FLUOPYRAM (243)

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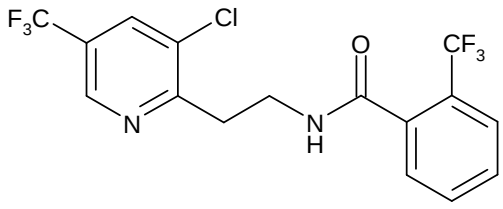
EXPLANATION

Fluopyram, a pyridylethylamide broad spectrum fungicide, is being developed for protection against a range of Ascomycete and Deuteromycete diseases in many horticultural and arable crops. Fungicidal action is by the inhibition of succinate dehydrogenase (complex II) within the fungal mitochondrial respiratory chain, thus blocking electron transport. Fluopyram inhibits spore germination, germ tube elongation, mycelium growth and sporulation. Within plants, fluopyram shows translaminar activity and some movement within the xylem.

Authorisations exist for the use of fluopyram (SC formulation) in China and are currently being progressed in Europe and North America under an OECD Joint Review exercise.

Residue and analytical aspects of fluopyram were considered for the first time by the present meeting. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability, environmental fate in soil and rotational crop residues.

IDENTITY

ISO common name:	Fluopyram
Code number	AE C656948
IUPAC name:	*N*-{2-[3-chloro-5-(trifluoromethyl)-2-pyridyl]ethyl}-α,α,α-trifluoro-*o*-toluamide
Chemical Abstracts name:	*N*-[2-[3-chloro-5-(trifluoromethyl)-2-pyridinyl]ethyl]-2-(trifluoromethyl)benzamide-
CAS number	658066-35-4
CIPAC number	not allocated
Molecular mass:	396.72 g/mol
Molecular formula	$C_{16}H_{11}ClF_6N_2O$
Structural formula:	

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

A detailed chemical and physical characterisation of the active ingredient is given in Table 1.

Table 1 Physical and chemical data of fluopyram, 99.8% pure active ingredient (RCF633-04)

Property	Findings	Reference
Melting point	117.5 °C at 1013 hPa	20070359.01
Boiling point	318–321 °C	20070359.01

Property	Findings	Reference
Temperature of decomposition	decomposition range 300–395 °C (exothermic reaction – 329 J/g)	20070359.01
Relative density	D_4 = 1.53 at 20 °C	PA07/024
Vapour pressure (extrapolated)	1.2 ×10^{-6} Pa for 20 °C 3.1 × 10^{-6} Pa for 25 °C 2.9 × 10^{-4} Pa for 50 °C	20050612.01
Henry's law constant (calculated)	2.98 ×10^{-5}Pa m^3 mol^{-1} at 20 °C (distilled water) 3.17 ×10^{-5}Pa m^3 mol^{-1} at pH 4 2.98 ×10^{-5}Pa m^3 mol^{-1} at pH 7 3.17 ×10^{-5}Pa m^3 mol^{-1} at pH 9	AF06/048
Appearance	white powder, no noticeable odour	PA07/025
Solubility in water	pH 4 15 mg/L at 20 °C pH 7 16 mg/L at 20 °C pH 9 15 mg/L at 20 °C In distilled water: pH 6.7 16 mg/L at 20 °C	PA05/074
Solubility in organic solvents	heptane 0.66 g/L at 20 °C toluene 62.2 g/L at 20 °C dichloromethane > 250 g/L at 20 °C methanol > 250 g/L at 20 °C acetone > 250 g/L at 20 °C ethyl acetate > 250 g/L at 20 °C DMSO > 250 g/L at 20 °C	PA05/113
n-Octanol/water partition coefficient	log P_{ow} 3.3 at pH 6.5 (shake flask method) for NLL7687-2 (99.4% pure fluopyram). Not dependant on pH (within pH range of 2–12) or water solubility	PA06/065 AF08/016
Hydrolysis	> 99% pure radiolabelled fluopyram is stable in sterile buffer solutions (50 °C) at pH 4, 7 and 9. No half-life calculated.	CX/06/015
Phototransformation purity > 99% [Phenyl-UL-^{14}C] radiolabel purity > 98% [Pyridyl-2,6-^{14}C] radiolabel	Degradation products in sterile aqueous 0.02 M phosphate buffer, pH 7 exposed to artificial sunlight equivalent to *ca* 32 days (Phoenix) or ca 50 days (Athens) summer light include numerous minor metabolites and one major rearrangement reaction product – fluopyram-lactame (up to 12.8% applied radiolabel at the end of the 31 day study. Derived DT_{50} (Phoenix AZ) 52-63 days Derived DT_{50} (Athens Greece) 81-97 days	CX/06/016
Dissociation in water	No pKa at environmentally relevant pH pKa = 0.5 at 23 °C at pH 1 (protonation of pyridyl ring)	AF07/048
pH	pH = 6.6 (1% solution in distilled water)	PA05/114

Technical material

Table 2 Physical and chemical data of technical fluopyram, 97.5% active ingredient (PFV064E001)

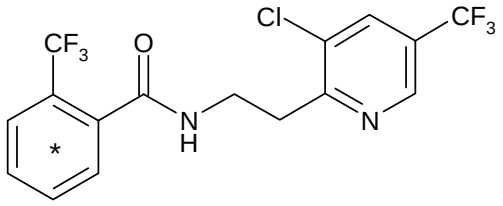
Property	Findings	Reference
Melting point	118 °C at 1013 hPa.	20070286.01
Boiling point	319 °C.	20070286.01
Temperature of decomposition	decomposition range 310–400 °C (exothermic reaction – 285 J/g)	20070286.01
Relative density	D_4 = 1.53 at 20 °C	PA07/024
Appearance	white powder, no noticeable odour	PA07/025

FORMULATIONS

Fluopyram is formulated as a 500 g ai/L suspension concentrate (SC), containing 41.5% or 4.16 lbs fluopyram/US gallon). Combination products with 50:50 mixtures of fluopyram and prothioconazole (200+200 SC), tebuconazole (200+200 SC) or trifloxystrobin (250 + 250 SC) and a 1:3 mixture with prothioconazole (125 + 375 SC) have also been formulated.

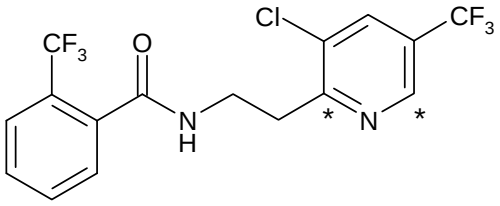
METABOLISM AND ENVIRONMENTAL FATE

The Meeting received fluopyram metabolism studies on animals (rats, lactating goats and laying hens), plants, soil and rotational crops. Fluopyram radiolabelled on the phenyl ring or the pyridyl ring were both used in these studies. The label positions are given below:



* denotes position of radiolabel, uniformly labelled in the phenyl ring

[phenyl-UL-^{14}C]-fluopyram



* denotes position of radiolabel, labelled in the 2- and 6-position of the pyridyl ring

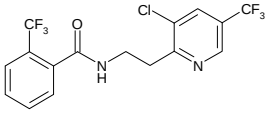
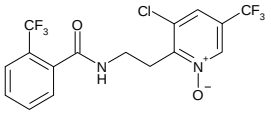
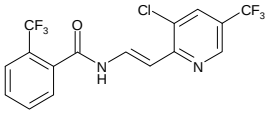
[pyridyl-2,6-^{14}C]-fluopyram

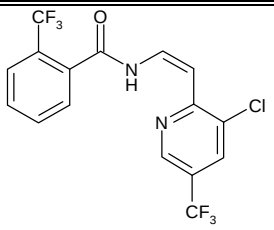
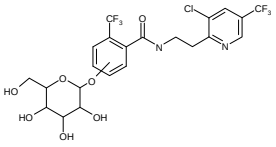
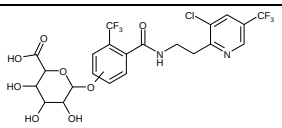
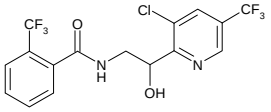
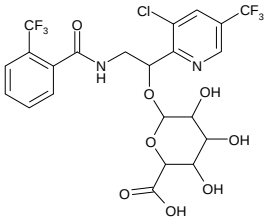
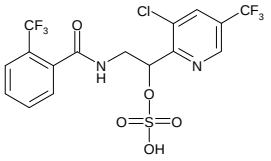
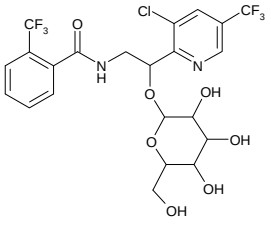
In this evaluation, the values presented in the tables are as reported in the various studies, but in the accompanying text, they have generally been rounded to two significant digits. Abbreviations have also been used for the various fluopyram metabolites mentioned in the study reports. These are:

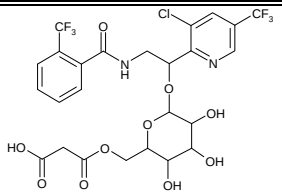
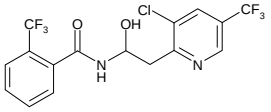
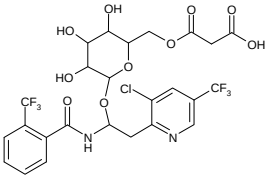
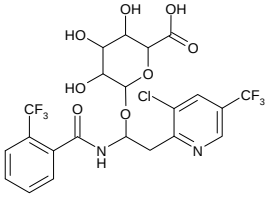
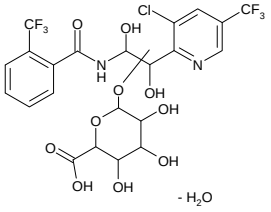
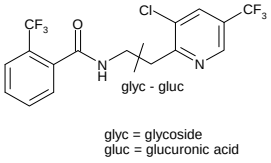
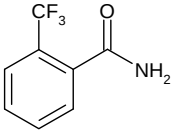
BZM = AE C656948-benzamide
PAA = AE C656948-pyridyl-acetic acid
PCA = AE C656948-carboxylic acid
7-OH = AE C656948-7-hydroxy
8-OH = AE C656948-8-hydroxy
gluc = GA = glucuronic acid
glyc = glycoside
glc = glucoside
MA = malonic acid
SA = sulfate

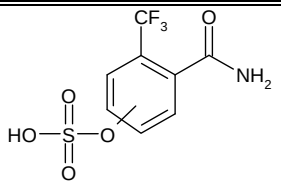
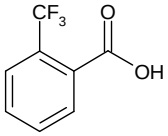
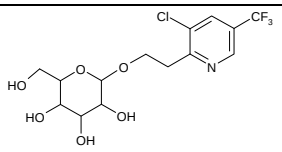
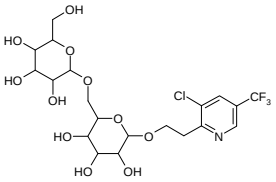
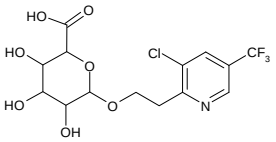
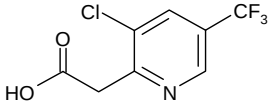
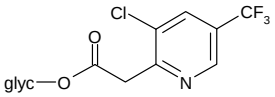
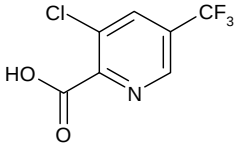
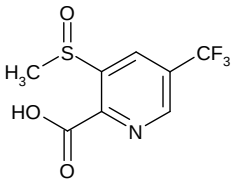
Major metabolites identified in these studies and discussed in this evaluation are listed below.

Table 3 Major fluopyram metabolites identified in plant and animal matrices

Codes	Names		Molecular formula	Occurrence
	Fluopyram AE C656948	*N*-[2-[3-chloro-5-(trifluoromethyl)-2-pyridinyl]ethyl]-2-(trifluoromethyl)benzamide		All matrices
N-oxide (M01)	AE C656948-N-oxide	*N*-{2-[3-chloro-1-oxido-5-(trifluoromethyl) pyridin-2-yl]ethyl}-2-(trifluoromethyl)benzamide		pepper
E-olefine (M02)	AE C656948-E-olefine BCS-AA10627	*N*-{(E)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]}-2-(trifluoromethyl)benzamide		rat goat hen

Codes	Names		Molecular formula	Occurrence
Z-olefine (M03)	AE C656948-Z-olefine BCS-AA10650	*N*-{(Z)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-(trifluoromethyl)benzamide		rat goat hen
phenol-glc (M06)	AE C656948-phenol-gucoside			rat rotational crops
phenol-GA (M07)	AE C656948-phenol-glucoronic acid			rat goat
7-OH (M08)	AE C656948-7-hydroxy BCS-AA10065	*N*-{2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-2-hydroxyethyl}-2-(trifluoromethyl) benzamide		rat goat hen plants rotational crops soil
7-OH-GA (M09)	AE C656948-7-hydroxy-glucuronic acid	1-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-2-{[2-(trifluoromethyl)-benzoyl]amino}ethyl beta-*D*-glucopyranosiduronic acid		rat goat
7-OH-SA (M10)	AE C656948-7-hydroxy-sulfate	1-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-2-({[2-(trifluoromethyl)phenyl]-carbonyl}amino)ethyl hydrogen sulfate		rotational crops
7-OH-glc (M11)	AE C656948-7-hydroxy-glucoside	*N*-[2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-2-(beta-*D*-glucopyranosyloxy)ethyl]-2-(trifluoromethyl) benzamide		grapes beans pepper rotational crops

Codes	Names		Molecular formula	Occurrence
7-OH-glc-MA (M12)	AE C656948-7-hydroxy-glucoside-malonic acid	1-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-2-{[2-(trifluoromethyl)-benzoyl]amino}ethyl 6-*O*-(carboxyacetyl)-beta-*D*-glucopyranoside		beans pepper rotational crops
8-OH (M18)	AE C656948-8-hydroxy	*N*-{2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-1-hydroxyethyl}-2-(trifluoromethyl) benzamide		rat goat plants rotational crops
8-OH-glc-MA (M19)	AE C656948-8-hydroxy-gucoside-malonic acid	1-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-1-{[2-(trifluoromethyl)-benzoyl]amino}ethyl 6-*O*-(carboxyacetyl)-beta-*D*-glucopyranoside		rotational crops
8-OH-GA (M20)	AE C656948-8-hydroxy-glucuronic acid	2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-1-{[2-(trifluoromethyl)-benzoyl]amino}ethyl beta-*D*-glucopyranosiduronic acid		rat goat
di-OH-GA (M21)	AE C656948-di-hydroxy-glucuronic acid			rat goat
8-OH-glyc-gluc (M22)	AE C656948-hydroxy-glycoside-glucuronic acid			beans
benzamide (M25)	AE C656948-benzamide AE F148815 BCS-AA10014	2-(trifluoromethyl)-benzamide		rat goat hen plants rotational crops aerobic soil

Codes	Names		Molecular formula	Occurrence
benzamide-SA (M31)	AE C656948-benzamide-sulfate			rat goat
benzoic acid (M33)	AE C656948-benzoic acid	2-(trifluoromethyl)-benzoic acid		rat hen rotational crops
hydroxyethyl-glc M35	AE C656948-hydroxyethyl-glucoside	2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethyl beta-*D*-glucopyranoside		beans
hydroxyethyl-di-glc (M36)	AE C656948-hydroxyethyl-di-glucoside	2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethyl 6-*O*-beta-*D*-glucopyranosyl-beta-D-glucopyranoside		pepper
hydroxyethyl-GA (M37)	AE C656948-hydroxyethyl-glucuronic acid	2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethyl beta-*D*-glucopyranosiduronic acid		rat goat
pyridyl-acetic acid (PAA) (M40)	AE C656948-pyridyl-acetic acid (PAA)	[3-chloro-5-(trifluoromethyl)pyridin-2-yl]acetic acid		rat goat hen beans pepper
PAA-glycoside (M42)	AE C656948-pyridyl-acetic acid -glycoside			pepper
pyridyl-carboxylic acid (PCA) (M43)	AE C656948-pyridyl-carboxylic acid (PCA)	3-chloro-5-(trifluoromethyl)pyridine-2-carboxylic acid		rat plants rotational crops aerobic soil
methyl-sulfoxide (M45)	AE C656948-methyl-sulfoxide	3-(methylsulfinyl)-5-(trifluoromethyl) pyridine-2-carboxylic acid		rotational crops aerobic soil

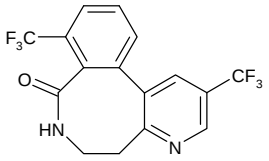
Codes	Names		Molecular formula	Occurrence
lactame	AE C656948-lactame	2,9-bis(trifluoromethyl)-6,7-dihydropyrido[2,3-e][2]benzazocin-8(5H)-one		Photolysis (aqueous buffer)

Table 4 Other major fluopyram metabolites identified only in rat matrices

Codes		Name	
M04	enol-GA	AE C656948-enol-glucuronic acid	
M13	7-OH-phenol	AE C656948-7-hydroxy-phenol	
M14	7-OH-phenol-GA	AE C656948-7-hydroxy-phenol-glucuronic acid	
M15	7-OH-phenol-SA	AE C656948-7-hydroxy-phenol-sulfate	
M16	7-OH-methyl-sulfone	AE C656948-7-hydroxy-methyl-sulfone	
M17	7-OH-hydroxy-phenol-SA	AE C656948-7-hydroxy-hydroxy-phenol-sulfate	
M23	methoxy-di-OH-GA	AE C656948-methoxy-di-hydroxy-glucuronic acid	
M24	benzoyl-serine	AE C656948-benzoyl-serine	*N*-acetyl-*O*-[2-(trifluoromethyl)benzoyl]-serine
M26	hydroxy-benzamide	AE C656948-hydroxy-benzamide	
M27	benzamide-OH-GA	AE C656948-benzamide-hydroxy-glucuronic acid	
M28	benzamide-cysteine	AE C656948-benzamide-cysteine	
M29	BA-methyl-sulfoxide	AE C656948-benzamide-methyl-sulfoxide	
M30	BA-methyl-sulfone	AE C656948-benzamide-methyl-sulfone	
M32	benzamide-N,O-GA	AE C656948-benzamide-N,O-glucuronic acid	1-*O*-{[2-(trifluoromethyl)-benzoyl]amino}-beta-*D*-glucopyranuronic acid
M38	ethyl-diol	AE C656948-ethyl-diol	1-[3-chloro-5-(trifluoromethyl)pyridin-2-yl] ethane-1,2-diol
M39	ethyl-diol-GA	AE C656948-ethyl-diol-glucuronic acid	
M41	hydroxy-PAA	AE C656948-hydroxy-pyridyl-acetic acid	[3-chloro-5-(trifluoromethyl)pyridin-2-yl](hydroxy)acetic acid
M44	pyridyl-methyl-cystein	AE C656948-pyridyl-methyl-cystein	*N*-acetyl-*S*-{[3-chloro-5-(trifluoromethyl) pyridin-2-yl]methyl}cysteine

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens, following oral dosing with [phenyl-UL-^{14}C]-fluopyram and [pyridyl-2,6-^{14}C]-fluopyram.

Rats

Metabolism studies in rats were reviewed by the JMPR at this Meeting, where it was concluded that Fluopyram absorption was rapid and residues in tissues at 168 hr accounted for < 0.5% (pyridyl ring-label) or 3–5% (phenyl ring-label) of the administered dose, with liver and kidney containing the highest concentrations of residues. Elimination of the radiolabel was via the faeces (39–64%) and the urine (35–60%).

The metabolism of fluopyram in rats was principally oxidative and took place mainly at the ethylene bridge of the molecule. Also, hydrolytic cleavage of the molecule and subsequent oxidation was observed, as was conjugation of several hydroxylated metabolites with glucuronic acid and to a lesser extent with sulphate.

Lactating goat

In two metabolism studies reported by Weber & Koester, 2008 [Ref: MEF-06/519][Ref: MEF-06/327], single 32 month old lactating goat (35 and 42 kg) was orally dosed five times at 24 hour intervals with 1.91 mg [phenyl-UL-^{14}C]-fluopyram or 2 mg/kg [pyridyl-2,6-^{14}C]-fluopyram per kg bw per day (equivalent to 46.26 ppm and 44.6 ppm respectively in the diet) and sacrificed about 24 hours after the last dose. Total radioactivity was measured in milk, plasma and excreta at timed sampling intervals, and in the edible organs and tissues liver, kidney, muscle and fat at sacrifice. The milk (1.2–1.7 kg/day), edible organs and tissues and excreta (urine and faeces) were analysed for parent compound and metabolites.

Samples were double or triple-extracted with acetonitrile/water mixtures and purified using reversed phase SPE cartridge clean-up. In the phenyl-label study, for the muscle, liver, and kidney samples, additional solvent extraction steps were performed with acetonitrile/water under microwave assistance at 120 °C. Extraction efficiencies ranged from 94–99% of the TRR.

In the pyridyl-label study, extraction efficiencies for all matrices except liver ranged from 89-97% of the TRR. For liver the initial solvent extraction was supplemented by further extraction steps involving microwave assistance to give a combined extraction efficiency of about 76%. A second liver extraction involving enzymatic digestion achieved 44% efficiency and solvent extraction of the residual solids achieved a further 36% efficiency (total 80%).

Extracts were analysed for parent compound and metabolites by reversed phase HPLC using ^{14}C-flow-through detection as the primary analytical method. Samples were kept frozen at ≤ -18 °C for up to 4 months (6 weeks in the pyridyl-label study) before extraction and analysis. A comparison of the extraction efficiencies and residue profiles in the initial samples stored for up to 6 weeks with those from samples stored for up to 4 months showed no significant differences. Limits of determination, at about 2.5 × background level, were from 0.001–0.009 mg/kg except for the phenyl-labelled liver extracts where the determination limit was 0.046 mg/kg.

In the phenyl-label study, the overall recovery (sum of radioactivity in the excreta, milk as well as organs and tissues) was 93.5% of the total administered dose, mostly in urine (53%) and faeces (36%). The residual balance of 6.5% at sacrifice may have been present in the gastro-intestinal tract.

The TRR in plasma and milk, determined over the whole testing period in timed sampling intervals increased continuously without reaching a plateau, suggesting ongoing absorption, rapid distribution and delayed excretion. The equivalent concentrations in milk samples ranged from 0.045 mg/kg (8 hours after the first dose) to 0.454 mg/kg (24 hours after the last of 5 doses), with a cumulative total representing 0.56% of the total applied dose.

The highest TRR was determined in the liver (8.38 mg/kg) with residues of 2.3 mg/kg in kidneys, 0.74 mg/kg in muscle and 0.4 mg/kg.

Table 5 Total radioactive residues in dissected organs and tissues, excreta and milk of the lactating goat following 5 daily oral administrations of [phenyl-UL-^{14}C]-fluopyram

Matrix	% administered dose	TRR (mg/kg)
Urine, 0-120 h	52.62	29.717
Faeces, 0-120 h	35.69	7.258
Total excreted	88.31	-
Liver	1.71	8.379
Kidney	0.07	2.295

Matrix	% administered dose	TRR (mg/kg)
Total body muscle	2.31	0.737
Total body fat	0.50	0.399
Total of organs/tissues	4.58	-
Milk 1 hr (dose 1)		-
Milk 8 hr		0.045
Milk 24 hr (dose 2)		0.091
Milk 32 hr		0.170
Milk 48 hr (dose 3)		0.223
Milk 56 hr		0.257
Milk 72 hr (dose 4)		0.290
Milk 80 hr		0.317
Milk 96 hr (dose 5)		0.384
Milk 104 hr		0.420
Milk 120 hr		0.454
Milk, 0-120 h	0.56	0.259 (mean)
Total	93.46	-

In the phenyl-label study, the major residue in milk was the BZM metabolite, present at 88-89% TRR, with no other components present at levels higher than 4.3% TRR. BZM was also the major metabolite in muscle (97.6% TRR), liver (83% TRR) and kidney (77% TRR). In fat, the major components were BZM (49% TRR), the parent fluopyram (18% TRR) and the Z-olefine (13% TRR). Residues of fluopyram and other identified metabolites were found in various tissues at less than 10% TRR.

Table 6 TRR levels and distribution of parent compound and metabolites in edible tissues of the goat after application of [phenyl-UL-^{14}C]-fluopyram

	morning milk	evening milk	muscle	fat	liver	kidney
TRR [ppm] =	0.276	0.228	0.737	0.399	8.379	2.295
Compound	% TRR	% TRR	% TRR	% TRR	% TRR	% TRR
Total analysed	96.8	97.9	98.9	96.7	93.5	97.7
Fluopyram (parent)	0.7	1.7	---	18.2	0.6	0.4
-benzamide-SA	4.3	4.3	0.7	---	1.6	2.5
-BZM	89.2	88.4	97.6	49.1	82.8	77.1
-7-OH-GA (isomer 1)	0.4	0.3	0.3	---	4.3	7.3
-7-OH-GA (isomer 2)	0.2	0.3	---	---	---	2.1
-di-OH-GA	---	---	---	---	---	0.7
-phenol-GA	---	---	---	---	0.8	1.2
-8-OH-GA (isomer 2)	---	---	---	---	2.1	3.6
-7-OH	0.8	1.8	0.3	7.7	0.9	0.6
-E-olefine	---	---	---	8.6	0.3	---
-Z-olefine	0.7	---	---	13.1	0.2	---
Total identified	96.3	96.8	98.9	96.7	93.5	95.4
Total characterised [a]	0.5	1.1	---	---	---	2.2
Losses [b]	0.6	1.0	0.2	2.5	0.1	---
Volatiles [c]	2.2	0.5	0.3	---	0.4	0.7
Total extracted [d]	99.5	99.4	99.4	99.2	94.0	98.4
Total bound (PES)	0.5	0.6	0.6	0.8	6.0	1.6
Accountability	100.0	100.0	100.0	100.0	100.0	100.0

[a] compounds were at least characterised by extraction and chromatographic behaviour (HPLC)

[b] dichloromethane/methanol eluates of SPE during clean up (SPE eluate and heptane phase for fat)

[c] distillates from concentration steps

[d] Total extracted for fat = sum of solvent extract (96.9% of TRR) and heptane phase (2.3% of TRR)

The metabolic reactions of [phenyl-UL-^{14}C]-fluopyram identified in the lactating goat involve hydroxylation of the ethylene bridge of the molecule resulting in 7-OH, 8-OH and a dihydroxylated compound, hydroxylation of the phenyl ring leading to the phenol metabolite, conjugation of the hydroxylated metabolites with glucuronic acid, elimination of water from compounds hydroxylated in the ethylene bridge, leading to the Z-olefine and E-olefine isomers (and able to isomerise into each other), cleavage of the aliphatic chain to form BZM and hydroxylation of BZM followed by conjugation with sulphate.

