56	1.7 1.7 mean 1.7 [a]	gsb064002 trial no ns; [Simon, 2003, PP5/1337]
Wallersdorf-See Germany, 2002 (Corinna)	EC 125 (P)	1	0.38	0.12	BBCH 49; 60-65 cm tall; 90% crop cover; 27 August	CL	MAT	52	0.48 0.58 Mean 0.53 [a]	gsb064202 trial no ns [Simon, 2003, PP5/1336]
Location ns; UK, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	63	< 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns Sweden, 1981, (var ns)	EC 250 (rac)	2	0.50	ns	GS ns date ns	ns	ns	84 91	0.17 < 0.05	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
42110; Feurs; S-France, 2004 (Laetitia)	EC 125 (P)	1	0.37	0.12	BBCH 38; 21 July	LC	49	56	0.72 [SS]	CEMR-2310 AF/7840/SY/1; [Kang, 2005, PP5/1441]
42450; Sury le Comtal; S-France, 2004 (Laetitia)	EC 125 (P)	1	0.36	0.12	BBCH 39; 21 July	LC	49	56	0.40 [SS]	CEMR-2310 AF/7840/SY/2 [Kang, 2005, PP5/1441]
S Agata Bolognese; Emilia Romagna; Italy, 1998 (Nubia)	EC 125 (P)	1	0.38	0.11	BBCH 39; 40-50 cm tall; 6 July	L	ns	56	1.1	RJ2779B IT20-98-H321; [Mason and Volpi; 1999, PP5/0124]
Voghera; Lombardia; Italy, 1998 (Asso)	EC 125 (P)	1	0.38	0.094	BBCH 39 – 41; 40-50 cm tall; 5 June	L	ns	56	0.83	RJ2779B IT30-98-H320; [Mason and Volpi; 1999, PP5/0124]
40016; Mascarino Venezzano; Italy, 2004 (Flavia)	EC 125 (P)	1	0.37	0.12	BBCH 39-49; 13 July	CL	49	57	1.4 [SS]	CEMR-2310 AF/7840/SY/3 [Kang, 2005, PP5/1441]
Banded foliar spray over rows										
Biota; Zaragoza; Aragón; Spain, 1998 (Oryx)	EC 125 (P)	1	0.38	0.094	BBCH 85; 50 cm tall; 7 Oct	C	93	54	2.2 [b] [SS]	RJ2833B ES10-98-SH007; [Ryan and Gallardo, 1999, PP5/0121]
Biota; Zaragoza; Aragón;	EC 125 (P)	1	0.37	0.094	BBCH 85; 50 cm tall; 7 Oct	C	93	54	1.8 [b] [SS]	RJ2833B ES10-98-SH107; [Ryan and Gallardo,

SUGAR BEET TOPS; Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Spain, 1998 (Korif)										1999, PP5/0121]
Sevilla; Andalucia; Spain, 1999 (Lola)	EC 125 (P)	1	0.43	0.093	BBCH 49; 60 cm tall; 24 May	L	MAT	56	0.89	RJ2995B ES51-99-S021; [Mason and Gallardo, 2000, PP5/0308]

BBCH 27-29: leaf development;

BBCH 30-39: rosette growth (leaves cover 10-90% of ground);

BBCH 40-49: development of roots (beets have 10-90% of harvestable size).

IMM = immature (trial TRS 211.85, DAT 30-45 harvested at 12-16 leaves)

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

[SS] Sample size not stated (M3701B) or less than the required 12 plants (1.4-3.4 kg in RJ2833, 1 kg in CEMR-2310).

[a] Replicate samples were taken from the same plot; the mean is taken for MRL derivation, , if according to cGAP.

[b] Each field sample was analysed in triplicate; the mean result is reported in the table

[c] () results of second analysis.

Additional trial information;

RR-00-066B GLP study. Apart from early frost (no impact on study) no unusual weather conditions. Spray application bytractor mounted, equipment or backpack sprayer. Spray volume 75-280 L/ha. Sugar beet plants (at least 12 items) were sampled by hand. Tops were cut from the roots and bagged separately. They were stored frozen for up to 2.5 months. Samples were analysed for total fluazifop using **GC-MS method RR91-014B with a valid LOQ of 0.01 mg/kg**. Concurrent recoveries of total fluazifop ranged from 67-110% at 0.01 and 1.0 mg/kg, Control samples were < 0.01 mg.kg.

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.03 mg/kg.

M3701B Non GLP study. No unusual weather conditions. Application by knapsack sprayer with 1.5-2.5 boom. Spray volume 400 L/ha. Sample size not stated. Dry leaves were removed. Storage at -30°C, maximum 231 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (78% at 0.5-10 mg/kg). Control samples < 0.05 mg/kg.

RJ1424B GLP study. Weather conditions did not affect growth except at Rosenow, where the growth of the sugar beets was slower due to dry weather conditions. Application by mobile small plot sprayers. Spray volume 300 L/ha. Sample size 12 plants at each sampling interval (> 2 kg leaves, except 1.2 kg leaves at Rosenow DAT0). Storage at -18 °C for maximum 7 months. Samples were analysed for total fluazifop using **NMR method ARAM 197 with a valid LOQ of 0.05 mg/kg**. Concurrent mean internal standard recovery (97-107% at 0.1-5 mg/kg). Control samples < 0.05 mg/kg.

gsb064002. GLP study. No unusual weather conditions. Broadcast foliar application by plot sprayer. Spray volume 300 L/ha. Sugar beet plants were sampled by hand (12 plants). Roots and tops were separated. The lab sample >1.7 kg leaves is a representative part of the field sample (weight not recorded). Storage at -18°C, maximum 169 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recoveries (107-113% at 0.01-1.0 mg/kg). Control samples < 0.01 mg/kg.

gsb064202. GLP study. No unusual weather conditions. Application by knapsack sprayer using a spray volum of 300 L/ha. 12 plants were sampled . Roots and tops were separated. Storage at -18°C, maximum 144 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg** was used. Mean internal standard recovery (92-111% at 0.01-1.0 mg/kg). Control samples < 0.01 mg/kg.

CEMR-2310. GLP study. No unusual weather conditions. Broadcast foliar spray using a plot sprayer. Spray volume 290-300 L/ha. Sugar beet tops (1 kg, no of items not stated, but weight too low) were harvested by hand. Storage at -17°C, maximum 285 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recoveries (85-88% at 0.01–0.1 mg/kg). Control samples < 0.01 mg/kg

RJ2779B. GLP study. No unusual weather conditions. Broadcast foliar spray with motor knapsack sprayer with boom. Spray volume 350-400 L/ha. Sugar beet plants (12 items; 2.1-2.8 kg) were sampled by hand and taken systematically from across the plots. The roots with tops and leaves were subsampled by dividing the roots, longitudinally, into four parts and

retaining one quarter of each.. Roots and tops were separated thereafter. Storage at -18°C for 74-105days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for averageconcurrent recoveries (104% at 0.1 mg/kg). Control samples < 0.01 mg/kg.

RJ2833B. GLP study. No unusual weather conditions. **Banded spray over rows** using a gas knapsack sprayer. Spray volume 400 L/ha. Sugar beet plants (1.45-3.45 kg; no of items not stated, but weight too low) were sampled by hand and taken systematically from across the plots. Roots and leaves were separated. Storage at -18°C for 206-234 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were corrected for individual concurrent recoveries (109-125% at 0.25-1.0 mg/kg). Uncorrected results were not reported. Control samples 0.01 mg/kg, therefore the LOQ needs to be increased to 0.01/0.3=0.04 mg/kg. Since residue levels were > 0.04 mg/kg, this has no effect on the results.

RJ2995B. GLP study. No unusual weather conditions. **Banded spray over rows** using a gas knapsack sprayer. Spray volume 464 L/ha. Sugar beet whole plants (3-6 kg; no of items not stated, but weight is sufficiently high) were sampled by hand and taken systematically from across the plots. Roots and leaves were separated. Storage at -18°C for 122-129 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for individual concurrent recoveries (89-104% at 0.5-1.0 mg/kg). Control samples < 0.01 mg/kg.

Table 248 Supervised field trials on fodder beet (tops, foliage), treated with a broadcast foliar fluazifop-butyl spray

FODDER BEET TOPS Location; Country; year; (variety)	For-mu-lation	no. of appl; (inter-val)	kg ai/ha	kg ai/hL	GS; at last treat-ment day	Soil type	GSH	DAT days	Total fluazifop[1] (mg/kg)	Report Trial no. [ref]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	24 0.10 < 0.05 < 0.05 n.a. < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 21 42 63 84 91	21 0.68 0.33 0.09 0.09 0.07 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	23 0.49 < 0.05 < 0.05 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.75	ns	GS ns date ns	ns	ns	0 21 42 63 84 91	41 1.3 0.31 0.14 < 0.05 < 0.05 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
Location ns; Germany, 1981 (var ns)	EC 250 (rac)	1	0.38	ns	GS ns date ns	ns	ns	0 23 41 62 86 107	14 0.11 < 0.05 < 0.05 na < 0.05	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]

FODDER BEET TOPS Location; Country; year; (variety)	For-mu-lation	no. of appl; (inter-val)	kg ai/ha	kg ai/hL	GS; at last treat-ment day	Soil type	GSH	DAT days	Total fluazifop[1] (mg/kg)	Report Trial no. [ref]
									[QU]	
2057 Schwarzenbek; Germany; 1983; (Rote Eckern-dorfer)	EC 125 (P)	1	0.38	0.094	12-14 leaves; 7 July	ns	ns	0 21 42 62 77	22 2.7 1.1 0.49 0.33 [SS]	M3701B; RS 8369 B2A; [Upton, 1984, PP9/0054]
idem	EC 250 (rac)	1	0.75	0.19	12-14 leaves; 7 July	ns	ns	0 21 42 63 77	31 3.5 0.91 0.84 0.47 [SS]	M3701B; RS 8369 B2B; [Upton, 1984, PP9/0054]
Almind; Denmark, 1988 (Magna-Mono)	EW 125 (P)	2; (30)	0.19 0.19	0.075 0.075	6-8 leaves; 50% crop cover; 19 June	L	ns	46 61 75	0.17 0.15 0.11 [SS]	M4870B DK10-88-H070; [Hayward and Harradine; 1989; PP5/0519]
Almind; Denmark, 1988; (Magna-Mono)	EW 125 (P)	1	0.38	0.19	4-5 leaves; 40% crop cover; 2 June	L	ns	44 63 77	0.10 0.06 0.05 [SS]	M4870B DK10-88-HI41 [Hayward and Harradine; 1989; PP5/0519]

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported
[SS] Sample size not stated (M3701B, M4870B) or less than the required 12 plants. Not suitable for MRL derivation

Additional trial information:
RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported.. Storage conditions not stated. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.03 mg/kg.
M3701B. Non GLP study. No unusual weather conditions. Application by knapsack sprayer with 1.5-2.5 boom. Spray volume 400 L/ha. Sample size not stated. Dry leaves were removed. Storage at -30°C, maximum 231 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (78% at 0.5-10 mg/kg). Control samples < 0.05 mg/kg.
M4870B Non GLP study. No unusual weather conditions. Broadcast foliar spray application by plot sprayer. Spray volume 200-250 L/ha. Fodder beets were sampled by hand and roots and tops were separated. Sample size not stated.. Storage -18 ºC for a maximum of 138days. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (91% at unknown level). Control samples < 0.01mg/kg.

Swede tops

Two cGAPs for swedes are available:

- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 42 days
- cGAP from the Netherlands, Belgium and the UK with 1 ×0.38 kg ai/ha and a PHI 56 days

Swede tops are harvested at the same time as the roots. Trials that could be matched to these cGAPs were summarized.

Table 249 lists trials conducted in the UK (1984, 1989). A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 249. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample size was less than the required 12 plants.

Table 249 Supervised field trials on swede (tops), treated with a broadcast foliar fluazifop-butyl spray

SWEDE TOPS Location, Country, year (Variety)	Form (g ai/L)	No (inter val in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Balerno, Mid Lothian; UK; 1984; (Ruta Otofte)	EC 125 (P)	1	0.38	0.19	4 leaves; 25 June	L	MAT	56	0.58 [SS]	M4001B; 9R/84 SAI37; [Harradine, 1985, PP5/0100]
Winchburgh, West Lothian; UK; 1984; (Ruta Otofte)	EC 125 (P)	1	0.38	0.19	4 leaves; 25 June	L	MAT	56	0.63 [SS]	M4001B; 9R/84 SAI38; [Harradine, 1985, PP5/0100]
Kirknewton, West Lothian; UK; 1984; (Ruta Otofte)	EC 125 (P)	1	0.38	0.19	4-6 leaves; 25 June	L	MAT	56	0.67 [SS]	M4001B; 9R/84 SAI39; [Harradine, 1985, PP5/0100]
Upper Largo, Fife; UK; 1984; (Ruta Otofte)	EC 125 (P)	1	0.38	0.19	4-6 leaves; 25 June	ns	MAT	56	0.68 [SS]	M4001B; 9R/84 SAI41; [Harradine, 1985, PP5/0100]
South Queensferry; UK; 1984; (Marian Greentop)	EC 125 (P)	1	0.38	0.19	4 leaves; 25 June	L	MAT	56	0.64 [SS]	M4001B; 9R/84 SAI42; [Harradine, 1985, PP5/0100]
Linlithgow, West Lothian; UK; 1989; (Ruta Otofte)	EW 125 (P) + Agral	1	0.42	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.98	M5318B; GB18-89-S421; [Cullen, 1991, PP5/0101]
idem	EW 250 (P) + Agral	1	0.42	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.56	idem
idem	EC 125 (P) + Agral	1	0.38	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.77	idem
Linlithgow, West Lothian; UK; 1989; (Doon Major)	EW 125 (P) + Agral	1	0.38	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.23	M5318B; GB18-89-S422 [Cullen, 1991, PP5/0101]
idem	EW 250 (P) + Agral	1	0.42	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.35	idem
idem	EC 125 (P) + Agral	1	0.42	ns	12 true leaves; 50% crop cover; 18 Aug	SiL	CH	70	0.75	idem

MAT = mature (roots 7.6-15 cm diameter);

[SS] Sample size was not stated.

Additional trial information:

M4001B. Non-GLP. No unusual weather conditions. Spray application using a CO_2 knapsack sprayer. Spray volume 200 L/ha. Sample sizes not stated. Storage at -20 °C, storage time not stated but less than 12 months. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with internal standard with a valid LOQ of 0.05 mg/kg**. Mean internal standard recovery (77% at 0.5 mg/kg). Control samples < 0.03 mg/kg.

M5318B. GLP. No unusual weather conditions. Samples of 12 plants. Roots and leaves were separated. Roots were sub-sampled in the field: cut longitudinally in 4 quarters and 1 quarter was retained for analysis. Storage at -18 °C; storage time not stated but less than 12 months. Samples were analysed for total fluazifop using **NMR method PPRAM 83 with a valid LOQ of 0.05 mg/kg.** Mean internal standard recovery (108% at 0.5 mg/kg). Control samples < 0.05 mg/kg.

Oilseed rape forage

Four cGAPs for oilseed rape are available:

- cGAP from Brazil and the UK with 1 ×0.19 kg ai/ha and a PHI of 14 days
- cGAP from France with 1 ×0.38 kg ai/ha and a PHI of 90 days
- cGAP from the Netherlands with 1 ×0.38 kg ai/ha before winter (i.e. end of December) and post-emergence of the crop
- cGAP from Belgium with 1 × 0.19 kg ai/ha before winter (i.e. end of December) and up to 15 cm crop height

Canola (oilseed rape) can be grazed when the canopy height is 15–20 cm tall. Since the GAP does not have grazing restrictions, oilseed forage can be harvested at any time after treatment (PHI = 0 days). Trials that could be matched to these cGAPs were summarized.

Table 250 lists trials conducted in Germany (1981, 1982-1983, 1993). A broadcast foliar spray application with fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 250.

[QU] indicates that the quality of the study was very poor, since field conditions (weather, plot size, sample size, application equipment) and storage conditions were not reported.

[SS] indicates that the sample size was less than the required 1 kg rape forage.

Additional trials from Germany (1979–1980) were available on rape forage with 2 × 0.50 kg ai/ha or 1 × 1.0 kg ai/ha and harvest at 0–216 DAT [Atreya and Froggatt, 1981, PP9/0384, report RJ0226B]. These trials were summarized in the metabolism section, because only the fluazifop-butyl and free fluazifop residues were analysed.

Table 250 Supervised field trials on oilseed rape (forage), treated with a broadcast foliar fluazifop-butyl spray

OILSEED RAPE FORAGE Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Location ns; Germany, 1981 (spring oilseed rape; var ns)	EC 250 (rac)	1	0.31	ns	GS ns date ns (April/May)	ns	ns	0 9 18 30 42	6.0 2.9 3.1 2.2 0.97 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns (April/May)	ns	ns	0 9 18 30 42	10 5.8 5.6 4.1 2.1	idem

OILSEED RAPE FORAGE Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg [QU]	Report; Trial no [ref]
Location ns; Germany, 1981 (spring oilseed rape; var ns)	EC 250 (rac)	1	0.31	ns	GS ns date ns (April/May)	ns	ns	0 9 17 30 42	6.4 3.6 3.6 1.4 0.78 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns (April/May)	ns	ns	0 9 17 30 42	11 9.4 8.2 3.9 1.6 [QU]	idem
Location ns; Germany, 1981 (spring oilseed rape; var ns)	EC 250 (rac)	1	0.31	ns	GS ns date ns (April/May)	ns	ns	0 8 17 28 42	6.5 4.4 2.6 1.4 0.51 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns (April/May)	ns	ns	0 8 17 28 42	24 14 6.7 3.8 1.2 [QU]	idem
Location ns; Germany, 1981 (spring oilseed rape; var ns)	EC 250 (rac)	1	0.31	ns	GS ns date ns (April/May)	ns	ns	0 7 14 28 42	4.6 2.7 2.5 1.3 0.42 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns (April/May)	ns	ns	0 7 14 28 42	24 11 12 3.5 1.1 [QU]	idem
Location ns; Germany, 1981 (spring oilseed rape; var ns)	EC 250 (rac)	1	0.31	ns	GS ns date ns (April/May)	ns	ns	0 7 14 28 42	5.8 1.9 2.5 1.3 0.38 [QU]	RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns (April/May)	ns	ns	0 7 14 28 42	21 9.1 7.1 4.0 1.5 [QU]	idem
Location ns; Germany, 1981	EC 250 (rac)	1	0.31	ns	GS ns date ns (April/May)	ns	ns	0 7 14	8.6 2.6 0.54	RJ0291B summary [Atreya and

OILSEED RAPE FORAGE Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
(spring oilseed rape; var ns)								28 42	0.09 0.08 [QU]	Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	1	0.75	ns	GS ns date ns (April/May)	ns	ns	0 7 14 28 42	31 9.6 1.9 0.72 0.33 [QU]	idem
2411 Klein-Zecher/Mollin Germany; 1982-83; (winter oil seed rape; Belinda)	EC 125 (P)	1	0.38	0.094	30 cm high; 7- 9 leaves; soil cover ns; 10 Apr 1983	ns	ns	0 14 28 41	7.3 2.7 1.4 0.7 [SS]	M3685B; RS 8370 B1 (A); [Upton, 1984, PP9/0399]
idem	EC 250 (rac)	1	0.75	0.19	30 cm high; 7- 9 leaves; soil cover ns; 10 Apr 1983	ns	ns	0 14 28 42	16 6.2 3.1 2.0 [SS]	M3685B; RS 8370 B1 [b]; [Upton, 1984, PP9/0399]
3141 Brakeded-Bleckede Germany; 1982-83; (winter oilseed rape; Jet neuf)	EC 125 (P)	1	0.38	0.094	30 cm high; 7-9 leaves; soil cover ns; 18 Apr 1983	ns	ns	0 14 28 42	8.7 2.8 1.4 0.74 [SS]	M3685B; RS 8370 B3; [Upton, 1984, PP9/0399]
idem	EC 250 (rac)	1	0.75	0.19	30 cm high; 7-9 leaves; soil cover ns; 18 Apr 1983	ns	ns	0 14 28 42	12 5.8 4.0 1.6 [SS]	M3685B; RS 8370 B3; [Upton, 1984, PP9/0399]
Pirmasens-Windberg; Germany; 1982-83; (winter oilseed rape; Belinda)	EC 125 (P)	1	0.38	0.094	40 cm high; Flower buds developing; soil cover 75%; 19 Apr 1983	ns	ns	0 14 28 42	11 3.0 0.93 1.1 [SS]	M3685B; RS 8370 E1; [Upton, 1984, PP9/0399]
idem	EC 250 (rac)	1	0.75	0.19	40 cm high; Flower buds developing; soil cover 75%; 19 Apr1983	ns	ns	0 14 28 42	20 4.5 1.4 2.0 [SS]	M3685B; RS 8370 E1; [Upton, 1984, PP9/0399]
2059 Krukow-Lavenberg E; Germany; 1983 (spring oilseed rape; Ergula)	EC 125 (P)	1	0.38	0.094	30 cm high; 7-9 leaves; soil cover ns; 10 May	ns	ns	0 14 28 42	16 3.7 2.0 1.4 [SS]	M3685B; RS 8370 B2 (A); [Upton, 1984, PP9/0399]
idem	EC 250 (rac)	1	0.75	0.19	30 cm high; 7-9 leaves; soil cover ns; 10 May	ns	ns	0 14 28 42	22 6.7 3.5 2.0 [SS]	M3685B; RS 8370 B2; [Upton, 1984, PP9/0399]

OILSEED RAPE FORAGE Location; Country; year; (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
23919 Berkenthin; Germany; 1993; (winter oilseed rape; Lirajet)	EC 125	1	0.38	0.125	(30 cm high; BBCH 39; 90% crop cover); at 8 April	SaL	39 57 64 69	0 14 27 40	13 4.5 3.0 1.5	RJ1660B; RS-9304-B1; [Bolygo, 1994; PP5/0217]
idem	ME 125	1	0.38	0.125	(30 cm high; BBCH 39; 90% crop cover); at 8 April	SaL	39 57 64 69	0 14 27 40	12 <u>4.6</u> 3.7 1.2	RJ1660B; RS-9304-B1; [Bolygo, 1994; PP5/0217]
85375 Neufarn; Germany; 1993; (winter oilseed rape; Lirabon)	EC 125	1	0.38	0.19	(20 cm high; BBCH 37; 80% crop cover); at 14 April	L	37 61 65 71	0 13 29 41	21 <u>3.8</u> 1.9 0.73	RJ1660B; RS-9304-G1; [Bolygo, 1994; PP5/0217]
idem	ME 125	1	0.38	0.19	(30 cm high; BBCH 37; 80% crop cover); at 14 April	L	37 61 65 71	0 13 29 41	21 3.6 1.5 0.63	RJ1660B; RS-9304-G1; [Bolygo, 1994; PP5/0217]
Klein Zecher; Germany; 1993; (autumn sown; Lirajet)	ME 125 (P)	1	0.38	0.125	15 cm; BBCH 23; 29 Oct	SaL	23 23 37-39 61	0 12 171 185	29 [SS] 16 [SS] 0.82 0.23	RJ1846B; RS-9305-B1; [Bolygo, 1995, PP5/0220]
idem	EC 125 (P)	1	0.38	0.12	15 cm; BBCH 23; 29 Oct	SaL	23 23 37-39 61	0 12 171 185	29 [SS] 18 [a][SS] 1.0 0.20	RJ1846B; RS-9305-B1; [Bolygo, 1995, PP5/0220]
068888 Dabrun; Germany; 1993; (autumn sown: Idol)	ME 125 (P)	1	0.38	0.094	17 cm; BBCH 25; 28 Oct	SaL	25 25 39 61	0 16 169 179	19 9.3 1.6 0.50	RJ1846B; RS-9305-K1; [Bolygo, 1995, PP5/0220]
idem	EC 125 (P)	1	0.38	0.094	17 cm; BBCH 25; 28 Oct	SaL	25 25 39 61	0 16 169 179	17 <u>10</u> 1.4 0.84	RJ1846B; RS-9305-K1; [Bolygo, 1995, PP5/0220]

BBCH 20-29: Formation of side shoots. BBCH 30-39: stem elongation. BBCH 50: flower buds present, still enclosed by leaves.

[QU] Quality of the study insufficient for MRL derivation since the field and storage conditions were not reported

SS Sample sizes not stated in the report and may be insufficient for MRL derivation.

[a] Mean of triplicate analysis.

Additional trial information:

RJ0291B. non-GLP. Poor quality of the study. Weather, equipment, soil type, plot size, sample size, growth stage at harvest were not reported. Storage conditions not stated. Samples were analysed for total fluazifop using HPLC-UV method **PPRAM 62 with a valid LOQ of 0.05 mg/kg**. Concurrent method recoveries (79-92% at 0.05-10 mg/kg in various crops). Control samples were < 0.05 mg/kg.

M3685B, GLP study. Winter oilseed rape, sown on 18-28 August 1982, treated in April 1983. Spring oilseed rape, sown on 3 March 1983, treated in May 1983. Weather conditions not stated. Boom sprayer or knapsack boom sprayer with spray volume 400 L/ha. Foliage was harvested by hand. Sample sizes were not stated. Storage at -18 °C or lower for maximum 226 days. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg**. Average internal standard recoveries were 66% at 0.5 mg/kg 71-85% at 1-10 mg/kg. Control samples were < 0.05 mg/kg.

RJ1660B, GLP study. Winter oilseed rape, sown on 20-24 August 1992, treated in April 1993. No unusual weather conditions. Spray volume 200-300 L/ha. Sampling were harvested by hand (B1) or with a small plot harvester (G1). Sample size 20-40 plants for forage (0.8-2.9 kg). Storage less at -18 °C for maximum 11 months. Samples were analysed for total fluazifop using **NMR method RAM 197/01 with a valid LOQ of 0.05 mg/kg**. Concurrent individual internal standard recoveries were 78-98% at 1 mg/kg. Concurrent individual method recoveries (80-109% at 0.05-1.0 mg/kg). Control samples were < 0.05 mg/kg.

RJ1846B. Winter oilseed rape. Weather at application and soil type were reported. Spray volume 300-400 L/ha. At least 20 plants/sample or 0.8 kg seed/sample was taken by hand systematically from across the plots avoiding boundaries. Samples sizes up to BBCH 23 were 0.8 kg in trial RS-9305-B1. Sample sizes at higher growth stages and the other trial were ≥1.0 kg. Storage less than 7 months at -18 °C. Samples were analysed for total fluazifop using **RAM 197/02 with a valid LOQ of 0.05 mg/kg**. Samples were not corrected for concurrent method recoveries (72-118% at 1-15 mg/kg. Control samples < 0.05 mg/kg.

Sunflower forage

Although trials were submitted for sunflower forage, these trials were not summarized because sunflower forage is not in the OECD feed table for dietary burden calculations.

Fodder and forage of grasses

Grass forage

One cGAP for grass is available:

- cGAP from the Netherlands with 1 ×0.25 kg ai/ha with a PHI of 49 days.

Trials that could be matched to this GAP were summarized.

Table 251 lists trials conducted in the USA (2010, 2012) and the Netherlands (1997) on red fescue (Festuca rubra) and fine fescue (Festuca rubra var trichopylla) grass. A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 251. Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample size was less than the required 1 kg grass forage.

Red fescue grass is tolerant to the treatment with fluazifop-P-butyl and so a harvestable forage crop is possible after application.

Table 251 Supervised field trials on grass (forage), treated with a broadcast foliar fluazifop-butyl spray

GRASS FORAGE Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Silverton, Oregon, USA, 2010 (LaCross fine fescue)	EC 125 (P) + COC	3 (12, 13)	0.29 0.29 0.30	0.15 0.15 0.15	vegetative, 15 cm high; 31 March	SiCL	forage	14	1.9, 1.8 mean = 1.8 [SS]	IR-4 PR 09825; 10-OR19 ; [Jolly, 2014, PP5_50554]
Silverton, Oregon, USA, 2010 (Shadow II fine fescue)	EC 125 (P) + COC	3 (12, 13)	0.28 0.28 0.29	0.086 0.086 0.086	vegetative, 15 cm high; 31 March	SiCL	forage	14	1.3, 1.5 mean = 1.4 [SS]	IR-4 PR 09825; 10-OR20 ; ; [Jolly, 2014, PP5_50554]
Silverton, Oregon, USA, 2010 (Lustrous fine fescue)	EC 125 (P) + COC	3 (12, 13)	0.28 0.28 0.29	0.11 0.11 0.11	vegetative, 15 cm high; 31 March	SiCL	forage	14	1.3, 1.5 mean = 1.4 [SS]	IR-4 PR 09825; 10-OR35 ; ; [Jolly, 2014, PP5_50554]
Plymouth, Washington, USA,	EC 125 (P)	3 (14, 14)	0.29 0.29 0.27	0.12 0.12 0.13	vegetative, 30 cm high; 01 May	SaL	forage	13	1.4, 1.1 mean = 1.2	IR-4 PR 09825; 12-WA*01 ; ;

GRASS FORAGE Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
2012 (Fienesto fescue)	+ NIS								[SS]	[Jolly, 2014, PP5_50554]
Patterson, Washington, USA, 2010 (Quatro sheeps fescue)	EC 125 (P) + NIS	3 (14, 15)	0.28 0.28 0.28	0.14 0.13 0.13	vegetative, 15-18 cm high; 28 May	SaL	forage	13	0.64, 0.77 mean=0.70 [SS]	IR-4 PR 09825; 10-WA16 ; ; [Jolly, 2014, PP5_50554]
Achthuizen; Netherlands; 1997; (red fescue: Mocassin)	EC 125 (P)	1	0.18	0.035	crop height 15-25 cm; 1 May	C	Fl	47	0.16	RJ2496B; NL10-97-H301 [Mason and Bouwman, 1998, PP5/0201]
idem	EC 125 (P)	1	0.34	0.068	crop height 15-25 cm; 1 May	C	Fl	47	0.43	idem
Dirksland; Netherlands; 1997; (red fescue: Koket)	EC 125 (P)	1	0.19	0.037	crop height 10-20 cm; 1 May	C	Fl	47	0.09	RJ2496B; NL10-97-H302 [Mason and Bouwman, 1998, PP5/0201]
idem	EC 125 (P)	1	0.36	0.072	crop height 10-20 cm; 1 May	C	Fl	47	0.15	idem

GSH: Fl = flowering 70-80 cm crop height

[SS] Sample size not reported and may be below the required 1 kg.

Additional trial information:

IR-4 09825. GLP. No unusual weather conditions. Plot size 118-141 m^2. Foliar spray using back pack sprayer. Spray volume 197-327 L/ha. Forage samples were harvested with sickle bar from at least 12 areas over the plot plot (weight of samples not reported). Storage at -22 °C for maximum of 999 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method MRID 40831305 with a valid LOQ of 0.02 mg/kg**. Concurrent recoveries were 70-92% (at 0.02-5.0 mg/kg). Control samples were < 0.02 mg/kg.

RJ2496B. GLP. Unusual weather conditions had no effect on crop health. Grass was sown in October 1995 (1.5 years before application). Foliar spray using an air pressurised knapsack sprayer.. Spray volume 500 L/ha. Grass leaves were sampled from at least 12 areas over the plot (2.1-2.5 kg grass). Storage at -11 °C for 60 days. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/01 with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average concurrent method recovery (100% at 0.1 mg/kg). Control samples < 0.01 mg/kg.

Grass hay

One cGAP for grass is available:

- cGAP from the Netherlands with 1 ×0.25 kg ai/ha with a PHI of 49 days.

Trials that could be matched to this GAP were summarized.

Table 252 lists trials conducted in the USA (2010, 2012) and Germany (1998) on red fescue (Festuca rubra) and fine fescue (Festuca rubra var trichopylla) grass. A broadcast foliar spray

application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 252.

Results marked with "[SS]" are not selected for derivation of the MRL, if according to cGAP.

[SS] indicates that the sample size was less than the required 0.5 kg grass hay.

Table 252 Supervised field trials on grass (hay), treated with a broadcast foliar fluazifop-butyl spray

GRASS HAY Location, Country, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Silverton, Oregon, USA, 2010 (LaCross fine fescue)	EC 125 (P) + COC	3 (12, 13)	0.29 0.29 0.30	0.15 0.15 0.15	vegetative, 15 cm high; 31 March	SiCL	hay	21	4.7, 4.5 Mean =4.6 [SS]	IR-4 PR 09825; 10-OR19 ; ; [Jolly, 2014, PP5_50554]
Silverton, Oregon, USA, 2010 (Shadow II fine fescue)	EC 125 (P) + COC	3 (12, 13)	0.28 0.28 0.29	0.086 0.086 0.086	vegetative, 15 cm high; 31 March	SiCL	hay	21	5.2, 4.5 mean =4.8 [SS]	IR-4 PR 09825; 10-OR20 ; ; [Jolly, 2014, PP5_50554]
Silverton, Oregon, USA, 2010 (Lustrous fine fescue)	EC 125 (P) + COC	3 (12, 13)	0.28 0.28 0.29	0.11 0.11 0.11	vegetative, 15 cm high; 31 March	SiCL	hay	21	4.4, 4.0 mean =4.2 [SS]	IR-4 PR 09825; 10-OR35 ; ; [Jolly, 2014, PP5_50554]
Plymouth, Washington, USA, 2012 (Fienesto fescue)	EC 125 (P) + NIS	3 (14, 14)	0.29 0.29 0.27	0.12 0.12 0.13	vegetative, 30 cm high; 01 May	SaL	hay	41	0.86 [a], 0.815 [a] mean =0.84 [SS]	IR-4 PR 09825; 12-WA*01 ; [Jolly, 2014, PP5_50554]
Patterson, Washington, USA, 2010 (Quatro sheeps fescue)	EC 125 (P) + NIS	3 (14, 15)	0.28 0.28 0.28	0.14 0.13 0.13	vegetative, 15-18 cm high; 28 May	SaL	hay	21	3.7, 3.0 mean = 3.3 [SS]	IR-4 PR 09825; 10-WA16 ; [Jolly, 2014, PP5_50554]
Radegast; Niedersachsen; Germany; 1998 (red fescue: Lifolia)	EC 125 (P)	1	0.19	0.094	BBCH 59; crop height 40-45 cm; 12 May	SaL	89	51	0.94	RJ2764B; RS-9815-B1 [Mason and Kappes, 1999, PP5/0203]
idem	EC 125 (P	1	0.38	0.19	BBCH 59; crop height 40-45 cm; 12 May	SaL	89	51	2.5	idem
Löwenberg; Brandenburg; Germany; 1998; (red fescue: Roland)	EC 125 (P)	1	0.19		BBCH 60; crop height 40-60 cm; 16 May	SaL	87	47	0.50	RJ2764B; RS-9815-K1; [Mason and Kappes, 1999, PP5/0203]
idem	EC 125 (P	1	0.38		BBCH 60; crop height 40-60 cm; 16 May	SaL	87	47	1.2	idem

BBCH 80-89 = ripening of seeds

[a] These residues are the average of two separate extractions and analyses

Additional trial information:

IR-4 PR 09825 GLP. No unusual weather conditions. Plot size 118-141 m^2. Foliar spray using back pack sprayer. Spray volume 197-327 L/ha. Hay samples were harvested at early seed head with sickle bar from at least 12 areas over the plot (weight of samples not reported). Samples were allowed to dry until moisture content of 10-20% (21-41 days). Storage at -22 °C for maximum of 989 days for hay. Samples were analysed for total fluazifop using **HPLC-MS/MS method MRID 40831305 with a valid LOQ of 0.02 mg/kg**. Method validation and concurrent recoveries ranged from 69-96% (at 0.02-5.0 mg/kg). Controls were < 0.02 mg/kg.

RJ2764B. GLP. No unusual weather conditions. Grass was sown in November 1994 or July 1996 (> 1 yr before application). Foliar spray using an air pressurised knapsack sprayer. Spray volume 200 L/ha. Hay was sampled from the field after seed harvest. Hay was taken from at least 30 areas over the plot (0.9-1.1 kg plot B1, 0.55-0.60 kg plot K1), which was reduced to >0.4 kg as laboratory sample. Storage at -18 °C for 147 days. Temperature reached -6 ºC for 2 days. This is considered to have no effect on the residue levels, since samples remained frozen at all times. Samples were analysed for total fluazifop using **HPLC-MS/MS method RAM 287/02, modification A with a valid LOQ of 0.01 mg/kg**. Samples were corrected for individual concurrent method recovery (95-103% at 0.01–0.1 mg/kg). Uncorrected results are not stated in the report. Control samples < 0.01–0.01 mg/kg. LOQ needs to be increased to 0.01/0.3 = 0.04 mg/kg. Since residue levels are higher, this has not impact on the study results.

By-products

Almond hulls

One cGAP for almonds is available:

- cGAP from France with 1 ×0.25 kg ai/ha and a PHI of 21 days for almonds, chestnuts, hazelnuts, macadamia nuts and walnuts

Trials that could be matched to this cGAP were summarized.

Table 253 lists trials conducted in the USA (1990). A weed directed spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 253. Residues of fluazifop found in the hull samples were probably the result of contact between the hulls and the treated ground cover under the trees.

Table 253 Supervised field trials on almonds (hulls), treated with fluazifop-butyl at the base of the trees

ALMOND HULL Location, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
College City, Colusa, CA, USA; 1990; (NonPareil)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	Hulls cracking; 03 Aug	SaL	NH	14	0.06	RR 92-041B; 17-CA-90-601; [Roper, 1992, PP5/0572]
Idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	Hulls cracking; 03 Aug	SaL	NH	14	0.07, 0.21 [b], 0.09 [b], 0.09 [b] mean 0.12 [a]	idem
College City, Colusa, CA, USA; 1990; (NonPareil)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	Hulls cracking; 03 Aug	SaL	NH	14	0.01	RR 92-041B; 17-CA-90-602; [Roper, 1992, PP5/0572]
Idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	Hulls cracking; 03 Aug	SaL	NH	14	0.01	idem

ALMOND HULL Location, year (Variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Durham, Butte, CA; USA; 1990 (Nonpareil)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	GS ns; 17 Aug	L	At hull split	14	< 0.01	RR 92-041B; 72-CA-90-603; [Roper, 1992, PP5/0572]
Idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	GS ns; 17 Aug	L	At hull split	14	0.01	idem
Ord Bent, Glenn, CA, USA; 1990 (Texas)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	GS ns; 31 Aug	L	At hull split	14	0.03	RR 92-041B; 72-CA-90-604; [Roper, 1992, PP5/0572]
idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	GS ns; 31 Aug	L	At hull split	14	0.03	idem
Lost Hills, Kern, CA; USA; 1990; (Nonpareil)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	GS ns; 30 Aug	SaL	MAT	14	0.03	RR 92-041B; 81-CA-90-605; [Roper, 1992, PP5/0572]
idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	GS ns; 30 Aug	SaL	MAT	14	0.06	idem
Lost Hills, Kern, CA, USA; 1990 (Carmel)	EC 120 (P) + 0.25% NIS	1	0.84	0.90	GS ns, 01 Oct	SiL	MAT	14	0.03	RR 92-041B; 81-CA-90-606; [Roper, 1992, PP5/0572]
idem	EC 120 (P) + 0.25% NIS	2 (21)	0.42 0.42	0.45 0.45	GS ns, 01 Oct	SiL	MAT	14	0.03	idem

[a] Results came from 4 replicate samples; the mean is taken for MRL derivation if according to cGAP

[b] Replicate analyses of the same sample.

Additional trial information:

RR 92-041B; GLP study. No unusual weather conditions. Application by ground sprayer. Spray volume 93.46 L/ha. Plot size 13-310 m^2, with 4-11 trees/plot, except in trial 605 with 3 trees/plot. Nuts were shaken from the tree mechanisally and picked by hand. One hulls (>1.5 kg) sample was taken per plot. Within 24 hours after sampling, samples were stored at <-20 °C for max 411 days. Samples were analysed for total fluazifop using **GC-MS method RR91-014B with a valid LOQ of 0.01 mg/kg**. Samples were not corrected for average method recoveries (73-91% at 0.01–0.5 mg/kg). Control samples < 0.01 mg/kg.

Cabbage wrapper leaves

Residue trials for head cabbages were summarized under the relevant section. No separate data were submitted on wrapper leaves alone.

Cotton gin trash

Two cGAPs for cotton are available:

- cGAP from the USA with 2 ×0.42 kg ai/ha and a PHI of 90 days
- cGAP from Brazil with 1 ×0.25 kg ai/ha and a PHI of 60 days

Trials that could be matched to these cGAPs were summarized.

Table 254 lists trials conducted in the USA (2008). A broadcast foliar spray application with fluazifop-P-butyl (R-enantiomer) was conducted under the conditions listed in Table 254.

Table 254 Supervised field trials on cotton (gin trash), treated with a broadcast foliar fluazifop-butyl spray

COTTON GIN TRASH Location, Country year, (variety)	Form (g ai/L)	No (interval in days)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop mg/kg	Report; Trial no [ref]
Proctor, AR, USA, 2008 (DG2215 B2RF)	EC 240 (P) + +0.25% NIS	2 (14)	0.42 0.42	0.47 0.46	Squaring 29 June, 2008	SiCL	MAT	90	0.025, 0.010 (mean = 0.018)	T002224-07, C24AR 081313; [Mazlo, 2009; PP5_50076]
Wharton, TX, USA, 2008 (DP 445/BG/RR)	EC 240 (P) + +0.25% NIS	2 (14)	0.42 0.42	0.21 0.22	BBCH 59-63; 30 June, 2008	SiL	MAT	90	0.087, 0.074 (mean=0.080)	T002224-07, W05TX 081315; [Mazlo, 2009; PP5_50076]
Uvalde, TX, USA, 2008 (DP434)	EC 240 (P) + +0.5% NIS	2 (14)	0.42 0.42	0.39 0.18	BBCH 59; June 6, 2008	CL	MAT	90	0.043, 0.043 (mean=0.043)	T002224-07, W07TX 081316; [Mazlo, 2009; PP5/50076]
Claude, TX, USA, 2008 (ST4554RF)	EC 240 (P) + +0.5% NIS	2 (14)	0.43 0.42	0.18 0.17	BBCH 66; 15 Aug, 2008	CL	MAT	90	0.63, 0.51 (mean=0.57)	T002224-07, E13TX 081317; [Mazlo, 2009; PP5_50076]
Levelland, TX, USA, 2008 (FM9063 B2F)	EC 240 (P) + 0.3–0.4% NIS	2 (14)	0.41 0.41	0.22 0.22	Bloom; 05 Aug, 2008	L	MAT	90	0.15, 0.18 (mean=0.16)	T002224-07, W39TX 081318; [Mazlo, 2009; PP5_50076]
Sanger, CA, USA, 2008 (PHY725RF Acala)	EC 240 (P) + +0.3% NIS	2 (14)	0.42 0.42	0.16 0.16	BBCH 65; 07 Aug, 2008	SaL	MAT	90	0.56, 0.70 (mean=0.63)	T002224-07, W31CA 081319; [Mazlo, 2009; PP5_50076]

Additional trial information:

T002224-01; GLP study. No unsual weather conditions. Tractor mounted or backpack ground sprayer. Spray volume 47.7-246 L/ha. Replicate samples of seed cotton were picked by mechanical picking (100 lbs, >45 kg) or by mechanical stripper (75 lbs, > 34 kg). Seed cotton samples were processed into commercially representative undelinted cottonseed and cotton by-product fractions (gin trash). Samples were stored frozen at <-23 °C for max 228 days. Samples were analysed for total fluazifop using HPLC-MS/MS method GRM044.01A. Samples were not corrected for average concurrent recoveries (99-110% 0.01-1.0 mg/kg). Control samples were < 0.01 mg/kg.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data on the fate of fluazifop-butyl derived residues during storage under warehouse conditions are available.

In processing

Fate of residues during processing

No studies were submitted on the fate of residues during processing.

Hydrolysis studies at ambient temperature (see physical-chemical section) indicated that fluazifop-P-butyl was stable at pH 5 but degraded at pH 7 (DT_{50} = 78 days) and pH 9 (DT_{50} = 29 hrs). The only degradation product was fluazifop acid (II). Hydrolysis studies at ambient temperature (see physical-chemical section) indicated that fluazifop acid (II) is stable at pH 5, 7 and 9. Hydrolysis studies with fluazifop acid (II) at elevated temperatures simulating boiling, pasteurisation and sterilisation were not submitted. However, the manufacturer indicated that:

"*Information on the hydrolytic behaviour of fluazifop*-P-*butyl under extreme conditions is available from the residue analytical method (SOP RAM 287/02), which employs an acid hydrolysis step in order to convert any fluazifop*-P-*butyl and conjugates of fluazifop acid (II) down to a common fluazifop acid (II) moiety, which is subsequently analysed. This conversion occurs in essentially quantitative yield, which indicates that fluazifop acid (II) is highly stable, even under extreme pH and temperature conditions. Therefore, the effect of simulated processing conditions on the residue of concern is well understood and no additional work is required*."

The plant metabolism section contains information on stability of fluazifop acid (II) under various hydrolysis conditions. Fluazifop acid (II) is stable after 1-3 hr reflux in 0.1 M HCl or 0.1 M NaOH, which reflect more stringent conditions than normally met during cooking, pasteurisation or sterilisation.

Processing of oranges

Processing study 1

A field trial with 13 trees per plot was conducted in Florida, USA, 1986 (trial 75FL86-929) to provide sufficient orange samples for processing and analysis [Francis, 1989, PP5/0586, RR 89-052B]. Oranges (variety Hamilin) were treated with 3 ×4.2 kg ai/ha fluazifop-P-butyl (EC formulation). The PHI was 14 days. Treatment was applied as a directed spray to the ground beneath and around each tree. Interval between the treatments was approximately 21 days. Approximately 408 kg of mature oranges were harvested from the untreated plot and the treated plot. Samples were stored at 5 °C until processing. Samples were processed one week after harvest. The processing procedures were representative of typical commercial practices.

Orange juice: Oranges were washed and the juice extracted with a commercial in-line juice extractor. The extracted juice was then passed through a finisher to remove the pulp and the resulting juice was collected in a cold wall cooling tank. The juice was sampled, placed in cans and stored frozen at -18 °C.

Finisher pulp: A sample of the finisher pulp was taken, placed in a cambric mailing bag with an inner polyethylene coating and liner and stored at -18 °C.

Oil: The oil/water/peel-frit emulsion from the juice extractor was passed through a modified finisher and the emulsion collected in a stainless steel tank. The emulsion was allowed to stand for a minimum of 5 hours before draining off the lower water phase. The concentrated oil emulsion was stored at 0 °C until further processed. The concentrated oil emulsion was passed through a vibrating screen and then centrifuged. The highly concentrated oil emulsion was the stored at <-18 °C for at

least 16 hours. After thawing the cold pressed oil was filtered. A sample of oil was taken and placed in a glass jar and stored at 5 °C.

Molasses: The peel-membrane-seed fraction from the commercial in line juice extractor and from the modified finisher were transferred to a pilot plant feed mill. As the peel left the receiving hopper, dehydrated lime as a liquid slurry was added. The limed-chopped-reacted peel was passed through a continuous press to produce the press liquor. The press liquor was heated to boiling under vacuum and concentrated in a concentrator to 68–72°Brix for final molasses. The molasses was sampled, sealed in cans and stored at <-18 °C.

Dried pulp: The citrus peel was dried in triple-pass, direct-fired drier to produce a dried citrus pulp of approximately 8% moisture content. The dried citrus pulp was sampled, placed in double layered 25 lb Kraft bags and stored at -18 °C.

From 389 kg of oranges, 151 kg of chopped peel, 203 kg of juice and 0.47 kg of oil were produced.

All samples were stored for up to 23 months at <-18 °C prior to analysis; storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using NMR method PPRAM 83 modification A or B with a valid LOQ of 0.05 mg/kg. This method is considered valid for the determination of total fluazifop in orange (at 0.05 mg/kg only). Average concurrent recoveries were 70–94% (at 0.05 mg/kg) in orange and its processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.03 mg/kg).

The residue levels and processing factors are summarized in Table 255. A residue of 0.016 mg/kg was detected in oranges that had been treated with 3 ×4.2 kg ai/ha, but this is below the valid LOQ (< 0.05 mg/kg). Fluazifop is concentrated in the peel, dried pulp, molasses, orange oil, but not in juice. However, since residues in the RAC were <LOQ no reliable processing factors can be determined.

Processing study 2

Two crop field trials were initiated in the USA on orange trees to determine the magnitude of fluazifop-butyl residues in processed commodities (juice, dried pulp, and oil) of citrus fruit (oranges) [Mazlo, 2013, A12460A_50019, report TK0058357]. Each plot was treated three times with 2.1 kg ai/ha (5× label rate) EC formulation as a soil directed broadcast spray application under the canopy and within the dripline of the canopy and with an interval of 21 days. One bulk sample was collected from each treated (190–215 kg) and untreated (204–209 kg) plot at PHI 14 days.

Oranges were removed from storage and weighed. Representative unwashed fruit samples fractions were removed, packaged, labeled, and placed in frozen storage (RAC). The remaining fruit was hand inspected for undesirable fruit and filed debris, which was discarded.

Washing: The oranges were batch tub washed for 5 minutes. The washed oranges were transferred to the modified Hobart Abrasive Peeler for scarifying. An average of 2.4 kg of oranges per batch was abraded for 90–150 seconds to scarify the flavedo for oil recovery. The scarified fruit was weighed and retained for juice processing.

Oil processing*:* The collected oil-water emulsion was transferred to the Sweco Sifter and screened using a 180 µm screen (94TBC screen) to separate any flavedo fragments from the oil-water emulsion. The scarified flavedo was set aside for alter addition to the shredded peel. The first-run oil-water emulsion was processed through the cream separator and IEC centrifuge to separate the oil. The free oil was removed and measured with a 500 mL volumetric pipette and frozen. The residual emulsion was frozen overnight, thawed, centrifuged, and the oil removed. This oil was added to the oil collected the rpvious day. The entire collected combined sampl of the oil revcovered from both processing days was weighed, packaged, labeled and placed in frozen storage for analysis.

Juice processing*:* An aliquot of the scarified oranges was weighed and transferred to the Hollymatic Juice extractor to recover the juice from the peel. The juice and the peel recovered were

weighed and Brix taken on the fresh juice. The collected juice was transferred to the pulper finisher and screened using a 1.19 mm screen to remove vesicular membranes, seeds, segment membranes, and peel fragments from the juice. The collected rag and seeds were set aside for later addition to the shredded peel. A representative sample of the fresh juice was removed, packaged, labeled and placed in frozen storage for analyses.

Pulp processing*:* The peel of the Hollymatic Juice Extractor was shredded using the Robot Coupe Food Processor. The shredded peel was combined with the sacrified flavedo from the scarification process and rag and seeds from the juice finisher extraction process to generate wet peel. Lime (95% CaO) was added to the wet peel and mixed on the Hobert mixer for 17 minutes. pH was adjusted to at least pH 8 by addition of lime and water and mixing again. The limed peel was pressed using the Suntech Fruit press and the expressed liquid weighed, checked for pH and Bris and discarded.

The wet peel pulp was placed on the Laboratory Bin Air Dryer and dried to below 10% moisture. The dried pulp was milled using the Suntech Fruit Press hammermill. A representative sample of the dreid pulp was removed, packaged, labeled and placed in frozen storage for analysis. The remaining dried pulp was discarded.

For oil, 199/177 kg of oranges (before washing) via 227/209 kg after scarification, resulted in 39 g orange oil. For juice, 33 kg oranges (194/176 kg discarded) produced 9.2/7.2 kg fresh juice and 24/25 kg peel; 24/25 kg peel resulted in 10/9.3 kg limed wet pulp, which subsequently resulted in 2.3/2.1 kg dried pulp (after milling) for untreated/treated samples from one location, respectively.

For oil 198 kg oranges (before washing) via 206/205 kg after scarification, resulted in 60/139 g oil. For juice, 33/30 kg oranges (172/175 kg discarded) produced 11/10 kg fresh juice and 21/19 kg peel; 21/19 kg peel resulted in 12/9.8 kg limed wet pulp, which subsequently resulted in 3.4/2.5 kg dried pulp (after milling) for untreated/treated samples from one location, respectively.

Samples were stored frozen for a maximum 9.7 months; storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using HPLC-MS/MS method GRM44.01A with modifications with a valid LOQ of 0.01 mg/kg for orange commodities. The method is considered valid for the determination of total fluazifop in orange juice (0.01–1.0 mg/kg), orange oil (0.01–1.0 mg/kg) and orange dried pulp (0.01–1.0 mg/kg). Average concurrent method recoveries were 91–113% (0.01–1.0 mg/kg) in orange and its processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg). The residue levels and processing factors are summarized in Table 255.

Table 255 Residue levels of fluazifop-butyl in orange processed commodities

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Commodity	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
Wauchula, FL, USA, 1986, (var Hamilin) EC 120 (P) + 1% v/v COC; 3 ×4.2 kg ai/ha, interval 21 days, DALT = 14 days, GS: 2.9 - 3.2 inch diameter, 11 Nov 1986 Soil: not stated	Orange (RAC) Chopped peel Dried pulp Molasses Orange juice Orange oil Finisher pulp	< 0.03 1.7 2.6 1.1 < 0.03 0.09 < 0.03 [LOQ=0.05]	-	RR 89-052B 75FL86-929 [Francis, 1989, PP5/0586]
Chuluota, FL, USA, 2011, (Hamilin) EC 240 (P) + COC 0.75%, 3 × 2.1 kg ai/ha, interval 21 days, DALT = 14 days, GS: BBCH 81, 23 Oct, 2011 Soil: Sand	Whole fruit	< 0.01, < 0.01 (mean: < 0.01) [a]	-	TK0058357, TK0058357-01 [Mazlo, 2013, A12460A_50019]
	Juice	< 0.01, < 0.01 (mean: < 0.01) [a]	-	
	Oil	< 0.01, 0.0124 (mean: 0.011) [a]	-	
	Dried pulp	0.0433, 0.0453	-	

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Commodity	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
		(mean: 0.044) [a]		
Porterville, CA, USA, 2011, (Valencia) EC 240 (P) +COC 0.75%; 3 × 2.1 kg ai/ha, interval 21 days; DALT: 14 days GS: BBCH 89, 03 Aug, 2011 Soil: Sandy clay loam	Whole fruit	0.0141, 0.0155, 0.0149 (mean: 0.015) [a]	-	TK0058357, TK0058357-02 [Mazlo, 2013, A12460A_50019]
	Juice	< 0.01, < 0.01 (mean: < 0.01) [a]	< 0.7	
	Oil	0.0788, 0.0700 (mean: 0.074) [a]	5.0	
	Dried pulp	0.0865, 0.0918 (mean: 0.089) [a]	6.0	

[a] Results are the mean of two replicate samples taken from the processed commodities

Processing of apple

Processing study 3

In a non-GLP study, apple trees were sprayed around the base with a single application of 0.5 kg ai/ha fluazifop-P-butyl (EC formulation) in 1991 in Germany [Gardyan, 1992, PP5/0183, report AZ84661A/91]. Further details on the field part were not reported. Apples were processed into apple sauce, apple juiceandpomace and dried apples. Since the treated apples (RAC) contained no residues (< 0.02 mg/kg total fluazifop) the processed products were not analysed and no processing factors could be derived from this study. Processing details were therefore not summarized.

Processing of cherries

Processing study 4

In a non-GLP study, cherry trees were sprayed around the base with a single application of 0.5 kg ai/ha fluazifop-P-butyl (EC formulation) in 1991 in Germany [Gardyan, 1992, PP5/0192, report AZ83558/91]. Further details on the field part were not reported. Cherries were processed into cherry jam, preserves, and juice. Since the treated cherries (RAC) contained no residues (< 0.02 mg/kg total fluazifop) the processed products were not analysed and no processing factors could be derived from this study. Processing details were therefore not summarized.

Processing of plums

Processing study 5

In a non-GLP study, plum trees were treated around the base with an EC formulation of fluazifop-P-butyl at 3 x 0.42 kg ai/ha (Washington, USA, 1986) or 3 × 2.1 kg ai/ha (California, USA, 1986) [Watford and Francis, 1987, PP5/0480, report TMU3311/B]. Details of the trials are summarized in the section residues resulting from supervised residue trials (trials 32WA86-910R and 45CA86-910R). Plums were harvested at commercial harvest at PHI 14–15 days. Plum samples were processed into dried plums (prunes). Processing details are not available.

Plums and prunes were stored for a maximum of 8 months at -20 °C. Storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using NMR method PPRAM 83, with a valid LOQ of 0.05 mg/kg for fruits. The method is considered insufficiently validated for the determination of total fluazifop in plums. Average concurrent method recoveries were 113–124% (0.05–0.1 mg/kg) in plums, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.02 mg/kg).

Since both the treated plums (RAC) and the dried plums contained no residues (< 0.02 mg/kg total fluazifop), no processing factors could be derived from this study.

Processing study 6

In a non-GLP study, plum trees were sprayed around the base with a single application of 0.5 kg ai/ha fluazifop-P-butyl (EC formulation) in 1991 in Germany [Gardyan, 1992, PP5/0192, report AZ83558/91]. Further details on the field part were not reported. Plums were processed into plum jam. Since the treated plums (RAC) contained no residues (< 0.02 mg/kg total fluazifop), the processed products were not analysed and no processing factors could be derived from this study. Processing details were therefore not summarized.

Processing of grapes

Processing study 7

Grape vines were treated with a weed directed spray of fluazifop-P-butyl (EC formulation) at the base of the vines at an application rate of 2 ×0.42 kg ai/ha in Visalia, CA, USA in 1984 [Watford and Francis, 1987, PP5/1113, TMU3330/B]. Details of the trials are summarized in the section residues resulting from supervised residue trials (trial US02-84-S08). Grapes were dried for an unspecified period to get 1.1 kg raisins of unspecified moisture content.

Grapes and raisins were stored at -20 ºC for a maximum period of 16 months until analysis. Storage stability is considered not an issue for total fluazifop. The products were analysed for total fluazifop using HPLC-UV method PPRAM 62/2 at 230 nm with a valid LOQ of 0.05 mg/kg. This method is considered valid for the determination of total fluazifop in grapes (0.05–0.5 mg/kg). Average concurrent method recoveries were 85–92% (0.025–0.1 mg/kg) in grapes, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in control samples (< 0.03 mg/kg).

Residues in grapes (RAC) were < 0.03 mg/kg total fluazifop, and also the raisins had residues < 0.03 mg/kg total fluazifop. No processing factors can be derived from this study.

Processing study 8

In a non-GLP study, a grape vine was treated at the base with an EC formulation of ^{14}C-labelled fluazifop-P-butyl (50:50 mixture of phenyl- and pyridyl-labelled compound) in the USA in 1986 [French and Leahey, 1987, PP5/0082, report RJ0569B]. For further details, see the metabolism section. The application was intended to consist of two treatments, each at a targeted rate of 0.84 kg ai/ha. After the first application at early bunch formation, analysis of the spray mix showed that in the first application approximately 20% less active ingredient had been applied. This shortfall was made up on day 42 with a supplementary application. The final (third) application was made 71 days after the first application (growth stage not stated). Analysis showed that for the third treatment 91% of the required rate of 0.84 kg ai/ha had been achieved. A total of 1.598 kg ai/ha had been applied, approximating 2 × 0.84 kg ai/ha. Mature grapes (500 g) were harvested 14 days after the final treatment and grapes were processed into juice and wet pomace. For this purpose the grapes were de-stalked and pressed into juice and wet pomace using a winepress for home wine making. The pomace (114 g) was allowed to dry (74 g). Samples were stored at -15 ± 5 °C until analysis (maximum of 8 months). Total radioactivity was determined by LSC.

The distribution of the total radioactive residues was:

Radioactive residue in grapes:	0.007 mg/kg eq
Radioactive residue in grape juice:	0.006 mg/kg eq
Radioactive residue in grape dried pomace:	0.013 mg/kg eq

Since a mass balance was not given in the study report, the recovery of radioactivity is unknown. Composition of the radioactive residues was not further investigated and therefore no processing factors can be derived from this study.

Processing study 9

A processing trial was carried out on grapes (variety Thompson Seedless) in California, USA in 2000 [Stewart, 2001, 406498, report RR 00-067B]. Three applications of fluazifop-P-butyl (EC formulation) were applied broadcast to the vineyard floor at an exaggerated rate of 2.11 kg ai/ha with a spray interval of 14 days. Details of the trials are summarized in the section residues resulting from supervised residue trials (trial 197GRCA1). Grapes were harvested at normal harvest, 50 days after the last application. Samples were kept below 8°C until processing, which started maximally 2 weeks later. The samples were processed into grape juice and raisins.

Grape juice (cold press): Fresh grapes were hand fed into a Crusher/Stemmer. The grape pulp was collected. The stems were discarded. The pulp was pressed to separate the juice and pulp. The wet pomace was discarded. The fresh juice was filtered. Filtered fresh juice was sampled.

Raisins: The grapes were distributed into drying trays and dried at a temperature of 60-74°C. An aliquot of raisins were sorted to remove cap stems, panicles and undesirable raisins. Sorted raisins were washed with cold water. A representative sample of raisins was collected.

From the treated plot, 45.2 kg of grapes was available, of which 20.4 kg was taken for juice processing. There was 13.2 kg fresh juice recovered from the press. For raisins, 21.8 kg grapes was available, of which 21 kg was used for processing. 5.5 kg raisins were recovered after the drying process.

After processing and homogenization samples were stored at -15°C or lower for 33 days prior to analysis. Storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using GC-MS Method RR91-014B with a valid LOQ of 0.01 mg/kg. The method is considered valid for the determination of total fluazifop in grapes (0.01-1.0 mg/kg) and grape raisins (0.01–0.1 mg/kg). Average concurrent method recoveries were 84-99% (0.01-1.0 mg/kg) in grapes and its processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

All samples (grapes, grape juice and raisins) had residues below LOQ (< 0.01 mg/kg total fluazifop). Therefore, no reliable processing factors could be determined.

Processing of brassica vegetables and leafy brassicas

Processing study 10

Brassica were treated in the field with a broadcast foliar spray of fluazifop-P-butyl at an application rate of 1 ×0.19 kg ai/ha (kale, Savoy cabbage, Brussels sprouts) or 1 ×0.20 kg ai/ha (cauliflower) in 1991 in Germany [Gardyan, 1992, PP5/0129, Report AZ83592/91]. Further details on the field part were not reported. Brassica (2–3 kg each) from these sites were processed (directly after harvest) by household methods.

Washing: Stalks were removed from the kale; the outside leaves were removed from the cauliflower, withered leaves and stalks were removed from the Savoy cabbage. The remaining part of the cabbage was cut and washed. Brussels sprouts were washed as they were. Brassica were divided for cooking and canning.

Cooking: Washed and cut kale, cauliflower, Savoy cabbage or Brussels sprouts (2–3 kg each) were cooked for 20 min in 4+–5 L water with 20 g salt. The cooked cabbage was allowed to drain and the cooking liquid was kept separately.

Canning: Washed and cut kale (2 kg) was cooked for 10 min in 4-5 water with 20 g salt. The cabbage was tightly filled into jars together with salted water. The sealed jars were heated for 80 min at 100 ºC.

Brassica (RAC) samples and processed samples were stored homogenised at –20ºC for a maximum period of 330 days until analysis. Storage stability is considered not an issue for total fluazifop. The products were analysed for total fluazifop using HPLC-UV method PPRAM 62/2. A second HPLC clean-up was used and analysis was without internal standard. The valid LOQ is 0.05 mg/kg for most commodities, but 0.1 mg/kg for head cabbage. This method is considered insufficiently validated for the determination of total fluazifop in kale, cauliflower, head cabbage and Brussels sprouts and their processed commodities. Average concurrent method recoveries were 70-114% (0.05-5.0 mg/kg) in kale, cauliflower, head cabbage and Brussels sprouts, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.05 mg/kg), except in Savoy cabbage (0.06 mg/kg).