In the pyridyl-label study, the overall recovery (sum of radioactivity in the excreta, milk as well as organs and tissues) was 82% of the total administered dose, mostly in urine (52%) and faeces (29%). The residual balance (18%) at sacrifice may have been present in the gastro-intestinal tract.

The TRR levels in plasma and milk, determined over the whole testing period in timed sampling intervals followed a diurnal course, increasing during the 8 hour period after each dosing and then decreasing, suggesting rapid absorption, distribution and elimination. TRR levels reached plateau-levels after about 50–60 hours in plasma and within 24 hours in milk. The highest TRR-value in milk (0.063 mg/kg) was detected 32 hours after the first dose, with a cumulative total TRR of 0.08% of the total applied dose.

The highest TRR was found in liver (1.43 mg/kg) with 0.4 mg/kg reported in kidneys, 0.37 mg/kg in fat and 0.042 mg/kg in muscle. The combined TRR in these tissue samples was about 0.85% of the total applied dose.

Table 7 Total radioactive residues in dissected organs and tissues, excreta and milk of the lactating goat following 5 daily oral administrations of [pyridyl-2,6-^{14}C]-fluopyram

Matrix	% administered dose	TRR (mg/kg)
Urine, 0-120 h	52.33	13.682
Faeces, 0-120 h	28.62	5.444
Total excreted	80.95	-
Liver	0.31	1.427
Kidney	0.01	0.403
Total body muscle	0.12	0.042
Total body fat	0.42	0.372
Total of organs/tissues	0.85	-
Milk 1 hr (dose 1)		-
Milk 8 hr		0.051
Milk 24 hr (dose 2)		0.017
Milk 32 hr		0.063
Milk 48 hr (dose 3)		0.02
Milk 56 hr		0.052
Milk 72 hr (dose 4)		0.018
Milk 80 hr		0.048
Milk 96 hr (dose 5)		0.021
Milk 104 hr		0.055
Milk 120 hr		0.026
Milk, 0-120 h	0.08	0.032 (mean)
Total	81.89	-

In the pyridyl-label study, the parent compound was the major component in fat (46% TRR), milk (43% TRR) and muscle (27% TRR) but was only a minor residue in liver (7.7% TRR) and was not found in kidney. The Z-olefine metabolite was the major metabolite in milk (13% TRR), muscle (22% TRR) and fat (34% TRR), but was a minor residue (5.7% TRR) in liver and was not found in kidney. The 7-OH metabolite was also a major component of milk (16% TRR), muscle (22% TRR) and fat (13% TRR).

Other major identified metabolites included the 7-OH glucuronic acid (isomer 1) at 24% TRR in liver and 35% TRR in kidney; the 7-OH glucuronic acid (isomer 2) at 16% TRR in kidney and the 8-OH glucuronic acid (isomer 2) at 18% TRR in kidney. Residues of these and other identified metabolites were found in various tissues at less than 10% TRR.

Table 8 TRR levels and distribution of parent compound and metabolites in edible tissues of the goat after application of [pyridyl-2,6-^{14}C]-fluopyram

	Morning milk	muscle	fat	liver	kidney
TRR [ppm] =	0.053	0.042	0.372	1.427	0.403
Compound	% TRR	% TRR	% TRR	% TRR	% TRR
Fluopyram (parent)	42.5	27.3	46.4	7.7	---
- PAA	---	---	---	---	8.6
-hydroxyethyl-GA	---	---	---	---	4.3
-7-OH-GA (isomer 1)	1.7	6.6	---	24.2	35.1
-7-OH-GA (isomer 2)	3.1	5.1	---	2.1	16.3
-di-OH-GA	---	---	---	1.0	1.7
-phenol-GA	---	---	---	2.0	3.2
-8-OH-GA (isomer 2)	1.1	2.5	---	9.5	17.7
-7-OH	16.2	21.6	12.8	6.1	1.0
-E-olefine	2.0	1.9	2.2	4.1	---
-Z-olefine	12.9	21.6	33.7	5.7	---
Total identified	79.4	86.7	95.1	62.4	87.9
Total characterised [a]	11.6	---	---	2.4	1.0
Losses [b]	2.4	---	1.1	1.0	0.5
Volatiles [c]	---	---	1.0	3.7	2.0
Not analysed	---	2.5	---	6.4	---
Total extracted [d]	93.4	89.2	97.1	75.8	91.4
Post extraction solids	6.6	10.8	2.9	24.2	8.6
Accountability	100.0	100.0	100.0	100.0	100.0

[a] compounds were at least characterised by extraction and chromatographic behaviour (HPLC)

[b] dichloromethane/methanol eluates of SPE column during clean up

[c] distillates from concentration steps

[d] muscle: by solvent extraction; liver: by solvent extraction and first microwave extraction

The metabolic reactions of [pyridyl-2,6-^{14}C]fluopyram identified in the lactating goat involve hydroxylation of the ethylene bridge of the molecule resulting in 7-OH, 8-OH metabolites and a dihydroxylated compound, hydroxylation of the phenyl ring (leading to the phenol), conjugation of the hydroxylated metabolites with glucuronic acid, elimination of water from compounds hydroxylated in the ethylene bridge leading to the Z-olefine and the E-olefines, molecular cleavage to the pyridyl-hydroxyethyl metabolite followed by conjugation with glucuronic acid and oxidation of the pyridyl-hydroxyethyl to PAA.

In conclusion, when the two metabolism studies are combined using appropriate scaling to obtain an overall picture of the metabolism, the scaled results indicated that residues of BZM represented 98% of the TRR in muscle, 89% TRR in milk, 81% TRR in liver, 73% TRR in kidney and

48% in fat. The Z- and E-olefine isomers made up about 13% and 8% TRR in fat respectively. Residues of the parent compound were about 18% TRR in fat and were less than 1% TRR in all other matrices. Other metabolites were present at less than 10% in any matrix.

The proposed metabolic pathway in goat includes hydroxylation of the parent compound to the 7-OH and 8-OH metabolites, the formation of the Z/E-olefines, cleavage of the fluopyram molecule to produce BZM (from the phenyl-label) and PAA (from the pyridyl-label) and conjugation of the hydroxylated parent compound, mainly with glucuronic acid.

Table 9 Relative residue distribution of fluopyram and metabolites in goat milk and fat, 24 hours after 5 daily oral doses of 45 ppm fluopyram.

	morning milk				fat			
Radio-label	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]
Compound	mg/kg	mg/kg	mg/kg	%	mg/kg	mg/kg	mg/kg	%
Fluopyram (parent)	0.002	0.023	0.002	0.73	0.073	0.173	0.073	17.9
-benzamide-SA	0.012			4.35	--			--
-BZM	0.246			89.2	0.196			48.2
-7-OH-GA (isomer 1)	0.001	0.001	0.0001	0.36	--	--	--	--
-7-OH-GA (isomer 2)	0.001	0.002	0.0002	0.36	--	--	--	--
-di-OH-GA	--	--	--	--	--	--	--	--
-phenol-GA	--	--	--	--	--	--	--	--
-8-OH-GA (isomer 2)	--	0.001	0.0001	0.00	--	--	--	--
-7-OH	0.002	0.009	0.0008	0.36	0.031	0.048	0.0203	7.62
-E-olefine	--	0.001	0.0001	0.00	0.034	0.008	0.0034	8.35
-Z-olefine	0.002	0.007	0.0006	0.73	0.052	0.125	0.0527	12.8
-PAA		--	--	0.00		--	--	--
- hydroxyethyl-GA		--	--	0.00		--	--	--
Total characterised	0.001	0.006	0.0005	0.55	--	--	--	--
Losses and volatiles	0.008	0.001	--	2.9	0.01	0.008	0.0034	3.29
Bound (PES)	0.001	0.003	0.0003	0.46	0.003	0.011	0.0046	1.88
Total (label specific)	0.275 (A)	0.01	0.0008 (B)	100	0.399 (A)	0.019	0.008 (B)	100

[a] scaling factor =phenyl-labelled parent residues/pyridyl-labelled parent residues

[b] percentage of the total label-specific (adjusted) residues (i.e., A+B)

Table 10 Relative residue distribution of fluopyram and significant metabolites in goat liver and kidney, 24 hours after 5 daily oral doses of 45 ppm fluopyram

	liver				kidney			
Radio-label	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]
Compound	mg/kg	mg/kg	mg/kg	%	mg/kg	mg/kg	mg/kg	%
Fluopyram (parent)	0.047	0.11	0.047	0.55	0.009	--	--	0.373
-benzamide-SA	0.133			1.55	0.058			2.4
-BZM	6.94			81	1.769			73.3
-7-OH-GA (isomer 1)	0.363	0.345	0.1474	4.24	0.168	0.142	0.168	6.96
-7-OH-GA (isomer 2)	--	0.03	0.0128	0.0	0.048	0.066	0.0781	1.99

	liver				kidney			
Radio-label	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]
Compound	mg/kg	mg/kg	mg/kg	%	mg/kg	mg/kg	mg/kg	%
-di-OH-GA	--	0.014	0.006	0.0	0.015	0.007	0.0083	0.62
-phenol-GA	0.068	0.029	0.0124	0.79	0.027	0.013	0.0154	1.12
-8-OH-GA (isomer 2)	0.174	0.136	0.0581	2.03	0.082	0.071	0.084	3.4
-7-OH	0.073	0.086	0.0367	0.85	0.014	0.004	0.0047	0.58
-E-olefine	0.021	0.058	0.0248	0.25	--	--	--	0.0
-Z-olefine	0.015	0.081	0.081	0.18	--	--	--	0.0
-PAA		--	--	0.0		0.035	0.0414	1.72
- hydroxyethyl-GA		--	--	0.0		0.017	0.0201	0.83
Total characterised	--	0.035	0.015	0.17	0.051	0.004	0.0047	2.31
Losses and volatiles	0.039	0.066	0.0282	0.78	0.016	0.01	0.0118	1.15
Bound (PES)	0.507	0.346	0.1478	7.64	0.037	0.035	0.0414	3.25
Total (label specific)	8.381 (A)	0.447	0.191 (B)	100	2.294 (A)	0.101	0.1195 (B)	100

[a] scaling factor =phenyl-labelled parent residues/pyridyl-labelled parent residues

[b] percentage of the total label-specific (adjusted) residues (i.e., A+B)

Table 11 Relative residue distribution of fluopyram and significant metabolites in goat muscle, 24 hours after 5 daily oral doses of 45 ppm fluopyram.

	muscle			
Radio-label	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]
Compound	mg/kg	mg/kg	mg/kg	%
Fluopyram (parent)	--	0.011	0.0024	0.0
-benzamide-SA	0.005			0.678
-BZM	0.719			97.5
-7-OH-GA (isomer 1)	0.002	0.003	0.0007	0.271
-7-OH-GA (isomer 2)	--	0.002	0.0004	0.0
-di-OH-GA	--	--	--	0.0
-phenol-GA	--	--	--	0.0
-8-OH-GA (isomer 2)	--	0.001	0.0002	0.0
-7-OH	0.002	0.009	0.002	0.271
-E-olefine	--	0.001	0.0002	0.0
-Z-olefine	--	0.009	0.002	0.0
-PAA		--	--	0.0
- hydroxyethyl-GA		--	--	0.0
Total characterised	--	--	--	0.0
Losses and volatiles	0.004	--	--	0.543
Bound (PES)	0.004	0.005	0.0011	0.693
Total (label specific)	0.736 (A)	0.005	0.0011 (B)	100

[a] scaling factor =phenyl-labelled parent residues/pyridyl-labelled parent residues

[b] percentage of the total label-specific (adjusted) residues (i.e., A+B)

Laying hens

In metabolism studies reported by Justus & Koester, 2008 [Ref: MEF-06/329][Ref: MEF-06/405], six 24 week-old White Leghorn hens were orally dosed 14 times at 24 hour intervals with either [phenyl-UL-^{14}C]-fluopyram or [pyridyl-2,6-^{14}C]-fluopyram at 2.0 mg per kg body weight per day (equivalent to about 26 ppm in the feed) and sacrificed about 24 hours after the last dose. The dose was tolerated without any observable toxicological effects. Total radioactivity (TRR) was measured as fluopyram equivalents in the excreta and eggs collected daily, as well as in the dissected tissues: muscle, fat, liver and skin collected at sacrifice (24 hours after the last dose). The eggs, tissues and excreta were analysed for parent compound and metabolites.

Samples were extracted with acetonitrile/water, purified by reversed phase SPE cartridge clean-up, the radioactivity was measured by combustion and/or liquid scintillation counting and analysed by reversed phase HPLC using ^{14}C-flow-through detection. Identification was by co-chromatography with reference compounds using HPLC and TLC chromatography and comparison of retention times and elution patterns. Extracts after purification represented between 92% and 99% of the TRR in the samples. Limits of determination, at about 2.5×background level, were from 0.001-0.002 mg/kg.

In the pyridyl-label study, fat was additionally extracted with n-heptane and after purification the resulting extracts of eggs, muscle, fat and excreta represented between 28% and 95% of the total radioactive residue (TRR). In addition, egg pools and liver samples from the pyridyl-label study were extracted after enzymatic digestion to solubilize more of the radioactive residues. Residual solids from both extraction procedures were further extracted with microwave assistance. This alternative extraction method resulted in extraction efficiencies of 99% in eggs and 90% in liver.

Samples were kept frozen at ≤ -18 °C for up to 18 weeks (6 months in the pyridyl-label study) before extraction and analysis. A comparison of the first chromatogram of a sample matrix (used for quantification) with later chromatograms from samples stored for up to 12 months showed no changes in the metabolic profile of each matrix.

In the phenyl-label study, the overall radioactive recovery from the excreta, eggs as well as organs and tissues was 94.8% of the applied dose, the majority of which (83% of the applied dose) was detected in the excreta collected before sacrifice and the balance (5%) may have remained in the gastro-intestinal tract. The excretion rate was relatively constant over the 14-day study period. About 4.3% of the applied dose was detected in the eggs and at sacrifice, 5% of the applied dose was measured in muscle and about 3% in other edible organs and tissues.

The TRR levels in eggs increased from 0.46 (day 1) to 3.2 mg/kg after 7 days and then leveled out to reach a maximum of 3.9 mg/kg (day 14). The residue-level in the eggs collected from the ovary and oviduct (5.77 mg/kg) was about 1.5 times higher compared to the laid eggs at the test end (3.9 mg/kg), suggesting that the egg yolk was probably the preferred site for the secretion.

The highest TRR levels were measured in liver (9.54 mg/kg, 0.86% applied dose) and kidney (5.76 mg/kg, 0.15% applied dose). Lower residues were found in muscle (3.29 mg/kg), skin (2.53 mg/kg), and subcutaneous fat (1.7 mg/kg). The residues in muscle corresponded to about 5% of the total dose assuming a value of 40% of the body weight for this tissue. Assuming a value of 12% and 4% of the body weight for fat and skin, respectively, the compound-related residue in both tissues accounted for a total of about 1% of the total applied dose.

Table 12 Mean total recovered radioactivity and TRR levels in excreta, eggs, organs and tissues from laying hen following 14 daily oral administrations of [phenyl-UL-^{14}C]-fluopyram

Matrix	% administered dose*	TRR (mg/kg)
Excreta day 1 – 14	82.67	10.66
Liver	0.86	9.54
Kidney	0.15	5.76
Eggs from ovary/oviduct	0.73	5.77

Matrix	% administered dose*	TRR (mg/kg)
Total body muscle	4.94	3.29
Total body skin	0.38	2.53
Total body fat	0.76	1.7
Total of organs/tissues	7.83	-
Eggs day 0 (1 dose)	not collected	-
Eggs day 1 (2 doses)	0.051	0.46
Eggs day 2 (3 doses)	0.166	1.02
Eggs day 3 (4 doses)	0.319	1.65
Eggs day 4 (5 doses)	0.547	2.09
Eggs day 5 (6 doses)	0.817	2.45
Eggs day 6 (7 doses)	1.137	2.87
Eggs day 7 (8 doses)	1.438	3.24
Eggs day 8 (9 doses)	1.795	3.22
Eggs day 9 (10 doses)	2.179	3.38
Eggs day 10 (11 doses)	2.590	3.57
Eggs day 11 (12 doses)	3.014	3.72
Eggs day 12 (13 doses)	3.452	3.8
Eggs day 13 (14 doses)	3.89	3.83
Eggs day 14	4.335	3.9
Eggs day 1 – 14	4.34	2.87 (overall mean)
Total	94.83	-

* cumulative values expressed as percentage of total administered dose

In the phenyl-label study, the major component in all the edible matrices was the benzamide metabolite (BZM) at 69% to 99% of the TRR. Other metabolites identified were the Z-olefine (26% in fat, up to 1.2% in eggs and 0.5% in muscle), the E-olefine (2.3% in fat) and the benzoic acid (0.3% in liver). Parent compound was only detected in eggs (up to 1.4% TRR) and in fat (2.5% TRR).

Table 13 TRR levels and distribution of parent compound and metabolites in edible tissues of the hen after application of [phenyl-UL-^{14}C]-fluopyram

	egg pool day 1–6	egg pool day 7–14	muscle	fat	liver
TRR [ppm]	1.81	3.58	3.28	1.641	9.466
Compound	% TRR	% TRR	% TRR	% TRR	% TRR
Fluopyram (parent)	1.4	0.7	---	2.5	---
-benzoic acid	---	---	---	---	0.3
-BZM	95.8	96.3	98.6	68.6	92.3
-E-olefine	---	---	---	2.3	0.3
-Z-olefine	0.5	1.2	0.5	25.9	0.2
Total identified	97.7	98.2	99.1	99.3	93.0
Analysed extract(s)	97.7	98.2	99.1	99.3	93.0
Volatiles in distillates	1.1	0.3	0.5	---	0.5
Extracts not analysed	0.1	0.1	0.0	0.1	0.1
Total extractable	98.9	98.6	99.6	99.4	93.6
Unextractable (solids)	1.1	1.4	0.4	0.6	6.4
Accountability	100.0	100.0	100.0	100.0	100.0

The principal metabolic reactions of [phenyl-UL-^{14}C]-fluopyram in the laying hen involve the cleavage of the aliphatic chain, yielding BZM as the major metabolite. A further metabolic reaction was the hydroxylation of the aliphatic chain followed by elimination of H_2O, yielding the two olefin isomers. Hydrolysis of BZM to the pyridyl-carboxylic acid (PCA) metabolite was observed as a minor reaction.

In the pyridyl-label study, the overall recovery (sum of radioactivity in the excreta, eggs as well as organs and tissues) was 96% of the total administered dose, the majority (95%) being measured in the excreta collected before sacrifice and the balance (4.4%) may have remained in the gastro-intestinal tract. The excretion was high and started immediately after the first administration and was relatively constant throughout the test period. About 0.36% of the total applied radio-label was detected in the eggs and at sacrifice the compound-related residues in the edible organs and tissues collected from the hens amounted to 0.48% of the total dose, about a half (0.22%) being in the fatty tissue.

The TRR levels in eggs increased from 0.05 mg/kg (day 1) to 0.32 mg/kg (day 8) when a residue plateau-level was reached. The residue-level in the eggs collected from the ovary and oviduct (0.83 mg/kg) was about 3x higher compared to the laid eggs at the test end (0.26 mg/kg), suggesting that the egg yolk was probably the preferred site for the secretion.

The highest TRR levels were found in liver (0.54 mg/kg) and subcutaneous fat (0.5 mg/kg) with levels of 0.24 mg/kg in kidney and 0.15 mg/kg in skin. Assuming a value of 12% and 4% of the body weight for fat and skin, respectively, the compound-related residue in both tissues accounted for 0.24% of the radioactivity total dose administered. The lowest TRR was measured in the muscle (0.05 mg/kg) which corresponded to 0.07% of the total dose assuming a value of 40% of body weight for this tissue.

Table 14 Mean total recovered radioactivity and TRR levels in excreta, eggs, organs and tissues from laying hen following 14 daily oral administrations of [pyridyl-2,6-^{14}C]-fluopyram

Matrix	% administered dose*	TRR (mg/kg)
Excreta day 1 – 14	94.71	12.642
Liver	0.05	0.538
Kidney	0.01	0.242
Eggs from ovary/oviduct	0.10	0.831
Total body muscle	0.07	0.048
Total body skin	0.02	0.152
Total body fat	0.22	0.498
Total of organs/tissues	0.48	-
Eggs day 0 (1 dose)	not collected	- **
Eggs day 1 (2 doses)	0.005	0.047
Eggs day 2 (3 doses)	0.013	0.093
Eggs day 3 (4 doses)	0.03	0.154
Eggs day 4 (5 doses)	0.047	0.177
Eggs day 5 (6 doses)	0.072	0.223
Eggs day 6 (7 doses)	0.093	0.272
Eggs day 7 (8 doses)	0.123	0.284
Eggs day 8 (9 doses)	0.158	0.321
Eggs day 9 (10 doses)	0.185	0.304
Eggs day 10 (11 doses)	0.216	0.293
Eggs day 11 (12 doses)	0.247	0.289
Eggs day 12 (13 doses)	0.279	0.282
Eggs day 13 (14 doses)	0.306	0.262
Eggs day 14	0.356	0.262

Matrix	% administered dose*	TRR (mg/kg)
Eggs day 1 – 14	0.36	0.235
Total	95.55	-

* cumulative values expressed as percentage of total administered dose

In the pyridyl-label study, the major component in all the edible matrices was the Z-olefine metabolite which accounted for about 70% of the TRR in the fat, 33% of the TRR in muscle and up to 19% in eggs. The E-olefine metabolite was found at up to 14% TRR in liver and 12% in fat. Parent compound was detected at significant levels in eggs (up to 18% TRR) and fat (12% TRR). The pyridyl-acetic acid (PAA) and the 7-hydroxy (7-OH) metabolites were minor components, present at 1-6% of the TRR.

Table 15 TRR levels and distribution of parent compound and metabolites in eggs after application of [pyridyl-2,6-^{14}C]-fluopyram to laying hens

	egg pool day 1 – 6, conventional extraction	egg pool day 1 – 6, enzymatic treatment	egg pool day 7 – 14, conventional extraction	egg pool day 7 – 14, enzymatic treatment
TRR [ppm]	0.156	0.156	0.286	0.286
Compound	% TRR	% TRR	% TRR	% TRR
Fluopyram	14.7	17.9	6.0	9.5
-PAA	6.4	---	3.7	---
-7-OH	3.9	5.8	0.9	1.6
-E-olefine	1.0	---	1.0	1.8
-Z-olefine	4.1	4.1	15.4	19.3
Total identified	30.2	27.8	26.9	32.2
Total characterised *	17.6	29.9	11.1	23.3
Analysed extract(s)	47.8	57.7	38.0	55.5
Volatiles in distillates	2.1	8.2	1.9	6.2
Microwave extracts	---	---	51.2	---
Extracts not analysed	3.7	3.8	7.9	2.4
Total extractable	53.6	69.7	99.0	64.1
Unextractable (solids)	46.4	30.3	1.0	35.9
Accountability	100.0	100	100	100

* compounds were at least characterised by extraction and chromatographic behaviour (HPLC)

Table 16 TRR levels and distribution of parent compound and metabolites in muscle, fat and liver after application of [pyridyl-2,6-^{14}C]-fluopyram to laying hens

	muscle pool	fat pool	liver pool conventional extraction	liver pool enzymatic treatment
TRR [ppm]	0.049	0.496	0.532	0.532
Compound	% TRR	% TRR	% TRR	% TRR
Fluopyram	1.0	12.2	---	---
-PAA	---	---	1.3	---
-7-OH	---	---	0.8	2.9
-E-olefine	3.9	12.4	11.8	13.9
-Z-olefine	33.0	70.5	1.9	3.1
Total identified	37.9	95.2	15.7	20.0
Total characterised *	16.7	---	12.5	47.3
Analysed extract(s)	54.6	95.2	28.2	67.3
Volatiles in distillates	2.9	1.1	0.7	8.6
Microwave extracts	---	---	---	7.4
Extracts not analysed	1.8	2.9	3.4	7.0

	muscle pool	fat pool	liver pool conventional extraction	liver pool enzymatic treatment
TRR [ppm]	0.049	0.496	0.532	0.532
Compound	% TRR	% TRR	% TRR	% TRR
Total extractable	59.3	99.2	32.3	90.3
Unextractable (solids)	40.7	0.8	67.7	9.7
Accountability	100.0	100.0	100.0	100.0

* compounds were at least characterised by extraction and chromatographic behaviour (HPLC)

The major metabolic reactions of [pyridyl-2,6-^{14}C]-fluopyram in the laying hen involve the hydroxylation of the aliphatic chain followed by elimination of H_2O to form the olefines, and the oxidative cleavage of the aliphatic chain, yielding the PAA metabolite.