The results are presented in Table 256. Since residues in cauliflower (RAC) were < 0.05 mg/kg total fluazifop, processed commodities were not analysed and no processing factors could be derived for cauliflower. Cooking and canning of kale, Savoy cabbage and Brussels sprouts reduced the total fluazifop residues while part of the residue remained in the cooking liquid. Processing factors from trials with high control values are not taken into account.

Table 256 Residues and processing factors for brassica commodities treated with fluazifop-P-butyl

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Commodity	Total fluazifop mg/kg	P-factor	Report no Trial no [Reference]
Location ns; Germany, 1991 (variety ns) EC 125 (P) 1 × 0.19 kg ai/ha; DALT 42 days GS: ns Soil type: ns	Brussels sprouts (RAC) cooked (household) cooking liquid	3.28 1.11 0.82	- 0.34 -	AZ83592/91; 91HJ068Ext 2, [Gardyan, 1992, PP5/0129]
Location ns; Germany, 1991 (variety ns) EC 125 (P) 1 × 0.19 kg ai/ha; DALT 47 days GS: ns Soil type: ns	kale (RAC) cooked (household) cooking liquid canned (household)	0.95 0.19 0.13 0.28	- 0.20 - 0.29	AZ83592/91; 91HJ068B1, [Gardyan, 1992, PP5/0129]
Location ns; Germany, 1991 (variety ns) EC 125 (P) 1 × 0.19 kg ai/ha; DALT 41 days GS: ns Soil type: ns	Savoy cabbage (RAC) cooked (household) cooking liquid	0.18 0.05 0.08 [cntrl = 0.06]	- 0.28 [a] -	AZ83592/91; 91HJ068Ext 1, [Gardyan, 1992, PP5/0129]

[a] untreated RAC sample contained 0.06 mg/kg residue, no processing factor can be derived from this study.

Additional trial information:

AZ83592/91. GLP. Weather, spray equipment and soil type were not reported. Spray volume not stated. Samples contained 2-3 kg brassica. Growth stage at harvest not stated. Storage conditions not stated. HPLC-UV method **PPRAM 62/2 with additional clean-up with column chromatography**. Residues not corrected for concurrent method recoveries (70-114% at 0.05-5.0 mg/kg). Control samples were < 0.05 mg/kg, except in Savoy cabbage (0.06 mg/kg).

Processing of tomatoes

No processing studies were submitted for tomato. Such studies are desirable, since tomato wet pomace is an animal feed item.

Processing of green pea seeds

Processing study 11

Two residue trials on peas were conducted in Germany and the United Kingdom in 2010 [Langridge, 2013, A12791B_11029, report CEMR-4751-REG]. Fluazifop-P-butyl was applied as an EC 125 formulation at 1 × 0.76 kg ai/ha or 1 ×1.8 kg ai/ha at 37 or 32 days before harvest. Details of the trials are summarized in the section residues resulting from supervised residue trials (trials CEMS-4751-02 and CEMS-4751-03). At normal commercial harvest for fresh green pea seeds, samples (17.5–35 kg) of treated and untreated pea seeds were harvested by hand and transported chilled to the processing facility. Green pea seeds were processed into canned and cooked peas according to relevant industrial practices and standardised procedures. One balance study and one follow-up study were carried out on each trial.

Cooked peas*:* During cooked pea production, unwashed peas (2.538 kg) without pods were thoroughly washed with water, wash water recovered (1.094 kg). For the balance studies, washed pea sub-samples (0.504 kg) and washing water (1.094 kg) were sub-sampled and analysed. The washed peas (2.548 kg) were put in boiling (100 °C) tap water (3.074 kg) for about 8 minutes. An amount of 1.294 kg peas were retrieved after cooking. For all studies cooked pea sub-samples (0.506 kg) were analysed, whilst a sub-sample (0.525 kg) of the recovered cooking water (2.462 kg) was analysed in the balance study only.

Canned peas*:* During canned pea production, peas without pods (5.014 kg) were thoroughly washed with water (2.506 kg), resulting in 5.0 kg washed peas en 2.406 kg wash water. For all processing studies washed pea sub-samples (0.508 kg) were analysed, whilst a subsample of the washing water (0.500 kg) was analysed in the balance study only. The washed peas (3.99 kg) were blanched in boiling (100 °C) tap water (7.98 kg) for about 1 minute. For the balance studies subsamples of blanched peas (0.500 kg) and blanching water (7.32 kg recovered, 0.522 kg sub samples) were analysed. The blanched peas (2.00 kg) were then canned and a solution of brine tapwater (1 kg), table salt and citric acid to adjust to pH 3.5) was added. A total of 1.472 kg of blanched peas was not used for canning. The cans were sealed and then sterilised for 10 minutes at 115–120 °C. Canned peas in brine afer sterilation were subsampled (0.750 kg), leaving 2.25 kg canned peas in brine in the rest of the sample.

Samples were stored frozen (≤-18 °C) for a maximum period of 13 months from sampling to analysis. Extract solutions were stored for a maximum of 14 days before analysis. Storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop with HPLC-MS/MS method GRM44.02A with a valid LOQ of 0.01 mg/kg. The method is considered valid for the determination of total fluazifop in green pea seeds (0.01–1.0 mg/kg) and green pea seeds blanced, cooked or canned (0.01–0.1 mg/kg). Average concurrent method recoveries were 75–94% (0.01–0.5 mg/kg) green pea seeds and its processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. Positive control residues were seen in the untreated samples of the balance trial of CEMS-4751-03 for fresh pea seeds (0.21–0.29 mg/kg), washed pea seeds (0.28–0.32 mg/kg), cooked pea seeds (0.24 mg/kg), canned pea seeds (0.19–0.21 mg/kg) and blanched pea seeds (0.34 mg/kg). In trial CEMS-4751-02 control levels were < 0.01 mg/kg, except for one sample of canned peas after sterilization (0.19–0.23 mg/kg).

Residue results and processing factors are shown in Table 257 respectively. Processing factors from trials with high control values are not taken into account.

Table 257 Residue levels of total fluazifop in processed pea commodities

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Pea commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
Lueneburg, Germany, (var Maxigold) EC 125 (P), 1 ×0.76 kg ai/ha	Fresh pea seeds	0.83, 0.86 (mean= 0.845) [a]	[b]	CEMR-4751-REG, CEMS-4751-03

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Pea commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
DALT = 37 days, GS: BBCH 35-36, 10 Aug, 2010 Soil: Sandy		[cntrl = 0.21]		[Langridge, 2013, A12791B_11029] (balance)
	Washed peas	0.77 [cntrl = 0.32]	0.91 [b]	
	Blanched peas	1.07 [cntrl = 0.34]	1.3 [b]	
	Canned non-sterile peas	0.81 [cntrl = 0.19]	0.96 [b]	
	Canned sterilised peas	0.79 [cntrl = 0.21]	0.93 [b]	
idem	Fresh pea seeds	0.88, 0.82 (mean = 0.85) [a]	-	idem (follow up)
	Canned sterilised peas	0.69	0.81	
idem	Fresh pea seeds	0.73, 0.78 (mean 0.755) [a] [cntrl = 0.29]	[b]	idem (balance)
	Washed peas	0.75 [cntrl = 0.28]	0.99 [b]	
	Cooked peas	0.92 [cntrl = 0.24]	1.2 [b]	
idem	Fresh pea seeds	0.95, 1.0 (mean 0.975) [a]	-	idem (follow up)
	Washed peas	0.74	0.76	
	Cooked peas	0.92	0.94	
Stratford upon Avon, Warwickshire, United Kingdom, (var Samish) EC 125 g ai/L, 1 ×1.8 kg ai/ha, DALT = 32 days, GS: BBCH 38-39; 03 June 2010 Soil: Silty loam	Fresh pea seeds	2.44, 2.57 (mean= 2.50) [a]	-	CEMR-4751-REG, CEMS-4751-02 [Langridge, 2013, A12791B_11029] (balance)
	Washed peas	2.41	0.96	
	Blanched peas	1.97	0.79	
	Canned non-sterile peas	1.01	0.40	
	Canned sterilised peas	1.44 [cntrl 0.21]	0.58	
idem	Fresh pea seeds	2.87, 1.56 (mean = 2.22) [a]	-	idem (follow up)
	Canned peas	1.58	0.71	
idem	Fresh pea seeds	2.66, 1.92 (mean = 2.29) [a]	-	idem (balance)
	Washed peas	2.43	1.1	
	Cooked peas	1.97	0.86	
idem	Fresh pea seeds	1.59, 2.59 (mean =2.09) [a]	-	idem (follow up)
	Washed peas	1.97	0.94	
	Cooked peas	1.74	0.83	

Cntrl Control samples contained 0.21–0.34 mg/kg total fluazifop.

[a] Results are the mean of two replicate samples taken from the processed commodities

[b] Processing factors not taken into account, since significant residues were found in the control samples

Processing of dry harvested peas

Processing study 12

Two residue trials on peas without pods were conducted in Northern France and Germany in 2011 [Devine, 2013, A12791B_11068, report CEMR-5037-REG]. Fluazifop-P-butyl was applied as a EC 125 at 1 ×1.9–2.0 kg ai/ha with harvest at 63 or 79 days days after treatment. Details of the trials are summarized in the section residues resulting from supervised residue trials (trials CEMS-5037-01 and CEMS-5037-02). At normal commercial harvest (BBCH 89) samples (5–10 kg) of treated and untreated dried peas were harvested transported chilled to the processing facility in France (Staphyt

Processing). Dried peas were processed into canned and cooked peas according to relevant industrial practices and standardised procedures. One balance study and one follow-up study were carried out on each trial. Sub samples (0.5–0.75 kg) of the (processed) seeds were analysed for total fluazifop.

Initial preparation*:* The peas (4.04/7.51 kg, MB trial 01 and 8.44/7.56 kg MB trial 02) were cleaned with a rubber roller peeler. Samples "cleaned seed" and "impurities (husks)" were taken. The cleaned seed (6.32 kg MB trial 01 and 7.28/6.93 kg MB trial 02) was then steeped in cold tap water overnight under ambient conditions (ratio seed/water 0.66–0.67), resulting in 11.06 kg and 14.07/13.75 kg steeped seed in MB trials 01 and 02, respectively. Samples "steeping water" and "steeped seed" were taken. Before washing the treated samples were divided into 2 parts: one for mass balance and one for follow-up. The peas (2.99/10.50 kg and 12.69/12.86 kg, respecivelt) were washed in tap water (ratio 0.75, 1.15 and 0.81, respectively), and samples "washing water" and "washed seed" were taken. The washed peas (3.13/10.73 kg and 10.72/9.83 kg, respectively) were again separated into two parts, one for cooking and the other for canning.

Cooked peas*:* The washed peas (0.66/1.45 kg and 1.52/1.54 kg, respectively) were added to boiling water at a ratio of 0.24/ 0.62 and 0.61/0.58, respectively for both trials. The peas were cooked for 27–68 minutes at 99.5–101 °C until they were soft and boiled. The samples "cooking water" and "cooked seeds" were taken. The weights of the cooked peas were 1.26/1.70 and 1.69/1.70 kg, respectively.

Canned peas*:* The washed peas 1.46/2.38 and 2.71/2.58 kg were blanched in boiling water at a ratio of 0.97/1.22 and 1.34/1.46, respectively, resulting in batches of 1.61/2.35 and 2.71/2.57 kg, respectively. The samples "blanching water" and "blanched seed" were taken. Batches of blanched peas (0.33/0.40 and 0.62/0.41 kg, respectively) were filled into containers and were covered, respectively with 0.30/1.15 and 0.84/1.14 kg brine (brine = 1.5% NaCl and 0.05% citric acid). A second set was prepared to generate samples of canned seeds with brine separated. For this set batches of blanched peas (0.74/0.62 and 1.29/0.37 kg, respectively) were filled into containers and were covered, respectively with 0.67/2.89 and 1.63/1.12 kg brine (brine = 1.5% NaCl and 0.05% citric acid). The containers were transferred to an autoclave for sterilisation at 123.4–126 °C for 10–12.5 minutes. The samples "canned seed" were taken. Total weights of the canned seeds were 0.63/1.54 and 1.23/1.55 kg for the canned samples including brine. Total weights before separation of the brine of canned seeds were 1.41/3.51 and 2.39/1.45 kg in the MB trials, respectively. After sterilisation the brine was separated from the peas and the samples "brine" and canned seed (excl brine)" were taken. The weights of the canned seeds with brine separated were 1.39/0.86 and 2.39*/0.55 kg, respectively. In one sample*) separation was not possible, because the brine was soaked up from the seed.

Samples were stored frozen (≤-18 °C) for a maximum period of 24 months from sampling to analysis. Storage stability is considered not an issue for total fluazifop. The samples were analysed for total fluazifop using HPLC-MS/MS method GRM044.02A with a valid LOQ of 0.01 mg/kg. The method is considered valid for the determination of total fluazifop in dry pea seeds (0.01–5.0 mg/kg), dry pea seeds cooked, steeped, blanched or canned (0.01–0.5 mg/kg). Average concurrent method recoveries were 71–121% (0.01–5.0 mg/kg) in dry peas and its processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg). Residue results and processing factors are shown in Table 258.

This study is considered not acceptable. Steeping and cooking leads to an increase in volume (and weight) of the peas and this should lead to a decrease in concentration level. If the residue is stable, the processing factor can maximally be equal to this dilution factor (1/1.85=0.54 for steeping and 1/2.5=0.4 for cooking). However, in this study, steeping and cooking leads to an increase in residue concentration and processing factors >1. Based on absolute values, the total residues seem to increase up to 2.4–3.4 times, which is not realistic.

A possible explanation for this apparent increase in residue after steeping could be that the pulses were not analysed properly. The analytical method requires that the pulses are soaked for a minimum of 2 hours in 1 M HCl or overnight in water before being extracted and hydrolysed. Steeping and cooking involves overnight soaking of the pulses. This could indicate that raw pulses

(RAC) were not soaked before extraction. Since this and other study reports do not indicate whether and how long the pulses have actually been soaked before extraction and hydrolysis, this hypothesis could not be confirmed. However, for acceptance of residue values and processing factors in pulses, the soaking period needs to be reported.

In trial CEMS 5037-01 plants were fallen on the ground, were only 10–15 cm high and crop yield was lower than in the control plot, due to phytotoxicity. These seeds do not comply with commercial standards.

Table 258 Residue levels of total fluazifop in processed dried pea commodities

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Pea commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
Beaumetz les Cambrai, Nord Pas-de-Calais, Northern France (var Pactole) EC 125 g ai/L, 1 ×2.0 kg ai/ha, DALT = 63 days, GS: BBCH 39, 09 May, 2011 Soil: Silt loam	Dried seed (RAC)	3.65, 3.65 (mean 3.65) [WC]	-	CEMR-5037-REG, CEMS-5037-01 [Devine, 2013, A12791B_11068] (balance)
	Cleaned seed	3.74, 3.72 (mean 3.73) [b]	1.0 [c d]	
	Steeped seed	5.04 [a]	1.4 [c d]	
	Washed seed	5.68 [a], 4.94 [a] (mean 5.31) [b]	1.5 [c d]	
	Blanched seed	4.45	1.2 [c d]	
	Canned seed (incl brine)	1.25	0.34 [c d]	
	Brine	0.56	-	
	Canned seed (excl brine)	1.18	0.32 [c d]	
	Cooked seed	2.74	0.75 [c d]	
idem	Dried seed (RAC)	3.65 [WC]	-	idem (follow-up)
	Canned seed (incl brine)	1.60	0.44 [c d]	
	Cooked seed	2.75	0.75 [c d]	
Baden-Württemberg, Germany (var Crackerjack) EC 125 g ai/L, 1 ×1.9 kg ai/ha, DALT = 79 days, GS: BBCH 33-34; 31 May 2011 Soil: Clay loam	Dried seed (RAC)	0.68, 0.63 (mean = 0.655)	-	CEMR-5037-REG, CEMS-5037-01 [Devine, 2013, A12791B_11068] (balance)
	Cleaned seed	0.66, 0.67 (mean = 0.665)	1.0 [d]	
	Steeped seed	1.15 [a]	1.8 [d]	
	Washed seed	1.37 [a], 1.46 [a] (mean 1.415) [b]	2.1 [d]	
	Blanched seed	1.11	1.7 [d]	
	Canned seed (incl brine)	0.46	0.70 [d]	
	Brine	0.15	-	
	Canned seed (excl brine)	0.38	0.58 [d]	
	Cooked seed	0.56	0.85 [d]	
idem	Dried seed (RAC)	0.67	-	idem (follow-up)
	Canned seed (incl brine)	0.22	0.33 [d]	
	Cooked seed	0.53	0.80 [d]	

[a] Average of 3 replicate analyses

[b] Results are the mean of two replicate samples taken from the processed commodities

[c] Processing factors not reliable, because RAC samples were not of commercial standards.

[d] Processing factors not reliable because RAC samples were not soaked before analysis

[WC] In trial CEMS 5037-01 plants were fallen on the ground, were only 10-15 cm high and crop yield was lower than in the control plot, due to phytotoxicity. Samples did not comply with commercial standards

Processing of dry harvested soya beans

Processing study 13

In a non-GLP study, soya bean plants had been sprayed with fluazifop-butyl (racemate), harvested and then processed into hulls, oil, meal and soapstock [Atreya *et al*., 1981, PP9/0527, PP009B040]. Samples were derived from trials HU1-79-06 and 48-MO80-009, which have been reported in [Atreya *et al*, 1981, PP9/0736, report PP009B036]. Further information was not provided.

Storage conditions were not reported. Samples were analysed for total fluazifop using a modified GC-MS method PPRAM 53 (= PPRAM 62). Concurrent method recoveries and results for control samples were not reported. Since the residues in the hulls were < 0.03 mg/kg total fluazifop, it was decided that the remainder of the samples would not be analysed. Processing factors could not be derived from this study.

Processing study 14

In a non-GLP study, soya beans were processed into oil and cake under laboratory conditions [Atreya *et al*., 1981, no code, PP009B059]. The samples had been treated with fluazifop-butyl (racemate) at 2 × 0.25 kg ai/ha (USA) or 1 × 1.0 kg ai/ha (Canada) and harvested after 98 and 77 days after the last treatment, respectively. Although the study author indicated that results from these trials have been reported in [Atreya *et al*, 1981, PP9/0736, report PP009B036] (USA) and [Atreya *et al*., 1981, PP9/0510, report PP009B016] (Canada), no trials matched the sampling interval or the residues reported for the dry seeds. Information on the history of the samples is therefore not clear.

Oil processing: Whole seed samples were finely ground and exhaustively extracted with hexane by Soxhlett extraction for 6 hrs. The hexane was evaporated, leaving the crude oil. Cake samples were air dried overnight at room temperature. The crude oil was further refined. The crude oil (5 g) was mixed with orthophosphoric acid (0.2 ml) and 5 M NaOH (20 ml) and centrifuged. The soap layer was discarded and the oil layer was collected. The oil layer was mixed with 0.1 M NaOH (20 mL) and centrifuged. The soap layer was discarded and the oil layer was collected. The oil layer was mixed with distilled water at 90–95 °C (water/oil = 25/75 v/v) and the water layer was discarded. The remaining oil layer was dried on a rotary evaporator at 60 °C for 5–10 min. Fullers Earth (2–3 g) was added and the oil was filtered to leave the refined oil.

Storage conditions were not reported. Samples were analysed for total fluazifop (method not indicated) and results were corrected for respective recovery results (83% for soya cake, 81% for soya oil). Results for control samples were not reported.

Results are shown in Table 259. Residues in refined oil were lower than in crude oil, but no clear concentration of residues in the crude oil or the cake was found.

Processing factors from this study are not acceptable. Sample history, storage conditions and analytical methods were not reported and the processing procedure seems to be a very small scale laboratory experiment.

Processing study 15

In a non-GLP commercial processing study [Atreya, 1982, PP9/0700, report PP009B152] performed in 1981/82 on composite soya bean samples (10 kg) from unknown origin. Subsamples of soya bean seeds were removed prior to processing to provide residues in the RAC.

Hulls: Soya beans (0.1 kg) were processed in the laboratory to provide hull and meal (i.e. ground seeds without hulls) and establish the distribution of the residue between each. The ratio hull to meal was 7.93 to 92.07 (weight percentage).

Oil processing: Soya beans (9.9 kg) were dried and ground by a commercial processing facility. The ground seeds were extracted in a soxhlet with n-hexane. Extraction was continued until no colour was apparent in the percolating solvent; the oil content of the remaining cake was then about 2%. The solvent was removed from the cake by warming in a current of air and the cake was

sampled for analysis. The crude oil was analysed for free fatty acid content. De crude oils were vigorously agitated at 90 °C with 2% w/v of a 10% solution of phosphoric acid. The crude oil and phosphoric dispersion were cooled to 40 °C and sufficient 14% caustic soda solution was added to neutralise the phosphoric acid and give a 30% excess of alkali. The oil was agitated at 40 °C for 20–30 minutes and the soap formed was allowed to settle. The oil was decanted from the soap layers, washed twice with water and finally with citric acid. The aqueous layers were settled and the oil decanted off. After the final wash, the oil was vacuum dried. Bleaching was accomplished by agitating the oil at 90 °C in the presence of 1% 237 Fullers earth and filtering after 30 minutes contact. Finally the product was deodorized with a steam distillation of the bleached oil carried out for one hour under 240 °C under vacuum (0.5 mm pressure) with 1% steam/hour.

Aliquots of whole seed samples, hulls, meal, cake (i.e. oil extracted meal), crude oil, refined oil and soapstock were retained for analysis. Storage information was not provided. Samples were analysed for total fluazifop using GC-MS method PP009B152 with a reported LOQ of 0.1 mg/kg. The method is considered not valid for soya bean commodities. Duplicate analyses were carried out on all samples. Average concurrent method recoveries were 72–134% (0.1–1.0 mg/kg) in soya refined and crude oil, soya hulls, meal, cake, soapstock, confirming method performance. However, method performance could not be confirmed for dry soya seeds (recovery 50% at 1.0 mg/kg). Residue levels were corrected for concurrent recoveries; uncorrected values are not reported. Positive residues were detected in all control samples: dry soya beans (0.21mg/kg), crude oil (0.25 mg/kg), refined oil (0.25 mg/kg), hulls (0.04 mg/kg), meal (0.08 mg/kg). Residue levels and processing factors are reported in Table 259.

Processing factors from this study are not acceptable: field trial and storage information is not provided, the analytical method is not valid, control levels were up to 0.30 mg/kg and recovery for the RAC is 50%.

Processing study 16

In a non-GLP study, composite samples from field treated soya beans were used for processing [Koubek, 1982, 406228, TMU0975B]. The soya bean contained 1.75 mg/kg total fluazifop. Soya bean seeds were processed into soya oil (crude) and solvent extracted meal by simulated commercial practices.

Soya beans were ground to separate hulls and then extracted with a non-polar solvent to release crude oils from the meal. Crude oil was treated with phosphoric acid and then treated with a caustic soda solution which released the soapstock. Refined oil was decanted, washed, dried, bleached and deodorised. Further details were not available.

Soya bean seeds were kept at -23 ºC for 2–12 months prior to processing and analysis. Storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using HPLC-UV method PPRAM 62/2 with a valid LOQ of 0.05 mg/kg. The method is considered valid for the determination of dry soya bean seeds (0.05–0.5 mg/kg). Average concurrent recovery was 99% (0.2–1.0 mg/kg) in soya bean seeds, confirming method performance. Residue levels were corrected for recoveries. Residues in control samples were not reported.

Results are shown in Table 259. Material balance for the fractions was 100%.

Processing study 17

A residue trial was carried out on soya (variety Nikir) during 1998 in Italy, Emilia Romagna region [Mason and Volpi, 1998, PP5_50435, RJ2914B]. A single application of fluazifop-P-butyl (EC formulation) was applied at a rate of 0.31 kg ai/ha by broadcast foliar spray. Details of the trial are summarized in the section residues resulting from supervised residue trials (trial IT20-98-H319). Replicate samples were taken from one trial 74 days after application and used for processing. Samples of soya bean seeds were stored at 5–25 °C until processing. Processing started at 36 days after harvest. Total fluazifop residues were monitored during this period and residue levels were 2.9, 2.7 and 3.5 mg/kg total fluazifop after 16, 25, 36 days of storage at ambient temparature. The residue

levels immediately before processing were used to calculate the processing factors. The samples were processed into soya flour, oil and milk.

Soya bean flour*:* Soya bean seeds were conditioned at approximately 60 °C, crushed and wind sifted to separate the flour from the hulls. Both flour and hull samples were frozen.

The process was started with 5 kg soya beans. After this process, 4.5 kg was in the soya bean flour and 0.4 kg was in the hulls.

Soya bean oil*:* Soya bean seeds were crushed, flocculated and heated to 44–51 °C. The flocs were then extracted into n-hexane at 60 °C. The n-hexane was distilled at 42 °C and 440 mbar for up to 3.5 hours and the remaining n-hexane was removed. The crude oil and oil-extracted meal were weighed and samples were frozen. The remainder of the oil-extracted meal was used for processing to milk and the remaining crude oil was refined. The crude oil was heated to 60 °C under stirring, aqueous solutions were added and the resultant aqueous phase was removed and frozen (slime/water mixture). The oil was neutralized using 4% NaOH and the resultant aqueous 'soap' sample was frozen. The oil was heated to 90 °C and washed repeatedly with water until pH 6–7 was achieved. The resultant aqueous phases were combined. The crude oil was dried and bleached. The resultant solid residue was frozen as the filter cake sample. Finally the crude oil was deodorised under vacuum to produce the refined oil sample. Both the refined oil and the resultant condensed fatty acids were frozen.

The process was started with 23.6 kg treated soya beans. For the treated beans, 4.5 kg was in the crude oil and 18.9 kg in the oil meal. From 1.8 kg crude oil, 1.2 kg refined oil could be processed.

Soya milk*:* The oil-extracted meal was processed by slurrying with water at pH 7.2. The slurry was filtered and the resulting solid was re-slurried prior to a second filtration step. The resultant solid material (protein-extracted meal) was frozen. The two soya extracts were combined and heated to approximately 90°C. Sugar and commercially available soya oil were added. The soya extracts were homogenised and the resulting soya milk was frozen.

The process was started with 1 kg soyameal and finished with 17.4 kg soya milk.

Samples were stored at -18°C for up to 5 months prior to analysis. Storage stability is considered not an issue for total fluazifop.

Samples of whole soya bean, flour, hulls, oil extracted meal, filter cake and protein extracted meal were analysed for total fluazifop using HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01–0.1 mg/kg depending on the commodity. The method is considered valid for the determination of total fluazifop in soya bean meal (0.01–0.25 mg/kg), soya bean flour (0.01–0.1 mg/kg), soya bean flocs (0.01–0.1 mg/kg), dry soya bean seeds (0.05–5.0 mg/kg) and soya bean hulls (at 0.25 mg/kg only). Average concurrent method recoveries were 93–117% (0.01–5.0 mg/kg) in soya beans and hulls, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg), except in soya hulls (0.02 mg/kg). The residue level in soya hulls is unlikely to affect processing factors derived for soya hulls.

Samples of fatty acids, soya bean crude and refined oil were analysed for total fluazifop using HPLC-MS/MS method RAM 122/04 with a valid LOQ of 0.01 mg/kg. The method is considered valid for the determination of free fluazifop acid (II) in soya oil (0.01–0.25 mg/kg) but not valid for the determination of fluazifop (II) conjugates. Average concurrent recoveries ranged from 68–111% (0.01–5.0 mg/kg) in soya oil and fatty acids, confirming method performance for free fluazifop acid (II). Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

Samples of soya bean soapstock, slime/water and soya milk were analysed for total fluazifop using method RAM 336/01, with a valid LOQ of 0.01 mg/kg for soya milk. The method is considered valid for for the determination of total fluazifop in soya milk (0.01–0.25 mg/kg), soya based infant formula (0.01–0.1 mg/kg), soya bean wash water (0.01–0.1 mg/kg), soya bean aqueous phase (0.01–0.1 mg/kg) and soya bean filter cake (0.01–0.1 mg/kg). Average concurrent recoveries ranged from

95–118% (0.001–1.0 mg/kg) in soya milk, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.001 mg/kg).

Results are shown in Table 259. The production of soya hulls, refined oil, soya milk and protein extracted meal resulted in a reduction in the total fluazifop residues. Concentration of residues was observed in flour and oil extracted meal. Processing factors for fatty acids and oil are not taken into account because of lack of radiovalidation for fluazifop conjugates.

Analysis of the protein content in the soya milk revealed a much lower protein concentration (0.8% protein) than is seen in the commercially produced milk (3.6% protein). Thus, the milk processing did not result in a product comparable to commercially available soya milk, and therefore, the data from the soya milk process are questionable. Processing factors for soya milk will not be taken into account.

Processing study 18

Four residue trials were carried out on soya in Italy in 1999 [Mason and Volpi, 2002, PP5/1144, RJ3149B]. One application of an EW formulation containing fluazifop-P-butyl was applied atat 0.25 kg ai/ha at growth stage R1 (Fehr and Caviness scale). Samples were taken at PHI 98–120 days for processing. Details of the trials are summarized in the section residues resulting from supervised residue trials (trials IT20-99-H385, H386, H387, H388). Samples for processing (12–18 kg) were obtained by mechanical threshing. The seeds were separated from the debris by sieving. The soya beans from the four trials were combined to give a bulk sample for processing. Samples for processing were stored at room temperature (18–24 °C) for 2–13 days until transport and than at 5 °C for 1 month until processing. Four distinct processes were carried out to produce soya flour, soya oil, soya protein isolate and soya milk. The production methods were designed to mimic commercial processing.

Soya bean flour processing*:* Soya bean seeds (5 kg) were crushed in contra rotating rollers and conditioned at approximately 60 °C in a drum dryer. A portion of the resulting flocs (5 kg) were wind sifted to separate the soya bean flour from the hulls. Both flour and hull samples were stored fozen prior to residue analysis.

Soya bean oil processing*:* Soya bean bean oil production was carried out in two stages crude oil production and refined oil production. Soya bean seeds (35 kg) were crushed in contra rotating rollers and condition at approximately 60 °C in a drum dryer. The resulting flocs were extracted in a pilot plant by continuous extraction into n-hexane (75 L) at 60 °C for 4 hours. The n-hexane was distilled at 40–46 °C and 470 mbar, the remaining n-hexane was removed by evaporation. The crude oil was weighed and a sample was frozen for residue analysis. The oil-extracted meal was vented at room temperature overnight to remove excess n-hexane, weighed and a sample was taken for frozen storage prior to residue analysis. The remainder of oil-extracted meal was used in the production of the protein isolate. Up to 1.3 kg of the crude oil was heated to 60 °C using two infrared lamps under stirring, aqueous solutions were added and heated to 85 °C. The resultant aqueous phase was removed and frozen for residue analysis as slime/water fraction. The oil was neutralised (4% sodium hydroxide at 90 °C) and the resulting aqueous soap was frozen for residue analysis. The oil was heated to 90 °C and washed repeatedly with water until pH7; the resultant aqueous phases were combined and labelled wash water prior to being frozen for residue analysis. The crude oil was dried and bleached with bleaching earth, the resultant filter cake was frozen for residue analysis. Finally, the crude oil was deodorised under vacuum to produce the refined oil sample. The refined oil sample and fatty acids condensed during deodorisation were frozen prior to residue analysis.

Soya bean milk production*:* A portion of the de-hulled soya bean seeds (0.8 kg) was stirred with 1.6 L of alkalised water (pH 8.5) and allowed to swell overnight at room temperature. Approximately 1.2 L of water was added to the swelled beans and the resultant mixture was heated to 100 °C. The heated mixture was ground using an ultr turrax macerator and stirred for 2 hours at 1700 RPM. The mixture was allowed to cool to 50 °C and filtered. The filtrate was soya milk with a nominal protein content of 3.6 % (w/v). The filter residue was further washed with water to give a

sample of soya milk (second extract) and filter cake, both samples were retained for residue analysis. Though soya milk with a nominal protein content of 3.6 % (w/v) is usually available in food retailers for child and adult consumption, in this processing study a concentration of 33.2–35.1 g/L was achieved. The soya isolate was diluted to 1.8% (w/v) to reproduce the concentration found in soyabased infant formula (SBIF) after preparation. A concentration of 17.6–20.3 g/L was achieved. All weight fractions have been corrected for subsampling.

Production of diluted soya protein isolate*:* About 3 kg of the oil-extracted meal resulting from the oil production were flocculated using corrugated and smooth rollers. The flocs were de-fatted in two batches using n-hexane in a Soxhlet extractor and the de-fatted flocs were left to stand overnight to remove excess n-hexane. The defatted flocs were ground using a Retsch mill. A portion of the de-fatted flocs (2 kg) was added to a stirred vessel containing 2 L of alkalised water (pH 8.5) pre-heated to 50 °C. The pH was maintained at 8.5 by addition of sodium hydroxide solution (3M). The hot mixture was homogenised, centrifuged and the protein-rich supernatant was decanted, the remaining solid material was extracted again in the same manner and the protein-rich supernatants were combined. This procedure was repeated using a second batch of de-fatted flocs (0.2 kg). The liquid extracts were combined to give one aqueous protein sample and the remaning solid material from both extractions was combined as the extraction residue and stored frozen for residue analysis. The protein-rich supernatant was acidified to pH 4.5 using HCl solution (3M). The mixture was centrifuged and the liquid phase was stored frozen for residue analysis, the solid remaining was dissolved in water and adjusted to pH 7 by addition of sodium hydroxide solution (3M). The suspension was filtered over a 0.25 mm sieve and dried in a lab spray drier. The resulting protein isolate was diluted with water to give a nominal protein content of 1.8 %.

Soya bean seeds and processed fractions were stored at -18 °C for up to 16 months prior to analysis. Storage stability is considered not an issue for total fluazifop.

Samples of soya bean, flour, hulls and oil-extracted meal were analysed for total fluazifop using HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01–0.1 mg/kg depending on the commodity. The method is considered valid for the determination of total fluazifop in soya bean meal (0.01–0.25 mg/kg), soya bean flour (0.01–0.1 mg/kg), soya bean flocs (0.01–0.1 mg/kg), dry soya bean seeds (0.05–5.0 mg/kg) and soya bean hulls (at 0.25 mg/kg only). Average concurrent method recoveries were 75–108% (0.01–1.0 mg/kg) in soya beans and it processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg.

Samples of residual oil, crude oil and refined oil were analysed for total fluazifop using HPLC-MS/MS method RAM 122/05 with a valid LOQ of 0.01 mg/kg. The method is considered valid for the determination of fluazifop acid (II) in soya oil (0.01–0.25 mg/kg), but not valid for the determination of fluazifop (II) conjugates. Average concurrent recoveries ranged from 74–112% (0.01–0.1 mg/kg) in soya oil, confirming method performance for free fluazifop acid (II). Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

Samples of soya milk and diluted protein isolate were analysed for total fluazifop using method RAM 336/01, with a valid LOQ of 0.01 mg/kg for soya milk. The method is considered valid for for the determination of total fluazifop in soya milk (0.01–0.25 mg/kg), soya based infant formula (0.01–0.1 mg/kg), soya bean wash water (0.01–0.1 mg/kg), soya bean aqueous phase (0.01–0.1 mg/kg) and soya bean filter cake (0.01–0.1 mg/kg). Average concurrent recoveries ranged from 95–97% (0.05–0.1 mg/kg) in soya milk, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

Residue results and processing factors are shown in Table 259. Processing factors for oil are not taken into account because of lack of radiovalidation for fluazifop conjugates.

Processing study 19

Three residue trials were carried out on soya in Italy in 2000 [Mason and Giacomelli, 2001, PP5/1122, RJ3208B]. One application of an EW formulation containing fluazifop-P-butyl was applied at 0.25 kg ai/ha at growth stage R1 (Fehr and Caviness scale). Samples were taken at PHI 90–96 days to confirm residue. Details of the trials are summarized in the section residues resulting from supervised residue trials (trials IT20-00-S356, S357, S358). At PHI 98-104 days samples were harvested for processing. Plants were harvested with a mower and collected by hand. The seeds were obtained by mechanical treshing on the same day and cleaned of the remaining debris by sieving. The seeds for processing (36–39 kg per trial) were kept at ambient temperature until processing (2 months). A portion of the soya beans was used for processing to soya flour and refined soya oil. The remainder of the soya bean bean samples were processed to soya milk and diluted protein isolate. In both cases the production method was designed to mimic the commercial process. All weight fractions have been corrected for subsampling.

Soya flour processing - Soya bean seeds (30 kg) were crushed in contra rotating rollers and conditioned at approximately 60 °C in a drum dryer. A portion of the resulting flocs (5 kg) was wind sifted to separate the soya bean flour from the hulls. Both flour and hull samples were stored fozen prior to residue analysis.

Soya oil processing - Soya bean bean oil production was carried out in two stages crude oil production and refined oil production.

Soya bean seeds (30 kg) were crushed in contra rotating rollers and condition at approximately 60 °C in a drum dryer. A portion of the resulting flocs (24 kg) was extracted in a pilot plant by continuous extraction into n-hexane (75 L) at 60 °C. The n-hexane was distilled at 40–49 °C and 450 mbar. Extraction and distillation was repeated and subsequently the remaining n-hezane was removed by evaporation. The crude oil was weighed and a sample was frozen for residue analysis. The oil-extracted meal was vented at room temperature overnight to remove excess n-hexane, weighed and a sample was taken for frozen storage rior to residue analysis. The remainder of the crude oil was removed. Crude oil (1.3 kg) was heated to 60 °C using two infor red lapms under stirring, aqueous solutions were added and heated to 85 °C. The resultant aqueous phase was removed and discarded. The oil was neutralised (4% sodium hydroxide) and the resulting aqueous soap was discarded. The oil was heated and washed repeatedly with water until pH7–8; the resulting aqueous phase was discarded. Partially refined crude oil was dried and bleached with bleaching earth, the resultant filter cake discarded and finally, the oil was deodorised under vacuum to produce the refined oil sample, which was frozen prior to residue analysis.

Soya milk production – Soya bean seeds (1 kg) were de-hulled as described for the other processes. De-hulled seeds (0.8 kg) was stirred with 1.6 L of alkalised water (pH 8.5) and allowed to swell overnight at room temperature. Approximately 1.5 L of water was added to 1 kg of swelled beans and the resultant mixture was ground using an ultra turrac macerator and heated to 80 °C for 2 hours. The mixture was allowed to cool to 40 °C and filtered. The filtrate was diluted with water to give soya milk.

Soya milk as available in food retailers for child and adult consumption has a nominal protein content of 3.6 % (w/v). A concentration of 33.2–35.1 g/L (assumed to be 3.3–3.5% w/v) was actually achieved.

Production of diluted soya protein isolate – Soya bean seeds (3 kg) were de-hulled and flocculated. The flocs were de-fatted in two batches using n-hexane in a Soxhlet extractor and the de-fatted flocs were left to stand overnight to remove excess n-hexane, followed by grounding in a mill fitted with a 0.5 mm sieve. De-fatted flocs (0.4 kg) were added to a stirred vessel containing 3.6 L of alkalised water (pH 8.5) pre-heated to 50 °C. The pH was maintained at 8.5 by addition of sodium hydroxide solytuin (3M). The hot mixture was centrifuged and the protein-rich supernatant was decanted, the remaining solid material was extracted again in the same manner and the protein-rich supernatant was acidified to pH 4.5 using hydrochloric acid solution (3M). The mixture was centrifuged and the liquid phase discarded, the solid remaining was dissolved in water and adjusted to

pH 7 by addition of sodium hydroxide solution (3M). The suspension was filtered and dried in a lab spray drier. The resulting protein isolate was diluted with water to give a the soyabased infant formula (SBIF).

Soya based infant formula (SBIF) as available in food retailers contains a nominal protein content of 1.8% (w/v). A concentration of 17.6–20.3 g/L (assumed to be 1.8–2.0% w/v) was actually achieved.

Soya bean seeds and processed fractions were stored at -18 °C for 6–9 months prior to analysis. Storage stability is considered not an issue for total fluazifop.

Samples of soya bean, soya flour, hulls and oil-extracted meal were analysed for total fluazifop using HPLC-MS/MS method RAM 287/02 with a valid LOQ of 0.01–0.1 mg/kg depending on the commodity. The method is considered valid for the determination of total fluazifop in soya bean meal (0.01–0.25 mg/kg), soya bean flour (0.01–0.1 mg/kg), soya bean flocs (0.01–0.1 mg/kg), dry soya bean seeds (0.05–5.0 mg/kg) and soya bean hulls (at 0.25 mg/kg only). Average concurrent method recoveries were 83–104% (0.01–1.0 mg/kg) in soya beans and it processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg.

Samples of crude and refined oil were analysed for total fluazifop using HPLC-MS/MS method RAM 122/05 with a valid LOQ of 0.01 mg/kg. The method is considered valid for the determination of fluazifop acid (II) in soya oil (0.01–0.25 mg/kg), but not valid for the determination of fluazifop conjugates. Average concurrent recoveries ranged from 78–94% (0.05–0.1 mg/kg) in soya oil, confirming method performance for free fluazifop acid (II). Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

Samples of soya milk and diluted protein isolate were analysed for total fluazifop using method HPLC-MS/MS RAM 336/01, with a valid LOQ of 0.01 mg/kg for soya milk. The method is considered valid for for the determination of total fluazifop in soya milk (0.01–0.25 mg/kg), soya based infant formula (0.01–0.1 mg/kg), soya bean wash water (0.01–0.1 mg/kg), soya bean aqueous phase (0.01–0.1 mg/kg) and soya bean filter cake (0.01–0.1 mg/kg). Average concurrent recoveries ranged from 86–104% (0.01 mg/kg) in soya milk and protein isolate, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

Soya bean seeds were analysed immediately before processing. Residue results and processing factors are shown in Table 259. Processing factors for oil are not taken into account because of lack of radiovalidation for fluazifop conjugates.

The production of soya bean hulls, refined oil, soya milk and diluted soya protein isolate resulted in a reduction of the total fluazifop residue in the processed commodity compared with the RAC.

Processing study 20

Field grown soya bean (indeterminate type) was treated with fluazifop-P-butyl in the USA in 2000 [Stewart, 2011, 406508, report RR 00-069B]. Soya bean was treated at the beginning of first buds forming with a spray application of fluazifop-P-butyl (EC formulation) at an application rate of 1 ×0.86 kg ai/ha plus 1 × 0.21 kg ai/ha at a 26 day interval. Details of the trials are summarized in the section residues resulting from supervised residue trials (trial 251SYMO1). Seeds were sampled at 79 DAT and stored at -12 ºC until processing for a maximum period of 19 days. Seeds from one location were processed into refined oil, simulating industrial processing.

Oil processing: Soya bean seeds (28.7/34.2 kg) were dried in an oven at 54–71 °C until moisture content of 7–10%. Dried seeds (26.5/31.5kg) were aspirated to separate small impurities. A Bauer disc mill was used to crack the hull and liberate the kernel. Hull (3.3/3.1 kg) and kernel (21.2/26.1 kg) were separated by aspiration. Kernels were heated to 71–79 °C and flaked and subsequently fed to an expander/extruder (exit temp 77–113 °C). After expansion, the collets are dried

in an oven at 54–71 °C (9/9 kg) and taken through solvent extraction and washing with hexane (8.73/9 kg). The collets are toasted (instead of run through with warm air). Crude oil (2.087/2.019 kg) and meal (6.48/6.6 kg) is separated. The crude oil is refined according to AOCS method Ca 9b-52. After refining, the refined oil (1.477/1.478 kg) and soapstock (160.1/153.1 g) are separated.

Samples were taken from soya seeds, hulls, kernels, solvent extracted meal, crude oil, refined oil and soapstock. Seeds and processed products were stored at -18 ºC for a maximum of 24 months. Storage stability is considered not an issue for total fluazifop. Samples were analysed twice. Seeds, kernel, hull, meal and oil were analysed for total fluazifop using GC-MS method RR 91-014B with a valid LOQ of 0.01 mg/kg. This method is considered valid for the determination of total fluazifop in dry soya bean seeds (0.01–0.5 mg/kg) and soya bean oil (0.01–0.1 mg/kg), but insufficiently validated for soya bean meal and hulls. Average concurrent method recoveries were 74–100% (0.01–1.0 mg/kg) in soya bean and its processed commodities, confirming method performance. Samples were not corrected for average concurrent recoveries. In control samples no residues were found (< 0.01 mg/kg). Residue results and processing factors are shown in Table 259.

Processing study 21

Field grown soya bean (indeterminate type) was treated with a formula containing fluazifop-P-butyl in the USA in 2010 [Hampton and Mazlo, 2013, A12460A_50026, report TK0016832]. The first application was applied pre-bloom and second at the beginning of bud formation. Fluazifop-P-butyl (EC formulation) was applied as foliar over the top broadcast spray at an application rate of 1 × 0.42 kg ai/ha plus 1 x 0.11 kg ai/ha. Details of the trials are summarized in the section residues resulting from supervised residue trials (trial C13-0222). Seeds from one location were processed into aspirated grain.

Generation of aspirated grain fraction: Soya bean seeds (308/294 kg) were not dried as the moisture content was 12.75 and 9.23 %, respectively (within range of 7-10%). The samples were placed in a dust generator. Samples were moved for 120 minutes. Aspiration fractions were 90 g in both samples. Light impurities were classified with sieves 2360 micron, 2000 micron, 1180 micron, 850 micron, and 425 micron. The material through the 2360 micron sieve was recombined to the aspirated grain fraction (AGF). The material passing through the 425 micron sieve (20.7 g/38.6 g) was greater than half the weight of the total material passing through the 2360 micron sieve, so all the material that went through the 2360 was recombined and the ash content was determined (40.7/25.1%)

Samples were stored below -10 ºC until shipment and below -20 °C for further storage (max 13 months). Storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using HPLC-MS/MS Method GRM044.01A with a valid LOQ of 0.01 mg/kg. This method is considered valid for the determination of total fluazifop in dry soya bean seed (0.01-1.0 mg/kg), soya bean meal (at 0.2 mg/kg only), soya bean hull (at 0.2 mg/kg only), soya bean oil (at 0.2 mg/kg only). Average concurrent method recoveries were 107-121% (0.01-10 mg/kg) in soya bean seed and its processed commodities, confirming method performance. Results were not corrected for concurrent method recoveries. Control samples were < 0.01 mg/kg.

Residue results and processing factors are shown in Table 259. Total fluazifop residues did not concentrate (<1 ×) in the aspirated grain fraction. Mass balance was sufficiently addressed.

Table 259 Residues and processing factors in soya bean seeds and processed soya commodities.

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Soya bean commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
Canada, 1980 1 × 1.0 kg ai/ha, DALT 77 (racemate) No field report available.	Dry seeds Cake (i.e. oil extracted meal) crude oil	0.92 1.02 0.32	- 1.1 [d] 0.35 [d] [SK*]	PP009B059 5562/80 [Atreya *et al*., 1981, PP9/0544]

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Soya bean commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
Laboratory experiment, does not represent commercial practice				
USA, 1981 2 × 0.25 kg ai/ha, DALT 98 (racemate) No field report available. Laboratory experiment, does not represent commercial practice	Dry seeds Cake (i.e. oil extracted meal) crude oil refined oil	0.54 0.47 0.74 0.44	- 0.87 [d] 1.4 [d] 0.81 [d] [SK*]	PP009B059 713/81 [Atreya *et al*., 1981, PP9/0544]
USA, 1981 2 × 0.25 kg ai/ha, DALT 98 (racemate) Laboratory experiment, does not represent commercial practice	Dry seeds Cake (i.e. oil extracted mea) crude oil refined oil	0.52 0.47 0.80 0.49	- 0.90 [d] 1.5 [d] 0.94 [d] [SK*]	PP009B059 714/81 [Atreya *et al*., 1981, PP9/0544]
USA, 1981 No field report available. Commercial processing Quality of the study is insufficient [QU]	Dry seeds (RAC) Hull Meal (before oil extraction) Cake (i.e. oil extracted meal) Crude oil Soapstock Refined oil	1.2 [a] 0.67 [a] 1.5 [a] 1.6 [a] 3.0 [a] 1.9 [a] 1.6 [a]	- 0.54 [d] - 1.3 [d] 2.4 [d] 1.5 [d] 1.3 [d] [SK*]	PP009B152 [Atreya *et al*. 1982, PP9/0700]
USA, 1990 No field report available. Commercial processing	Dry seeds (RAC) Hull Crude Oil Oil extracted meal	1.75 0.89 1.46 1.64	- 0.51 0.83 0.94	TMU0975/B [Koubek, 1982, 406228]
Filetto, Emilia Romagna, Italy, 1998, (Nikir) EC 125 (P); 1 × 0.31 kg ai/ha, DAT 74 days. GS: R3, 50 cm high; 9 July 1998 Soil: loam Dry seeds were stored for 36 days at room temperature before the start of processing. Residues in dry seeds were analysed just before processing.	Dry seeds (RAC) Flour Hull [cntrl 0.02] Oil extracted meal Crude oil Slime/water Soap Wash water Filter cake Fatty acids Refined oil Soya milk Protein extr meal	3.50 [a] 3.85 [a] 0.785 [a] 3.95 [a] 0.940 [a] 0.90 [a] 0.51 [a] 0.03 [a] 0.21 [a] < 0.01 [a] 0.270 [a] 0.18 mg/L [a] 0.215 [a]	- 1.1 0.22 1.1 0.27 [b] - - - - < 0.003 [b] 0.08 [b] 0.05 [c] 0.06	RJ2914B; IT20-98-H319 [Mason and Volpi, 1998, PP5_50435]
Italy, 1999 (4 trials) EW 250 (P) + TF8035 mineral oil; 1 × 0.25 kg ai/ha; DALT: 98-120 days GS: (R1, 30-50 cm), 15-29 June, 1999 Soil: loam or silty sand Bulk sample from 4 sites in Italy; samples taken from plot 3 at each site	Dry seeds (RAC) Flour Hulls Flocs Oil extracted meal Crude oil Slime Soap Wash water Filter cake Fatty acids Refined oil Oil extracted meal	0.210 [a] 0.230 [a] 0.110 [a] 0.235 [a] 0.260 [a] 0.065 [a]] 0.015 [a] 0.075 [a] < 0.01 [a] 0.02 not analysed 0.03 [a] 0.245 [a]	- 1.1 0.52 - 1.2 0.31 [b - - - - - 0.14 [b] 1.2	RJ3149B; IT20-99-H385; IT20-99-H386; IT20-99-H387; IT20-99-H388 [Mason and Volpi, 2002, PP5/1144]
idem	Dry seeds (RAC) Flour Hulls Flocs Filter cake Soya milk Aqueous phase	0.25 [a] 0.20 [a] 0.175 [a] 0.265 [a] 0.04 0.045 [a] 0.02 [a]	- 0.80 0.70 - - 0.18 -	idem

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Soya bean commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
	Extraction residue Diluted protein isolate Residual oil Soya milk (2nd extract) Flocs without oil	0.01 [a] < 0.01 [a] 0.06 [a] 0.035 [a] < 0.01 [a]	- < 0.04 - [b] 0.14 -	
Glorie di Mezzano (48010, RA), Italy, 2000, (var Mixer) EW 250 (P) + TF8035 mineral oil, 1 x 0.25 kg ai/ha; DALT: 104 days GS: R1, 30 cm, 07 June, 2000 Soil: not indicated (friable)	Dry seeds (RAC) Flour Hull Oil extracted meal Crude oil Refined oil	0.200 [a] 0.235 [a] 0.130 [a] 0.265 [a] 0.085 [a] 0.040 [a]	- 1.2 0.65 1.3 0.42 [b] 0.20 [b]	RJ3208B, IT20-00-S356 [Mason and Giacomelli, 2001, PP5/1122]
idem	Dry seeds (RAC) Diluted protein isolate Soya milk	0.210 [a] < 0.01 [a] 0.03 [a]	- < 0.05 0.14	idem
Lavezzola (18021, RA), Italy, 2000, (var Albir) EW 250 (P) + TF8035 mineral oil, 1 x 0.25 ai kg/ha; DALT: 103 days GS: R1, 40 cm, 07 June, 2000 Soil: clay loam	Dry seeds (RAC) Flour Hull Oil extracted meal Crude oil Refined oil	0.615 [a] 0.670 [a] 0.225 [a] 0.865 [a] 0.195 [a] 0.07 [a]	- 1.1 0.37 1.4 0.32 [b] 0.11 [b]	RJ3208B; IT20-00-S357 [Mason and Giacomelli, 2001, PP5/1122]
idem	Dry seeds (RAC) Diluted protein isolate Soya milk	0.68 [a] 0.03 [a] 0.14 [a]	- 0.044 0.21	idem
Bando di Argenta (44010, FE), Italy, 2000, (var Lynda) EW 250 (P) + TF8035 mineral oil; 1 x 0.25 kg ai/ha; DALT: 98 days GS: R1, 30 cm, 13 June, 2000 Soil: loam	Dry seeds (RAC) Flour Hull Oil extracted meal Crude oil Refined oil	0.555 [a] 0.590 [a] 0.210 [a] 0.545 [a] 0.130 [a] 0.08 [a]	- 1.1 0.38 0.98 0.23 [b] 0.14 [b]	RJ3208B; IT20-00-S358 [Mason and Giacomelli, 2001, PP5/1122]
idem	Dry seeds (RAC) Diluted protein isolate Soya milk	0.555 [a] 0.02 [a] 0.05 [a]	- 0.036 0.090	idem
Leonard, MO, USA, 2000, (var NK 3911) EC 240 (P) +NIS 0.5%, 1 × 0.86 +1 × 0.21 kg ai/ha, interval 26 days, DALT = 79 days, GS: R3, 11 July, 2000 Soil: Silty loam	Dry seeds (RAC) Oil extracted Meal Hull Refined Oil	2.15 [a] 2.75 [a] 1.65 [a] 0.01 [a]	- 1.3 0.77 0.005 [SK*]	RR 00-069B S251SYMO1 [Stewart, 2001, 406508]
Northwood, ND, USA, 2010 (var PFS 0905) EC 240 (P) + 1.0% COC; 1 × 0.42 + 1 ×0.11 kg ai/ha, interval 27 days DALT: 82 days GS: R3, 21 July, 2010 Soil: Sandy clay loam	Dry seeds (RAC)	0.84, 0.79, 0.84 (mean: 0.82)	-	TK0016832; C13-0222 [Hampton and Mazlo, 2013, A12460A_50026]
	Aspirated grain	0.26 [a]	0.32 [SK*]	

V and R growth stages are Fehr-Caviness growth stages. V5 = plants with 4 nodes (counting the unifoliate node) with fully developed trifoliate leaves. No blooms present a V5 stage. R3 = beginning of pod growth; at least one pod 5 mm long at one of the four uppermost fully developed leaf nodes on the main stem

QU Quality of the study is insufficient. Since field trial information is not provided, storage information is not provided, control levels were up to 0.30 mg/kg and recovery for the RAC is 50%, information from this study cannot be used for derivation of processing factors

[cntrl] Residue level found in the untreated control sample

[a] Results are the mean of two replicate analyses

[b] Processing factors for fatty acids and oil are not taken into account because of lack of radiovalidation for fluazifop conjugates for HPLC-MS/MS method RAM 122.

[c] Processing factors for soya milk are not taken into account, because the final product contained a lower protein content than in commercial products.

[d] Processing factor not taken into account because of poor quality of the study or because the process does not represent commercial practice.

[SK*] Its not clear from the report, whether a soaking step has been performed prior to extraction of the RAC samples. Processing factors not taken into account.

Processing of potatoes

Processing study 22

Potatoes were treated in the field with a single broadcast foliar spray of fluazifop-butyl (racemate) at an application rate of 0.75 kg ai/ha at four locations in the UK in 1982 [Atreya *et al*., 1982, PP9/0702, report PP009B153]. Details of the trials are summarized in the section residues resulting from supervised residue trials (report PP009B153). Tubers were taken at 21–70 days after application. Potatoes were washed, peeled and boiled using household methods.

Washed potatoes*:* A representative sample of potatoes from each trial (0.3–0.6 kg) was washed with cold water to remove soil. The washings from 3 samples were kept for analysis.

Peeled potatoes: Potatoes (0.37–1.3 kg) were peeled to represent household practice and the weight of the peel and flesh were recorded. About 14–20% of the weight was peel. Potato flesh was cut in half. One part was analysed and the other part was further processed into boiled potatoes.

Boiled potatoes: Potato flesh halves (0.37–1.3 kg) were either boiled in twice the weight salted water on a hot plate or on a gas cooker. Potatoes were drained and potatoes and cooking water was analysed. The potatoes took up to 3 hours to boil on a hot plate but only 30 minutes on the gas cooker. The boiled potatoes from the hot plate resulted in greater disintegration of the flesh, which was analysed together with the cooking water. Processing factors derived for boiled potatoes on a hot plate are not taken into account, because the boiling procedure does not represent household practice.

Storage conditions were not reported. Storage stability is considered not an issue for total fluazifop. The crop samples and processed commodities were analysed for total fluazifop using HPLC-UV method PPRAM 62 (with internal standard) with a valid LOQ of 0.05 mg/kg for most commodities. This method is considered valid for the determination of total fluazifop in potato tubers (0.1–1.0 mg/kg), boiled potatoes (1.0 mg/kg only), potato peels (1.0 mg/kg only). Average concurrent method recoveries were 80–88% (1.0 mg/kg) in potato flesh, peel and boiled flesh, confirming method performance. Residue levels were corrected for internal standard recovery. Control samples were not reported.

The results are presented in Table 260. Mass balance calculations indicate that cooking on a gas cooker only slightly affects total fluazifop residues in the edible portions of the potatoes. Residues in the flesh (73–80%) + cooking water (8–15%) add up to 88–95% of the residues in the raw flesh.

Processing study 23

Potatoes were treated in the field with a broadcast foliar spray of fluazifop-P-butyl at an application rate of 0.375 kg ai/ha with an ME formulation at two locations in Germany in 1993 [Weeren, 1994, PP5/0102 = Pelz, 1994, PP5_50062, report AZ13430/93]. Details of the trials are summarized in the section residues resulting from supervised residue trials (trials RS-9307-B1 and B2). Potato tubers were stored at 4–6 °C until processing for a maximum of 8 days. Potatoes were processed using household methods.

Peeled potatoes: Potatoes (10.0/9.17 kg) were peeled and cut, yielding 8.67/7.84 kg potatoes without peel and 1.29/1.33 kg peels. The peels were kept separately. Peeled potatoes were processed further into cooked potatoes, chips and dried potatoes.

Cooked potatoes: peeled potatoes (2.0/2.1 kg) were cooked for 20–25 min in 2 L water with 10 g salt. The cooked potatoes (2.60/1.94 kg) were allowed to drain and the cooking liquid (1.44/1.60 kg) was kept separately.

Chips: peeled potatoes (2.1/2.0 kg) were cut into thin slices and were deep-fried during 20 min in 1 kg fat. This yielded 0.89/0.91 kg of chips. Since this concerns thinly sliced potatoes, the reviewer assumes that chips refer to crisps and not to French fries.

Dried potatoes: peeled potatoes (1.56/1.35 kg) were sliced, distributed evenly on the racks of the desiccation apparatus and dried for 6–8 hrs in a desiccation apparatus. This yielded 0.41/0.29 kg of dried potatoes.

Potato tubers and processed samples were stored at –18 ºC for maximum of 170 days. Storage stability is considered not an issue for total fluazifop. The crop samples and processed commodities were analysed for total fluazifop using GC-MS method P-14.077 with a reported LOQ of 0.01 mg/kg. This method is considered valid for the determination of free fluazifop acid (II) in potatoes (0.1 mg/kg only) but not valid for the determination of fluazifop (II) conjugates. Average concurrent method recoveries were 74–109% (0.01–0.1 mg/kg) in potatoes and its processed commodities, confirming method performance for free fluazifop acid (II). Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg), except in peels (< 0.01–0.01 mg/kg) and crisps (< 0.01–0.01 mg/kg.

The results are presented in Table 260. After peeling and cooking, the residues remained in the raw edible portion. Residues concentrated after deep-frying and drying. Processing factors from this study are not taken into account because of lack of radiovalidation for fluazifop conjugates.

Mass balance calculations indicate that the total fluazifop residues remain in the edible portions of the potatoes. Residues in the peel (8–16%)+ peeled potato (97–109%) add up to 105–125% of the residues in the RAC, residues in peeled boiled potato (71–105%)+ cooking liquid (8–14%) add up to 79–119% of the residues in the RAC, residues in chips represent 95–116% and residues in dried potatoes represent 97–108% of the residues in the RAC.

Table 260 Residues and processing factors in potatoes and processed potato matrices

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Potato commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
Manthorpe, Lincolnshire, UK, 1982 (var Estima) racemate 1 × 0.75 kg ai/ha, post-flowering, DAT 3 weeks	Raw potato with peel (RAC) Wash water (soil sludge) Washed raw peel Raw potato, without peel Cooked potato, without peel Boiling water	1.2 [a] < 0.01 1.1 1.2 0.95 0.08	- - 0.92 1.0 0.79 -	PP009B153 ADJ No 82/3013 [Atreya *et al*., 1982, PP9/0702]
Manthorpe, Lincolnshire, racemate; UK, 1982 (var Estima) 1 × 0.75 kg ai/ha, post-flowering, DAT 6 weeks	Raw potato with peel (RAC) Wash water (soil sludge) Washed raw peel Raw potato, without peel Cooked potato, without peel Boiling water	- < 0.01 - 1.0 0.73 0.04	-	PP009B153 ADJ No 82/ 3015 [Atreya *et al*., 1982, PP9/0702]
Manthorpe, Lincolnshire, racemate UK, 1982 (var Estima) 1 × 0.75 kg ai/ha, post-flowering, DAT 3 weeks	Raw potato with peel (RAC) Raw peel Raw potato, without peel Cooked potato, without peel Boiling water	1.2 [a] 0.34 1.3 0.96 0.11	- 0.28 1.1 0.80 -	PP009B153 ADJ No 82/3017 [Atreya *et al*., 1982, PP9/0702]
Manthorpe, Lincolnshire, racemate UK, 1982 (var Estima) 1 × 0.75 kg ai/ha, post-flowering, DAT 3 weeks	Raw potato with peel (RAC) Washed raw peel Raw potato, without peel Cooked potato, without peel Boiling water	- - 1.1 0.76 0.08	-	PP009B153 ADJ No 82/3019 [Atreya *et al*., 1982, PP9/0702]

Location, year, (variety), formulation, dose rate, interval, DALT Growth stage and date at last treatment Soil type	Potato commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
DAT 6 weeks				
Swillington Common, racemate UK, 1982 (var Wilja) 1 × 0.75 kg ai/ha, 25 cm high, DAT 5 weeks	Raw potato with peel (RAC) Wash water (soil sludge) Washed Raw peel Raw potato, without peel Cooked potato, without peel Boiling water	1.2 [a] < 0.01 0.77 1.3 0.66 0.69	- - 0.64 1.1 0.55 [b] -	PP009B153 ADJ No 82/1927 [Atreya *et al*., 1982, PP9/0702]
Cornwall, UK, 1982 (var Maris Peer) racemate 1 × 0.75 kg ai/ha, before meeting in rows, DAT 10 weeks	Raw potato with peel (RAC) Washed raw peel Raw potato, without peel Cooked potato, without peel Boiling water	0.64 [a] 0.34 0.70 0.39 0.42	- 0.53 1.1 0.61 [b] -	PP009B153 ADJ No 82/2265, [Atreya *et al*., 1982, PP9/0702]
Suffolk, UK, 1992 (var Desira), racemate 1 × 0.75 kg ai/ha, before meeting in rows, DAT 3 weeks; soil type ns	Raw potato with peel (RAC) Washed raw peel Raw potato, without peel Cooked potato, without peel Boiling water	1.3 [a] 0.44 1.4 0.76 0.68 [	- 0.34 1.1 0.58 [b] -	PP009B153 ADJ No 82/2590 [Atreya *et al*., 1982, PP9/0702]
Bienenbüttel-Varendorf, Germany, 1993 (variety not specified) ME 125 (P), 1 x 0.38 kg ai/ha, DALT = ca. 6 weeks (42 days), Soil type ns	Raw potatoes, with peel (RAC) Raw potatoes, without peel Peel, not washed [cntrl 0.01] Cooked potatoes, without peel Cooking liquid Chips (i.e. crisps) [cntrl 0.01] Dried potatoes	0.17 0.19 0.11 [c] 0.13 0.02 0.40 [c] 0.70	- 1.1 [c] 0.65 [c] 0.76 [c] - 2.4 [c] 4.1 [c]	AZ13430/93 RS-9307-B1 [Weeren, 1994, PP5/0102] = [Pelz, 1994, PP5_50062]
Büchen, Germany, 1993 Potato (variety not specified) ME 125 (P), 1 x 0.38 kg ai/ha, DALT = ca 6 weeks (42 days) Soil type ns	Raw potatoes, with peel (RAC) Raw potatoes, without peel Peel, not washed Cooked potatoes, without peel Cooking liquid Chips (i.e. crisps) Dried potatoes	0.22 0.28 0.25 0.32 0.05 0.72 1.41	- 1.3 [c] 1.1 [c] 1.5 [c] - 3.3 [c] 6.4 [c]	AZ13430/93 RS-9307-B2 [Weeren, 1994, PP5/0102] = [Pelz, 1994, PP5_50062]

[cntrl] Residue level found in the untreated control sample

[a] Calculated by the reviewer from the residues in peel and flesh and the weight fractions of the peel and flesh

[b] Boiled on a hot plate for 3 hrs, whereby potatoes disintegrated into the cooking water; Processing factor not taken into account

[c] untreated peel and chips contained 0.01 mg/kg residue; thereby increasing LOQ to 0.01/0.3=0.04 mg/kg. Since residues in peel and chips are >0.04 mg/kg, processing factors can be derived from this study.