In conclusion, when the two metabolism studies are combined using appropriate scaling to obtain an overall picture of the metabolism, the scaled results indicated that the BZM metabolite was the major component of the extracted radioactivity identified in muscle (98% TRR), liver (89% TRR), eggs (90% TRR) and fat (68% TRR). The only other metabolite found above 10% TRR was the Z-olefine isomer, found in fat at about 26% TRR. Fluopyram was found in fat at about 2.5% TRR and made up less than 1% TRR in other matrices.

The proposed metabolic pathway in hens includes hydroxylation of the parent compound to the 7-OH and 8-OH metabolites, the formation of the Z/E-olefines, cleavage of the fluopyram molecule to produce BZM (from the phenyl-label) and PAA (from the pyridyl-label) and conjugation of the hydroxylated parent compound, mainly with glucuronic acid.

Table 17 Relative residue distribution of fluopyram and significant metabolites in hen eggs and fat, 24 hours after 14 daily oral doses of 26 ppm fluopyram

	eggs (days 7-14)				fat			
Radio-label	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]
Compound	mg/kg	mg/kg	mg/kg	%	mg/kg	mg/kg	mg/kg	%
Fluopyram (parent)	0.024	0.017	0.024	0.642	0.042	0.061	0.042	2.53
-benzoic acid	--			0.0	--			0.0
-BZM	3.447			89.6	1.126			67.9
-E-olefine	--	0.003	0.0042	0.0	0.037	0.062	0.0427	2.23
-Z-olefine	0.044	0.044	0.0621	1.14	0.425	0.35	0.241	25.6
-7-OH	--	0.002	0.0028	0.073	--	--	--	0.0
-PAA		0.011	0.0155	0.404		--	--	0.0
Extracts not analysed	0.003	0.023	0.0325	0.922	0.001	0.014	0.0096	0.642
Losses and volatiles	0.012	0.152	0.2146	5.89	--	0.006	0.0041	0.249
Bound (PES)	0.051	0.003	0.0042	1.44	0.01	0.004	0.0028	0.769
Total (label specific)	3.581 (A)	0.189	0.2668 (B)	100	1.641 (A)	0.0024	0.0165 (B)	100

[a] scaling factor =phenyl-labelled parent residues/pyridyl-labelled parent residues

[b] percentage of the total label-specific (adjusted) residues (i.e., A+B)

Table 18 Relative residue distribution of fluopyram and significant metabolites in hens muscle and liver, 24 hours after 14 daily oral doses of 26 ppm fluopyram

	muscle				liver			
Radio-label	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]	phenyl	pyridyl	scaled [a] pyridyl	fraction [b]
Compound	mg/kg	mg/kg	mg/kg	%	mg/kg	mg/kg	mg/kg	%
Fluopyram (parent)	--	0.001	0.0009	0.0	--	--	--	0.0
-benzoic acid	--			0.0	0.024			0.244
-BZM	3.233			98	8.737			88.9
-E-olefine	--	0.002	0.0019	0.0	0.028	0.063	0.059	0.28
-Z-olefine	0.015	0.016	0.015	0.46	0.016	0.01	0.0094	0.16
-7-OH	--	--	--	0.0	--	0.004	0.0038	0.038
-PAA		--	--	0.0		0.007	0.0066	0.067
Extracts not analysed	--	0.001	0.0009	0.028	0.006	0.018	0.017	0.232
Losses and volatiles	0.016	0.001	0.0009	0.51	0.05	0.004	0.0038	0.545
Bound (PES)	0.015	0.02	0.0188	1.02	0.605	0.36	0.338	9.56
Total (label specific)	3.279 (A)	0.022	0.0206 (B)	100	9.486 (A)	0.389	0.365 (B)	100

[a] scaling factor =phenyl-labelled parent residues/pyridyl-labelled parent residues

[b] percentage of the total label-specific (adjusted) residues (i.e., A+B)

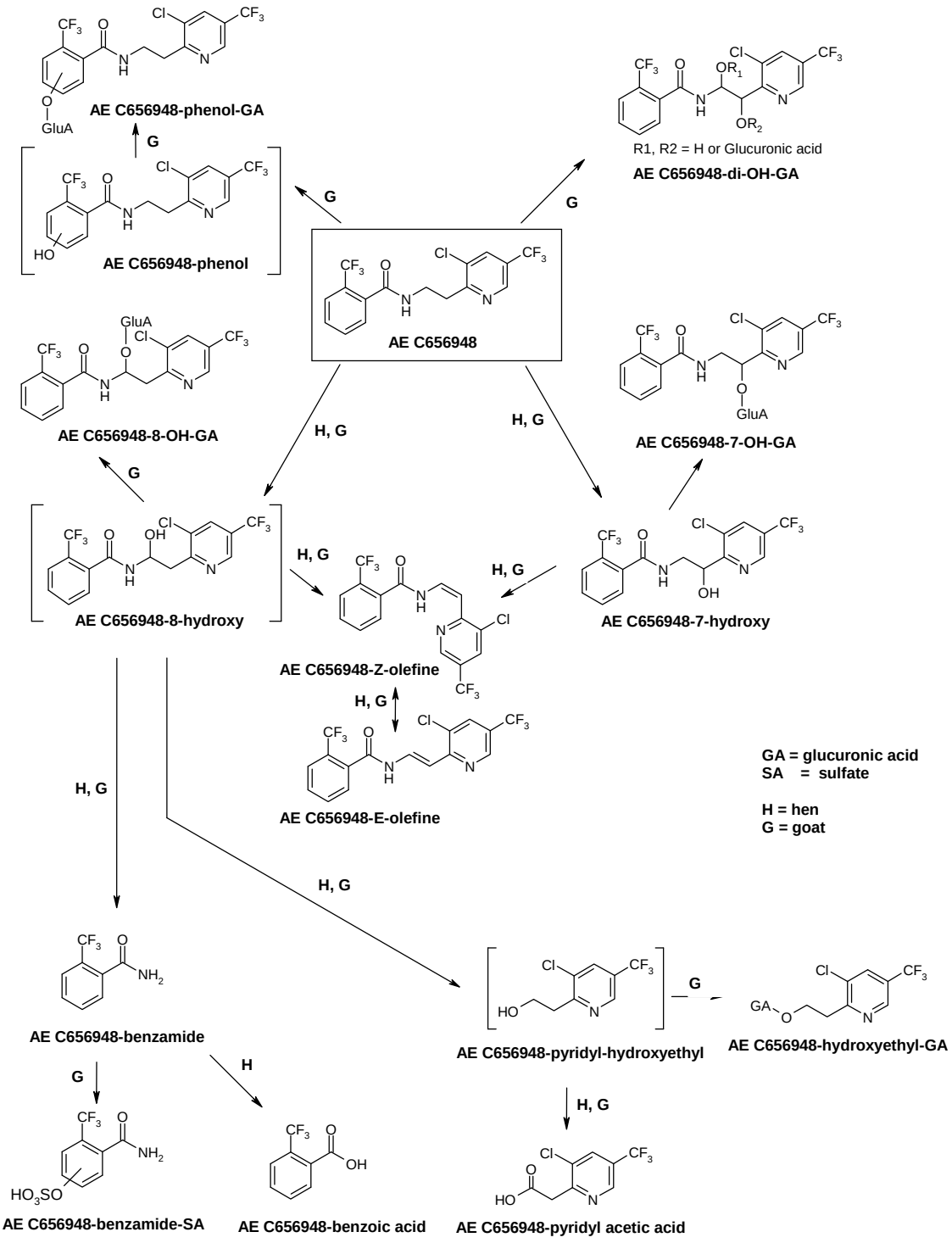


Figure 1 Proposed metabolic pathway of fluopyram in animals (derived from both radiolabels)

Plant metabolism

The Meeting received plant metabolism studies on grapes, beans, potatoes and red pepper with [phenyl-UL-^{14}C]-fluopyram and [pyridyl-2,6-^{14}C]-fluopyram.

Grapes

In two metabolism studies reported by Haas & Henk, 2006 [Ref: MEF-06/087] and Haas, 2006 [Ref: MEF-06/004], Muller-Thurgau grape vines were treated with three foliar sprays of [phenyl-UL-^{14}C]-fluopyram or [[pyridyl-2,6-^{14}C]-fluopyram (SC 500) respectively, the first applications at 9.7 or 9.6 mg ai/plant, approximating 100 g ai/ha, at growth stage BBCH 17-19 (6–9 leaves unfolded), the second at 19.5 or 21.5 mg ai/plant, approximating 200 g ai/ha, 42 days later at BBCH 71 (fruit set) and the last application at 21.2 or 18.6 mg ai/plant, approximating 200 g ai/ha, 49 days later at BBCH 81 (beginning of ripening). Mature grapes and leaves were harvested at maturity with a pre-harvest interval (PHI) of 18 days for grapes and 19 days for leaves. Young leaves were also sampled immediately after the second application. All samples and extracts were stored at ≤ -18 °C and were extracted and quantified within three days after sampling.

The leaf samples were triple-extracted in acetonitrile/water and the TRR in the young leaves measured 28.6 mg/kg and 48 mg/kg in the mature leaves in the phenyl-label study and 64 mg/kg and 43 mg/kg respectively in the pyridyl-label study. In grapes, of the total 1.7–1.9 mg/kg TRR found in the two studies, 1.3–1.5 mg/kg was extracted by surface washing with acetonitrile and a further 0.34 mg/kg TRR was obtained by triple-extraction in acetonitrile/water.

Parent compound and metabolites in the extracts were quantified by reversed phase HPLC coupled to a radioactivity detector with glass scintillation cell and identified by co-chromatography with reference compounds using HPLC and TLC chromatography and comparison of retention times and elution patterns. A comparison of the extraction efficiencies and residue profiles in the initial samples with those from samples stored for 8–10 months showed no significant differences in the composition or the proportions of the compounds measured. The reported determination limit, about 2.5 × background level was 0.002 mg/kg.

In the phenyl-label study, a total of 99% of the TRR was identified in grapes and 94% in leaves. Fluopyram was the major component found, 1.8 mg/kg (98% of the TRR) in grapes and 44 mg/kg (92% of the TRR) in leaves. Minor components present were the BZM and 7-OH metabolites in grapes and the 7-OH, 7-OH-glucoside and the 8-OH metabolites in grape leaves.

Table 19 TRR levels and distribution of parent compound and metabolites in grapes after application of [phenyl-UL-^{14}C]AE C656948 to grape vines, PHI 18 days

Compound	Young leaves	Grapes	Mature leaves
	(TRR = 28.55 mg/kg)	(TRR = 1.86 mg/kg)	(TRR = 48.06 mg/kg)
	% TRR	% TRR	% TRR
Surface wash (acetonitrile)	-	80.0	-
Acetonitrile/water extracted	98.2	18.6	93.9
Fluopyram (parent)	98.2	97.6	91.8
-8-OH	n.d.	n.d.	0.6
-7-OH	n.d.	0.3	0.7
-7-OH-glc	n.d.	n.d.	0.7
-BZM	n.d.	0.7	n.d.
Total identified	98.2	98.6	93.8
Total characterised a)	n.d.	0.1	n.d.
Total extractable	98.2	98.6	93.9
Total bound residues (PES)	1.8	1.4	6.1

[a] Radioactivity in this category is at least characterised by extraction and chromatographic behaviour.

n.d. =not detected.

The proposed metabolic pathway of fluopyram in grapes involves hydroxylation of parent compound leading to 7-OH and the 8-OH metabolites, conjugation of the 7-OH with glucose and cleavage of the activated molecule forming BZM.

In the pyridyl-label study, a total of 97% of the TRR was identified in grapes and 95% in leaves. Fluopyram was the major component found, 1.63 mg/kg (96% of the TRR) in grapes and 39 mg/kg (91% of the TRR) in leaves. Minor metabolites present were PCA and 7-OH in grapes and 7-OH and its glucoside conjugate, 8-OH and PCA in grape leaves.

Table 20 TRR levels and distribution of parent compound and metabolites in grapes after application of [pyridyl-2,6-^{14}C]-fluopyram to grape vines, PHI 18 days

Compound	Young leaves	Grapes	Mature leaves
	(TRR = 64.18 mg/kg)	(TRR = 1.70 mg/kg)	(TRR = 42.66 mg/kg)
	% TRR	% TRR	% TRR
Surface wash (acetonitrile)	-	76.9	-
Acetonitrile/water extracted	97.3	20.2	94.7
Fluopyram (parent)	95.7	95.8	91.3
-8-hydroxy	0.2	n.d. [b]	0.8
-7OH	0.3	0.3	1.0
-7-OH-glc	0.2	n.d.	0.8
-PCA	0.3	0.9	0.8
Total identified	96.7	97.0	94.7
Total characterised [a]	0.6	0.1	n.d.
Total extractable	97.3	97.1	94.7
Total bound residues (PES)	2.7	2.1	5.2

[a] Radioactivity is at least characterised by extraction and chromatographic behaviour.

[b] nd = not detected.

The proposed metabolic pathway of [pyridyl-2,6-^{14}C]-fluopyram in grapes involves the hydroxylation of parent compound leading to 7-OH and 8-OH, conjugation of the 7-OH with glucose and cleavage of the activated molecule forming PCA.

Potato

The metabolism of fluopyram in potatoes was investigated in two studies reported by Bongartz, 2007 [Ref: MEF-05/512] and [ref: MEF-05/513]. In these studies, three foliar applications of [phenyl-UL-^{14}C]-fluopyram or [pyridyl-2,6-^{14}C]-fluopyram (SC 500), equivalent to 165–176 g ai/ha, were applied to potato plants at growth stages BBCH 16, 16 days later at BBCH 55 and 11 days later at BBCH 71. Potato leaves and tubers were harvested at maturity, 51 days after the last application and stored at ca. -20 °C. All samples were extracted within 4–6 days of sampling and stored at approximately -20 °C for up to 35–44 days before analysis. A comparison of the extraction efficiencies and residue profiles in the initial samples in the phenyl-label study with those from samples stored for about 1 month showed no significant differences.

In the two studies, the TRR in tubers (0.008–0.012 mg/kg) was very low while the TRR in leaves was 48 mg/kg (phenyl-label) and 22 mg/kg (pyridyl-label). Triple extraction in acetonitrile/water extraction retrieved 95–97% of the TRR in tubers and 99% of the TRR in leaves with 74–77% TRR (tubers) and 99% TRR (leaves) being quantified by reversed phase HPLC coupled to a radioactivity detector with glass scintillation cell. The determination limits, about 2.5 × background level were 0.001–0.002 mg/kg.

In the phenyl-label study, fluopyram (parent) accounted for 69% TRR in tubers and 98% TRR in leaves. BZM was measured in tubers (7.1% TRR) and in leaves (0.5% TRR). The 7-OH metabolite was also detected in low amounts (≤ 1.2% TRR) in both tubers and leaves.

Table 21 TRR levels and distribution of parent compound and metabolites in potato after application of 3 × 167 g [phenyl-UL-^{14}C]-fluopyram to potato vines, PHI 51 days.

Compound	Potato tuber	Potato leaves
	TRR = 0.008 mg/kg	TRR = 47.64 mg/kg
	% of TRR	% of TRR
Fluopyram (parent)	68.8	98.0
-7-OH	1.2	0.8
-BZM	7.1	0.5
Total identified	77.1	99.2
Total characterised	19.5	0.1
Total extracted (combined extracts)	96.7	99.4
Bound residues (solids)	3.3	0.6

The proposed metabolic pathway of [phenyl-UL-^{14}C]-fluopyram in potatoes involves hydroxylation of parent compound leading to 7-OH, which is cleaved to BZM.

In the pyridyl-label study, fluopyram (parent) accounted for 23% TRR in tubers and 98% TRR in leaves. The PCA metabolite was measured in tubers (50% TRR) and in leaves 0.5% TRR) and the 7-OH metabolite was also detected in low amounts (≤ 1.1% TRR) in both tubers and leaves.

Table 22 TRR levels and distribution of parent compound and metabolites in potato after application of 3 × 167 g [pyridyl-2,6-^{14}C]-fluopyram to potato vines, PHI 51 days

Compound	Potato tuber	Potato leaves
	TRR = 0.012 mg/kg	TRR = 21.67 mg/kg
	% TRR	% TRR
Fluopyram (parent)	23.2	98.1
-7-OH	1.1	0.6
-PCA	49.8	0.5
Total identified	74.1	99.2
Total characterised	21.2	0.4
Total extracted (combined extracts)	95.3	99.6
bound residues (solids)	4.7	0.4

The proposed metabolic pathway of [pyridyl-2,6-^{14}C]-fluopyram in potatoes involves hydroxylation of parent compound leading to 7-OH, which is cleaved to PCA.

Beans

The metabolism of fluopyram in beans was investigated in two studies reported by Haas, 2006 [Ref: MEF-06/005] and Haas & Henke, 2006 [Ref: MEF-06/004], where two foliar applications of [phenyl-UL-^{14}C]-fluopyram or [pyridyl-2,6-^{14}C]-fluopyram (SC 500), equivalent to or 269 or 265 g ai/ha and 259 or 245 g ai/ha respectively, were applied to bean plants at growth stage BBCH 51 and 28 days later at BBCH 75. Leaves and green beans (with pods) were sampled 4 days after the last application, mature succulent beans (without pods) and 'straw' (vines and empty pods) were sampled 29 days after the last application. A subsample of the succulent beans (without pods) were dried for a further 11 days (until there was no further moisture loss) to simulate 'dry beans'. Samples were stored ≤ -18 °C. All samples were extracted within 4–12 days of sampling and stored at or below -18 °C until analysis.

In the phenyl-label study, the TRR in green beans (with pods) was 1.4 mg/kg, in succulent beans (without pods) the TRR was 0.07 mg/kg and in the dried beans the TRR was 0.12 mg/kg. The TRR in fresh leaves (foliage) was 36 mg/kg while in the straw (including the empty pods) the TRR was 16.55 mg/kg. Triple extraction in acetonitrile/water retrieved 94–98% of the TRR in all samples with 84–98% TRR being identified by HPLC (LOQ of 0.001 mg/kg).

In succulent and dry beans, 11–13% of the respective TRR was measured as fluopyram with BZM being the major metabolite, at 51.6% TRR in succulent beans and 64% TRR in dry beans. In green beans, foliage and straw more that 90% of the TRR was identified as fluopyram, with BZM being either not detected or < 1% TRR.

Table 23 TRR levels and distribution of parent compound and metabolites in beans after foliar applications of [phenyl-UL-^{14}C]-fluopyram

Compound	Green beans	Foliage	Succulent beans	Dry beans	Straw
TRR (mg/kg)	1.4	35.97	0.07	0.12	16.55
	% TRR	% TRR	% TRR	% TRR	% TRR
Fluopyram (parent)	93.9	93.8	11.4	12.6	90.2
-8-hydroxy	-	0.3	6.0	2.1	0.6
-7OH	-	0.7	4.0	2.5	0.7
-7-OH-glc-MA [b]	-	2.2	2.2	-	4.1
-7-OH-glc	-	0.4	1.7	-	0.4
-hydroxy-glyc-gluc [b]	-	-.	6.7	10.4	-
-BZM	-	0.5	51.6	64.0	0.6
Total identified	93.9	97.9	83.6	91.6	96.7
Total characterised [a]	n.a.	0.2	11.3	5.7	n.a.
Total extractable (combined extracts)	93.9	98.1	94.9	97.3	96.7
Total bound residues	6.1	1.9	5.0	2.7	3.3

[a] Radioactivity at least characterised by extraction and chromatographic behaviour.

[b] glyc = glycoside, gluc = glucuronic acid, MA = malonic acid

The proposed metabolic pathway of [phenyl-UL-^{14}C]AE C656948 in beans involves the hydroxylation of parent compound leading to 7-OH and AE C656948-8-hydroxy, conjugation of the 7-OH metabolite with glycosides and malonic acid and cleavage to form BZM.

In the pyridyl-label study, the TRR in green beans (with pods) was 3.9 mg/kg, in succulent beans (without pods) the TRR was 0.17 mg/kg and in the dried beans the TRR was 0.13 mg/kg. The TRR in fresh leaves (foliage) was 38.5 mg/kg while in the straw (including the empty pods) the TRR was 19 mg/kg. Triple extraction in acetonitrile/water retrieved 96–99% of the TRR in all samples with 76–99% TRR being identified by HPLC.

In succulent and dry beans, 4.8–5.7% of the respective TRR was measured as fluopyram with PAA (23–30% TRR) and PCA (31–33% TRR) being the major metabolites found, with the hydroxyethyl-glycoside metabolite also being found at ≤ 3.1% TRR. Fluopyram was the predominant residue in green beans (99% TRR), in foliage (92% TRR) and in straw (87% TRR).

Table 24 TRR levels and distribution of parent compound and metabolites in beans after foliar applications of [pyridyl-2,6-^{14}C]-fluopyram

Compound	Green beans	Foliage	Succulent beans	Dry beans	Straw
TRR (mg/kg)	3.88	38.53	0.17	0.31	19.02
	% TRR	% TRR	% TRR	% TRR	% TRR
Fluopyram (parent)	99.3	92.3	4.8	5.7	87.1
-8-OH	-	0.5	2.7	1.6	0.9

Compound	Green beans	Foliage	Succulent beans	Dry beans	Straw
TRR (mg/kg)	3.88	38.53	0.17	0.31	19.02
	% TRR	% TRR	% TRR	% TRR	% TRR
-7OH	-	1.6	4.0	4.0	1.1
-7-OH-glc-MA [b]	-	3.2	[c]	[c]	4.7
-7-OH-glc [b]	-	0.6	1.4	1.3	0.7
-hydroxy-glyc-gluc [b]	-	-	4.5	5.6	0.2
-PAA	-	-	29.5	22.6	0.2
-hydroxyethyl-glyc [b]	-	0.2	1.9	3.1	-
-PCA	-	0.5	31.0	32.5	0.6
Total identified	99.3	98.9	79.8	76.4	95.5
Total characterised [a]	n.a.	0.2	17.9	21.0	0.3
Total extractable (combined extracts)	99.3	99.1	97.6	97.4	95.7
Total bound residues	0.7	1.0	2.3	2.6	4.3

[a] Unidentified metabolites characterised by extraction and chromatographic behaviour.

[b] glyc = glycoside, gluc = glucuronic acid, MA = malonic acid

[c] higher conjugates (e.g. -7-OH-glc-MA) detected in a multiple residue HPLC-region

The proposed metabolic pathway of [pyridyl-2,6-^{14}C]-fluopyram in beans involves hydroxylation of parent compound leading to the 7-OH and 8-OH metabolites, conjugation of the 7-OH with glycosides and malonic acid and cleavage of the activated molecule to form PAA and PCA and to a much lesser extent the hydroxyethyl-glycoside.

Pepper, Sweet

The metabolism of fluopyram in red bell pepper was investigated in two studies reported by Henk, Kuhnke & Spiegel, 2008 [Ref: MEF-06/315] and Henk, Spiegel & Weber, 2008 [Ref: MEF-06/314] using [phenyl-UL-^{14}C]-fluopyram and [pyridyl-2,6-^{14}C]-fluopyram respectively. In these studies the labelled fluopyram (SC 500) was applied by drip irrigation to plants growing on a stone wool substrate. A single application, equivalent to 5 mg ai/plant, was applied when the plants were at BBCH 15-17 growth stage (5th–7th leaf unfolded). Fruit were harvested at maturity at three time points (55-96 days after application) and the remaining plants (without roots and fruit) were sampled one day after the third fruit harvest (97 days after application). To facilitate identification of metabolites, an additional 4× treatment was also applied, with young plant being taken at the start of flowering (33 days after application).

The plant samples were extracted and quantified within a day of harvest and the fruit samples were stored at about -18 °C for up to 42 days before being combined, extracted and analysed. Samples were triple-extracted in acetonitrile/water (extracting more than 95% TRR) and residues were measured by reversed phase HPLC coupled to a radioactivity detector with glass scintillation cell and identified using co-chromatography (HPLC, TLC).

In the phenyl-label study, the TRR in the mature fruits was low (0.04 mg/kg) and consisted mainly of the parent fluopyram (49% TRR), with the cleavage product BZM representing 16% TRR. In plants, the TRR was 3.5 mg/kg, again mainly the parent fluopyram (64% TRR) with BZM being found at 10% of the TRR. Other metabolites detected were 7-OH and its glucoside conjugate (each at ≤ 9% TRR) in both fruits and plants. The plant samples also contained the 8-OH metabolite and a malonic acid conjugate of the 7-OH-glc at levels of < 1% TRR.

Table 25 TRR levels and distribution of parent compound and metabolites in red bell peppers after a drip irrigation treatment with [phenyl-UL-^{14}C]-fluopyram

Compound	Intermediate plant (4×overdose)	Fruits (1×)	Rest of plant (1×)
	(TRR 6.24 mg/kg)	(TRR 0.038 mg/kg)	(TRR 3.54 mg/kg)
	% TRR	% TRR	% TRR
Fluopyram (parent).	86.6	48.9	64.0
-BZM	3.8	16.1	10.1
-7-OH-glc-MA	-	-	0.7
-7-OH-glc	2.7	3.9	8.9
-7-OH	3.8	9.0	6.8
-8-OH	0.6	-	0.5
Total identified	97.4	77.8	90.9
Total characterised [a]	1.1	18.4	5.4
Total extractable (combined extracts)	98.5	96.2	96.3
Total bound residues (PES)	1.5	3.8	3.7

[a] Radioactivity at least characterised by extraction and chromatographic behaviour.

The proposed metabolic pathway of [phenyl-UL-^{14}C]-fluopyram in red bell pepper involves the hydroxylation of parent compound leading to 7-OH and 8-OH metabolites, conjugation of the 7-OH with glycosides and malonic acid and cleavage of the activated molecule to form BZM.