[c] processing factors are not taken into account because the hydrolysis step used in the analytical method is not specified nor radiovalidated.

Processing of sugar beets

Processing study 24

In a non-GLP study, sugar beets were treated in the field with two broadcast foliar sprays of fluazifop-butyl (racemate) at an application rate of 1.5 kg ai/ha plus 1.0 kg ai/ha at a single location in the UK in 1982 [Atreya, 1982, PP9/0366, report PP009B089]. Sugar beets were harvested at 73 DAT. Details of the trials are summarized in the section residues resulting from supervised residue trials (trial 981-SBB-EA7). Sugar beets were processed into white sugar in a laboratory pilot comparable with industrial processing.

Sugar processing: Dirty sugar beets (27.8 kg) were washed in a beet washer for 5–10 min. Washed sugar beets (25 kg) were sliced into cossettes with standard beet knives. The cossettes (25 kg)

were subjected to counter current diffusion with water at 70 ºC to get 32 kg raw juice (a dilute impure sugar solution) and 25 kg wet pulp. The wet pulp (5% dry substance) was pressed for 1 min at 1500 psi, leaving 6 kg pressed pulp (20 % dry substance) and 19.2 kg press water. The pressed pulp was dried to leave 1.33 kg dried pulp (90% dry substance). The raw juice underwent several processes: carbonation at 80–85 ºC at pH = 11 and at 90 ºC at pH=9 (leaving 33.7 kg thin juice + precipitate), evaporation under vacuum at 40–50 ºC (leaving 6.5 kg thick juice + distillate), filtration (leaving 6.5 kg standard liquor + filter cake), boiling under vacuum at 80 ºC (leaving 4.24 kg massecuite + distillate). The massecuite was centrifuged and washed with hot water leaving damp sugar and 2.1 kg green syrup (i.e. washings and spun off syrup). The damp sugar is passed through a rotary hot air drier and sieved to remove coarse lumps to leave 1.8 kg white granulated sugar.

The residue in the pressed pulp can be used as estimate for the residue in ensiled sugar beet pulp, while the residue in the green syrup can be used as estimate for the residue in sugar beet molasses.

Storage conditions for RAC and processed samples were not indicated. Storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using HPLC-UV method PPRAM 62 or its modification A with a valid LOQ of 0.05 mg/kg. Selected samples were methylated and residue results were confirmed by GC-MS. The method is considered insufficiently validated for sugar beet roots and its processed commodities. Average concurrent method recoveries were 68–97% (0.1–1.0 mg/kg) in sugar beets and sugar, confirming method performance. Residue levels were corrected for concurrent recoveries; uncorrected results were not available. Residues in control samples were not reported.

Results were are shown in Table 261. Residues concentrate in the thick juice and the green syrup, but diminish in pressed pulp. Mass balance calculations indicate that residues were reduced in sugar: only 0.3% of the total fluazifop present in the RAC ends up in the white sugar.

Processing study 25

A second processing trial was carried out on sugar beets (variety Canyon) during 2000 in Washington, USA [Stewart, 2001, 406493, report RR 00-070B]. Fluazifop-P-butyl was applied as a broadcast foliar spray at an exaggerated dose of 2 ×2.11 kg ai/ha with a spray interval of 14 days. Sugar beet roots were sampled at normal harvest, 90 days after the last application. Details of the trials are summarized in the section residues resulting from supervised residue trials (trial 270 (SBWA1)). Sugar beet roots were processed into sugar (refined), dry pulp and molasses.

Sugar processing: Sugar beets were washed. Sugar was extracted from the cossettes in a series of steam heated kettles with a mixture of fresh water and pulp press water. The kettles were heated to 65–80°C. Extracted beet pulp was pressed to recover pulp press water and pressed wet pulp. The pressed wet pulp was dried within a target range of 80–93 °C to less than 10% moisture. The obtained dried pulp was milled and a sample was collected. Pulp press water (raw juice) from the diffuser was frozen prior to purification. The juice was purified by addition of lime and carbon dioxide at a temperature range of 80–90 °C. Clear juice was decanted and screened to remove suspended larger particles and the settled sludge was vacuum filtered. Filtrate was combined with the clear decanted liquid and filter cake was discarded. The clarified liquid was further purified and vacuum filtered. Filter cake was discarded. The clarified juice was concentrated and then heated within a range of 75–85 °C and vacuum filtered. The filter cake was discarded. The filtered thick juice was warmed and then crystallized to sugar. The massecuite was centrifuged. The intial spin-off syrup (molasses) was collected from the centrifuge and sampled. Sugar retained in the basket was washed with 86–96 °C clean water.

For the treated beets, 161.6 kg was available for processing, of which 116.9 kg went into the washing process. Then, 72.6 kg continued to the diffusion process. Subsequently, 8.6 kg of pressed pulp was discarded, and from the other 12.4 kg pressed wet pulp, 1.9 kg could be recovered as dry pulp. Out of the 72.6 kg continuing into the diffusion process, 92.10 kg was retrieved as raw juice (water has been added). Then, 13.8 kg concentrated juice was available, of which 9.1 kg went into the

crystallization process, with finally 2.4 kg molasses recovered and 1532.2 gram of wet sugar. From the wet sugar, 1481.3 gram of dry refined sugar was recovered.

Samples were stored at <-15 °C for up to 53 days prior to analysis. Storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using GC-MS method RR 91-014B with a valid LOQ of 0.01 mg/kg. The method is considered valid for the determination of total fluazifop in sugar beet roots and tops (0.01–1.0 mg/kg). Average concurrent method recoveries were 69–108% (0.01–15 mg/kg) in sugar beet and its processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

Results are shown in Table 261. Residues were found to concentrate in dry pulp and molasses. Residues were reduced in sugar (refined).

Table 261 Residues and processing factors in sugar beets and sugar beet commodities.

Trial, Location, year, (variety), dose rate, interval, DALT Soil type	Sugar beet commodities	Total fluazifop (mg/kg)	PF]	Report Trial no. [ref]
Location ns; UK, 1982, (var ns) EC 250 (racemate) 1 × 1.5 kg ai/ha + 1 × 1.0 kg ai/ha DALT 73 days no field data available	sugar beet roots (RAC)	0.455 [a]	-	PP009B089 981-SBB-EA7 [Atreya, 1982, PP9/0366]
	cosettes	0.625 [a]	-	
	pressed pulp	0.040 [a]	0.087	
	pulp pressed water	0.035 [a]	-	
	raw juice	0.19 [a]	-	
	thin juice	0.195 [a]	-	
	thick juice	2.6 [a]	-	
	green syrup	3.45 [a]	-	
	white sugar	0.02 [a]	0.043	
Ephrata, WA, USA, 2000, (Canyon) EC 240 (P), 2 x 2.1 kg ai/ha interval of 14 days, DALT = 90 days GS: post-emergence; Soil: Sandy Loam	sugar beet roots (RAC)	0.855 [a]	-	RR 00-070B 270 (SBWA1) RS-9307-B1 [Stewart, 2001, 406493]
	sugar (refined)	0.31 [a]	0.36	
	dry pulp	3.35 [a]	40	
	molasses	12.0 [a]	14	

[a] average residue obtained from duplicate analysis of the processed fraction

Processing of asparagus

Processing study 26

Three field trials were conducted to determine the effect of washing and cooking of asparagus [Roper and Graham, 1992, PP5/0584, report RR 92-057B]. Asparagus was treated with 2 × 0.42 kg ai/ha fluazifop-P-butyl with an interval of 21 days and green asparagus spears were harvested 1 day after the last treatment. Details of the trials are summarized in the section residues resulting from supervised residue trials (trials 17-CA-91-321, 15-WA-91-327, 28-MI-91-325).

Washing*:* Bulk samples of asparagus was first trimmed to 7 inch lengths and any broken or undersized spares were removed. The trimmed asparagus were washed in 0.45 kg lots using approximately 3.8 L of water. Subsamples of washed asparagus and wash water were collected for analysis.

Boiling: Washed asparagus were boiled in 0.45 kg lots. These were covered with approximately 600 mL water, heated to boiling and allowed to simmer for 10–15 min. Four lots were cooked for each treatment and the four lots were combined after cooking. Subsamples of cooked asparagus and cooking water were collected for analysis.

Steaming*:* Washed asparagus were cooked by steaming (10–15 minutes) in a steaming pot using approximately 240 mL of water. Four lots of 0.45 kg were steamed for each treatment. The

steamed spears from four lots were combined and the water and steamed spears were taken for analysis.

Microwave cooking: For microwave cooking lots of 0.45 kg of asparagus were cut into pieces of 2.5–5 cm. The cut-up spears were placed in a 2–quart casserole with about 60 mL of water. The casserole was placed in a microwave oven and cooked at high energy for 9-10 minutes. This was done for four lots per treatment and the four lots were combined after microwaving.. Subsamples of the microwaved asparagus were collected for analysis.

Samples were stored at -23 °C or lower for 71–221 days before extraction and another 1–12 days from extraction to analysis. Storage stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using GC-MS method RR 91-014B with a valid LOQ of 0.01 mg/kg. This method is considered valid for the determination of total fluazifop in asparagus (0.01–5.0 mg/kg) and asparagus cooked (at 5.0 mg/kg only). Average concurrent method recoveries were 78–102% (0.01–5.0 mg/kg) in asparagus and its processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

Results are shown in Table 262. Results indicate that residues in asparagus are slightly reduced after boiling, steaming and microwave cooking. Mass balance indicates that these residues are transferred from the spears to the cooking liquid.

Table 262 Residues and processing factors in asparagus commodities

Trial information	Asparagus commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
Stockton, San Joaquin, CA, USA, 1991, (UC157) EC 125 (P) + 0.5% (v/v) NIS, 2 x 0.42 kg ai/ha, interval 21 days, DALT = 1 days, GS: 50 cm; date: 06 March, 1991 Soil: Clay loam	Asparagus (RAC) [a]	5.0 [b]	-	RR 92-057B 17-CA-91-321 [Roper and Graham, 1992, PP5/0584]
	Washed spears	4.8 [b]	-	
	Boiled spears	5.1 [b]	1.0	
	Steamed spears	3.4 [b]	0.68	
	microwave cooked spears	4.7	0.94	
Benton Harbor, Berrie, MI, USA, 1991 (variety ns) EC 125 (P) + 0.5% (v/v) NIS 2 x 0.42 kg ai/ha, interval 21 days, DALT = 1 days, GS: not indicated; Soil: sandy loam	Asparagus (RAC) [a]	1.8 [b]	-	RR 92-057B 28-MI-91-325 [Roper and Graham, 1992, PP5/0584]
	Washed spears	1.0	-	
	Boiled spears	0.9	0.50	
	Steamed spears	1.4	0.78	
	microwave cooked spears	1.3	0.72	
	Wash water from washing	0.02	-	
	Water from boiling	0.8	-	
	Water from steaming	0.4	-	
Ephrata, Grant, WA, USA, 1991, (Comman) EC 125 (P) +0.5% (v/v) X-77, 2 x 0.42 kg ai/ha, interval 21 days, DALT = 1 days, GS: 23 cm; date: 21 April, 1991 Soil: Sandy loam	Asparagus (RAC) [a]	0.6	-	RR 92-057B 15-WA-91-327 [Roper and Graham, 1992, PP5/0584]
	Washed spears	0.4	-	
	Boiled spears	0.2	0.33	
	Steamed spears	0.3	0.50	
	microwave cooked spears	0.4 [b]	0.67	
	Wash water from washing	< 0.01	-	
	Water from boiling	0.2 [b]	-	
	Water from steaming	0.1	-	

[a] Residue level in asparagus from field samples 7 only (supervised residue trial table lists the individual and means for field samples 5, 6, 7). Residue levels from field sample 7 in the table above differ from those reported for the supervised residue trial, because samples were re-analysed prior to processing. 17-CA-91-321 (supervised trial result for field sample 7= 4.1 mg/kg), 28-MI-91-325 (no supervised trial result), 15-WA-91-327 (supervised trial result for field sample 7=0.50 mg/kg).

[b] Results are the means of two replicate analyses on the same sample.

Processing of sugar cane

No processing studies were submitted for sugarcane. Such studies are desirable, since sugarcane bagasse and sugarcane molassis is an animal feed item.

Processing of cotton seed

Processing study 27

In a non-GLP commercial processing study [Atreya *et al*., 1982, PP9/0700, PP009B152] performed in 1981/82 on composite cotton whole seed samples (4.0 kg) from unknown origin. Subsamples of cotton whole seeds were removed prior to processing to provide residues in the RAC.

Hulls: Cotton whole seeds (0.1 kg) were processed in the laboratory to provide hull and meal (i.e. ground seeds without hulls) and establish the distribution of the residue between each. The ratio hull to meal was 46.8 to 53.2 (weight percentage).

Oil processing: Cotton whole seeds (3.9 kg) were dried and ground by a commercial processing factility. The ground seeds were extracted in a soxhlet with n-hexane. Extraction was continued until no colour was apparent in the percolating solvent; the oil content of the remaining cake was then about 2%. The solvent was removed from the cake by warming in a current of air and the cake was sampled for analysis. The crude oil was analysed for free fatty acid content. De crude oils were vigorously agitated at 90 °C with 2% w/v of a 10% solution of phosphoric acid. The crude oil and phosphoric dispersion were cooled to 40 °C and sufficient 14% caustic soda solution was added to neutralise the phosphoric acid and give a 30% excess of alkali. The oil was agitated at 40 °C for 20–30 minutes and the soap formed was allowed to settle. The oil was decanted from the soap layers, washed twice with water and finally with citric acid. The aqueous layers were settled and the oil decanted off. After the final wash, the oil was vacuum dried. Bleaching was accomplished by agitating the oil at 90 °C in the presence of 1% 237 Fullers earth and filtering after 30 minutes contact. Finally, the product was deodorized with a steam distillation of the bleached oil carried out for one hour under 240 °C under vacuum (0.5 mm pressure) with 1% steam/hour.

Aliquots of whole seed samples, hulls, meal, cake (i.e. oil extracted meal), crude oil, refined oil and soapstock were retained for analysis. Storage information was not provided. Samples were analysed for total fluazifop using GC-MS method PP009B152 with a reported LOQ of 0.1 mg/kg. The method is considered insufficiently validated for cottonseed commodities, but is considered not valid for the determination of fluazifop conjugates (no radiovalidation). Duplicate analyses were carried out on all samples. Average concurrent method recoveries were 68–142% (0.1–1.0 mg/kg) in cotton seeds, cotton refined and crude oil, cotton hulls, meal, cake, soapstock, confirming method performance. However, method performance could not be confirmed for dry soya bean seeds (recovery 50% 0.1 mg/kg). Residue levels were corrected for concurrent recoveries; uncorrected values are not reported. Control samples were < 0.1 mg/kg. Residue levels and processing factors are reported in Table 263.

Residue levels and processing factors are reported in Table 263. Processing factors from this study are not taken into account, because no radiovalidation is available for the hydrolysis step used in the analytical method.

Table 263 Residues and processing factors in cotton commodities

Trial information	Cotton commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
USA, 1981 No field report available.	Cotton whole seed	0.41 [a]	-	PP009B152; trial ns [Atreya *et al*., 1982, PP9/0700]
	Cotton hulls	0.20 [a]	0.49 [b]	
	Meal (before extraction)	0.12 [a]	0.29 [b]	
	Cake (i.e. oil extracted meal)	0.31 [a]	0.76 [b]	
	Cotton Crude oil	0.64 [a]	1.6 [b]	
	Soapstock	0.46 [a]	1.1 [b]	
	Cotton refined oil	0.42 [a]	1.0 [b]	

[a] Results are the mean of two replicate analyses

[b] Processing factors are not taken into account, because no radiovalidation is available for the hydrolysis step used in the analytical method.

Processing of oilseed rape seed

Processing study 28

Field grown winter oilseed rape was treated in spring in Germany in 1993 when plants were 20–30 cm high with a spray application of fluazifop-P-butyl (ME formulation) at an application rate of 1 × 0.38 kg ai/ha [Bolygo, 1994, PP5/1105, report RJ1684B]. Details of the trials are summarized in the section residues resulting from supervised residue trials (trials RS-9304-B1 and G1). Seeds were stored at -18 ºC until processing for a maximum period of 170 days. Seeds from two locations were commercially processed into refined oil, simulating industrial processing.

Oil processing: Seeds (8.353/7.240 kg) were dried for about 4 hours at 80 ºC until the moisture level was 6–8%. Dried seeds (8.033/6.940 kg) were cold pressed using a Komet spindle press CA 59, leaving 5.833/5.480 kg oil cake (= meal after cold press) and 1.973/1.210 kg cold pressed crude oil. The oil cake was subsequently extracted with 8 cycles of n-hexane at about 70 ºC (hot extraction) until 90% of the oil was extracted. The separation of the oil n-hexane mixture was made by distillation in a rotary vacuum evaporator. The necessary vacuum was created by means of a waterjet pump so that n-hexane evaporated at temperatures below 50 °C. This resulted in 5.422/5.103 kg oil cake (meal after hot extract) and 1.055/1.174 kg hot extracted crude oil. Refinement was conducted separately for each kind of crude oil, resulting in 0.947/0.290 kg refined oil (cold pressed) and 0.517/0.152 kg refined oil (hot extracted).

Oilseed rape seeds and processed products were stored at -18 ºC prior to analysis for a maximum of 250 and 90 days respectively. Storage stability is considered not an issue for total fluazifop.

Oilseed rape seeds and meal were analysed in duplicate or triplicate for total fluazifop using NMR method RAM 197/02 with a valid LOQ of 0.1 mg/kg. This method is considered valid for the determination of total fluazifop in oilseed rape seeds (0.1 mg/kg only). Average concurrent method recoveries were 74–117% (0.1–1.0 mg/kg) in oilseed rape seed and meal, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.05 mg/kg).

Oilseed rape oil was analysed in duplicate or triplicate for total fluazifop using NMR method RAM 122/02 with a valid LOQ of 0.5 mg/kg for oil. The method is considered valid for the determination of free fluazifop acid (II) in oilseed rape oil (0.5 mg/kg only) but not valid for the determination of fluazifop (II) conjugates (no radiovalidation). Average concurrent recoveries ranged from 95–99% (0.1–0.5 mg/kg) in oilseed rape oil, however precision at 0.1 mg/kg was only 24%, not confirming method performance for free fluazifop acid (II) at levels below 0.5 mg/kg. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.02 mg/kg).

The results are shown in Table 264. Mass balance calculations indicate that most of the fluazifop found in the seeds is accounted for in the meal: 85–99% after cold press or 75–90% after hot extraction. Processing factors for oil are not taken into account because of imprecision of the analytical method at 0.1 mg/kg and because no radiovalidation is available for the hydrolysis step used in the analytical method.

Table 264 Residues and processing factors in processed commodities of rape seed

Trial information	Oilseed commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
Berkenthin, Germany, 1993, (winter oilseed rape: Lirajet)	RAC (seeds) meal after cold press meal after hot extract	3.3 4.5 4.4	- 1.4 1.3	RJ1684B; RS-9306-B1, [Bolygo, 1994,

Trial information	Oilseed commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
ME 125 (P), 1 x 0.38 kg ai/ha, DALT = 116 days, GS: BBA 39, 8 Apr 1993 Soil: Sandy Loam	cold pressed crude oil hot extracted crude oil cold pressed refined oil hot extracted refined oil	0.30 0.31 0.11 0.21	0.09 [a] 0.09 [a] 0.03 [a] 0.06 [a]	PP5/1105]
Neufahrn, Germany, 1993, (winter oilseed rape, Lirabon) ME 125 (P), 1 x 0.38 kg ai/ha, DALT = 101 days, GS: BBA 37, 14 April Soil: Loam	RAC (seeds) meal after cold press meal after hot extract cold pressed crude oil hot extracted crude oil cold pressed refined oil hot pressed refined oil	2.4 2.6 2.4 0.22 0.26 0.08 0.07	- 1.1 1.0 0.092 [a] 0.11 [a] 0.033 [a] 0.029 [a]	RJ1684B; RS-9306-G1; [Bolygo, 1994, PP5/1105]

[a] Processing factors for oil are not taken into account because of imprecision of the analytical method at 0.1 mg/kg and because no radiovalidation is available for the hydrolysis step used in the analytical method.

Processing of sunflower seed

Processing study 29

A GLP commercial processing study was performed in 1989 on sunflower [Alferness and Kleinschmidt, 1991, PP5/0233, report RR91-010B]. Two field trials were conducted in the USA. In each trial broadcast foliar spray applications were made at a rate of 2 ×0.56 kg ai/ha fluazifop-P-butyl and an interval of 14 days. Details of the trials are summarized in the section residues resulting from supervised residue trials (trials 13-TX-89-851, 33-ND-89-852). Mature, marketable seeds were harvested at 66 to 99 DAT. Flower heads were harvested to produce 9.5 -17 kg for processing. After threshing, seed samples were stored frozen within 5 hours after harvest for shipment to the processing facility. Portions of these samples were processed into hulls, meal, crude oil, and refined oil mimicing the typical commercial practices.

Commercial oil processing: After trash removal, sunflower seeds were dried (if needed) until the moisture level was less than 10 percent. Seeds from trial 33ND89-852 contained 20% moisture and were dried in a forced air oven for 65 minutes at 65 °C. The seeds were then hulled to separate the seeds in hulls and kernels. Tap water was added to the kernels to adjust the moisture content to 10 %. The kernels were then cooked to a temperature of 93-104 °C (30-45 minutes). At the end of the cooking period, the oil was removed mechanically with an Anderson expeller. The presscake (i.e. cold press oil extracted meal) and crude oil were collected for analysis. The crude oil was refined by addition of NaOH solution to the crude oil followed by heating for 1 hr at 60-65 °C. The refined oil is decanted and sampled for analysis [AOCS, 1997].

All samples were kept frozen (-0 °C or lower) for up to 6 months. Storage stability is considered not an issue for total fluazifop.

Samples were analysed in duplicate for total fluazifop using modification A of GC-MS method RR89-073B with a valid LOQ of 0.01 mg/kg. The method is considered insufficiently validated for the determination of total fluazifop in sunflower seed commodities. Average concurrent method recoveries were 76-101% (0.01–0.1 mg/kg) in sunflower seed and its processed commodities, confirming method performance. Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

Sunflower seeds, meal and hulls were analysed in duplicate for total CF3-pyridone (X) using method R90-384B with a valid LOQ of 0.01 mg/kg. Sunflower crude and refined oil were analysed in duplicate for total CF3-pyridone (X) using modification A of GC-MS method RR90-384B. The method is considered insufficiently validated for the determination of free CF3-pyridone (X) in sunflower seed commodities and not valid for the determination of CF3-pyridone (X) conjugates because a radiovalidation is lacking. Average concurrent method recoveries were 71-93% (0.01–

0.1 mg/kg) in sunflower seed and its processed commodities, confirming method performance for free CF3-pyridone (X). Residue levels were not corrected for concurrent recoveries. No residues were detected in the control samples (< 0.01 mg/kg).

Residue levels and processing factors are reported in Table 265.

Table 265 Residues and processing factors in sunflower seed commodities

Location, year, (variety), Formulation Dose rate, interval, DALT GS and last application date Soil type	Sunflower commodities	Total fluazifop (mg/kg)	Total CF3-pyridone (X) mg/kg	PF [a]	Report Trial no. [ref]
Petersburg, Hale, TX, USA, 1989, (Large Grey Stripe) EC, 120 (P) + AG98; 2 x 0.56 kg ai/ha, interval 14 days, DALT = 66 days, GS: 12-13 leaves, 19 June, 1989 Soil: not stated	Sunflower seed (RAC)	0.37 [a]	0.01 [a]	-	RR 91-010B 13TX89-851 [Alferness, 1991, PP5/0233]
	Oil extracted meal (cold press)	1.15 [a]	0.02 [a]	3.1	
	Hulls	0.05 [a]	< 0.01 [a]	0.14	
	Sunflower crude oil	< 0.01 [a]	< 0.01 [a]	< 0.03	
	Sunlfower refined oil	< 0.01 [a]	< 0.01 [a]	< 0.03	
Mooreton, Richland, ND, USA, 1989 (Sigco 468) EC 120 (P) + COC t 2 x 0.56 kg ai/ha, interval 14 days DALT: 99 days GS: 10-12 leaves, 22 June, 1989 Soil: not stated	Sunflower seed (RAC)	< 0.01 [a]	< 0.01 [a]	-	RR 91-010B 33ND89-852 [Alferness, 1991, PP5/0233]
	Oil extracted meal (cold press)	< 0.01 [a]	< 0.01 [a]	-	
	Hulls	< 0.01 [a]	< 0.01 [a]	-	
	Sunflower crude oil	< 0.01 [a]	< 0.01 [a]	-	
	Sunlfower refined oil	< 0.01 [a]	< 0.01 [a]	-	

[a] PF is calculated based on total fluazifop in processed commodity divided by total fluazifop in RAC; CF3-pyridone (X) is not taken into account in calculating the processing factor.

[b] Residues are the mean of a duplicate analysis

Processing of coffee beans

Processing study 30

In a GLP study, coffee plants were treated in the field with a spray solution of fluazifop-P-butyl at an application rate of 3 ×0.28 or 3 × 1.4 kg ai/ha (interval 13-14 days) in Hawaii in 2008 [Barney, 2011, PP5_50291, report IR-4 PR 03432 (2011)]. Details of the trials are summarized in the section residues resulting from supervised residue trials (trial 08-HI04). Coffee bean cherries were harvested 1 day after the last application, dried for 2 days in the field and subsequently husked in the field. The resulting green coffee beans (RAC) were sampled for analysis and were further processed by roasting, grinding, extracting and freeze drying.

Coffe bean roasting: Green coffee beans were roasted in a coffee roaster, which consists of a rotating drum that is heated to approximately 208 °C. Smoke and papery silver skin were removed during roasting. During roasting, the beans release water vapour at a rapid rate, sounding as if beans are cracking in unison = “first pop”. This is the light roast. Dark roasters require longer roasting times and are characterised by a second “pop”.

Coffee grinding: A small capacity grinder was used to grind the roasted coffee beans for this processing study.

Extracting: is the actual brewing of ground coffee in boiling water, then filtering out the coffee grounds and retaining the extract for freeze drying.

Freeze drying: This process entails a basic drying process at a low temperature at e.g. -50 °C, then placing the frozen extract into the freeze dryer and obtaining freeze dried coffee powder after about 4 days.

The green coffee beans (RAC) were stored frozen for 934 days at ≤-20 °C. The processed fractions were stored frozen for a maximum time period of 493 days at the same conditions. Storage

stability is considered not an issue for total fluazifop. Samples were analysed for total fluazifop using HPLC-UV method PPRAM62 with a valid LOQ of 0.05 mg/kg. This method is considered valid for the determination of total fluazifop in coffee green beans (0.05–2.0 mg/kg), coffee roasted beans (at 0.05 mg/kg only) and coffee freeze dried (at 0.05 mg/kg only). Average concurrent method recoveries were 77–81% (at 0.05 mg/kg only) in green coffee beans, roasted coffee and freeze dried coffee, confirming method performance. Residues in some processed fractions were corrected with an average overall recovery of 73%; uncorrected results were not available. No residues were detected in the control samples (< 0.05 mg/kg).

Residues were < 0.05 mg/kg total fluazifop in the RAC and processed fractions and therefore, no processing factors could be derived from this study.

Table 266 Residues and processing factors in coffee commodities

Trial information	Coffee commodities	Total fluazifop (mg/kg)	PF	Report Trial no. [ref]
Kauai, HI, USA, 2008, (Red Caturra) EC240 (P) + 0.25% NIS; 3 x 0.28 kg ai/ha interval 13 and 14 days or 3 ×1.4 kg ai/ha, DALT = 1 day GS: ripe berries; 19 Nov soil type: silty clay loam	green beans	< 0.05	-	IR-4 PR 03432 (2011); 08-HI04; [Barney, 2011; PP5_50291]
	roasted beans	< 0.05	-	
	freeze-dried coffee	< 0.05	-	
Kauai, HI, USA, 2008, (Red Caturra) EC240 (P) + 0.25% NIS; 3 x 1.4 kg ai/ha interval 13 and 14 days DALT = 1 day GS: ripe berries; 19 Nov soil type: silty clay loam	green beans	< 0.05	-	idem
	roasted beans	< 0.05	-	
	freeze-dried coffee	< 0.05	-	

Overview of processing factors

Table 267 presents an overview of processing factors which were considered acceptable for refinement of the dietary risk assessment and/or dietary burden calculations. Processing factors are calculated based on total fluazifop in the processed commodity divided by total fluazifop in the RAC.

Table 267 Overview of acceptable processing factors

Commodities	PF (total fluazifop) individual acceptable results	PF (total fluazifop) median or best estimate
Oranges		
- orange juice	< 0.7	< 0.7 (n = 1)
- orange oil	5.0	5.0 (n = 1)
- dried pulp	6.0	6.0 (n = 1)
Brussels sprouts		
- cooked Brussels sprouts	0.34	0.34 (n = 1)
Kale		
- cooked kale	0.20	0.20 (n = 1)
- canned kale	0.29	0.29 (n = 1)
Green pea seeds		
- cooked green peas	0.83, 0.86, 0.94	0.88 (median, n = 3)
- canned green peas (sterilised)	0.58, 0.71, 0.81	0.71 (median, n = 3)
Dry harvested soya bean seeds [a]	-	
- soya bean hulls	0.22, 0.37, 0.38, 0.51, 0.52, 0.65, 0.70	0.51 (median, n = 7)
- soya oil, crude	0.83	0.83 (n = 1)
- soya bean oil extracted meal	0.94, 0.98, 1.1, 1.2, 1.2, 1.3. 1.4	1.2 (median, n = 7)

Commodities	PF (total fluazifop) individual acceptable results	PF (total fluazifop) median or best estimate
- soya bean protein extracted meal	0.06	0.06 (n = 1)
- soya flour	0.80, 1.1, 1.1, 1.1, 1.1, 1.2	1.1 (median, n = 6)
- soya milk	0.090, 0.14, 0.18, 0.21	0.16 (median, n = 4)
- soya diluted protein isolate	< 0.04, < 0.05, 0.036, 0.044,	0.040 (best estimate, n = 2)
Potatoes [a]		
- raw potato peels	0.28, 0.34, 0.53, 0.64, 0.92	0.53 (median, n = 5)
- raw potato flesh	1.0, 1.1, 1.1, 1.1, 1.1	1.1 (median, n = 5)
- cooked potato without peel	0.79, 0.80,	0.80 (median, n = 2)
Sugar beet roots		
- sugar beet sugar (refined)	0.043, 0.36	0.36 (best estimate)
- sugar beet molasses	14	14 (n = 1)
- sugar beet dry pulp	40	40 (n = 1)
- sugar beet wet pulp (pressed pulp)	0.087	0.087 (n = 1)
Asparagus		
- boiled spears	0.33, 0.50, 1.0	0.50 (median, n = 3)
- steamed spears	0.50, 0.68, 0.78	0.68 (median, n = 3)
- microwave cooked spears	0.67, 0.72, 0.94	0.72 (median, n = 3)
Oilseed rape [a]		
- oil extracted meal (cold press)	1.1, 1.4	1.2 (median, n = 2)
- oil extracted meal (hot extraction)	1.0, 1.3	1.2 (median, n = 2)
Sunflower seed		
- oil extracted meal (cold press)	3.1	3.1 (n = 1)
- hulls	0.14	0.14 (n = 1)
- sunflower crude oil	< 0.03	< 0.03 (n = 1)
- sunflower refined oil	< 0.03	< 0.03 (n = 1)

[a] Some processing factors were not taken into account (see text for further details)

Residues in the edible portion of food commodities

The Meeting received information on residues in the edible portions of food commodities for oranges, limes and bananas.

Residues in the edible portion and peels of oranges and limes

The Meeting received data on the distribution between residues in the peel and the pulp for oranges and limes. Since the samples contained residues below the LOQ in both the peel and the flesh, these studies were not summarized here [Culoto and Mallmann, 1985, PP9/0130, report RIC1933 (lime); O'Brien and Harradine, 1987, PP5/0191, report M4533B (orange)]. The residues in the RAC for these trials have been summarized in the supervised residue trials section.

Only one study from Brazil (1981) contained residues where the distribution between peel and pulp could be calculated. This study is summarized in Table 268.

Table 268 Residues of oranges (whole fruit) after pre-harvest treatment at the base of the trees

Location, Country, year (Variety)	Form	kg ai/ha	kg ai/hL	GS and last treatment day;	GSH	DALT (days)	Crop Part	Total fluazifop (mg/kg)	Report; Trial no [ref]
Brazil, 1981	EC 250 (rac)	2 × 1.0	2 × 0.33	ns	ns	7 7 7 14 14 14 28 28 28	peel pulp RAC peel pulp RAC peel pulp RAC	< 0.05 < 0.05 < 0.05 [a] < 0.05 < 0.05 < 0.05 [a] < 0.05 < 0.05 < 0.05 [a]	PP009B117 1Ct/81 ou 6LS [Atreya and Harradine, 1981, PP9/0613] and RJ0291B summary [Atreya and Harradine, 1982, PP9/0062]
idem	EC 250 (rac)	2 × 2.0	2 × 0.67	ns	ns	7 7 7	peel pulp RAC	0.11 < 0.05 0.068 [a]	idem

Location, Country, year (Variety)	Form	kg ai/ha	kg ai/hL	GS and last treatment day;	GSH	DALT (days)	Crop Part	Total fluazifop (mg/kg)	Report; Trial no [ref]
						14	peel	< 0.05	
						14	pulp	< 0.05	
						14	RAC	< 0.05 [a]	
						21	peel	< 0.05	
						21	pulp	< 0.05	
						21	RAC	< 0.05 [a]	
						28	peel	< 0.05	
						28	pulp	< 0.05	
						28	RAC	< 0.05 [a]	

ns = not specified or not stated; rac = racemate of fluazifop-butyl;

[a] Residue levels in the whole orange were calculated from flesh and peel, assuming 30% weight as peel and 70% weight as flesh

Additional trial information:

PP009B117: non-GLP study. Weather conditions, number of trees, application equipment not stated. Spray volume 300 L/ha. Sample sizes are not stated. The peel was removed from the orange and the flesh and peel were analysed separately. Residue levels in the whole orange were calculated from flesh and peel, assuming 30% weight as peel and 70% weight as flesh. Storage for a maximum of 158 days at unstated conditions. Results are the average of four replicate analytical samples. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62 with** in internal standard calibration with a valid LOQ of 0.05 mg/kg. Individual internal standard recovery 71-81% at 0.2 mg/kg for peel or flesh. Control samples were < 0.05 mg/kg.

Residues in the edible portion of bananas

The Meeting received data on the residues in the edible portion of bananas (pulp). The studies were performed in Honduras [Pay, 1987, PP/0185, report M4388B] and Martinique [Culoto and Mallmann, 1985, PP9/0130, report RIC1933]. Fluazifop-butyl (racemate) or fluazifop-P-butyl (R-enantiomer) were applied around the base of the trees. Residue levels were only determined in the edible portion of the fruit and not in the peel and as such not relevant for MRL derivation. The results of the studies are summarized in Table 269.

Study results from report M4388B and RIC1933 cannot be used for MRL derivation or dietary risk assessment since plot sizes and/or sample sizes were not indicated.

Table 269 Supervised field trials on bananas (unbagged; pulp only), treated with fluazifop-butyl at the base of the banana plants

BANANAS Location; Country; year; (variety)	For-mu-lation	no. of appl; (inter val)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop (mg/kg)	Report Trial no. [ref]
Honduras, 1984-1986 (Valery)	EC 125 (P)	1	0.25	ns	All three growth stages [a], 16 October, 1984	L	ripe	41	< 0.04 plp < 0.04 plp < 0.04 plp [SS] [b]	M4388B LaLima (lab) [Pay, 1987, PP/0185]
idem Follow up trial	EC 125 (rac)	6; (30-30-30-30-30)	0.25 0.25 0.25 0.25 0.25 0.25	ns	All 3 growth stages [a] 29 May, 1985	L	ripe	14	< 0.04 plp < 0.04 plp < 0.04 plp [SS] [b]	idem
idem Follow up trial	EC 125 (rac)	11; (30-30-30-30-30-	0.25 0.25 0.25 0.25	ns	All three growth stages [a], 21 Jan,	L	ripe	29	< 0.04 plp < 0.04 plp < 0.04 plp	idem

BANANAS Location; Country; year; (variety)	For-mu-lation	no. of appl; (inter val)	kg ai/ha	kg ai/hL	GS and last treatment day	Soil type	GSH	DALT (days)	Total fluazifop (mg/kg)	Report Trial no. [ref]
		30-30-30-30-30)	0.25 0.25 0.25 0.25 0.25 0.25 0.25		1986				[SS] [b]	
Irfa, Martinique, France (Carribean), 1984 (Mura accuminata)	EC 250 (rac)	1	0.50	ns	GS ns; 16 May	ns	ns	2 8 15 30	< 0.05 plp < 0.05 plp < 0.05 plp < 0.05 plp [SS]	RIC1933 [Culoto and Mallmann, 1985, PP9/0130]
idem	EC 250 (rac)	1	1.0	ns	GS ns; 16 May	ns	ns	2 8 15 30	< 0.05 plp < 0.05 plp < 0.05 plp < 0.05 plp [SS]	idem

plp = residues in the pulp

[a] Bananas have a continual harvest, therefore three generations represent all the time. The trials first received one application and ripe bananas were harvested 41 days later. Over the next 6 months, a further 5 applications were made and ripe bananas were harvested 14 days after the last application. Over the next 8 months, a further 5 applications were made and ripe bananas were harvested 29 days after the last application.

[b] Results came from three replicate trials.

[SS] Sample size not reported; result from this trial cannot be selected for MRL derivation

Additional trial information:

M4388B. non GLP. Weather conditions not reported. Plot sizes 2000 m^2. Application equipment and spray volume not reported. Sample size not reported. Storage at -18° C or lower for a maximum of 1 year. Samples were analysed for total fluazifop using **HPLC-UV method PPRAM 62/2**. Samples were corrected for average internal standard recovery (83% at 0.2–0.5 mg/kg). Control samples were < 0.04 mg/kg.

RIC1933, non GLP. Weather conditions not reported. Plot sizes not reported. Application equipment and spray volume not reported. Sample size not reported. Storage at -18 °C for unknown period. Samples were analysed for total fluazifop using HPLC-UV method PPRAM 62/1.Samples were not corrected for average concurrent method recovery (85% at 0.1–0.3 mg/kg). Control samples were < 0.05 mg/kg.

RESIDUES IN ANIMAL COMMODITIES

Direct animal treatments

Not relevant for the present use pattern.

Farm animal feeding studies

The Meeting received information on feeding studies in dairy cows and laying hens.

Feeding studies with dairy cows

In a non-GLP study, dairy cows (Friesian; 4-9 years; 420-625 kg; n = 15) were fed twice daily with a basal diet (n = 3) or with a diet containing fluazifop-butyl (RS) at nominal levels of 0.2, 0.8, 3.0 and 12.0 ppm dry food (n = 3 per group) during 29 consecutive days [Atreya *et al*, 1981, PP9/0182, report RJ0215B]. Actual levels were 111%, 107%, 104% and 102% of the intended dose of 0.2, 0.8, 3.0 and 12.0 ppm dry feed. Treatment had no effect on milk yield, bodyweight, food consumption or on general health of the cows. Milk was sampled with 1-3 days intervals within the trial period. The AM

and PM milk was bulked per animal. Two cows per group were slaughtered 24 hrs after the last dose and the remaining cow was fed untreated diet for a further 7-8 days before it was slaughtered. Samples (2 ×500 g) were taken from milk, liver, kidney, muscles and fat. Samples were stored frozen at -20 ± 2 °C. Milk samples were analysed after maximally 4 months of storage; liver samples after maximally 3.5 months; kidney samples after maximally 4 months; muscle samples after 4 months with duplicate analysis for some samples after 5 months; fat samples after maximally 4.5 months [Swain, 2009, PP9_50000, report T008915/08].

Individual tissue samples as well as individual and daily bulked milk samples were analysed for fluazifop residues using HPLC-UV and GC-MS method PPRAM 61. Residues of parent compound and free fluazifop are analysed separately from lipophilic conjugates of fluazifop in milk and tissues. In fat the total fluazifop residues were measured as a single analyte. All residues are expressed as fluazifop. Samples were not corrected for average concurrent recoveries (88–121% at 0.05–0.1 mg/kg). Control samples were < 0.01 mg/kg total fluazifop in milk (n = 10) and < 0.02 mg/kg total fluazifop in tissues, except in kidney and muscle (< 0.02–0.02 mg/kg eq).

Fluazifop-butyl was not found (< 0.01 mg/L eq) in individual and bulk samples of milk at any of the feeding levels. Free fluazifop was found at levels of 0.01 mg/L in 4 out of 14 bulk milk samples at the 12 ppm feeding level (not shown in the table), while the individual milk samples showed no residues (< 0.01 mg/L). Lipophilic fluazifop conjugates reached mean plateau levels of 0.042 and 0.15 mg/L eq within three days at the 3 and 12 ppm feeding levels, respectively (see Table 270).

Lipophilic fluazifop conjugates were not found (< 0.02 mg/kg eq) in the tissue samples at any of the feeding levels. Polar fluazifop related residues (fluazifop-butyl, fluazifop and polar fluazifop conjugates) were only found in the highest dose group with maxima of 0.13, 0.03, 0.03 and 0.06 mg/kg eq in kidney, liver, cardiac muscle or peritoneal fat (see Table 271). Residues in fat represent total fluazifop residues (with unknown composition). The results further indicate that the total fluazifop residues do not accumulate and rapidly decline after the application of the fluazifop containing diet has stopped.

Table 270 Lipophilic fluazifop conjugates (expressed as fluazifop) in the bulked and individual milk samples (mg/L eq)

Group:	B	C	D				E			
Fluazifop-butyl (RS) Feeding rate (mg ai/kg food):	0.2	0.8	3				12			
Cow number	bulk sample	bulk sample	bulk sample	10	11	12	bulk sample	13	14	15
Day:										
-1	n.a.	n.a.	< 0.01	n.a.	n.a.	n.a.	< 0.01	< 0.01	< 0.01	< 0.01
1	n.a.	< 0.01	< 0.01	n.a.	n.a.	n.a.	0.02	n.a.	n.a.	n.a.
3	n.a.	< 0.01	0.05	n.a.	n.a.	n.a.	0.16	n.a.	n.a.	n.a.
5	n.a.	0.01	0.05	< 0.01	< 0.01	0.04	0.14	0.04	0.07	0.10
8	n.a.	0.01	0.04	n.a.	n.a.	n.a.	0.12	n.a.	n.a.	n.a.
12	n.a.	0.01	0.05	0.01	0.02	**0.07**	0.12	0.11	0.08	0.09
17	n.a.	0.01	0.01	n.a.	n.a.	n.a.	0.13	n.a.	n.a.	n.a.
23	n.a.	0.01	0.03	0.01	0.02	0.05	**0.18**	0.06	0.09	0.05
26	n.a.	< 0.01	0.05	n.a.	n.a.	n.a.	0.15	n.a.	n.a.	n.a.
28	n.a.	n.a.	0.06	n.a.	n.a.	n.a.	0.17	0.14	0.13	0.10
29	n.a.	< 0.01	0.04	0.03	0.04	0.05	0.14	0.12	0.09	0.13
30	n.a.	n.a.	n.a.	n.a.	0.04	< 0.01	n.a.	n.a.	0.14	< 0.01
31	n.a.	n.a.	n.a.	n.a.	n.a.	< 0.01	n.a.	n.a.	n.a.	< 0.01
32	n.a.	n.a.	n.a.	n.a.	n.a.	< 0.01	n.a.	n.a.	n.a.	< 0.01
Mean day 3-29	**n.a.**	**0.01**	**0.042**				**0.15** [a]			
Max day 3-29	**n.a**	**0.01**	**0.07**				**0.18** [a]			

n.a. = not analysed

[a] At the 12 ppm feeding level, < 0.01–0.01 mg/kg free fluazifop was detected. This should be added to this level.

Table 271 Polar fluazifop related residues (fluazifop-butyl, fluazifop, polar conjugates), expressed as fluazifop, in individual cow tissues (mg/kg eq)

Group:	Cow number:	Fluazifop-butyl (RS) Feeding rate (mg ai/ kg food):	Liver	Kidney	Adductor muscle	Pectoral muscle	Cardiac muscle	Subcu-taneous fat [a]	Peritoneal fat [a]
A	1	control	< 0.02	**0.02**	< 0.02	**0.02**	< 0.02	< 0.02	< 0.02
	3		< 0.02	< 0.02	n.a.	n.a.	n.a.	n.a.	< 0.02
B	4	0.2	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	5		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	6*		n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
C	7	0.8	< 0.02	< 0.02	n.a.	n.a.	n.a.	n.a.	n.a.
	8		< 0.02	< 0.02	n.a.	n.a.	n.a.	n.a.	n.a.
	9*		< 0.02	< 0.02	n.a.	n.a.	n.a.	n.a.	n.a.
D	10	3	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
	11		< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
	12*		< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
E	13	12	**0.03**	**0.13**	< 0.02	< 0.02	**0.02**	**0.03**	**0.06**
	14		**0.03**	**0.06**	< 0.02	< 0.02	< 0.02	**0.02**	**0.05**
	mean (13and14)		**0.03**	**0.10**	< 0.02	< 0.02	**0.02**	**0.025**	**0.055**
	15*		< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02

n.a. = not analysed

* = animals which were allowed a seven day recovery period

[a] residues in fat represent total fluazifop (polarity unknown).

Feeding studies with laying hens

In a non-GLP study hens (22–24 weeks; 1.2–2.5 kg; n = 160 females and 16 males) were fed once daily with a basal diet or with a diet containing fluazifop-butyl (RS) (40 females and 4 males per feeding group) during 28 days [Swaine and Francis, 1981, PP9/0183, report RJ0217B]. The actual amounts of fluazifop-butyl in the feed of the 4 groups were 0, 0.4, 2.5 and 10.3 mg/kg. Eggs were sampled at intervals throughout the trial (n = 10 per group) and albumen and yolk were separated. Eggs from the same treatment group and day were pooled. No effects on body weights, food consumption or egg production were observed during treatment. Hens were sacrificed on days 21 and 28 (n = 6 per group). The report does not state the period after last treatment. The remaining hens were fed untreated diet for 7 and 14 days and they were sacrificed on day 35 and 42 (n = 6 per group). Fat and mixed samples of muscle with skin and underlying fat from the left pectoral region and left leg were taken after sacrifice. Tissue samples were pooled per group and minced, except for day 28, where samples for individual animals were minced. Samples were stored at -20 ± 2 °C for less than 4 months [Swain, 2009, PP9_50001, report T008916/08].

Samples of mixed tissues (muscle, skin and fat) and liver, and egg samples were analysed using HPLC-UV and GC-MS method PPRAM 58. Average recovery values for tissue and egg samples at 0.02–0.2 mg/kg ranged between 88–121%. Samples were corrected for average recoveries and uncorrected residue levels were nr in the study. Control samples were < 0.01 mg/kg eq for muscle and fat (GC-MS) and < 0.02 mg/kg eq for eggs, yolks, albumen and liver (HPLC-UV).

The results of the analysis of eggs and tissues are presented in Tables 272 and 273. Residue levels in eggs were only measurable in eggs from hens treated with the highest dose at 10.3 ppm and the plateau level reached was 0.04 mg/kg eq at day 7. After separation of yolk and albumen, residues were detected only in the yolk (maximum of 0.11 mg/kg eq). The mixed tissues (muscle, fat and skin) and the liver samples of hens treated with the highest dose of 10.3 ppm contained residues in the range of 0.01–0.04 mg/kg eq and 0.03–0.13 mg/kg eq, respectively. Residues declined rapidly when the birds returned to an untreated diet.

Table 272 Residues of total fluazifop (expressed as fluazifop) in egg samples.

Group:	B	C	D
Fluazifop-butyl (RS) Feeding rate (ppm dry feed):	0.4	2.5	10.3
	Whole eggs Total fluazifop mg/kg eq	Whole eggs Total fluazifop mg/kg eq	Whole eggs Total fluazifop mg/kg eq
Day			
-1	n.a.	< 0.02	< 0.02 (whole egg)
1	n.a.	< 0.02	< 0.02 (whole egg)
3	n.a.	n.a.	- 0.02 (yolk) 0.02 (albumen) 0.02 (whole egg) [a]
7	n.a.	< 0.02	0.04 (whole egg)
15	n.a.	< 0.02	0.04 (whole egg)
17	n.a.	n.a.	0.05 (yolk) < 0.02 (albumen) 0.03 (whole egg) [a]
21	n.a.	< 0.02	0.04 (whole egg)
25	n.a.	n.a.	0.11 (yolk) < 0.02 (albumen) 0.05 (whole egg) [a]
27	n.a.	< 0.02	0.04 (whole egg)
31	n.a.	n.a.	0.08 (yolk) < 0.02 (albumen) < 0.02 (whole egg) [a]
35	n.a.	< 0.02	0.02 (whole egg)
42	n.a.	< 0.02	< 0.02 (whole egg)
Average (day 7-27)	n.a.	< 0.02	0.04 (whole egg)
Maximum (day 7-27)			0.05 (whole egg)

n.a. = not analysed

[a] Calculated assuming mass fractions in egg white (31.6 g) and egg yolk (15.6 g) as used in the laying hen metabolism study

Table 273 Residues of total fluazifop (expressed as fluazifop) in hen tissues

Group	Fluazifop-butyl (RS) Feeding rate (ppm dry feed)	Day	Hen number	Mixed tissues (muscle, fat and skin) total fluazifop mg/kg eq	Liver total fluazifop mg/kg eq
A	control	-	-	< 0.01	< 0.02
B	0.4	-	-	n.a.	n.a.
C	2.5	21	bulked (n = 6)	0.01	**0.050**
		28	bulked (n = 6)	**0.015**	< 0.02
		28	96	n.a.	< 0.02
		28	105	n.a.	**0.04**
		28	115	n.a.	< 0.02
		28	123	n.a.	< 0.02
		28	124	n.a.	< 0.02
		28	1403	n.a.	< 0.02
		35	bulked (n = 6)	< 0.01	< 0.02
D	10.3	21	bulked (n = 6)	**0.020**	**0.075**
		28	bulked (n = 6)	**0.020**	**0.060**
		28	136	< 0.01[a]	< 0.02
		28	143	**0.01**	**0.03**
		28	152	**0.01**	**0.08**
		28	167	**0.04**	**0.05**
		28	171	**0.02**	**0.06**
		28	172	**0.02**	**0.13**
		35	bulked (n = 6)	**0.015**	< 0.02

Group	Fluazifop-butyl (RS) Feeding rate (ppm dry feed)	Day	Hen number	Mixed tissues (muscle, fat and skin) total fluazifop mg/kg eq	Liver total fluazifop mg/kg eq
		42	bulked (n = 6)	**0.015**	< 0.02

n.a. = not analysed

Residues in food in commerce or at consumption

No data submitted; no data required for the current uses.

National residue definitions

No information provided.

APPRAISAL

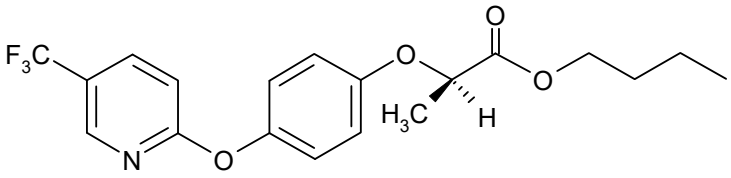
Fluazifop-P-butyl was scheduled for residue evaluation as a new compound by the 2015 JMPR at the 46th Session of the CCPR (2014). Because the dossier was considered incomplete at the start of the 2015 JMPR, the evaluation was postponed until the 2016 JMPR. Fluazifop-P-butyl is used for the post-emergence control of grass (graminaceous) weeds in a wide range of broad-leaved crops. Fluazifop-P-butyl is quickly absorbed across leaf surfaces. Its hydrolysis product, fluazifop-P-acid (or fluazifop-P), then distributes throughout the plant through both xylem and phloem transport and accumulates in the meristem tissue of the growing points of both shoots and roots. The speed of the herbicidal action increases with weed vigour.

The Meeting received information from the manufacturer on identity, metabolism, storage stability, residue analysis, use pattern, residues resulting from supervised trials on various crops, fate of residue during processing, and livestock feeding studies.

Chemical name

Fluazifop-P-butyl

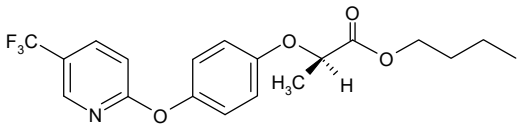
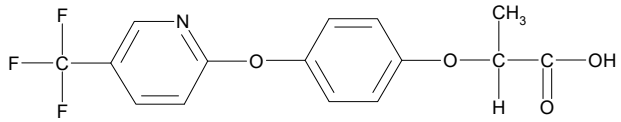
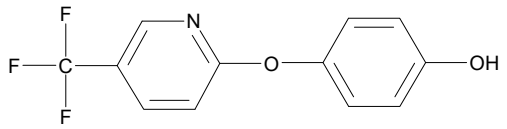
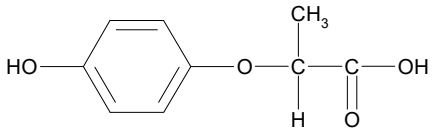
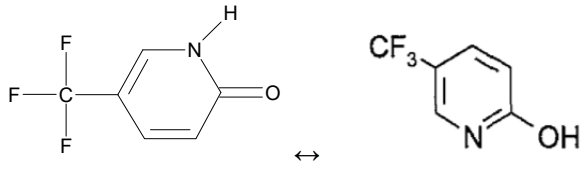
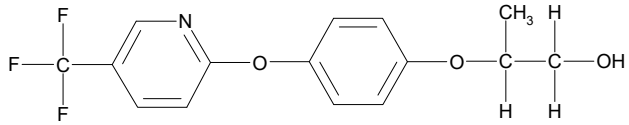
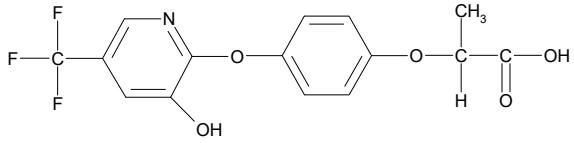
Butyl (R)-2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy]propionate



Fluazifop-P-butyl is the active purified (resolved) R-enantiomer of the racemate (RS)-fluazifop-butyl. This R-enantiomer possesses the majority of the herbicidal activity. The enantiomeric purity of fluazifop-P-butyl is 96–99% R-enantiomer and 1–4% S-enantiomer. The chiral carbon atom of the R-enantiomer is indicated in the figure above.

Fluazifop-butyl (racemate) contains R and S-enantiomers in a 50:50 w/w ratio. The biological activity of the racemate is due primarily to the R-enantiomer which gives equal herbicidal activity at half the rate of racemic (RS)-fluazifop-butyl. A formulation based on the racemate was marketed first and was replaced by a formulation based on the R-enantiomer in 1984. Several of the available studies have been performed with the racemate.

Compounds referred to in the appraisal:

Fluazifop-P-butyl (I) MW 383.4	
Fluazifop-P-acid (II) MW 327.3	
Pyr-Ph ether (IV) MW 255.20	
Despyridinyl acid (III) MW 182.17	
CF3-pyridone (X) MW 163.10	
Fluazifop alcohol (34, XXXIV)	
Hydroxyfluazifop acid (XL) MW 343.3	

Plant metabolism

The Meeting received plant metabolism studies for fluazifop-butyl after soil directed or foliar applications on fruits and fruiting vegetables (grapes and cucumbers), stem and leafy vegetables (lettuce, celery, and endive), cereals (maize), pulses and oilseeds (alfalfa, cotton, oilseed rape, and soya bean) and root and tuber vegetables (carrot, potato tubers, and sugar beet roots). All radioactive residue levels in the metabolism studies are expressed as fluazifop-butyl equivalents, and all percentages are expressed as %TRR (total radioactive residues) in the specified commodity. As a large number of metabolism studies were received, they were summarized together.

In most crop commodities, residues could be extracted sequentially by acetonitrile and acetonitrile/water at levels > 80%, except cotton forage 78–95%, carrot roots 61–88%, and carrot foliage 40–74%. Oilseed commodities were sequentially extracted by hexane, diethyl ether or dichloromethane, acetonitrile/water and methanol or water at levels > 80%, except cotton seeds 64–65% TRR.

Organo-soluble and polar conjugates present in the extracts were cleaved by alkaline or acid hydrolysis to investigate to which exocon they were attached. In some commodities the remaining solids were hydrolysed as well. The Meeting noted that harsh hydrolysis conditions can lead to degradation of fluazifop-butyl and its metabolites, and interpretation of the metabolism studies needs

to take this into account. Studies showed that fluazifop acid remained intact (92–96% recovery) with 0.1 M NaOH for 1–3 hrs reflux or 6 M HCl for 6 hrs 60 ºC (90% recovery). Higher alkaline concentrations (1 M NaOH) or higher temperatures under acid conditions (reflux in 1–6 M HCl) resulted in degradation of fluazifop acid into CF3-pyridone (X) and despyridinyl acid (III). CF3-pyridone degraded under alkaline hydrolysis conditions, but remained intact with 6 M HCl for 1 hr reflux (93% recovery). Stability of despyridinyl acid (III), Pyr-Ph ether (IV) and hydroxyfluazifop acid (XL) under these conditions has not been investigated, but is desirable.

Since fluazifop-P-butyl could possibly convert to S-enantiomeric forms during hydrolysis or metabolism, the Meeting considered epimerisation studies in plants. Epimerisation of [^{14}C]phenyl-fluazifop-butyl R- or S-enantiomers was studied in lettuce and cotton plants treated with a topical leaf and stem spot application. Plants were harvested 27 days later and then extracted and hydrolysed. The R/S ratio remained unchanged for fluazifop acid, indicating that no epimerisation occurred in the plant or during sample extraction and acid or alkaline hydrolysis. Contrary, analysis of samples from supervised trials treated with fluazifop-butyl (RS) showed an increase in the proportion of the fluazifop acid R-enantiomer with a crop to crop variation in the rate and content of conversion. The R-enantiomer proportion of the total fluazifop remained approximately the same in carrot roots at 21 days after treatment (46–54%), but increased to 74–82% in apple at 35–49 days after treatment, 78% in head cabbage at 49 days after treatment, 62% in kale at 27–41 days after treatment, 69–77% in dry peas at 54 days after treatment and 76–84% in oilseed rape seeds. Since fluazifop-P-butyl (96–99% R-enantiomer) is currently the only compound that is available in trade, it is unlikely that S-enantiomeric levels will be higher than specified for fluazifop-P-butyl in the FAO JMPS specifications.

Translocation studies showed that fluazifop-butyl derived residues translocated rapidly throughout the plants. In a study on cucumbers following a single foliar application, a high proportion of the residues (88%) was present on the peel of the fruit after 1 day, while residues had distributed evenly between the peel and flesh after 14 days. Whole plant autoradiograms of soya bean plants following a topical leaf application or a topical stem injection showed very little translocation after 1 day, but the radiocarbon had spread throughout the soyaplants including the roots and the new growth after 7–14 days.

Fluazifop-P-butyl may be applied:

- to grass-like crops as a desiccant (grass seed production) or as a ripener to increase the sucrose concentration (sugar cane)
- as a weed directed spray application at the base of trees, shrubs or vines or a banded inter-row soil application to field crops
- as a broadcast or banded (over-the-top) foliar application to various crops.

Treatment of grass-like crops was studied in maize plants. Fluazifop-P-butyl is an herbicide effective against graminaceous weeds and is therefore phytotoxic to cereals and grasses. This was confirmed in a metabolism study on maize plants where maize plants died after 13–28 days after a topical leaf or stem application. In a study, where maize plants were stem injected with [^{14}C]phenyl-fluazifop-butyl (RS) at an unknown dose rate, only parent and fluazifop acid were identified in the extracts. Parent compound decreased from 15–40% AR at 1–7 days to 5% AR at 14–28 days. Free fluazifop acid decreased from 65% at Day 1 to 5% AR at Day 28. The presence of other compounds was not investigated and hydrolysis was not conducted. Fluazifop-butyl and fluazifop acid were the only compounds found in cereal forage.

Application around the base of trees or shrubs was studied on grape vines. Field grown grapes were sprayed at the base of the vine with a mixture of [^{14}C]phenyl- and [^{14}C]pyridyl-fluazifop-P-butyl (R-enantiomer). The vine was treated with 1–2 applications at 0.84 kg ai/ha with an interval of 71 days, whereby the first application was at early bunch formation. Total radioactive residues (TRR) in immature grape berries at DAT 21, 30, 45, 60 after a single treatment and mature grape berries at DAT 14, 30 after a double treatment were 0.004–0.009 mg/kg eq. Composition of the residue was not further investigated. This study indicates that application of fluazifop-P-butyl around the base of trees,

shrubs or vines is not expected to result in significant residues in the fruits (or nuts) as long as the spray does not reach the fruits (or nuts).

Foliar applications to various crops were investigated in 21 different metabolism studies. These are summarized in the table below. Studies, where severe hydrolysis conditions are used are not taken into account for residue characterisation.

In addition, individual compounds were analysed in samples treated with fluazifop-butyl (RS) in supervised residue trials. Fluazifop-butyl (parent compound) is found at significant quantities at the day of application, but is found at low levels (up to 0.1 mg/kg) up to 8 days in fruits, up to 12 days in roots, up to 16 days in oilseed forage and up to 98 days in root forage. In trials on celery, where extraction conditions did not degrade fluazifop acid, low levels (0.06–0.08 mg/kg) of total CF3-pyridone (free and conjugates) were found up to 30 days after application. Total fluazifop (i.e. sum of fluazifop-butyl, fluazifop acid and its conjugates, expressed as fluazifop acid) levels ranged from 1.2–2.7 mg/kg for these samples. When corrected for molecular weight (× 327.3/163.10 = 2.01), levels of CF3-pyridone were equivalent to 5.2–13% of total fluazifop in celery stems. This is similar to levels found in the celery metabolism study.

							Parent (I) and metabolites as %TRR								
No	Crop	Treatment	Label		PHI	TRR Mg/kg	I	II	I + II	IV	III	X	34	XL	NH
2	Cucumber	1 × 0.50 kg ai/ha foliar spray	Ph	RS	1	1.3	7.3	69	76	-	–	Nr	Na	Na	24
2	Cucumber	1 × 0.50 kg ai/ha foliar spray	Ph	RS	14	4.9	–	72	72	–	3.3	Nr	Na	Na	17
2	Cucumber	1 × 0.52 kg ai/ha foliar spray	Py	RS	14	2.4	–	69	69	–	Nr	–	Na	Na	11
9	Rape seeds	1 × 0.84 kg ai/ha; topical leaf and stem and soil	Py	RS	70–91	0.65	–	69	69	–	Nr	D (–)	Na	Na	6
15	Soyaseeds	1 × 1.0 kg ai/ha; Broadcast pods present	Ph	RS	63	11	–	77	77	Na	3.7	Nr	Na	Na	10
15	Soyaseeds	1 × 1.0 kg ai/ha; Broadcast pods present	Ph	RS	43	6.0	–	81	81	Na	Na	Nr	Na	Na	13
16	Soyaseeds	1 × 0.56 kg ai/ha; Broadcast BBCH 15	Ph	R	104	0.04	–	50	50	–	2.3	Nr	Na	Na	19
16	Soyaseeds	1 × 0.56 kg ai/ha; Broadcast BBCH 15	Py	R	104	0.09	–	40	40	–	Nr	D (–)	Na	Na	24
16	Soyaseeds	0.56 + 0.21 kg ai/ha; Broadcast BBCH 69	Ph	R	82	0.57	–	57	57	–	3.9	Nr	Na	Na	13
16	Soyaseeds	0.56 + 0.21 kg ai/ha; Broadcast BBCH 69	Py	R	82	1.0	0.2	59	60	–	Nr	D 0.9	Na	Na	16
17	Carrot roots	1 × 0.25 kg ai/ha; broadcast	Ph	R	45	0.15	–	63	63	–	6.4	Nr	–	Na	12
17	Carrot roots	1 × 0.53 kg ai/ha; broadcast	Ph	RS	45	0.18	–	46	46	–	4.8	Nr	13	Na	23
17	Carrot roots	1 × 0.51 kg ai/ha; broadcast	Py	RS	45	0.33	–	44	44	–	Nr	1.0	11	Na	25
18	Carrot roots	1 × 0.42 kg ai/ha; broadcast	Ph	R	20	0.38	0.5	62	62	–	13	Nr	Na	Na	25
18	Carrot roots	1 × 0.43 kg ai/ha broadcast	Py	R	20	0.54	–	49	49	–	Nr	37	Na	Na	14
18	Carrot roots	0.42 + 0.42 kg ai/ha; broadcast	Ph	R	45	0.091	–	64	64	–	18	Nr	Na	Na	19
18	Carrot roots	0.42 + 0.42 kg ai/ha; broadcast	Py	R	45	0.13	–	59	59	–	Nr	29	Na	Na	12

							Parent (I) and metabolites as %TRR								
No	Crop	Treatment	Label		PHI	TRR Mg/kg	I	II	I + II	IV	III	X	34	XL	NH
19	Potato tubers	1 × 0.86 kg ai/ha; topical leaf and soil	Ph	RS	56	0.37	–	42	42	–	18	Nr	Na	13	15
19	Potato tubers	1 × 0.84 kg ai/ha; topical leaf and soil	Py	RS	56	0.29	–	25	25	–	Nr	–	Na	15	30
20	Sugar beet roots	1 × 2.8 kg ai/ha; topical leaf and soil	Ph	RS	87	0.049	–	25	25	–	18	Nr	Na	Na	4
21	Sugar beet roots	1 × 0.25 kg ai/ha; broadcast	Ph	R	90	0.09	–	52	52	–	17	Nr	–	Na	18
21	Sugar beet roots	1 × 0.52 kg ai/ha; broadcast	Ph	RS	90	0.08	–	40	40	–	15	Nr	–	Na	17
21	Sugar beet roots	1 × 0.51 kg ai/ha; broadcast	Py	RS	90	0.20	–	34	34	–	NR	IC 3.4	–	Na	18
4	Celery stems	0.45 + 0.18 kg ai/ha; broadcast	Ph	R	30	0.05	–	43	43	Na	18	Nr	1.0	4.4	1
4	Celery stems	0.42 + 0.36 kg ai/ha; broadcast	Py	R	30	0.08	–	39	39	Na	Nr	2.7	–	1.2	8
3	Lettuce	1 × 0.45 kg ai/ha; topical leaf and stem	Ph	R	27	NA	52	19	71	0.4	8.7	Nr	–	Na	5
3	Lettuce	1 × 0.45 kg ai/ha; Topical leaf and stem	Ph	S	27	NA	49	19	68	1.7	4.1	Nr	5.3	Na	7
5	Endive	1 × 0.42 kg ai/ha; broadcast	Ph	R	20	0.65	–	48	48	11	25	Nr	Na	Na	3
5	Endive	1 × 0.42 kg ai/ha; broadcast	Py	R	20	0.88	–	37	37	25	Nr	14	Na	Na	12
5	Endive	0.42 + 0.42 kg ai/ha; broadcast	Ph	R	28	1.4	–	49	49	0.5	40	Nr	Na	Na	1
5	Endive	0.42 + 0.42 kg ai/ha; broadcast	Py	R	28	1.8	–	43	43	–	Nr	11	Na	Na	2
4	Celery leaves	0.45 + 0.18 kg ai/ha; broadcast	Ph	R	30	0.31	2.4	52	54	Na	7.1	Nr	0.3	1.6	–
4	Celery leaves	0.42 + 0.36 kg ai/ha; broadcast	Py	R	30	0.64	–	63	63	Na	Nr	14	–	0.7	6
11	Maize forage	Dose rate ns; topical stem injection	Ph	RS	1	NA	15	65 fr	80	Na	Na	Nr	Na	Na	20
11	Maize forage	Dose rate ns; topical stem injection	Ph	RS	7	NA	40	25 fr	65	Na	Na	Nr	Na	Na	35
6	Alfalfa forage	1 × 0.49 kg ai/ha; foliar spray	Ph	RS	20	3.2	–	70	70	–	–	Nr	Na	Na	6
6	Alfalfa forage	1 × 0.49 kg ai/ha; foliar spray	Py	RS	20	2.5	–	70	70	–	Nr	Na	Na	Na	6
6	Alfalfa forage	1 × 0.49 kg ai/ha; foliar spray	Ph	RS	87	0.13	–	37	37	–	–	Nr	Na	Na	13
7	Cotton forage	1 × 0.45 kg ai/ha; topical leaf and stem	Ph	R	27	NA	24	38	61	2.7	7.3	Nr	–	Na	11
7	Cotton forage	1 × 0.45 kg ai/ha; topical leaf and stem	Ph	S	27	NA	23	56	79	2.5	1.5	Nr	–	Na	6
10	Soyaforage	1 × 0.75 kg ai/ha; topical leaf and stem	Ph	RS	1	NA	15	40 fr	55	Na	Na	Nr	Na	Na	45
10	Soyaforage	1 × 0.75 kg ai/ha; topical leaf and stem	Ph	RS	2	NA	1.0	50 fr	51	Na	Na	Nr	Na	Na	49
10	Soyaforage in nutrient	1 × 0.75 kg ai/ha; topical leaf and stem	Ph	RS	6	NA	–	76	76	Na	Na	Nr	Na	Na	12
10	Soyaforage in nutrient	1 × 0.75 kg ai/ha; topical leaf and stem	Ph	RS	29	NA	–	15	15	Na	Na	Nr	Na	Na	85
11	Soyaforage	Dose rate ns; topical stem injection	Ph	RS	1	NA	65	15 fr	80	Na	Na	Nr	Na	Na	20
16	Soyaforage	1 × 0.56 kg ai/ha; Broadcast BBCH 15	Ph	R	22	5.2	0.2	71	72	0.3	–	Nr	Na	Na	2
16	Soyaforage	1 × 0.56 kg ai/ha; Broadcast	Py	R	22	4.3	–	70	70	0.2	Nr	D 0.2	Na	Na	3

							Parent (I) and metabolites as %TRR								
No	Crop	Treatment	Label		PHI	TRR Mg/kg	I	II	I + II	IV	III	X	34	XL	NH
		BBCH 15													
18	Carrot foliage	1 × 0.42 kg ai/ha; broadcast	Ph	R	20	0.86	–	82	82	–	1.7	Nr	Na	Na	16
18	Carrot foliage	1 × 0.42 kg ai/ha; broadcast	Py	R	20	1.3	–	42	42	–	Nr	48	Na	Na	10
18	Carrot foliage	0.42 + 0.42 kg ai/ha; broadcast	Ph	R	45	1.0	–	82	82	–	5.9	Nr	Na	Na	13
18	Carrot foliage	0.42 + 0.42 kg ai/ha;Broadcast	Py	R	45	1.5	–	47	47	–	Nr	31	Na	Na	22

No = number of the study, referring to the study number in the evaluation

I = parent, II = fluazifop acid, IV = Pyr-Ph ether; III = despyridinyl acid, X = CF3-pyridone, 34 = fluazifop alcohol, XL = hydroxyfluazifop acid

NH = extracted or solid fractions not subjected to hydrolysis, may contain some additional II, IV, III, X conjugates

IC = incomplete hydrolysis

fr = free fluazifop acid only—no hydrolysis conducted

D = degraded

nr = not relevant (compound doesn't contain the label)

na = not analysed (presence not verified)

– = not detected

Py = pyridinyl label

Ph = phenyl label

R = R-enantiomer

S = S-enantiomer

RS = racemate

bold indicates > 0% TRR

These studies show that metabolism is similar in all five crop categories, but the quantity of the different metabolites is different between fruits, seeds, roots, stems or leaves of the crops.

Significant residues appear in fruits after foliar application (cucumbers). Fluazifop acid and its conjugates comprise the major residue (69–72%). Fluazifop-butyl (I) and despyridinyl acid (III) are found at low levels (7.3% and 3.3%, respectively). Residues are distributed evenly throughout the peel and the pulp within 14 days of treatment. Samples from supervised residue trials show that fluazifop-butyl may be present up to 8 days after treatment in strawberries.

Significant residues appear in the seeds of pulses and oilseeds (oilseed rape seeds and soyaseeds), when the application is performed at pod formation stage. Residues are lower when application is performed at 3–6 trifoliate stage. The principal component of the residue is fluazifop acid in free or conjugated form (40–81%). Fluazifop-butyl (I) and despyridinyl acid (III) are found at low levels (0.2% and < 4%, respectively) up to 82 days after treatment. Fluazifop conjugates were identified as glyceride esters (glycerol dioleate, glycerol dilinoleate and a hybrid oleate-palmitate ester of glycerol) in soya bean seeds.

Significant residues appear in root and tuber vegetables (carrots, potatoes, and sugar beet roots). The principal component of the residue is fluazifop acid in free or conjugated form (25–64%). Despyridinyl acid (conjugates) and CF3-pyridone (free and conjugates) were found at significant levels (4.8–18% and 1–37%, respectively). CF3-pyridone was found at higher levels than its despyridinyl counterpart in some root crops and could indicate additional uptake from soil. Fluazifop alcohol (free and conjugates) was only found in carrot roots (11–13%) treated with fluazifop-butyl (RS) and is thought to be derived from the S-enantiomer. Hydroxyfluazifop acid (XL, free) was found in potatoes at significant levels (13–15%). The fluazifop acid, despiridinyl acid and CF3-pyridone conjugates were identified as hexosides and/or malonylhexosides. Fluazifop-butyl was found at low levels (0.5%) up to 20 days after treatment.

Low residues appear in stem vegetables (celery). The principal component of the residue is fluazifop acid in free or conjugated form (39–43%). Despyridinyl acid (free and conjugates) is found at significant levels (18%). CF3-pyridone (free and conjugated) is found at low levels (2.7% TRR). Supervised residue trials show that CF3-pyridone is present up to 30 days after application at levels equivalent to 13% total fluazifop in celery stems. Hydroxyfluazifop acid (XL) was found at low levels (< 5%). Parent compound was not detected.

Significant residues appear in leafy vegetables (lettuce, endive, and celery leaves) with fluazifop-butyl or fluazifop acid (free and conjugated) as the main compound (up to 52% and 19–63%, respectively). Parent compound was found at significant levels (49–52%) in lettuce leaves at 27 days after treatment, and at low levels (2.4%) in celery leaves at 30 days after treatment. Pyr-Ph ether (IV) (free and conjugated) was found at significant levels (11–25%) in immature endive (DAT 20) and at lower levels in lettuce and mature endive (< 2%). Despyridinyl acid (III) conjugates and CF3-pyridone (free and conjugated) were found at significant levels in endive, lettuce and celery leaves (7.1–41% and 11–14%, respectively). The fluazifop acid, despiridinyl acid and CF3-pyridone conjugates were identified as hexosides, malonylhexosides or pyridinyl N-sugars.

Significant residues appear in forage of pulses and oilseeds. The principal components of the residue are parent (up to 25%) and fluazifop acid in free or conjugated form (37–76%). Pyr-Ph ether (IV) (free and conjugated) and despyridinyl acid (III) conjugates were found at low levels (< 3% and < 8% TRR, respectively). Supervised residue trials show that fluazifop-butyl may be present up to 16 days after treatment in oilseed rape forage.

Significant residues appear in forage of roots and tubers. The principal component of the residue was fluazifop acid in free or conjugated form (42–82%). Despyridinyl acid (conjugates) were found at low levels (< 6% TRR). CF3-pyridone (conjugates) was found at higher levels (31–48% TRR) than its despyridinyl counterpart (< 6% TRR) and could indicate additional uptake from soil. Pyr-Ph ether (IV) was not detected. The fluazifop acid, despiridinyl acid and CF3-pyridone conjugates were identified as hexosides and/or malonylhexosides. Supervised residue trials show that fluazifop-butyl may be present up to 98 days after treatment in sugar beet forage.

Fate in rotational crops

Metabolism of fluazifop-butyl was investigated in two confined rotational crops following a single bare soil treatment.

In the first confined rotational crop study, [^{14}C]phenyl- or [^{14}C]pyridyl-fluazifop-butyl (RS) was applied to a bare sandy loam soil at 1 × 0.25 kg ai/ha under greenhouse conditions. Rotational crops (lettuce, wheat and sugar beet) were sown at 30, 120 and 327 Day plant back intervals for the phenyl label and 60, 120 and 365 day PBI for the pyridyl label. Total radioactive residues were < 0.01 mg/kg eq in the phenyl-labelled crop samples at all plant back intervals. Total radioactive residues were < 0.01 mg/kg eq in the pyridyl-labelled sugar beet roots and lettuce leaves at all plant back intervals. Total radioactive residues were 0.011–< 0.01–< 0.01 mg/kg in wheat grain, 0.10–0.080–0.031 mg/kg eq in wheat straw and 0.027–0.018–< 0.01 mg/kg eq in sugar beet tops, respectively for the three PBIs. The radioactive residues were not further characterised.

In the second confined rotational crop study, [^{14}C]phenyl- or [^{14}C]pyridyl-fluazifop-P-butyl (R-enantiomer) was applied to a bare sandy loam soil at 0.44–0.50 kg ai/ha under indoor conditions. Rotational crops (lettuce, wheat and carrot) were sown at 30, 60 and 270 Day plant back intervals (PBI).

Analysis of the soil samples showed that only 1.2%AR remained as parent compound after 30 days. In soil treated with [^{14}C]phenyl-labelled fluazifop-P-butyl, free fluazifop-P-acid were the main compounds. In soil treated with [^{14}C]pyridyl labelled fluazifop-P-butyl, free CF3-pyridone was the main compound.

Crops grown in soil treated with [^{14}C]phenyl-labelled fluazifop-P-butyl had very low residues. Total radioactive residues in lettuce leaves and carrot roots were below 0.01 mg eq/kg at all plant back intervals. Residues in wheat grains and feed crops were below 0.04 mg/kg eq except wheat straw

at the 60 Day plant back interval (PBI) where the residue was 0.1 mg/kg eq. In wheat straw of the 60 Day PBI 60% TRR was organo- and/or acid soluble. Individual extracted components of wheat straw did not exceed 0.014 mg/kg eq, post extraction solids represented a residue of 0.03 mg/kg eq. No known metabolites were found.

Crops grown in soil treated with [^{14}C]pyridyl-fluazifop-P-butyl had radioactive residues > 0.01 mg/kg eq at all plant back intervals. Total radioactivity in edible crop commodities ranged from 0.01–0.25 mg/kg eq at 30 Day PBI, 0.03–0.46 mg/kg eq at 60 Day PBI and 0.02–0.34 mg/kg eq at 270 Day PBI, while residues up to 1.5 mg/kg were found in forage (PBI 60) and up to 6.7 mg/kg eq were found in wheat straw (PBI 270). Characterisation and identification was carried out on all crops grown after a 60-day rotation period. Fluazifop-P-butyl, fluazifop acid and Pyr-Ph ether (IV) were not detected. CF3-pyridone (X) including its conjugates represented > 60% TRR in most crop commodities.

The Meeting noted that analyses in soil and rotational crops indicate that fluazifop-P-butyl and fluazifop acid are not taken up from the soil and concluded that total fluazifop residues are therefore not expected in rotational crops. CF3-pyridone is the only residue that is taken up from the soil under confined conditions at all plant back intervals (30, 60, and 270 Days).

Animal metabolism

The Meeting received results of metabolism studies in laboratory animals, humans, lactating goats and laying hens. Metabolism in laboratory animals and humans was summarized and evaluated by the WHO panel of the 2016 JMPR.

One lactating cow was dosed orally twice daily for 7 consecutive days with a gelatin capsule containing a 50:50 mixture of [^{14}C]phenyl and [^{14}C]pyridyl-fluazifop-butyl (racemate). The equivalent actual mean daily dose in the dry feed was 2.5 ppm (or 0.075 mg/kg bw). The cow was sacrificed 4 hours after the last dose. Total recovered radioactivity amounted to 82% of the administered dose. The majority of the radioactivity was recovered in urine (80%) with small amounts recovered in faeces (1.7%) and milk (1.1%).

The highest radioactivity concentrations were found in kidney (0.039 mg/kg eq) and liver (0.024 mg/kg eq), followed by fat (0.002–0.005 mg/kg eq) and muscle (0.001 mg/kg eq). Total radioactive residues in milk reached a plateau concentration of approximately 0.034 mg/kg eq following 2 days of dosing.

Following solvent extraction, residue extractabilities were > 89% TRR for milk and all tissues of cow. In milk, the majority of the residues (94% TRR) were extracted with hexane, representing the residues in the milkfat fraction. Extracts from milk and liver were hydrolysed to cleave possible conjugates.

Parent was not detected in milk or tissues of cow. The most significant metabolite (including conjugates) identified in all tissues and milk was fluazifop acid (32–68% TRR). Pyr-Ph ether (IV) (including conjugates) was identified in liver and kidney (10–12% TRR, < 0.01 mg/kg eq). These levels must be seen as minimum levels, since several extracted or solid fractions of these commodities were not subjected to hydrolysis and may contain additional amounts of metabolites. These unhydrolysed fractions accounted for 16%, 19%, 26%, 63% and 68% TRR in milk, liver, kidney, muscle and fat, respectively. Muscle and fat residue characterisation was not pursued further because of the low total radioactive residue levels (< 0.01 mg/kg eq).

Two lactating goats (one per label) were dosed orally twice daily for 7 consecutive days with a gelatin capsule containing [^{14}C]phenyl or [^{14}C]pyridyl-fluazifop-P-butyl (R-enantiomer). The equivalent actual mean daily doses in the dry feed were 9.6 or 9.7 ppm (or 0.28 or 0.23 mg/kg bw) for the phenyl or pyridyl label, respectively. Goats were sacrificed 16 hours after the last dose. Total recovered radioactivity amounted to 87% and 99% of the administered dose for the phenyl and pyridyl radiolabelled forms, respectively. The majority of the radioactivity was recovered in urine (70–82% AR) with lower amounts recovered in recovered in faeces (10–11% AR), milk (0.8–0.9% AR) and tissues (< 0.2% in total).

The highest radioactivity concentrations were found in kidney (0.62/0.46 mg/kg eq) and liver (0.060/0.045 mg/kg eq), followed by fat (0.006–0.015/0.005–0.011 mg/kg eq) and muscle (0.004/0.002–0.003 mg/kg eq). Total radioactive residues in milk reached a plateau concentration of approximately 0.15–0.16 mg/kg eq following 96–104 hours dosing.

Following solvent extraction, residue extractabilities were 54–55% TRR for kidney and 62–66% TRR for liver. A further 9–11% TRR and 37–43% TRR could be extracted from kidney and liver, respectively, with mild alkaline and/or acid solutions at room temperature. Milk was separated into skimmed milk (30%/1% TRR, phenyl/pyridyl label) and milk fat (70%/92% TRR, phenyl/pyridyl label). Muscle and fat were not analysed further. Extracted residues from kidney and milk were subjected to more severe hydrolysis conditions to release the exocons from the conjugates, but these conditions were too harsh for an acceptable residue characterisation.

Parent was not detected in milk or tissues of goat. The most significant metabolite identified in liver was free fluazifop acid (21–25% TRR). Pyr-Ph ether (IV) (including conjugates) was not detected in liver.

Two laying hens (one per radiolabel) were dosed orally once daily for 14 consecutive days with a gelatin capsule containing [^{14}C]phenyl or [^{14}C]pyridyl-fluazifop-butyl (racemate). The equivalent actual mean daily doses in the dry feed were 3.1 or 2.6 ppm dry feed (0.22 or 0.18 mg/kg bw) for the phenyl or pyridyl label, respectively. Hens were sacrificed 4 hours after the last dose. The majority of the radioactivity was recovered in excreta (97–98% AR).

The highest radioactivity concentrations were found in kidney (0.056/0.44 mg/kg eq, phenyl/pyridyl) and liver (0.027/0.077 mg/kg eq), followed by fat (0.040–0.045/0.029–0.039 mg/kg eq) and muscle (0.004–0.005/0.008–0.011 mg/kg eq). Total radioactive residues in egg yolks achieved a plateau concentration of 0.02 mg/kg eq after 6–7 days of dosing. Total radioactive residues in egg whites achieved a plateau concentration of 0.002–0.003 mg/kg eq after 3 days of dosing. Following solvent extraction, residue extractabilities were ≥ 82% TRR for eggs and tissues.

Parent was not detected in eggs and tissues of hens. The most significant metabolite (including conjugates) identified in eggs and all tissues was fluazifop acid (51–71% TRR). Despyridinyl acid (III), Pyr-Ph ether (IV) and CF3-pyridone (X) were not detected. These levels must be seen as minimum levels, since several extracted or solid fractions of these commodities were not subjected to hydrolysis and may contain additional amounts of metabolites. These unhydrolysed fractions accounted for 38.9% TRR (eggs), 25–27% (liver), 22–40% (kidney), 33–49% (muscle), 8.9–25% (fat) TRR.

In a second metabolism study on hens, ten laying hens (five per radiolabel) were dosed orally twice daily for 10 consecutive days with a gelatin capsule containing [^{14}C]phenyl or [^{14}C]pyridyl-fluazifop-P-butyl (R-enantiomer). The equivalent actual mean daily dose in the dry feed was 9 ppm (or 0.84 mg/kg bw). Hens were sacrificed 24 hours after the last dose. Total recovered radioactivity amounted to 93% and 95% of the administered dose for the phenyl and pyridyl radiolabelled forms, respectively. The majority of the radioactivity was recovered in excreta (90%/93%, phenyl/pyridyl).

The highest radioactivity concentrations were found in abdominal fat (0.14/0.24 mg/kg eq, phenyl/pyridyl), followed by skin with fat (0.041/0.064 mg/kg eq), liver (0.007/0.028 mg/kg eq) and muscle (0.002–0.009/0.005–0.012 mg/kg eq). Total radioactive residues in egg yolks achieved a plateau concentration of 0.072 mg/kg eq after 144 hrs of dosing. Total radioactive residues in egg whites achieved a plateau concentration of 0.033 mg/kg eq after 120–168 hrs of dosing.

Following solvent extraction, residue extractabilities were ≥ 88% TRR for egg yolk and egg white, fat or skin with fat, 48% TRR for liver and 23% TRR for muscle. Selected extracts were treated under hydrolytic conditions to cleave the conjugates. Hydrolysis conditions in liver were too soft for an acceptable residue characterisation.

Parent was only detected as a minor component in liver (0.7% TRR). The most significant metabolite (including conjugates) identified in fat and eggs was fluazifop acid (56–86% TRR). Pyr-Ph ether (IV) was only detected in pyridyl-labelled egg white (1.1% TRR). These levels must be seen as

minimum levels, since several extracted or solid fractions of these commodities were not subjected to hydrolysis and may contain additional amounts of metabolites. These unhydrolysed fractions accounted for 7.4–20.1% TRR (eggs), 1.0–2.7% (abdominal fat), 11–22% (fat with skin).

In summary, metabolism between cows, goats, hens, laboratory animals and humans is similar. Fluazifop-butyl is metabolized via hydrolysis to form fluazifop acid (all tissues, milk, and eggs) and further conjugation of fluazifop- acid possibly to lipids. Pyr-Ph ether was detected at significant levels in cow liver and kidney (10–12%). Since extracts of milk, eggs and tissues contained significant amounts of compounds that were not subjected to hydrolysis, the absence or presence of other significant metabolites could not be confirmed.

In general, metabolism between plants and animals is similar. Despyridinyl acid (III) was detected in minor quantities in rats and mice, and CF3-pyridone was detected in minor quantities in rats. Pyr-Ph ether (IV) was not detected in laboratory animals, but it was detected in livestock. Hydroxyfluazifop acid (XL) was not detected in animals.

Although fluazifop acid or its conjugates are the main residues in plants, no livestock metabolism and/or feeding studies were conducted with fluazifop acid. However, since fluazifop-butyl is rapidly absorbed and de-esterified into fluazifop acid, studies with fluazifop-butyl are satisfactory.

Environmental fate in soil

The Meeting received information on soil photolysis, aerobic degradation and field dissipation.

Soil photolysis of [^{14}C]phenyl or [^{14}C]-pyridyl-fluazifop-P-butyl (i.e. R-enantiomer) indicated that photo-degradation is not a major route of degradation for fluazifop-P-butyl. The average DT_{50} for fluazifop-P-butyl in the irradiated soils was 116 days, whereas it was 272 days for the dark controls.

Soil studies with fluazifop-butyl (RS) showed that the metabolite fluazifop acid largely comprised of the R-enantiomer and that the proportion of the R-enantiomer increased with time. This was confirmed in a supplemental study with the separate R- and S-enantiomers of [^{14}C]phenyl-fluazifop-butyl. These studies indicate that fluazifop-P-butyl degradation products will remain as R-enantiomer when applied to soil.

Aerobic degradation of [^{14}C]-phenyl and/or [^{14}C]pyridyl-fluazifop-butyl (racemate) under laboratory conditions indicated that fluazifop-butyl (RS) degraded rapidly to 1.2–3.7% AR after 3 weeks. The major metabolites identified were fluazifop acid and CF3-pyridone. Fluazifop acid reached a maximum of 45–83% AR (phenyl label) after 2 days of incubation in most soils, except 72% AR after 21 weeks in the sandy soil. CF3-pyridone (X) reached a maximum of 22–25% AR after 12 weeks of incubation (pyridyl label only). Pyr-Ph ether (IV) was found as a minor metabolite (< 4% AR at all time points, except in the sandy soil with 8.9% AR at 21 weeks). Carbon dioxide was formed from Day 1 onwards and these levels increased with time (up to 25–36% AR after 45 weeks of incubation). An additional study confirmed that fluazifop-P-butyl degrades similar to fluazifop-butyl (racemate). Half-lives for fluazifop-butyl, fluazifop acid, Pyr-Ph ether (IV) and CF3-pyridone (X) are listed in the table below.

Compound	Geometric DT_{50} (days) Aerobic laboratory conditions	Geometric DT_{90} (days) Aerobic laboratory conditions
Fluazifop-butyl (RS)	1.0	3.4
Fluazifop-P-acid	6.5–8.3 From different kinetic endpoint studies	32–35 From different kinetic endpoint studies
CF3-pyridone (X)	12	134
Pyr-Ph ether (IV)	31	42–348 (individual, mean not calculated)

Aerobic degradation under less favourable laboratory conditions indicates that the degradation of fluazifop acid is mediated by microbial activity. This is evidenced by the virtual absence of degradation in sterilised soils.

Field dissipation studies on bare soil or cotton and soyaplots indicated that the CF3-pyridone levels in soil were very low (< 0.01–0.05 mg/kg) and were < 0.01 mg/kg at 75–270 days after the last application. When more sensitive analytical methods were used, CF3-pyridone could be detected at levels of < 0.001–0.01 mg/kg for a longer period (359–373 days after the last application). CF3-pyridone levels resulting from sequential application of fluazifop-butyl do not differ from single fluazifop-butyl applications. Half-lives for CF3-pyridone in the field dissipation studies were estimated between 100–241 days; these are longer than those estimated in the aerobic field studies (12–134 days).

In conclusion, aerobic soil degradation studies demonstrate that fluazifop-butyl and fluazifop acid degrade in soil, but that CF3-pyridone and Pyr-Ph ether are semi-persistent. Under standardized aerobic soil conditions, CF3-pyridone reaches a maximum after 4–12 weeks of fluazifop-butyl treatment, while Pyr-Ph ether is present at constant low levels. Considering a worst case DT_{90} of 255 days (36 weeks) for CF3-pyridone obtained in the aerobic soil studies, and peak appearance after 12 weeks of fluazifop-butyl treatment, most of the CF3-pyridone compound has disappeared by 48 weeks. CF3-pyridone levels in soil resulting from sequential application of fluazifop-butyl do not differ from single fluazifop-butyl applications. CF3-pyridone is expected to degrade in soils within a year after application and field dissipation studies confirm this. CF3-pyridone is not expected to accumulate to a soil plateau level equivalent to 125% (or higher) of the residue level following the maximal seasonal application rate for fluazifop-butyl. Thus, no adjustment is needed for crop residues obtained in the rotational crop studies.

Methods of analysis

The Meeting received description and validation data for analytical methods for the determination of total fluazifop (i.e. sum of fluazifop-butyl, fluazifop acid and its conjugates, expressed as fluazifop acid) in plant and animal commodities. Total fluazifop is not determined by the existing multi-residue method, since hydrolysis is needed to release fluazifop acid from its conjugates.

Fluazifop-butyl and fluazifop acid occur in two isomeric forms–the R- and S-enantiomer. The R- and S-enantiomers are not separated by the chromatographic techniques applied in the analytical methods.

HPLC-MS/MS method GRM44.02A was submitted as the enforcement/monitoring method for the determination of total fluazifop in plant commodities. Plant commodities were extracted with acetonitrile/concentrated HCl (plants with > 60% water content) or acetonitrile/1 M HCl (grains, pulses, oilseeds, and dry crops) after soaking for at least 2 hrs in 1 M HCl or overnight in water. Residues in the extracts were then hydrolysed in 6 M HCl (1 hr, 60 ºC) to convert fluazifop-P-butyl and fluazifop conjugates to fluazifop acid. Samples were cleaned-up by SPE prior to quantification by HPLC-MS/MS. Radio-validation confirmed that total fluazifop is adequately extracted from endive (69%) and carrots (99%) under these conditions. The Meeting considers validation sufficient for all plant commodities. The LOQ was 0.01 mg/kg, expressed as fluazifop acid, in each matrix.

GC-MS method RAM 331/01 was submitted as the enforcement/monitoring method for the determination of total fluazifop in animal commodities. Animal commodities were extracted with dichloromethane/methanol and the residues in the extract were then hydrolysed with 0.2 M NaOH in methanol (1 hr at 60 ºC) to convert fluazifop-P-butyl and fluazifop conjugates to fluazifop acid. The hydrolysate is cleaned-up by liquid-liquid partition and solid phase extraction (SPE). The fluazifop acid residues are then derivatised to the methyl ester, followed by clean-up on SPE and determination by GC-MS. Radio-validation confirmed that total fluazifop is quantitatively extracted from milk (102%), liver (87%), and eggs (89%) under these conditions. The Meeting considers validation sufficient for all animal commodities (meat, liver, kidney, fat, milk and eggs). The LOQ was 0.01 mg/kg, expressed as fluazifop acid, in each matrix.

Several other analytical methods were submitted for the determination of total fluazifop in plant and animal material. The extraction and hydrolysis conditions for most of the methods were the same as described above for plant or animal commodities. Radio-validation was available for alternative hydrolysis conditions. Further, the methods differed in their clean-up procedures and

detection techniques. Various detection techniques were used: HPLC-UV, 19F-NMR, HPLC-MS/MS, GC-NPD or GC-MS. The LOQs were 0.01–0.05 mg/kg. Methods were not fully validated according to current guidelines and in some cases the valid LOQ is higher than reported. The Meeting considered these methods adequate for the residue trials, unless specified otherwise in the supervised trials section.

A few analytical methods were submitted for the determination of despyridinyl acid or CF3-pyridone and its conjugates in plant material. Extracts were hydrolysed with 1 M HCl (1 hr reflux) to convert CF3-pyridone conjugates into CF3-pyridone or 6 M HCl (1 hr reflux) to convert despyridinyl acid to its conjugates. Since fluazifop acid partly degrades under these conditions, the levels of despyridinyl acid and CF3-pyridone are overestimated. The Meeting considers these analytical methods not acceptable.

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability in plant, animal or soil commodities fortified with fluazifop-P-butyl, fluazifop acid or CF3-pyridone (X) and on storage stability of total fluazifop in plant and animal commodities with incurred residues.

Parent fluazifop-P-butyl is stable for at least 28 months at -18 ºC in onions.

Fluazifop acid is stable for at least 27 months at -1 °C, 8 months at -15 ºC and for 31 months at -20 °C in raspberries, blueberries, strawberries, sweet potatoes, rhubarb, macadamia nuts, and green coffee beans. Studies with incurred residues to assess the stability of total fluazifop residues (including conjugates) were inconclusive, since the samples were not analysed immediately after harvest. Since fluazifop conjugates are converted to fluazifop acid by hydrolysis in the analytical method and fluazifop acid is resistant to a whole range of hydrolysis conditions (acid, alkaline, and enzymatic), it is likely that any degradation of the fluazifop conjugates proceeds through formation of fluazifop acid upon frozen storage.

CF3-pyridone is stable for at least 24–28 months at -18 ºC in apples, onions, lettuce, and peanut kernels.

Fluazifop acid is stable for at least 12–18 months at -16 ºC in various processed commodities: soya bean meal, soya bean hulls, soya bean oil, soya bean milk, potato flakes, potato wet peel, potato chips, wheat flour, wheat middlings, wheat shorts, tomato paste and tomato puree.

Fluazifop acid is stable for at least 12 months at -20 ºC in milk, eggs and tissues.

The Meeting concluded that total fluazifop and CF3-pyridone (X) is stable during frozen storage in all plant and animal commodities as long as the samples stay frozen.

Definition of the residue

In primary crops, parent compound was detected at significant levels in fruits and edible leaves and represented 7.3% (0.092 mg/kg) in cucumbers and 49–52% TRR in lettuce.

Fluazifop acid and its sugar or glyceride conjugates represented the principal part of the residue in most edible crop commodities (25–77% TRR).

Despyridinyl acid (III) and its conjugates were detected in all crop categories investigated, but were only found at levels above 10% TRR or above 0.01 mg/kg eq in cucumbers, celery leaves, endive, soya bean seeds, carrot roots, and potato tubers).

CF3-pyridone (X) and its conjugates are expected in all crop categories, but were often not identified because only the phenyl label was investigated or alkaline hydrolysis conditions were used to release conjugates. CF3-pyridone and its conjugates were found at levels above 10% TRR or above 0.01 mg/kg eq in celery leaves, endive and carrot roots.

Pyr-Ph ether (IV) and its conjugates were detected in various leafy commodities at low levels, but were found at levels above 10% TRR in immature endive.

Hydroxyfluazifop acid (XL) was found at levels above 10% TRR in potato tubers.

Fluazifop alcohol (XXXIV) was only found at levels above 10% TRR or 0.01 mg/kg eq in crops treated with fluazifop-butyl (RS) and is considered to be derived from the S-enantiomer of fluazifop-butyl. Since fluazifop-butyl (RS) is replaced by fluazifop-P-butyl since 1984, this compound is not expected to appear in crops.

In rotational crops CF3-pyridone and its conjugates were the principal components in all crop categories. No residues above the LOQ (0.02 or 0.05 mg/kg) of CF3-pyridone were found in edible crops in the field rotational crop studies submitted.

Fluazifop-P-butyl belongs to the aryloxyphenoxypropionate herbicides, and despyridinyl acid (III) may be a common metabolite to all compounds belonging to this group: chlorazifop, clodinafop, clofop, clofop-iso-butyl, cyhalofop, cyhalofop-butyl, diclofop, fenoxaprop-, fenoxaprop-ethyl, fenthiaprop, fenthiaprop-ethyl, fluazifop-methyl, haloxyfop-, haloxyfop-methyl, haloxyfop-etotyl, kuicaoxi, propaquizafop, quizalofop, trifop and trifop-methyl. CF3-pyridone (X) may be a common metabolite to fluazifop-methyl, trifop and trifop-methyl. Despyridinyl acid (III) and CF3-pyridone (X) are therefore not suitable for markers of fluazifop-butyl in primary crop commodities.

Analytical methods for enforcement have been validated for the common moiety fluazifop acid, which is released from fluazifop-butyl and fluazifop conjugates. Since a hydrolysis procedure is required to be able to release fluazifop acid from its conjugates, the residue is unlikely to be measured by a multi-residue method.

The Meeting concluded that fluazifop-butyl, fluazifop acid and its conjugates represent the major residue and these compounds are suitable for markers for MRL compliance in primary crops.

Regarding the inclusion of metabolites for dietary risk assessment, the Meeting decided to estimate the overall toxicological burden of relevant metabolites. Apart from fluazifop acid, metabolites found at levels > 10% TRR or > 0.01 mg/kg eq in plant commodities were: despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X), hydroxyfluazifop acid (XL) and their conjugates. The Meeting made some conservative dietary exposure estimates to decide whether these metabolites need to be selected for inclusion in the residue definition for dietary risk assessment. Since the supervised residue trials only analysed total fluazifop, residue levels for these metabolites are estimated based on the ratio of this metabolite relative to total fluazifop residues obtained from the metabolism studies. The median and maximum ratios are listed in Table 1 below.

Potential dietary exposure to total fluazifop was calculated assuming 0.01 mg/kg total fluazifop in fruits and tree nuts, 0.02 mg/kg in sugar cane, 0.03 mg/kg in cucurbits and seeds for beverages, 0.05 mg/kg in leafy vegetables, 0.2 mg/kg in berries and fruiting vegetables other than cucurbits, 0.3 mg/kg in bulb, stalk and stem vegetables, 0.8 mg/kg in Brassicas, 1.5 mg/kg in legumes, roots and tubers, 5 mg/kg in pulses and 9 mg/kg in oilseeds and using the average consumption in the GEMS/Food 17 cluster diets.

Potential long-term dietary exposure to each metabolite is calculated by multiplication of the total fluazifop residues by the median ratio metabolite/total fluazifop listed in the table below for each individual metabolite and using the GEMS/Food 17 Cluster diet. Results are presented in Table 2 below. For the potential short-term dietary exposure, the ratios between total fluazifop residues and the respective metabolites is taken into account.

Despyridinyl acid (III) and CF3-pyridone (X) individually contribute significantly to the total long-term dietary exposure (7.5–17% and 7.6–19%, expressed as fluazifop acid equivalents, respectively). Percentages of despyridinyl acid (III) or CF3-pyridone (X) to total fluazifop were up to 76% in roots crops and up to 110% in leafy crops, suggesting significant contribution to the short-term dietary exposure. Additional uptake from soil is expected for CF3-pyridone (X), but not for despyridinyl acid (III).

Despyridinyl acid (III) is found in rats and mice, where it is excreted in small amounts (approximately 0.7% and 2% of the applied dose in rats and mice, respectively) in the urine. Based on toxicity studies, the Meeting concluded that despyridinyl acid (III) is not genotoxic in vitro. On the

basis of structural considerations, the Meeting concluded that despyridinyl acid (III) is unlikely to be of greater toxicity than the parent.

CF3-pyridone (X) is not found in rats or dogs but is present in mice to a limited extent, where it is excreted in small amounts (approximately 1.1% of the applied dose) in the urine. Based on toxicity studies conducted with CF3-pyridone, the Meeting concluded that CF3-pyridone (X) is covered by the ADI and ARfD for fluazifop-P-butyl.

The Meeting noted that despyridinyl acid (III) and CF3-pyridone (X) are counter pieces, resulting from cleavage of fluazifop acid. Therefore, adjustment of molecular weights to fluazifop acid equivalents for the sum of both cleavage products would result in an overestimation of the total toxicological burden. Both compounds were present in comparable relative amounts in primary treated crops. For CF3-pyridone (X), additional uptake from soil into plant commodities is expected, making it a conservative indicator for the combined residue of both counter pieces, when expressed as fluazifop acid equivalents.

The Meeting considered that if CF3-pyridone (X) is included into the residue definition for dietary intake purposes, this would also accommodate for residues of despyridinyl acid (III), when expressed as fluazifop acid equivalents.

Pyr-Ph ether (IV) was estimated to contribute insignificantly (0–0.03%, expressed as fluazifop acid equivalents) to the total long-term dietary exposure. Percentages of Pyr-Ph ether (IV)/total fluazifop found in specific crop commodities were generally below 5%, except for immature endive with percentage of 68% while the mature plant was present at 4.4%. Pyr-Ph ether (IV) was not found in laboratory animals and no toxicity studies are available. Its estimated exposure based on uses considered by the present Meeting is below the threshold of toxicological concern for Cramer Class III (1.5 µg/kg bw/day). Therefore, Pyr-Ph ether (IV) does not need to be considered further.

Hydroxyfluazifop acid (XL) gives significant contribution to the total long term-intake (6–31% compared to total fluazifop and expressed as fluazifop acid equivalents), primarily based on root crops, for which only one plant metabolism study included analysis of this metabolite. Percentages of hydroxyfluazifop acid (XL)/total fluazifop found in specific crop commodities (in metabolism studies) were low in leafy crops (3%) but significant in root crops (62%), suggesting potential contribution to the short-term dietary exposure. Hydroxyfluazifop acid (XL) was not found in laboratory animals. No toxicological information is available. However, owing to its structural similarity with the parent, the Meeting concluded that hydroxyfluazifop acid XL is unlikely to be of greater toxicity than the parent. The Meeting decided to include hydroxyfluazifop acid (XL) into the residue definition for dietary intake purposes.

The Meeting decided to include fluazifop-butyl, fluazifop acid, CF3-pyridone (X) and hydroxyfluazifop acid (XL) and their conjugates in the residue definition for dietary risk assessment for plant commodities.

The major compounds identified in cow or hen tissues, milk or eggs is fluazifop acid in free or conjugated form. Parent compound was only detected at trace levels in hen liver. Fluazifop acid and its lipophilic conjugates were identified at levels of 32–37% TRR (< 0.01 mg/kg eq) in cow muscle and fat, 61–68% (0.015–0.032 mg/kg eq) in cow milk, liver and kidney, 51–85% (< 0.01–0.012 mg/kg eq) in hen muscle, egg yolks, egg whites and whole eggs and 51–74% (0.019–0.24 mg/kg eq) in hen kidney, liver and fat.

Since animal feeds contain fluazifop acid conjugates as well as despyridinyl acid (III), Pyr-Ph ether (IV), CF3-pyridone (X), and hydroxyfluazifop acid (XL), animal feeding studies with these compounds are considered desirable to investigate whether any of these metabolites accumulate in tissues.

Analytical methods for enforcement of animal commodities have been validated for the common moiety fluazifop acid, which is released from fluazifop-butyl and fluazifop conjugates. Since

a hydrolysis procedure is required to be able to release fluazifop acid from its conjugates, the residue is unlikely to be measured by a multi-residue method.

Since fluazifop-butyl, fluazifop acid and fluazifop conjugates represent the major part of the residue in all livestock commodities and no other metabolites have been identified in significant quantities, the Meeting decided to define the residue for enforcement and for dietary risk assessment in animal commodities as total fluazifop (i.e. the sum of fluazifop-butyl, fluazifop acid and its conjugates).

The cow and hen metabolism studies indicated that total fluazifop residues are a Factor 5 higher in fat than in muscle and a Factor 5 higher in egg yolk than in egg white. Fluazifop acid is found as lipophilic conjugates in the fat fraction of the milk and in hen fat and egg yolk. The Meeting considers total fluazifop fat soluble.

The Meeting recommended the following residue definition for fluazifop-P-butyl:

Definition of the residue for compliance with the MRL in plant commodities: *total fluazifop, defined as the sum of fluazifop-P-butyl, fluazifop-P-acid (II) and their conjugates, expressed as fluazifop-P-acid.*

Definition of the residue for dietary risk assessment in plant commodities: *the sum of fluazifop-P-butyl, fluazifop-P-acid (II), 2-[4-(3-hydroxy-5-trifluoromethyl-2-phenoxy)pyridyloxy] propionic acid (XL), 5-trifluoromethyl-2-pyridone (X) and their conjugates, expressed as fluazifop-P-acid*.

Definition of the residue for compliance with the MRL and for dietary risk assessment in animal commodities*: total fluazifop, defined as the sum of fluazifop-P-butyl, fluazifop-P-acid (II) and their conjugates, expressed as fluazifop-P-acid.*

The Meeting considers the residue fat soluble.

Since CF3-pyridone (X) and hydroxyfluazifop acid (XL) have not been analysed in the supervised residue trials it is proposed to use an adjustment factor to correct for the additional contribution of these metabolites to the total residue by multiplying the median and highest residues of total fluazifop residues with the factors for the various plant groups as indicated in Table 274 below.

Table 274 Median and maximum ratios between metabolite and total fluazifop

	Median ratios metabolite/total fluazifop from metabolism				Median residue
Crop group	Pyr-Ph ether	Despyridinyl acid	CF3-pyridone	Hydroxyfluazifop acid	multiplication factor
	IV	III	X	XL	1.00 + (III or X) + XL [a]
Fruits and fruiting vegetables; cereals, tree nuts; seeds for beverages; sugar cane, oil fruits, fruit and bud and tree spices, hops, tea from shrubs	0	0.046	0	0	1.05
Leafy vegetables, Brassicas, fresh herbs, saffron, herb tea	0.01	0.12	0.235	0.03	1.27
Bulb, stalk and stem vegetables	0	0.43	0.07	0.10	1.53
Legume vegetables, oilseeds and pulses, seed spices	0	0.05	0.05	0	1.05
Roots and tubers, root spices, herbal root tea	0	0.28	0.33	0.62	1.95
	Maximum ratios metabolite/total fluazifop from metabolism				Highest residue
Crop group	Pyr-Ph ether	Despyridinyl acid	CF3-pyridone	Hydroxyfluazifop acid	multiplication factor
	IV	III	X	XL	1.00 + (III or X) + XL [a]
Fruits and fruiting vegetables; cereals, tree nuts; seeds for beverages; sugar cane, oil fruits,	0	0.046	0	0	1.05

	Median ratios metabolite/total fluazifop from metabolism				Median residue
fruit and bud and tree spices, hops, tea from shrubs					
Leafy vegetables, Brassicas, fresh herbs, saffron, herb tea	0.044	0.82	1.13	0.03	2.16
Bulb, stalk and stem vegetables	0	0.43	0.07	0.10	1.53
Legume vegetables, oilseeds and pulses, seed spices	0	0.07	0.07	0	1.07
Roots and tubers, root spices, herbal root tea	0	0.44	0.76	0.62	2.38

[a] Contribution for CF3-pyridone (X) is also estimated from despyridinyl acid (III). Both compounds were present in comparable relative amounts in primary treated crops and therefore CF3-pyridone levels were taken from despyridinyl acid (III) levels for crop commodities, where the presence of CF3-pyridone (III) was not investigated or where CF3-pyridone levels were lower.

Table 275 TMDI using median multiplication factors and assumed residue levels in crop commodities [a]

Compound	GEMS/food Cluster with maximum intake	Residue intake (ug/person/day) as fluazifop acid	Residue intake (ug/kg bw/day) as fluazifop acid	Percentage of total fluazifop G01–G17
Total fluazifop	G11 (bw 60 kg)	2364.9	39.4	100%
Pyr-Ph ether (IV)	G15 (bw 60 kg)	0.5	0.0083	0.00–0.03%
Despyridinyl acid (III)	G03 (bw 60 kg)	335.0	5.58	7.5–17%
CF3-pyridone (X)	G03 (bw 60 kg)	383.7	6.39	7.6–19%
Hydroxyfluazifop acid (XL)	G03 (bw 60 kg)	622.4	10.4	6.0–31%

[a] Assumed residue levels of 0.01 mg/kg total fluazifop in fruits and tree nuts, 0.02 mg/kg in sugar cane, 0.03 mg/kg in cucurbits and seeds for beverages, 0.05 mg/kg in leafy vegetables, 0.2 mg/kg in berries and fruiting vegetables other than cucurbits, 0.3 mg/kg in bulb, stalk and stem vegetables, 0.8 mg/kg in Brassicas, 1.5 mg/kg in legumes, roots and tubers, 5 mg/kg pulses, 9 mg/kg in oilseeds.

Results of supervised residue trials on crops

Trials submitted to the Meeting were conducted from 1979 to 2014 and the quality of these trials differed considerably. The older trials were conducted when no guidelines existed. Only trials that were conducted according to current standards were taken into account for maximum residue level estimation.

Fluazifop-P-butyl is phytotoxic to grass-like crops (cereals, grasses, and sugar cane), but other crops do not show phytotoxicity at any growth stage. Proportionality from high to low dose rates is therefore used in the selection of data for estimation of maximum residue levels in crops other than grasses.

Weed directed spray applications at the base of trees or vines

Since metabolism studies indicated that no residues are expected above 0.01 mg/kg for weed directed spray applications at the base of trees, shrubs or vines, the Meeting decided to evaluate all supervised residue trials with weed directed spray applications at the base of trees together.

Field trials involving citrus fruit were performed in the USA (grapefruits, lemons, and oranges), Southern France and Martinique (lemon and lime) and Italy (oranges).

Critical GAP for citrus fruit is the US GAP with three applications at the base of the tree at 0.42 kg ai/ha with a PHI of 14 days.

One grapefruit trial from the USA (3 ×0.42–0.43 kg ai/ha, PHI 12 days) matched the US cGAP within 25%. Five additional grapefruit trials from the USA at a higher dose (3 ×0.56 kg ai/ha, PHI 14 days) confirmed the non-residue situation. Total fluazifop residues were: < 0.01 and < 0.05 (5) mg/kg (n = 6).

Four lemon trials from the USA at a higher dose (3 × 0.56 kg ai/ha, PHI 14 days) indicated a non-residue situation. Total fluazifop residues were: < 0.05 (4) mg/kg (n = 4).

Six orange trials from the USA (3 × 0.41–0.43 kg ai/ha, PHI 12–14 days) matched the US cGAP within 25%. Five additional orange trials from the USA at higher dose (3 × 0.56 kg ai/ha, PHI 14 days) confirmed the non-residue situation. Total fluazifop residues were:< 0.01 (6) and < 0.05 (5) mg/kg (n = 11).

Additional grapefruit (5), lemon (4) and orange (5) trials from the USA (3 × 0.84 kg ai/ha, PHI 14 days), confirmed residues were below LOQ (< 0.05 mg/kg for each). One trial on oranges from the USA with a 5 × higher dose rate (3 × 2.1 kg ai/ha, PHI 14 days), indicated residues at 0.015 mg/kg. One trial on oranges from Brazil (2 × 2.0 kg ai/ha, PHI 7 days), where the sample size was insufficient to generate a representative sample, indicated residues at 0.068 mg/kg.

Field trials involving pome fruit were performed in Germany (apples and pears), France (apples), Italy (apples) and the USA (apples).

Critical GAP for apples and pears in the Netherlands or Belgium is one application at the base of the tree at 0.38 kg ai/ha and a PHI of 28 days.

Two apple trials from Southern France and Italy (1 × 0.38–0.39 kg ai/ha, PHI 28 days) matched the Dutch or Belgian cGAP within 25%. Total fluazifop residues were: < 0.01 and < 0.01 mg/kg (n = 2).

Three apple trials from the USA (2 × 0.42 kg ai/ha, PHI 14 days) confirmed residues below LOQ: < 0.05 (3) mg/kg. Two apple trials from Northern France (1 × 0.75–0.96 kg ai/ha, PHI 7 days) confirmed residues below LOQ: < 0.01 and < 0.03 mg/kg (n = 2). However, one apple trial and three pear trials in Germany did not confirm the non-residue situation. One apple trial from Germany (1 × 1.0 kg ai/ha, PHI 0 days), which was inadequately described, indicated total fluazifop residues at 0.07 mg/kg. Three pear trials from Germany (1 × 1.0 kg ai/ha, PHI 7, 7, 13 days), which were inadequately described, indicated total fluazifop residues at 0.05, 0.05 and 0.07 mg/kg, respectively.

Field trials involving stone fruits were performed in Germany (cherries, plums, and peaches), Italy (peaches) and the USA (cherries, plums, and peaches).

The cGAP for cherries, plums, apricots, peaches and nectarines is the US cGAP with weed directed applications at the base of the tree at 3 × 0.42 kg ai/ha with a PHI of 14 days.

Four cherry trials from the USA (3 × 0.42 kg ai/ha, PHI 14–15 days) matched the US cGAP within 25%. Total fluazifop residues were: < 0.05 (4) mg/kg.

Four plum trials from the USA (3 × 0.42 kg ai/ha, PHI 14–15 days) matched the US cGAP within 25%. Total fluazifop residues were: < 0.05 (4) mg/kg (n = 4)

Three peach trials from the USA (3 × 0.42 kg ai/ha, PHI 14 days) matched the US cGAP within 25%. Total fluazifop residues were: < 0.05 (3) mg/kg.

One plum trial from the USA (3 × 2.1 kg ai/ha, PHI 14 days) confirmed residues were below LOQ (< 0.05 mg/kg). One peach trial from the USA (3 × 0.42 kg ai/ha, PHI 9 days) also confirmed residues were below LOQ (< 0.05 mg/kg).

Field trials involving grapes were performed in Germany, Spain, Greece and the USA.

The cGAP for grapes in Belgium is one application at 0.38 kg ai/ha and PHI of 28 days. Three grape trials from Spain and Greece (1 × 0.75 kg ai/ha with PHI 27–28 days) indicated a non-residue situation. Total fluazifop residues were: < 0.01, < 0.01 and < 0.01 mg/kg (n = 3).

The cGAP for grapes in the USA is 3 × 0.42 kg ai/ha and PHI of 50 days. Grape trials from the USA (3 × 0.42 kg ai/ha with PHI 50 days) could be matched to this GAP within 25%. Total fluazifop residues were: < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, and < 0.01 mg/kg (n = 6).

Furthermore, one grape trial from the USA (3 × 2.1 kg ai/ha, PHI 50 days) confirmed residues were below LOQ (< 0.01 mg/kg). Three trials from Germany (1 × 1.0 kg ai/ha, PHI 0, 7, 22), which

were poorly described, could not confirm the non-residue situation for grapes, as residues of fluazifop found were: 0.05, 0.06 and 0.14 mg/kg.

Field trials involving olives were performed in Italy.

The cGAP for olives is the French cGAP with one application at 0.25 kg ai/ha with PHI of 21 days. None of the trials could be matched to this GAP. One olive trial in Italy (1 × 0.75 kg ai/ha, PHI 28 days) at a higher dose confirmed the non-residue situation: < 0.01 mg/kg.

Field trials involving bananas were performed in the USA, Australia, Honduras and Martinique (i.e. French overseas territory).

Critical GAP for bananas is the US GAP with 3 × 0.42 kg ai/ha with a PHI of 0 days. Trials from the USA (3 × 0.42 kg ai/ha, PHI 0 days) matched this cGAP within 25%. Residues from bagged and unbagged bananas were equal. Total fluazifop residues were: < 0.01, < 0.01, < 0.01 and < 0.01 mg/kg (n = 4).

Field trials involving tree nuts were performed in the USA (almonds, macadamia nuts, pecans, and walnuts), UK (hazelnuts) and Italy (hazelnuts).

Critical GAP for macadamia nuts and pecans in the USA is three applications at the base of the trees at 3 × 0.42 kg ai/ha and PHI of 1 day. None of the trials could be matched to the USA cGAP.

Critical GAP for almonds, chestnuts, hazelnuts, macadamia nuts and walnuts in France is one application at the base of the trees with 1 × 0.25 kg ai/ha and PHI of 21 days. None of the trials could be matched to the cGAP from France.

Four almond trials from the USA at higher dose rate and shorter PHI (1 × 0.84 kg ai/ha, PHI 14 days) indicated residues below the LOQ for the French cGAP. Total fluazifop residues were: < 0.01, < 0.01, < 0.01 and < 0.01 mg/kg (n = 4).

Three walnut trials from the USA at higher dose rate and shorter PHI (1 × 0.84 kg ai/ha, PHI 14 days) indicated residues below the LOQ for the French cGAP. Total fluazifop residues were < 0.01, < 0.01 and < 0.01 mg/kg (n = 3).

One hazelnut trial from the UK at a higher dose rate but longer PHI (1 × 0.75 kg ai/ha, PHI 28 days) did not confirm the non-residue situation. Total fluazifop residues were: 0.01 mg/kg (n = 1) for nuts sampled by hand. Furthermore, one hazelnut trial from Italy (1 × 2.5 kg ai/ha, PHI 49, 73 days) did not confirm the non-residue situation. Total fluazifop residues were: 0.07 and 0.08 mg/kg (n = 2). Since the non-residue situation could not be confirmed for hazelnuts, the Meeting did not estimate a maximum residue level for hazelnuts.

Field trials involving coffee beans were performed in Brazil and the USA (Hawaii).

Critical GAP for coffee beans is the GAP from the USA with a weed directed application at 2 × 0.42 kg ai/ha with a PHI of 1 day. None of the trials could be matched to the USA GAP.

One trial from the USA (3 × 1.4 kg ai/ha, PHI 1 day) confirmed that no residues are to be expected in green coffee beans. Total fluazifop residues were < 0.05 mg/kg (n = 1).

The Meeting concluded that incidental residues that were found on citrus fruit, pome fruit and grapes are likely to result from unintentional sprays onto fruit due to spray drift, and these do not represent good agricultural practice. Furthermore, the Meeting concluded that the trials on citrus fruit, pome fruit, stone fruit, tree nuts, grapes, olives, bananas, and coffee beans mutually supported each other. Taking into account the LOQ of 0.01 mg/kg for the enforcement method, the Meeting estimated a maximum residue level of 0.01* mg/kg for citrus fruit, pome fruit, stone fruit, grapes, table olives and olives for oil production, bananas, macadamia nuts, pecans, almonds, walnuts and coffee beans. The Meeting estimated a median and highest residue of 0.01 mg/kg.

Using multiplication factors of 1.05 and 1.05 for the median and highest residues, the Meeting estimated an STMR and HR of 0.011and 0.011 mg/kg eq.

Cane berries

Field trials on cane berries were performed in Germany (blackberries and raspberries), the UK (raspberries), Southern France (raspberries) and the USA (blackberries and raspberries).

The cGAP for raspberries and blackberries in the Netherlands is 1 × 0.38 kg ai/ha and PHI of 45 days for a weed directed spray between bushes.

Blackberry trials did not match the Dutch GAP. Raspberry trials from the UK (1 × 0.38 kg ai/ha, PHI 56 days, base application) matched the Dutch cGAP within 25%. Total fluazifop residues were: < 0.05 and < 0.05 mg/kg (n = 2).

The Meeting estimated a maximum residue level of 0.01* mg/kg for cane berries based on the non-residue situation for weed directed sprays and the LOQ of 0.01 mg/kg for the enforcement method. The Meeting estimated a median and highest residue of 0.01 mg/kg.

Using multiplication factors of 1.05 and 1.05 median and highest residues, the Meeting estimated an STMR and HR of 0.011 and 0.011 mg/kg eq.

Bush berries

Field trials were performed in Germany (bilberries), USA (blueberries) and the UK (gooseberries and currants).

The only cGAP for bilberries and blueberries is the French GAP with one application at 0.25 kg ai/ha and PHI of 42 days. None of the trials could be matched to this GAP. The Meeting decided not to derive maximum residue levels for bilberries and blueberries.

The cGAP for currants and gooseberries is the cGAP from the UK at 1 × 0.38 kg ai/ha for a weed directed spray (where possible) before bloom or after harvest.

Currant trials from the UK (1 × 0.38 kg ai/ha, leaves unfolding to bud burst, over the top spray) matched the UK cGAP within 25%. Total fluazifop residues were: < 0.05 and < 0.05 mg/kg (n = 2).

Gooseberry trials from the UK (1 × 0.38 kg ai/ha, leaves unfolding to bud burst, over the top spray) matched the UK cGAP within 25%. Total fluazifop residues were: < 0.05 mg/kg (n = 1).

Since the cGAP applications are applied early in the growing season or after harvest, no residues are expected. The Meeting estimated a maximum residue level of 0.01* mg/kg for currants and gooseberries. The Meeting estimated a median and highest residue of 0.01 mg/kg.

Using multiplication factors of 1.05 and 1.05 for median and highest residues, the Meeting estimated an STMR and HR of 0.011and 0.011 mg/kg eq.

Strawberries

Field trials involving strawberries were performed in Germany, Sweden, the UK, Southern France, Italy and Spain.

The cGAP for strawberries in the Netherlands and France is 1 × 0.38 kg ai/ha with a PHI of 42 days.

Strawberry trials from Sweden, the UK, Southern France, Spain and Italy (1 × 0.36–0.39 kg ai/ha, PHI 39–43 days) matched the French and Dutch cGAP within 25%. Additional trials from Southern France (1 × 0.18–0.19 kg ai/ha, PHI 42 days) could be matched to this cGAP using proportionality. Two additional trials from the UK (1 × 0.38 kg ai/ha, PHI 55–57 days) were taken into account, since significant residues were found at these longer PHIs. Total fluazifop residues were: 0.02, 0.02, 0.02, 0.01 × 0.38/0.18, 0.01 × 0.38/0.18, < 0.05, 0.06, 0.06, 0.03 × 0.38/0.19, 0.07, 0.08, 0.11, 0.11, 0.12, 0.12 mg/kg, which becomes 0.02, 0.02, 0.02, 0.021, 0.021, < 0.05, 0.06, 0.06, 0.06, 0.07, 0.08, 0.11, 0.11, 0.12 and 0.12 mg/kg (n = 15).