In the pyridyl-label study, the TRR in the mature fruits was also low (0.06 mg/kg) and consisted mainly of the cleavage product PCA and two glycoside isomers of the PAA metabolite consisting of 43.5%, 23.8% 14.2% TRR respectively, with parent residues making up 16.2% TRR. The non-conjugated PAA and 7-OH were additionally found in the fruits of the 4× overdose experiments.

In plants, where the TRR was 2.34 mg/kg, the major component was parent fluopyram (70% TRR) with minor metabolites (each < 10% TRR) being 7-OH and its glucose conjugate, the N-oxide metabolite and a higher glucose conjugate of the hydroxyethyl cleavage product. In the young 4×-treated plant the 8-OH metabolite was also identified.

Table 26 TRR levels and distribution of parent compound and metabolites in red bell peppers after a drip irrigation treatment with [pyridyl-2,6-^{14}C]-fluopyram

Compound	Intermediate plant (4× overdose)	Fruits (4× overdose)	Fruits (1×)	Rest of plant (1×)
	TRR 18.24 mg/kg	TRR 0.149 mg/kg	TRR 0.06 mg/kg	TRR 2.34 mg/kg
	% TRR	% TRR	% TRR	% TRR
Fluopyram (parent)	88.1	32.8	16.2	70.1
-PCA	0.4	19.5	43.5	-
-PAA	-	9.8	-	-
-PAA-glycoside (isomer 1)	-	12.5	23.8	-
-PAA-glycoside (isomer 2)	-	19.7	14.2	-
-8-OH-di-glc	1.5	-	-	7.0
-7-OH-glc	1.9	-	-	9.2
-N-oxide	0.5	-	-	2.9
-7OH	3.5	3.7	-	5.1
-8-OH	0.7	-	-	-
Total identified	96.7	98.1	97.8	94.3

Compound	Intermediate plant (4× overdose)	Fruits (4× overdose)	Fruits (1×)	Rest of plant (1×)
	TRR 18.24 mg/kg	TRR 0.149 mg/kg	TRR 0.06 mg/kg	TRR 2.34 mg/kg
	% TRR	% TRR	% TRR	% TRR
Total characterised [a]	1.1	-	-	1.0
Total extractable (combined extracts)	97.8	98.1	97.8	95.3
Total bound residues	2.2	1.9	2.2	4.7

[a] Radioactivity in this category is at least characterised by extraction and chromatographic behaviour.

The proposed metabolic pathway of [pyridyl-2,6-^{14}C]AE C656948 in red bell pepper involves hydroxylation of parent compound leading to 7-OH and 8-OH metabolites, conjugation of the 7-OH with glucose and cleavage of the activated molecule to form PCA, PAA and glycoside conjugates and hydroxyethyl conjugates. A minor pathway in the plant (but not fruit) involves the formation of an N-oxide.

Apple – cell culture

In a study reported by Haas, 2005 [Ref: MEF-05/142], the metabolism of fluopyram was investigated in heterotrophic plant cell suspension cultures originating from apple fruit following incubation with [phenyl-UL-^{14}C]-fluopyram and with [pyridyl-2,6-^{14}C]-fluopyram in order to facilitate metabolite identification and to produce radiolabelled reference compounds for the plant and animal metabolism studies.

The radiolabelled fluopyram compounds were dissolved in acetonitrile and diluted with non-radiolabelled fluopyram to obtain the desired specific radioactivity (concentration: 50 µM = 794 µg/40 mL cell suspension; radioactivity ca. 185 kBq = 5 µCi). The solution was applied directly to the plant suspension cells at the beginning of the exponential growth phase and samples were taken 7 days later.

Samples were filtered to separate the cells from the nutrient medium. The cells were washed intensively with water to remove soluble radioactivity from the cell surfaces. The wash solution was combined with the decanted supernatant from the nutrient medium before being concentrated and re-dissolved in acetonitrile or acetonitrile/water and stored at approximately 4 °C during processing and then at about -20 °C in a freezer.

The cells were triple-extracted with acetonitrile/water and the combined extracts were concentrated to the aqueous remainder. The extracted fibrous solids were air dried at room temperature and radio-assayed without further extraction since only a low residual amount of radioactivity was evident.

Metabolites were isolated and identified from the concentrated medium (phenyl label) and from the cell extracts (both labels) by HPLC fractionation and purification of the fractions. Purified metabolites were identified by LC-NMR, HPLC-MS/MS, FT-MS and various NMR techniques.

Table 27 Fluopyram metabolites identified in apple cell cultures treated with radio-labelled fluopyram

Peak No	Compound name	Isolated fraction (Rt: minutes)[a]	Isolated from ... , Identified by...
1	-hydroxymethyl-BZM	HF3307R1 (24.3)	nutrient, phenyl-label LC-NMR, FT-MS/MS, LC-MS/MS [m/z 219]
2	-BZM	HF3307P5 (25.0)	nutrient, phenyl-label LC/MS/MS [m/z 189]
3	-PCA	HF3308R1 (32.8)	cell extract, pyridyl label LC-MS, LC/MS/MS [m/z 225]
4	-pyridyl-hydroxymethyl conjugate	HF3308R2 (39.2)	cell extract, pyridyl label LC-NMR, FT-MS, LC/MS/MS [m/z 517]

Peak No	Compound name	Isolated fraction (Rt: minutes)[a]	Isolated from ... , Identified by...
5	-pyridyl-hydroxyethyl malonyl-hexose conjugate	HF3308P3 (43.6)	cell extract, pyridyl label LC-NMR, FT-MS, LC/MS/MS [m/z 473]
6	-deschloro-3-hydroxy-glc	HF3307P3 (44.3)	nutrient and cell extract, phenyl-label LC-NMR, LC-MS/MS [m/z 540]
7	-7-OH-glc	HF3307P1 and HF330P1 (48.1)	nutrient & cell extract, phenyl & pyridyl labels LC-NMR, H,H-COSY-NMR, HMBC-NMR, HMQC-NMR, LC-MS/MS [m/z 574]
8	-8-OH-glc	HF3307R5 (50.1)	nutrient, phenyl-label LC-NMR, H,H-COSY-NMR, HMBC-NMR, HMQC-NMR, LC-MS/MS [m/z 574]
9	-7-OH	HF3307P2 (54.2)	nutrient, phenyl-label LC-NMR, H,H-COSY-NMR, LC-MS/MS [m/z 412]

[a] Method AEC948_HF33_B

In summary, residues of unchanged fluopyram account for the major part of the residues in grapes and green beans and also occur at lower levels in potatoes, drip-irrigated red peppers, dry and succulent beans (without pods), where the commodities are not directly exposed to fluopyram spray applications. In these latter commodities, significant levels of the cleavage products BZM and to a lesser extent the PCA and PAA metabolites also occur, generally at concentrations close to the combined fluopyram plus BZM levels. The PCA metabolite residues were up to 0.1 mg/kg in dry beans, less than 0.05 mg/kg in succulent beans (without pods) and peppers (drip irrigated) and less than 0.01 mg/kg in potatoes. Residues of the PAA metabolite in beans were 0.05–0.07 mg/kg in both the fresh and dry beans (without pods).

The proposed metabolic pathway in plants involves hydroxylation of the parent compound to the 7-OH and 8-OH metabolites and subsequent conjugation (mainly with sugars), cleavage of the parent molecule to produce the BZM, PAA and PCA metabolites.

Table 28 Residue distribution of fluopyram and significant metabolites [a] in edible plant tissues following foliar or drip irrigation treatments with radio-labelled fluopyram.

	grapes		potatoes		green beans		succulent beans		dry beans		pepper (drip)	
Radio-label	phenyl	pyridyl	phenyl	pyridyl	phenyl	pyridyl	phenyl	pyridyl	phenyl	pyridyl	phenyl	pyridyl
TRR [mg/kg][b]	1.9	1.7	0.008	0.012	1.4	3.9	0.07	0.17	0.12	0.31	0.04	0.06
Compound	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]
Total analysed	99	97	97	95	94	99	95	98	97	97	96	98
Fluopyram (parent)	98 [1.8]	96 [1.6]	69 [0.006]	23 [0.003]	94 [1.3]	99 [3.9]	11 [0.008]	4.8 [0.008]	13 [0.015	5.7 [0.018]	49 [0.019]	16 [0.01]
-BZM	0.7 [0.01]		7.1 [0.001]		---		52 [0.036]		64 [0.08]		16 [0.006]	
-hydroxy-glyc-gluc	---	---	---	---	---	---	6.7 [0.005]	4.5 [0.008]	10 [0.013]	5.6 [0.017]	---	---
-PCA		0.9 [0.02]		50 [0.006]		---		31 [0.05]		33 [0.1]		44 0.026]
-PCA-glycosides [c]		---		---		---		---		---		38 [0.023]
-PAA		---		---		---		30 [0.05]		23 [0.07]		---

[a] above 10% TRR in any one matrix

[b] fluopyram equivalents. LOQs (about 2.5× background levels) = 0.001–0.002 mg/kg

[c] sum of isomers

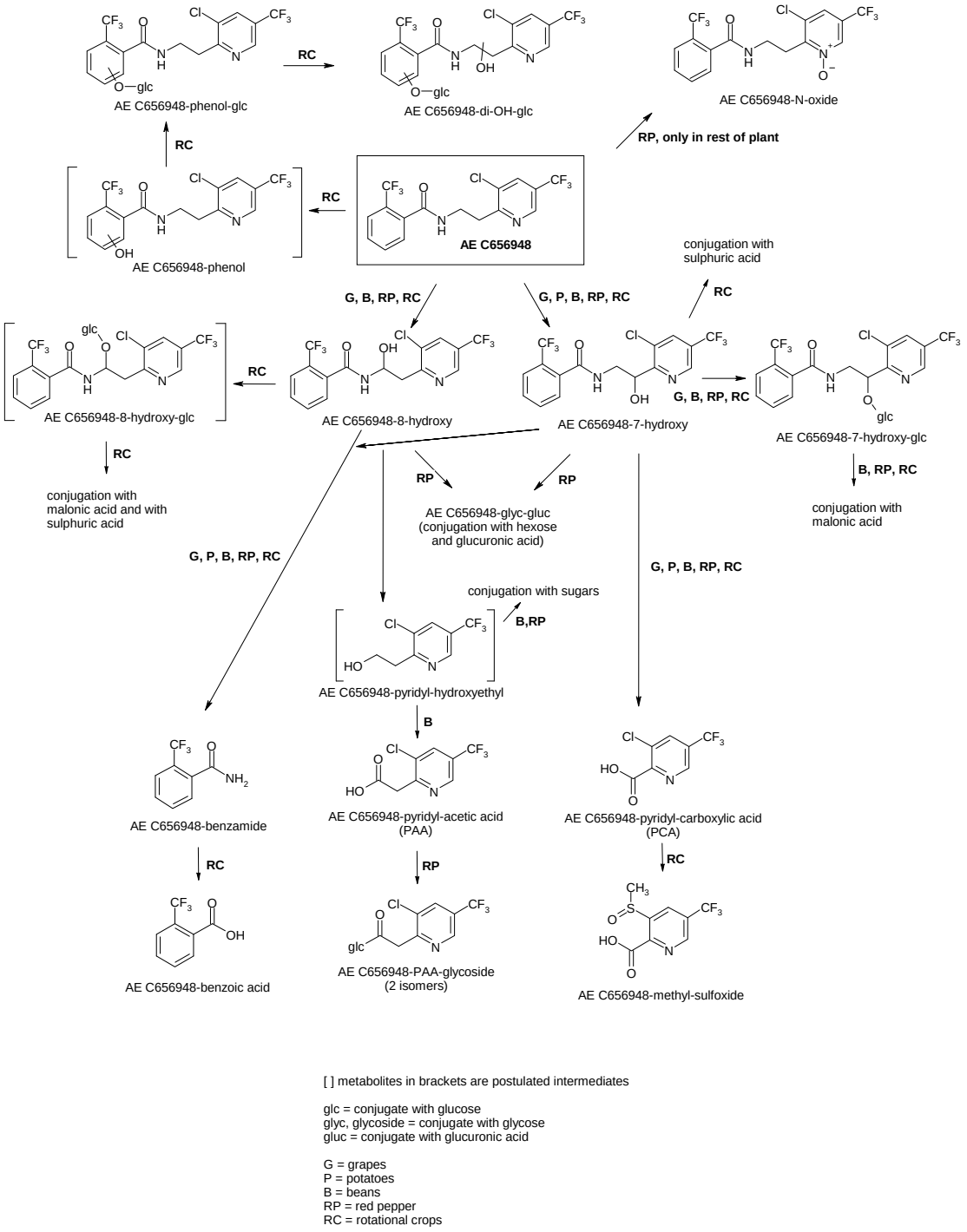


Figure 2 Proposed metabolic pathway of fluopyram in plants (derived from both radiolabels)

ENVIRONMENTAL FATE

The meeting received information on aerobic and anaerobic degradation in soil, photolysis on the soil surface, field dissipation, adsorption/desorption in soil, leaching into groundwater, aquatic degradation and confined and field rotational crop studies. Because fluopyram is intended for use as a foliar treatment close to harvest, as a seed treatment and drip irrigation treatment, only hydrolysis, aerobic soil degradation, soil dissipation, soil photolysis and the rotational crop studies were considered relevant for the present evaluation. The other information was not summarised.

Hydrolysis

The hydrolytic stability of fluopyram was reported by Doble & Oddy, 2006 [Ref: CX/06/015]. Fluopyram was stable in sterile aqueous buffered solutions at pH 4, 7 and 9 when stored at 50 °C in the dark for 5 days. One minor degradate was measured, up to 1.6% applied radiolabel, in the pH 7 and pH 9 solutions at the end of the test period.

Photolysis

Photolytic degradation of fluopyram in sterile aqueous buffer solutions (pH 7) at 25 °C was reported by Oddy & Brett, 2008 [Ref: CX/06/016]. After exposure to continuous artificial sunlight for 13 days, radio-labelled fluopyram residues decreased from 99.5% to 64% of the applied phenyl-label and from 100% to 72% of the applied pyridyl label. In both cases, the one major degradation product was the lactame degradate, at 12–13% of the TRR. A number of minor degradation products were reported, all at less than 4.2% of the applied amount. Based on simple first order kinetics, experimental DT_{50} values of 21–25 days and DT_{90} values of 70-83 days were calculated.

Aerobic soil metabolism

The degradation of [phenyl-UL-^{14}C]-fluopyram and [pyridyl-2,6-^{14}C]-fluopyram was investigated in two studies reported by Babczinski, 2008 [Ref: MEF-06/295] and Menke & Telscher, 2008 [Ref: MEF-07/424] respectively. In these studies, four European soils (loam soils, silt-loam and sandy-loam) mixed with either phenyl-labelled or pyridyl-labelled fluopyram (at a target rate of 0.67 mg/kg soil, equivalent to 250 g/ha, mixed to 2.5 cm depth in soil with a bulk density of 1.5 g/mL), were incubated in the dark for 121–128 days at 20 °C. Samples were collected at intervals up to 121–128 days after application, extracted, and analysed within 3 days. Residues were extracted in acetonitrile/water and more aggressively using microwave-assisted extraction (at 70 °C) and the extracts were analysed by LSC and HPLC.

At the end of the 121 day incubation period, non-extractable radioactivity in the two studies were about 8.6–15% of the applied radiolabel (AR), with $^{14}CO_2$ accounting for up to 16–24% of the AR. Metabolites found at concentrations greater than 0.5% of the applied dose were 7-OH (maximum 4.2% AR) and BZM (maximum 1.1% AR) in the phenyl-label study and were 7-OH (maximum 3.3% AR), PCA (0.7% AR) and the PCA methyl-sulfoxide (maximum 1% AR).

Table 29 Degradation of [phenyl-UL-^{14}C]-fluopyram in soils under aerobic conditions

		Residues (% AR) on the following days after treatment (DAT)							
Compound	Soil type	0	1	2	7	14	30	62	121
Fluopyram (parent)	Silt-loam [a]	97.8	96.9	95.5	95.3	91.0	87.6	77.5	67.6
	Sandy-loam [b]	93.5	94.5	94.1	94.8	92.2	89.0	76.8	66.7
	Loam-1 [c]	97.7	102.4	97.9	97.1	93.6	91.6	90.3	76.1
	Loam-2 [d]	96.5	96.6	95.6	92.8	89.5	85.2	75.5	57.3
-7-OH	Silt-loam [a]	0.1	0.5	0.6	1.0	2.0	2.5	3.0	2.7
	Sandy-loam [b]	0.2	0.3	0.3	1.3	1.9	3.2	3.7	3.7
	Loam-1 [c]	n.d.	0.3	0.6	1.5	2.0	3.3	3.5	3.3

		Residues (% AR) on the following days after treatment (DAT)							
Compound	Soil type	0	1	2	7	14	30	62	121
	Loam-2 [d]	n.d.	0.4	0.5	1.7	2.6	3.2	4.2	4.0
-BZM	Silt-loam [a]	0.4	0.3	0.3	0.2	0.4	0.5	0.6	0.6
	Sandy-loam [b]	0.4	0.4	0.3	-	0.5	0.5	0.7	0.8
	Loam-1 [c]	0.1	0.1	0.1	-	0.3	0.2	0.1	0.3
	Loam-2 [d]	0.4	0.4	0.5	0.6	0.9	1.1	1.1	1.1
Total unidentified	Silt-loam [a]	1.4	1.8	1.3	1.8	2.2	0.2	1.1	2.4
	Sandy-loam [b]	1.6	1.5	1.5	1.8	1.7	0.3	1.2	1.9
	Loam-1 [c]	1.7	1.2	1.7	2.1	2.0	0.4	0.7	1.6
	Loam-2 [d]	1.0	1.3	1.3	1.6	2.4	0.2	0.7	2.7
Total extractable residues	Silt-loam [a]	99.7	99.5	97.6	98.3	95.6	90.9	82.2	73.2
	Sandy-loam [b]	95.7	96.7	96.2	97.9	96.3	93.0	82.4	73.1
	Loam-1 [c]	99.5	104.1	100.3	100.6	97.9	95.4	94.5	81.3
	Loam-2 [d]	97.9	98.7	97.9	96.8	95.4	89.7	81.4	65.1
$^{14}CO_2$	Silt-loam [a]	n.a.	0.1	0.1	0.5	1.3	3.0	7.8	14.5
	Sandy-loam [b]	n.a.	< 0.1	0.1	0.5	1.1	2.8	7.7	13.9
	Loam-1 [c]	n.a.	0.1	0.1	0.4	0.7	1.8	4.0	13.4
	Loam-2 [d]	n.a.	< 0.1	0.1	0.5	1.5	3.8	9.8	16.2
Volatile organics	Silt-loam [a]	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.0
	Sandy-loam [b]	n.a.	< 0.1	< 0.1	< 0.1	0.0	0.1	< 0.1	0.1
	Loam-1 [c]	n.a.	0.0	0.0	< 0.1	0.1	0.0	0.1	0.0
	Loam-2 [d]	n.a.	0.0	0.1	0.0	< 0.1	0.0	0.1	0.1
Non-extractable residues	Silt-loam [a]	1.8	1.9	2.0	2.9	3.3	4.7	8.5	11.3
	Sandy-loam [b]	2.0	1.7	2.1	2.9	2.9	4.2	8.1	10.8
	Loam-1 [c]	2.3	1.7	2.2	3.0	3.1	3.9	7.1	10.1
	Loam-2 [d]	1.3	1.5	1.7	2.9	3.1	5.1	9.9	13.8
Total recovery	Silt-loam [a]	101.5	101.4	99.8	101.8	100.2	98.5	98.5	99.1
	Sandy-loam [b]	97.7	98.4	98.3	101.3	100.3	100.1	98.1	97.9
	Loam-1 [c]	101.8	105.9	102.6	104.0	101.9	101.2	105.8	104.9
	Loam-2 [d]	99.1	100.2	99.8	100.2	100.0	98.6	101.2	95.1

a Hoefchen am Hohenseh silt-loam soil (sand 8:silt 75:clay 17), 3.6% OM, CEC 14.4 meq/100 g

b Laacherhof AXXa sandy loam soil (sand 73:silt 16:clay 11), 2.6% OM, CEC 9.4 meq/100 g

c Laacherhof Wurmwiese loam soil (sand 48:silt 36:clay 15.9), 3.5% OM, CEC 11.6 meq/100 g

d Laacherhof AIIIa loam soil (sand 42:silt 40:clay 17), 1.3% OM, CEC 11.3 meq/100 g

n.a - not analysed

The experimental data from the phenyl-label study were evaluated according to the FOCUS guidance document on degradation kinetics and based on a simple first order model, selected as the best-fit, DT_{50s} and DT_{90s} for [phenyl-UL-^{14}C]-fluopyram were estimated. Modelling for the 7-OH metabolite was also conducted on the basis of the results achieved using the SFO model for parent compound and DT_{50} and DT_{90} values were determined.

Table 30 Degradation calculations of [phenyl-UL-^{14}C]-fluopyram and the 7-OH metabolite in four soils under aerobic conditions (Simple First Order model)

Compound	Soil	DT_{50} (days)	DT_{90} (days)	Chi^2 error (%)
Fluopyram	Silt loam (Hoefchen a. Hohenseh)	221	735	1.0508
	Sandy loam (Laacherhof AXXa)	231	769	1.2716
	Loam (Laacherhof Wurmwiese)	339	> 1000	1.8053
	Loam (Laacherhof AIIIa)	165	549	0.7347

Compound	Soil	DT_{50} (days)	DT_{90} (days)	Chi^2 error (%)
	SFO- Geomean	231	> 365	
-7-OH	Silt loam (Hoefchen a. Hohenseh)	13.2	43.7	9.3
	Sandy loam (Laacherhof AXX)	17.3	57.6	6.3
	Loam (Laacherhof Wurmwiese)	14.1	46.8	6.6
	Loam (Laacherhof AIIIa)	17.7	58.7	9.0
	SFO - Geomean	15.5	51.3	

Fluopyram was slowly degraded during the test period of 121 days. Under aerobic conditions, the average simple first order (SFO) half-lives for fluopyram in the tested soils was 231 days and 15.5 days for the 7-OH metabolite.

The proposed metabolic breakdown of fluopyram in soil involves the hydroxylation in the 7-position of the parent molecule and molecule cleavage yielding BZM (presumably originating from 7-OH) followed by mineralization to CO2 and the formation of non-extractable residues.

Table 31 Degradation of [pyridyl-2,6-^{14}C]-fluopyram in soils under aerobic conditions

		Residues (% AR) on the following days after treatment (DAT)							
Compound	Soil type	0	1	3	7	15	30	58	128
Fluopyram (parent)	Silt-loam [a]	97.4	96.3	96.2	93.7	93.9	87	78.8	64.1
	Sandy-loam [b]	97.5	96.4	97.6	95.4	94.8	90	87.2	81
	Sandy-loam [c]	97.3	95.6	95.6	93.9	92.1	88.2	80.1	68.4
	Clay-loam [d]	96.1	95.3	93.3	89.4	87.7	81.1	71.2	56.5
-7-OH	Silt-loam [a]	n.d.	n.d.	0.9	1.5	1.7	2.3	2.3	1.7
	Sandy-loam [b]	n.d.	n.d.	n.d.	0.5	1.8	2.1	1.7	2
	Sandy-loam [c]	n.d.	1	1.7	2.6	2.8	2.8	2.6	2.2
	Clay-loam [d]	n.d.	0.7	1.9	2.8	3	3.3	2.6	1.1
-PCA	Silt-loam [a]	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	Sandy-loam [b]	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	Sandy-loam [c]	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	Clay-loam [d]	n.d.	n.d.	n.d.	0.4	0.6	0.7	0.4	n.d.
-methyl sulfoxide	Silt-loam [a]	n.d.	n.d.	n.d.	n.d.	n.d.	0.6	0.9	1
	Sandy-loam [b]	n.d.	n.d.	n.d.	n.d.	n.d.	0.5	0.6	0.8
	Sandy-loam [c]	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	Clay-loam [d]	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Total unidentified	Silt-loam [a]	0.7	0.4	0.5	0.6	0.5	1.3	1.7	2.2
	Sandy-loam [b]	0.6	0.5	0.5	0.5	0.4	1.2	2	2.7
	Sandy-loam [c]	0.6	0.7	0.6	0.5	0.5	0.5	1	0.6
	Clay-loam [d]	0.5	0.6	0.6	1.3	1.5	2.4	2.6	2.3
Total extractable residues	Silt-loam [a]	98.1	96.7	97.6	95.7	96.1	91.2	83.7	68.4
	Sandy-loam [b]	98.2	96.9	98.1	96.5	97	93.9	91.5	86.6
	Sandy-loam [c]	97.9	97.2	97.9	97	95.4	91.5	83.8	71.2
	Clay-loam [d]	96.6	96.6	95.7	93.9	92.8	87.5	76.9	59.9
$^{14}CO_2$	Silt-loam [a]	n.a.	< 0.1	< 0.1	0.2	0.7	2.3	6.8	18.3
	Sandy-loam [b]	n.a.	< 0.1	< 0.1	0	0.2	0.7	1.9	4.7
	Sandy-loam [c]	n.a.	< 0.1	0.1	0.3	1.1	3.2	7.8	19.5
	Clay-loam [d]	n.a.	< 0.1	< 0.1	0.2	1.1	4.1	10.9	24
Volatile organics	Silt-loam [a]	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	Sandy-loam [b]	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	Sandy-loam [c]	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1

		Residues (% AR) on the following days after treatment (DAT)							
Compound	Soil type	0	1	3	7	15	30	58	128
	Clay-loam [d]	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Non-extractable residues	Silt-loam [a]	1.9	2.3	2.5	3.1	3.8	5.5	8.5	13.2
	Sandy-loam [b]	1.8	2.2	2	2.7	3.1	4.6	6.1	8.6
	Sandy-loam [c]	2.1	2.3	2.3	2.9	4	5.9	8	11.7
	Clay-loam [d]	3.4	3.9	3.5	5.1	6.1	8.7	11.4	15.1
Total recovery	Silt-loam [a]	100.0	99.0	100.1	99.0	99.8	99.1	98.9	99.9
	Sandy-loam [b]	100.0	99.2	100.1	99.2	100.3	99.2	99.5	99.9
	Sandy-loam [c]	100.0	99.6	100.2	100.1	100.6	100.6	99.6	102.4
	Clay-loam [d]	100.0	100.5	99.3	99.2	100.0	100.3	99.2	98.9

[a] Hoefchen am Hohenseh silt loam soil (sand 27:silt 56:clay 17), 4.1% OM, CEC 13.4 meq/100 g

[b] Laacherhof AXXa sandy loam soil (sand 75:silt 14:clay 11), 3.8% OM, CEC 9.5 meq/100 g

[c] Laacherhof Wurmwiese loam soil (sand 59:silt 24:clay 17), 3.1% OM, CEC 9.6 meq/100 g

[d] Dollendorf, II DD clay loam soil (sand 35:silt 36:clay 29), 8.8% OM, CEC 21.2 meq/100 g

n.a.: not analysed; n.d.: not detected

The experimental data from the pyridyl-label study were evaluated according to the FOCUS guidance document on degradation kinetics and based on a simple first order model, selected as the best-fit, DT_{50}s and DT_{90}s were estimated. Modelling for the 7-OH metabolite was also conducted on the basis of the results achieved using the SFO model for parent compound and DT_{50} and DT_{90} values were determined.