The Meeting estimated a maximum residue level of 0.3 mg/kg for strawberries, based on the cGAP for the Netherlands and France. The Meeting estimated a median residue of 0.06 mg/kg and a highest residue of 0.12 mg/kg.

Using multiplication factors of 1.05 and 1.05 for median and highest residues, the Meeting estimated an STMR and HR of 0.063 and 0.13 mg/kg eq.

Onion, bulb (dry harvested)

Field trials involving bulb onions were performed in the United Kingdom, the Netherlands, Spain, Italy, Southern France, USA and Brazil.

Critical GAP for onions in the USA is for 2 × 0.42 kg ai/ha with a PHI of 45 days. Trials from the USA (2 × 0.42 kg ai/ha, PHI 39–46 days) matched this GAP within 25%. Additional trials from the USA (2 × 1.1 kg ai/ha, PHI 45–46 days) could be matched to this GAP through proportionality. Total fluazifop residues were: < 0.05, < 0.05, < 0.05, < 0.06, 0.06, 0.26 × 0.42/1.1, 0.11, 0.34 × 0.42/1.1, 0.18, 0.48 × 0.42/1.1 mg/kg (n = 10), which becomes < 0.05 (3), < 0.06, 0.06, 0.099, 0.11, 0.13, 0.18 and 0.18 mg/kg (n = 10).

The Meeting estimated a maximum residue level of 0.3 mg/kg on bulb onion (dry harvested) on the basis of the cGAP for the USA. The Meeting estimated a median residue of 0.080 and a highest residue of 0.18 mg/kg.

Using multiplication factors of 1.53 and 1.53 for median and highest residues, the Meeting estimated an STMR and HR of 0.12and 0.28 mg/kg eq.

The Meeting decided to extrapolate the maximum residue level, STMR and HR to shallots (dry harvested) and garlic.

Leeks

Field trials involving leeks were performed in the Netherlands, UK and Northern France.

cGAP for leeks is the GAP from France with one application at 0.38 kg ai/ha with a PHI of 42 days. Trials from the Netherlands (1 × 0.38 kg ai/ha, PHI 43 days) matched this GAP within 25%. Total fluazifop residues were: < 0.05 and < 0.05 mg/kg (n = 2). The non-residue situation could not be confirmed, since trials in the UK at 1 × 0.38 kg ai/ha at longer PHIs of 76–108 days showed residues of 0.02–0.06 mg/kg. The Meeting considered two trials insufficient.

Cabbages, Head

Field trials involving head cabbages were performed in Northern France, Germany, Greece, Spain and Brazil.

Critical GAP for head cabbages is the GAP from Brazil with one foliar application at 0.25 kg ai/ha with a PHI of 28 days. Trials from Brazil (1 × 0.19 kg ai/ha, PHI 28 days) matched this GAP within 25%. Total fluazifop residues were: 0.27, 0.29, 0.29 and 0.51 mg/kg (n = 4). The Meeting considered four trials insufficient.

Critical GAP for head cabbages in France is for one foliar application at 0.19 kg ai/ha with a PHI of 42 days. Trials from Northern France and Germany (1 × 0.19 kg ai/ha, PHI 42–49 days) matched this GAP within 25%. Total fluazifop residues were: 0.06, 0.12, 0.15, 0.16, 0.56 and 1.7 mg/kg (n = 6).

The Meeting estimated a maximum residue level of 3 mg/kg on head cabbages, based on the French GAP. The Meeting estimated a median residue of 0.155 mg/kg and a highest residue of 1.7 mg/kg.

Using multiplication factors of 1.27 and 2.16 for median and highest residues, the Meeting estimated an STMR and HR of 0.20 and 3.7 mg/kg eq.

Cucumbers and Summer squash

Field trials involving cucumbers were performed under outdoor and indoor conditions in Italy and Spain. Field trials involving summer squash were performed under outdoor conditions in Italy and South Africa.

Critical GAP for cucumber, summer squash and gherkins is the GAP from France with one foliar application of 0.19 kg ai/ha with a PHI of 28 days.

One indoor trial on cucumber from Spain (1 × 0.31 kg ai/ha, PHI 28 days, broadcast foliar application) could be matched to this GAP through proportionality. Total fluazifop residues were: 0.02 × 0.19/0.31 mg/kg (n = 1) which following the application of proportionality becomes 0.012 mg/kg (n = 1).

Outdoor trials on cucumber from Spain and Italy (1 × 0.31 kg ai/ha, PHI 27 days) could be matched to this GAP through proportionality. Total fluazifop residues were: < 0.01 mg/kg (n = 1).

One outdoor trial on summer squash from Italy (1 × 0.31 kg ai/ha, PHI 29) could be matched to this GAP through proportionality. Total fluazifop residues were: < 0.01 mg/kg (n = 1).

The Meeting considered three trials insufficient.

Tomato

Field trials involving tomatoes were performed under outdoor conditions in Spain, Italy and France.

Critical GAP for tomatoes is the French GAP for tomatoes, aubergines and peppers with one foliar application at 0.38 kg ai/ha with a PHI of 35 days. Trials from Spain, Italy and Southern France (1 × 0.31 kg ai/ha, PHI 35–42 days) matched this GAP within 25%. Total fluazifop residues were: < 0.01, < 0.01, < 0.01, < 0.05, < 0.05, 0.06, 0.12 and 0.25 mg/kg (= 8).

The Meeting estimated a maximum residue level of 0.4 mg/kg on tomatoes based on the cGAP from France. The Meeting estimated a median residue of 0.05 mg/kg and a highest residue of 0.25 mg/kg.

Using multiplication factors of 1.05 and 1.05 for median and highest residues, the Meeting estimated an STMR and HR of 0.053and 0.26 mg/kg eq.

The Meeting decided to extrapolate the maximum residue level, STMR and HR to eggplants.

Kale

Field trials involving kale were performed under outdoor conditions in the UK.

Critical GAP for kale for human consumption in France is 1 × 0.19 kg ai/ha with a PHI of 42 days. One trial from Germany (1 × 0.19 kg ai/ha, PHI 42 days) matched this GAP within 25%. Total fluazifop residues were 0.95 mg/kg (n = 1). The Meeting considered one trial insufficient.

Lettuce

Field trials involving head lettuce, leaf lettuce and Cos lettuce were performed under outdoor conditions in Greece, Spain, Italy, Southern and Northern France, Brazil, and the USA.

The cGAP for lettuces is the GAP from Brazil with one foliar application at 0.25 kg ai/ha with a PHI of 28 days. Trials from Northern France, Italy, Spain and Brazil (1 × 0.25–0.31 kg ai/ha, PHI 28–31 days) matched this GAP within 25%. Total fluazifop residues in head lettuce were < 0.01 and 0.66 mg/kg (n = 2). None of the Cos lettuce trials matched the GAP. Total fluazifop residues in leaf lettuce were: < 0.01 (7) mg/kg (n = 7).

Since head lettuce contained one high residue, the Meeting decided to estimate maximum residue levels for leaf lettuce only. The Meeting estimated a maximum residue level of 0.01* mg/kg for leaf lettuce. The Meeting estimated a median and highest residue of 0.01 mg/kg.

Using multiplication factors of 1.27 and 2.16 for long- and short-term dietary exposure, the Meeting estimated an STMR and HR of 0.013 and 0.022 mg/kg eq.

Turnip greens

Field trials involving turnips were performed in the United Kingdom.

Critical GAP for turnips in Belgium is 1 × 0.38 kg ai/ha and a PHI of 56 days. Trials from the UK (1 × 0.38 kg ai/ha, PHI 62–68 days) on turnip tops matched this cGAP within 25%. Total fluazifop residues were 1.3 and 1.6 mg/kg (n = 2). The Meeting considered two trials insufficient.

Common bean (pods and/or immature seeds) (Phaseolus spp)

Field trials involving green beans with pods were performed in Canada, Germany, the Netherlands, UK, France, Italy, and Spain.

Critical GAP for green beans with pods is the Belgium GAP with one foliar application at 0.38 kg ai/ha with a PHI of 28 days. Trials from Germany, the Netherlands, UK, France and Spain (1 × 0.30–0.38 kg ai/ha, PHI 27–35 days) matched this cGAP within 25%. Total fluazifop residues in green beans with pods were: 0.06, 0.08, 0.17, 0.23, 0.25, 0.27, 0.29, 0.32, 0.35, 0.38, 0.48, 0.84, 1.6 and 4.6 mg/kg (n = 14).

The Meeting estimated a maximum residue level of 6 mg/kg on beans (green pods and immature seeds, *Phaseolus* spp) based on the cGAP from Belgium. The Meeting estimated a median residue of 0.305 mg/kg and a highest residue of 4.6 mg/kg.

Using multiplication factors of 1.05 and 1.07 for median and highest residues, the Meeting estimated an STMR and HR of 0.32 and 4.9 mg/kg eq.

Peas (pods and succulent = immature seeds) (Pisum spp, Vigna spp)

Field trials involving green peas with pods were performed in the Netherlands, Germany, Denmark, UK, Northern France, Spain and Canada.

Critical GAP for green peas with pods is the Belgium GAP with one foliar application at 0.38 kg ai/ha with a PHI of 28 days. Trials from the UK and Northern France (1 × 0.37–0.38 kg ai/ha, PHI 34–35 days) matched this cGAP within 25%. Total fluazifop residues in green peas with pods were: 0.08, 0.23, 0.42, 0.85 and 0.90 mg/kg (n = 5).

The Meeting estimated a maximum residue level of 2 mg/kg on peas, pods and succulent immature peas (*Pisum spp, Vigna spp*) based on the cGAP from Belgium. The Meeting estimated a median residue of 0.42 mg/kg and a highest residue of 0.90 mg/kg.

Using multiplication factors of 1.05 and 1.07 for median and highest residues, the Meeting estimated an STMR and HR of 0.44 and 1.0 mg/kg eq.

Peas, shelled (succulent seeds) (Pisum spp, Vigna spp)

Field trials involving green pea seeds were performed in the Netherlands, Germany, UK, France, Italy, Spain and Canada.

Critical GAP for green peas without pods in Belgium is one foliar application at 0.38 kg ai/ha with a PHI of 28 days. However, since higher residues were observed at longer pre-harvest intervals, this cGAP was not explored further.

Critical GAP for green peas without pods in the Netherlands is one foliar application at 0.38 kg ai/ha with a PHI of 56 days. Trials from Canada, Germany, the UK (1 × 0.38–0.40 kg ai/ha, PHI 42–66 days) matched this GAP within 25%. Total fluazifop residues in green pea seeds were: < 0.05, 0.16, 0.27, 0.53, 3.8 and 7.6 mg/kg (n = 6)

The Meeting noted that despite the longer pre-harvest interval, residues according to the Dutch cGAP were higher than those for the Belgian cGAP. The Meeting estimated a maximum

residue level of 15 mg/kg on peas, shelled (succulent seeds) (*Pisum spp, Vigna spp*) based on the cGAP from the Netherlands. The Meeting estimated a median residue of 0.40 mg/kg and a highest residue of 7.6 mg/kg.

Using multiplication factors of 1.05 and 1.07 for median and highest residues, the Meeting estimated an STMR and HR of 0.42and 8.1 mg/kg eq.

Pulses

Since the processing study on dry peas has shown that soaking is essential for quantitative analysis of total fluazifop, trials were not taken into account when the soaking step was omitted or when it is not clear whether soaking was performed.

Beans (dry) (Phaseolus spp)

Field trials involving dry beans were performed in the USA, Canada, Brazil and Spain.

Critical GAP for dry beans is the USA GAP for dry beans with two foliar applications at 0.42 kg ai/ha with a PHI of 60 days. Trials from the USA with dry beans (2 × 0.42 kg ai/ha, PHI 59–75 days) matched this cGAP within 25%. Total fluazifop residues in dry beans, where a pre-extraction soaking step was included in the analytical method, were: 0.32, 0.46, 0.76, 0.82, 1.1, 1.2, 3.4, 3.6, 5.0, 9.4, 16 and 20 mg/kg (n = 12).

The Meeting estimated a maximum residue level of 40 mg/kg on beans (dry, *Phaseolus* spp). The Meeting estimated a median residue of 2.3 mg/kg.

Using multiplication factors of 1.05 for median residues, the Meeting estimated an STMR of 2.4 mg/kg eq.

Broad bean (dry) (Vicia spp)

Field trials involving dry broad beans were performed in UK, Germany, Southern France, Spain and Italy.

Critical GAP for dry broad beans is the French GAP with one foliar application at 0.38 kg ai/ha with a PHI of 56 days. Only one trial, which was not conducted to current standards, could be matched to this cGAP.

Critical GAP for pulses in the Netherlands is one foliar application at 0.38 kg ai/ha with PHI 90 days. Trials from UK with dry broad beans (1 × 0.38 kg ai/ha, PHI 97–98 days) matched the cGAP from the Netherlands within 25%. Total fluazifop residues in dry broad beans, where a pre-extraction soaking step was included in the analytical method, were: 0.08 and 0.09, mg/kg (n = 2). The Meeting considered two trials insufficient upon which to base a maximum residue level estimation.

Field pea (dry) (Pisum spp)

Field trials involving dry peas and dry field peas were performed in Netherlands, UK, Germany, and France.

Critical GAP for dry peas in France is one foliar application at 0.38 kg ai/ha with PHI 56 days. Trials from the UK, Northern France with dry peas, dry field peas or dry fodder peas (1 × 0.38 kg ai/ha, PHI 46–68 days) matched the cGAP from France within 25%. Total fluazifop residues in dry field peas were: 0.26 (no soaking step included), 0.59, 0.91 × 0.38/0.31, 2.0, mg/kg (n = 4), which becomes 0.59, 1.1 and 2.0 mg/kg (n = 3). The Meeting considered three trials insufficient upon which to base a maximum residue level estimation.

Critical GAP for dry peas in Belgium is 1 × 0.38 kg ai/ha with an application just before bloom. Trials from Germany, the UK, Northern France (1 × 0.31–0.38 kg ai/ha, BBCH 35–39) matched the cGAP from Belgium within 25%. Total fluazifop residues in dry field peas, where a pre-extraction soaking step was included in the analytical method, were: 0.02, 0.10, 0.10, 0.17, 0.18, 0.24, 0.27, 0.49, 0.54, 0.59, 0.91, 1.0, 1.1 and 2.0 mg/kg (n = 14).

The Meeting estimated a maximum residue level of 3 mg/kg on peas (dry, Pisum spp). The Meeting estimated a median residue of 0.38 mg/kg.

Using multiplication factors of 1.05 for median residues, the Meeting estimated an STMR of 0.40 mg/kg eq.

Soya bean (dry)

Field trials involving soya beans (dry) were performed in the USA, Canada, Brazil, Switzerland, Italy and France.

Critical GAP for dry soya beans in Brazil consists of one broadcast application of 0.25 kg ai/ha with a PHI of 60 days. Trials from Brazil, Italy and Northern France (0.24–0.31 kg ai/ha with PHI 56–68 days) matched the cGAP from Brazil within 25%. Additional trials from Italy and Southern France (0.38 kg ai/ha with PHI 57–60 days) could be matched to the Brazilian GAP using the proportionality principle. The Meeting decided to apply the proportionality principle on all residues where the dose rate deviated from 0.25 kg ai/ha. Total fluazifop residues in dry soya beans, where a pre-soaking step was included in the analytical method, were: 0.49, 0.93, 1.2, 1.7, 2.1, 2.4 × 0.25/0.26, 3.2 × 0.25/0.24, 4.7 × 0.25/0.31, 6.3 × 0.25/0.38^{BF}, 5.4 × 0.25/0.31, 9.8 × 0.25/0.38 and 11 × 0.25/0.31 mg/kg (n = 12), which resulted in the following dataset: 0.49, 0.93, 1.2, 1.7, 2.1, 2.3, 3.3, 3.8, 4.1^{BF}, 4.4, 6.4 and 8.9 mg/kg (n = 12), where BF indicates a banded foliar application.

The Meeting estimated a maximum residue level of 15 mg/kg on soya beans (dry, Glycine spp). The Meeting estimated a median residue of 2.8 mg/kg.

Using multiplication factors of 1.05 for median residues, the Meeting estimated an STMR of 2.9 mg/kg eq.

Carrots

Field trials involving carrots were performed in the UK, Spain, Italy, France, Brazil and the USA in different growing seasons. As it is not clear which GAP leads to the highest residues, the Meeting evaluated the residues matching the different cGAPs.

Critical GAP for carrots in the USA is 2 × 0.42 kg ai/ha with a PHI of 45 days. Trials from the USA (2 × 0.42 kg ai/ha, PHI 44–48 days) matched this GAP within 25%. Additional trials from the USA (2 × 0.56 kg ai/ha, PHI 45 days) could be matched to the US GAP using the proportionality principle. Total fluazifop residues were 0.019, 0.027, < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, < 0.05 and 0.072 mg/kg (n = 9).

Critical GAP for carrots in Brazil is 1 × 0.25 kg ai/ha with a PHI of 30 days. Trials from Brazil (1 × 0.25 kg ai/ha, PHI 30 days) matched this GAP within 25%. Additional trials from Southern France, Italy and Spain with carrots (1 × 0.31–0.32 kg ai/ha with PHI 28–29 days) could be matched to the Brazilian GAP using the proportionality principle. The Meeting decided to apply the proportionality principle on all residues where the dose rate deviated from 0.25 kg ai/ha. Total fluazifop residues were: 0.02 × 0.25/0.33^{BF}, 0.03 × 0.25/0.32^{BF}, 0.04, 0.04, 0.05 × 0.25/0.31, 0.05, 0.07 × 0.25/0.31, 0.07 × 0.25/0.31, 0.19 × 0.25/0.31, 0.17, mg/kg, which becomes 0.015, 0.023, 0.04, 0.04, 0.040, 0.05, 0.056, 0.056, 0.15 and 0.17 mg/kg (n = 10), where BF indicates a banded foliar application.

Critical GAP for carrots in France is 1 × 0.38 kg ai/ha with a PHI of 42 days. Two trials from the UK and Southern France (1 × 0.38 kg ai/ha, PHI 34–42 days) matched this GAP within 25%. Total fluazifop residue levels were < 0.05 and 0.29 mg/kg. The Meeting considered two trials insufficient.

Critical GAP for the Netherlands, UK and Belgium is 1 × 0.38 kg ai/ha with a PHI of 56 days. Trials from the UK, Southern France (1 × 0.38 kg ai/ha, PHI 42–64 days) matched this GAP within 25%. Total fluazifop residues in trials using an adjuvant were < 0.05, < 0.05, 0.09, 0.09, 0.21, 0.23 and 0.29 mg/kg (n = 7).

These data show that the residue levels based on the Dutch, UK and Belgian GAP are higher than those from the US and Brazilian GAPs. The Meeting estimated a maximum residue level of 0.6 mg/kg on carrots based on the GAP applied in the Netherlands, the United Kingdom and Belgium. The Meeting estimated a median residue of 0.09 mg/kg and a highest of 0.29 mg/kg.

Using multiplication factors of 1.95 and 2.38 for median and highest residues, the Meeting estimated an STMR and HR of 0.18, 0.69 mg/kg eq.

Celeriac

Field trials involving celeriac were performed in Northern France in two growing seasons.

Critical GAP for celeriac is the GAP from Belgium and the Netherlands with 1 × 0.38 kg ai/ha with a PHI of 56 days. Four trials from Northern France (1 × 0.38 kg ai/ha, PHI 50-56 days) matched this GAP within 25%. Total fluazifop residues were $< 0.01^{BF}$, $\underline{\leq 0.01}^{BF}$, $\underline{0.11}$, and 0.17 mg/kg, where BF indicates a banded foliar application.

The Meeting estimated a maximum residue level of 0.4 mg/kg on celeriac based on the Belgian and Dutch GAP. The Meeting estimated a median residue of 0.060 mg/kg and a highest residue of 0.17 mg/kg.

Using multiplication factors of 1.95 and 2.38 for median and highest residues, the Meeting estimated an STMR and HR of 0.12 and 0.40 mg/kg eq.

Potato

Field trials involving potatoes were performed in Brazil, Canada and Europe in various growing seasons.

Critical GAP for potatoes in Brazil is 1 × 0.25 kg ai/ha with a PHI of 28 days. Trials from Brazil and Southern France (1 × 0.25 kg ai/ha, PHI 27–29 days) matched this GAP within 25%. Additional trials from Germany (1 × 0.38 kg ai/ha with PHI 27–29 days) could be matched to the Brazilian GAP using the proportionality principle. The Meeting decided to apply the proportionality principle on all residues where the dose rate deviated from 0.25 kg ai/ha. Total fluazifop residues were: < 0.01 (3), 0.06 × 0.25/0.38, < 0.05 (3), 0.07, 0.11 and 0.44 mg/kg (n = 10), which becomes < 0.01, < 0.01, < 0.01, 0.039, $\underline{\leq 0.05}$, $\underline{\leq 0.05}$, < 0.05, 0.07, 0.11 and 0.44 mg/kg (n = 10).

The Meeting estimated a maximum residue level of 0.6 mg/kg on potato based on the Brazilian GAP. The Meeting estimated a median residue of 0.05 mg/kg and a highest residue of 0.44 mg/kg.

Using multiplication factors of 1.95 and 2.38 for median and highest residues, the Meeting estimated an STMR and HR of 0.10, 1.0 mg/kg eq.

Radish

Field trials involving radish were performed in the UK.

Critical GAP for radishes for France is 1 × 0.38 kg ai/ha with a PHI of 42 days. None of the trials could be matched to this GAP.

Critical GAP for radishes from Belgium is 1 × 0.38 kg ai/ha with a PHI of 56 days. One trial of poor quality from the UK (1 × 1.0 kg ai/ha, PHI 55 days) could be matched to this GAP using proportionality. The Meeting considered the data insufficient.

Sugar beet

Field trials involving sugar beets were performed in the United Kingdom, Germany, Spain, Italy, France, Greece, Canada and the USA. Trials from fodder beets can be used to derive maximum residue levels for sugar beets and vice versa.

Critical GAP for sugar beets in the USA is 2 × 0.42 kg ai/ha and a PHI of 90 days. Trials from the USA (2 × 0.42 kg ai/ha, PHI 89–90 days) on sugar beets matched this GAP within 25%. Total fluazifop residues were: < 0.01, 0.02, 0.06, 0.06, 0.06, 0.06, 0.08, 0.08, 0.10, 0.10, 0.11, 0.22 mg/kg (n = 12).

Critical GAP for sugar beets and fodder beets in the UK is 1 × 0.38 kg ai/ha and a PHI of 56 days. Trials on sugar beets from Germany, Greece, Italy, Spain (1 × 0.37–0.43 kg ai/ha, PHI 47–60 days) matched this GAP within 25%. Total fluazifop residues were: 0.08, 0.08^{BF}, 0.09, 0.09, 0.09, 0.10, 0.12^{BF}, 0.14^{BF}, 0.26 and 0.32 mg/kg (n = 10), where BF indicates a banded foliar spray.

The Meeting estimated a maximum residue level of 0.5 mg/kg on sugar beets based on the GAP in the United Kingdom. The Meeting estimated a median residue of 0.095 mg/kg and a highest residue of 0.32 mg/kg in roots of sugar beets and fodder beets.

Using multiplication factors of 1.95 and 2.38 for median and highest residues, the Meeting estimated an STMR and HR of 0.19 and 0.76 mg/kg eq.

Swede and Turnip

Field trials involving swedes were performed in the UK. Field trials involving turnips were performed in the United Kingdom and Canada.

Critical GAP for swedes and turnips in France is 1 × 0.38 kg ai/ha with PHI of 42 days. None of the swede trials and only one turnip trial, which was inadequately reported, could be matched to this GAP using proportionality.

Critical GAP for swedes and turnips in Belgium is 1 × 0.38 kg ai/ha with a PHI of 56 days. Two swede trials from the UK (1 × 0.38 kg ai/ha, PHI 56–70 days) matched this GAP within 25%. Total fluazifop residues were: 0.43 and 0.55 mg/kg (n = 2).

Two turnip trials from the UK (1 × 0.38 kg ai/ha, PHI 62–68 days) could be matched to this cGAP within 25%. Total fluazifop residues were: 0.74 and 2.0 mg/kg (n = 2).

The Meeting considered the trials on swedes and turnips mutually supportive and decided to combine the trials. Total fluazifop residues were: 0.43, 0.55, 0.74 and 2.0 mg/kg (n = 4).

The Meeting estimated a maximum residue level of 4 mg/kg on turnips and swedes based on the GAP in Belgium. The Meeting estimated a median residue of 0.645 mg/kg and a highest residue of 2.0 mg/kg.

Using multiplication factors of 1.95 and 2.38 for median and highest residues, the Meeting estimated an STMR and HR of 1.3 and 4.8 mg/kg eq.

Sweet potato

Field trials involving sweet potatoes were performed in the USA in the 2008 growing season.

Critical GAP for sweet potato and yam is from the USA with 4 × 0.21 kg ai/ha and a PHI of 14 days. Trials from the USA (4 × 0.21 kg ai/ha, PHI 12–16 days) matched this GAP within 25%. Total fluazifop residues were: 0.11, 0.12, 0.51, 0.52, 0.57 and 0.85 mg/kg (n = 6).

The Meeting estimated a maximum residue level of 2 mg/kg on sweet potato based on the GAP in the USA. The Meeting estimated a median residue of 0.515 mg/kg and a highest residue of 0.85 mg/kg.

Using multiplication factors of 1.95 and 2.38 for median and highest residues, the Meeting estimated an STMR and HR of 1.0, and 2.0 mg/kg eq.

The Meeting decided to extrapolate the maximum residue level, STMR and HR to yams.

Asparagus

Field trials involving asparagus were performed in the USA, Northern France and Spain.

Critical GAP for asparagus in the USA is 2 × 0.42 kg ai/ha and a PHI of 1 day. Trials from the USA (2 × 0.42 kg ai/ha, PHI 1 days) on asparagus matched this GAP within 25%. Total fluazifop residues were 1.7, 1.8 and 3.9 mg/kg (n = 3). The Meeting considered three trials insufficient.

Rhubarb

Field trials involving rhubarb were performed in the USA.

Critical GAP for rhubarb is the GAP from France with 1 × 0.19 kg ai/ha with a PHI of 42 days. None of the trials could be matched to this GAP. The Meeting considered the data insufficient.

Witlof chicory (sprouts)

Field trials involving witlof roots and sprouts were performed in Northern France and the Netherlands.

Critical GAP for witlof roots for sprout production is the GAP from Belgium, France or the Netherlands with 1 × 0.38 kg ai/ha with a PHI of 56 days for the roots. Trials from the Netherlands and Northern France (1 × 0.38 kg ai/ha, PHI 55–57 days for the roots) matched this GAP within 25%. Total fluazifop residues in the sprouts (endives) grown from these roots on hydroponic solutions were: < 0.01 and < 0.01 mg/kg (n = 2).

Trials from the Netherlands (1 × 0.38 or 0.75 kg ai/ha, PHI 101 days for the roots) with a much longer PHI, could not confirm the non-residue situation in the sprouts, since total fluazifop residues of 0.02 and 0.03 mg/kg were found in the sprouts grown from these roots. Since the non-residue situation in sprouts could not be confirmed, the Meeting considered two trials insufficient.

Sugar cane

Field trials involving sugar cane were performed in Brazil and the USA.

Critical GAP for sugar cane is the Brazilian GAP with one foliar spray with 1 × 0.075 kg ai/ha and a PHI of 42 days. This spray application is used as a desiccant to increase the sucrose content of the sugar cane.

Sugar cane trials from Brazil (1 × 0.075 kg ai/ha, PHI 35 days) could be matched to the Brazilian GAP within 25%. Total fluazifop residues were: < 0.01, < 0.01, < 0.01 and < 0.01 mg/kg (n = 4).

Since all trials were below the LOQ, the Meeting considered four trials sufficient. The Meeting estimated a maximum residue level of 0.01* mg/kg for sugar cane based on the Brazilian cGAP. The Meeting estimated a median and highest residue of 0.01 mg/kg.

Using multiplication factors of 1.05 and 1.05 for median and highest residues, the Meeting estimated an STMR and HR of 0.011 and 0.011 mg/kg eq.

Oilseeds

Cotton seed

Field trials involving cotton were performed in the USA, Brazil and Spain.

Critical GAP for cotton in Brazil is 1 × 0.25 kg ai/ha with a PHI of 60 days. Trials from Brazil (1 × 0.25 kg ai/ha with PHI 60 days) matched this GAP within 25%. Total fluazifop residues were: < 0.01 (4 ×) mg/kg (n = 4). The Meeting considered four trials insufficient.

Critical GAP for cotton in the USA is 2 × 0.42 kg ai/ha and a PHI of 90 days. Trials from the USA (2 × 0.41–0.43 kg ai/ha with PHI 88–97 days) matched this GAP within 25%. Total fluazifop residues were: < 0.01 (6), 0.016, 0.044, 0.046, < 0.05 (8), 0.08, 0.089 and 0.71 mg/kg mg/kg (n = 20).

The Meeting estimated a maximum residue level of 0.7 mg/kg on cotton seed, based on the USA GAP. The Meeting estimated a median residue of 0.05 mg/kg.

Using multiplication factors of 1.05 for median residues, the Meeting estimated an STMR of 0.053 mg/kg eq.

Rape seed

Field trials involving rape seed were performed in the UK, Germany, Spain, Southern France and Italy.

Critical GAP for oilseed rape in Brazil and the UK is 1 × 0.19 kg ai/ha with a PHI of 14 days. None of the trials could be matched to this GAP.

Critical GAP for oilseed rape in France is 1 × 0.38 kg ai/ha with a PHI of 90 days. Trials from Germany, Spain, Southern France (1 × 0.37–0.39 kg ai/ha, PHI 81–112 days) matched this GAP within 25%. Total fluazifop residues in the seeds were: 1.5, 2.0, 2.2, 2.2 and 2.3 mg/kg (n = 5). The Meeting considered five trials insufficient.

Sunflower seeds

Field trials involving sunflower seed were performed in Brazil, Germany, France, Italy, Spain, Hungary, and the USA.

Critical GAP for sunflower seed in France is 1 × 0.38 kg ai/ha with a PHI of 90 days. Trials from Germany and France (1 × 0.37–0.38 kg ai/ha, PHI 83–109 days) matched this GAP within 25%. One additional trial from Northern France (1 × 0.38 kg ai/ha, PHI 113 days) was taken into account, since significant residues were found at this longer PHI. Total fluazifop residues were: < 0.01, < 0.01, < 0.01^{BF}, 0.02, 0.04, < 0.05, < 0.05, < 0.05, < 0.05 and 0.06 mg/kg (n = 9), where BF indicates a banded foliar spray.

Critical GAP for sunflower seed in Brazil is 1 × 0.25 kg ai/ha and a PHI of 59 days. Trials from Brazil (1 × 0.25 kg ai/ha, PHI 59–67 days) matched this GAP within 25%. Additional trials from Italy and Spain (1 × 0.34–0.40 kg ai/ha, PHI 60 days) could be matched to this GAP using proportionality. Total fluazifop residues were: < 0.02, < 0.02, < 0.02, < 0.02, 0.90 × 0.25/0.40, 2.2 × 0.25/0.38, 4.0 × 0.25/0.34, 5.6 × 0.25/0.38 mg/kg, which becomes < 0.02, < 0.02, < 0.02, < 0.02, 0.56, 1.4, 2.9 and 3.7 mg/kg (n = 8).

The Meeting estimated a maximum residue level of 7 mg/kg on sunflower seed, based on the Brazilian GAP. The Meeting estimated a median residue of 0.29 mg/kg.

Using multiplication factors of 1.05 for median residues, the Meeting estimated an STMR of 0.30 mg/kg eq.

Bean forage (green)

Field trials involving green Phaseolus bean forage (haulms) were performed in southern France and Spain. The bean haulms in these trials were harvested at BBCH 49–79 and can be considered as forage.

Critical GAP for green *Phaseolus* beans in Belgium is 1 × 0.38 kg ai/ha and PHI of 28 days. Phaseolus bean forage is not grazed and is harvested at the same time as the green beans with or without pods as a by-product. Trials from Southern France and Spain (1 × 0.30–0.32 kg ai/ha, PHI 27–28 days) matched this GAP within 25%. Total fluazifop residues in green bean forage were: 0.19, 1.0, 2.1 and 2.3 mg/kg (n = 4) on an as received basis.

The Meeting estimated a median and highest residue level on the cGAP in Belgium of 1.55 mg/kg and 2.3 mg/kg, on an as received basis, respectively, for green *Phaseolus* bean forage.

Bean fodder

Three field trials involving Phaseolus bean straw were performed in Southern France and Spain. Bean straws were harvested at BBCH 89 and should be considered as fodder.

Critical GAP for dry Phaseolus beans in the USA or Brazil is 2 × 0.42 kg ai/ha with a PHI of 60 days or 1 × 0.25 kg ai/ha with a PHI of 60 days, respectively. Bean fodder is harvested at the same time as the dry Phaseolus beans. No trials matched these GAPs.

Five field trials involving Vicia bean straw were performed in Southern France, Spain and Italy in 2006. Fava bean straw was harvested at BBCH 89 and should be considered as fodder.

Critical GAP for dry Vicia beans in the Netherlands is 1 × 0.38 kg ai/ha and PHI of 90 days. Bean fodder is harvested at the same time as the dry Vicia beans. Trials from Southern France, Spain and Italy (1 × 0.31–0.32 kg ai/ha, PHI 90–93 days) matched this GAP within 25%. Total fluazifop residue levels in bean straw (PHI 90–93 days) were: 0.05, 0.37, 0.38, 1.6 and 3.1 mg/kg (n = 5) on an as received basis. On a dry-weight basis (DM = 88%), total fluazifop residue levels in bean straw were: 0.057, 0.42, 0.43, 1.8 and 3.5 mg/kg (n = 5).

The Meeting estimated a maximum residue level of 7 mg/kg (dry weight). The Meeting estimated a median and highest residue based on the cGAP in the Netherlands of 0.43 mg/kg and 3.5 mg/kg (dry weight), respectively.

Pea forage

Field trials involving green pea forage were performed in the United Kingdom, Denmark, France, Spain, and Canada. Since the GAPs do not have grazing restrictions, pea forage can be harvested at any time after treatment of either peas intended for green pea pods, green pea seeds or dry peas. According to the FAO manual green pea vines are ready for harvest from any time after pods begin to form (BBCH 70–79).

Critical GAP for dry peas in France is 1 × 0.38 kg ai/ha and critical GAP for green peas in Belgium is 1 × 0.38 kg ai/ha. Trials from the UK and Northern France matched this GAP (1 × 0.31–0.39 kg ai/ha) within 25% of the dose rate. Total fluazifop residue levels in pea forage (BBCH 77–79) were: 0.06, 0.18^{BF}, 0.31, 0.49, 0.65, 0.68, 0.92, 1.0^{BF}, 1.3, 1.8, 1.8, 2.2 and 2.3 mg/kg (n = 13) on an as received basis, where BF indicates banded foliar spray.

The Meeting estimated a median and highest residue of 0.92 and 2.3 mg/kg on pea forage an as received basis, respectively, based on the cGAP of green peas from Belgium and dry peas from France.

Pea fodder (dry)

Field trials involving dry straw or haulms from dry peas were performed in the Netherlands, Denmark, Germany, UK, Southern France, Italy, and Spain. Pea fodder is harvested at the same time as the dry pea seeds.

Critical GAP for dry peas from France is 1 × 0.38 kg ai/ha and PHI 56 of days. Field trials performed in the UK, the Netherlands, Southern France (1 × 0.31–0.38 kg ai/ha, PHI 54–65 days) matched this GAP within 25%. Total fluazifop residue levels in pea straw were: 1.1, 1.2 and 6.1 mg/kg (n = 3) on an as received basis. On a dry-weight basis (DM = 88%), total fluazifop residue levels in pea straw/haulms were: 1.3, 1.4 and 6.9 mg/kg (n = 3). The Meeting considered three trials insufficient.

Soya bean forage (green)

Field trials involving soya bean forage were performed in Canada and in South Africa. Since the GAPs do not have grazing restrictions, soya bean forage can be harvested at any time after treatment. Soya bean forage can be grazed. According to the FAO manual soya bean forage can be harvested when plants are 15–20 cm tall (sixth node) to beginning of pod formation (i.e., BBCH 16–69 or V6–R2).

Critical GAP for dry soya beans in Brazil is 1 × 0.25 kg ai/ha. Trials from Canada and South Africa (1 × 0.24–0.27 kg ai/ha) matched the GAP for Brazil within 25% of the dose rate. Total fluazifop residue levels in soya bean forage (BBCH 59–69 or V6–R2) were: 0.21, 0.53, 1.4, 1.6^{CDM},

1.9 and 4.0 mg/kg (n = 6) on an as received basis The dry matter content of the sample with superscript CDM was 35%, confirming the default value for DM content in forage.

The Meeting estimated a median and highest residue of 1.5 mg/kg and 4.0 mg/kg, respectively, for green soya bean forage on as received basis.

Soya bean hay and straw

Field trials involving soya bean hay (as dried forage) were performed in Canada. Since the GAP does not have grazing restrictions, soya bean forage for hay can be harvested at any time after treatment. According to the FAO manual soya bean forage for hay is harvested from mid-to-full bloom and before bottom leaves begin to fall or when pods are approximately 50% developed (BBCH 65–75 or R2–R3).

Critical GAP for dry soya bean in Brazil is 1 × 0.25 kg ai/ha. Trials from Canada and South Africa (1 × 0.25–0.26 kg ai/ha) matched the GAP for Brazil within 25% of the dose rate. Total fluazifop residue levels in soya bean hay (BBCH 67–75) were: 0.072^{CDM}, 0.27, 0.28, 0.58 and 1.7^{CDM} mg/kg (n = 5) on an as received basis. Drying forage to hay is expected to lead to a content of about 88% DM (default for fodder). This is confirmed in some soya bean hay samples indicated with superscript [CDM]. Forage was left to dry to hay to a moisture content between 10–20%. On a dry-weight basis (DM = 88% or study specific value), fluazifop residue levels in soya bean hay 0.085, 0.31, 0.32, 0.66 and 2.1 mg/kg (n = 5).

The Meeting estimated a maximum residue level of 4 mg/kg (dry weight) for soya bean fodder based on the cGAP in Belgium. The Meeting estimated a median and highest residue of 0.32 kg and 2.1 mg/kg, (dry weight), respectively,

One field trial involving soya bean fodder was performed in South Africa (1991). Soya bean fodder is harvested at the same time as the dry soya bean seeds.

Critical GAP for dry soya beans in Brazil is 1 × 0.25 kg ai/ha and a PHI of 60 days. One trial from South Africa matched the cGAP for Brazil. Total fluazifop residues were: 0.23 mg/kg (n = 1). The Meeting considered one trial insufficient.

Alfalfa forage (green)

Field trials involving medic pasture were performed in South Africa. Medic pastures are the *Medicago* species, commonly known as medick of burclover. This family covers over 87 species. *Medicago sativa* (alfalfa) is the best known member, which grows to 1 metre in height. Most members are low, creeping herbs, resembling clover, but with burs (seed or dry fruit). The creeping members are often used as forage crops (e.g. *M. lupulina* and *M. trunculata*). Only alfalfa (M. sativa) is in the Codex Classification.

Critical GAP from Belgium for clover and lucerne (also known as alfalfa) is 1 × 0.38 kg ai/ha with a PHI of 28 days. Trials from Saudi Arabia (1 × 0.25 kg ai/ha, PHI 28 days) could be matched to the Belgium GAP through proportionality. Total fluazifop residues were: 3.7 × 0.38/0.25, 5.1 × 0.38/0.25 and 5.3 × 0.38/0.25 mg/kg (n = 3), which becomes 5.6, 7.7 and 8.0 mg/kg (n = 3). The Meeting considered three trials insufficient.

Fodder beet

Trials from sugar beets can be used to derive maximum residue levels for fodder beet. As the Meeting estimated an STMR of 0.095 mg/kg and an HR of 0.32 mg/kg on an as received basis in roots of sugar beets, these STMR and HR values it was agreed to also apply these values to fodder beet.

Sugar beet/Fodder beet leaves or tops

Field trials involving sugar beet and fodder beet tops were performed in the United Kingdom, Denmark, Germany, Spain, Italy, France, Greece, Canada and the USA. Trials from fodder beets tops can be used to derive maximum residue levels for sugar beet tops and vice versa.

Critical GAP for sugar beets and fodder beets is the GAP from the UK with 1 × 0.38 kg ai/ha and a PHI of 56 days. Trials from Germany (1 × 0.37–0.43 kg ai/ha, PHI 47–56 days) matched this GAP within 25%. Total fluazifop residues in sugar beet tops were: 0.36, 0.37, 0.47, 0.83, 0.89^{BF}, 1.1 and 1.7 mg/kg (n = 7) on an as received basis, where BF indicates a banded foliar application.

The Meeting estimated a median and highest residue of 0.83 mg/kg and 1.7 mg/kg, respectively, on an as received basis.

Swede/Turnip leaves or tops

Field trials involving swede tops were performed in the UK. Field trials involving turnip tops were performed in the United Kingdom.

Critical GAP for swedes and turnips Belgium is 1 × 0.38 kg ai/ha and a PHI of 56 days.

Residue trials from the UK (1 × 0.38–0.42 kg ai/ha, PHI 56–70 days) on swede tops matched this cGAP within 25%. Total fluazifop residues in swede tops were: 0.75 and 0.98 mg/kg (n = 2) on an as received basis.

Trials from the UK (1 × 0.38 kg ai/ha, PHI 62–68 days) on turnip tops matched this cGAP within 25%. Total fluazifop residues in turnip tops were 1.3 and 1.6 mg/kg (n = 2) on an as received basis.

The Meeting considered the trials on swede tops and turnip tops mutually supportive and decided to combine the data. Total fluazifop residues were 0.75, 0.98, 1.3 and 1.6 mg/kg (n = 4).

The Meeting estimated a median and highest residue of 1.1 and 1.6 mg/kg, respectively, on an as received basis for swede and turnip tops for animal fodder only.

Kale forage

Field trials involving kale were performed under outdoor conditions in the UK.

Critical GAP for kale for animal fodder in the UK is one foliar application at 0.38 kg ai/ha with a PHI of 56 days. Trials from the UK (1 × 0.38 kg ai/ha, PHI 49–56 days) matched this GAP within 25%. Total fluazifop residues were: 0.10, 0.16, 0.22, 0.33, 0.97 and 0.97 mg/kg (n = 6) on an as received basis.

The authorised use in the UK for kale is for animal fodder. As animal forages are not traded, the Meeting decided not to propose a maximum residue level. The Meeting estimated a median residue of 0.275 mg/kg and a highest residue of 0.97 mg/kg on an as received basis for kale for animal fodder only.

Forage of oilseed rape

Field trials involving rape forage were performed in Germany. Canola (oilseed rape) can be grazed when the canopy height is 15–20 cm tall.

On the Dutch label it is stated that the growth of the weeds stops within 1–2 days, the weeds start dying within 1 week, and will be completed in 3–5 weeks. Immature crops used for forage will not be treated with pesticides unless they are expected to survive. After two weeks the success of application of the pesticide on crop survival will be evident. Therefore, the residue levels observed at a PHI of 14 days are used for estimation of maximum residue levels. Note that the Australian label (not submitted) includes a grazing restriction of 21 days.

Critical GAP for rape forage is from France with 1 × 0.38 kg ai/ha (leaving about 14 days for the pesticide to kill the weeds). Trials from Germany (1 × 0.38 kg ai/ha, PHI 12–18 days) matched this GAP within 25%. Total fluazifop residues were: 3.8, 4.6 and 10 mg/kg (n = 3) on an as received basis. The Meeting considered three trials insufficient.

Forage and fodder of grasses

Field trials involving grasses were performed in the Netherlands, Germany (red fescue) and the USA (fine fescue).

Critical GAP for grasses is the GAP from the Netherlands with 1 × 0.25 kg ai/ha and a PHI of 49 days. Only one trial from the Netherlands on grass forage (1 × 0.19 kg ai/ha, PHI 47 days, BBCH 47 at harvest) matched the GAP within 25%. Total fluazifop residues were: 0.09 mg/kg (n = 1) on as received basis. The Meeting considered one trial insufficient.

Two trials from Germany on grass hay (1 × 0.19 kg ai/ha, PHI 47–51, BBCH 89 at harvest) matched the GAP within 25%. Total fluazifop residues were: 0.50 and 0.94 mg/kg (n = 2) on as received basis. The Meeting considered two trials insufficient.

The Meeting did not estimate a maximum residue level, or a median and highest residue level for grasses (forage or hay).

Almond hulls

Field trials involving almond hulls were performed in the USA.

Critical GAP from France for almonds is one application at the base of the trees with 1 × 0.25 kg ai/ha and PHI of 21 days. None of the trials could be matched to the cGAP from France.

Cotton gin trash

Field trials involving cotton gin trash were performed in the USA. Cotton gin trash is harvested as a by-product at the same time as the harvest of the cotton seeds.

Critical GAP for cotton is the GAP from the USA with 2 × 0.42 kg ai/ha and a PHI of 90 days. Trials from the USA (2 × 0.41–0.43 kg ai/ha with PHI 88–97 days) matched this GAP within 25%. Total fluazifop residues in cotton gin trash were: 0.018, 0.043, 0.080, 0.16, 0.57 and 0.63 mg/kg (n = 6) on as received basis.

The Meeting estimated a median and highest residue level of 0.12 mg/kg and 0.63 mg/kg, respectively for dry cotton fodder (gin trash), based on the USA GAP.

Rotational crops

The meeting received two field rotational crop studies to investigate the actual uptake of residues from soil.

In the first field rotational crop study at four different locations in the USA fluazifop-butyl (RS) was applied onto a fallow plot at a single application of 1.1 kg ai/ha. Various rotational crops were planted at 15, 30, 60, 90 and120 days after soil treatment. Soil samples were not analysed.

No residues above the LOQ (0.02 or 0.05 mg/kg) of total fluazifop were found in any of the crop commodities at any of the plant back intervals. CF3-pyridone was not analysed.

In the second field rotational crop study at two different locations in the UK, fluazifop-P-butyl was applied onto bare soil or to oilseed rape plants at a single application of 0.38 or 0.48 kg ai/ha. Rotational crops (lettuce, wheat and carrots) were sown 1, 2, 4 or 6 months after application.

No residues above the LOQ (0.01 or 0.05 mg/kg) of total fluazifop were found in any of the crop commodities at any of the plant back intervals. CF3-pyridone was only found in carrot tops at levels < 0.01–0.13 mg/kg (at all plant back intervals) and in wheat forage at < 0.01–0.02 mg/kg wheat forage at the 4-month plant back interval.

The Meeting concluded that CF3-pyridone is the only residue that is taken up from the soil under field conditions. The Meeting concluded that it was not necessary to estimate maximum residue levels for total fluazifop in rotational crops.

CF3-pyridone is a relevant metabolite for dietary risk assessment. The dose rates as used in the field rotational crop studies (1 × 0.38 kg ai/ha or 1 × 0.48 kg ai/ha) are equal to or higher than the maximum seasonal rate listed in the GAP information for field crops (0.19–0.38 kg ai/ha) in the EU and Brazil, but they are lower than the maximum seasonal rate listed in the GAP information for field crops (0.42–0.84 kg ai/ha) or fruiting vegetables (0.84 kg ai/ha) in the USA. Therefore, dose rates as used in the available field rotational crop studies are too low to estimate CF3-pyridone levels in rotational crops. In addition, proportionality cannot be used to correct the CF3-pyridone levels found in the crop commodities, since many of the residue levels are below the LOQ. A field rotational crop study at the maximum seasonal rate for the USA, where CF3-pyridone is quantified in rotational crops is desirable.

Fate of residues during processing

Studies on the fate of residues under conditions simulation boiling, pasteurisation or sterilisation were not conducted.

Hydrolysis studies at ambient temperatures indicated that fluazifop-P-butyl was stable at pH 5 but degraded at pH 7 (DT_{50} = 78 days) and pH 9 (DT_{50} = 29 hrs). The only degradation product was fluazifop acid. Hydrolysis studies at ambient temperature indicated that the fluazifop acid is stable at pH 5, 7 and 9.

Stability of fluazifop acid was investigated under various hydrolysis conditions. Fluazifop acid is stable after 1–3 hr reflux in 0.1 M HCl or 0.1 M NaOH, which reflect more stringent conditions than normally met during cooking, pasteurisation or sterilisation.

Processing studies were undertaken for oranges, apples, cherries, plums, grapes, cauliflower, Brussels sprouts, Savoy head cabbage, kale, green pea seeds, dry harvested peas, dry harvested soya beans, potatoes, sugar beets, asparagus, cotton seed, oilseed rape seed, sunflower seed and coffee beans. Acceptable processing factors based on total fluazifop are listed in the table below. Using the $STMR_{RAC}$ values obtained from fluazifop-butyl use, the Meeting estimated STMR-Ps for processed commodities for use in the livestock dietary burden calculations and/or dietary intake calculations.

The Meeting decided to extrapolate processing factors derived from oranges to the whole group of citrus fruits,

No processing factors could be derived for commodities where the residue in the RAC was below the LOQ (apples, cherries, plums, grapes, cauliflower, Savoy head cabbage and coffee beans). Dried plums (prunes), dried grapes (raisins), roasted coffee beans and freeze-dried coffee powder had residues below the LOQ.

No processing factors could be derived for dry harvested peas, because total fluazifop residues increased after steeping and cooking, which indicates that the original RAC sample was not soaked sufficiently long before extraction and hydrolysis to release all fluazifop conjugates. Soaking is therefore a critical parameter for the analysis of pulses.

No processing factors could be derived for several soya bean oils, cottonseed oil and rape seed oil, because the hydrolysis step used in the analytical method was not radio-validated.

Commodity	Processing factors (PF) Residue: total fluazifop	PF	STMR-P (mg/kg)	HR-P (mg/kg)	Median-P residue (mg/kg)	Highest-P residue (mg/kg)
Oranges			Citrus fruit			
–orange juice	< 0.7	< 0.7 (n = 1)	= 0.011*0.7 = 0.0077	–	–	–
–orange oil	5.0	5.0 (n = 1)	= 0.011*5.0 = 0.055	–	= 0.01*5.0 = 0.05	–
–dried pulp	6.0	6.0 (n = 1)	–	–	= 0.01*6.0 = 0.06	–
Green pea seeds						
–cooked	0.83, 0.86, 0.94	0.86 (median,	0.42 × 0.86 =	8.1 × 0.86 =	–	–

Commodity	Processing factors (PF) Residue: total fluazifop	PF	STMR-P (mg/kg)	HR-P (mg/kg)	Median-P residue (mg/kg)	Highest-P residue (mg/kg)
green peas		n = 3)	0.36	7.0		
–canned green peas	0.58, 0.71, 0.81	0.71 (median, n = 3)	0.42 × 0.71 = 0.30	8.1 × 0.71 = 5.8	–	–
Dry harvested soya bean seeds	–					
–soya bean hulls	0.22, 0.37, 0.38, 0.51, 0.52, 0.65, 0.70	0.51 (median, n = 7)	–	–	2.8 × 0.51 = 1.4	–
–soya bean oil, crude	0.83	0.83 (n = 1)	2.9 × 0.83 = 2.4			–
–soya bean oil extracted meal	0.94, 0.98, 1.1, 1.2, 1.2, 1.3. 1.4	1.2 (median, n = 7)	–	–	2.8 ×1.2 = 3.4	–
–soya bean flour	0.80, 1.1, 1.1, 1.1, 1.1, 1.2	1.1 (median, n = 6)	2.9 × 1.1 = 3.2			–
–soya bean milk	0.090, 0.14, 0.18, 0.21	0.16 (median, n = 4)	2.9 × 0.16 = 0.46			–
Potatoes						
–raw potato peels	0.28, 0.34, 0.53, 0.64, 0.92	0.53 (median, n = 5)	–	–	0.05 × 0.53 = 0.026	0.44 × 0.53 = 0.23
–raw potato flesh	1.0, 1.1, 1.1, 1.1, 1.1	1.1 (median, n = 5)	0.10 × 1.1 = 0.11	1.0 × 1.1 = 1.1		
–cooked potato without peel	0.79, 0.80	0.80 (mean, n = 2)	0.10 × 0.80 = 0.080	1.0 × 0.80 = 0.80		
Sugar beet roots						
–sugar beet sugar (refined)	0.043, 0.36	0.36 (best estimate)	0.19 × 0.36 = 0.068			–
–sugar beet molasses	14	14 (n = 1)			0.095 × 14 = 1.33	–
–sugar beet dry pulp	40	40 (n = 1)			0.095 × 40 = 3.8	–
–sugar beet wet pulp (pressed pulp)	0.087	0.087 (n = 1)			0.095 × 0.087 = 0.0083	–
Sunflower seed						
–oil extracted meal (cold press)	3.1	3.1 (n = 1)			0.29 × 3.1 = 0.90	–
–hulls	0.14	0.14 (n = 1)			0.29 × 0.14 = 0.041	–
–sunflower refined oil	< 0.03	< 0.03 (n = 1)	0.30 × 0.03 = 0.0090			–

NR = no recommendation

PF based on total fluazifop only

Median-P and highest-P residues based on total fluazifop only, are used for dietary burden calculations and maximum residue level estimation)

STMR-P and HR-P are used for the long-term and short-term dietary exposure estimates and are based on the residue definition for dietary risk assessment.

Total fluazifop was shown to concentrate in orange oil (PF = 5.0, n = 1), orange dried pulp (PF = 6.0, n = 1), sugar beet molasses (PF = 14, n = 1), sugar beet dry pulp (PF = 40, n = 1) and oil extracted meal from sunflower seed (PF = 3.1, n = 1). Oil extracted meal from sunflower seed is not a commodity in trade.

The Meeting estimated a maximum residue level of 0.01 × 5 = 0.05* mg/kg for orange oil, 0.01 × 6 = 0.06* mg/kg for orange dried pulp, 0.5 × 14 = 7 mg/kg for sugar beet molasses, 0.5 × 40 = 20 mg/kg for sugar beet dry pulp. A dry matter conversion was not considered necessary.

Livestock dietary burden

The Meeting estimated the dietary burden of fluazifop-P-butyl in livestock on the basis of diets listed in the OECD Feed table 2009. Calculation from highest residue, and median-P values (some bulk commodities) provide the levels in feed suitable for estimating maximum and highest residue levels, while calculation from median and median-P values for feed is suitable for estimating STMR values for animal commodities.

The dietary burden calculation of fluazifop for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6. The calculations were made according to the livestock diets from US/CAN, EU, Australia and Japan in the OECD Feed Table 2009.

Some processed and forage commodities do not appear in the Recommendations Table (because no maximum residue level is needed) but they are used in estimating livestock dietary burdens. Those commodities are listed below.

Codex classification	Commodity	Median residue (-P) (mg/kg)	Highest residue (-P) (mg/kg)
AB 0660	Almond hulls	NR	
AB 0226	Apple pomace, dry (no suitable PF available; 0.05 (highest loqs) used)	0.05	–
VD 0071	Beans (pulses)	2.3	–
AL 1030	Bean forage (green)	1.55	2.3
AL 0061	Bean fodder	0.43 (dw)	3.5 (dw)
VB 0041	Cabbages, head	0.155	1.7
no code	Carrot, culls (root values are used)	0.09	0.29
AB 0001	Citrus pulp (0.01* × PF 6)	0.06	–
AB1203	Cotton meal (no reliable PF, 0.05 used)	0.05	–
SO 0691	Cotton undelinted seed (no reliable PF, 0.05 used)	0.05	–
AB 0691	Cotton hulls (no reliable PF, 0.05 used)	0.05	–
AM 0691	Cotton gin by products (cotton gin trash) = fodder	0.12	0.63
VD 0561	Field pea (dry)	0.38	
AB 0269	Grape pomace, dry (no reliable PF, 0.01* used)	0.01	0.01
AV 0480	Kale, as animal fodder	0.275	0.97
AL 0528	Pea, vines (green) = forage	0.92	2.3
AL 0072	Pea, hay or fodder	NR	NR
VR 0589	Potato, culls (tuber values are used)	0.05	0.44
no code	Potato dried pulp (STMR 0.05 × PF 4.4 = 0.22, PF assumed based on dry matter in dried pulp and whole potato (88/20 = 4.4)	0.22	
no code	Potato process waste (0.05 × PF 0.53 = 0.277; wet peel values are used)	0.0277	
AV 0495	Rape greens (rape forage is considered here)	NR	NR
no code	Rape seed meal	NR	
AL 1265	Soya bean, forage (green)	1.5	4.0
AL 0541	Soya bean hay	0.32 (dw)	2.1 (dw)
VD0541	Soya bean (dry)	2.8	–
no code	Soya bean, aspirated grain fractions (soya bean values used)	2.8	–
AB 1265	Soya bean, meal (2.8 × PF 1.2 = 4.7, values for oil extracted meal used)	3.4	–
AB 0541	Soya bean, hulls (2.8 × PF 0.51 = 1.4)	1.4	–
no code	Soya bean okara (pulp or tofu, fibrous part of the bean, data for oil extracted meal used)	3.4	–
DM 0659	Sugar cane, molasses (no PF 0.01* used)	0.01	–
no code	Sugar cane, bagasse (no PF 0.01* used)	0.01	–
no code	Sugar cane tops (sugar cane values used)	0.01	
no code	Sugar beet, mangel* (values of tops used)	0.83	1.7

Codex classification	Commodity	Median residue (-P) (mg/kg)	Highest residue (-P) (mg/kg)
AV 0596	Sugar beet tops	0.83	1.7
AB 0596 (dry)	Sugar beet, pulp, dry (0.095 × 40)	3.8	
AB 1201 (wet)	Sugar beet, ensilaged pulp (v residue for sugar beet root is used)	0.095	–
DM 0596	Sugar beet, molasses (0.095 × PF 14 = 1.33)	1.33	
no code	Sunflower meal (0.29 × PF 3.1 = 0.90)	0.90	
VR0497	Swede, roots	0.645	2.0
VW 0448 (paste)	Tomato, pomace, wet (no processing data, residue for tomato used)	0.05	–
VR 0506	Turnip, roots	0.645	2.0
AV 0506	Turnip, leaves or tops	1.1	1.6

		Livestock dietary burden for fluazifop-P-butyl (based on total fluazifop, expressed as fluazifop acid), ppm of dry matter diet			
		US/CAN	EU	Australia	Japan
Max	beef cattle	1.92	13.8 [A]	9.66	8.94
	dairy cattle	5.90	10.3 [C]	8.97	6.55
	poultry—broiler	1.00	4.03	2.79	1.28
	poultry—layer	1.00	4.76 [E]	2.79	1.10
Mean	beef cattle	1.28	6.63	4.40	8.94 [B]
	dairy cattle	3.41	6.13	4.42	6.55 [D]
	poultry—broiler	1.00	2.68	2.79	1.28
	poultry—layer	1.00	3.05 [F]	2.79	1.10

[A] Highest maximum beef or dairy cattle dietary burden suitable for maximum residue level estimates for mammalian meat.

[B] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.

[C] Highest maximum dairy cattle dietary burden suitable for maximum residue level estimates for milk.

[D] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

[E] Highest maximum poultry dietary burden suitable for maximum residue level estimates for poultry meat and eggs.

[F] Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs.

Residues in animal commodities

The Meeting received a lactating dairy cow feeding study, which provided information on likely residue resulting in animal tissues and milk from fluazifop-butyl residues in animal diets.

Fifteen lactating Friesian cows were fed a basal diet or a diet containing fluazifop-butyl (RS) at nominal levels of 0.2, 0.8, 3.0 and 12.0 ppm dry feed, twice daily, for 29 consecutive days, corresponding to 0.17, 0.68, 2.55 and 10.2 ppm fluazifop acid.

Parent fluazifop-butyl was not found (< 0.01 mg/L fluazifop acid eq) in individual and bulk samples of milk at any of the feeding levels. Free fluazifop acid was found at levels of 0.01 mg/L in 4 out of 14 bulk milk samples at the 12 ppm feeding level, while the individual milk samples showed no residues (< 0.01 mg/L). Lipophilic fluazifop conjugates reached mean plateau levels of 0.042 and 0.15 mg/L fluazifop acid eq within three days at the 3 and 12 ppm fluazifop-butyl feeding levels, respectively, corresponding to 2.55 and 10.2 ppm fluazifop acid in dry feed, respectively.

Lipophilic fluazifop conjugates were not found (< 0.02 mg/kg fluazifop acid eq) in the tissue samples at any of the feeding levels. Polar fluazifop related residues (fluazifop-butyl, fluazifop acid and polar fluazifop conjugates) were only found in the highest dose group with maxima of 0.13, 0.03, 0.03 and 0.06 mg/kg total fluazifop in kidney, liver, cardiac muscle or peritoneal fat, respectively. Residues in fat represent total fluazifop residues with unknown composition. The results further

indicate that the total fluazifop residues do not accumulate and rapidly decline after the application of the fluazifop-butyl containing diet has stopped.

Laying hens were fed with a basal diet or with a diet containing fluazifop-butyl (RS), once daily, for 28 days. The actual amounts of fluazifop-butyl in the feed of the four groups were 0, 0.4, 2.5 and 10.3 ppm dry feed, corresponding to 0, 0.32, 2.1 and 8.8 ppm fluazifop acid in dry feed, respectively.

Residue levels in eggs were only measurable in eggs from hens treated with the highest dose at 10.3 ppm fluazifop-butyl and the plateau level reached was 0.04 mg/kg total fluazifop at Day 7. After separation of yolk and albumen, residues were detected only in the yolk (maximum of 0.11 mg/kg total fluazifop). The mixed tissues (muscle, fat and skin) and the liver samples of hens treated with the highest dose of 10.3 ppm contained total fluazifop residues in the range of 0.01–0.04 mg/kg and 0.03–0.13 mg/kg, respectively. Total fluazifop residues declined rapidly when the birds returned to an untreated diet.

Animal commodities maximum residue levels

The animal feeding studies were performed using fluazifop-butyl, but for the estimation of the maximum residue levels in animal commodities, the feeding levels are expressed in ppm fluazifop acid dry feed.

Mammals

For maximum residue level estimation, the high residues in the tissues were calculated by extrapolating the maximum dietary burden (13.8 ppm) from the relevant feeding level (10.2 ppm fluazifop acid eq) from the dairy cow feeding study and using the highest tissue concentration from the individual animal within this feeding group.

The STMR values for the tissues would usually be calculated by interpolating the mean dietary burden (8.95 ppm) between the relevant feeding levels (2.55 and 10.2 ppm fluazifop acid eq) from the dairy cow feeding study and using the mean tissue concentrations from those feeding groups. Because residue levels at 2.55 ppm fluazifop acid eq are below LOQ, the dietary level of 0 ppm was used to establish the linear relationship, rather than the 2.55 ppm level.

For whole milk maximum residue level estimation, the high residues in the milk were calculated by extrapolating the maximum dietary burden (10.3 ppm) from the relevant feeding level (10.2 ppm fluazifop acid eq) from the dairy cow feeding study and using the highest mean milk concentration from this feeding group.

The STMR value for whole milk was calculated by interpolating the calculated mean dietary burden (6.55 ppm) between the relevant feeding levels (2.55 and 10.2 ppm fluazifop acid eq) from the dairy cow feeding study and using the mean milk concentration from those feeding groups (0.042 mg/L and 0.16 mg/L).

Dietary burden (ppm total fluazifop)	Total fluazifop (mg/kg)				
Feeding level [ppm, fluazifop acid eq]	Milk	Muscle	Liver	Kidney	Fat
Maximum residue level					
	Mean	Highest	Highest	Highest	Highest
Beef cattle (13.8) [0, 10.2]	–	0.027 [0, 0.02]	0.041 [0, 0.03]	0.18 [0, 0.13]	0.081 [0, 0.06]
Dairy cattle (10.3) [2.55, 10.2]	0.19 [0.07, 0.19 mg/L]	–	–	–	–
STMR					
	Mean	Mean	Mean	Mean	Mean
Beef cattle (8.94) [0, 10.2]	–	0.018 [0, 0.02]	0.026 [0, 0.03]	0.088 [0, 0.10]	0.048 [0, 0.055]
Dairy cattle		–	–	–	–

Dietary burden (ppm total fluazifop)	Total fluazifop (mg/kg)				
Feeding level [ppm, fluazifop acid eq]	Milk	Muscle	Liver	Kidney	Fat
(6.55) [2.55, 10.2]	0.10 [0.042, 0.16 mg/L]				

The data from the cattle feeding studies were used to support the estimation of maximum residue levels for mammalian meat and whole milk.

Residues in whole milk were estimated as 0.19 and 0.10 mg/kg, resulting from the maximum (10.3 ppm) and mean (6.55 ppm) dietary burdens, respectively.

The Meeting estimated a maximum residue level for total fluazifop in whole milk of 0.2 mg/kg. The Meeting also estimated an STMR for whole milk of 0.10 mg/kg.

Based on the mean (8.95 ppm) dietary burden, median residues were estimated as 0.018, 0.026, 0.088 and 0.048 mg/kg, respectively for mammalian muscle, liver, kidney and fat. Resulting from the maximum (13.8 ppm) dietary burden, highest residues in tissues were estimated as 0.027, 0.041, 0.18 and 0.081 mg/kg for mammalian muscle, liver, kidney and fat, respectively.

Since the residue is fat soluble, the maximum residue level for meat is based on residues in fat tissues. The Meeting estimated a maximum residue level for total fluazifop in mammalian meat, edible offal and fat of 0.09, 0.2 and 0.09 mg/kg, respectively. The Meeting estimated an STMR of 0.024 (= 0.8 × 0.018 + 0.2 × 0.048), 0.088 and 0.048 mg/kg and an HR of 0.038 (= 0.8 × 0.027 + 0.2 × 0.081), 0.18 and 0.081 mg/kg in mammalian meat, edible offal and fat, respectively.

Poultry

The fluazifop-P-butyl maximum dietary burden for poultry is 4.76 mg/kg and the mean dietary burden is 3.05 ppm.

For maximum residue level estimation in eggs, the high residues in eggs were calculated by interpolating the maximum dietary burden (4.76 ppm) between the relevant feeding levels (2.1 and 8.8 ppm) from the poultry study and using the highest residue concentrations in eggs from those feeding groups. Because residue levels at 2.1 ppm and 8.8 ppm feeding levels are below LOQ or near the LOQ of the method, the dietary level of 0 ppm was used to establish the linear relationship, rather than the 2.1 ppm level.

The STMR value for eggs was calculated by interpolating the STMR dietary burden (3.05 ppm) between the relevant feeding levels (2.1 and 8.8 ppm) from the poultry study and using the mean egg concentrations from those feeding groups. Because residue levels at 2.1 ppm and 8.8 ppm feeding levels are below LOQ or near the LOQ of the method, the dietary level of 0 ppm was used to establish the linear relationship, rather than the 2.1 ppm level.

For maximum residue level estimation in tissues, the high residues in mixed and liver poultry tissues were calculated by interpolating the maximum dietary burden (4.76 ppm) between the relevant feeding levels (2.1 and 8.8 ppm) from the poultry study and using the highest residue concentrations in tissues from those feeding groups.

The STMR value for poultry tissues was calculated by interpolating the STMR dietary burden (3.05 ppm) between the relevant feeding levels (2.1 and 8.8 ppm) from the poultry study and using the mean tissue concentrations from those feeding groups.

Dietary burden (ppm total fluazifop)	Total fluazifop residues		
Feeding level [ppm, fluazifop acid eq]	Eggs	Mixed tissues Of fat and muscle	Liver
Maximum residue level	Highest	Highest	Highest
Poultry (4.76) [0, 8.8] [2.1, 8.8]	 0.027 [0, 0.05]	 0.025 [0.015, 0.04]	 0.082 [0.05, 0.13]
STMR	Mean	Mean	Mean

Dietary burden (ppm total fluazifop)	Total fluazifop residues		
Feeding level [ppm, fluazifop acid eq]	Eggs	Mixed tissues Of fat and muscle	Liver
Maximum residue level	Highest	Highest	Highest
Poultry (3.05) [0, 8.8] [2.1, 8.8]	0.014 [0, 0.04]	0.016 [0.015, 0.020]	0.054 [0.05, 0.075]

The data from the poultry study were used to support the estimation of maximum residue levels for poultry meat and eggs.

Residues in whole eggs were estimated as 0.027 and 0.014 mg/kg, resulting from the maximum (4.76 ppm) and mean (3.05 ppm) dietary burden respectively.

The Meeting estimated a maximum residue level in eggs of 0.03 mg/kg total fluazifop. The Meeting also estimated an STMR and HR of 0.014 and 0.027 mg/kg, respectively for poultry eggs.

Total fluazifop residues estimated from the mean dietary burden (3.05 ppm) were 0.016 and 0.054 mg/kg, respectively for mixed tissues (of fat and muscle) and liver. Total fluazifop residues in mixed tissues and liver were estimated as 0.025 and 0.082 mg/kg, respectively resulting from the maximum (4.76 ppm) dietary burden.

Since the residue is fat soluble, the maximum residue level for meat is based on residues in fat tissues. The Meeting estimated a maximum residue level for poultry meat, edible offal, and fat of 0.03, 0.09, and 0.03 mg/kg, respectively. The meeting estimated an STMR of 0.016, 0.054 and 0.016 mg/kg and an HR of 0.025, 0.082 and 0.025 mg/kg, respectively, for poultry meat, edible offal and fat tissue.

RECOMMENDATIONS

On the basis of the data obtained from supervised residue trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

The Meeting recommended the following residue definition for fluazifop-P-butyl:

Definition of the residue for compliance with the MRL in plant commodities: *total fluazifop, defined as the sum of fluazifop-P-butyl, fluazifop-P-acid (II) and their conjugates, expressed as fluazifop-P-acid.*

Definition of the residue for dietary risk assessment in plant commodities: *the sum of fluazifop-P-butyl, fluazifop-P-acid (II), 2-[4-(3-hydroxy-5-trifluoromethyl-2-phenoxy)pyridyloxy] propionic acid (XL), 5-trifluoromethyl-2-pyridone (X) and their conjugates, expressed as fluazifop-P-acid.*

Definition of the residue for compliance with the MRL and for dietary risk assessment in animal commodities: *total fluazifop, defined as the sum of fluazifop-P-butyl, fluazifop-P-acid (II) and their conjugates, expressed as fluazifop-P-acid.*

The residue is fat soluble.

CCN	Commodity name	MRL mg/kg	STMR-P (mg/kg)	HR-P (mg/kg)	median residue mg/kg	highest residue mg/kg
	Commodities of plant origin					
FC 0001	Citrus fruit	0.01*	0.011	0.011	0.01	0.01
FP 0009	Pome fruits	0.01*	0.011	0.011	0.01	0.01

CCN	Commodity name	MRL mg/kg	STMR-P (mg/kg)	HR-P (mg/kg)	median residue mg/kg	highest residue mg/kg
FS 0012	Stone fruits	0.01*	0.011	0.011	0.01	0.01
FB 2005	Caneberries	0.01*	0.011	0.011	0.01	0.01
FB 0021	Currants, black, red, white	0.01*	0.011	0.011	0.01	0.01
FB 0268	Gooseberries	0.01*	0.011	0.011	0.01	0.01
FB 0269	Grapes (table, wine)	0.01*	0.011	0.011	0.01	0.01
FB 0275	Strawberries	0.3	0.063	0.13	0.06	0.12
FT 0305	Olives	0.01*	0.011	0.011	0.01	0.01
	Olives for oil production	0.01*	0.011	0.011	0.01	0.01
FI 0327	Bananas/	0.01*	0.011	0.011	0.01	0.01
VA 0385	Onion, Bulb	0.3	0.12	0.28	0.08	0.18
VA 0381	Garlic	0.3	0.12	0.28	0.08	0.18
VA 0388	Shallots	0.3	0.12	0.28	0.08	0.18
VB 0041	Cabbages, Head	3	0.20	3.7	0.155	1.7
VO 0440	Eggplant	0.4	0.053	0.26	0.05	0.25
VO 0448	Tomato	0.4	0.053	0.26	0.05	0.25
VL 0483	Lettuce, Leaf	0.01*	0.013	0.022	0.01	0.01
VP 0061	Beans, except broad bean and soya bean	6	0.32	4.9	0.305	4.6
VP 0063	Peas (pods and succulent = immature seeds)	2	0.44	1.0	0.42	0.90
VP 0064	Peas, shelled (succulent seeds)	15	0.42	8.1	0.40	7.6
VD 0071	Beans (dry)	40	2.4		2.3	
VD 0561	Field peas (dry)	3	0.40		0.38	
VD 0541	Soya bean (dry)	15	2.9		2.8	
VR 0577	Carrot	0.6	0.18	0.69	0.09	0.29
VR 0578	Celeriac	0.4	0.12	0.40	0.06	0.17
VR 0589	Potato	0.6	0.10	1.0	0.05	0.44
VR 0596	Sugar beet	0.5	0.19	0.76	0.095	0.32
VR 0497	Swede	4	1.3	4.8	0.645	2.0
VR 0506	Turnip, Garden	4	1.3	4.8	0.645	2.0
VR 0508	Sweet potato	2	1.0	2.0	0.515	0.85
VR 0600	Yams	2	1.0	2.0	0.515	0.85
GS 0659	Sugar cane	0.01*	0.011	0.011	0.01	0.01
TN 0660	Almonds	0.01*	0.011	0.011	0.01	0.01
TN 0669	Macadamia nuts	0.01*	0.011	0.011	0.01	0.01
TN 0672	Pecan	0.01*	0.011	0.011	0.01	0.01
TN 0678	Walnuts	0.01*	0.011	0.011	0.01	0.01
SO 0691	Cotton seed	0.7	0.053		0.05	
SO 0702	Sunflower seed	7	0.30		0.29	
SB 0716	Coffee beans	0.01*	0.011	0.011	0.01	0.01
Commodities of animal origin						
MM 0095	Meat (from mammals other than marine mammals)	0.09 (fat)	0.024	0.038		
MF 0100	Mammalian fats (except milk fats)	0.09	0.048	0.081		
MO 0105	Edible offal (mammalian)	0.2	0.088	0.18		
ML 0106	Milks	0.2	0.10	–		
PM 0110	Poultry meat	0.03	0.016	0.025		
PF 0111	Poultry fats	0.03	0.016	0.025		
PO 0111	Poultry, Edible offal of	0.09	0.054	0.082		
PE 0112	Eggs	0.03	0.014	0.027		
Animal feed						
AL 1030	Bean forage (green)				1.55	2.3
AL 0061	Bean fodder	7 (dw)			0.43 (dw)	3.5 (dw)
AL 0528	Pea vines (green)				0.92	2.3
AL 0541	Soya bean fodder	4 (dw)			0.32 (dw)	2.1 (dw)
AL 1265	Soya bean forage (green)				1.5	4.0
AM 1051	Fodder beet (i.e. roots)	0.5			0.095	0.32
AV 1051	Fodder beet leaves or tops				0.83	1.7
AV 0596	Sugar beet leaves or tops				0.83	1.7
AV 0506	Turnip leaves or tops				1.1	1.6
	Swede leaves or tops				1.1	1.6
AM 0506	Turnip fodder (i.e. roots)				0.645	2.0

CCN	Commodity name	MRL mg/kg	STMR-P (mg/kg)	HR-P (mg/kg)	median residue mg/kg	highest residue mg/kg
AM 0497	Swedish turnip or Swede fodder (i.e. roots)				0.645	2.0
AV 0480	Kale forage				0.275	0.97
AB 0001	Citrus pulp, dry	0.06*			0.06	
AM 0691	Cotton fodder, dry (gin trash)				0.12	0.63
AB 0541	Soya bean hulls				1.4	
AB 1265	Soya bean meal (oil extracted)				3.4	
AB 0596	Sugar beet pulp, dry	20			3.8	
AB 1201	Sugar beet pulp, wet				0.0083	
DM 0596	Sugar beet molasses	7			1.33	
	Sunflower seed, oil extracted meal (cold press)				0.90	
	Sunflower seed, hulls				0.041	
	Potato, peel				0.027	0.23
Processed foods						
OC 0541	Soya bean oil, crude		2.4		2.3	
OR 0702	Sunflower seed oil, edible		0.0090			
JF 0001	Citrus juices		0.0077			
	Orange oil	0.05*	0.055			
	Peas, green, cooked		0.36	7.0		
	Peas, green, canned		0.30	5.8		
	Potato, flesh		0.11	1.1		
	Potato, cooked without peel		0.080	0.80		
	Soya bean flour		3.2			
	Soya bean milk		0.46			
	Sugar beet, refined sugar		0.068			

FURTHER WORK OR INFORMATION

Desirable:

- A field rotational crop study at the maximum seasonal rate according to cGAP in the USA, where CF3-pyridone is quantified in rotational crops
- Supervised residue trials where hydroxyfluazifop acid (XL) is quantified using validated analytical methods.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The International Estimated Daily Intakes (IEDI) for fluazifop-P-butyl were calculated from recommendations for STMRs for raw and processed commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 3to the 2016 Report.

The IEDIs of the 17 GEMS/Food cluster diets, based on the estimated STMRs represented 40–160% of the maximum ADI of 0.004 mg/kg bw, expressed as fluazifop acid. The estimate of acceptable daily intake applies to fluazifop-P-butyl and its metabolites fluazifop acid (II), despyridinyl acid (III), CF3-pyridone (X) and hydroxyfluazifop acid (XL). An exceedance was found for GEMS/Food cluster diet G16 (160%).

The Meeting concluded that the long-term dietary exposure to residues of fluazifop-P-butyl from uses considered by the Meeting may present a public health concern.

Short-term dietary exposure

The International Estimated Short Term Intake (IESTI) for fluazifop-p-butyl was calculated from recommendations for STMRs/HRs for raw and processed commodities in combination with

consumption data for corresponding food commodities. The results are shown in Annex 4 to the 2016 Report.

For fluazifop-P-butyl the IESTI represented 40% of the ARfD (0.4 mg/kg bw, expressed as fluazifop acid). The ARfD applies to fluazifop-P-butyl and its metabolites fluazifop acid (II), despyridinyl acid (III), CF3-pyridone (X) and hydroxyfluazifop acid (XL).

On the basis of the information provided, the Meeting concluded that the short-term dietary exposure to residues of fluazifop-P-butyl, from uses considered by the Meeting, is unlikely to present a public health concern.

REFERENCES

References are listed based on alphabetical order of author name, year and then manufacturer code. For a particular author, all the references with that single author are listed, then the references with that particular author plus a second author and then the references with that particular author plus two or more other authors In case of two authors, references are listed based on author name 1, author name 2, year, manufacturer code In case of more than two authors, the entries are listed based on the first author only, then year, then manufacturer code (such references are listed as "author 1 et al" in the text).

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
PP9/0121	Agnes P	1988	Report on supervised trials for residue analysis 1988 (Fusiflex, soybean) Plant Protection and Agrochemistry Centre of the Ministry of Agriculture and Food, Budapest Report RIC1927, 19 December 1988 Non-GLP, not published Syngenta File No PP9/0121
			Not summarized because trials could not be matched to the cGAP
PP5/0607	Alferness PJ	1990	Determination of total fluazifop in crops by gas chromatography ICI Americas Inc, Richmond, CA, USA Report RR 89-073B, 8 May 1990 GLP, not published Syngenta File No: PP5/0607
			Available as Appendix G in Alferness et al, 1991, PP5/0233
PP5/0066	Alferness PJ	1992	Determination of Total Fluazifop in Crops by Gas Chromatography (WRC-91-029) ICI Agricultural Products, ICI Americas Inc, Richmond, CA, USA Report RR 91-014B, Study FLUA-91-MV-01, 25 February 1992 GLP, not published Syngenta File No PP5/0066
PP5/0233	Alferness PJ, Kleinschmidt MG	1991	Fusilade® 2000: Magnitude of the residue study on processed sunflower products (WRC-91-012) ICI Agricultural Products, Western Research Center, Richmond, CA, USA Report RR 91-010B, Project 0005-89-PR-02, 31 July 1991 GLP, not published Syngenta File No PP5/0233
-	AOCS	1997	Sampling and analysis of commercial fats and oils – Refining Loss AOCS Official Method Ca 9a-52 Revised 1997 Published
PP5/0194	Armstrong A, Mak C	1989	Fluazifop-P-butyl: Residues determined in strawberries from trials carried out in Sweden during 1988 ICI Agrochemicals, Bracknell, Berkshire, UK Report M4883B, Study 88JH197, 2 March 1989 Non-GLP, not published Syngenta File No PP5/0194
PP9/0270	Arnold DJ,	1980	PP009 : Degradation in Soil under Aerobic and Flooded Conditions in the

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
	Rapley JH, Weissler MS White RD		Laboratory ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0131B, 24 September 1980 GLP, not published Syngenta File No PP9/0270
PP5_50552	Arsenovic M	2013	Fluazifop-P-Butyl - Magnitude of the Residue on Rhubarb Rutgers State University of New Jersey, Princeton, NJ, USA Project IR-4 PR A2404 (2013), Lab ID A240410-FLR16, 2 October 2013 GLP, not published Syngenta File No PP5_50552
			Two reports available with the same number: Baron, 1987, 464387 and Arsenovic, 2013, PP5_50552
PP5_50561	Arsenovic M	2013	Fluazifop-P-Butyl: Magnitude of the Residue on Lettuce (Head and Leaf) Rutgers State University of New Jersey of New Jersey, Princeton, NJ, USA Project IR-4 PR 02072, Lab ID 00207210-FLR17, 18 October 2013 GLP, not published Syngenta File No PP5_50561
			Not summarized because trials could not be matched to the cGAP
PP5_50555	Arsenovic M	2014	Fluazifop-P-Butyl: Magnitude of the Residue on Onion (Green) Rutgers State University of New Jersey of New Jersey, Princeton, NJ, USA Project IR-4 PR 03405, Lab ID 03405/11/FLR09, 15 April 2014 GLP, not published Syngenta File No PP5_50555
			Not summarized because trials could not be matched to the cGAP
PP5_50553	Arsenovic M	2014	Fluazifop-P-Butyl: Magnitude of Residue on Strawberry, Perennial Rutgers State University of New Jersey, Princeton, NJ, USA Project IR-4 PR A2085, Lab ID A208511-FLR16, 27 May 2014 GLP, not published Syngenta File No PP5_50553
			Trials not summarized because trials could not be matched to the cGAP; Storage stability has been summarized
PP5_50556	Arsenovic M, Jolly C	2013	Fluazifop-P-Butyl: Magnitude of the Residue on Caneberry Rutgers State University of New Jersey, Princeton, NJ, USA Project IR-4 PR 03947, Lab ID 03947-10-FLR17, 21 October 2013 Not GLP, not published Syngenta File No PP5_50556
PP5_50557	Arsenovic M, Jolly C	2013	Fluazifop-P-Butyl: Magnitude of the Residue on Blueberry Rutgers State University of New Jersey, Princeton, NJ, USA Project IR-4 PR 02083, Lab ID 0208310-FLR16, 21 October 2013 Not GLP, not published Syngenta File No PP5_50557
			Trials not summarized because they did not match cGAP **Storage stability has been summarized**
444863	Askew PD, Hill IR	1985	A Comparison of the Microflora and Physicochemical Properties of Soils used in UK Laboratory Studies with those of USA Soils ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0429B, Study PP000CK10, 23 July 1985 GLP, not published Syngenta File No 444863
PP9/0512	Atreya NC,	1981	Fluazifop-butyl and its acid metabolite (fluazifop) in sugarbeet ICI Plant Protection Division, Bracknell, Berkshire, UK Report PP009B019, QA 505/PP009B019, 9 April 1981 GLP, not published Syngenta File No PP9/0512
PP9/0435	Atreya NC	1982	Fluazifop-Butyl: Residue Data Report on Pears in West Germany (1982) Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B163, 1 December 1982 Non-GLP, not published Syngenta File No PP9/0435
PP9/0366	Atreya NC	1982	Fluazifop-butyl: Commercial Processing Study on Sugar Beet

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B089, 8 February 1982 Non-GLP, not published Syngenta File No PP9/0366
			Trials not summarized because trials could not be matched to the cGAP. Processing data are summarized.
PP5/0935	Atreya NC	1982	The determination of residues of total fluazifop (fluazifop-butyl, fluazifop and conjugate esters) in crops – an internal standard procedure ICI Plant Protection Division, Bracknell, Berkshire, UK PPRAM 62/1, December 1982 Non-GLP, not published Syngenta File No PP5/0935
463114	Atreya NC	1990	Phase 3 Summary: Residue data report of PPRAM 58 The determination of residues of fluazifop-butyl (PP009), fluazifop and conjugate esters of fluazifop in eggs and chicken tissue ICI Agrochemicals, Bracknell, Berkshire, UK Report M5159B, June 1990 GLP, not published Syngenta File No 463114
			This report is relevant since original report PPRAM 58 no longer available.
463113	Atreya NC	1990	Phase 3 Summary of PPRAM 61 The determination of residues of fluazifop-P-butyl and fluazifop in milk and bovine tissues ICI Agrochemicals, Bracknell, Berkshire, UK Report M5164B, June 1990 GLP, not published Syngenta File no 463113
			Relevant since it contains radiovalidation and description of PPRAM 61.
PP9/0390	Atreya NC	1990	Phase 3 Summary of PPRAM 103: The determination of residues of 5-trifluoromethyl-2-pyridone in crops ICI Agrochemicals, Bracknell, Berkshire, UK Report M5166B, June 1990 GLP, not published Syngenta File No PP9/0390
			Relevant, contains method validation results for PPRAM 103
PP5/0799	Atreya NC	1990	Phase 3 Summary of M8941B and M4843B Fluazifop-butyl: Storage stability of Residue in Deep Frozen Crop Samples ICI Agrochemicals, Bracknell, Berkshire, UK Report M5163B, June 1990 GLP, not published Syngenta File No PP5/0799
			Contains summaries of Report PP009B157, 496/PP009B017, TMU3074, TMU3079, M8941B and M4843B. M8941B in the title probably should have been M4891B. M4891B was not submitted.
PP5/0776	Atreya NC	1993	The determination of fluazifop and reference X in Soil A liquid chromatographic method using external standardisation for fluazifop A gas chromatographic method (GC-MSD) using external standardisation for Reference X (R154719) ICI Agrochemicals, Bracknell, Berkshire, UK Report RAM 195/01, January 1993 Non-GLP, not published Syngenta File No PP5/0776
			This document is identical to Bolygo, 1990, PP5/0776, report ARAM 195, except that Atreya, 1993, PP5/0776 has an extra front page
PP9/0497	Atreya NC, Collis WMD	1980	PP009 Acid: Extractability study ICI Imperial Chemical Studies Ltd, Bracknell, Berkshire, UK Report PP009B003, QA 359/PP009B003, 19 August 1980 Non-GLP, not published Syngenta File No PP9/0497

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
PP9/0431	Atreya NC, Collis WMD	1981	Fluazifop and fluazifop-butyl: Storage Stability study for soyabean (Canada, 1979) ICI, Imperial Chemical Studies Ltd, Bracknell, Berkshire, UK Report QA 496/PP009B017, Study R3/301, 16 March 1981 Non-GLP, not published Syngenta File No PP9/0431 Syngenta File No PP5/0969
PP9/0697	Atreya NC, Collis WMD	1982	Fluazifop-butyl & fluazifop: recovery data and GC-MS confirmation, soya and cotton, USA, 1982 ICI Imperial Industries Ltd, Bracknell, Berkshire, UK Report PP009B151, 14 October 1982 Non-GLP, not published Syngenta File No PP9/0697 **Also submitted as appendix in Bussey, 1990, 407595, report RR 90-103B**
PP9/0606	Atreya NC, Collis WMD	1983	Fluazifop-butyl: Residue Data Report for Soybeans in South Africa (1982) ICI Imperial Chemical Studies Ltd, Bracknell, Berkshire, UK, Report PP009B176, QA 1056/PP009B176, 10 January 1983 Non-GLP, not published Syngenta File No PP9/0606 **Trials on soya forage/straw were summarized. Trials on soya seeds were not summarized because they could not be matched to the cGAP**
PP9/0728	Atreya NC, Dick JP	1984	Fluazifop-butyl: Residue Data Report on Lettuce, Cucumber, Carrots, Onions in USA (1983) ICI Plant protection Division, Bracknell, Berkshire, UK Report PP009B272, QA 1324/PP009B272, 25 January 1984 GLP, not published Syngenta File No PP9/0728
PP9/0384	Atreya NC, Froggatt DA	1981	Fluazifop-butyl and fluazifop: Residue levels on crop samples taken from trials during 1979-1980 ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0226B, 21 December 1981 Non-GLP, not published Syngenta File No PP9/0384 This report is a compilation of report on several crops filed under PP009B001-B005, PP009B007-B011, PP009B013-B016, PP009B18-B019, PP009B021-B029, PP009B033-B036, PP009B038-B039, PP009B043-B058, PP009B060, and PP009B062-B063 Trials in sugar beets, potatoes, carrots, radisch, onions, lettuce, cabbage/broccoli, peas, beans, soya beans, cucumber, pome fruit, grapes, strawberry, linseed, cotton, sunflower, rapeseed, tobacco, soil The trials were not performed according to a GAP submitted for the current evaluation, except for a few trials on potatoes
PP9/0039	Atreya N, Froggatt DA	1983	Fluazifop-butyl: Storage Stability in sugarbeet, strawberries, oilseed rape, green beans, cauliflower Imperial Chemical Industries PLC, Bracknell, Berkshire, UK Report PP009B157, Study PP009BCO4, QA 996/PP009B157, 1 July 1983 Non-GLP, not published Syngenta File No PP9/0039
PP9/0613	Atreya NC, Harradine KJ	1982	Fluazifop-butyl: Residue Data Report on Oranges in Brazil ICI Plant Protection Division, Bracknell, Berkshire, UK Report PP009B117, QA 818/PP009B117, 15 April 1982 GLP, not published Syngenta File No PP9/0613
PP9/0062	Atreya NC, Harradine KJ	1982	Fluazifop-butyl: Residue levels on Crop Samples taken from Trials During 1980-81 ICI Imperial Chemical Industries PLC, Bracknell, Berkshire, UK Report RJ0291B, 26 November 1982 Non-GLP, not published Syngenta File No PP9/0062 This report is a compilation of reports on several crops filed from PP009B037, PP009B064-B075, PP009B078-B079, PP009B082-B085, PP009B087-B088, PP009B090-B123, PP009B127-B136, PP009B138, PP009B141-B146 Trials in

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			sugar and fodder beets, potatoes, carrots, turnips, beetroot, onions, leeks, lettuce and spinach, red-, savoy-, white- cabbage and cauliflower, kale, peas, beans, soya beans, tomato, melon, oranges, pome fruit, cherries, peaches, plums, black currants, red currants, gooseberries, raspberries, strawberries, grapes, pineapple, lupin, rapeseed, rapeseed, tobacco, soil. The trials were not performed according to a GAP submitted for the current evaluation, except for some trials on sugar and fodder beets, peas, strawberries, rapeseed, sunflower, and coffee
PP9/0722	Atreya NC, Harradine KJ	1983	Fluazifop-butyl: Residue Data Report on Soyabean in Canada (1982) ICI Imperial Chemical Industries PLC, Bracknell, Berkshire, UK, Report PP009B261, QA 1275/PP009B261, 4 November 1983 Non-GLP, not published Syngenta File No PP9/0722 **This trial is also available in Atreya et al, 1983, PP9/0669, report PP009B229** **Not summarized because trials could not be matched to the cGAP**
PP9/0726	Atreya NC, Harradine KJ	1983	Fluazifop-butyl: Residue Data Report on Soyabeans in Canada (1983) ICI Imperial Chemical Industries PLC Bracknell, Berkshire, UK, Report PP009B265, QA 1296/PP009B265, 22 November 1983 Non-GLP, not published Syngenta File No PP9/0726
PP9/0355	Atreya NC, Houlden AC	1980	The determination of residues of fluazifop-butyl (PP009) in soil – a high pressure liquid chromatographic method ICI Agrochemicals, Bracknell, Berkshire, UK Report PPRAM 54, December 1980 Non-GLP, not published Syngenta File No PP9/0355 **See also Jones, 1991, no code, PPRAM 54 addendum**
PP9/0271	Atreya nC, Houlden AC	1981	Fluazifop-butyl: PP009 - Laboratory Degradation in Two Standard Soils ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0183B, 16 January 1981 GLP, not published Syngenta File No PP9/0271
PP5/0779	Atreya NC, Jones SD	1995	The determination of residues of fluazifop-P-butyl in soil An external recovery method with determination by either High Performance Liquid Chromatography (HPLC) or Gas-Liquid Chromatography (GLC) Zeneca Agrochemicals, Bracknell, Berkshire, UK SOP RAM 054/02, 4 August 1995 Non-GLP, not published Syngenta File No PP5/0779
PP9/0633	Atreya NC, Upton B	1982	Fluazifop-butyl: Residue Data Report on Coffee in Brazil (1981) Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B122, 26 April 1982 Non-GLP, not published Syngenta File No PP9/0633
PP9/0102	Atreya NC, Upton BP	1984	Extractability study on weathered residues, fluazifop butyl, lettuce and fodder beet ICI Plant Protection Division, Bracknell, Berkshire, UK Report PP009B281, QA 1382/PP009B281, 10 April 1984 Non-GLP, not published Syngenta File No PP9/0102, Syngenta File No PP9/0382
PP9/0731	Atreya NC, Upton BP	1984	Fluazifop-butyl - Reference X: Residue Data Report for Carrots, Onions and Sugarbeet in the USA (1983) ICI Agrochemicals, Bracknell, Berkshire, UK Report PP009B290, QA 1381/PP009B290, 7 February 1984 GLP, not published Syngenta File No PP9/0731
PP9/0498	Atreya NC, Collis WMD, Houlden AC	1980	PP009: Residues Determined in Soybean from Trials Carried out in Canada During 1979 Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B004, QA 360/PP009B004, 25 July 1980 Non-GLP, not published Syngenta File No PP9/0498

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Not summarized because trials could not be matched to the cGAP
PP9/0504	Atreya NC, Harradine KJ, Rounds LB	1980	Fluazifop and Fluazifop-butyl Determined in Potatoes from Trial carried out in Holland During 1980 ICI Plant Protection Division, Bracknell, Berkshire, UK, Report PP009B010, 2 December 1980 Non-GLP, not published Syngenta File No PP9/0504
			Not summarized because trials could not be matched to the cGAP
PP9/0499	Atreya NC, Houlden AC, Ross P	1980	PP009: Residues Determined in Sugarbeet from Trials Carried out in Canada During 1979 Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report 367/PP009B005, 15 August 1980 Non-GLP, not published Syngenta File No PP9/0499
			Not summarized because trials could not be matched to the cGAP
PP9/0502	Atreya NC, Houlden AC, Tummon OJ	1980	Fluazifop-Butyl: Residues Determined in Sugarbeet from Trials Carried out in the UK During 1979 Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B008, QA No 400/PP009B008, 18 September 1980 Non-GLP, not published Syngenta File No PP9/0502
			Not summarized because trials could not be matched to the cGAP
PP9/0501	Atreya NC, Tummon OJ, Harradine KJ	1980	Fluazifop-Butyl: Residues Determined in Sunflower Seed from Trials Carried out in Canada During 1979 Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B007, QA No 374/PP009B007, 15 August 1980 Non-GLP, not published Syngenta File No PP9/0501
			Trials not summarized because trials could not be matched to the cGAP **Only fluazifop-butyl and free fluazifop acid were analysed** **Addressed in the metabolism section**
PP9/0385	Atreya NC, Tummon OJ, Houlden AC	1980	The Determination of Residues of Fluazifop-butyl (PP009) in Crops – a High Pressure Liquid Chromatographic Method ICI Imperial Chemical Industries, Bracknell, Berkshire, UK PPRAM 51, 22 October 1980 Non-GLP, not published Syngenta File No PP9/0385
PP9/0508	Atreya NC, Collis WD, Freeman BL, Rounds LB	1981	Fluazifop-Butyl and its acid metabolite: Residues in Sugar Beet from Trials Carried out in West Germany in 1980 Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B014, QA 458/PP009B014, 14 January 1981 Non-GLP, not published Syngenta File No PP9/0508
			Not summarized because trials could not be matched to the cGAP
PP9/0510	Atreya NC, Collis WMD, French DA, Rounds LB	1981	Fluazifop-butyl: Residue Data Report for Soyabean in Canada (1980) ICI Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK Report PP009B016, QA 495/PP009B016, 16 March 1981 Non-GLP, not published Syngenta File No PP9/0510
			Not summarized because trials could not be matched to the cGAP
PP9/0182	Atreya NC, Dick JP, Harradine K	1981	Fluazifop-butyl: Residue Transfer Study with Dairy Cows Fed on a Diet Containing the Herbicide ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0215B, 6 August 1981 GLP, not published Syngenta File No PP9/0182
PP9/0544	Atreya NC, Dick JP,	1981	Fluazifop: Fractionation Study on Soyabean (America/Canada) ICI Imperial Chemical Industries, Bracknell, Berkshire, UK

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
	Harradine KJ		Residue Data Report PP009B059, QA 618/PP009B059, 3 September 1981 Non-GLP, not published Syngenta File No PP9/0544
PP9/0734	Atreya NC, Freeman BL, Froggatt DA	1981	Fluazifop-butyl and its acid metabolite in cotton in the USA in 1979, 1980 ICI Imperial Chemical Industries, Bracknell, Berkshire, UK Report PP009B035, QA 549/PP009B035, 15 June 1981 Non-GLP, not published Syngenta File No PP9/0734
			Report also present as appendix in Ussary, 1981, 405792,TMU0679/B
PP9/0525	Atreya NC, Freeman BL, Froggatt DA	1981	Fluazifop-butyl: Residue Data Report for Field Peas in Australia (1979) ICI Plant Protection Division, Bracknell, Berkshire, UK Report PP009B038, QA 555/PP009B038, 22 June 1981 Non-GLP, not published Syngenta File No PP9/0525
			Not summarized because trials could not be matched to the cGAP
PP9/0554	Atreya NC, Freeman BL, Froggatt DA	1981	Fluazifop-butyl: Residue Data Report for Dried Peas in the UK (1981) ICI Plant Protection Division, Bracknell, Berkshire, UK Report PP009B070, QA 655/PP009B070, 3 November 1991 Non-GLP, not published Syngenta File No PP9/0554
			Part of the dry pea trials were not summarized because trials could not be matched to the cGAP; other dry pea trials were summarized
PP9/0509	Atreya NC, Freeman BL, Rounds LB	1981	Fluazifop-butyl and its Acid Metabolite, Residue Trials in Winter Oil Seed Rape (1979-1980) Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B015, QA No 489/PP009B015, 9 March 1981 Non-GLP, not published Syngenta File No PP9/0509
			Not summarized because trials could not be matched to the cGAP **Only the fluazifop-butyl and free fluazifop residues were analysed**
PP5/0606	Atreya NC, Froggatt DA, Tummon OJ	1981	The Determination of Residues of Fluazifop-Butyl, Fluazifop and its Conjugates in Crops ICI Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, PPRAM 62, 24 August 1981 Non-GLP, not published Syngenta File No PP5/0606
PP9/0552	Atreya NC, Harradine KJ, Froggatt DA	1981	Fluazifop-butyl: Residue Data Report for Winter Field Beans in the UK (1981) ICI Plant protection Division, Bracknell, Berkshire, UK Report PP009B068, QA 650/PP009B068, 2 November 1981 Non-GLP, not published Syngenta File No PP9/0552
			Not summarized because trials could not be matched to the cGAP
PP9/0507	Atreya NC, Harradine KJ, Rounds LB	1981	Fluazifop-butyl and Fluazifop Resdiues Determined in Potatoes from Trial carried out in West Germany During 1980 ICI Plant Protection Division, Bracknell, Berkshire, UK, Report PP009B013, QA 446/PP009B013, 8 January 1981 Non-GLP, not published Syngenta File No PP9/0507
			Trials not summarized because only fluazifop-butyl and free fluazifop acid were analysed; addressed in the metabolism section.
PP9/0733	Atreya NC, Kipps MR, Stanley PD	1981	Fluazifop-butyl Ref X: Residue Data Report for Cotton seed & Soya bean in the USA (1980) ICI Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B061, QA 629/PP009B061, 9 October 1981 Non-GLP, not published Syngenta File No PP9/0733
462775	Atreya NC, Tummon OJ, Froggatt DA	1981	Fluazifop-butyl Acid hydrolysis of fluazifop conjugate esters to fluazifop ICI Imperial Chemical Indus0tries, Bracknell, Berkshire, UK Report PP009B030, QA 527/PP009B030, 25 August 1981

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Non-GLP, not published Syngenta File No 462775
PP9/0523	Atreya NC, Upton B, Froggatt DA	1981	Fluazifop-Butyl and its Acid Metabolite in Sunflower (Canada, 1980) Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report 546/PP009B034, 12 June 1981 Non-GLP, not published Syngenta File No PP9/0523
PP9/0736	Atreya NC, Upton B, Froggatt DA	1981	**Not summarized because trials could not be matched to the cGAP** Fluazifop and Fluazifop-butyl Residues Determined in Soyabean in the USA (1980, 1979, 1978) ICI Imperial Chemical Industries, Bracknell, Berkshire, UK Report PP009B036, QA 664/PP009B036, 11 November 1981 Non-GLP, not published Syngenta File No PP9/0736
PP9/0527	Atreya NC, Upton B, Froggatt DA	1981	**Not summarized because trials could not be matched to the cGAP** **Study summarized in metabolism section. Additional trial information is available in Ussary, 1981, 406278, report TMU0678/B revised.** Fluazifop-butyl: Residue Data Report for Soya in the USA (1980) – Soya Fractionation study ICI Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B040, QA 557/PP009B040, 22 June 1981 Non-GLP, not published Syngenta File No PP9/0527
PP9/0702	Atreya NC, Froggatt DA, Harradine KJ	1982	Processing Study Fluazifop-butyl, Potato, UK, 1982 ICI Plant Protection Division, Bracknell, Berkshire, UK Report PP009B153, QA 990/PP009B153, 28 October 1982 GLP, not published Syngenta File No PP9/0702
PP9/0710	Atreya NC, Pay J, Harradine KJ	1982	Fluazifop-butyl: Residue Data Report on Peaches in West Germany (1982) Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK Report PP009B159, 19 November 1982 Non-GLP, not published Syngenta File No PP9/0710
PP9/0436	Atreya NC, Pay J, Harradine KJ	1982	Fluazifop-butyl: Residue Data Report for Grapes in West Germany (1981) Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK Report PP009B139, 15 September 1982 Non-GLP, not published Syngenta File No PP9/0436
PP9/0432	Atreya NC, Pay J, Harradine KJ	1982	Fluazifop-butyl: Residue Data on Apples in West Germany (1982) Imperial Chemical Industries PLC, Bracknell, Berkshire, UK, Report PP009B167, 14 December 1982 Non-GLP, not published Syngenta File No PP9/0432
PP9/0700	Atreya N, Upton BP, Freeman BL	1982	Fluazifop-butyl : Commercial processing study on soyabean and cotton ICI Imperial Chemical Industries, Bracknell, Berkshire, UK, Report PP009B152, QA 978/PP009B152, 15 October 1982 Non-GLP, not published Syngenta File No PP9/0700
ASF64_10000	Atreya N, Upton BP, Freeman BL	1982	Total Fluazifop Residues in Swedes from trials in the UK During 1981 ICI Imperial Chemical Industries, Bracknell, Berkshire, UK, Report PP009B169, QA 1043/PP009B169, 13 December 1982 Non-GLP, not published Syngenta File No ASF64_10000
PP9/0433	Atreya NC, Upton B, Rounds LB	1982	Fluazifop-Butyl: Residue Data on Apples in West Germany (1981) Imperial Chemical Industries PLC, Bracknell, Berkshire, UK Report PP009B120, 4 April 1982 Non-GLP, not published Syngenta File No PP9/0433
PP9/0434	Atreya NC, Upton B, Rounds LB	1982	Fluazifop-Butyl: Residue Data Report on Pears in West Germany (1981) Imperial Chemical Industries PLC, Bracknell, Berkshire, UK, Report PP009B127, 19 April 1982 Non-GLP, not published Syngenta File No PP9/0434

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
PP9/0644	Atreya NC, Upton B, Rounds LB	1982	Fluazifop-butyl: Residue Data Report on Peaches in West Germany (1981) Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK Report PP009B132, 19 May 1982 Non-GLP, not published Syngenta File No PP9/0644
PP9/0669	Atreya NC, Collis WMD, French DA, Harradine KJ	1983	Fluazifop-butyl: Residue Data Report for Soya bean in Canada (1981) ICI Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, Report PP009B229, QA 1175/PP009B229, 25 April 1983 Non-GLP, not published Syngenta File No PP9/0669 **See also Atreya & Harradine, 1983, PP9/0722, report PP009B261 for additional information on some trials. Only trial Mordon-U4 was summarized. Other trials were not summarized because trials could not be matched to the cGAP.**
PP9/0034	Atreya NC, Dick JP, Harradine KJ	1983	The determination of residues of fluazifop-butyl and fluazifop (free and lipophilic conjugates) in milk and bovine tissues – A high pressure liquid chromatography gas chromatography – mass spectrometry method Report PPRAM 61/1, 28 March 1983 Non-GLP, not published Syngenta File No PP9/0034 Syngenta File No PP9/0342
PP5/1047	Atreya NC, Dick JP, Upton B	1983	The determination of residues of total fluazifop (fluazifop-butyl, fluazifop and conjugate esters) in crops – an internal standard procedure ICI Plant Protection Division, Bracknell, Berkshire, UK Report PPRAM 62/2, 28 March 1983 Non-GLP, not published Syngenta File No PP5/1047
PP9/0621	Atreya NC, Pay J, Harradine KJ	1983	Fluazifop-butyl: Residue Data Report for Peaches in Italy (1982) Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK Report PP009B187, 17 January 1983 Non-GLP, not published Syngenta File No PP9/0621
PP9/0628	Atreya NC, Pay J, Harradine KJ	1983	Fluazifop-butyl: Residue Data Report for Hazelnuts in Italy (1982) Imperial Chemical Industries PLC, Bracknell, Berkshire, UK, Report PP009B194, 9 February 1983 Non-GLP, not published Syngenta File No PP9/0628
PP9/0437	Atreya NC, Pay J, Harradine KJ	1983	Fluazifop-Butyl: Residue Data Report for Grapes in West Germany (1982) Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK Report PP009B180, 7 January 1983 Non-GLP, not published Syngenta File No PP9/0437
PP9/0065	Atreya NC, Dick JP, Upton, BP	1984	Fluazifop-butyl: 'Total Fluazifop', Reference III, Reference X, Carrots, USA, 1983 ICI Plant Protection Division, Bracknell, Berkshire, UK Report PP009B300, 5 March, 1984 GLP, not published Syngenta File No PP9/0065 **One trial summarized; other trials not summarized because trials could not be matched to the cGAP. Data on metabolites summarized in metabolism section**
PP5/0590	Atreya NC, Davy GS, Patel A, Cassidy E	1997	Fluazifop-P-butyl: Field Crop Rotation Study Carried out in the UK During 1993-95 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2202B, Study 93JH167, TK0258193, 27 March 1997 GLP, not published Syngenta File No PP5/0590
PP5/1178	Atreya NC, Jones SD, Hargreaves SL	2000	Residue analytical method for the determination of residues of fluazifop-P-butyl in soil Zeneca Agrochemicals, Bracknell, Berkshire, UK SOP RAM 054/03, 30 October 2000 Non-GLP, not published Syngenta File No PP5/1178

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
-	Aver E, Bolygo E, Williams JR	1993	Fluazifop-P-butyl: Residues in Alfalfa From Field Study (91JH330F) carried Out in Saudi Arabia During 1991-92. ICI Agrochemicals, Bracknell, Berkshire, UK Report RJ1338B, Study 91JH330F, 9 August 1993 GLP, not published Syngenta File No no code
PP5_50403	Bang J	2013	Fluazifop-P-Butyl: Evaluating the Comparability of Non-US Soils Used for Environmental Fate Studies for Fluazifop-P-Butyl with the Use Area Soils in the United States Syngenta Crop Protection, LLC, Greensboro, NC, USA Report TK0058358, Task TK0058358, 16 July 2013 Not GLP, not published Syngenta File No PP5_50403
A13680A_10002	Baptista GC, Bahia, O	2006	Fusiflex – Magnitude de resíduos de Fomesafen e Fluazifop em grãos de soya - Brasil, 2005 Laboratório de Resíduos e Meio Ambiente, Sao Paulo, Brasil Report M04064, 15 February 2006 GLP, not published, Syngenta File No A13680A_10002
			Trial on green soya seeds not summarized because a cGAP is not available. Trials on dry soya seeds were summarized.
PP5_50290	Barney WP	2011	Fluazifop-P-butyl – Magnitude of the Residue on Sweet Potato IR-4 Project Headquarters, Princeton, NJ, USA Project IR-4 PR 02328 (2011), Task TK0001590, 26 May 2011 GLP, not published Syngenta File No PP5_50290
			Two reports with the same number: PR 02328 (1990) and PR 02328 (2011)
PP5_50291	Barney WP	2011	Fluazifop-P-butyl – Magnitude of the Residue on Coffee IR-4 Project Headquarters, Princeton, NJ, USA Project IR-4 PR 03432 (2011), Task TK0001591, 9 November 2011 GLP, not published Syngenta File No PP5_50291
			Two reports with the same number. PR 03432 (1988) and PR 03432 (2011)
-	Baron JJ	1986	Fluazifop-Magnitude of Residue on Cucumbers: Corrected Reports North Carolina State University, NC, USA IR-4 PR 1878, 20 February 1986 Unpublished, 54 p Syngenta File no: no code available
			Corrected report for for IR-4, PR1878 (NC), 1984, no code
464387	Baron, J	1987	Fluazifop - Magnitude of the Residue on Rhubarb Northeast Regional Laboratory, Cornell University, Geneva, NY, USA Project IR-4 PR 2404 (1987), 10 April 1987 GLP, not published Syngenta File No 464387
			Contains original reports for trials in Maryland 1984-1985 **Two reports available with the same number: Baron, 1987, 464387 and Arsenovic, 2013, PP5_50552**
464389	Baron J	1987	Fluazifop - Magnitude of Residue on Asparagus USDA-ARS Yakima Analytical Laboratory, Yakima, WA, USA Project IR-4 PR 2201, 1 September 1987 Not GLP, not published Syngenta File No 464389
471695	Baron, J	1988	Fluazifop – Magnitude of Residue on Coffee University of Hawaii, Honolulu, HI, USA Project IR-4 PR 03432 (1988), 27 June 1988 No GLP, not published Syngenta File No 471695
			Two reports with the same number: 03432 (1988) and 03432 (2011)
464386	Baron J	1989	Fluazifop – Magnitude of Residue on Macadamia Nut

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			University of Hawaii, Department of Agricultural Biochemistry, Honolulu, HI, USA Project IR-4 PR 3431, 21 February 1989 GLP, not published Syngenta File No 464386
-	Baron J	1989	Fluazifop: Magnitude of Residue on Asparagus IR-4 Northcentral Analytical Laboratory Michigan State University, MI, USA Study IR-4 PR 3944, 10 April, 1989 GLP, unpublished report Syngenta File no no code
463130	Baron J	1990	Phase 3 Summary of MRID 40241901 - Fluazifop - Magnitude of the Residue on Rhubarb University of Maryland, New York, USA IR-4 Project Headquarters, Rutgers University, New Brunswick, NJ, USA Project IR-4 PR 2073, 10 May 1990 GLP, not published Syngenta File No 463130 **Summary of Baron, 1987, 464387, report IR-4 PR 2404 (1987), but contains some additional informaton on the trials conducted in MD, USA, 1984-1985**
-	Baron JJ	1990	Phase 3 summary of MRIDs 144014 and 164500; Fluazifop: Magnitude of the residue on Sweet Potato North Carolina State University, Louisiana State University and ICI Americas, Inc Project IR-4 2328 (1990), 10 May 1990 GLP, not published Syngenta File No no code available. **Summary report. Original data were not submitted.** **Trials not summarized because they did not match with the cGAP** **Two reports with the same number: PR 02328 (1990) and PR 02328 (2011)**
PP5/1488	Bell, A	2006	Fluazifop-P-butyl (PP5) - Residue study in or on Sunflowers in Southern France and Italy during 2005 CEMAS, North Ascot, UK Report CEMR-2690, 31 August 2006 GLP, not published Syngenta File No PP5/1488
PP5/1489	Bell A	2006	Fluazifop-P-butyl (PP5) – Residue study on Leeks in Northern France CEMAS, North Ascot, Berkshire, UK, Report CEMR-2687, Study CEMS-2687, 11 August 2006 GLP, not published Syngenta File No PP5/1489
PP5/1487	Bell A	2006	Fluazifop-P-butyl (PP5) – Residue study in or on Potatoes in the UK and Northern France during 2005 CEMAS, North Ascot, Berkshire, UK, Report CEMR-2688, Study CEMS-2688, 25 August 2006 GLP, not published Syngenta File No PP5/1487
PP5/1486	Bell A	2006	Fluazifop-P-butyl (PP5) – Residue study in or on Potatoes in Spain during 2005 CEMAS, North Ascot, Berkshire, UK, Report CEMR-2689, Study CEMS-2689, 31 August 2006 GLP, not published Syngenta File No PP5/1486
PP5/1545	Bell A	2007	Fluazifop-P-Butyl (A12791B): Residue study on dried beans in southern France, Spain and Italy in 2006 CEMAS, North Ascot, UK Report CEMR-3008, Study CEMS-3008, 2 November 2007 GLP, not published Syngenta File No PP5/1545 (47 pages) Syngenta File No A12791B_10428 (47 pages)
A12791B_10432	Bell A	2007	Fluazifop-P-Butyl (PP5) : Residue study on potatoes in southern France in 2006 Syngenta Crop Protection AG, Basel, CH, CEMAS, North Ascot, Berkshire, UK, Report CEMR-3374, Study T002032-06, 18 October 2007

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			GLP, not published Syngenta File No A12791B_10432
PP5/1555	Bell A	2007	Fluazifop-P-Butyl (PP5): residue study on potatoes in Spain in 2006 CEMAS, North Ascot, Berkshire, UK, Report CEMR-3375, Study T000825-06, 19 October 2007 GLP, not published Syngenta File No PP5/1555
PP5/1550	Bell A	2007	Fluazifop-P-butyl (PP5) – Residue study on Fresh Peas without pods in Spain in 2006 CEMAS, North Ascot, Berkshire, UK, Report CEMR-3012, Study CEMS-3012, 18 October 2007 GLP, not published Syngenta File No PP5/1550
PP5/1552	Bell A	2008	Fluazifop-P-butyl (PP5): Residue study on Fresh Peas without pods in the UK and Northern France in 2006 CEMAS, North Ascot, Berkshire, UK, Report CEMR-3009, Study CEMS-3009, 22 January 2008 GLP, not published Syngenta File No PP5/1552
PP5/1544	Bell A	2008	Fluazifop-P-butyl (PP5): Residue study on dried peas in Southern France, Spain and Italy in 2006, CEMAS, North Ascot, Berkshire, UK Report CEMR-3373, Study T000800-06, Project CEMS-3373, 22 January 2008 GLP, not published Syngenta File No PP5/1544 Syngenta File No PP5/1556
A1279B_10430	Bell A	2008	Fluazifop-P-butyl (PP5): Residue Study on Fresh Beans with Pods in Southern France in 2006 ICI Agrochemicals, Bracknell, Berkshire, UK Report CEMR-3014, Study CEMS-3014, 22 January 2008 GLP, not published Syngenta File No A1279B_10430
PP9/0175	Bell EG, Cavell BD	1983	Fluazifop-butyl: Quantification of Radioactive Residues in rotational Crops Following Soil Treatment with ^{14}C-fluazifop-butyl ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0216B, 7 January 1983 (original and supplement) Non-GLP, not published Syngenta File No PP9/0175
PP9/0049	Bell EG, Evans JDHL, Cavell BD	1984	Fluazifop-butyl: Quantification and characterisation of radioactive residues in alfalfa treated with ^{14}C-fluazifop-butyl ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0340B, 29 February 1984 Non-GLP, not published Syngenta File No PP9/0049
PP9/0277	Bewick DW	1982	Fluazifop: Stereochemistry of Residues Derived from the Hydrolysis of Fluazifop-butyl in Soil ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0270B, 30 September 1982 Non-GLP, not published Syngenta File No PP9/0277
PP9/0276	Bewick DW	1983	Fluazifop-butyl: Fate of the Separate R and S-Enantiomers in Soil ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0306B, 30 March 1983 GLP, not published Syngenta File No PP9/0276
-	Bewick DW	1986	Stereochemistry of fluazifop-butyl transformations in soil. Pestic. Sci. 17 (1986) 349-356 Published
PP5/1028	Bill Z, Kamienski VLG	1992	Fusiflex – Residue Analysis Report (Dry Beans) Instituto de Tecnologia do Parana, Brasil Report TECPAR 81975/92, 12 February 1992 Non-GLP, not published Syngenta File No PP5/1028
PP5/0411	Bill Z,	1992	Fluazifop-P-Butyl – Study to determine residues in soya

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
	Kamienski VLG		Instituto de Tecnologia do Parana, Brasil Report TECPAR 81976/92, 28 February 1992 Non-GLP, not published, Syngenta File No PP5/0411
PP5/1027	Bill Z, Kamienski VLG	1992	Fluazifop-P-butyl and Fomesafen – Residue Levels in Soyabeans from Trials in Brazil 1991 Instituto de Tecnologia do Parana, Brasil Report TECPAR 81978/92, 24 March 1992 Non-GLP, not published, Syngenta File No PP5/1027
PP5/1072	Bill Z, Kamienski VLG	1992	Fluazifop-P-butyl + Fomesafen –Study to determine residues in soya Instituto de Tecnologia do Parana, Brasil Report TECPAR 81979/92, 27 February 1992 Non-GLP, not published, Syngenta File No PP5/1072
PP5/0389	Bill Z, Kamienski VLG	1992	FUSILADE – Residue Analysis Report (Dry Beans) Instituto de Tecnologia do Parana, Brasil Report TECPAR 81980/92, 28 February 1992 Non-GLP, not published Syngenta File No PP5/0389
PP5/0390	Bill Z, Kamienski VLG	1992	FUSILADE – Residue Analysis Report (Dry Beans) Instituto de Tecnologia do Parana, Brasil Report TECPAR 81981/92, 27 February 1992 Non-GLP, not published Syngenta File No PP5/0390
PP5/1029	Bill Z, Kamienski VLG	1992	Fusiflex – Residue Analysis Report (Dry Beans) Instituto de Tecnologia do Parana, Brasil Report TECPAR 83030/92, 13 February 1992 Non-GLP, not published Syngenta File No PP5/1029
PP5/0381	Bolygo E	1992	Residues in field beans from trials in the UK during 1988 (EC formulation) Addendum to report M5002B ICI Agrochemicals, Bracknell, Berkshire, UK Report M5002B addendum Study 88JH199, 3 June 1992 Non-GLP, not published Syngenta File No PP5/0381
PP5/0388	Bolygo E	1992	Residues in Field Beans from Trials Carried Out in the UK During 1989 Addendum ICI Agrochemicals, Bracknell, Berkshire, UK Report M5316B addendum, Study 88JH420, 3 June 1992 GLP, not published Syngenta File No PP5/0388 **See also Cullen & Jones, 1991, PP5/0387 (original M5316B report)**
PP5/0817	Bolygo E	1992	Fluazifop-P-butyl: Dissipation in Soil from a Trial Carried Out in Canada During 1990 ICI Agrochemicals, Bracknell, Berkshire, UK, Report RJ1323B, Study 90JH160, 21 December 1992 GLP, not published Syngenta File No: PP5/0817 **Not summarized because no information on the persistent soil metabolite CF3-pyridone was provided**
PP5/0521	Bolygo E	1992	Fluazifop-P-butyl: Residues in Medic Pasture from a Trial Carried out in South Africa During 1990 ICI Agrochemicals, Bracknell, Berkshire, UK, Report RJ1068B, Study 91JH118, 27 July 1992 GLP, not published Syngenta File No PP5/0521
PP5/0425	Bolygo E	1992	Fluazifop-P-butyl: Residues in Melon from a Trial Carried out in South Africa During 1991 ICI Agrochemicals, Bracknell, Berkshire, UK, Report RJ1082B, Study 91JH103, June 1992 GLP, not published Syngenta File No PP5/0425

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Not summarized because no registered use pattern is available
PP5/0422	Bolygo E	1992	Fluazifop-P-butyl: Residues in Cucurbits from a Trial Carried out in South Africa During 1991 ICI Agrochemicals, Bracknell, Berkshire, UK, Report RJ1085B, Study 91JH237A, 28 July 1992 GLP, not published Syngenta File No PP5/0422
PP5/0576	Bolygo E	1992	Fluazifop-P-butyl: Residues in Cotton Seeds from a Trial Carried out in South Africa During 1991 ICI Agrochemicals, Bracknell, Berkshire, UK, Report RJ1131B, Study 91JH102, 2 October 1992 GLP, not published Syngenta File No PP5/0576
PP5/0095	Bolygo E	1993	Fluazifop-P-butyl: Residue Levels in Potatoes from Trials Carried out in Germany During 1992 ICI Agrochemicals, Bracknell, Berkshire, UK Report RJ1405B, Study 92JH120, 4 March 1993 GLP, not published Syngenta File No PP5/0095
PP5/0098	Bolygo E	1993	Fluazifop-P-butyl: Residue Levels in Sugarbeet Foliage and Roots from Trials Carried out in Germany During 1992 ICI Agrochemicals, Bracknell, Berkshire, UK Report RJ1424B, 8 March 1993 GLP, not published Syngenta File No PP5/0098
PP5/0818	Bolygo E	1993	Fluazifop-P-butyl: Dissipation and Build-up in Soils from Trials Carried Out in Italy During 1990-92 ICI Agrochemicals, Bracknell, Berkshire, UK Report RJ1386B, Study 89JH276, 29 June 1993 GLP, not published Syngenta File No PP5/0818
PP5/0819	Bolygo E	1993	Fluazifop-P-butyl: Dissipation and Build-up in Soils from Trials Carried Out in Italy During 1990-1993 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ1512B, Study 89JH276, 24 September 1993 GLP, not published Syngenta File No: PP5/0819
			Not summarized because no information on the persistent soil metabolite CF3-pyridone was provided
PP5/0217	Bolygo E	1994	Fluazifop-P-butyl: Magnitude of Residues in Oilseed Rape Following Spring Application from a Field Study (RS-9304) carried out in Germany During 1993 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ1660B, 13 June 1994 GLP, not published Syngenta File No PP5/0217
PP5/1105	Bolygo E	1994	Fluazifop-P-butyl: Residues in Oilseed Rape and its Processed Products Following Spring Application from a Field Study (RS-9306) and Processing Studies (93 10 47 017 & p 66232503) Carried out in Germany During 1993-94 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ1684B, Study 93JH098, 28 June 1994 GLP, not published Syngenta File No PP5/1105
PP5/0220	Bolygo E	1995	Fluazifop-P-butyl: Magnitude of Residues in Oilseed Rape Following Autumn Application from a Field Study (RS-9305) carried out in Germany During 1993-1994 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ1846B, Study 93JH097, 19 July 1995 GLP, not published Syngenta File No PP5/0220
PP9/0391	Bolygo E	1998	The Determination of Residues of Total fluazifop (fluazifop-P-butyl, fluazifop and conjugate esters) in oily crops and oil Zeneca Agrochemicals, Bracknell, Berkshire, UK SOP RAM 122/04, 11 August 1997

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Non-GLP, not published Syngenta File No PP9/0357
PP5/0067	Bolygo E	1998	Fluazifop-P-butyl: Residue analytical method for total fluazifop analysis in crops An external standard procedure using liquid chromatography with MS-MS or UV detection Zeneca, Bracknell, Berkshire, UK SOP RAM 287/02, 26 June 1998 Non-GLP, not published Syngenta File No PP5/0067
PP9/0357	Bolygo E, Kipps MR	1994	The Determination of Residues of Total fluazifop (fluazifop-P-butyl, fluazifop and conjugate esters) in oily crops and oil Zeneca Agrochemicals, Bracknell, Berkshire, UK SOP RAM 122/02, 20 January 1994 Non-GLP, not published Syngenta File No PP9/0357
PP5/0776	Bolygo E, Brunel D, Jones SD	1991	The determination of fluazifop and reference X in Soil A liquid chromatographic method using external standardisation for fluazifop A gas chromatographic method (GC-MSD) using external standardisation for Reference X (R154719) ICI Agrochemicals, Bracknell, Berkshire, UK Report ARAM 195, 13 June 1991 Non-GLP, not published Syngenta File No PP5/0776 **This document is identical to Atreya 1993, PP5/0776, report RAM 195/01, except that Atreya, 1993, PP5/0776 has an extra front page**
PP5/0196	Bolygo E, Myles P, Dack F	1995	Fluazifop-P-butyl: Residue levels in strawberries from trials carried out in the UK during 1994 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ1817B, Study 94JH050, 6 June 1995 GLP, not published Syngenta File No PP5/0196
PP9/0188	Bolygo E, Sidhu P, Mason R, McGill C	2000	The Determination of Residues of Total Fluazifop (Fluazifop-Butyl, Fluazifop & Conjugate Esters) in Oily Crops & Oil Zeneca Agrochemicals, Bracknell, Berkshire, UK SOP RAM 122/05, 21 September 2000 Non-GLP, not published Syngenta File No PP9/0188
PP5/0235	Bramley YM, Leahey JP, Skidmore MW	2004	Fluazifop-P-butyl: Confined Crop Rotation Syngenta, Bracknell, Berkshire, UK, Report RJ1457B, Study 90JH005, original, 15 June 1994 Report RJ1457B, Study 90JH005, Amendment no 1, 1 December 2004 GLP, not published Syngenta File No PP5/0235
PP5_50066	Brown S	2009	Fluazifop-P Butyl: Independent Laboratory Validation of Syngenta Analytical Method (GRM04401A) for the Determination of Fluazifop-P-Butyl as Fluazifop-P Acid in Crops by LC-MS/MS Morse Laboratories, LLC, Sacramento, CA, USA Report ML09-1552-SYN, Task T009024-08, 10 November 2009 GLP, not published Sungenta File No PP5_50066
PP5/0460	Bunker M, Jones S	1991	Fluazifop-P-butyl: Residues in blackcurrants from trials in the UK during 1989 ICI Agrochemicals, Bracknell, Berkshire, UK Report M5091B, Study 89JH070, 30 April 1991 Non-GLP, not published Syngetna File No PP5/0460
PP5/0474	Bunker M, Jones S	1991	Fluazifop-P-butyl: Residues in gooseberries from trials in the UK during 1989 ICI Agrochemicals, Bracknell, Berkshire, UK Report M5092B, Study 89JH071, 29 April 1991 Non-GLP, not published Syngenna File No PP5/0474
407594	Bussey RJ	1990	Phase 3 summary of MRIDs 40831303, 40831304, 40831305, 40831307 and related MRIDs 152494, 151494, 40361110, 40704805: FUSILADE Residue analytical method Report RR 90-098B, 13 April 1990