Table 32 Degradation calculations of [pyridyl-2,6-^{14}C]-fluopyram and the 7-OH metabolite in four soils under aerobic conditions (Simple First Order model)

Compound	Soil	DT_{50} (days)	DT_{90} (days)	Chi^2 error (%)
Fluopyram	Silt loam (Hoefchen a. Hohenseh)	210	697	0.852
	Sandy loam (Laacherhof AXXa)	464	> 1000	1.034
	Loam (Laacherhof Wurmwiese)	250	829	0.739
	Clay loam (Dollendorf)	162	538	1.588
	SFO- Geomean	250	> 365	
-7-OH	Silt loam (Hoefchen a. Hohenseh)	5.9	19.5	9.7
	Sandy loam (Laacherhof AXXa)	10.8	35.9	15.7
	Loam (Laacherhof Wurmwiese)	8.5	28.2	27.7
	Clay loam (Dollendorf)	5.8	19.3	24.5
	SFO - Geomean	7.5	24.8	

Fluopyram degraded slowly during the test period of 128 days. Under aerobic conditions, the Geomean of the half-lives for fluopyram in the tested soils was 250 days and 7.5 days for the 7-OH metabolite.

A proposed metabolic pathway for the aerobic degradation of fluopyram in soil involves hydroxylation in the 7-position of the parent molecule, cleavage yielding PCA (presumably originating from 7-OH), sulfoxidation with the elimination of the chlorine atom of PCA yielding methyl-sulfoxide, mineralization to CO2 and the formation of bound residues.

The aerobic biotransformation of fluopyram radiolabelled in the phenyl ring and in the pyridyl ring was studied in a silty clay loam (Nebraska) and a sandy loam (California) and reported by Meyer, 2008 [Ref: MEGMP069]. In this study, [phenyl-UL-^{14}C] and [pyridyl-2,6-^{14}C]-fluopyram was applied to soil at 0.11-0.13 mg ai/kg, equivalent to 250 g/ha (15 cm depth, 1.5 g/cm^3 bulk density) and incubated in the dark at 25 °C and at 75% of water holding capacity at 1/3 bar for 365 days.

Duplicate soil samples were collected at 12 intervals during the 1-year study period, extracted the same day and stored for up to 23 days before analysis. The samples were triple-extracted in acetonitrile/water and analysed by HPLC co-chromatography using non-radioactive reference standards to identify and characterise metabolites with confirmation by electronspray ionisation LC/MS. If the total radioactivity in the ambient soil extract was less than 90% of the total applied activity, the soil was subjected to aggressive extraction with acetonitrile/water in an ASE cell.

In the Nebraska silty-clay loam soil, the average extracted radioactive residues decreased from 97.4% (day 0) to 60.1% at day 273 in the phenyl label experiment, and from 98% (day 0) to 60.5% at day 365 in the pyridyl label experiment. Bound residues increased from 2.6% (day 0) to a maximum of 14.9% at day 273 (phenyl label) and from 2% (day 0) to14.7% at day 365 (pyridyl label). Radioactive carbon dioxide increased to a maximum of 24.4% at day 273 (phenyl label) and to 27.2% at day 365 (pyridyl label). Fluopyram (parent) residues decreased to 59.9% at day 273 in the phenyl label experiment, and to 60.3% at day 365 in the pyridyl label experiment.

In the California sandy loam soil, the average extracted radioactive residues decreased from 99.3% (day 0) to 80.2% at day 365 in the phenyl label experiment, and from 99.3% (day 0) to 68.5 at day 272 in the pyridyl label experiment. Bound residues increased from 0.7% (day 0) to 9.4% at day 272 (phenyl label) and from 0.7% (day 0) to 10.6% at day 272 (pyridyl label). Radioactive carbon dioxide increased to 9.4% at day 365 (phenyl label) and to 14% at day 272 (pyridyl label). Residues of fluopyram (parent) decreased to 71.2% at day 365 (phenyl label) and to 61.3% at day 365 (pyridyl label).

Apart from CO_2, no metabolite of significance was detected in either soil and volatile residues were insignificant. No intermediate metabolites accumulated in the aerobic soil system.

Table 33 Degradation of [phenyl-^{14}C]-fluopyram and [pyridyl-2,6-^{14}C]-fluopyram in soils under aerobic conditions, expressed as a percentage of the applied radioactivity.

Compound	Soil	Radio-label	Sampling Times (days)											
			0	3	7	14	30	59 [a] 60 [b]	90 [a] 91 [b]	120 [a] 133 [b]	150 [a] 157 [b]	182 [a] 183 [b]	272 [a] 273 [b]	365
Fluopyram	silty clay loam (NE)[a]	phenyl	96.3	93.8	90.1	78.2	78.2	74.4	68.2	73	71.5	69.6	59.9	60.7
		pyridyl	97.4	92.3	89.6	82.1	85.4	77.1	70.5	76.7	73.2	68.2	63.2	60.3
	sandy loam (CA) [b]	phenyl	98.9	98.2	98.6	94.2	88	92.7	89.2	83.2	82.1	79.3	75.8	71.2
		pyridyl	98.5	96.5	89.2	87.4	88.7	83.3	86.5	78.9	79.9	78.3	61.4	61.3
Unknown A	silty clay loam (NE)	phenyl	0.2	0.6	0.7	0.9	1	0.5	0.2	0.4	-	-	0.2	0.1
		pyridyl	0.1	0.6	0.7	0.9	1.2	-	0.8	-	-	-	0.1	0.1
	sandy loam (CA)	phenyl	0.4	0.6	-	-	-	-	-	-	-	-	-	0
		pyridyl	0.2	0.6	1.3	-	-	-	-	-	-	-	0.3	0
Unknown B	silty clay loam (NE)	phenyl	0.3	0.2	0	0.2	-	-	-	-	-	-	-	0
		pyridyl	0.2	0.3	0	0.4	0.4	-	-	-	-	-	-	0
	sandy loam (CA)	phenyl	0	0.2	0.3	0.4	-	-	-	-	-	-	0.6	0
		pyridyl	0.4	0.3	0.4	0.3	-	-	-	-	-	-	0.7	0
Unidentified	silty clay loam (NE)	phenyl	1.1	0.8	0.7	1	1	0.5	0.2	0.4	-	-	0.2	0.1
		pyridyl	0.5	0.8	0.7	1.4	1.6	-	0.8	-	-	-	0.1	0.1
	sandy loam (CA)	phenyl	0.4	0.8	0.3	0.4	-	-	-	-	-	-	0.6	0
		pyridyl	0.8	0.8	1.7	0.3	-	-	-	-	-	-	0.9	0
ASE extract	silty clay loam (NE)	phenyl	n.e	n.e	n.e	7.7 [c]	9.4 [c]	10.4 [c]	9.9 [c]	[d]	[d]	[d]	[d]	[d]
		pyridyl	n.e	n.e	n.e	7.1 [c]	8.2 [c]	10.5 [c]	11.7 [c]	[d]	[d]	[d]	[d]	[d]
	sandy loam (CA)	phenyl	n.e	n.e	n.e	n.e	n.e	2.8 [c]	2.7 [c]	3.3 [c]	3.1 [c]	3.8 [c]	5.7 [c]	9.0 [c]
		pyridyl	n.e	n.e	n.e	n.e	n.e	2.9 [c]	2.3 [c]	3.7 [c]	3.4 [c]	4.1 [c]	6.2 [c]	9.1 [c]

Compound	Soil	Radio-label	Sampling Times (days)											
			0	3	7	14	30	59 [a] 60 [b]	90 [a] 91 [b]	120 [a] 133 [b]	150 [a] 157 [b]	182 [a] 183 [b]	272 [a] 273 [b]	365
Total Extractable*	silty clay loam (NE)	phenyl	97.4	94.6	90.8	86.9	88.5	85.2	78.4	73.4	71.5	69.6	60.1	60.8
		pyridyl	98	93.1	90.4	90.6	95.2	87.6	83.1	76.7	73.2	68.2	63.3	60.5
	sandy loam (CA)	phenyl	99.3	99	98.9	94.6	88	95.6	91.9	86.6	85.2	83.1	82.1	80.2
		pyridyl	99.3	97.4	90.9	87.6	88.7	86.2	88.8	82.7	83.3	82.4	68.5	70.4
CO_2	silty clay loam (NE)	phenyl	n.e	0.5	0.8	2.2	5.1	8.5	12.2	14.3	16.3	17	24.4	21.6
		pyridyl	n.e	0.8	0.8	1.7	4.5	7.8	12.1	14.3	18.3	19	23.9	27.2
	sandy loam (CA)	phenyl	n.e	0.3	0.6	0.6	0.9	2.9	3.5	4.1	5.1	6.7	6.8	17.3
		pyridyl	n.e	0.2	0.4	0.6	1.2	2.5	3.6	4.9	5.4	6.5	14	12.4
Total Volatile	silty clay loam (NE)	phenyl	0	0.5	0.9	2.2	5.1	8.5	12.2	14.4	16.3	17.1	24.4	21.6
		pyridyl	0	0.8	0.9	1.8	4.5	7.8	12.1	14.3	18.4	19	23.9	27.2
	sandy loam (CA)	phenyl	0	0.3	0.6	0.6	0.9	2.9	3.5	4.2	5.2	6.7	6.8	17.3
		pyridyl	0	0.2	0.4	0.6	1.2	2.5	3.6	5	5.5	6.5	14	12.4
Bound Residues	silty clay loam (NE)	phenyl	2.6	8.2	9.2	3.6	7.3	9.1	10.5	11.1	13.2	13	14.9	14.1
		pyridyl	2	7	10.4	5	7.3	9.4	10.2	11.6	13.7	13.8	13.6	14.7
	sandy loam (CA)	phenyl	0.7	1.7	2.7	4.5	6.2	4.5	6.1	7	6.5	8.3	9.4	9
		pyridyl	0.7	1.4	3.1	4.1	4.7	4.7	4.8	6.2	6.1	6.8	10.6	8.3
Total% Recovery	silty clay loam (NE)	phenyl	100	103.3	100.9	92.7	100.9	102.8	101	98.8	101	99.7	99.4	96.5
		pyridyl	100	101	101.6	97.4	107	104.8	105.4	102.6	105.2	101	100.8	102.4
	sandy loam (CA)	phenyl	100	101	102.1	99.8	95.1	103	101.5	97.7	97	98.1	98.3	106.6
		pyridyl	99.9	99	94.4	92.3	94.6	93.3	97.1	93.8	94.9	95.7	93.1	91.1

[a] Nebraska silty clay loam (sand 12:silt 60:clay 32), 2.9% OM, CEC 18.7 meq/100 g

[b] California sandy loam (sand 65:silt 21:clay 14), 0.9% OM, CEC 11.5 meq/100 g

n.e not extracted

[c] extract not analysed by HPLC to identify residue component

[d] ASE extracts were analysed by HPLC and included in the v fluopyram and other components above.

The degradation/dissipation rates of fluopyram were calculated using a simple first-order (SFO) kinetics model to calculate estimated half-lives of 922 days (California sandy loam) and 484 days (Nebraska silty clay loam).

Table 34 Degradation calculations of fluopyram in two US soils under aerobic conditions (Simple First Order model)

Soil/Study site	First order kinetic		Chi^2
	DT_{50} [days]	DT_{90} [days]	
Sandy loam - Porterville, CA	922	> 1000	2.4
Silty clay loam - Springfield, NE	484	> 1000	2.7

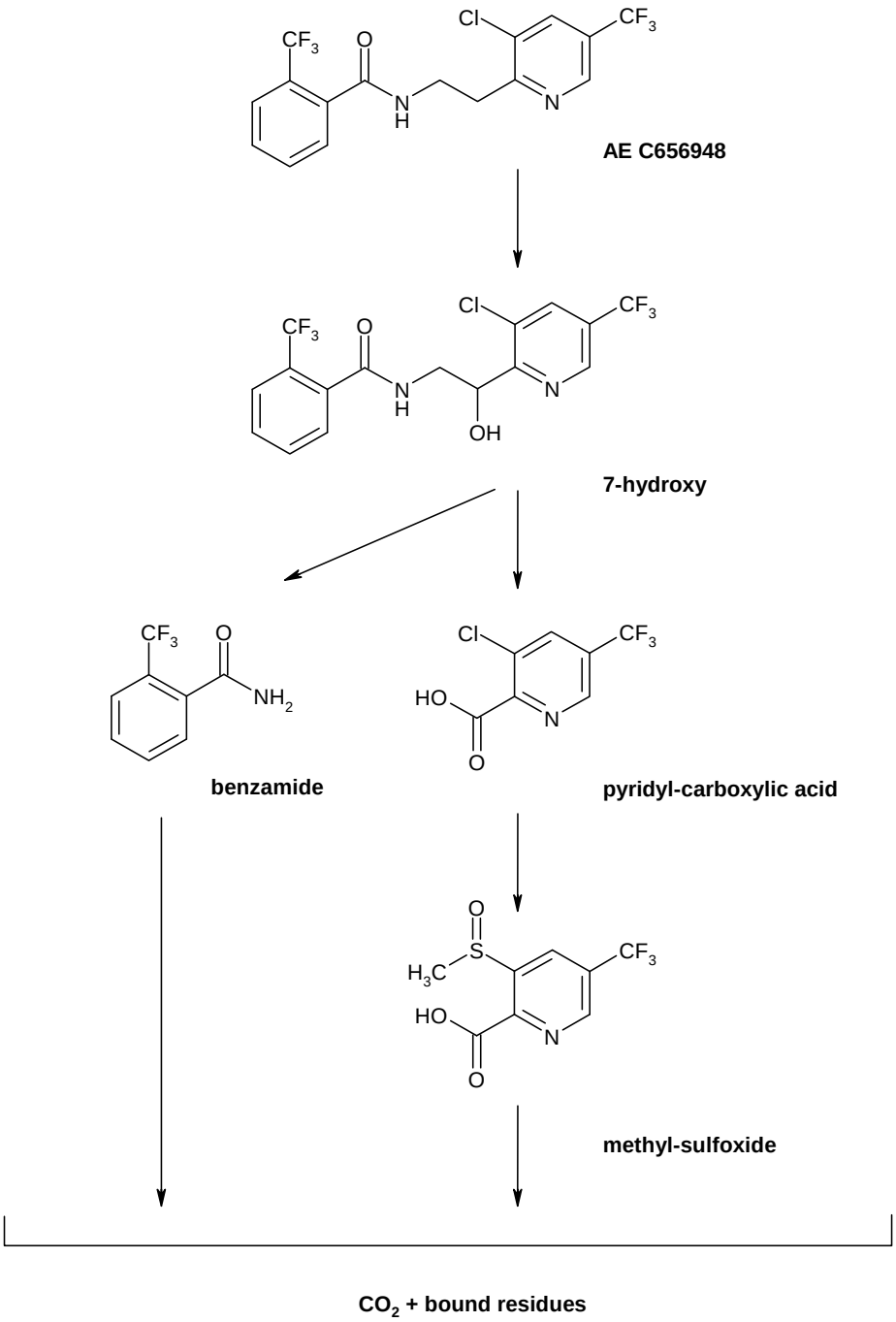


Figure 3 Proposed degradation pathway of AE C656948 in soil

Soil photolysis

In a study reported by Doble & Oddy, 2007 [Ref: CX/06/022], the photo-transformation of fluopyram was investigated on a sandy loam soil at 20 °C and 75% soil moisture at 1/3 bar. [Phenyl-UL-^{14}C]-fluopyram was applied to the soil surface at a rate 7.8 µg/g soil, approximating a field use of 250 g ai/ha and the treated soil was continuously exposed to artificial irradiation (xenon lamp with < 290 nm cut-off filter, light intensity 276 Wm^{-2}) for 23 days. Duplicate samples were taken after 1, 3, 7, 14 and 23 days exposure and analysed for fluopyram and transformation products. Soil samples were triple-

extracted with acetonitrile/water and analysed by HPLC with radioactivity detection after concentration. Identification and confirmation was performed by HPLC with comparison to certified analytical standards, TLC and LC/MS.

Extractable radioactivity, as the fluopyram parent compound, ranged from 99–105% of the applied radioactivity (AR) over the 23-day study period, with about 0.2% of the applied dose being mineralized to $^{14}CO_2$ and no organic volatiles were detected. Non-extractable residues increased from 0.4% AR to 2.3% AR at the end of the study.

Table 35 Phototransformation of [phenyl-UL-^{14}C]-fluopyram on a sandy loam soil, expressed as percentage of applied radioactivity

Compound		Sampling times (days)					
		0	1	3	7	14	23
Total extractable residues (fluopyram)	irradiated	105.2	100.2	99.3	101.4	100.7	101.4
	dark	105.2	97.4		104	103	99.5
Non-extractable residues	irradiated	0.7	0.4	0.7	0.9	1.6	2.3
	dark	0.7	0.4		0.9	2.2	2.7
CO_2	irradiated	-	-	-	0.2	0.1	0.2
	dark	-	-		0.2	0.1	0.1
Volatile organic compounds	irradiated	-	-	-	-	-	-
	dark	-	-		-	-	-
Total% recovery	irradiated	105.8	100.6	100	102.4	102.4	103.9
	dark	105.8	97.8		105.1	105.3	102.3

Soil: Porterville sandy loam (sand 68:silt 22:clay 10), 1.03% OM, CEC 7.4 meq/100 g

The study showed that fluopyram was photolytically stable under the test conditions. Mineralisation during the course of the study was minimal (about 0.2% AR). No degradation products were formed. The extractable part was identified as unchanged parent compound. Photolysis on soil surfaces is not considered a relevant degradation pathway.

Soil dissipation – field studies

Study 1: European field studies

Soil dissipation of fluopyram under European field conditions was investigated in a study reported by Heinemann & Telscher, 2007 [Ref: RA-2052/05]. Bare soil plots at six sites in Europe were sprayed with the equivalent of 250 g ai/ha fluopyram (250 SC) in 300 litres water and grass was sown within 10 days after application. Application was with knapsack sprayers with 3–8 nozzle booms. Soil samples (to 50 cm deep) were taken 0 to 740 days after application, deep-frozen within 24 hours and stored at -18 °C for up to 409 days before analysis.

Soil samples were microwave extracted in acetonitrile/water and analysed by HPLC using MS/MS detection in the Multiple Reaction Monitoring mode (BCS Method No.: 00973). The overall mean recovery of the method was 101% (RSD of 2.2%) and the LOQ was 0.005 mg/kg.

Quantifiable residues of fluopyram were detected to a maximum depth of 20 cm (with residues < LOQ being found at a single sampling event in the 20–30 cm soil layer). Fluopyram residues at the end of the study periods were between 3.2% and 26.5% of the applied amount.

Table 36 Fluopyram soil dissipation in six European soils after grass pre-emergent spraying with the equivalent of 250 g ai fluopyram/ha

	Burscheid (Germany)	Little Shelford (UK)	Staffanstorp (Sweden)	Vatteville (France)	Vilobi d'Onyar (Spain)	Albaro (Italy)
Site	R2005 0326/1	R2005 0328/8	R2005 0329/6	R2005 0331/8	R2005 0332/6	R2005 0333/4
Soil type [a]	silt loam	sandy loam	loam	silt loam	loam	silt loam
% OC [b]	1.07	1.08	1.38	0.79	0.68	1.21
pH (water)	6.9	8.1	8.1	7.3	6.7	8.2
S:S:C [c]	15:65:20	64:21:15	51:30:19	18:70:12	47:38:15	22:53.5:24.5
CEC [d]	11.3	16.4	18	6.9	7.4	19.1
Bulk density[e]	1.29	1.26	1.27	1.28	1.28	1.65
DAT [f]	(%) [g]	(%) [g]	(%) [g]	(%) [g]	(%) [g]	(%) [g]
0	91.9	106	111	85	92.1	83.7
12-15	72.3	100	73.7	86.2	57.7	46.5
25-28	74.7	78.1	76.5	64.6	61.3	38.4
55-56	68.7	63.8	73.7	66.2	68.5	39.8
100-107	51.1	65.4	63.3	52.3	49.7	27.9
146-168	40.8	49.8	50.1	65.4	13.5	22.3
201-223	34.7	58.2	50.5	53.1	36.2	20.2
266-273	29.9	46.6	45.7	38.3	26.5	17.8
363-377	30.2	38	41.7	48.3	19.1	13.6
479-485	21.2	39.4	27.5	31.5	6.6	8.1
592-613	14.1	27.5	28.9	26.1	3.6	9.5
726-740	16	24.9	26.2	26.5	3.2	6.5

[a] soil type of the 0-30 cm depth core sample

[b] % organic carbon

[c] sand:silt:clay composition

[d] cation exchange capacity (meq/100 g)

[e] soil bulk density in g/cm^3

[f] days after treatment

[g] % of the applied residue (250 g ai/ha equiv)

Kinetic modelling was used to estimate the fluopyram dissipation rate with the goodness of fit being assessed by visual inspection and an error criterion based on a chi-square ($\chi 2$) significance test. The coefficient of determination (R^2) was also used as a secondary measure of goodness of fit. Four kinetic models were considered: single first-order (SFO), first-order multiple-compartment (FOMC), the hockey-stick model (HS) and the double-first-order in parallel (DFOP).

The results of the modelling indicated that fluopyram exhibits abi-phasic degradation behaviour. Fluopyram DT_{50} values ranged from 21 to 347 days and the estimated DT_{90} values were from 497 to > 1000 days.

Table 37 Calculated soil dissipation times for fluopyram in six European soils

Location and Trial No.	Kinetic Model	DT_{50} [days]	DT_{90} [days]	Visual Assessment [a]	Chi^2 error
Burscheid, Germany R 2005 0326/1	SFO	206	684	o	11.0
	FOMC	146	> 1000	+	7.0
	DFOP [b]	146	> 1000	+	7.3
Little Shelford, UK R 2005 0328/8	SFO	315	> 1000	o	13.6
	FOMC	149	> 1000	+	8.0
	DFOP [c]	239	> 1000	+	8.7

Location and Trial No.	Kinetic Model	DT_{50} [days]	DT_{90} [days]	Visual Assessment [a]	Chi^2 error
Staffanstorp, Sweden R 2005 0329/6	SFO	312	> 1000	o	13.6
	FOMC	128	> 1000	+	9.1
	DFOP	179	> 1000	+	5.9
Vatteville, France R 2005 0331/8	SFO	391	> 1000	o	11.0
	FOMC	319	> 1000	+	10.4
	DFOP	347	> 1000	+	10.1
Vilobi d'Onyar, Spain R 2005 0332/6	SFO	147	487	+	22.1
	FOMC	123	586	o	22.6
	DFOP	100	497	+	20.0
Albaro, Italy R 2005 0333/4	SFO	118	391	--	26.9
	FOMC	27.5	> 1000	+	10.5
	DFOP	21.3	512	+	7.0

[a] Visual assessment of the model graphs: + good, o medium, -- bad

[b] The fit of the DFOP and FOMC model are similar (visual assessment and marginal difference in chi^2 error). The DFOP model was chosen as best fit for long term assessments, especially where residues > 10% applied dose remain at study end.

[c] The DFOP model was chosen as best fit despite a marginally higher chi^2 error because the FOMC model overestimates the last two data points (day 613, 740), crucial for the long term behaviour of the FOMC model.

Further modelling of the data from these six European soil studies was conducted by Kley & Ellerich, 2008 [Ref: MEF-07/448] to derive kinetic soil dissipation parameters for modelling and environmental risk assessments according to FOCUS kinetics. Temperature and moisture normalised DT_{50} values were derived for standard reference conditions (20 °C, 100% field capacity). Selection of the most appropriate kinetic model was based on a detailed statistical analysis including visual assessment, chi^2 statistics, significance t-test and correlation analysis.

After the kinetic evaluation of the field dissipation trials comparing simple first-order (SFO) and biphasic models, the best description could be given using a SFO fit for 5 sites and a double-first-order in parallel (DFOP) fit for one site (Albaro).