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Non-GLP, not published Syngenta File No 407594
407595	Bussey RJ	1990	**Summary report contains some additional validation results on HPLC-UV method PPRAM 62/2 and PPRAM 122 is relevant for this evaluation.** Phase 3 Reformat of MRIDs 40831303, 40831304, 40831305, 40831307 and related MRIDs 152494, 151494, 40361110, 40704805: FUSILADE Residue analytical method Report RR 90-103B, 13 April 1990 Non-GLP, not published Syngenta File No 407595
PP9/0369	Cavell BD, Evans JDHL	1981	**Contains description of PPRAM 83 and is relevant for this evaluation**. Metabolism of fluazifop-butyl in sugar beet roots Origin and report number not stated, 13 August 1981 Non-GLP, not published Syngenta File No PP9/0369
PP9/0194	Cavell BD, Evans JDHL	1985	Fluazifop-butyl: Additional Hydrolysis Studies on Polar Metabolites in Sugarbeet ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0373B, 14 February 1985 GLP, not published Syngenta File PP9/0194
PP9/0200	Cavell BD, Hignett RR, MacNeil RM	1981	Characterisation of the radioactive residue in soya beans at harvest following treatment of immature soya plant with ^{14}C-fluazifop-butyl ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0171B, 6 April 1981 Non-GLP, not published Syngenta File No PP9/0200
PP5/0380	Crook SJ	1988	PP5: Residues in Green Beans, Bean Seeds, Cotton, Cabbage and Onions from Trials in Spain during 1987 ICI Agrochemicals, Bracknell, Berkshire, UK Report M4799B, 10 October 1988 Non-GLP, not published Syngenta File No PP5/0380
PP5/0613	Crook SJ	2000	**Trials on onion tops were not summarized because they did not match cGAP** **Other crops were summarized** Fluazifop-P-butyl Validation of an analytical Method for the Determination of Residues of Total Fluazifop in Bovine Muscle Tissue, Liver, Kidney, Fat, Milk and Hen Eggs Zeneca Agrochemicals, Bracknell, Berkshire, UK Report TMJ4388B, 30 March 2000 Non-GLP, not published Syngenta File No PP5/0613
PP5/0161	Crook SJ, Harradine KJ	1986	Fluazifop-P-butyl Residues determined in peas and pea straw from trials carried out in the Netherlands during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4261B, 19 September 1986 Non-GLP, not published Syngenta File No PP5/0161
PP5/0612	Croucher A	2000	**Green peas with pods and some green pea seeds not summarized because trial could not be matched to the cGAP. Other green pea seeds and dry peas and dry pea straw were summarized**. Fluazifop: Independent Laboratory Validation of an Analytical Method for the Determination of Residues of Total Fluazifop in Animal Tissues Covance Laboratories, Harrogate, North Yorkshire, UK Report 38/263-D2140, Study 38/263, April 2000 Non-GLP, not published Syngenta File No PP5/0612
PP5/0085	Cullen GM	1991	Fluazifop-P-butyl: Residues in carrots from trials carried out in the United Kingdorm during 1989 ICI Agrochemicals, Bracknell, Berkshire, UK ,

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Report M5317B, Study 89JH073, 28 May 1991 GLP, not published Syngenta File No PP5/0085
PP5/0101	Cullen GM	1991	Fluazifop-P-butyl: Residues in swedes from trials carried out in the United Kingdorm during 1989 ICI Agrochemicals, Bracknell, Berkshire, UK Report M5318B, Study 89JH091, 28 May 1991 GLP, not published Syngenta File No PP5/0101
PP5/0195	Cullen GM, Jones SD	1991	Fluazifop-P-butyl: Residues determined in strawberries from trials carried out in in the UK during 1989 ICI Agrochemicals, Bracknell, Berkshire, UK , Report M5319B, Study 89JH069, 23 May 1991 GLP, not published Syngenta Fie No PP5/0195
PP5/0387	Cullen GM, Jones SD	1991	Residues in field beans from trials carried out in the UK during 1989 ICI Agrochemicals, Bracknell Berkshire, UK Report M5316B, Study 88JH420, 28 May 1991 GLP, not published Syngenta File No PP5/0387 **See also Bolygo, 1992, PP5/0388 (addendum to M5316B)**
PP5/0091	Cullen GM, Jones SD	1991	Fluazifop-P-butyl: Residues in Onions from Trials Carried out in the UK During 1989 ICI Agrochemicals, Bracknell Berkshire, UK , Report M5264B, 4 June 1991 GLP, not published Syngenta File No PP5/0091
PP5/0540	Culoto B	1984	RECHERCHE De Residus De : Fluazifop/P/Butyl Dans Les Graines De Tournesol SOPRA (Société Pour la Protection de l'Agriculture), Clamart, France Report H19/834-P, November 1984 GLP, not published Syngenta File No PP5/0540
-	Culoto B, Mallman RJ	1983	Fusilade report apples and pears SOPRA, Bernay Laboratory FB/EM, France Report RIC2815, 25 January, 1983 Non-GLP, unpublished report Syngenta file no. no code **Not summarized because trials could not be matched to the cGAP and method PPRAM 52 was used where fluazifop conjugates are not taken into account**
PP5/0280	Culoto B, Mallmann RJ	1983	Rapport General Fusilade (endive, tomate) SOPRA (Société Pour la Protection de l'Agriculture), Clamart, France RIC2816, 8 March 1983 Non-GLP, not published Syngenta File No PP5/0280
PP9/0050	Culoto B, Mallmann RJ	1984	Recherche comparative des residues sur des carrots après application de la formule commercial Fusilade et d'une formule experimentale fluazifop-P-butyl SOPRA (Société Pour la Protection de l'Agriculture), Clamart, France Report RIC1913, February 1984 Non-GLP, not published Syngenta File No PP9/0050
PP9/0130	Culoto B, Mallmann RJ	1985	Residue analysis of fluazifop-butyl: Test for residues in bananas and limes from trials carried out in Martinique in 1984 SOPRA (Société Pour la Protection de l'Agriculture), Clamart, France Report RIC1933, January 1985 Non-GLP, not published Syngenta File No PP9/0130
PP5/1447	Das R	2006	PP5 Density Syngenta Crop Protection AG, Münchwilen, CH, Study 115848, 22 March 2006 GLP, not published Syngenta File No PP5/1447

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
PP5/1446	Das R	2006	PP5 Color, physical state and odor Syngenta Crop Protection AG, Münchwilen, CH, Study 115847, 8 March 2006 GLP, not published Syngenta File No PP5/1446
PP9/0390	Davy GS	1986	Plant Protection Division Residue Analytical Method No 103 The Determination of Residues of 5-trifluoromethyl-2-pyridone in crops – A nuclear magnetic resonance spectroscopic method ICI Imperial Chemical Industries PLC, Bracknell, Berkshire, UK PPRAM 103, 9 May 1986 Non-GLP, not published Syngenta File No PP9/0390
PP9/0192	Davy GS, Atreya NC	1983	Fluazifop-butyl: R-fluazifop content of residues derived from fluazifop-butyl in crops ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0298B, 28 January 1983 Non-GLP, not published Syngenta File No PP9/0192
PP9/0356	Davy GS, Harradine KJ, Kipps MR	1991	The Determination of Residues of Total Fluazifop (Fluazifop-Butyl, fluazifop and conjugate esters) in crops Zeneca Agrochemicals Report ARAM 197, 6 June 1991 Non-GLP, not published Syngenta File No PP9/0356
PP9/0358	Davy GS, Harradine KJ, Kipps MR, Bolygo E	1994	The Determination of Residues of Total Fluazifop (Fluazifop-Butyl, fluazifop and conjugate esters) in crops Zeneca Agrochemicals, Bracknell, Berkshire, UK SOP RAM 197/02, 21 January 1994 Non-GLP, not published Syngenta File No PP9/0358
PP9/0181	Day SR, Evans JDHL, MacNeil RM, Cavell BD	1981	Quantification and characterization of radioactive residues in eggs and tissues of hens dosed with ^{14}C-fluazifop-butyl ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0212B, 14 August 1981 Non-GLP, not published Syngenta File No PP9/0181
PP9/0047	Day SR, Hignett RR and Cavell BD	1981	Fluazifop-butyl: Characterisation of radioactive residues in oilseed rape after foliar and soil application of 14C-fluazifop-butyl ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0187B, 17 June 1981 Non-GLP, not published Syngenta File No PP9/0047
PP5/0080	Devine HC	1999	Independent Laboratory Validation for Zeneca Agrochemicals of the Method Described in ICI Agricultural Product Report RR 91-014B CEMAS, North Ascot, Berkshire, UK, Report CEMR-1159, Study CEMS-1159, 5 November 1999 Non-GLP, not published Syngenta File No PP5/0080
A12791B_10841	Devine C	2012	Fluazifop-P-Butyl – Residue Study on Apples in Southern France and Italy in 2011 CEMAS, North Ascot, Berkshire, UK, Report CEMR-4968, Study CEMS-4968, Task TK0055807, 20 September 2012, GLP, not published Syngenta File No A12791B_10841
A12791B_11068	Devine C	2013	Fluazifop-P-butyl – Residue Study on Dried Peas (Processing) in Northern France and Germany in 2011 CEMAS, North Ascot, Berkshire, UK, Report CEMR-5037-REG, Study CEMS-5037, Task TK0057345, 26 Sept 2013 GLP, not published Syngenta File No A12791B_11068
PP5/0088	Dick JP	1984	Fluazifop-P-butyl: Residues in Onions from Trials in the UK During 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3872B, 18 December 1984

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			GLP, not published Syngenta File No PP5/0088
PP5/0458	Dick JP	1984	Residues in blackcurrants from trials in the UK during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3870B, 27 November 1984 Non-GLP, not published Syngenta File No PP5/0458
PP5/0473	Dick JP	1984	Fluazifop-P-butyl: Residues in gooseberries from trials in the UK during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3869B, 29 November 1984 Non-GLP, not published Syngenta File No PP5/0473
PP5/0488	Dick JP	1984	Fluazifop-P-butyl: Residues in raspberries from trials in the UK during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3847B, 3 October 1984 Non-GLP, not published Syngenta File No PP5/0488
PP5/0410	Dick JP	1988	Fluazifop-P-Butyl : Residues in Soybeans from trials in Canada during 1987 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4644B, 7 January 1988 GLP, not published, Syngenta File No PP5/0410
			Not summarized because trials could not be matched to the cGAP
PP5/0092	Dick JP, Atreya NC	1984	Fluazifop-P-butyl: Residues in main crop potatoes from trials in the UK during 1983 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3694B, 16 January 1984 GLP, not published Syngenta File No PP5/0092
PP5/0094	Dick J and Rounds LB	1985	Fluazifop-P-butyl: Residues in potatoes from trials in the Netherlands during 1984 ICI Agrochemicals, Bracknell, Berkshire, UK Report M3977B, 7 June 1985 GLP, not published Syngenta File No PP5/0094
PP5/0412	Dick JP, Rounds LB	1985	Residues in Canning Peas from trials in the Netherlands during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3976B, 13 June 1985 Non-GLP, not published Syngenta File No PP5/0412
PP5/0089	Dick JP, Rounds LB	1985	Fluazifop-P-butyl: Residues in Onions from Trials in the Netherlands During 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK, Report M3975B, 13 June 1985 GLP, not published Syngenta File No PP5/0089
PP5/0238	Dick JP, Rounds LB	1985	Fluazifop/P/butyl: Residues in Carrots and Sugar Beet from Trials in the USA During 1983 and 1984 ICI Plant Protection Division, USA Report M4041B, 16 August 1985 GLP, not published Syngenta File No PP5/0238
A12530B_10016	Draetta M	2012	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-butyl em Cebola – Brasil, 2010-11 (onion) Laboratório de Resíduos e Meio Ambiente, São Paulo, Brazil, Report M11026, 12 April 2012 GLP, not published Syngenta File No A12530B_10016
A12530B_10014	Draetta M	2012	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-butyl em Caraços de Algodão – Brasil, 2010-11 (cotton) Laboratório de Resíduos e Meio Ambiente, São Paulo, Brazil,, Report M11027, 28 March 2012 GLP, not published Syngenta File No A12530B_10014
A12530B_10013	Draetta M	2012	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-butyl em Alface –

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Brasil, 2010-11 (lettuce) Laboratório de Resíduos e Meio Ambiente, São Paulo, Brazil, Report M11028, 20 March 2012 GLP, not published Syngenta File No A12530B_10013
A12530B_10012	Draetta M	2012	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-butyl em Cenoura – Brasil, 2010-11 (carrot) Laboratório de Resíduos e Meio Ambiente, São Paulo, Brazil, Report M11030, 13 March 2012 GLP, not published Syngenta File No A12530B_10012
A12530B_10015	Draetta M	2012	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-butyl em Grãos de Soja – Brasil, 2011 (soybean) Laboratório de Resíduos e Meio Ambiente, São Paulo, Brazil, Report M11032, 12 April 2012 GLP, not published Syngenta File No A12530B_10015
A12530B_10020	Draetta M	2012	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-butyl em Tomate – Brasil, 2010-11 (tomato) Laboratório de Resíduos e Meio Ambiente, São Paulo, Brazil, Report M11033, 25 June 2012 GLP, not published Syngenta File No A12530B_10020
A12530B_10018	Draetta M	2012	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-butyl em Grãos de Feijão – Brasil, 2010-11 (beans) Laboratório de Resíduos e Meio Ambiente, São Paulo, Brazil, Report M11034, 04 May 2012 GLP, not published Syngenta File No A12530B_10018
A12530B_10019	Draetta M	2012	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-butyl em Tubérculos de Batata – Brasil, 2010-11 Laboratório de Resíduos e Meio Ambiente, São Paulo, Brazil,, Report M11031, 4 May 2012 GLP, not published Syngenta File No A12530B_10019
A12530B_10011	Draetta M	2012	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-Butyl em Cana-de-Açúcar – Brasil, 2010-11 (sugar cane) Laboratório de Resíduos e Meio Ambiente, São Paulo, Brazil,, Report M11029, 13 February 2012 GLP, not published Syngenta File No A12530B_10011
PP5_10112	Edwards J, Braid S	2010	Fluazifop – Analytical Method for the Determination of Total Fluazifop in Crops Final Determination by LC-MS/MS CEMAS, North Ascot, Berkshire, UK Report GRM044.02A, Task T000934-08, 23 February 2010 Non-GLP, not published Syngenta File No PP5/10112
R154719/0002	Emburey SN	2002	R154719 (Metabolite X of Fluazifop-P-butyl) Laboratory Degradation Study in Four Soil Types Syngenta, Bracknell, Berkshire, UK Report RJ3259B, Study 01JH080, 8 February 2002 GLP, not published Syngenta File No R154719/0002
PP5/0824	Emburey GT, Leahey JP	1994	Fluazifop-P-butyl: Identification of a Product Formed During Aqueous Photolysis Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ1537B, 22 March 1994 GLP, not published Syngenta File No PP5/0824
PP9/0285	Evans JDHL, Cavell BD	1980	PP009: Preliminary Hydrolysis Studies ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0121B, 27 May 1980 Non-GLP, not pulished Syngenta File No PP9/0285
PP9/0043	Evans JDHL,	1984	Fluazifop-butyl: Comparative Metabolism of Separated R and S Enantiomers in

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
	Cavell BD		Lettuce Plants ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0353B, 13 March 1994 GLP, not published Syngenta File No PP9/0043
PP9/0048	Evans JDHL, Cavell BD	1984	Fluazifop-butyl: Comparative Metabolism of Separated R and S Enantiomers in Cotton Plants ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0356B, 24 May 1984 GLP, not published Syngenta File No PP9/0048
PP9/0180	Evans JDHL, Bell EG and Cavell BD	1981	Quantification and characterisation of radioactive residues in milk and tissues of a cow after dosing with 14C-fluazifop-butyl ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0207B, 9 July 1981 Non-GLP, not published Syngenta File No PP9/0180
PP9/0193	Evans JDHL, Hignett RR, Cavell BD	1982	Fluazifop-butyl: Metabolism of ^{14}C-fluazifop-butyl in sugar beet ICI Plant Protection Division Report RJ0221B, Study PP009 AC04, 22 January 1982 Non-GLP, not published Syngenta File PP9/0193
-	FOCUS	2006	Guidance document on estimating persistence and degradation kinetics from environmental fate studies on pesticides in EU registration FOCUS Work Group on Degradation Kinetics, EU Document Reference SANCO/10058/2005, version 20, 434 pp
406311	Francis P	1985	Fluazifop-P-butyl/Fluazifop-butyl: A Comparison of the level of Residues found in Carrots from Trials conducted in 1983 and 1984 ICI Americas Inc, Goldsboro, NC, USA Report TMU1812/B, 5 September 1985 GLP, not published Syngenta File No 406311
434142	Francis PD	1985	Fluazifop-P-Butyl/Fluazifop-Butyl: A Comparison of the Level of Residues Found in Onions from Trials conducted during 1983 and 1984 ICI Americas Inc, Goldsboro, NC, USA, Report TMU1815/B, 25 September 1985 GLP, not published Syngenta File No 434142
PP5/0466	Francis PD	1989	FUSILADE 2000 (Fluazifop-P-Butyl) Magnitude of the Residue Study on Citrus ICI Agricultural Products, Richmond, CA, USA Report RR 89-051B, 14 November 1989 Non-GLP, not published Syngenta File No PP5/0466
PP5/0586	Francis PD	1989	Fusilade 2000 (Fluazifop-P-butyl)-Magnitude of the Residue on Processed Orange Products ICI Americas Inc, Richmond, CA, USA Report RR 89-052B, Lab ID 005-86-07, 11 October 1989 GLP, not published Syngenta File No PP5/0586
407582	Francis P, Kennedy S	1981	Residue data report PP009 – Ref III cotton seed USA 1980 Imperial Chemical Industries Ltd, Bracknell, Berkshire, UK, PP009B042, QA no 568/PP009B042, 10 July 1981 non-GLP, not published Syngenta File No 407582 (NAFTA SD)
			Report present as appendix in Ussary, 1981, 405793, report TMU0680B
PP5/0384	Freeman BL	1990	Fluazifop-P-Butyl: Residues in Field Beans From Trials in the UK During 1988 (EW Formulation) ICI Agrochemicals, Bracknell, Berkshire, UK Report M4994B, June 1990 Non-GLP, not published Syngenta File No PP5/0384
PP5/0382	Freeman BL, Mak C	1989	Fluazifop-P-Butyl: Residues In Field Beans From Trials Carried Out In The UK During 1988 (EC Formulation)

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			ICI Agrochemicals, Bracknell, Berkshire, UK Report M5002B, 1 December 1989 Non-GLP, not published Syngenta File No PP5/0382 **See also Bolygo, 1992, PP5/0381 for an addendum to this report**
PP5/0082	French DA, Leahey JP	1987	Fluazifop-P-butyl: Determination of Radioactive Residues in Grapes from a Vine Treated with 2 Basal Applications of ^{14}C-Fluazifop-P-butyl ICI Plant Protection Division, Bracknell, Berkshire, UK, Report RJ0569B, 22 April 1987 GLP, not published Syngenta File No PP5/0082
PP5/0801	French DA, Matharu KK	1989	Fluazifop-P-butyl: Photodegradation on a Soil Surface ICI Agrochemicals, Bracknell, Berkshire, UK, Report RJ0795B, Study 89JH269, December 1989 GLP, not published Syngenta File No PP5/0801
PP5/0081	French DA, Brown PM, Leahey JP	1987	Fluazifop-P-butyl: Quantification and Characterisation of Radioactive Residues in Celery ICI Plant Protection Division Report RJ0590B, 12 August 1987 GLP, not published Syngenta File No PP5/0081
PP5/0183	Gardyan C	1992	Determination of the Residues of Fluazifop-P-butyl in/on Apples Dr Specht & Partner Chemische Laboratorien, Hamburg, Germany Report AZ84661A/91, Study ICI-9111, 18 December 1992 GLP, not published Syngenta File No PP5/0183
PP5/0192	Gardyan C	1992	Determination of the Residues of Fluazifop-P-butyl in/on Stone-Fruit (Cherries and Plums) Dr Specht & Partner Chemische Laboratorien, Hamburg, Germany Report AZ83558/91, Study ICI-9102, 16 December 1992 GLP, not published Syngenta File No PP5/0192
PP5/0129	Gardyan C	1992	Determination of the Residues of Fluazifop-P-butyl in/on Cabbage (Kale, Cauliflower, Savoy Cabbage and Brussels Sprouts) and Processed Products Dr Specht & Partner Chemische Laboratorien, Hamburg, Germany, Report AZ83592/91, Study ICI-9103, 9 December 1992 GLP, not published Syngenta File No PP5/0129
PP5_10101	Gemrot F	2010	Fluazifop – Independent Laboratory Validation of a Method for the Determination of Total Fluazifop Residues in Crops Eurofins – ADME Bioanalyses, Vergeze, France, Report S10-01917-REG, Study S10-01917, Task TK0001720, 10 June 2010 GLP, not published Syngenta File No PP5/10101
PP5/1549	Geoffroy A	2006	Boiling- and freezing point of PP 5 Solvias AG, Basel, Switzerland, Study L06-001139, 23 May 2006 GLP, not published Syngenta File No PP5/1459
PP5/1458	Geoffroy A	2006	Vapour pressure curve of PP 5 Solvias AG, Basel, Switzerland, Study L06-001140, 23 May 2006 GLP, not published Syngenta File No PP5/1458
R156172_50003	Ghebremichael L, Bang J	2014	Comparability of UK "18 Acres", "Gore Hill" and "Rosedean" Soils Used for Environmental Fate Studies for Fluazifop-Butyl with the Use Area Soils in the United States Syngenta Crop Protection, LLC, USA Report TK0256375, 15 December 2014 Not GLP, not published Syngenta File No R156172_50003
PP5/1357	Gill JP	2003	Residue Study with Fluazifop-P-butyl (PP5) in or on Potatoes in the UK Syngenta, Bracknell, Berkshire UK,

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Report 02-7068, 5 November 2003 GLP, not published Syngenta File No PP5/1357
PP5/1359	Gill JP	2003	Residue Study with Fluazifop-P-butyl in or on Potatoes in the UK Syngenta Bracknell, Berkshire, UK Report 02-7069, 5 November 2003 GLP, not published Syngenta File No PP5/1359
PP5/1355	Gill JP	2003	Residue Study with Fluazifop-P-butyl (PP5) in or on Potatoes in Spain Syngenta, Bracknell, Berkshire, UK, Report 02-7044, 4 November 2003 GLP, not published Syngenta File No PP5/1355
PP5/1353	Gill JP	2003	Residue Study with Fluazifop-P-butyl (PP5) in or on Potatoes in Spain Syngenta, Bracknell, Berkshire, UK Report 02-7045, 27 October 2003 GLP, not published Syngenta File No PP5/1353
PP9/0203	Goddard C, Hignett RR, Cavell BD	1981	Fluazifop-butyl: Metabolism in cotton plants grown under field conditions and quantification and characterisation of residues in mature cotton seeds ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0196B, 3 July 1981 Non-GLP, not published Syngenta File No PP9/0203
PP5/0825	Goodyear A	1995	(^{14}C)-Fluazifop-P: Hydrolysis in Sterile Aqueous Solution Hazleton Europe Ltd, Harrogate, North Yorkshire, UK, Report 38/187-1015, 21 April 1995 GLP, not published Syngenta File No PP5/0825
PP5/0808	Goodyear A	1998	(^{14}C)-Fluazifop-P: Soil Degradation at 20 ºC Covance, Harrogate, North Yorkshire, UK, Report 38/200-D2142, October 1998 GLP, not published Syngenta File No PP5/0808
PP5/10033	Graham R, Gilbert J	2009	[14C] Fluazifop-P-butyl – Route of Degradation under Aerobic Laboratory Conditions, in One Soil, at 20°C Covance Laboratories, Harrogate, North Yorkshire, UK, Report 1983/104-D2149(2), Study 1983/104, Task T001733-08, 23 November 2009 GLP, not published Syngenta File No PP5/10033
R156172_10000	Graham R, Fletcher T, Gilbert J	2013	R156172 Photodegradation of [14C]Fluazifop-P, R156172, in sterile aqueous solution Smithers Viscient (ESG) Ltd, Harrogate, North Yorkshire, UK Covance Laboratories Ltd, Harrogate, North Yorkshire, UK Covance Laboratories Ltd, Alnwick, Northumberland, UK Report 1983/106, Study: 1983/106, Task: T001736-08, 15 May 2013 GLP, not published Syngenta File No R156172_10000
PP5/10019	Greener M	2009	R150397 (Soil metabolite of fluazifop-P-butyl) – Calculation of Kinetic Endpoints from Laboratory Study Data according to FOCUS Kinetics Guidelines Syngenta, Bracknell, Berkshire, UK, Report RAJ0708B, SYPOS no T001740-08, 22 May 2009 Non-GLP, not published Syngenta File No PP5/10019
A12460A_50026	Hampton M, Mazlo J	2013	Fluazifop-P-Butyl (A12460A) – Residues Levels in or on Soybeans, Including Aspirated Grain, from Decline Trials Conducted in the United States during 2010 Syngenta Crop Protection LLC, Greensboro, NC, USA JRF America, Audubon, PA, USA, GLP Technologies, Navasota, TX, USA, Report TK0016832, Study AU-2010-22, Task TK0016832, 20 August 2013 GLP, not published Syngenta File No A12460A_50025 (protocol)

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Syngenta File No A12460A_50026 Syngenta File No A12460A_50027
-	Hagan M, Bertrand N	2013	Fluazifop-P-butyl Dissipation of fluazifop-P-butyl EC (240) in soil under soybean production conditions and agriculture fallow/non-crop land use conditions in the Southeastern United States Analytical Phase Report Amendment #1 ALS Environmental, Edmonton, Alberta, Canada Report 12SYN323 amendment #1, study TK0015266, 18 July 2013 GLP, not published Syngenta File No: no code not available **Present as addendum in Wiepke et al, 2013, A12460A_50023, report TK0015266**
PP5/0593	Hand LH, Robertson TA	1999	Fluazifop-P-butyl: Metabolism in the Goat Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2799B, 29 October 1999 GLP, not published Syngenta File No PP5/0593
PP5/1062	Hargreaves SL	2000	Residue analytical method for the determination of residues of fluazifop-P and R154719 in soil Zeneca Agrochemicals, Bracknell, Berkshire, UK SOP RAM 354/01, 30 October 2000 Non-GLP, not published Syngenta File No PP5/1062
PP5/1061	Hargreaves SL	2001	Residue analytical method for the determination of residues of fluazifop-P and R154719 in soil Zeneca Agrochemicals, Bracknell, Berkshire, UK SOP RAM 354/02, 30 March 2001 Non-GLP, not published Syngenta File No PP5/1061
PP9/0052	Harradine KJ	1984	Fluazifop-butyl and Fluazifop-P-butyl: Residues in Potatoes from Trials in West Germany During 1983 ICI Agrochemicals, Bracknell, Berkshire, UK Report M3676B, 12 January 1984 No GLP, not published Syngenta File No PP9/0052
PP9/0057	Harradine KJ	1984	Fluazifop-butyl: Residues in brassica trials in West Germany during 1983 ICI Agrochemicals, Bracknell, Berkshire, UK Report M3681B, 12 January 1984 GLP, not published Syngenta File No PP9/0057
PP9/0071	Harradine KJ	1984	Fluazifop-P-butyl: Residues in Chicory from trials in Holland during 1983 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3690B, 09 January 1984 Non-GLP, not published Syngenta File No PP9/0071
PP9/0116	Harradine KJ	1984	Fluazifop-butyl/fluazifop-P-butyl: Residues in Peas and Dried Peas From Comparative Trials Carried Out in Canada During 1983 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3754B, 3 April 1984 Non-GLP, not published Syngenta File No PP9/0116 **Dry peas not summarized because trials could not be matched to the cGAP Green peas with pods and green pea seeds were summarized**
PP9/0117	Harradine KJ	1984	Fluazifop-butyl/fluazifop-P-butyl: Residues in Dried Peas and Pea straw From Comparative Trials Carried Out in Holland During 1983 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3759B, 11 April 1984 Non-GLP, not published Syngenta File No PP9/0117
PP9/0119	Harradine KJ	1984	Fluazifop butyl: Residues in Dried Peas from Comparative Trials in the UK During 1983 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3724B, Study PP009B296, 12 March 1984

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Non-GLP, not published Syngenta File No PP9/0119
PP5/0815	Harradine KJ	1984	Fluazifop-P-butyl: Dissipation in Soils from Trials in West Germany during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report No. M3858B, 18 October 1984 GLP, not published Syngenta File No PP5/0815
			Not summarized because study contains no information on CF3-pyridone
PP5/0122	Harradine KJ	1985	Residues in Cucumbers from a trial carried out in Canada during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4097B, 4 November 1985 Non-GLP, not published Syngenta File No PP5/0122
			Not summarized because trials could not be matched to the cGAP
PP5/0421	Harradine KJ	1985	Residues in Cucumbers from a trial carried out in Canada during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4106B, 4 December 1985 Non-GLP, not published Syngenta File No PP5/0421
			Not summarized because trials could not be matched to the cGAP
PP5/0084	Harradine KJ	1985	Fluazifop-P-butyl: Residues in carrots from trials in the UK during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3954B, 11 April 1985 Non-GLP, not published Syngenta File No PP5/0084
PP9/0089	Harradine KJ	1985	Fluazifop-P-butyl: Residues determined in Chicory from trials carried out in the Netherlands during 1983/1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4058B, 28 August 1985 Non-GLP, not published Syngenta File No PP9/0089
PP5/0100	Harradine KJ	1985	Fluazifop-P-butyl: Residues in swedes from trials in the UK during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4001B, 04 June 1985 Non-GLP, not published Syngenta File No PP5/0100
PP5/0397	Harradine KJ	1985	Fluazifop-P-butyl: Residues in vining peas from trials in the UK during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4008B, Study PP005B021, 4 June 1985 Non-GLP, not published Syngenta File No PP5/0397
PP5/0408	Harradine KJ	1985	Fluazifop-P-Butyl : Residues in Soyabeans from trials carried out in Canada during 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4010B, 4 June 1985 GLP, not published, Syngenta File No PP5/0408
PP9/0120	Harradine KJ	1985	Fluazifop-butyl : Residues Determined in Soyabeans From Trials Carried Out in Brazil During 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4140B, 12 December 1985 GLP, not published Syngenta File No PP9/0120
PP5/0407	Harradine KJ	1985	Fluazifop-P-Butyl : Residues determined in Soyabeans carried out in Brazil during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4141B, 12 December 1985 GLP, not published, Syngenta File No PP5/0407
PP5/0273	Harradine, KJ	1985	Fluazifop-P-butyl: Residues determined in Rutabaga from trials carried out in Canada during 1984

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4052B, 29 August 1985 Non-GLP, not published Syngenta File No PP5/0273
PP5/0272	Harradine KJ	1986	Fluazifop-P-butyl: Residues determined in Swedes from trials in the UK during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4204B, 17 April 1986 Non-GLP, not published Syngenta File No PP5/0272
PP5/0376	Harradine KJ	1986	Fluazifop-P-butyl : Residues in Common Dry Beans From Trials Carried Out in Canada During 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4130B, 6 January 1986 Non-GLP, not published Syngenta File No PP5/0376
PP5/0398	Harradine KJ	1986	Fluazifop-P-butyl : Residues in Vining Peas and Dried Peas From Trials in the UK During 1983/84, treated at the label recommendation ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4209B, 17 April 1986 Non-GLP, not published Syngenta File No PP5/0398 **Dry peas not summarized because the trials could not be matched to the GAP.** **Green pea seeds and green pea forage were summarized**
PP5/0472	Harradine KJ	1986	Fluazifop-P-butyl: Residues in gooseberries from trials in the UK during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK, Report M4186B, 8 April 1986 Non-GLP, not published Syngenta File No PP5/0472
-	Harradine KJ, Atreya NC	1984	The detection of residues of total fluazifop (fluazifop-butyl, fluazifop and conjugate esters) in crops – A nuclear magnetic resonance spectroscopic method ICI Plant Protection Division, Bracknell, Berkshire, UK Report PPRAM 83, 16 January 1984 Non-GLP, not published Syngenta File No: not available **Submitted as appendix in Bussey, 1990, 407595**
PP5/0087	Harradine KJ, Crook SJ	1986	Fluazifop-P-butyl: Residues in leeks from trials carried out in the Netherlands during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4217B 11 June 1986 Non-GLP, not published Syngenta File No PP5/0087
PP5/0090	Harradine KJ, Crook SJ	1986	Fluazifop-P-butyl: Residues in Onions from Trials in the Netherlands During 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4205B, 6 June 1986 GLP, not published Syngenta File No PP5/0090
PP5/0087	Harradine K, Crook SJ	1986	Fluazifop-P-butyl: Residues leeks from trials carried out in the Netherlands during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4217B, 11 June 1986 Non-GLP, not published Syngenta File No PP5/0087
PP5/0457	Harradine K, Pay J	1986	Fluazifop-P-butyl: Residues in blackcurrants from trials in the UK during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4197B, Study PP005/B060, 17 April 1986 Non-GLP, not published Syngenta File No PP5/0457
PP9/0274	Harvey BR, Hill IR	1983	Fluazifop-butyl: Extraction and Fractionation of Bound Residues in Soil (Addendum to RJ0197B)

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			ICI Plant Protection Division, Bracknell, Berkshire, UK, Report RJ0336B, 14 November 1983 Non-GLP, not published Syngenta File No PP9/0274
PP9/0737	Harvey B, Mistry R, Hill I	1981	Fluazifop-butyl: Further Work Regarding Acetone/HCl Extractable Material from Soil (Addendum to RJ0197B) ICI Plant Protection Division, Bracknell, Berkshire, UK, No report number, no date available Non-GLP, not published Syngenta File No PP9/0737
PP9/0273	Harvey BR, Vincent J, Mistry R, Arnold DJ	1981	Fluazifop-butyl: Degradation in Soil ICI Plant Protection Division, Bracknell, Berkshire, UK, Report RJ0197B, 24 August 1981 Non-GLP, not published Syngenta File No PP9/0273 **See also the two addenda to this study (Harvey et al, 1981, PP9/0737 and Harvey & Hill, 1983, PP9/0274)**
PP5/0251	Hayward G	1987	Fluazifop-P-butyl and Reference X: Residues in Onions from Trials Carried in the USA during 1986 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4545B, Study PP005B121, 27 July 1987 GLP, not published Syngetna File No PP5/0251
462746	Hayward GJ	1988	Fluazifop-P-butyl (reference X): Storage Stability of Residues in Deep Frozen peanut kernel samples ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4841B, Study PP009BC15, 9 November 1988 GLP, not published Syngenta File No 462746
PP5/0076	Hayward GJ	1988	Fluazifop-P-butyl (reference X): Storage Stability of Residues in Deep Frozen apples, lettuce and soyabean samples ICI Agrochemicals, Bracknell, Berkshire, UK Report M4842B, Study PP009BC15, 9 November 1988 GLP, not published Syngenta File No PP5/0076
PP5/0077	Hayward GJ	1988	Fluazifop-P-butyl and Reference X: Storage Stability of Residues in Deep Frozen Onion Samples ICI Agrochemicals, Bracknell, Berkshire, UK Report M4843B, 9 November 1988 GLP, not published Syngenta File No PP5/0077
462671	Hayward GJ, Atreya NC	1987	The Determination of Residues of Total Fluazifop (Fluazifop-Butyl, fluazifop and conjugate esters) in oily crops ICI Industries PLC, Bracknell, Berkshire, UK PPRAM 122, 15 October 1987 Non-GLP, not published Syngenta File No: 462671 **Submitted as appendix in Davy et al, 1991, PP9/0356**
PP5/0519	Hayward GJ, Harradine KJ	1989	Residues in Fodder Beet from Trials in Denmark during 1988 ICI Agrochemicals, Bracknell, Berkshire, UK Report M4870B, January 1989 Non-GLP, not published Syngenta File No PP5/0519
PP9/0197	Hignett RR, Cavell BD	1979	Translocation and Metabolism of ^{14}C-phenyl Labelled PP009 in Soya and Maize Following Injection ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0101B, 28 November 1979 Non-GLP, not published Syngenta File No PP9/0197
PP9/0199	Hignett RR, Godddard C, Evans JDHL, Cavell BD	1979	The Uptake and Degradation of ^{14}C-PP009 following its foliar application to soya and maize plants ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0085C, 9 November 1979

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Non-GLP, not published Syngenta File No PP9/0199
PP9/0198	Hignett RR, MacNeil RM, Goddard C, Cavell BD	1980	Metabolism of ^{14}C-PP009 in soya plants grown under field conditions and quantification of the radioactive residue ni the harvested bean Interim Report ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0134B, 16 June 1980 Non-GLP, not published Syngenta File No PP9/0198
			This is an interim report; the final report was not submitted
PP5_50103	Huang S	2010	Analytical Method for the Determination of Fluazifop-P-Butyl (R154875, PP5), Fluazifop-P Acid (R156172) and Compound X (R154719, CGA142110) in Soil Using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS) - Analytical method amendment Syngenta Crop Protection, LLC, Greensboro, NC, USA Report GRM044.03A, Task TK0019659, 27 November 2010 Not GLP, not published Syngenta File No PP5_50103
R150397_50000	Huang S	2012	Fluazifop-P-butyl - Soil Extraction Efficiency Evaluation for the Fluazifop-P-butyl Degradation Product, Compound IV (R150397/CGA181847), During Method Development – Method Validation Syngenta Crop Protection, LLC, Greensboro, NC, USA Report TK0172993, Task TK0172993, 4 December 2012 Not GLP, not published Syngenta File R150397_50000
PP9/0042	Hughes P, Evans JDHL, Cavell BD	1985	Fluazifop-butyl: Comparative Metabolism of Fluazifop-butyl (PP009) and Fluazifop-P-butyl (PP005) in Carrots ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0418B, 26 April 1985 GLP, not published Syngenta File No PP9/0042
PP9/0040	Hughes P, Evans JDHL, Leahy JP, Cavell BD	1986	Fluazifop-butyl: Comparative Metabolism of Fluazifop-butyl (PP009) and Fluazifop-P-butyl (PP005) in Sugar Beets ICI Plant Protectin Division, Bracknell, Berkshire, UK Report RJ0490B, 6 November 1986 GLP, not published Syngenta File No PP9/0040
-	IR-4	1984	Results of Tests on the Amount of Fluazifop-butyl Residues Remaining in or on Cucumbers Including a Description of the Analytical Method Used Location Delmar, Delaware, USA IR-4 PR 1878 (DE) Non-GLP, not published Syngenta File no no code
			Not summarized because trials could not be matched to the cGAP
-	IR-4	1984	Results of Tests on the Amount of Fluazifop-P-butyl Residues Remaining in or on Cucumbers Including a Description of the Analytical Method Used Location North Carolina, USA IR-4 PR 1878 (NC) Syngenta File no no code
			Description of the same trial as in Baron, 1986, IR-4 PR1878
PP5/0882	Jessop K, Embury G, Leahey J	1991	Fluazifop-P-butyl: Photodegradation in Aqueous Solution at pH 5 ICI Agrochemicals, Bracknell, Berkshire, UK, Report RJ0992B, 16 August 1991 GLP, not published Syngenta File No PP5/0822
PP5/1031	Johnson TD, Aver E, Bolygo E, French DA	1993	Fomesafen/Fluazifop-P-butyl – Residues in Soya bean from a Trial Carried Out in South Africa During 1991 ICI Agrochemicals, Bracknell, Berkshire, UK Report TMJ3065B, Study 91JH131, 4 March 1993 GLP, not published, Syngenta File No PP5/1031

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Residues in forage were summarized. Residues in dry haulms and dry seed were not summarized because trials could not be matched to the GAP
PP5_50554	Jolly C	2014	Fluazifop-P-Butyl: Magnitude of the Residue on Fine Fescue Grasses (Seed Crop) Rutgers State University of New Jersey of New Jersey, Princeton, NJ, USA Report IR-4 PR 09825, 0982510-FLR18, MRID 49460507, 29 August 2014 GLP, not published Syngenta File No PP5_50554
PP5/0150	Jones SD	1991	Fluazifop-P-butyl: Residues in peas from a trial carried out in Denmark during 1989 ICI Agrochemicals, Bracknell, Berkshire, UK Report M5347B, Study 89JH185, 4 June 1991 Non-GLP, not published Syngenta File No PP5/0150
			Trials on green peas with pods not summarized because trials could not be matched to the cGAP. Dry peas, green pea forage and dry pea straw were summarized.
-	Jones SD	1991	Addendum to PPRAM 54 ICI Agrochemicals, Bracknell, Berkshire, UK Report PPRAM 54 addendum, June 1991 Non-GLP, not published Syngenta File No not available
			Submitted as addendum to Atreya & Houlden, 1980, PPRAM 54
PP5/0193	Jones SD	1991	Fluazifop-P-butyl: Residues in raspberries from trials in the UK during 1989 ICI Agrochemicals, Bracknell, Berkshire, UK Report M5320B, Study 89JH072, 2 May 1991 Non-GLP, not published Syngenta File No PP5/0193
PP5/0386	Jones SD	1991	Fluazifop-P-butyl: Residues in Dry beans from Trials in Canada During 1990 ICI Agrochemicals, Bracknell, Berkshire, UK Report M5386B, Study 90JH204, 21 October 1991 Non-GLP, not published Syngenta File No PP5/0386
			Not summarized because trials could not be matched to the cGAP
PP5/0099	Jones SD	1992	Fluazifop-P-butyl: Residues in Turnips from Trials in the UK During 1990 ICI Agrochemicals, Bracknell Berkshire, UK Report RJ0997B, 21 January 1992 Non-GLP, not published Syngenta File No PP5/0099
PP5/0405	Jones SD	1992	Fluazifop-P-Butyl: Residues in Field Peas from trials in Canada during 1990 ICI Agrochemicals, Bracknell, Berkshire, UK Report RJ1059B, Study 90JH159, 1 April 1992 GLP, not published Syngenta File No PP5/0405
			Dry peas not summarized because trials could not be matched to cGAP. Green peas with pods and gree pea forage were summarized.
PP5/1291	Jones RN	2003	Fluazifop acid: Determination of half-life and DT50 values for laboratory and field dissipation studies using ModelManager (version 11) Syngenta, Bracknell, Berkshire, UK Report RAJ0161B, 8 October 2003 GLP, not published Syngenta File No: PP5/1291
			Not summarized because no kinetic trigger endpoints were derived for the persistent soil metabolite CF3-pyridone
PP5/0814	Jones SD, Atreya NC	1991	Fluazifop-P-butyl: Dissipation in West German Soils Following Autumn or Spring Applications ICI Agrochemicals, Bracknell, Berkshire, UK, Report RJ0952B, Study 88JH384, 8 April 1991 GLP, not published Syngenta File No: PP5/0814

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
PP5/1024	Jones SD, Bonfanti F	1998	Fluazifop-P-butyl – Residue Levels in Soyabeans Trials Carried Out in Italy During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2442B, Study 96JH177, Task TK0219472, 6 January 1998 GLP, not published, Syngenta File No PP5/1024 Syngenta File No PP5_50431
PP5/0223	Jones SD, Hughes A	1999	Fluazifop-P-butyl: Residue Levels in Hazelnuts from Trials carried out in the UK during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2656B, 23 February 1999 GLP, not published Syngenta File No PP5/0223
PP5/0113	Jones SD, Kenny D	1999	Fluazifop-P-butyl: Residue Levels in Asparagus from Trials carried out in France during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2701B, Study 97JH112, 22 february 1999 GLP, not published Syngenta File No PP5/0113
PP5/0295	Jones SD McGill C	1999	PP5: Residue Levels in Onions from Trials carried out in Spain & Italy during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2728B, 23 February 1999 GLP, not published Syngenta File No PP5/0295
PP5/0168	Jones SD, Volpi E	1998	Fluazifop-P-butyl: Residue Levels in Cucurbits from Trials Carried out in Italy During 1996 Zeneca, Agrochemicals, Bracknell, Berkshire, UK, Report RJ2265B, 24 June 1997 GLP, not published Syngenta File No PP5/0168
PP5/0221	Jones SD, Volpi E	1998	Fluazifop-P-butyl : Residue Levels in Sunflower from Trials carried out in Italy During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2284B, Study 96JH077, 17 November 1997 GLP, not published Syngenta File No PP5/0221
PP5/0152	Jones SD, Bouwman JJ, Ryan J	1997	Fluazifop-P-butyl: Residue levels in beans harvested green with edible pods from trials carried out in the Netherlands during 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2287B Study 96JH053, 01 September 1997 GLP, not published Syngenta File No PP5/0152
PP5/0171	Jones SD, Cowley P, Ryan J	1997	Fluazifop-P-butyl: Residue Levels in Cucurbits from a Trial Carried out in Spain During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2380B, 17 November 1997 GLP, not published Syngenta File No PP5/0171
PP5/0169	Jones SD, Gallardo E, Ryan J	1997	Fluazifop-P-butyl: Residue Levels in Tomatoes from Trials Carried out in Spain During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2268B, 8 July 1997 GLP, not published Syngenta File No PP5/0169
PP5/0135	Jones SD, Griehl T, Ryan J	1997	Fluazifop-P-butyl: Residue Levels in Savoy Cabbage from Harvest Trials carried out in Germany During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2306B, 19 June 1997 GLP, not published Syngenta File No PP5/0135
PP5/0151	Jones SD, Griehl T, Ryan J	1997	Fluazifop-P-butyl: Residue levels in French beans from trials carried out in Germany during 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2290B, 19 June 1997

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			GLP, no published Syngenta File No PP5/0151
PP5/0138	Jones SD, Renard C, Ryan J	1997	Fluazifop-P-butyl: Residue Levels in Lettuce from Trials carried out in France during 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2302B, Study 96JH052, 08 July 1997 GLP, not published Syngenta File No PP5/0138
PP5/0162	Jones SD, Volpi E, Elliott R	1997	Fluazifop-P-butyl: Residue Levels in Fresh Peas from Trials carried out in Italy during 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2254B, Study 96JH075, 06 May 1997 GLP, not published Syngenta File No PP5/0162
PP5/0164	Jones SD, Volpi E, Ryan J	1997	Fluazifop-P-Butyl – Residue Levels in Soya Bean from Trials Carried Out in Italy During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2405B, Study 96JH076, 20 November 1997 GLP, not published, Syngenta File No PP5/0164 Syngenta File No PP5/0419 Syngenta File No PP5_50430
PP5/0110	Jones SD, Gallardo E, Ryan J	1998	Fluazifop-P-butyl: Residue Levels in Asparagus from Trials Carried out in Spain during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2673B, 19 October 1998 GLP, not published Syngenta File No PP5/0110
PP5/0198	Jones SD, Renard C, Ryan J	1998	Fluazifop-P-butyl: Residue Levels in Apples from Trials Carried out in France During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2319B, 20 January 1998 GLP, not published Syngenta File No PP5/0198
PP5/1026	Jones SD, Volpi E, McGill CD	1998	Fomesafen and Fluazifop-P-butyl – Residue Levels in Soyabeans from Trials Carried Out in Italy During 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2720B, Study 97JH208, Task TK0219484, 21 October 1998 Report RJ2720B, Study 97JH208, 2 November 1998 (different layout) GLP, not published, Syngenta File No PP5/1026 (21 Oct 1998, 42 pages) Syngenta File No PP5_50433 (2 Nov 1998, 43 pages)
PP5/0140	Jones SD, Myles P, Mason R	1999	Fluazifop-P-butyl: Residue levels in kale from trials carried out in the UK during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2654B Study 97JH115, 01 March 1999 GLP, not published Syngenta File No PP5/0140
PP5/0175	Jones SD, Volpi E, Gallardo E, Kenny D	1999	Fluazifop-P-butyl: Residue Levels in Tomato from Trials carried out in Italy & Spain during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2657B, 13 April 1999 GLP, not published Syngenta File No PP5/0175
A12791B_10829	Jutsum L	2011	Fluazifop-P-Butyl – Residue Study on Field Beans with Pods in France (South) and Spain, in 2009 CEMAS, North Ascot, UK Report CEMR-4384-REG, Study CEMS-4384, Task T000906-09, 3 February 2011 GLP, not published Syngenta File No A12791B_10829
A12791B_10830	Jutsum L	2011	Fluazifop-P-Butyl – Residue Study on Dried Peas in France (South) and Spain, in 2008 CEMAS, North Ascot, UK, Report T009247-07-REG, Study CEMS-3911, Task T009237-07, 3 February

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			2011 GLP, not published Syngenta File No A12791B_10830
A12791B_ 10831	Jutsum L	2011	Fluazifop-P-Butyl (A12791B) – Residue Study on Field Peas (Dried) in France (South) and Spain, in 2009 CEMAS, North Ascot, UK, Report CEMR-4385-REG, Study CEMS-4385, Task T000907-09, 8 February 2011 GLP, not published Syngenta File No A12791B_10831
A1279B_10837	Jutsum L, Allen L	2011	Fluazifop-P-Butyl – Residue Study on Peas without Pods in Germany and the UK in 2010 CEMAS, North Ascot, Berkshire, UK, Report CEMR-4658-REG, Study CEMS-4658, Task TK0024934, 14 Sept 2011 GLP, not published Syngenta File No A12791B_10837
PP5/1440	Kang J	2005	Fluazifop-P-butyl (PP5): Residue Study in or on Potato in France (South) CEMAS, North Ascot, Berkshire, UK, Report CEMR-2309, Study CEMS-2309, 12 July 2005 GLP, not published Syngenta File No PP5/1440
PP5/1441	Kang J	2005	Fluazifop-P-butyl (PP5): Residue Study in or on Sugar Beet in Italy and France (South) CEMAS, North Ascot, Berkshire, UK, Report CEMR-2310, Study CEMS-2310, 18 July 2005 GLP, not published Syngenta File No PP5/1441
PP5/1438	Kang J	2005	Fluazifop-P-butyl (PP5): Residue Study in or on strawberry in Italy and France (South) CEMAS, North Ascot, Berkshire, UK, Report CEMR-2306, Study CEMS-2306, 14 May 2005 GLP, not published Syngenta File No PP5/1438
A12791B_11992	Kennedy S	2014	Fluazifop-P-butyl – Residue Study on Strawberries in Italy and Spain in 2013 CEMAS, North Ascot, UK, Report CEMR-6043, Study CEMS-6043, Task TK0178538, 19 June 2014 GLP, not published Syngenta File No A12791B_11992
-	KINGUI	2006	User interface for kinetic evaluations, version 11 Bayer Technical Services, Bayer CropScience
405683	Kleinschmidt M, Miller M	2000	Fluazifop-P-Butyl - Residue Levels in Banana from Trials Carried Out in the United States during 1999 Zeneca Ag Products, Inc Richmond, CA, USA Report RR 00-043B, TK0015161, 20 Sept 2000 GLP, not published Syngenta File No 405683
			This report has the same content as Miller, 2000, PP5/0454
406227	Koubek KG	1982	Fluazifop Residues in Soybeans ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU0922/B, 12 October 1982 GLP, not published, Syngenta File No 406227
			See also Koubek, 1983, 432235, TMU1172/B for additional information **Not summarized because trials could not be matched to the cGAP**
406305	Koubek KG	1982	Fluazifop Residues in Carrots ICI Americas Inc, Agricultural Chemicals Division, North Carolina, USA Report TMU0902/B, 17 August, 1982 GLP, not published Syngenta File No 406305
			Not summarized because trials could not be matched to the cGAP
405794	Koubek KG	1982	Fluazifop Residues in Cottonseed

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			ICI America Inc, Greensboro, NC, USA Report TMU0987/B, 26 October 1982 Non-GLP, not published Syngent File No 405794
405795	Koubek KG	1982	Fluazifop Residues in Cottonseed ICI America Inc, Greensboro, NC, USA Report TMU1027/B, 30 December 1982 Non-GLP, not published Syngent File No 405795
406228	Koubek KG	1982	Fusilade Resideus in Soybean Fractions ICI Americas Inc, Richmond, CA, USA Report TMU0975/B, 15 October 1982 Non-GLP, not published Syngenta File 406228
405726	Koubek KG	1983	Fluzifop Residues in Sugar Beets ICI Americas Inc, Agricultural Chemicals Division, NC, USA Report TMU1211/B, 16 September 1983 GLP, not published Syngenta File No 405726
406276	Koubek KG	1983	Fluazifop Residues in Soybeans ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU1037/B, 17 January 1983 GLP, not published, Syngenta File No 406276
			See also Koubek, 1983, 432235, TMU1172/B for additional information **Not summarized because trials could not be matched to the cGAP**
406307	Koubek KG	1983	Fluazifop Residues in Carrots ICI Americas Inc, Goldsboro, NC, USA Report TMU1182/B, 1 July 1983 GLP, not published Syngenta File No 406307
			Not summarized because trials could not be matched to the cGAP
406309	Koubek KG	1983	Fluzifop Residues in Carrots ICI Americas Inc, Agricultural Chemicals Division, North Carolina, USA Report TMU1231/B, 24 October 1983 GLP, not published Syngenta File No 406309
432235	Koubek KG	1983	Fluazifop Residues in Soybeans after Aerial Application ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU1172/B, 29 April 1983 GLP, not published Syngenta File No 432235
			See also Koubek, 1982, 406227, TMU0922/B for additional information **See also Koubek, 1983, 406276, TMU1037/B for additional information** **Not summarized because trials could not be matched to the cGAP**
406215	Koubek KG	1983	Fluzifop Residues in Bulb Onions ICI Americas Inc, Agricultural Chemicals Division, NC, USA Report TMU1257/B, 4 January 1984 GLP, not published Syngenta File No 406215
405796	Koubek KG	1984	Fluazifop Residues in Cotton ICI America Inc, Greensboro, NC, USA Report TMU1401/B, 8 March 1984 Non-GLP, not published Syngent File No 405796
PP5/0406	Koubek KG	1984	Fluazifop Residue in Soybeans ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU1403/B, 9 March 1984 GLP, not published, Syngenta File No PP5/0406 Syngenta File No 405716

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Not summarized because trials could not be matched to the cGAP
PP5/0608	Kukla D	1991	Determination of 5-(Trifluoromethyl)-2(1H)-pyridinone (R-154719) residues in sugar beets and sugar beet processed commodities by gas chromatography ICI Americas Inc, Richmond, CA, USA Report RR 90-384B, 25 July 1999 GLP, not published Syngenta File No PP5/0608
-	Kwiatkowski AS, Crook SJ	2009	Residue analytical method for the determination of total fluazifop in soya milk and soya-based infant formula using LC-MS/MS Syngenta, Bracknell, Berkshire, UK SOP RAM 336/01, 20 March 2009 GLP, not published Syngenta File No: not available
			Available as appendix in Mason, 2009, PP5/10004
A12791B_11035	Langridge G	2013	Fluazifop-P-butyl – Residue Study on Peas Without Pods In Southern France, Italy and Spain in 2012 CEMAS, North Ascot, Berkshire, UK, Report CEMR-5453, Study CEMS-5453, Task TK0112059, 9 August 2013 GLP, not published Syngenta File No A12791B_11035
A12791A_10077	Langridge G	2013	Fluazifop-P-butyl – Residue Study on Strawberries in Southern France, Italy and Spain in 2012 CEMAS, North Ascot, Berkshire, UK Report CEMR-5448, Study CEMS-5448, Task TK0112057, 11 June 2013 GLP, not published Syngenta File No A12791A_10077
A12791B_11028	Langridge G	2013	Residue Study on Head Lettuce in Southern France, Italy and Spain in 2012 CEMAS, North Ascot, UK, Report CEMR-5451, Study CEMS-5451, Task TK0112077, 31 July 2013, GLP, not published Syngenta File No A12791B_11028
A12791B_11249	Langridge G	2013	Fluazifop-P-butyl: Residue Study on Oilseed Rape in Southern France, Italy and Spain in 2012 CEMAS, North Ascot, Berkshire, UK, Report CEMR-5449, Study CEMS-5449, Task TK0112058, 20 November 2013 GLP, not published Syngenta File No A12791B_11249
A12791B_11029	Langridge G	2013	Fluazifop-P-butyl – Residue Study on Peas without Pods (Processing) in Germany and the UK in 2010 CEMAS, North Ascot, Berkshire, UK, Report CEMR-4751-REG, Study CEMS-4751, Task TK0024933, 31 July 2013 GLP, not published Syngenta File No A12791B_11029
-	Leahey J	1990	Fluazifop-butyl : Degradation in Soil : A comparison of the Microflora and Physicochemical Properties of Soils Used in UK Laboratory Studies with those of USA Soils ICI Agrochemicals, Bracknell, Berkshire, UK Report M5148B, June 1990 Not GLP, not published Syngenta File No not available
463828	Leahey JP, French DA	1991	Fluazifop-butyl (PP009): Extractability and Hydrolysis of Radioactive Residues in Soybeans ICI Agrochemicals, Bracknell, Berkshire, UK Report M4394B, Study PP009AC16, 22 August 1991 (cover page 15 June 1987) GLP, not published Syngenta File No 463828
PP5_50001	Lin K	2009	Fluazifop-P-butyl: Radiovalidation of Analytical Method GRM04401A for Determination of Fluazifop-P-Butyl as Fluazifop-P Acid in Crops by LC-MS/MS Syngenta Crop Protection Inc, Greensboro, NC, USA Report T002223-07, Task T002223-07, 27 January 2009

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			GLP, not published Syngenta File No: PP5_50001 (core) Syngenta File No: PP5_50006 (submission)
PP005_50017	Lin K	2009	Fluazifop-P-Butyl: Radiovalidation of ICI Plant Protection Division Residue Analytical Method No 62/2 for the Determination of Residues of Total Fluazifop (Fluazifop-Butyl, Fluazifop and Conjugate Esters) in Crops Syngenta Crop Protection Inc, Greensboro, NC, USA Report T009022-08, Task T009022-08, 30 January 2009 GLP, not published Syngenta File No: PP005_50017
A12530D_10013	Lopes KC	2013	Fusilade 250 EW – Magnitude de Resíduos de Fluazifop-P-Butyl em Repolho – Brasil, 2012 (cabbage) Laboratório de Resíduos e Meio Ambiente, Syngenta Proteção de Cultivos Ltda, São Paulo – SP – Brasil Report M12060, 7 February 2013 GLP, not published Syngenta File No: A12530D_10013
PP9/0202	MacNeil RM, Cavell BD	1984	Fluazifop-butyl: - Characterisation of the Radioactive Residue in Soya Beans following Foliar Appliction of ^{14}C-Fluazifop-butyl at two growth stages ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0342B, 7 June 1984 Non-GLP, not published Syngenta File No PP9/0202
PP9/0046	MacNeil RM, Cavell BD	1985	Fluazifop-butyl: - Characterisation of the radioactive residue in soya beans at harvest 63 days after treatment of soya plants with ^{14}C-fluazifop-butyl ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0374B, 24 September 1985 Non-GLP, not published Syngenta File No PP9/0046
PP9/0287	MacNeil RM, Hignett RR, Cavell BD	1981	Fluazifop-butyl: Photolysis of 14C-fluazifop-butyl in sterile aqueous solutions ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0176B, 12 June 1981 (22 pages) Non-GLP, not published Syngenta File Code No PP9/0287
PP9/0045	MacNeil RM, Hignett RR, Cavell BD	1981	Fluazifop-butyl: - Characterisation of radioactive soya bean residues arising from field applications of ^{14}C-fluazifop-butyl ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0211B, 24 July 1981 Non-GLP, not published Syngenta File No PP9/0045
PP9/0201	MacNeil RM, Hignett RR, Cavell BD	1981	Fluazifop-butyl: - Characterisation of non-polar radioactive residues in soya beans arising from field applications of ^{14}C-fluazifop-butyl to immature plants ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0213B, 14 August 1981 Non-GLP, not published Syngenta File No PP9/0201
PP9/0278	MacNeil RM, Hignett RR, Cavell BD	1981	Fluazifop-butyl : Photodegradation of $^{(14)}$C-Fluazifop-butyl on a Soil Surface ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0191B, 11 June 1981 GLP, not published Syngenta File No PP9/0278
-	Mak C, Atreya NC	1987	Addendum to PPRAM 122 ICI Industries PLC, Bracknell, Berkshire, UK Addendum to PPRAM 122, 21 December 1987 Non-GLP, not published Syngenta File No: not available
			Submitted as appendix in Davy et al, 1991, PP9/0356
PP5/0462	Mak C, Scott MH	1988	Fluazifop-P-butyl: Residues Determined in Blackberries, Bilberries and Raspberries from Trials in West Germany During 1987 ICI Agrochemicals, Bracknell, Berkshire, UK Report M4779B, 19 September 1988 Non-GLP, not published Syngenta File No PP5/0462
PP9/0286	Makin NGS,	1980	PP009: Hydrolysis of $^{(14)}$C-PP009 in Sterile Aqueous Solution

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
	Hignett RR, Cavell BD		ICI Plant Protection Divsion, Bracknell, Berkshire, UK Report RJ0145B, 3 September 1980 Non-GLP, not published Syngenta File No PP9/0286
PP5/0184	Markus K, Nguy T	1986	Residue data sheet Field information and Analytical Results Fluazifop-P-butyl Bananas ICI Australia Operations, Merindale, Australia Report RIC1934, 3 September 1986 Non-GLP, not published Syngenta File No PP5/0184
PP5_10084	Marshall L	2010	Fluazifop – Validation of a Method for the Determination of Total Fluazifop Residues in Crops CEMAS, North Ascot, Berkshire, UK, Report CEMR-4218-REG, Study CEMS-4218, Task T000934-08, 19 February 2010 GLP, not published Syngenta File No PP5/10084
A1279B_10788	Marshall, L	2009	Fluazifop-P-butyl: Residue Study on Beans with Pods in France (South) and Spain in 2008 ICI Agrochemicals, Bracknell, Berkshire, UK Report T009248-07-REG, Study CEMS-4008, 14 October 2009 GLP, not published Syngenta File No A1279B_10788
PP5/1449	Martin N	2006	Dissociation constant of PP 5 in water Solvias AG, Basel, CH, Study L06-001141, 12 May 2006 GLP, not published Syngenta File No PP5/1449
-	Maslowski K	2012	Determinaçao de resíduos de fluazifop total em amostras vegetais por LC/MS/MS Syngenta Proteçao de Cultivos Uda, Sao Paulo, Brazil Report POPIT.MET.138.Rev.02, 18 January 2012 Non-GLP, not published Syngenta File No, not available (confirmed)
PP5/0116	Mason R	1999	Fluazifop-P-butyl: Residue Levels in Celeriac from Trials carried out in Northern France during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2630B, 05 March 1999 GLP, not published Syngenta File No PP5/0116
PP5/0356	Mason R	2000	Fluazifop-P-Butyl: Residue Levels in Cabbage from a Trial carried out in Northern France during 1999 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2992B, 27 October 2000 GLP, not published Syngenta File No PP5/0356
PP5/0005	Mason R	2001	Residue Levels in Sunflowers from a Study conducted in Germany during 2000 Syngenta, Bracknell, Berkshire, UK Report RJ3234B, Study 00JH086, 17 October 2001 GLP, not published Syngenta File No PP5/0005
PP5/1112	Mason R	2001	Residue levels in dry peas from a study conducted in Germany during 2000 Syngenta, Bracknell, Berkshire, UK Report RJ3209B, Study 00JH085, 18 July 2001 GLP, not published Syngenta File No PP5/1112
PP5/1260	Mason R	2002	Fluazifop-P-butyl: Residue Levels in Vining Peas from a Trial conducted in the UK during 2001 Syngenta, Bracknell, Berkshire, UK Report RJ3336B, Study 01JH083, 2 December 2002 GLP, not published Syngenta File No PP5/1260
PP5/1241	Mason R	2002	Fluazifop-P-butyl Residue levels in potatoes from trials conducted in Southern France during 2001 Syngenta, Bracknell, Berkshire, UK Report RJ3295B, Study 01JH084, 2 December 2002