The overall geometric mean DT_{50} for modelling purposes according to FOCUS kinetics, was calculated as 118.8 days (normalised to 20 °C, 100% field capacity).

Table 38 Modelling parameter of field dissipation of fluopyram, normalised to 20 °C, 100% field capacity, using single first-order (SFO) or double-first-order in parallel (DFOP) fit

Site	Kinetic model	$C_{t\,0}$	k	DT_{50} total field	error ε of chi^2-test	R^2
		g/ha	1/d	d	%	
Hoefchen, D	SFO	207.0	0.0067	103.5	7.2	0.974
Little Shelford, UK	SFO	226.8	0.0048	144.6	12.8	0.868
Staffanstorp, S	SFO	218.5	0.0058	120.5	12.6	0.872
Vatteville, F	SFO	198.2	0.0050	139.7	10.3	0.881
Vilobí d'Onyar, E	SFO	202.5	0.0074	93.2	20.9	0.887
Albaro, I	DFOP	209.2	0.0058 [d]	119.5 [a]	6.4	0.991
Geomean				118.8		

[a] rate k and DT_{50} represent the second slow phase of a DFOP fit, appropriate for FOCUS kinetics modelling

Study 2: North American field studies

Five field soil dissipation studies were conducted in USA, involving bare soil plots treated with 0.5 kg ai/ha fluopyram (SC 500), applied in about 200 litres of water. The plots were irrigated to reflect at

least 110% of the long term average rainfall or to reflect irrigation practices (125–131% of orchard evapotranspiration).

Soil core samples were taken at day-0 and at various intervals over the subsequent 18 months, with additional samples being taken after about 22 months in three sites. Soil cores were collected to a depth of 122 cm, separated into eight 15 cm segments and stored frozen for up to 22 months before microwave-assisted extraction with acetonitrile/water and LC/MS/MS analysis. The limit of quantitation (LOQ) was 1.0 µg/kg for fluopyram and its transformation products with method detection limits of 0.2 µg/kg for fluopyram, 7-OH, BZM and 0.3µg/kg for PCA.

Initial residues of fluopyram in the top 15-cm soil layers ranged from 111–286 µg/kg with 21–44% remaining in soil at the end of the study periods. Final residues in the top 15 cm ranged from 6–30% of the initial concentration with 2-20% being measured at depths below 30 cm.

Most of the fluopyram residues remained in the top 30 cm segments with occasional detections at depths greater than 30 cm (less than 1% of the initial concentration during the first 12 months of the study and less than 3% after 2 years). In two sites involving high irrigation rates (Washington and California), about 11–12% of the initial concentration was found below 30 cm after 2 years, about 10% in the 30–60cm band and about 2% in the 60–90 cm band. The California site also reported residues below 90 cm after about 9 months, up to 4% of the initial concentration after about 18 months and 1.5% after 22 months.

Table 39 Average residues of fluopyram in moisture-corrected soil samples collected from bare soil plots in USA, treated with 0.5 kg ai/ha fluopyram (SC 500)

Site	Soil depth (cm)	Days After Treatment (DAT)													
		0	3	6-7	14-15	30	58-64	90-93	115-129	179-192	267-276	316	361-385	497-545	665-673
		Average Moisture-corrected Residues (µg/kg)													
Washington sandy loam MEGMP065 [a]	0-15	285.7	248.5	246.6	240.6	200.8	164.6	141.4	134.5	132.6	102.2		87.9	36.7	22.9
	15-30		(0.33)	(0.91)	(0.58)	1.57	4.4	1.49	10.9	5.96	22.2		24.2	41.9	23.1
	> 30		N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		(0.42)	11.8	33.32
	Total	285.7	248.8	247.5	241.2	202.3	169	142.9	145.4	138.6	124.4		112.5	90.4	79.3
New York loamy sand MEGMP084 [b]	0-15	262.6	197.3	234.5	190.4	154.9	153.1	155.6	189.2	140.8	176.8		116.4	117.6	80.6
	15-30		N.D.	(0.46)	N.D.	(0.65)	1.09	(0.54)	N.D.	N.D.	1.78		1.76	8.33	17.2
	> 30		N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	(0.28)		(0.33)	6.35	8.96
	Total	262.6	197.3	234.9	190.4	155.5	154.2	156.1	189.2	140.8	178.9		118.5	132.3	106.8
Nth Dakota loam MEGMP085 [c]	0-15	257.3	232.5	220.6	209.6	166.4	123.3	118.7	133.8			125.7	117.5	113.1	
	15-30		(0.77)	2.14	(0.86)	N.D.	N.D.	N.D.	N.D.			N.D.	(0.27)	(0.24)	
	> 30		N.D.	(0.38)	N.D.	N.D.	N.D.	N.D.	N.D.			N.D.	(0.22)	N.D.	
	Total	257.3	233.3	223.3	210.5	166.4	123.3	118.7	133.8			125.7	118	113.4	
Georgia loamy sand MEGMP086 [d]	0-15	153.9	131.1	119.9	95	89.8	62.2	75.7	59.6	68.6	54.3		51.9	37	
	15-30		94.37	(0.83)	3.73	(0.28)	N.D.	N.D.	N.D.	N.D.	(0.48)		(0.83)	3.26	
	> 30		1.56	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		N.D.	1.79	
	Total	153.9	227	120.8	98.8	90.1	62.2	75.7	59.6	68.6	54.8		52.7	40.9	
California sandy loam MEGMP087 [e]	0-15	110.8	130.9	136.3	76	94.1	60.6	40.5	58.6	45	31.2		18.2	6.98	6.78
	15-30		1.2	N.D.	1.36	7.63	9.25	11.8	12.4	14	15.7		10	8.45	6.07
	> 30		N.D.	N.D.	N.D.	1.91	(0.22)	4.99	4.02	8.77	14.51		23.03	22.69	12.09
	Total	110.8	132.3	136.4	77.5	103.6	70.1	57.3	75.1	67.8	61.4		51.2	38.1	23.3

[a] soil characteristics: (sand 80:silt 15:clay 6), 0.8% OM, pH (water) 8.1, CEC 9.3 meq/100 g

[b] soil characteristics: (sand 87:silt 9:clay 5), 2.2% OM, pH (water) 6.2, CEC 7 meq/100 g

[c] soil characteristics: (sand 34:silt 41:clay 26), 5.8% OM, pH (water) 7, CEC 21.9 meq/100 g

[d] soil characteristics: (sand 84:silt 10:clay 7), 0.8% OM, pH (water) 5.9, CEC 4.5 meq/100 g

[e] soil characteristics: (sand 57:silt 28:clay 16), 1.1% OM, pH (water) 7.8, CEC 9.7 meq/100 g

Note: values in brackets are reported below the LOQ

The observed transformation products were 7-OH, BZM, and PCA metabolites. The highest concentrations of the transformation products summed from each soil segment in the soil core were 3.0 µg/kg (7-OH), 9.7 µg/kg (BZM) and 10 µg/kg (PCA). Residues of BZM and PCA were below the LOQ at all sites within 30 days after treatment. In four of the sites, residues of 7-OH were below the LOQ after 9 months and at the New York site, were present at about 1.6 µg/kg (mostly in the top 15 cm segment) at the end of the 22-month study period.

Kinetic modelling was used to estimate the fluopyram dissipation rate with the goodness of fit being assessed by visual inspection and an error criterion based on a chi-square (χ^2) significance test. The coefficient of determination (R^2) was also used as a secondary measure of goodness of fit. Based on the chi-square error criterion and visual assessment a simple (single) first-order (SFO) kinetics and double first-order in parallel (DFOP) model were used to fit the measured data from bare soil plots for fluopyram.

The estimated degradation and half-life (DT_{50}) values ranged from 24.1 to 537 days. The time required for dissipation of 75% (DT_{75}) of the initial fluopyram concentration ranged from 502 to > 1000 days.

Table 40 Dissipation times for fluopyram in North American field soil dissipation studies

Site	Model	Estimated Initial Conc C_0 (µg/kg)	Rate Constant k1 (d^{-1})	Rate Constant k2 (d^{-1})	DT_{50} (days)	DT_{75} (days)	χ^2 (%)	R^2
Washington sandy loam	SFO	235.2	0.0023	-	295	603	12.9	0.79
	DFOP	273.4	0.0011	0.0307	166	784	3.7	0.95
New York loamy sand	SFO (replicates)	200.7	0.0010	-	677	> 1000	13.8	0.32
	DFOP (replicates)	250.4	0.1032	5.8×10^{-4}	537	> 1000	10.0	0.47
	DFOP (averaged)	250.5	0.1031	5.8×10^{-4}	537	> 1000	10.0	0.79
Nth Dakota loam	SFO	203.6	0.0018	-	382	770	17.0	0.52
	DFOP	253.9	8.9×10^{-5}	0.0373	86.7	> 1000	3.7	0.92
Georgia loamy sand	SFO	138.5	0.0047	-	147.5	295	31.1	0.50
	DFOP	185.2	0.0672	8.8×10^{-4}	24.1	502	21.0	0.75
California sandy loam	SFO	107.3	0.0024	-	284	578	18.2	0.54
	DFOP	125.7	0.0014	0.0430	175	665	14.6	0.63

Fluopyram showed abi-phasic degradation behaviour under the investigated North America conditions. The calculated DT_{50} dissipation times ranged between 24 and 537 days and at one site, fluopyram residues were observed below 90 cm, attributed to the excessive input of water to the bare soil plots. No residue of transformation products was found above the LOQ below the top 15 cm.

Table 41 Dissipation routes and conceptual model for fluopyram and transformation products in soil

Route of Dissipation	Washington sandy loam (385 DAT)	New York loamy sand (365 DAT)	North Dakota loam (363 DAT)	Georgia loamy sand (372 DAT)	California sandy loam (361 DAT)
Soil residue of fluopyram (% zero-time concentration)	39.4%	45.1%	45.9%	34.3%	46.2%
Transformation products [a]					
7-OH	0.3%	0.8%	0.3%	0.1%	0%
BZM	0%	0%	0%	0%	0%
PCA	0%	0%	0.5%	0%	0.6%
Leaching, if measured	0.0% residues not below 46 cm	0.0% residues not below 46 cm	0.0% residues not below 46 cm	0.0% residues not below 61 cm	1.1% fluopyram residue found at 107 -122 cm

Route of Dissipation	Washington sandy loam (385 DAT)	New York loamy sand (365 DAT)	North Dakota loam (363 DAT)	Georgia loamy sand (372 DAT)	California sandy loam (361 DAT)
Mineralization to CO2 [b]	27.2% of applied radioactivity liberated as CO_2 after one year (radiolabelled soil metabolism studies [Ref: MEGMP069])				
Non-extractable residues [b]	14.7% applied radiolabel-fluopyram not extracted by exhaustive extraction (radiolabelled soil metabolism studies [Ref: MEGMP069])				
Volatilization, if measured	0.0% (fluopyram is non-volatile - vapour pressure of 3.1 × 10-6 Pa at 25 °C)				
Plant uptake, if measured	0.0% - not measured				
Run off, if measured	0.0% - not measured since the test plots had a slope of 0-1%.				
Total accountability	81.6%	87.0%	88.6%	76.3%	89.8%

[a] Concentrations of transformation products (parent equivalent) as % of the initial fluopyram residue (day 0)

[b] Maximum/mean values derived from laboratory study

Soil accumulation – field studies

The interim results of the accumulation of fluopyram in soil under European field conditions following three annual fluopyram applications were reported by Heinemann & Telscher, 2007 [Ref: MEF-07/288]. In this study, fluopyram (250 SC) was applied annually at nominal rate of 250 g ai/ha in 300 L water/ha on bare harrowed and levelled soil plots at two sites in Monheim (Germany) and Tarascon (France) using knapsack sprayers with 3-8 nozzle spraying booms. Assuming a soil density of 1.5 kg/L, this would equate to a concentration of 167 µg/kg in the top 10 cm soil after a single application and without any degradation, would lead to a concentration of 334 µg/kg and 501 µg/kg after two and three consecutive applications.

Soil samples were taken before and after the annual applications and before winter dormancy. Samples were taken to a maximum depth of 50 cm before and after each application (end of winter) and before winter dormancy each year and stored at or below - 18 °C until analysis. Samples were microwave-extracted in acetonitrile/water and analysed by HPLC using MS/MS detection in the Multiple Reaction Monitoring mode (Method 00973), with an overall mean recovery of 101% (RSD 2.2%) and an LOQ of 5 µg/kg (LOD of 1.5 µg/kg).

Maximum fluopyram residues of 228–237 µg/kg were found in the 0–10 cm layers and 4.8–13.4 µg/kg maximum were measured in the 10–20 cm layers. Fluopyram residues were below the LOQ in all soil samples below 20 cm. Total fluopyram residues in soil increased from 39 µg/kg (Monheim) and 77 µg/kg (Tarascon) after one year, just before the 2nd application, to 86 µg/kg and 110 µg/kg respectively after 2 years, just before the 3rd application.

Table 42 Fluopyram residues in two European soils following three bare ground applications of 0.25 kg ai/ha fluopyram (250 SC).

Site		Fluopyram residues in soil (µg/kg)						
	DAT (days)	0	194-215	334-353[a]	335-354[b]	565-570	697-710[a]	698-711[b]
Monheim sandy loam [c)] R 2005 0334/2	0–10 cm	135	39.9	38.0	146	69.2	80.9	228
	10 -20 cm	0.00	0.75	0.75	3.30	2.38	4.08	4.82
	20 -30 cm	0.00	0.00	0.00	0.75	0.75	0.75	0.75
	> 30 cm	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Total (0-50 cm)	135	40.7	38.8	150	72.3	85.7	234
Tarascon silt loam [d)] R 2005 0335/0	0–10 cm	143	71.5	70.3	179	99.8	95.7	237
	10–20 cm	2.66	3.80	5.96	4.90	6.78	13.35	9.62
	20–30 cm	0.00	0.75	0.75	2.24	0.75	0.75	2.76
	> 30 cm	0.00	0.00	0.00	0.75	0.00	0.00	0.75
	Sum 0–50 cm	145	76.1	77.1	187	107	110	250

[a] mean concentration at the latest sampling date for the present interim report before the next application

[b] upper limit of the "saw teeth" curve (mean values of several soil samples per sampling date)

[c] soil characteristics (0-30cm): (sand 72:silt 18:clay 10), 0.89% OM, pH (water) 6.9, CEC 5.8 meq/100 g

[d] soil characteristics (0-30 cm): (sand 18:silt 63:clay 19), 0.67% OM, pH (water) 8.3, CEC 13.1 meq/100 g

Residues in rotational crops

Confined accumulation studies

Study 1: phenyl-labelled fluopyram

The metabolism of fluopyram in wheat, Swiss chard and turnips grown in three consecutive crop rotations.was studied by Klempner, 2008 [Ref: MEF-07/412] [Phenyl-UL-^{14}C]-fluopyram was sprayed onto bare sandy loam soil (2.3% organic matter, pH 6.4, CEC 5.9 meq/100 g) at a rate equivalent to 534 g ai/ha and crops of the 1st, 2nd and 3rd rotation were sown with plant-back intervals (PBIs) of 30, 139 and 280 days.

Wheat samples were taken at BBCH 29–31 (as forage), at BBCH 77-83 (and dried as hay) and at maturity (as grain and straw). The Swiss chard (without roots) and whole turnip plants (as leaves and roots) were sampled at maturity. Samples were stored at ca -18 °C for up to 4 weeks before extraction and analysis.

Total radioactive residues (TRR) above 0.01 mg/kg were found in all rotated crop matrices from all PBIs, except turnip roots from the 280-day PBI. TRR ranged from 0.009 mg/kg in turnip roots planted 280 days after soil application to 6.156 mg/kg in mature wheat straw planted 30 days after soil application. TRR generally declined with the later plant-back intervals. The exception was wheat forage where TRR increased in the 139-day PBI samples to ~2× the initial value but then decreased in the 280-day PBI sample.

Table 43 Total Radioactive Residues (TRRs), expressed as parent compound equivalents, mg/kg) in wheat, Swiss chard and turnips from three rotations in soil treated with [phenyl-UL-^{14}C]-fluopyram

TRR	Wheat				Swiss	turnip	
[mg/kg]	forage	hay	straw	grain	chard	leaves	roots
1st rotation (30 days)	0.100	1.783	6.156	0.167	0.540	0.884	0.065
2nd rotation (139 days)	0.785	1.120	3.450	0.054	0.377	0.113	0.013
3rd rotation (280 days)	0.197	1.527	1.032	0.023	0.164	0.103	0.009

Conventional extraction of the samples using acetonitrile/water released > 96% of the radioactive residues in Swiss chard and turnip leaves and roots, 87–95% of the radioactive residues in wheat forage, hay and straw and 77–85% of the radioactive residues in wheat grains. Turnip roots from the 280-day PBI were not subjected to extraction procedures due to low TRR.

The post extraction solids of wheat hay (all rotations) and wheat straw (1st and 2nd rotation) after conventional extraction were exhaustively extracted using acetonitrile/water in a microwave with increased temperature. Microwave extraction released further 3–5% of the TRR in wheat hay and straw. Conventional and microwave extracts of wheat hay and straw showed similar metabolite patterns. Diastase (starch cleaving enzyme) treatment of the post extraction solids of wheat grain released further a 9% of the TRR from solids of the 1st rotation, and 15% of the TRR from solids of the 2nd rotation. The solutions after diastase treatment were further characterised as polar and probably natural compounds by partitioning (1st rotation).

Unextracted residues were < 10% of the TRR in all rotated crop samples from all plant-back intervals, except the 280-day PBI wheat grain where the unextracted residues were 21.9% of the TRR, but represented 0.005 mg/kg.

Parent fluopyram and 28 metabolites were detected in the various samples of the three rotations. Of these fluopyram and 12 metabolites were identified by LC-MS and LC-MS/MS. The other 16 metabolites (unknowns) were characterised by their extraction and retention in radio-HPLC; each of them were < 0.04 mg/kg. Additionally, a very polar region was characterised in wheat grain accounting for up to 21.3% of the TRR (at a low level of 0.01 mg/kg) in the second rotation, this appeared to be due to assimilated $^{14}CO_2$ which was incorporated in the starch matrix.

Fluopyram accounted for the major part of the residues in all samples of all rotations and accounted for 56–84% of the TRR in the 1st rotation samples, 33–78% of the TRR in the 2nd rotation samples and 28–59% of the TRR in the 3rd rotation samples. In general, the levels of the parent compound decreased with subsequent plant-back intervals.

The 7-OH metabolite and its various conjugates with glucose, malonic acid (2 isomers) and sulphuric acid were important metabolites mainly in Swiss chard (non-conjugated metabolite at 21% of the TRR in the 1st rotation increasing to about 35% TRR in subsequent rotations). In the other samples, residues were < 10% TRR, except in wheat hay and straw from the 3rd rotation where they accounted for 12.3–12.6% TRR. The sulphuric acid conjugate of 7-OH was also a prominent metabolite in Swiss chard increasing from 7% of TRR in the 1st rotation to 16% and 12% of the TRR in the 2nd and 3rd rotation, respectively and was also detected at low levels in turnip leaves (0.7–1.0% TRR; 30 and 139-day PBIs).

The 8-OH metabolite and its conjugate were only of minor importance. Both or at least one of them were detected in all samples but at low levels (< 2.3% TRR in total). The phenol-glucoside was detected in turnip leaves only, where it amounted to 10%, 16% and 10% of the TRRs of the 1st, 2nd and 3rd rotation, respectively.

Two label-specific metabolites were identified: BZM and benzoic acid. The benzoic acid accounted for 0.6–6.9% TRR in wheat forage, hay and grain, and turnip leaves and roots from the 30-day PBI; 0.3–0.4% TRR in wheat forage and hay, and 13.6% TRR in wheat grain from the 139-day PBI; and 13% TRR in wheat grain from the 280-day PBI. The BZM metabolite accounted for 2.8–9.7% TRR in wheat forage, hay, straw and grain, and turnip leaves and roots, and 11.1% TRR in Swiss chard from the 30-day PBI; 3.2-7.4% TRR in all commodities from the 139-day PBI; and 5.9–8.0% TRR in wheat forage, hay, straw and grain, and 10.3–11.7% TRR in Swiss chard and turnip leaves from the 280-day PBI.

Table 44 Residues in wheat forage, hay and straw after a 30 day plant back interval (1st rotation, [phenyl-UL-^{14}C]-fluopyram)

1. Rotation		Wheat forage		Wheat hay		Wheat straw		Wheat grains	
	TRR [mg/kg]	0.1		1.783		6.156		0.167	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
26	fluopyram (parent)	74.8	0.075	76.9	1.370	74.1	4.557	61.9	0.104
2	-benzoic acid	1.7	0.002	0.6	0.011	-	-	6.9	0.012
4	-BZM	5.6	0.006	4.3	0.076	2.8	0.169	4.1	0.007
7	-7-OH-glc-MA (isomer 1)	3.2	0.003	5.1	0.090	4.5	0.278	1.7	0.003
8	-7-OH-glc-MA (isomer 2)	-	-	1.0	0.017	1.0	0.062	-	-
12A	-8-OH-glc-MA	1.0	0.001	0.8	0.014	0.7	0.046	-	-
12B	-7-OH-SA	-	-	-	-	-	-	-	-
13	-7-OH-glc [a]	2.2	0.002	1.4	0.026	2.8	0.169	1.2	0.002
17	-phenol-glc [b]	-	-	-	-	-	-	-	-
21	-7-OH	2.4	0.002	4.3	0.077	5.6	0.346	2.7	0.005
23	-8-OH	0.3	< 0.001	0.6	0.01	1.4	0.087	0.9	0.001
Total extractable		94.4	0.094	97.3	1.734	95.9	5.901	84.7	0.142
Total identified		91.3	0.091	94.9	1.691	92.9	5.715	79.4	0.133
Charact. by HPLC retention [c]		3.1	0.003	2.0	0.036	2.4	0.147	5.3	0.009

1. Rotation		Wheat forage		Wheat hay		Wheat straw		Wheat grains	
	TRR [mg/kg]	0.1		1.783		6.156		0.167	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Charact. by diastase treatment		-	-	-	-	-	-	8.5	0.014
Not analysed		-	-	0.4	0.007	0.7	0.040	-	-
Total bound residues (PES)		5.6	0.006	2.8	0.049	4.1	0.254	6.8	0.012
Accountability		100.0	0.100	100.0	1.783	100.0	6.156	100.0	0.167

[a] co-eluting with 8-hydroxy-glc-SA; representing about 20% of peak no. 13

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 16 minor metabolites characterised, all ≤ 5.1% TRR and < 0.04 mg/kg

Table 45 Residues in Swiss chard, turnip leaves and roots after a 30 day plant back interval (1st rotation, [phenyl-UL-^{14}C]-fluopyram)

1. Rotation		Swiss chard		Turnip leaves		Turnip roots	
	TRR [mg/kg]	0. 540		0.884		0.065	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
26	fluopyram (parent)	56.0	0.302	68.4	0.605	83.5	0.054
2	-benzoic acid	-	-	0.6	0.005	3.7	0.002
4	-BZM	11.1	0.060	9.7	0.086	4.0	0.003
7	-7-OH-glc-MA (isomer 1)	0.3	0.001	1.3	0.011	-	-
8	-7-OH-glc-MA (isomer 2)	-	-	0.9	0.008	-	-
12A	-8-OH-glc-MA	-	-	-	-	-	-
12B	-7-OH-SA	7.0	0.038	0.7	0.006	-	-
13	-7-OH-glc [a]	1.0	0.005	0.4	0.004	-	-
17	-phenol-glc [b]	-	-	10.4	0.092	-	-
21	-7-OH	20.9	0.113	3.3	0.029	3.6	0.002
23	-8-OH	0.7	0.004	0.3	0.003	0.9	0.001
Total extractable		99.4	0.536	99.5	0.880	98.7	0.064
Total identified		97.0	0.523	96.1	0.850	95.8	0.062
Characterised by HPLC retention [c]		2.2	0.012	3.4	0.030	2.9	0.002
Characterised by diastase treatment		-	-	-	-	-	-
Not analysed		0.3	0.001	-	-	-	-
Total bound residues (PES)		0.6	0.003	0.5	0.004	1.3	0.001
Accountability		100.0	0.540	100.0	0.884	100.0	0.065

[a] co-eluting with 8-OH-glc-sulfate; representing about 20% of peak no. 13

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 16 minor metabolites characterised, all ≤ 5.1% TRR and < 0.04 mg/kg

Table 46 Residues in wheat forage, hay and straw after a 139 day plant back interval (2nd rotation, [phenyl-UL-^{14}C]-fluopyram)

2. Rotation		Wheat forage		Wheat hay		Wheat straw		Wheat grains	
	TRR [mg/kg]	0.785		1.12		3.45		0.054	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
26	fluopyram (parent)	74.9	0.588	66.7	0.748	67.8	2.338	37.1	0.020
2	-benzoic acid	0.4	0.003	0.3	0.004	-	-	13.6	0.007

2. Rotation		Wheat forage		Wheat hay		Wheat straw		Wheat grains	
	TRR [mg/kg]	0.785		1.12		3.45		0.054	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
4	-BZM	3.5	0.028	3.7	0.041	3.2	0.111	3.3	0.002
7	-7-OH-glc-MA (isomer 1)	4.9	0.039	6.1	0.069	6.4	0.219	-	-
8	-7-OH-glc-MA (isomer 2)	1.3	0.010	0.7	0.008	1.3	0.046	-	-
12A	-8-hydroxy-glc-MA	1.2	0.009	1.4	0.016	1.2	0.043	-	-
12B	-7-OH-SA	-	-	-	-	-	-	-	-
13	-7-OH-glc [a]	2.0	0.016	2.0	0.023	2.8	0.097	-	-
17	-phenol-glc [b]	-	-	-	-	-	-	-	-
21	-7-OH	3.8	0.030	5.2	0.059	6.0	0.208	1.3	0.001
23	-8-OH	0.5	0.004	0.8	0.009	1.4	0.050	-	-
Total extractable		94.7	0.743	91.9	1.030	94.6	3.262	76.6	0.041
Total identified		92.4	0.726	87.2	0.977	90.2	3.111	55.3	0.030
Charact. by HPLC retention [c]		2.3	0.018	3.8	0.043	3.6	0.124	21.3	0.011
Charact. by diastase treatment		-	-	-	-	-	-	15.3	0.008
Not analysed		-	-	0.9	0.010	0.8	0.027	-	-
Total bound residues (PES)		5.3	0.041	8.0	0.090	5.4	0.188	8.1	0.005
Accountability		100.0	0.785	100.0	1.120	100.0	3.450	100.0	0.054

[a] co-eluting with 8-OH-glc-sulfate; representing about 20% of peak 13

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 14 minor metabolites characterised, 13 at ≤ 1.3% TRR and < 0.04 mg/kg, one at ≤ 21.3% TRR but very low levels (0.001-0.019 mg/kg)

Table 47 Residues in Swiss chard, turnip leaves and roots after a 139 day plant back interval (2nd rotation, [phenyl-UL-^{14}C]-fluopyram)

2. Rotation		Swiss chard		Turnip leaves		Turnip roots	
	TRR [mg/kg]	0.377		0.113		0.013	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
26	fluopyram (parent)	32.7	0.123	54.3	0.062	77.7	0.010
2	-benzoic acid	-	-	-	-	-	-
4	-BZM	7.4	0.028	5.6	0.006	5.8	0.001
7	-7-OH-glc-MA (isomer 1)	-	-	2.1	0.002	-	-
8	-7-OH-glc-MA (isomer 2)	-	-	0.8	0.001	-	-
12A	-8-OH-glc-MA	-	-	-	-	-	-
12B	-7-OH-SA	15.6	0.059	1.0	0.001	-	-
13	-7-OH-glc [a]	1.8	0.007	0.9	0.001	-	-
17	-phenol-glc [b]	-	-	16.2	0.018	-	-
21	-7-OH	35.4	0.134	6.3	0.007	4.1	0.001
23	-8-OH	0.9	0.003	0.4	< 0.001	2.3	< 0.001
Total extractable		98.8	0.373	98.7	0.112	96.7	0.013
Total identified		93.9	0.354	87.6	0.099	89.9	0.012
Characterised by HPLC retention [c]		4.9	0.019	11.1	0.013	6.8	0.001
Characterised by diastase treatment		-	-	-	-	-	-
Not analysed		-	-	-	-	-	-
Total bound residues (PES)		1.2	0.004	1.3	0.001	3.3	< 0.001
Accountability		100.0	0.377	100.0	0.113	100.0	0.013

[a] co-eluting with 8-OH-glc-sulfate; representing about 20% of peak 13

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 14 minor metabolites characterised, 13 at ≤ 1.3% TRR and < 0.04 mg/kg, one at ≤ 21.3% TRR but very low levels (0.001–0.019 mg/kg)

Table 48 Residues in wheat forage, hay and straw after a 280 day plant back interval (3rd rotation, [phenyl-UL-^{14}C]-fluopyram)

3. Rotation		Wheat forage		Wheat hay		Wheat straw		Wheat grains	
	TRR [mg/kg]	0.197		1.527		1.032		0.023	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
26	fluopyram (parent)	59.2	0.117	57.5	0.878	50.1	0.516	28.4	0.007
2	-benzoic acid	-	-	-	-	-	-	13.0	0.003
4	-BZM	8.0	0.016	6.2	0.095	6.6	0.068	5.9	0.001
7	-7-OH-glc-MA (isomer 1)	8.5	0.017	7.1	0.108	6.3	0.065	-	-
8	-7-OH-glc-MA (isomer 2)	1.2	0.002	4.0	0.062	2.9	0.030	-	-
12A	-8-OH-glc-MA	2.2	0.004	1.4	0.021	-	-	-	-
12B	-7-OH-SA	-	-	-	-	-	-	-	-
13	-7-OH-glc a	3.4	0.007	3.4	0.052	6.9	0.071	-	-
17	-phenol-glc [b]	-	-	-	-	-	-	-	-
21	-7-OH	6.3	0.012	12.6	0.193	12.3	0.127	3.4	0.001
23	-8-OH	0.5	0.001	1.1	0.016	1.3	0.014	-	-
Total extractable		92.4	0.182	95.7	1.461	90.8	0.937	78.1	0.018
Total identified		89.3	0.176	93.3	1.424	86.4	0.891	50.7	0.012
Charact. by HPLC retention [c]		3.1	0.006	1.5	0.023	3.3	0.034	27.4	0.006
Charact. by diastase treatment		-	-	-	-	-	-	-	-
Not analysed		-	-	0.9	0.014	1.1	0.011	-	-
Total bound residues (PES)		7.6	0.015	4.3	0.066	9.2	0.095	21.9	0.005
Accountability		100.0	0.197	100.0	1.527	100.0	1.032	100.0	0.023

[a] co-eluting with 8-OH-glc-sulfate; representing about 20% of peak no. 13

[b] co-eluting with -di-hydroxy-glc, which is only a very minor metabolite and not quantified

[c] up to 8 minor metabolites characterised, 7 at ≤ 7.6% TRR (< 0.01 mg/kg) and one at ≤ 19.9%

TRR but very low levels (0.004–0.034 mg/kg)

Table 49 Residues in Swiss chard, turnip leaves and roots after a 280 day plant back interval (3rd rotation, [phenyl-UL-^{14}C]-fluopyram)

3. Rotation		Swiss chard		Turnip leaves		Turnip roots	
	TRR [mg/kg]	0.164		0.103		0.009	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
26	fluopyram (parent)	33.7	0.055	58.6	0.060	Not extracted due to low residues < 0.01 mg/kg	
2	-benzoic acid	-	-	-	-		
4	-BZM	10.3	0.017	11.7	0.012		
7	-7-OH-glc-MA (isomer 1)	-	-	1.2	0.001		
8	-7-OH-glc-MA (isomer 2)	-	-	0.8	0.001		
12A	-8-OH-glc-MA	-	-	-	-		
12B	-7-OH-SA	12.1	0.020	-	-		
13	-7-OH-glc [a]	1.3	0.002	-	-		

3. Rotation		Swiss chard		Turnip leaves		Turnip roots	
	TRR [mg/kg]	0.164		0.103		0.009	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
17	-phenol-glc [b]	-	-	10.1	0.010		
21	-7-OH	34.9	0.057	8.2	0.008		
23	-8-OH	1.4	0.002	0.4	< 0.001		
Total extractable		98.4	0.161	99.0	0.102		
Total identified		93.6	0.153	91.0	0.094		
Characterised by HPLC retention [c]		4.8	0.008	8.0	0.008		
Characterised by diastase treatment		-	-	-	-		
Not analysed		-	-	-	-		
Total bound residues (PES)		1.6	0.003	1.0	0.001		
Accountability		100.0	0.164	100.0	0.103		

[a] co-eluting with 8-OH-glc-sulfate; representing about 20% of peak 13

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 8 minor metabolites characterised, 7 at ≤ 7.6% TRR (< 0.01 mg/kg) and one at ≤ 19.9% TRR but very low levels (0.004–0.034 mg/kg)

A significant decline of the radioactive residues was observed for all samples from crops planted 39, 139, and 280 days after application of [phenyl-U-^{14}C]-fluopyram to bare soil, except wheat forage and hay, where the residues remained at a similar level. The TRRs in all other samples declined by factors of 3–8. The TRR for all samples of the 3rd rotation apart from turnip roots exceeded 0.01 mg/kg.

Study 2: pyridyl-labelled fluopyram

A parallel rotational crop metabolism study involving pyridyl-labelled fluopyram was reported by Klempner, 2008 [Ref: MEF-07/413] in wheat, Swiss chard and turnips from three consecutive rotations. [Pyridyl-2,6-^{14}C]-fluopyram was sprayed onto bare sandy loam soil (2.3% organic matter, pH 6.4, CEC 5.9 meq/100 g) at a rate equivalent to 514 g ai/ha and crops of the 1st, 2nd and 3rd rotation were sown with plant-back intervals (PBIs) of 30, 139 and 280 days.

Wheat samples were taken at BBCH 29-31 (as forage), at BBCH 77-83 (and dried as hay) and at maturity (as grain and straw). The Swiss chard (without roots) and whole turnip plants (as leaves and roots) were sampled at maturity. Samples were stored at ca -18 °C for up to 4 weeks before extraction and analysis.

Total radioactive residues (TRR) above 0.01 mg/kg were found in all rotated crop matrices from all PBIs. TRR ranged from 0.01 mg/kg in turnip roots planted 280 days after soil application to 6.66 mg/kg in mature wheat straw planted 30 days after soil application. TRR generally declined with the later plant-back intervals, except in wheat forage which increased at the 139-day PBI, but decreased at the 280-day PBI, back to about the initial value.

Table 50 Total Radioactive Residues (TRRs), expressed as parent compound equivalents, mg/kg) in wheat, Swiss chard and turnips from three rotations in soil treated with [pyridyl-2,6-^{14}C]-fluopyram

TRR	Wheat				Swiss	turnip	
[mg/kg]	forage	hay	straw	Grain	chard	leaves	roots
1st rotation (30 days)	0.157	1.802	6.663	0.412	0.570	0.565	0.036
2nd rotation (139 days)	0.568	0.971	2.562	0.072	0.343	0.103	0.010
3rd rotation (280 days)	0.167	0.709	1.622	0.037	0.211	0.095	0.012

Conventional extraction of the samples using acetonitrile/water released > 97% of the radioactive residues in Swiss chard and turnip leaves and roots, 85–96% of the radioactive residues in wheat forage, hay and straw and 82–92% of the radioactive residues in wheat grains.

The post extraction solids of wheat hay (all rotations) and wheat straw (1st and 2nd rotation) after conventional extraction were exhaustively extracted using acetonitrile/water in a microwave with increased temperature. Microwave extraction released further 3 – 6% of the TRR in wheat hay and straw. Conventional and microwave extracts of wheat hay and straw showed similar metabolite patterns. Diastase (starch cleaving enzyme) treatment of the post extraction solids of wheat grain released further a 4% of the TRR from solids of the 1st rotation, and 9% of the TRR from solids of the 2nd rotation. The solutions after diastase treatment were further characterised as polar and probably natural compounds by partitioning (1st rotation).

Unextracted residues were < 10% TRR in all rotated crop samples from all plant-back intervals, except the 280-day PBI wheat grain where the unextracted residues were 17.8% TRR, but represented 0.007 mg/kg.

Parent fluopyram and 29 metabolites were detected in the various samples of the three rotations. Of these fluopyram and 12 metabolites were identified by LC-MS and LC-MS/MS. The other 17 metabolites (unknowns) were characterised by their extraction and retention in radio-HPLC; each of them were ≤ 0.011 mg/kg (0.1-2% of the respective TRRs) except in wheat straw, where the unknown metabolites comprised 0.4-1.3% TRRs (0.011–0.042 mg/kg).

Except for wheat grain, fluopyram accounted for the major part of the residues in all samples of all rotations and covered 57 – 86% TRR in the 1st rotation samples, 37–95% TRR in the 2nd rotation samples and 39 – 92% TRR in the 3rd rotation samples. In wheat grain, fluopyram accounted for 20.4–33.4% TRR in the three rotations.

The 7-OH metabolite and its various conjugates with glucose, malonic acid (2 isomers) and sulphuric acid were important metabolites mainly in Swiss chard (non-conjugated metabolite at 28% of the TRR in the 1st rotation increasing to about 38% TRR in subsequent rotations). In the other samples, residues were < 10.1% TRR. The 7-OH sulphuric acid conjugate was a prominent metabolite in Swiss chard increasing from 8% of TRR in the 1st rotation to 17% and 14% of the TRR in the 2nd and 3rd rotation, respectively and was also detected in turnip leaves at < 1% TRR from all plant-back intervals. The glucoside and glucoside-malonic acid metabolites of 7-OH were minor components, at < 10% TRR in wheat forage, hay and straw and in Swiss chard and turnip leaves.

The 8-OH metabolite and its conjugate were only of minor importance. Both or at least one of them were detected in all samples but at low levels (< 2.9% TRR in total). The phenol-glucoside was detected in turnip leaves only, where it amounted to 12%, 18% and 15% of the TRRs of the 1st, 2nd and 3rd rotation, respectively.

Two label-specific metabolites were identified: PCA and the methyl-sulfoxide, totalling 48.9–65.4% TRR in wheat grain from the three rotations. Residues of PCA in wheat grain were 56%, 16% and 29% of the TRRs of the 1st, 2nd and 3rd rotation, respectively, and the methyl-sulfoxide comprised 1.2%, 49% and 20% of the wheat grain TRRs of the 1st, 2nd and 3rd rotation, respectively. PCA also accounted for 17% TRR in 30-day PBI wheat forage, and was found at < 10% TRR in wheat forage from the 2nd and 3rd plant-back intervals, as well as in wheat hay, wheat straw (found in 1st rotation only), Swiss chard, turnip leaves, and turnip roots (1st rotation only). The methyl-sulfoxide was identified in wheat forage, hay and straw and Swiss chard at low levels (< 5% TRR).

Table 51 Residues in wheat forage, hay and straw after a 30 day plant back interval (1st rotation, [pyridyl-2,6-^{14}C]-fluopyram)

1. Rotation		Wheat forage		Wheat hay		Wheat straw		Wheat grains	
	TRR [mg/kg]	0.157		1.802		6.663		0.412	
Peak	Compound	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
27	Fluopyram (parent)	71.7	0.113	78.9	1.421	73.9	4.926	33.4	0.137

1. Rotation		Wheat forage		Wheat hay		Wheat straw		Wheat grains	
	TRR [mg/kg]	0.157		1.802		6.663		0.412	
Peak	Compound	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
2	-methyl-sulfoxide	-	-	-	-	-	-	1.2	0.005
3	-PCA	16.5	0.026	4.9	0.088	0.9	0.060	55.9	0.230
9	-7-OH-glc-MA (isomer 1)	3.3	0.005	4.2	0.076	5.6	0.376	-	-
10	-7-OH-glc-MA (isomer 2)	-	-	1.2	0.021	1.1	0.072	-	-
14A	-8-OH-glc-MA	-	-	0.6	0.010	0.4	0.028	-	-
14B	-7-OH-SA	-	-	-	-	-	-	-	-
15	-7-OH-glc [a]	1.3	0.002	0.9	0.016	3.1	0.203	-	-
19	-phenol-glc [b]	-	-	-	-	-	-	-	-
24	-7-OH	2.1	0.003	3.8	0.068	7.4	0.494	1.8	0.008
25	-8-OH	-	-	0.4	0.006	1.3	0.087	-	-
Total extractable		95.7	0.151	96.7	1.742	96.0	6.398	92.3	0.380
Total identified		95.0	0.149	94.7	1.707	93.7	6.248	92.3	0.380
Charact. by HPLC retention [c]		0.7	0.001	1.8	0.032	1.1	0.074	-	-
Charact. by diastase treatment		-	-	-	-	-	-	4.1	0.017
Not analysed		-	-	0.3	0.004	1.2	0.076	-	-
Total bound residues (PES)		4.3	0.007	3.3	0.059	4.0	0.265	3.6	0.015
Accountability		100.0	0.157	100.0	1.802	100.0	6.663	100.0	0.412

[a] co-eluting with 8-OH-glc-SA; representing about 20% of peak no. 15

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 17 minor metabolites characterised, all ≤ 2% TRR and < 0.043 mg/kg

Table 52 Residues in Swiss chard, turnip leaves and roots after a 30 day plant back interval (1st rotation, [pyridyl-2,6-^{14}C]-fluopyram)

1. Rotation		Swiss chard		Turnip leaves		Turnip roots	
	TRR [mg/kg]	0.57		0.565		0.036	
Peak	Compound	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
27	fluopyram (parent)	56.7	0.323	70.6	0.399	86.1	0.031
2	-methyl-sulfoxide	0.4	0.003	-	-	-	-
3	-PCA	1.8	0.010	4.3	0.024	6.0	0.002
9	-7-OH-glc-MA (isomer 1)	-	-	1.9	0.011	-	-
10	-7-OH-glc-MA (isomer 2)	-	-	0.6	0.003	-	-
14A	-8-OH-glc-MA	-	-	-	-	-	-
14B	-7-OH-SA	7.9	0.045	0.7	0.004	-	-
15	-7-OH-glc [a]	0.9	0.005	0.3	0.002	-	-
19	-phenol-glc [b]	-	-	12.0	0.068	-	-
24	-7-OH	28.0	0.160	3.5	0.020	3.1	0.001
25	-8-OH	0.9	0.005	0.3	0.002	1.5	0.001
Total extractable		99.8	0.569	99.5	0.562	98.7	0.036
Total identified		96.6	0.551	94.3	0.532	96.7	0.035
Characterised by HPLC retention [c]		3.2	0.018	5.2	0.030	2.0	0.001
Characterised by diastase treatment		-	-	-	-	-	-
Not analysed		-	-	-	-	-	-
Total bound residues (PES)		0.2	0.001	0.5	0.003	1.3	< 0.001

1. Rotation		Swiss chard		Turnip leaves		Turnip roots	
	TRR [mg/kg]	0.57		0.565		0.036	
Peak	Compound	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Accountability		100.0	0.570	100.0	0.565	100.0	0.035

[a] co-eluting with 8-OH-glc-SA; representing about 20% of peak no. 15

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 17 minor metabolites characterised , all ≤ 2% TRR, and < 0.043 mg/kg

Table 53 Residues in wheat forage, hay and straw after a 139 day plant back interval (2nd rotation, [pyridyl-2,6-^{14}C]-fluopyram)

2. Rotation		Wheat forage		Wheat hay		Wheat straw		Wheat grains	
	TRR [mg/kg]	0.568		0.971		2.562		0.072	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
27	fluopyram (parent)	77.9	0.442	66.9	0.650	73.5	1.881	20.4	0.015
2	-methyl-sulfoxide	2.2	0.012	4.7	0.046	1.2	0.031	49.0	0.035
3	-PCA	1.0	0.006	0.6	0.006	-	-	16.4	0.012
9	-7-OH-glc-MA (isomer 1)	5.6	0.032	7.0	0.068	3.5	0.089	-	-
10	-7-OH-glc-MA (isomer 2)	0.7	0.004	1.4	0.013	2.5	0.064	-	-
14A	-8-OH-glc-MA	1.2	0.007	1.4	0.014	0.9	0.022	-	-
14B	-7-OH-SA	-	-	-	-	-	-	-	-
15	-7-OH-glc [a]	1.0	0.005	2.5	0.024	2.3	0.059	-	-
19	-7-OH-glc [b]	-	-	-	-	-	-	-	-
24	-7-OH	3.1	0.018	5.9	0.057	6.1	0.156	0.8	0.001
25	-8-OH	0.7	0.004	0.7	0.007	1.2	0.030	-	-
Total extractable		95.2	0.540	91.6	0.890	92.7	2.373	86.5	0.062
Total identified		93.2	0.530	91.1	0.886	91.0	2.331	86.5	0.062
Characterised by HPLC retention [c]		2.0	0.011	0.5	0.004	0.8	0.020	-	-
Characterised by diastase treatment		-	-	-	-	-	-	9.1	0.007
Not analysed		-	-	-	-	0.9	0.022	-	-
Total bound residues (PES)		4.8	0.027	8.4	0.081	7.4	0.189	4.4	0.003
Accountability		100.0	0.568	100.0	0.971	100.0	2.562	100.0	0.072

[a] co-eluting with 8-OH-glc-SA; representing about 20% of peak 15

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 14 minor metabolites characterised, all ≤ 1.7% TRR and < 0.012 mg/kg

Table 54 Residues in Swiss chard, turnip leaves and roots after a 139 day plant back interval (2nd rotation, [pyridyl-2,6-^{14}C]-fluopyram)

2. Rotation		Swiss chard		Turnip leaves		Turnip roots	
	TRR [mg/kg]	0.343		0.103		0.01	
Peak	Compound	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
27	fluopyram (parent)	37.3	0.128	59.9	0.061	94.6	0.01
2	-methyl-sulfoxide	0.4	0.001	-	-	-	-
3	-PCA	0.2	0.001	1.5	0.001	-	-
9	-7-OH-glc-MA (isomer 1)	-	-	2.8	0.003	-	-
10	-7-OH-glc-MA (isomer 2)	-	-	1.2	0.001	-	-

2. Rotation		Swiss chard		Turnip leaves		Turnip roots	
	TRR [mg/kg]	0.343		0.103		0.01	
Peak	Compound	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
14A	-8-OH-glc-MA	-	-	-	-	-	-
14B	-7-OH-SA	16.8	0.058	0.7	0.001	-	-
15	-7-OH-glc [a]	1.8	0.006	1.4	0.001	-	-
19	-phenol-glc [b]	-	-	18.4	0.019	-	-
24	-7-OH	36.5	0.125	5.3	0.005	3.5	< 0.001
25	-8-OH	1.1	0.004	0.5	0.001	-	-
Total extractable		99.2	0.341	97.4	0.100	98.1	0.010
Total identified		94.0	0.323	91.7	0.094	98.1	0.010
Characterised by HPLC retention [c]		5.2	0.018	5.7	0.006	-	-
Characterised by diastase treatment		-	-	-	-	-	-
Not analysed		-	-	-	-	-	-
Total bound residues (PES)		0.8	0.003	2.6	0.003	1.9	< 0.001
Accountability		100.0	0.344	100.0	0.103	100.0	0.01

[a] co-eluting with 8-OH-glc-SA; representing about 20% of peak 15

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 14 minor metabolites characterised, all ≤ 1.7% TRR and < 0.012 mg/kg

Table 55 Residues in wheat forage, hay and straw after a 280 day plant back interval (3rd rotation, [pyridyl-2,6-^{14}C]-fluopyram)

3. Rotation		Wheat forage		Wheat hay		Wheat straw		Wheat grains	
	TRR [mg/kg]	0.167		0.709		1.622		0.037	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
27	fluopyram (parent)	70.2	0.118	66.8	0.473	58.3	0.945	31.0	0.012
2	-methyl-sulfoxide	2.0	0.003	3.5	0.025	1.2	0.019	20.3	0.008
3	-PCA	0.9	0.002	0.5	0.004	-	-	28.6	0.011
9	-7-OH-glc-MA (isomer 1)	7.5	0.013	9.5	0.067	7.3	0.118	-	-
10	-7-OH-glc-MA (isomer 2)	1.1	0.002	1.2	0.008	4.5	0.072	-	-
14A	-8-OH-glc-MA	1.7	0.003	1.7	0.012	1.2	0.019	-	-
14B	-7-OH-SA	-	-	-	-	-	-	-	-
15	-7-OH-glc [a]	3.0	0.005	3.0	0.021	3.7	0.060	-	-
19	-phenol-glc [b]	-	-	-	-	-	-	-	-
24	-7-OH	5.6	0.009	8.6	0.061	10.1	0.164	2.3	0.001
25	-8-OH	-	-	-	-	1.7	0.028	-	-
Total extractable		92.4	0.155	95.9	0.679	91.0	1.477	82.2	0.031
Total identified		92.0	0.154	95.4	0.671	87.9	1.425	82.2	0.031
Charact. by HPLC retention [c]		0.4	0.001	0.6	0.004	2.1	< 0.001	-	-
Charact. by diastase treatment		-	-	-	-	-	-	-	-
Not analysed		-	-	0.5	0.004	1.0	0.016	-	-
Total bound residues (PES)		7.6	0.013	4.1	0.029	9.0	0.146	17.8	0.007
Accountability		100.0	0.167	100.0	0.709	100.0	1.622	100.0	0.037

[a] co-eluting with 8-OH-glc-SA; representing about 20% of peak 15

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 13 minor metabolites characterised, all ≤ 1.3% TRR and < 0.022 mg/kg

Table 56 Residues in Swiss chard, turnip leaves and roots after a 280 day plant back interval (3rd rotation, [pyridyl-2,6-^{14}C]-fluopyram)

3. Rotation		Swiss chard		Turnip leaves		Turnip roots	
	TRR [mg/kg]	0.211		0.095		0.012	
Peak	Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
27	fluopyram (parent)	38.6	0.081	64.2	0.061	91.7	0.010
2	-methyl-sulfoxide	0.5	0.001	-	-	-	-
3	-PCA	0.2	0.000	1.1	0.001	-	-
9	-7-OH-glc-MA (isomer 1)	-	-	1.7	0.002	-	-
10	-7-OH-glc-MA (isomer 2)	-	-	1.9	0.002	-	-
14A	-8-OH-glc-MA	-	-	-	-	-	-
14B	-7-OH-SA	13.5	0.029	0.4	< 0.001	-	-
15	-7-OH-glc [a]	1.3	0.003	1.6	0.001	-	-
19	-phenol-glc [b]	-	-	14.7	0.014	-	-
24	-7-OH	38.6	0.081	8.3	0.008	6.1	0.001
25	-8-OH	1.3	0.003	0.4	< 0.001	-	-
Total extractable		98.2	0.207	98.7	0.094	97.8	0.011
Total identified		94.0	0.198	94.2	0.090	97.8	0.012
Characterised by HPLC retention [c]		4.2	0.009	4.5	0.004	-	-
Characterised by diastase treatment		-	-	-	-	-	-
Not analysed		-	-	-	-	-	-
Total bound residues (PES)		1.8	0.004	1.3	0.001	2.2	< 0.001
Accountability		100.0	0.211	100.0	0.095	100.0	0.012

[a] co-eluting with 8-OH-glc-SA; representing about 20% of peak 15

[b] co-eluting with -di-hydroxy-glc, a very minor metabolite and not quantified

[c] up to 13 minor metabolites characterised, all ≤ 1.3% TRR and < 0.022 mg/kg

A significant decline of the radioactive residues was observed for all samples from crops planted 39, 139, and 280 days after application of [pyridyl-2,6-^{14}C]-fluopyram to bare soil, except wheat forage, where the residues remained at a similar level. The TRRs in all other samples declined by factors of 2.5-11. The TRR for all samples of the 3rd rotation apart from turnip roots exceeded 0.01 mg/kg.