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			GLP, not published Syngenta File No PP5/1241
PP5/1256	Mason R	2003	Residue Study with Fluazifop-P-Butyl (PP5) in or on Winter Oilseed Rape in Southern France Syngenta, Bracknell, Berkshire, UK, Report 02-7015, 20 February 2003 GLP, not published Syngenta File No PP5/1256
PP5/1365	Mason R	2003	Residue Study with Fluazifop-P-butyl (PP5) in or on Winter Oilseed Rape in France (South) Syngenta, Bracknell, Berkshire, UK Report 03-7004, 23 December 2003 GLP, not published Syngenta File No PP5/1365
PP5/1367	Mason R	2003	Residue Study with Fluazifop-P-butyl (PP5) in or on winter oilseed rape in France (South) Syngenta, Bracknell, Berkshire, UK, Report 03-7005, 23 December 2003 GLP, not published Syngenta File No PP5/1367
PP5/1396	Mason R	2004	Residue Study with Fluazifop-P-Butyl (PP5) in or on Soya in France (North) Syngenta, Bracknell, Berkshire, UK Report 03-7072, 7 July 2004 GLP, not published, Syngenta File No PP5/1396
PP5/1397	Mason R	2004	Residue Study with Fluazifop-P-Butyl (PP5) in or on Soya in France (North) Syngenta, Bracknell, Berkshire, UK Report 03-7073, 7 July 2004 GLP, not published, Syngenta File No PP5/1397
PP5/1398	Mason R	2004	Residue Study with Fluazifop-P-Butyl (PP5) in or on Soya in Switzerland Syngenta, Bracknell, Berkshire, UK Report 03-7026, 27 July 2004 GLP, not published, Syngenta File No PP5/1398
PP5/1399	Mason R	2004	Residue Study with Fluazifop-P-Butyl (PP5) in or on Soya in France (North) Syngenta, Bracknell, Berkshire, UK Report 03-7074, 26 July 2004 GLP, not published Syngenta File No PP5/1399
PP5/1416	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on Potato in Spain Syngenta, Bracknell, Berkshire, UK Report 03-7028, 12 August 2004 GLP, not published Syngenta File No PP5/1416
PP5/1418	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on Potato in Spain Syngenta, Bracknell, Berkshire, UK, Report 03-7027, 12 August 2004 GLP, not published Syngenta File No PP5/1418
PP5/1408	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on Potatoes in Italy Syngenta, Bracknell, Berkshire, UK, Report 03-7038, 9 August 2004 GLP, not published Syngenta File No PP5/1408
PP5/1410	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on Potatoes in Italy Syngenta, Bracknell, Berkshire, UK Report 03-7037, 9 August 2004 GLP, not published Syngenta File No PP5/1410
PP5/1411	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on Potatoes in France (South) Syngenta Bracknell, Berkshire, UK Report 03-7057, 12 August 2004 GLP, not published Syngenta File No PP5/1411

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
PP5/1424	Mason R	2004	Residue Study with Fluzifop-P-butyl (PP5) in or on Potatoes in France (south) Syngenta Bracknell, Berkshire, UK, Report 03-7056, 23 August 2004 GLP, not published Syngenta File No PP5/1424
PP5/1395	Mason R	2004	Residue Analysis for Total Fluazifop in Head Cabbage Generated in German Field Study 02/038 – Analytical Study Syngenta, Bracknell, Berkshire, UK, Report 03-7076, 22 June 2004 includes: Production of Plant Samples for the residue determination of fluazifop-P-butyl after application of Fusilade Max – in two different application schemes – against grass weeds in head cabbage (Savoy cabbage) – Field Study Planzenschutzdienst und Rückstandslabor LUFA der Landwirtschaftskammer Rheinland, Bonn, Germany Report 02/038, AK-Luck-No RU-H-16 02 NW BN 2/2 GLP, not published Syngenta File No PP5/1395
PP5/1426	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on Dry Peas in France (South) Syngenta, Bracknell, Berkshire, UK, Report 03-7059, 7 September 2004 GLP, not published Syngenta File No PP5/1426
PP5/1394	Mason R	2004	Residue Analysis for Total Fluazifop in Head Cabbage Generated in German Field Study 02/037 – Analytical Study Syngenta, Bracknell, Berkshire, UK, Report 03-7068, 22 June 2004 Amendment 1, 26 October 2004, includes: Production of Plant Samples for the residue determination of fluazifop-P-butyl after application of Fusilade Max – in two different application schemes – against grass weeds in head cabbage (Savoy cabbage) – Field Study Planzenschutzdienst und Rückstandslabor LUFA der Landwirtschaftskammer Rheinland, Bonn, Germany Report 02/037, AK-Luck-No RU-H-16 02 NW BN 2/1 GLP, not published Syngenta File No PP5/1394
PP5/1412	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on Fresh Vining Peas without Pods in Spain Syngenta, Bracknell, Berkshire, UK Report 03-7031 13 August 2004 GLP, not published Syngenta File No PP5/1412
PP5/1413	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on Fresh Vining Peas without Pods in Spain Syngenta, Bracknell, Berkshire, UK Report 03-7032, 13 August 2004 GLP, not published Syngenta File No PP5/1413
PP5/1376	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on leeks in the Netherlands, Syngenta, Bracknell, Berkshire, UK, Report 02-7083, 26 February 2004 GLP, not published Syngenta File No PP5/1376
PP5/1377	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on leeks in the UK, Syngenta, Bracknell, Berkshire, UK, Report 02-7035, 26 February 2004 GLP, not published Syngenta File No PP5/1377
PP5/1405	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on leeks in the France (North), Syngenta, Bracknell, Berkshire, UK, Report 02-21401, 30 July 2004 GLP, not published Syngenta File No PP5/1405
PP5/1409	Mason R	2004	Residue Study with Fluazifop-P-butyl (PP5) in or on leeks in the the

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Netherlands Syngenta, Bracknell, Berkshire, UK, Report 03-7029, 9 August 2004 GLP, not published Syngenta File No PP5/1409
PP5/1414	Mason R	2004	Residue study with fluazifop-P-butyl (PP5) in or on potato in Greece Syngenta, Bracknell, Berkshire, UK, Report 03-7079, 12 August 2004, GLP, not published, Syngenta File No PP5/1414
PP5/1415	Mason R	2004	Residue study with fluazifop-P-butyl (PP5) in or on potato in Greece Syngenta, Bracknell, Berkshire, UK Report 03-7080, 12 August 2004 GLP, not published Syngenta File No PP5/1415
PP5/1417	Mason R	2004	Residue study with fluazifop-P-butyl (PP5) in or on potato in Spain Syngenta, Bracknell, Berkshire, UK Report 03-7030, 11 August 2004 GLP, not published Syngenta File No PP5/1417
PP5/1419	Mason R	2004	Residue study with fluazifop-P-butyl (PP5) in or on potato in France (South) Syngenta, Bracknell, Berkshire, UK Report 03-7047, 17 August 2004 GLP, not published Syngenta File No PP5/1419
PP5/1420	Mason R	2004	Residue study with fluazifop-P-butyl (PP5) in or on potato in France (South) Syngenta, Bracknell, Berkshire, UK Report 03-7048, 17 August 2004 GLP, not published Syngenta File No PP5/1420
PP5/1425	Mason R	2004	Residue study with fluazifop-P-butyl (PP5) in or on dry peas in France (South) Syngenta, Bracknell, Berkshire, UK Report 03-7058, Study 03-7058, 7 September 2004 GLP, not published Syngenta File No PP5/1425
PP5/10004	Mason R	2009	Fluazifop-P-butyl: Validation of a residue analytical method for the determination of residues in soya milk and soya based infant formula Syngenta, Bracknell, Berkshire, UK Report RJ3110B, Study 00JH129, 3 April 2009 GLP, not published Syngenta File No PP5/10004
			Includes SOP RAM 336/01 as appendix (see Kwiatkowski & Crook, 2009)
PP5/0006	Mason R, Alevra E	2001	Residue Levels in Head Cabbage from Trials conducted in Greece during 2000 Syngenta, Bracknell, Berkshire, UK, Report RJ3232B, 29 October 2001 GLP, not published Syngenta File No PP5/0006
PP5/1111	Mason R, Atger JC	2001	Residue Levels in Raspberries from trials Conducted in Southern France during 2000 Syngenta, Bracknell, Berkshire, UK, Report RJ3210, Study 00JH049, 18 July 2001 GLP, not published Syngenta File No PP5/1111
PP5/1090	Mason R, Bailey B	2001	Fluazifop-P-butyl Residue levels in dried peas from trials conducted in the UK during 2000 Syngenta, Bracknell, Berkshire, UK Report RJ3211B, Study 00JH065, 27 July 2001 GLP, not published Syngenta File No PP5/1090
PP5/0154	Mason R, Bouwman JJ,	1998	Fluazifop-P-butyl: Residue levels in fresh beans from trials carried out in the Netherlands during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2611B, 27 August 1998 GLP, not published

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Syngenta File No PP5/0154
PP5/0201	Mason R, Bouwman JJ	1998	Residue Levels in Red Fescue from Trials carried out in the Netherlands during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2496B, Study 97JH103, 24 September 1998 GLP, not published Syngenta File No PP/0201
PP5/0210	Mason R, Chamier O	1999	Fluazifop-P-butyl: Residue Levels in Oilseed Rape (Autumn Sown) from Harvest Trials carried out in Germany During 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2766B, 27 May 1999 GLP, not published Syngenta File No PP5/0210
PP5/0209	Mason R, Chamier O	1999	Fluazifop-P-butyl: Residue levels in oilseed rape (spring sown) from harvest trials carried out in Germany during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2765B, Study 98JH131, 27 May 1999 GLP, not published Syngenta File No PP5/0209
PP5/0211	Mason R, Chamier O	1999	Fluazifop-P-butyl: Residue levels in oilseed rape (spring sown) from trials carried out in Germany during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2806B, Study 98JH057, 26 June 1999 GLP, not published Syngenta File No PP5/0211
PP5/1333	Mason R, Clark T	2003	Fluazifop-P-butyl: Residues in Beans (with Pods) from Trials carried out in Southern France and Italy during 2001 Syngenta, Bracknell, Berkshire, UK Report RJ3294B, Study 01JH053, 16 October 2003 GLP, not published Syngenta File No PP5/1333
PP5/0212	Mason R, Codd M	1999	Fluazifop-P-butyl: Residue levels in oilseed rape from trials carried out in the UK during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2758B, 29 June 1999 GLP, not published Syngenta File No PP5/0212
PP5/0153	Mason R, Gallardo E	1998	Fluazifop-P-butyl: Residue Levels in Dried Beans from Trials carried out in Spain During 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2610B, Study 97JH097, 26 August 1998 GLP, not published Syngenta File No PP5/0153, Syngenta File No PP5/0362
PP5/0308	Mason R, Gallardo E	2000	Residue Levels in Sugar Beet from a Trial carried out in Spain during 1999 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2995B, 20 October 2000 GLP, not published Syngenta File No PP5/0308
PP5/1068	Mason R, Giacomelli	2001	Fluazifop-P-butyl – Residues Levels in Soybean from Trials Conducted in Italy During 2000 Syngenta, Bracknell, Berkshire, UK, Report RJ3206B, Study 00JH111, Task TK0219488, 20 June 2001 GLP, not published, Syngenta File No PP5/1068 Synenta File No PP5_50436
			Not summarized because trials could not be matched to the cGAP
PP5/1122	Mason R, Giacomelli G	2001	Fluazifop-P-Butyl : Residue Level in Soybean and Soya Products from Trials conducted in Italy during 2000 Syngenta, Bracknell, Berkshire, UK Report RJ3208B, Study 00JH114, Task TK0219489, 30 November 2001 Report RJ3208B, Study 00JH114, 4 December 2001 (different layout) GLP, not published Syngenta File No PP5/1122

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Syngenta File No PP5_50437
PP5/1091	Mason R, Henson S	2001	Fluazifop-P-butyl Residue levels in potatoes from trials conducted in the UK during 2000 Syngenta, Bracknell, Berkshire, UK Report RJ3200B, Study 00JH064, 19 June 2001 GLP, not publsihed Syngenta File No PP5/1091
PP5/0534	Mason R, Hill SE	1999	Fluazifop/P/butyl: Residue Levels in Sunflower from Trials carried out in Spain & Italy during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2726B, Study 97JH128, 19 May 1999 GLP, not published Syngenta File No PP5/0534
PP5/1118	Mason R, Iniesta L	2001	Fluazifop-P-butyl: Residue Levels in Sunflower from Trials conducted out in Spain during 2000 Syngenta, Bracknell, Berkshire, UK Report RJ3252B, Study 00JH043, 12 November 2001 GLP, not published Syngenta File No PP5/1118
PP5/0203	Mason R, Kappes E	1999	Residue Levels in Red Fescue from Trials carried out in Germany during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2764B, Study 98JH056, 24 August 1999 GLP, not published Syngenta File No PP5/0203
PP5/0158	Mason R, Myles P	1999	Fluazifop-P-butyl: Residue Levels in Dried Peas from Trials Carried out in UK During 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2785B, 29 June 1999 GLP, not published Syngenta File No PP5/0158 Syngenta File No PP5/0369
PP5/0111	Mason R, Picard JM	1999	Fluazifop-P-butyl: Residue levels in chicory witloof from trials carried out in France during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2646B, Study 97JH122, 01 February 1999 GLP, not published Syngenta File No PP5/0111
PP5/0165	Mason R, Volpi E	1998	Fluazifop-P-butyl: Residue Levels in Soyabean from Trials Carried out in Italy During 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2481B, Study 97JH126, Task TK0219483, 3 March 1991 GLP, not published, Syngenta File No PP5/0165 Syngenta File No PP5/0420 Syngenta File No PP5_50432
PP5/0351	Mason R, Volpi E	1999	Residue Levels in Lettuce from Trials carried out in Italy during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2786B, 22 November 1999 GLP, not published Syngenta File No PP5/0351
PP5/0124	Mason R, Volpi E	1999	Fluazifop-P-butyl: Residue Levels in Sugar Beet from Trials Carried out in Italy During 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2779B, 17 September 1999 GLP, not published Syngenta File No PP5/0124
PP5/0125	Mason R, Volpi E	1999	Fluazifop-P-butyl: Residue Levels in Carrots from Trials Carried out in Italy during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2659B, 22 November 1999 GLP, not published Syngenta File No PP5/0125
PP5/0119	Mason R, Volpi E	1999	Residue Levels in Potatoes from Trials carried out in Italy during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2757B, Study 98JH072, 24 May 1999

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			GLP, not published Syngenta File No PP5/0119
			Not summarized because trials could not be matched to the cGAP
PP5/0177	Mason R, Volpi E	1999	Residue Levels in Processing Tomatoes from trials carried out in Italy during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2780B, Study 98JH052, 16 August 1999 GLP, not published Syngenta File No PP5/0177
PP5_50435	Mason R, Volpi E	2000	Fluazifop-P-Butyl — Residue Levels in Soybean and Soya Products from Trials Carried Out in Italy During 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2914B, Study 98JH142, Task TK0219487, 27 April 2000 GLP, not published, Syngenta File No PP5_50435
PP5/1144	Mason R, Volpi E	2002	Residue Levels in Soybean and Soya Products from Trials carried out in Italy during 1999 Syngenta, Bracknell, Berkshire, UK Report RJ3149B, Study 99JH172, Task TK0219490, 9 January 2002 Report RJ3149B, Study 99JH172, 16 January 2002 (different layout) GLP, not published Syngenta File No PP5/1144 Syngenta File No PP5_50438
PP5/0365	Mason R, Gallardo E, Ryan J	1998	Fluazifop-P-butyl: Residues in Fresh Beans from Trials in Spain during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2493B, Study 97JH096, 24 September 1998 GLP, not published Syngenta File No PP5/0365
			Not summarized because trials could not be matched to the cGAP
PP5/0173	Mason R, Gallardo E, Volpi E	1998	Fluazifop-P-butyl: Residue Levels in Cucumbers from Trials in Spain & Italy during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2507B, 8 September 1998 GLP, not published Syngenta File No PP5/0173
PP5/0340	Mason R, Alevra E, Hill SE	1999	Residue Levels in Lettuce from Trials carried out in Greece during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2631B, 19 February 1999 GLP, not published Syngenta File No PP5/0340
PP5/0143	Mason R, Codd M, Myles P	1999	Fluazifop-P-butyl: Residue levels in kale from trials carried out in the UK during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2759B, Study 98JH068, 24 May 1999 GLP, not published Syngenta File No PP5/0143
PP5/0156	Mason R, Kappe E, Glass H	1999	Fluazifop-P-butyl: Residue levels in French beans from harvest trials carried out in Germany during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2629B, 2 February 1999 GLP, not published Syngenta File No PP5/0156
PP5/0139	Mason R, Kappes E, Glass H	1999	Residue Levels in Spinach from Harvest Trials carried out in Germany during 1997 Zeneca Agrochemicals, Bracknell, Berkshire UK Report RJ2632B, Study 97JH127, 4 February 1999 GLP, not published Syngenta File No PP5/0139
			Trials not summarized because no MRLs on spinach are intended **Analytical method validation has been summarized**
PP5/0112	Mason R, Laycock D,	1999	Fluazifop-P-butyl: Residue Levels in Carrot from Trials carried out in Spain and Southern France during 1997

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	Gallardo E, Glass H		Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2638B, 12 February 1999 GLP, not published Syngenta File No PP5/0112
PP5/0189	Mason R, Michalopoulos G, Gallardo E	1999	Fluazifop-P-butyl: Residue Levels in Grapes from Trials Carried out In Greece and Spain During 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2636B, 5 March 1999 GLP, not published Syngenta File No PP5/0189
PP5/0115	Mason R, Michalopoulos G, Gallardo E	1999	Fluazifop-P-butyl: Residue Levels in Sugarbeet from Trials Carried out in Greece and Spain During 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2553B, 5 March 1999 GLP, not published Syngenta File No PP5/0115
PP5/0157	Mason R, Renard C, McGill CD	1999	Fluazifop-P-butyl: Residue Levels in Field Peas from Trials carried out in Northern France During 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2510B, 24 February 1999 GLP, not published Syngenta File No PP5/0157
PP5/0137	Mason R, Renard CL, Hill SE	1999	Fluazifop-P-butyl: Residue Levels in Head Cabbages from Trials carried out in Northern France During 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2645B, 1 February 1999 GLP, not published Syngenta File No PP5/0137
PP5/0159	Mason R, Volpi E, Hill SE	1999	Fluazifop-P-Butyl : Residue Level in Soybean from Trials conducted in Italy during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2781B, Study 98JH055, Task TK0219485, 27 September 1999 GLP, not published, Syngenta File No PP5/0159 Syngenta File No PP5/0370 Syngenta File No PP5_50434
PP5/0188	Mason R, Volpi E, McGill CD	1999	Fluazifop-P-butyl: Residue Levels in olives from Trials in Italy during 1997 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2634B, Study 97JH119, 03 February 1999 GLP, not published Syngenta File No PP5/0188
PP5/0372	Mason R, Gallardo E, Ryan J	2000	Fluazifop-P-butyl: Residues in Fresh Beans from a Trial carried out in Spain during 1999 ICI Agrochemicals, Bracknell, Berkshire, UK Report RJ2993B, Study 99JH201, 30 October 2000 GLP, not published Syngenta File No PP5/0372
PP5/0373	Mason R, Ryan J, Gallardo E	2000	Fluazifop-P-butyl: Residue Levels in Dry Beans from a Trial carried out in Spain during 1999 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2994B, 27 October 2000 GLP, not published Syngenta File No PP5/0373
PP5/0096	Massenot F, Culoto B	1986	Recherche Comparative De Residus De Fluazifop-P-butyl Dans Des Betteraves Sucrieres Traitees Par Pulverisation Classique Ou Electrodyne Laboratoire Analyse de Residus de Bernay, Report D 26-EP, Code D321/78, July 1986 Non-GLP, not published Syngenta File No PP5/0096
A13680D_10051	Matarazzo V	2013	A13680D – Magnitude de Resíduos de Fluazifope-P-Butílico e Fomesafen em Grãos de Soja – Brasil, 2013 Syngenta Proteção de Cultivos Ltda, São Paulo, Brasil Report M13030, 1 November 2013 (Portuguese, 71 pages) Report M13030, 1 November 2013 (Portuguese, 212 pages) Report M13030, Task TK0044488 (English, 116 pages)

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			GLP, not published Syngenta File No A13680D_10051 Syngenta File No A13680D_10052 Syngenta File No A13680D_10056 Syngenta File No A13680D_10057
PP5/0615	Mathis SMG, Harris JE	2001	Fluazifop-P-butyl: Metabolism in Soya Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2948B, 31 January 2001 GLP, not published Syngenta File No PP5/0615
PP5_50036	Mayer L	2009	Analytical method for the determination of fluazifop-P-butyl as fluazifop-P acid in crops by LC-MS/MS Syngenta Crop Protection Inc, Greensboro, NC, USA Report GRM044.01A, Task T002220-07, 10 March 2009 GLP, not published Syngenta File No PP5_50036 (37 pp) Syngenta File No: PP5_50386a (40 pp)
PP5_50029	Mayer L	2009	Validation of Analytical method GRM044.01A for the determination of fluazifop-P-butyl as fluazifop-P acid in crops by LC-MS/MS Syngenta Crop Protection Inc, Greensboro, NC, USA Report T002220-07, Task T002220-07, 10 February 2009 GLP, not published Syngenta File No: PP5_50029 (core), Syngenta File No: PP5_50031 (submission)
PP5_50076	Mazlo J	2009	Fluazifop-P-Butyl – Magnitude of the Residues in or on Cotton Syngenta Crop Protection, Inc, Greensboro, NC, USA, JRF America, King of Prussia, PA, USA Report T002224-07, JRF Study KP-2009-23, 14 December 2009 GLP, not published Syngenta File No PP5_50076
PP5_50071	Mazlo J	2009	Fluazifop-P-Butyl – Magnitude of the Residues in or on Carrots Syngenta Crop Protection, Inc, Greensboro, NC, USA JRF America, King of Prussia, PA, USA Report T002222-07, Study KP-2009-22, Task T002222-07, 23 November 2009 GLP, not published Syngenta File No PP5_50071 (101 pages, two different layouts)
A12460A_50019	Mazlo J	2013	Fluazifop-P-butyl (A12460A) – Magnitude of the Residues in or on Citrus Processed Commodities after Applications of Fluazifop-P-butyl DX Herbicide USA 2011 Syngenta Crop Protection LLC, Greensboro, NC, USA, Morse Laboratories, LLC, Sacramento, USA, University of Idaho Food Technology Center, Caldwell, ID, USA, Report TK0058357, Morse Study 68388, 27 February 2013 GLP, not published Syngenta File No A12460A_50019
PP5/0821	McCarron E, Heath J	1989	Fluazifop-P-butyl: Hydrolysis in Sterile Aqueous Solution ICI Agrochemicals, Bracknell, Berkshire, UK Report RJ0779B, 22 November 1989 GLP, not published Syngenta File No PP5/0821
PP5/0455	McGill C	2000	Fluazifop-P-Butyl: Residue Levels in Strawberries (outdoor) from trials carried out in Southern France and Italy during 1999 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ3074B, 30 October 2000 GLP, not published Syngenta File No PP5/0455
PP5/1233	McGill C	2002	Fluazifop-P-Butyl : Residue Levels in Dried Peas from Trials conducted in Southern France and Italy during 2001 Syngenta, Bracknell, Berkshire, UK, Report RJ3300B, 16 September 2002 GLP, not published Syngenta File No PP5/1233
PP5/1150	McGill C	2002	Fluazifop-P-Butyl: Variability in Residue Levels in Potatoes from an Exploratory Study conducted in the UK during 2001 Syngenta, Bracknell, Berkshire, UK

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Technical Letter 01JH128/01, Study RPLAN050, no date No GLP, not published Syngenta File No PP5/1150
PP5/1281	McGill C	2003	Stability of Residues of Various Crops and Processed Fractions Stored for up to 18 months in Deep Freeze Syngenta, Bracknell, Berkshire, UK Report RJ3087B, Study 99JH217, 27 August 2003 GLP, not published Syngenta File No PP5/1281
PP5/1232	McGill C, Crawford M	2002	Fluazifop-P-Butyl: Residue Levels in Beans (with pods) from Trials Conducted in the UK and Northern France during 2001 Syngenta, Bracknell, Berkshire, UK, Report RJ3299B, 17 July 2002 GLP, not published Syngenta File No PP5/1232
PP5/1227	McGill C, Richards S	2002	Fluazifop-P-butyl Residue levels in dried peas from a trial conducted in the UK during 2001 Syngenta, Bracknell, Berkshire, UK Report RJ3266B, Study 01JH085, 15 July 2002 GLP, not published Syngenta File No PP5/1227
PP5/0309	McGill C, Sutra G	2000	Fluazifop-P-Butyl: Residue Levels in Carrots from Trials conducted in Southern France during 1999 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ3065B, 26 October 2000 GLP, not published Syngenta File No PP5/0309
405660	McKay JC	1989	Fusilade 2000 (fluazifop-P-butyl): magnitude of the residue study on dry beans ICI Americas Inc, Richmond, CA, USA Report RR 89-046B, Study 4-005-87-05, 18 August 1989 Non-GLP, not published Syngenta File No 405660 **Cowpea trials were not summarized, because no cGAP was available. Other trials were summarized. This report also contains the residue trials listed in Watson & Francis, 1986, PP5/0378, report TMU3094/B.**
PP5/0105	Miles PD, Cowley P	1997	Fluazifop-P-butyl: Residue levels in asparagus from a trial carried out in Spain during 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2281B, 21 November 1997 GLP, not published Syngenta File No PP5/0105
PP5/0134	Miles PD, Cowley P	1997	Fluazifop-P-butyl: Residue Levels in Lettuce from Trials carried out in Northern France and Spain During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2363B, 21 November 1997 GLP, not published Syngenta File No PP5/0134
PP5/0133	Miles P, Hill SE	1997	Fluazifop-P-butyl: Residue Levels in Cabbage from Trials Carried out in France During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2312B, 21 November 1997 GLP, not published Syngenta File No PP5/0133
PP5/0197	Miles PD, Nassoy G	1997	Fluazifop-P-butyl: Residue Levels in Lemons from Trials carried out in France During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2241B, 29 April 1997 GLP, not published Syngenta File No PP5/0197
PP5/0163	Miles PD, Nassoy G	1997	Fluazifop-P-butyl: Residue Levels in Soybeans from Trials carried out in France during 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2368B, Study 96JH057, Task TK0219470, 17 November 1997 GLP, not published,

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Syngenta File No PP5/0163 Syngenta File No PP5/0418 Syngenta File No PP5_50429
PP5/0170	Miles PD, Nassoy G	1997	Fluazifop-P-butyl: Residue Levels in Tomatoes from Trials Carried out in France During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2370B, 21 November 1997 GLP, not published Syngenta File No PP5/0170
PP5/0207	Miles PD, Cowley P, Hill SE	1997	Fluazifop-P-butyl : Residue Levels in Sunflower Seeds from Trials carried out in Spain During 1996 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2303B, Study 96JH065, 29 September 1997 GLP, not published Syngenta File No PP5/0207
PP5/0368	Miller MM	1999	Residue Levels in Soybean from Trials carried out in the United States during 1998 (WRC-99-044) (WINO 40034) Zeneca Ag Products, Richmond, CA, USA, Report RR 99-021B, Study FLUA-98-MR-01, Project 40034, 6 April 1999 GLP, not published Syngenta File No PP5/0368 Syngenta File No PP5_50413 Syngenta File No 405819
PP5/0454	Miller MM	2000	Fluazifop-P-butyl: Residue Levels in Banana from Trials carried out in the United States during 1999 Zeneca Ag Products, Inc, Richmond, CA, USA Report RR 00-043B, Project FLUA-99-MR-01 WINo 44068, 20 September 2000 GLP, not published Syngenta File No PP5/0454
			This report has the same content as Kleinschmidt & Miller, 2000, 405683
PP5/50544	Moore P	2014	Fluazifop-P-Butyl: Discussion on Analytical Methods for R and S Enantiomers of Fluazifop-Butyl and Fluazifop Acid Syngenta Crop Protection, LLC, USA Report TK0251855, 24 September 2014 Not GLP, not published Syngenta File No PP5/50544
PP5/0250	Morgan JL, Crook SJ	1986	Fluazifop-P-butyl (reference X): Residues in Onions from Trials Carried out in the USA during 1984 ICI Plant Protection Division, Greensboro, NC, USA Report M4266B, study PP005B072, 10 July 1986 GLP, not published Syngenta File No PP5/0250
			Analysis of CF3-pyridone (compound X) in the trials from report TMU1815/B
PP5/0191	O'Brien M, Harradine KJ	1987	Fluazifop-P-butyl: Residues in Oranges (Peel and Flesh) from Trials Carried out in Italy During 1986 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4533B, 30 June 1987 Non-GLP, not published Syngenta File No PP5/0191
PP5/0485	O'Brien M, Harradine KJ	1987	Fluazifop-P-butyl: Residues in Olives from Trials Carried out in Italy During 1986 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4526B, 19 June 1987 Non-GLP, not published Syngenta File No PP5/0485
CGA181847_50001	Oddy AM, Doble ML	2011	Fluazifop-P-butyl – Rate of Degradation of [14C]-R150397, a Soil Metabolite, in Three Soils under Aerobic Laboratory Conditions at 20°C Final Report – Amendment 1 Battelle UK Ltd, Ongar, Essex, CM5 0GZ, UK Report Number NC/09/015, Study NC/09/015, Task T000130-09, 25 October 2011

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			GLP, not published Syngenta file No: CGA181847_50001
PP5/0416	Patel A, Elliott GA	1996	Fluazifop-P-Butyl: Residue Levels in Field Beans from Trials Carried Out in the UK During 1994 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ1894B, 2 January 1996 GLP, not published Syngenta File No PP5/0416
PP5/0130	Patel A, Robinson WJ	1994	Fluazifop-P-Butyl: Residue Levels in Savoy Cabbage from Trials Carried Out in Germany During 1993 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ1583B, Study 93JH094, 28 April 1994 Includes Fluazifop-P-butyl: Residues in Savoy Cabbage, Federal Republic of Germany 1993, Chamler OD, Zeneca Study RS-9310, 22 December 1993 GLP, not published Syngenta File No PP5/0130
PP9/0044	Patel H, Cavell BD, Bell EG	1983	Fluazifop-butyl: Metabolism of ^{14}C-fluazifop-butyl in cucumber fruits ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0299B, 11 February 1983 GLP, not published Syngenta File No PP9/0044
PP5/0564	Patel A, Harradine KJ, Codd M, Leaper DJ	1993	Fluazifop-P-butyl: Residue levels in Spring Oilseed Rape from Trials Carried Out in the UK during 1992 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ1456B, Study 92JH055, 9 August 1993 GLP, not published Syngent File No PP5/0564
PP5/0103	Patel A, Atreya NC, Frost MJ	1995	Fluazifop-P-butyl: Residue levels in carrots from trials carried out in the UK during 1994 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ1884B, Study 94JH053, 30 June 1995 GLP, not published Syngenta File No PP5/0103
PP5/0396	Pay J	1986	Fluazifop-P-Butyl – Residues in Fodder Pea from trials in West Germany during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4234B, 18 June 1986 Non-GLP, not published Syngenta File No PP5/0396
PP5/0409	Pay J	1987	Fluazifop-P-Butyl : Residues in soybean from trials in Canada during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4322B, 19 January 1987 GLP, not published, Syngenta File No PP5/0409
			Not summarized because trials could not be matched to the cGAP
PP5/0185	Pay J	1987	Fluazifop-P-butyl: Residues in Bananas from Trials in Honduras During 1984, 1985 and 1996 ICI Plant Protection Division, Bracknell, Berkshire, UK, Report M4388B, Study PP005B071, 27 July 1987 GLP, not published Syngenta File No PP5/0185
PP5/0374	Pay J, Atreya NC	1986	Fluazifop-P-Butyl: Residues in Fodder Bean From Trials in West Germany During 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4233B, 27 August 1986 Non-GLP, not published Syngenta File No PP5/0374
PP5/0816	Pay J, Harradine KJ	1986	Fluazifop-P-butyl: Residues in soil from dissipation trials in Canada during 1985 ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0539B, 23 December 1986 Non-GLP, not published, Syngenta File No PP5/0816

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Not summarized, because no information on the persistent soil metabolite CF3-pyridone was provided
PP5_50062	Pelz S	1994	Determination of Residues of Total-Fluazifop (Fluazifop-P-Butyl + Fluazifop-P) in Potatoes and Their Processed Products Federal Republic of Germany 1993 Dr Specht and Partner Chemische Laboratorien GmbH, Germany Report AZ13430/93, Study ICI-9301, Task TK0022593, 16 May 1994 GLP, not published Syngenta File No PP5_50062
			This report is identical to Weeren, 1994, PP5/0102
PP9/0107	Plyler S, Francis P	1987	Fluazifop-Butyl: Residues of Fluazifop on Lettuce ICI Americas Inc, Greensboro, NC, USA Report TMU3005/B, original 4 June 1986 (17 pages) Report TMU3005/B, revision 4 March 1987 (19 pages) GLP, not published Syngenta File No PP9/0107
			Not summarized because trials could not be matched to the cGAP
PP5_50411	Pyles S, Hagan M	2013	Stability of Fluazifop-P-Butyl (R154875), Fluazifop-P Acid (R156172), and Compound X (R154719, CGA142110) in Soil Under Freezer Storage Conditions Syngenta Crop Protection, LLC, Greensboro, NC, USA ALS Environmental, Edmonton, Alberta, Canada Report TK0015285, ALS report 13SYN328REP, Task TK0015285, 2 August 2013 GLP, not published Syngenta File No PP5_50411
PP5_50002	Quistad GB	2008	^{14}C-fluazifop-P-butyl Nature of Residue in Carrot PTRL West Inc, Hercules, GA, USA Excel Research Services, Fresno, CA, USA Report 1689W, Task T010256-06, 14 October 2008 GLP, not published Syngenta File No: PP5_50002
PP005_50034	Quistad GB	2008	^{14}C-fluazifop-P-butyl Nature of Residue in Endive PTRL West Inc, Hercules, GA, USA Excel Research Services, Fresno, CA, USA Report 1690W, Task T010255-06, 5 December 2008 GLP, not published Syngenta File No: PP005_50034
PP9/0272	Rapley JH, Arnold DJ, Weissler MS, White RD	1981	Fluazifop-butyl: Development of Methods to Study Its Degradation in Soil ICI Plant Protection Division, Location not indicated Report RJ0158B, 28 April 1981 GLP, not published Syngenta File No PP9/0272
PP5/1222	Richards S	2002	Fluazifop-P-Butyl: Residue Levels in Leeks from Trials Conducted in the UK and Northern France during 2001 Syngenta, Bracknell, Berkshire, UK Report RJ3278B, 24 June 2002 GLP, not published Syngenta File No PP5/1222
PP5/0595	Robertson TA, Hand LH	1999	Fluazifop-P-butyl: Metabolism in the Hen Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2839B, 13 December 1999 GLP, not published Syngenta File No PP5/0595
PP5/0218	Robinson NJ, Patel A	1994	Fluazifop-P-butyl: Residue Levels in Sunflowers from Trials Carried out in Germany During 1993 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ1656B, Study 93JH134, 29 June 1994 GLP, not published Syngenta File No PP5/0218
PP5/0611	Robinson NJ, Crook SJ,	2000	Residue Analytical Method for Determination of Residues of Total Fluazifop in Bovine Muscle Tissue, Liver, Kidney, Fat, Milk and Hen Egg

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	Hargreaves SL		Zeneca Agrochemicals, Bracknell, Berkshire, UK, SOP RAM 331/01, 6 April 2000 Non-GLP, not published Syngenta File No PP5/0611
PP5/0572	Roper EM	1992	Fusilade 2000 : Magnitude of the Residues of Fluazifop-P-Butyl and Fluazifop in Almond Hulls and Nutmeats (WRC-92-060) ICI Americas Inc, Western Research Center, Richmond, CA, USA, Report RR 92-041B, Project ID 0005-90-MR-01, 25 September 1992 GLP, not published Syngenta File No PP5/0572
PP5/0582	Roper EM	1992	FUSILADE 2000 : Magnitude of the Residues of Fluazifop-P-Butyl and Fluazifop in Walnut Nutmeats (WRC-92-012) ICI Americas Inc, Western Research Center, Richmond, CA, USA, Report RR 92-009B, Study 0005-90-MR-3, 25 September 1992 GLP, not published Syngenta File No PP5/0582
430705	Roper E, Francis P	1987	Fluazifop-P-butyl: Residues of Fluazifop in Carrots ICI Americas Inc, Greensboro, NC, USA Reports RSR-027-87/C, 30 September 1987 GLP, not published Syngenta File No 430705 Syngenta File No 405742
405720	Roper E, Francis P	1988	Fluazifop-P-Butyl: Residues of Fluazifop in Sugarcane Following Spot Applications of Fusilade 2000 ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU3310/B, 23 March 1988 Not GLP, not published Syngenta File No 405720
PP5/0584	Roper EM, Graham DG	1992	FUSILADE: Residues of Fluazifop and Fluazifop-P-Butyl on Fresh Asparagus Spears and on Boiled, Steamed and Microwaved Spears (WRC-92-080) ICI America Inc, Western Research Center, Richmond, CA, USA, Report RR 92-057B, Study FLUA-91-MR-03, 4 August 1992 GLP, not published Syngenta File No PP5/0584
PP5/0126	Ryan J	1999	Fluazifop-P-butyl: Residue Levels in Onions from Trials Carried out in Southern France and Spain During 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2827B, 1 December 1999 GLP, not published Syngenta File No PP5/0126
PP5/0447	Ryan J	2000	Residue Levels in Cucumbers Grown Indoors from a Trial carried out in Spain during 1999 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ3058B, 31 October 2000 GLP, not published Syngenta File No PP5/0447
PP5/0145	Ryan J, Atger JC	1999	Fluazifop-P-butyl: Residue Levels in Lettuce from Trials Carried out in Southern France Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2782B, Study 98JH069, 24 May 1999 GLP, not published Syngenta File No PP5/0145
PP5/0160	Ryan J, Gallardo E	1999	Fluazifop-P-butyl: Residue Levels in Dried Beans from Trials Carried out in Spain During 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2826B, 30 September 1999 GLP, not published Syngenta File No PP5/0160 Syngenta File No PP5/0371
PP5/0121	Ryan J, Gallardo E	1999	Fluazifop-P-butyl: Residue Levels in Sugar Beet from Trials Carried out in Spain During 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2833B, 29 September 1999 GLP, not published Syngenta File No PP5/0121

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
PP5/0208	Ryan J, Gallardo E	1999	Fluazifop-P-butyl: Residue Levels in Oilseed Rape from Trials carried out in Spain during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2771B, 5 May 1999 GLP, not published Syngenta File No PP5/0208
PP5/0118	Ryan J, Gallardo E	1999	Fluazifop-P-butyl: Residue Levels in Carrots from Trials carried out in Spain during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2772B, Study 98JH039, 5 May 1999 GLP, not published Syngenta File No PP5/0118
PP5/1149	Ryan J, Iniesta L	2002	Fluazifop-P-Butyl : Residue Levels in Potatoes from Trials conducted in Spain during 2000 Syngenta, Bracknell, Berkshire, UK, Report RJ3222B, 18 February 2002 GLP, not published Syngenta File No PP5/1149
PP5/0610	Ryan J, Kenny D	1999	Fluazifop-P-butyl: Validation of a residue analytical method for the determination of total fluazifop in animal products Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2873B, Project ID 99JH225, 2 December 1999 GLP, not published Syngenta File No PP5/0610
PP5/0542	Ryan J, LeSiourd J	2000	Residue Levels in Sunflowers from Trials carried out in Northern France during 1999 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2940B, Study 99JH147, 31 October 2000 GLP, not published Syngenta File No PP5/0542
PP5/0120	Ryan J, Renard C	1999	Fluazifop-P-butyl: Residue Levels in Celeriac from Trials carried out in Northern France during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2804B, 23 June 1999 GLP, not published Syngenta File No PP5/0120
PP5/0146	Ryan J, Renard C	1999	Fluazifop-P-butyl: Residue Levels in Head Cabbage from Trials carried out in Northern France During 1998 Zeneca, Agrochemicals, Bracknell, Berkshire, UK, Report RJ2794B, 23 June 1999 GLP, not published Syngenta File No PP5/0146
PP5/0147	Ryan J, Sutra G	1999	Fluazifop-P-butyl: Residue levels in leafy cabbage from trials carried out in northern France during 1998 Zeneca Agrochemicals, Bracknell, Berkshire, UK Report RJ2834B, Study 98JH133, 28 June 1999 GLP, not published Syngenta File No PP5/0147
A12791B_50001	Sagan K	2008	Fluazifop EC (A12791N) – Residue Levels on Edible Beans from Trials Conducted in Canada During 2007 Syngenta Crop Protection Canada Inc, Guelph, Ontario, Canada Report CER 02607/07, 26 September 2008 GLP, not published Syngenta File No A12791B_50001
A12791B_50003	Sagan K	2008	Fluazifop EC (A12791B) - Residue Levels on Soybeans (Forage, Hay and Seed) from Trials Conducted in Canada during 2007 Syngenta Crop Protection Canada Inc, Guelph, Ontario, Canada Report CER 02605/07, 7 November 2008 GLP, not published, Syngenta File No A12791B_50003
A12791B_50005	Sagan K	2008	Fluazifop EC(A12791B) - Residue Levels on Potatoes from Trials Conducted in Canada during 2007 ALS Laboratory Group, Edmonton, Canada Report CER 02606/07, Task TK0021557, 30 September 2008 GLP, not published

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Syngenta File No A12791B_50005 Includes as appendix 2 : Tauber, R and Stilson, S 2008 Analytical Phase Report: Fluazifop EC (A12791B) – Residue Levels on Potatoes from Trials Conducted in Canada During 2007 Report 08SYN226REP, 7 March 2008
A12791N_50004	Sagan K	2009	Fluazifop EC (A12791N) - Residue Levels on Potatoes from Trials Conducted in Canada During 2008 ALS Laboratory Group, Edmonton, Canada Report CER 02608/08, Task TK0021556, 22 June 2009 GLP, not published Syngenta File No A12791N_50004 Includes as appendix 2 : Abetew, M 2009 Analytical Phase Report: Fluazifop EC (A12791N) – Residue Levels on Potatoes from Trials Conducted in Canada During 2008 ALS Report 09SYN248REP, 19 February 2009
A12791N_50001	Sagan K	2009	Fluazifop EC (A12791N) – Residue Levels on Edible Beans from Trials Conducted in Canada During 2008 Syngenta Crop Protection, Canada Inc, Guelph, Ontario, Canada Report CER 02609/08, 15 June 2009 GLP, not published Syngenta File No A12791N_50001
A12791B_50006	Sagan K	2010	Fomesafen/Fluazifop — Residue Levels on Soybeans (Forage, Hay and Seed) from Trials Conducted in Canada during 2006 Syngenta Crop Protection Canada Inc, Guelph, Ontario, Canada Report CER 02401/06, 12 April 2010 GLP, not published, Syngenta File No A12791B_50006
			Forage and hay data were summarized Residues in seeds were not summarized because trials could not be matched to the cGAP
PP5_50339	Schmitt J, Perez R	2013	Fluazifop-P-Butyl - Independent Laboratory Validation of Analytical Method (GRM04403A) for the Determination of Fluazifop-P-Butyl (R154875, PP5), Fluazifop-P Acid (R156172), Compound X (R154719, CGA142110) in Soil Using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS) ADPEN Laboratories, Inc, Jacksonville, FL, USA Report TK0114928, Task TK0114928, 6 February 2013 GLP, not published Syngenta File No PP5_50339
PP5/1148	Simon P	2002	Determination of Residues of Fluazifop-P-Butyl in Potatoes in Germany Syngenta Agro GmbH, Maintal, Germany, Report gpo11501, 30 January 2002 GLP, not published Syngenta File No PP5/1148
PP5/1147	Simon P	2002	Determination of Residues of Fluazifop-P-Butyl in Potatoes in Germany Syngenta Agro GmbH, Maintal, Germany Report gpo31501, 30 January 2002 GLP, not published Syngenta File No PP5/1147
PP5/1145	Simon P	2002	Determination of Residues of Fluazifop-P-Butyl in Potatoes in Germany Syngenta Agro GmbH, Maintal, Germany, Report gpo41501, 30 January 2002 GLP, not published Syngenta File No PP5/1145
PP5/1146	Simon P	2002	Determination of Residues of Fluazifop-P-Butyl in Potatoes in Germany Syngenta Agro GmbH, Maintal, Germany Report gpo91501, 30 January 2002 GLP, not published Syngenta File No PP5/1146
PP5/1342	Simon P	2003	Residues of Fluazifop-P-butyl after Application of A12791B in Potatoes, Germany 2002 Syngenta Agro GmbH, Maintal, Germany, Report gpo079002, 25 August 2003

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			GLP, not published Syngenta File No PP5/1342
PP5/1337	Simon P	2003	Residues of Fluazifop-P-butyl after Application of A12791B in Sugarbeets, Germany 2002 Syngenta Agro GmbH, Maintal, Germany Report gsb064002, 8 October 2003, includes: Mason, R, 2003: Residues of fluazifop-P-butyl after application of A12791B in sugarbeet, Germany 2002 Analytical Phase Report Syngenta, Bracknell, Berkshire UK, Report gsb064002, 3 September 2003 GLP, not published Syngenta File No PP5/1337
PP5/1336	Simon P	2003	Residues of Fluazifop-P-butyl after Application of A12791B in Sugarbeets, Germany 2002 Syngenta Agro GmbH, Maintal, Germany Report gsb064202, 8 October 2003, includes: Mason, R, 2003: Residues of fluazifop-P-butyl after application of A12791B in sugarbeet, Germany 2002 Analytical Phase Report Syngenta, Bracknell, Berkshire UK, Report gsb064202, 3 Sept 2003 GLP, not published Syngenta File No PP5/1336
PP5/1427	Simon P	2004	Residues of Fluazifop-P-butyl after Application of A12791B in Potatoes, Germany 2003 Syngenta Agro GmbH, Maintal, Germany Report gpo023103, 15 October 2004 GLP, not published Syngenta File No PP5/1427
PP9/0041	Snow AD, Hignett RR and Cavell BD	1983	Fluazifop-butyl: Metabolism of 14C-fluazifop-butyl in potatoes ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0325B, 29 September 1983 Non-GLP, not published Syngenta File No PP9/0041
PP5_50598	Sparrow K	2015	Fluazifop-P - Physical and Chemical Properties (Metabolite of Fluazifop-P-Butyl, PP005) Syngenta Crop Protection, LLC, Greensboro, NC, USA Report PC-15-037, TK0180660, 12 February 2015 GLP, not published Syngenta File No PP5_50598
			Study identical to Wollerton & Husband, 1992, R156172/0001, report RJ1263B. Only the front pages were different.
PP5/1069	Stewart ER	2001	Fluazifop-P-Butyl – Residue levels on dry beans from trials conducted in the United States during 2000 (WRC-00-086) (WINO 49034) Stewart Agricultural Research Services, Inc, Clarence, MO, USA, Report RR 00-061B, Zeneca FLUA-00-MR-01 & WINO 49034, 31 May 2001 GLP, not published Syngenta File No PP5/1069 (164 pages) Syngenta File No 404614 (163 pages)
406504	Stewart ER	2001	Residue Levels on Grapes from Trials Conducted in the United States during 2000 Stewart Agricultural Research Services, Inc, Clarence, MO, USA, Report RR 00-062B, Project FLUA-00MR-02 & T001432-01, 27 August 2001 GLP, not published Syngenta File No 406504
406466	Stewart ER	2001	Fluazifop-P-Butyl – Residue Levels on Citrus from Trials Conducted in the United States during 2000 Stewart Agricultural Research Services, Inc, Clarence, MO, USA, Report RR 00-063B, Task T001434-01, 27 August 2001 GLP, not published Syngenta File No 406466
406507	Stewart ER	2001	Residue levels on soybeans from trials conducted in the United States during 2000 Stewart Agricultural Research Services, Inc, Clarence, MO, USA, Report RR 00-065B, Project FLUA-00-MR-05 & WINO 37481, 27 August

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			2001 GLP, not published Syngenta File No 406507 Syngenta File No 406511 Syngenta File No 453979
PP5/1070	Stewart ER	2001	Fluazifop-P-Butyl: Residue Levels on Sugarbeets from Trials Conducted in the United States during 2000 Stewart Agricultural Research Services, Inc, Clarence, MO, USA, Report RR 00-066B, Study FLUA/00/MR/06, Task, T001479/01, 31 May 2001 GLP, not published Syngenta File No PP5/1070
406498	Stewart ER	2001	Fluazifop-P-Butyl: Residue Levels on Grape Juice and Raisins from a Trial Conducted in the United States in 2000 Stewart Agricultural Research Services, Inc MO, USA Report RR 00-067B, Study FLUA-00-PR-01, Task T001433-01, 27 August 2001 GLP, not published Syngenta File No 406498
406508	Stewart ER	2001	Residue Levels on Soybean Meal, Hulls, and Oil (Refined) from a Trial Conducted in the United States during 2000 Stewart Agricultural Research Services, Inc, Clarence, MO, USA, Report RR 00-069B, Project FLUA-00-PR-03 & T001478-01, 27 August 2001 GLP, not published Syngenta File No 406508
406493	Stewart ER	2001	Fluazifop-P-Butyl: Residue Levels on Sugarbeet Sugar (Refined), Dry Pulp and Molasses from a Trial Conducted in the United States during 2000 Stewart Agricultural Research Services, Inc MO, USA Report RR 00-070B, Study FLUA-00-PR-04, T001480-01, 10 September 2010 GLP, not published Syngenta File no 406493 **Some trials not summarized because they could not be matched to the cGAP. Processing data were summarized**.
PP5/1497	Suszter, G	2003	Residue analytical determination of active ingredient of Fusilade Forte (fluasifop-P-butyl) in sunflower PPSCS of Borsod-Abauj-Zemplen County, Miskolc, Hungary Report 02SYNAA0505, 25 March 2003 GLP, not published Syngenta File No PP5/1497
-	Suzuki L	2011	Determinaçao de resíduos de fluazifop total em amostras vegetais por LC/MS/MS Syngenta Protecao de Cultivos Ltda, Sao Paulo, Brazil Report POPIT.MET.138.Rev.00, 26 October 2011 Non-GLP, not published Syngenta File No: not available
PP9_50000	Swain WE	2009	Fluazifop-Butyl - Residue Transfer Study with Dairy Cows Fed on a Diet Containing the Herbicide (MRID 00093843) : Response Syngenta Crop Protection, Greensboro, NC, USA Report T008915/08, Task T008915/08, 19 February 2009 Not GLP, not published Syngenta File No PP9_50000
PP9_50001	Swain WE	2009	Fluazifop-Butyl - Residue Transfer Study with Laying Hens Fed on a Diet Containing the Herbicide (MRID 00093845): Response Syngenta Crop Protection, Greensboro, NC, USA Report T008916/08, Task T008916/08, 19 February 2009 Not GLP, not published Syngenta File No PP9_50001
PP9/0183	Swaine H, Francis PD	1981	Fluazifop-butyl: Residue Transfer Study with Laying Hens Fed on a Diet Containing the Herbicide ICI Plant Protection Division, Bracknell, Berkshire, UK Report RJ0217B, 29 September 1981 GLP, not published Syngenta File No PP9/0183
-	Syngenta	2015	Response to Questions 02, 26 March 2015
-	Syngenta	2016	Response to Questions 11, 2 May 2016
-	Syngenta	2016	Response to Questions 14, 26 August 2016

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
-	Syngenta	2016	Response to Questions 15, 26 August 2016
-	Syngenta	2016	Response to Questions 16, 5 September 2016
R156172_50001	Tauber R, Hagan M	2013	Storage of fluazifop-P-butyl residues as fluazifop acid (fluazifop-P) in processed fractions of potato, soybean, tomato and wheat under freezer storage conditions for up to 12 months Amendment #1 ALS Environmental, Edmonton, Alberta, Canada Report 13SYN331REP Amendment #1, Study TK0058356, 8 August 2013 GLP, not published Syngenta File No R156172_50001
A12530B_10010	Tomaz ML	2008	Determinação de resíduos totais de Fluazifop-P em sementes de girasol após applicação de Fusilade 250 EW Plantec Pianejamento e Tecnología Agrícola Ltda, Iracemápolis, SP, Brasil, Report 027-003-07B, Study T06030, 30 September 2008 GLP, not published Syngenta File No A12530B_10010
PP9/0036	Trumbo KE, Francis PD	1986	Fluazifop-butyl: Storage Stability of Residues in Deep Frozen Tomato Samples ICI Americas Inc, Goldsboro, NC, USA, Report TMU3079, 31 July 1986 GLP, not published Syngenta File No PP9/0036
PP9/0037	Trumbo KE, Francis PD	1986	Fluazifop-butyl: Storage Stability of Residues in Deep Frozen Celery Samples ICI Americas Inc, Goldsboro, NC, USA, Report TMU3074, 31 July 1986 GLP, not published Syngenta File No PP9/0037 (7 pages) Syngenta File No 405759 (11 pages, additional cover pages)
PP9/0399	Upton B	1984	Fluazifop butyl: Residues in Oil Seed Rape from Trials in West Germany During 1982-1983 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M3685B, 7 February 1984 GLP, not published Syngenta File No PP9/0399
PP9/0035	Upton B	1986	Fluazifop butyl: Reference X Method Validation Data Carrots 1984 ICI Plant Protection Division, Bracknell, Berkshire, UK Report M4239B, 15 May 1986 GLP, not published Syngenta File No PP9/0035
PP9/0054	Upton BP, Atreya NC	1984	Fluazifop-butyl: Residues in Sugarbeet and Fodderbeet From Trials in West Germany 1983 ICI Plant Protection Division, Bracknell, Berkshire, UK, Report M3701B, 7 February 1984 Non-GLP, not published Syngenta File No PP9/0054
406278	Ussary JP	1981	Fluazifop Residues in Soybeans ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU0678/B original, 2 October 1981 (25 pages) Report TMU0678/B revised, 2 December 1981 (30 pages) GLP, not published, Syngenta File No 406277 (original report) Syngenta File No 406278 (revised report) **This report describes the trials from Atreya et al, 1981, PP9/0736, report PP009B036, but contains some additional information** **Not summarized because trials could not be matched to the cGAP**
405792	Ussary JP	1981	Fluazifop Residues in Cottonseed ICI Americas Inc, Agricultural Chemicals Division, North Carolina, USA Report TMU0679/B, 2 October 1981 GLP, not published Syngenta File No 405792
405793	Ussary JP	1981	**Study contains report PP009B035 as appendix** Fluazifop-butyl Metabolite Reference III Residues in Cottonseed ICI Americas Inc, Agricultural Chemicals Division, North Carolina, USA Report TMU0680/B, September 1981 GLP, not published

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
			Syngenta File No 405793
			Study contains report PP009B042 as appendix.
PP9/0284	Ussary JP	1981	Fluazifop-butyl Dissipation in Soils ICI Americas Inc., Goldsboro, North Carolina, USA Report No. TMU0657/B, 13 July 1981 GLP, not published Syngenta File No PP9/0284
			Not summarized because field dissipation studies from 4 sites in the USA in 1979 contained no information on the soil persistent CF3-pyridone
405714	Ussary JP	1981	Fluazifop-butyl residues in soil from soybean and cotton field trials ICI Americas Inc, Greensboro, NC, USA Report TMU0676/B, 3 November 1981 GLP, not published Syngenta File No 405714 (NAFTA SD)
			Not summarized because field dissipation studies from 6 sites in the USA in 1980 contained no information on the soil persistent CF3-pyridone
PP9/0176	Ussary JP	1981	Fluazifop- butyl Crop Rotation Field Residue Study ICI Americas Inc, Greensboro, USA Report TMU0671/B, 17 August 1981 GLP, not published, Syngenta File No PP9/0176
			Contains Swaine H, 1981, Residue Data Report PP009/BO/41, QA 569/PP009/BO/41, Fluazifop-butyl in rotated crops in USA 1980 as appendix
PP5/0079	Walter DJ	1996	Fluazifop-P-butyl: Method Validation for the Determination of Residues of Total Fluazifop in Various Crops Zeneca Agrochemicals, Bracknell, Berkshire, UK, Report RJ2172B, Study 96JH163, 17 December 1996 Includes: Bolygo E Fluazifop-P-butyl: Residue analytical method for total fluazifop analysis in crops An external standard procedure using liquid chromatography with MS-MS or UV detection Standard Operating Procedure RAM 287/01 GLP, not published Syngenta File No PP5/0079
PP5/10008	Wang M	2009	Fluazifop-P-butyl – Calculation of Kinetic Trigger Endpoints in Soil from Laboratory Study Data according to FOCUS Kinetics Guidelines (including the soil metabolites R156172 and R154719) RIFcon GmbH, Heidelberg, Germany, Report R-09049-2, 31 March 2009 GLP, not published Syngenta File No PP5/10008
PP5/10009	Wang M	2009	Fluazifop-P-butyl – Calculation of Kinetic Modelling Endpoints in Soil from Laboratory Study Data according to FOCUS Kinetics Guidelines (including the soil metabolites R156172 and R154719) Rifcon GmbH, Heidelberg, Germany, Report R-09049-1, 31 March 2009 Non-GLP, not published Syngenta File No PP5/10009
405749	Watford SP, Francis PD	1986	Residue Levels of Fluazifop in Apples ICI Americas Inc, Goldsboro, NC, USA Report TMU3119/B including supplement, 18 November 1986 GLP, not published Syngenta File No 405749 Syngenta File No 463807
PP5/0378	Watford SP, Francis PD	1986	Fluazifop-P-butyl : Residue Levels of Fluazifop on Dry Beans ICI Amercicas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU3094/B, 7 November 1986 Non-GLP, not published Syngenta File No PP5/0378 (15 pages) Syngenta File No 405710 (14 pages)
405746	Watford SP,	1987	Fluazifop-P-butyl: Fluazifop Residues in Apple Samples (1986)

Code	Author(s)	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP Published/Unpublished
	Francis PD		ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU3291/B, 1 June 1987 GLP, not published Syngenta File No 405746
PP5/0468	Watford SP, Francis PD	1987	Fluazifop-P-butyl: Residues Levels of Fluazifop in Cherries (PP005) 1986 ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU3181/B, 1 March 1987 (46 pages) Report TMU3181/B, 1 March 1987 (43 pages, NMR supplement) Non-GLP, not published Syngenta File No PP5/0468
PP5/0476	Watford SP, Francis PD	1987	Fluazifop-P-butyl: Residue Levels of Fluazifop in Peaches (PP005) 1986 ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU3168/B, 5 March 1987 (41 pages) Report TMU3168/B, 5 March 1987 (38 pages, supplement) Non-GLP, not published Syngenta File No PP5/0476
434208	Watford SP, Francis PD	1987	Fluazifop-P-butyl: Fluazifop Residues in Pecans (1985) ICI Americas INC, Biological Research Center, Goldsboro, NC, USA Project ID TMU3251/B, 21 April 1987 Non-GLP, not published Syngenta File No 434208
PP5/0480	Watford SP, Francis PD	1987	Fluazifop-P-Butyl: Fluazifop Residues in Plum and Prune Samples (1986) ICI Americas INC, Biological Research Center, Goldsboro, NC, USA, Project ID TMU3311/B including supplement, 1 June 1987 (48 pages) Non-GLP, not published Syngenta File No PP5/0480 Syngenta File No 405689
PP5/1113	Watford SP, Francis PD	1987	Fluazifop-P-butyl: Fluazifop-P- butyl Residues in Grape Samples ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU3330/B, 11 September 1987 GLP, not published Syngenta File No PP5/1113
PP5/0471	Watford SP, Francis PD	1987	Fluazifop-P-butyl: Residue Levels of Fluazifop on Grapes (1986) ICI Americas Inc, Goldsboro, NC, USA Report TMU3144/B, 17 March 1987 Not GLP, not published Syngenta File No PP5/0471
PP5/0323	Watford SP, Francis PD	1988	Fluazifop-P-Butyl: Fluazifop and Reference X Residues in Celery (1986) ICI Americas Inc, Biological Research Center, Goldsboro, NC, USA Report TMU3418/B, 30 March 1988 GLP, not published Syngenta File No PP5/0323 **Trials not summarized since the manufacturer did not intend to have MRLs on celery. Addressed in the metabolism section.**
PP5/0102	Weeren RD	1994	Determination of Residues of Total-fluazifop (fluazifop-P-butyl + fluazifop-P) in Potatoes and their Processed Products - Federal Republic of Germany 1993 Dr Specht & Partner Chemische Laboratorien, Hamburg, Germany, Zeneca Agro Project No RS-9307 Report AZ13430/93, Study ICI-9301, 16 May 1994 GLP, not published Syngenta File No PP5/0102 **This report is identical to Pelz, 1994, PP5_50062**
18664401MDC2	Weissenberg L	2013	Determinacao de Residuos de fluazifop total em amostras vegetais por LC/MS/MS Syngenta Protecao de Cultivos Ltda, Sao Paulo, Brazil Report POPIT.MET.138.Rev.08, 21 October 2013 Non-GLP, not published **Study available as appendix in Matarazzo, 2013, A13680D_10051, report M13030 (Portuguese version)** Separate English translation provided: “Determination of fluazifop residues in vegetable samples by LC-MS/MS Standard operating procedure POPIT MET 138 Rev 08”

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			Syngenta File No 18664401MDC2
PP5/1480	Weissenfeld M	2006	Determination of the water solubility of fluazifop-P-butyl RCC Ltd, Itingen, Switzerland, RCC study A65700, sponsor archive 115849, 23 June 2006 GLP, not published Syngenta File No PP5/1480
PP5/1482	Weissenfeld M	2006	Determination of the partition coefficient (n-octanol/water) of fluazifop-P-butyl RCC Ltd, Itingen, Switzerland, RCC study A65698, sponsor archive 115890, 21 July 2006 GLP, not published Syngenta File No PP5/1482
PP5/0811	Wiebe LA	1989	Fusilade® 2000: Field Dissipation Study for Terrestrial Uses, Visalia, California, 1989 ICI Americas Inc, Richmond, CA, USA Report RR 89-066B, Protocol FUSI 89-SD-01, 29 November 1989 GLP, not published Syngenta File No PP5/0811
PP5/0812	Wiebe LA	1989	**Not summarized because study contains no information on CF3-pyridone** Fusilade® 2000: Field Dissipation Study for Terrestrial Uses, Porterville, California, 1989 ICI Americas Inc, Richmond, CA, USA Report RR 89-067B, Protocol FUSI 89-SD-01, 29 November 1989 GLP, not published Syngenta File No PP5/0812
PP5/0777	Wiebe LA	1989	**Not summarized because study contains no information on CF3-pyridone** Determination of fluazifop-P-butyl and fluazifop residues in soil by gas chromatography ICI Americas Inc, Richmond, CA, USA Report RR 89-072B, 30 November 1989 Non-GLP, not published Syngenta File No PP5/0777 Syngenta File No 407581
PP5/0778	Wiebe LA	1990	Determination of 5-(trifluoromethyl)-2(1H)-pyridinone (R-154719) residues in soil by gas chromatography ICI Americas Inc, Richmond, CA, USA Report RR 90-076B, 30 April 1990 Non-GLP, not published Syngenta File No PP5/0778 Syngenta File No 407628
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