The proposed metabolic pathway involves hydroxylation of the ethylene linking group of the parent compound forming the 7-OH and the 8-OH metabolites, hydroxylation of the phenyl ring and subsequent conjugation with glucose, leading to the -phenol-glucose conjugate, conjugation of the hydroxylated metabolites with glucose, malonic acid and sulphuric acid, hydrolytic cleavage and subsequent oxidation to the BZM and PCA metabolites and subsequently to the -methyl sulfoxide and the -benzoic acid metabolites (the PCA and -methyl sulfoxide metabolites may also be formed at low proportions in the soil or by plant root enzymes and selectively translocated into grains following phloem transport), and the formation of polar, probably natural compounds incorporated into the starch matrix of grains.

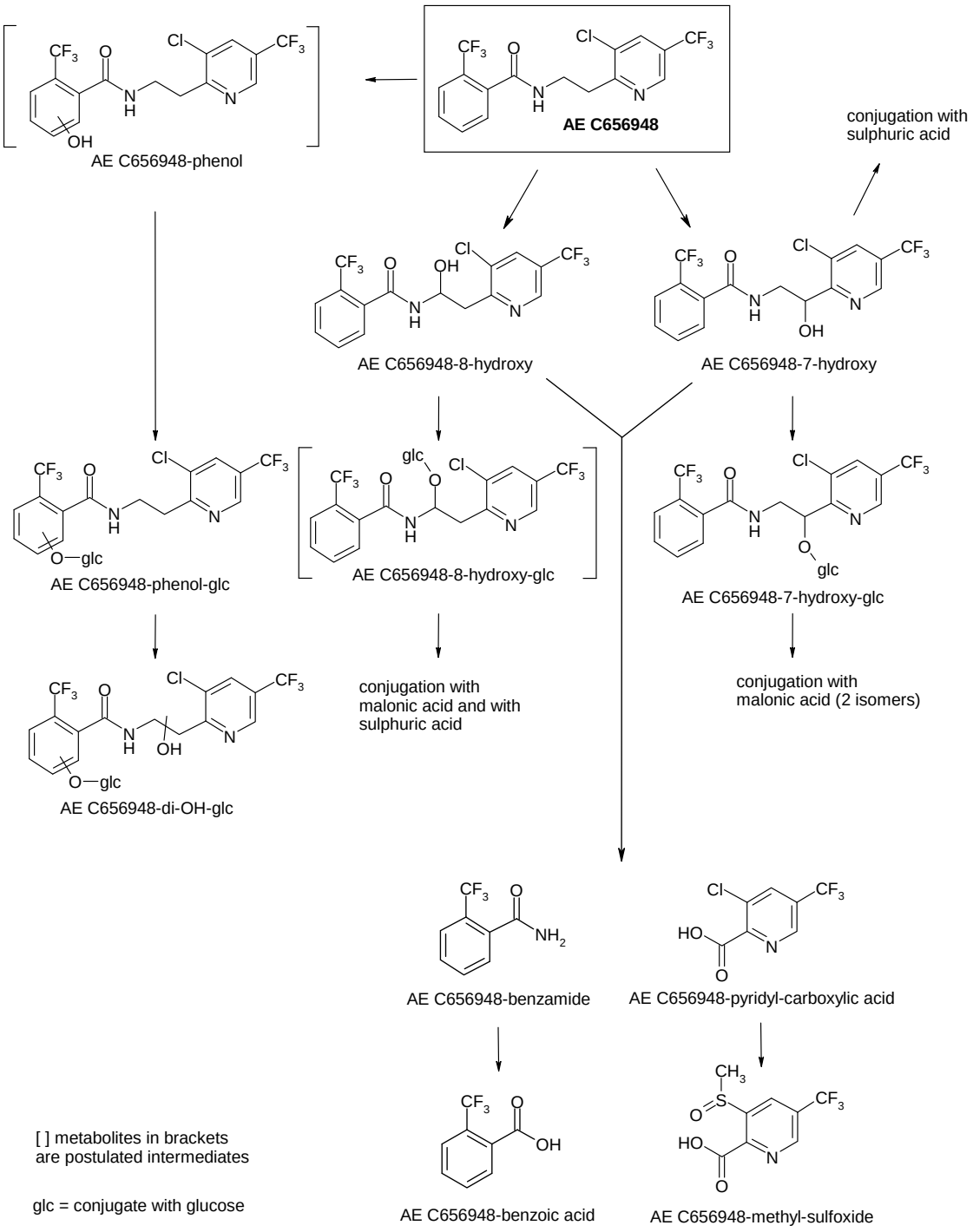


Figure 4 Proposed metabolic pathway for fluopyram in rotational crops (derived from both radiolabels)

Rotational crop field studies

Four field rotational crop trials conducted in Europe with fluopyram were reported by Noss & Erler, 2008 [Ref: RA-2648/06 to RA-2651/06]. Representative rotational crops used in these trials were turnips or carrots (tops and roots) to represent root crops, lettuce (head) to represent leafy vegetables and wheat (green material, grain, and straw) to represent cereal grains.

In these studies, three plots were sprayed with 0.5 kg ai/ha fluopyram (SC 500) using knapsack and 3–9 nozzle spray booms and the rotational crops were planted 28-49 days later (primary crops), 90–240 days after treatment (1st rotation crops) or 286-320 days after treatment (2nd rotation crops). In one plot, fluopyram was applied to bare ground, incorporated to at least 8 cm and planted/sown with lettuce (30 day PBI), turnip or carrot (30-36 day PBI) and wheat (28–49 day PBI). In a second plot, fluopyram was applied 2 weeks after planting lettuce as a primary crop (and harvested at maturity 32–86 days after planting) and the subsequent fallow plot was replanted (1st rotation) with lettuce, turnip or carrot (90–240 days after application) and with wheat (100–154 days after application). In the third plot, fluopyram was also applied 2 weeks after planting the primary lettuce crop (which was harvested at maturity 43–91 days after planting) and the subsequent fallow plot was replanted (2nd rotation) with lettuce and turnip (320 days after application) and with wheat (286 days after application).

All crop samples were harvested at normal maturity. For cereals, additional samples were taken at growth stage 29–30 BBCH (green material - forage). For root and leafy vegetables, additional samples were taken at early harvest (about 14 days before harvest). Samples were stored at or below -18 °C within 24 hours of harvest and stored frozen for up to 310 days (carrot/turnip leaf), 308 days (carrot/turnip roots), 404 days (lettuce), 221 days (wheat forage), 119 days (wheat straw) and 114 days (wheat grain).

Samples were extracted in acetonitrile/water and analysed for fluopyram and the major metabolites (7-OH, BZM, PCA and the methyl sulfoxide) using the LC/MS/MS Method 00984 with LOQs of 0.05 mg/kg for wheat straw and 0.01 mg/kg for all other analytes and matrices. Recovery rates for all analytes were measured in all matrices, spiked at 0.01, 0.05, 0.1 and also at 0.5, 2.5 and 5 mg/kg in the wheat matrices. Mean recovery rates ranged from 78–109% (fluopyram), 79–106% (7-OH), 78–133% (methyl sulfoxide), 84–124% (BZM) and 70–109% for PCA.

In turnip/carrot tops after 30-36 day plant-back intervals, residues of fluopyram ranged from < 0.01–0.1 mg/kg in the early harvest samples and 0.01–0.04 mg/kg at harvest. After the 1st rotation, fluopyram residues were 0.01–0.04 mg/kg and < 0.01–0.01 mg/kg after the 2nd rotation. No residues of the four metabolites were found above the LOQ of 0.01 mg/kg at any plant-back interval.

In turnip/carrot roots after 30-36 day plant-back intervals, residues of fluopyram ranged from < 0.01–0.05 mg/kg in both the early harvest samples and at harvest. After the 1st rotation, fluopyram residues were 0.01–0.03 mg/kg and were < 0.01 after the 2nd rotation. No residues of the four metabolites were found above the LOQ of 0.01 mg/kg at any plant-back interval.

In lettuce after a 30 day plant back interval, fluopyram residues ranged from < 0.01–0.11 mg/kg in the early harvest samples and < 0.01–0.03 mg/kg at harvest. After the 1st rotation, fluopyram residues were < 0.01–0.02 mg/kg and were below the LOQ (0.01 mg/kg) after the 2nd rotation. No residues of the four metabolites were found above the LOQ at any plant-back interval.

In wheat forage after 28-49 day plant back intervals, residues of fluopyram ranged from 0.07–0.12 mg/kg, declining after the 1st rotation to 0.05–0.08 mg/kg and 0.02–0.1 mg/kg after the 2nd rotation. Residues of the methyl-sulfoxide were < 0.01–0.05 mg/kg after 30 days plant back interval, 0.01–0.06 mg/kg after the 1st rotation and < 0.01–0.06 mg/kg after the 2nd rotation. Residues of the three other metabolites were below the LOQ of 0.01 mg/kg at all plant-back intervals except in one trial where the BZM metabolite was found at the LOQ after a 49 day plant-back interval.

In wheat grain after 28–49 day plant back intervals, residues of fluopyram ranged from < 0.01–0.01 mg/kg and were < 0.01 mg/kg after the 1st and 2nd rotations. Residues of the methyl-sulfoxide were 0.01–0.09 mg/kg after the 28–49 day plant-back intervals and 0.02–0.05 mg/kg after the 1st rotation, declining to 0.01–0.02 mg/kg after the 2nd rotation. No residues of the other three metabolites were found above the LOQ of 0.01 mg/kg at any plant-back interval.

In wheat straw after 28–49 day plant back intervals, residues of fluopyram ranged from < 0.05–0.28 mg/kg and were < 0.05–0.19 mg/kg after the 1st rotation and < 0.05–0.08 mg/kg after the 2nd rotation. Residues of the methyl-sulfoxide were < 0.05–0.07 mg/kg after the 28–49 day plant-back intervals and were < 0.05 mg/kg after the 1st and 2nd rotations. Residues of 7-OH were < 0.05–

0.11 mg/kg after the 28-49 day plant-back intervals, < 0.05–0.1 mg/kg after the 1st rotation and < 0.05 mg/kg after the 2nd rotation. Residues of the BZM metabolite were only found in one trial at 0.14 mg/kg in straw after a 49 day plant-back interval and no residues of PCA were found above the LOQ of 0.05 mg/kg at any plant-back interval.

Table 57 Residues in turnips and carrots grown as rotational crops following one application of 0.5 kg ai/ha fluopyram (SC 500) to bare ground or lettuce (primary crop) in Europe

Crop sample	PBI (days)	PHI (days)	Harvest stage	Residues (mg/kg)					Trial site Ref
				fluopyram	7-OH	methyl sulfoxide	BZM	PCA	
Turnip tops	30	86	14 dbh	0.02	< 0.01	< 0.01	< 0.01	< 0.01	Germany [a] (Burscheid) RA-2648/06
		100	Harvest	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
	216	323	14 dbh	0.04	< 0.01	< 0.01	< 0.01	< 0.01	
		337	Harvest	0.04	< 0.01	< 0.01	< 0.01	< 0.01	
	301	372	14 dbh	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		386	Harvest	0.01	< 0.01	< 0.01	0.01	< 0.01	
Turnip roots	30	86	14 dbh	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
		100	Harvest	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	216	323	14 dbh	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
		337	Harvest	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
	301	372	14 dbh	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		386	Harvest	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
Turnip tops	30	91	14 dbh	0.1	< 0.01	< 0.01	< 0.01	< 0.01	France [b] (Cergy) RA-2649/06
		105	Harvest	0.04	< 0.01	< 0.01	< 0.01	< 0.01	
	90	151	14 dbh	0.03	< 0.01	< 0.01	< 0.01	< 0.01	
		165	Harvest	0.03	< 0.01	< 0.01	< 0.01	< 0.01	
	320	381	14 dbh	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
Turnip roots	30	91	14 dbh	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		105	Harvest	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	90	151	14 dbh	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		165	Harvest	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	320	381	14 dbh	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		397	Harvest	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
Carrot tops	30	108	14 dbh	0.02	< 0.01	< 0.01	< 0.01	< 0.01	Italy [c] (Albaro) RA-2650/06
		122	Harvest	0.04	< 0.01	< 0.01	< 0.01	< 0.01	
	240	338	14 dbh	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		352	Harvest	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	289	363	14 dbh	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		377	Harvest	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
Carrot roots	30	108	14 dbh	0.05	< 0.01	< 0.01	< 0.01	< 0.01	
		122	Harvest	0.05	< 0.01	< 0.01	< 0.01	< 0.01	
	240	338	14 dbh	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
		352	Harvest	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
	289	363	14 dbh	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
		377	Harvest	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
Carrot tops	36	206	14 dbh	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	Spain [d] (Sevilla) RA-2651/06
		220	Harvest	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	154	271	14 dbh	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		285	Harvest	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
	344	532	14 dbh	0.01	< 0.01	< 0.01	< 0.01	< 0.01	

Crop sample	PBI (days)	PHI (days)	Harvest stage	Residues (mg/kg)					Trial site Ref
				fluopyram	7-OH	methyl sulfoxide	BZM	PCA	
		546	Harvest	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
Carrot roots	36	206	14 dbh	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
		220	Harvest	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
	154	271	14 dbh	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
		285	Harvest	0.03	< 0.01	< 0.01	< 0.01	< 0.01	
	344	532	14 dbh	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		546	Harvest	0.01	< 0.01	< 0.01	< 0.01	< 0.01	

dbh = days before harvest

PBI = plant-back interval

[a] soil characteristics: silty loam, %OM = 1.61, pH = 6.99

[b] soil characteristics: sandy loam, %OM = 0.94, pH = 8.6

[c] soil characteristics: silty loam, %OM = 1.27, pH = 8.4

[d] soil characteristics: loam, %OM = 0.47, pH = 8.8

Table 58 Residues in lettuce grown as a rotational crop following one application of 0.5 kg ai/ha fluopyram (SC 500) to bare ground or lettuce (primary crop) in Europe

Crop sample	PBI (days)	Harvest stage	PHI (days)	Residues (mg/kg)					Trial Site Ref
				fluopyram	7-OH	methyl sulfoxide	BZM	PCA	
lettuce	30	14 dbh	66	0.03	< 0.01	< 0.01	< 0.01	< 0.01	Germany [a] (Burscheid) RA-2648/06
		Harvest	80	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	230	14 dbh	261	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		Harvest	275	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	301	14 dbh	338	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		Harvest	352	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
lettuce	30	14 dbh	59	0.11	< 0.01	< 0.01	< 0.01	< 0.01	France [b] (Cergy) RA-2649/06
		Harvest	73	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
	90	14 dbh	129	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		Harvest	143	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	320	14 dbh	354	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		Harvest	368	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
lettuce	30	14 dbh	58	0.09	< 0.01	< 0.01	< 0.01	< 0.01	Italy [c] (Albaro) RA-2650/06
		Harvest	72	0.03	< 0.01	< 0.01	< 0.01	< 0.01	
	240	14 dbh	296	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
		Harvest	310	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	290	14 dbh	318	0.03	< 0.01	< 0.01	< 0.01	< 0.01	
		Harvest	332	0.01	< 0.01	< 0.01	< 0.01	< 0.01	
lettuce	30	14 dbh	136	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	Spain [d] (Sevilla) RA-2651/06
		Harvest	150	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	155	14 dbh	225	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		Harvest	239	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
	337	14 dbh	449	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
		Harvest	463	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	

dbh = days before harvest

PBI = plant-back interval

[a] soil characteristics: silty loam, %OM = 1.61, pH = 6.99

[b] soil characteristics: sandy loam, %OM = 0.94, pH = 8.6

[c] soil characteristics: silty loam, %OM = 1.27, pH = 8.4

[d] soil characteristics: loam, %OM = 0.47, pH = 8.8

Table 59 Residues in wheat forage, grain and straw grown as a rotational crop following one application of 0.5 kg ai/ha fluopyram (SC 500) to bare ground or lettuce (primary crop) in Europe

Sample Material	PBI (days)	Harvest stage	PHI (days)	Residues (mg/kg)					Trial Site Ref
				fluopyram	7-OH	methyl sulfoxide	BZM	PCA	
wheat forage	28	BBCH 29-30	190	0.07	< 0.01	< 0.01	< 0.01	< 0.01	Germany [a] (Burscheid) RA-2648/06
	100	BBCH 29-30	240	0.08	< 0.01	0.02	< 0.01	< 0.01	
	363	BBCH 29-30	414	0.02	< 0.01	< 0.01	< 0.01	< 0.01	
wheat grain	28	Harvest	314	0.01	< 0.01	0.01	< 0.01	< 0.01	
	100	Harvest	328	< 0.01	< 0.01	0.04	< 0.01	< 0.01	
	363	Harvest	691	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
wheat straw	28	Harvest	314	0.07	< 0.05	< 0.05	< 0.05	< 0.05	
	100	Harvest	328	0.09	0.05	< 0.05	< 0.05	< 0.05	
	363	Harvest	691	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	
wheat forage	30	BBCH 29-30	193	0.12	< 0.01	0.05	< 0.01	< 0.01	France [b] (Cergy) RA-2649/06
	146	BBCH 29-30	307	0.05	< 0.01	0.06	< 0.01	< 0.01	
	286	BBCH 29-30	336	0.1	< 0.01	0.01	< 0.01	< 0.01	
wheat grain	30	Harvest	308	< 0.01	< 0.01	0.09	< 0.01	< 0.01	
	146	Harvest	425	< 0.01	< 0.01	0.05	< 0.01	< 0.01	
	286	Harvest	425	< 0.01	< 0.01	0.02	< 0.01	< 0.01	
wheat straw	30	Harvest	308	0.28	0.11	0.07	< 0.05	< 0.05	
	146	Harvest	425	0.17	0.1	< 0.05	< 0.05	< 0.05	
	286	Harvest	425	0.06	< 0.05	< 0.05	< 0.05	< 0.05	
wheat forage	30	BBCH 29-30	139	0.11	< 0.01	0.02	< 0.01	< 0.01	Italy [c] (Albaro) RA-2650/06
	120	BBCH 29-30	242	0.07	< 0.01	0.01	< 0.01	< 0.01	
	363	BBCH 29-30	478	0.06	< 0.01	0.01	< 0.01	< 0.01	
wheat grain	30	Harvest	265	0.01	< 0.01	0.03	< 0.01	< 0.01	
	120	Harvest	352	< 0.01	< 0.01	0.02	< 0.01	< 0.01	
	363	Harvest	611	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
wheat straw	30	Harvest	265	0.15	0.08	< 0.05	< 0.05	< 0.05	
	120	Harvest	352	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	
	363	Harvest	611	0.08	0.06	< 0.05	< 0.05	< 0.05	
wheat forage	49	BBCH 29-30	126	0.11	< 0.01	0.02	0.01	< 0.01	Spain [d] (Sevilla) RA-2651/06
	150	BBCH 29-30	211	0.05	< 0.01	0.02	< 0.01	< 0.01	
	357	BBCH 29-30	437	0.02	< 0.01	0.06	< 0.01	< 0.01	
wheat grain	49	Harvest	219	< 0.01	< 0.01	0.01	< 0.01	< 0.01	
	150	Harvest	266	< 0.01	< 0.01	0.04	< 0.01	< 0.01	
	357	Harvest	582	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	
wheat straw	49	Harvest	219	0.05	< 0.05	0.06	0.14	< 0.05	
	150	Harvest	266	0.19	0.05	< 0.05	< 0.05	< 0.05	
	357	Harvest	582	< 0.05	< 0.01	< 0.05	0.05	< 0.05	

dbh = days before harvest

PBI = plant-back interval

[a] soil characteristics: silty loam, %OM = 1.61, pH = 6.99

[b] soil characteristics: sandy loam, %OM = 0.94, pH = 8.6

[c] soil characteristics: silty loam, %OM = 1.27, pH = 8.4

[d] soil characteristics: loam, %OM = 0.47, pH = 8.8

Table 60 Summary: Highest residues in rotational crops following one application of 0.5 kg ai/ha fluopyram (SC 500) to bare ground or lettuce (primary crop) in Europe

crop	Sample Material	PBI (days)	Harvest stage	PHI (days)	Highest residue (mg/kg)				
					fluopyram	7-OH	methyl sulfoxide	BZM	PCA
turnip carrot	tops	30-36	Harvest	100-220	0.04	< 0.01	< 0.01	< 0.01	< 0.01
		90-240	Harvest	165-352	0.04	< 0.01	< 0.01	< 0.01	< 0.01
		289-344	Harvest	377-546	0.01	< 0.01	< 0.01	0.01	< 0.01
	roots	30-36	Harvest	100-220	0.05	< 0.01	< 0.01	< 0.01	< 0.01
		90-240	Harvest	165-352	0.03	< 0.01	< 0.01	< 0.01	< 0.01
		289-344	Harvest	377-546	0.01	< 0.01	< 0.01	< 0.01	< 0.01
lettuce	head	30	Harvest	72-150	0.03	< 0.01	< 0.01	< 0.01	< 0.01
		90-240	Harvest	143-310	0.01	< 0.01	< 0.01	< 0.01	< 0.01
		290-337	Harvest	332-463	0.01	< 0.01	< 0.01	< 0.01	< 0.01
wheat	forage	28-49	BBCH 29-30	126-193	0.12	< 0.01	0.05	0.01	< 0.01
		100-150	BBCH 29-30	211-307	0.08	< 0.01	0.06	< 0.01	< 0.01
		286-363	BBCH 29-30	336-478	0.1	< 0.01	0.06	< 0.01	< 0.01
	grain	28-49	Harvest	219-314	0.01	< 0.01	0.09	< 0.01	< 0.01
		100-150	Harvest	266-425	< 0.01	< 0.01	0.05	< 0.01	< 0.01
		286-363	Harvest	425-691	< 0.01	< 0.01	0.02	< 0.01	< 0.01
	straw	28-49	Harvest	219-314	0.28	0.11	0.07	0.14	< 0.05
		100-150	Harvest	266-425	0.19	0.1	< 0.05	< 0.05	< 0.05
		286-363	Harvest	425-691	0.08	0.06	< 0.05	0.05	< 0.05

Rotational crop field trials were also conducted on cotton, alfalfa, wheat, turnip and mustard greens in USA.

Alfalfa was grown as a rotational crop after treatment with fluopyram in 12 USA trials reported by Krolski, 2008 [Ref: RAGMP105]. In these trials, 2 spray applications of fluopyram (500 SC) were made to bare soil (or a target crop at up to 3 sites), 5 days apart, at 0.24–0.26 kg ai/ha/application (0.5 to 0.51 kg ai/ha total) and alfalfa was planted 12–14 days after the last treatment.

Samples of alfalfa forage and hay were collected from each of three cuttings at commercial maturity, stored frozen for up to 11 months before acetonitrile/water extraction and analysis for fluopyram using HPLC (triple stage quadrupole mass spectrometry (method GM-001-P07-01). The limit of quantitation (LOQ) was 0.01 mg/kg in both alfalfa forage and alfalfa hay and mean recovery rates were 84–97% in samples spiked at 0.01, 0.5 and 1.0 mg/kg.

Residues in first-cut alfalfa forage sampled 69–186 days after the last treatment (12–14 days before planting) ranged from 0.01–0.39 mg/kg, with lower residues (< 0.01–0.19 mg/kg present in the second-cut and third-cut forage (generally about 40 days apart). In hay, sampled at the same times as forage, residues in first-cut hay ranged from 0.02–0.93 mg/kg and reduced to 0.01–0.46 mg/kg in subsequent cuts.

Table 61 Fluopyram residues in alfalfa forage and hay as a rotational crop planted 12–14 days after 2 applications of fluopyram (500 SC), 5 days apart, totalling 0.5 kg ai/ha

ALFALFA Site (soil type)	Application kg ai/ha	Application L/ha	PHI (days)	Commodity	%DM	Fluopyram residues (mg/kg)	Reference (Cover crop)
USA, 2007 Germansville, PA (clay loam)	0.257 0.257	179 179	82 121 159 82 121 159	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	23 20 24 64 76 73	0.04, 0.04 0.04, 0.04 0.02, 0.02 0.10, 0.10 0.11, 0.11 0.04, 0.04	GM111-07RA (bare soil)
USA, 2007 Sycamore, GA (loamy sand)	0.25 0.253	165 166	186 231 252 186 231 252	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	21 29 26 76 88 78	0.02, 0.01 0.03, 0.03 0.07, 0.07 0.09, 0.06 0.05, 0.05 0.13, 0.12	GM112-07RA (bare soil)
USA, 2007 Richland, IA (silty clay loam)	0.250 0.247	157 155)	69 105 144 69 105 144	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	27 31 21 74 82 85	0.08, 0.05 0.05, 0.05 0.05, 0.06 0.19, 0.17 0.13, 0.14 0.18, 0.20	GM113-07RA (bare soil)
USA, 2007 New Holland, OH (clay loam)	0.251 0.260	149 148)	106 140 181 106 140 181	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	32 25 28 85 64 52	0.04, 0.04 0.04, 0.04 0.01, 0.01 0.06, 0.06 0.09, 0.10 0.01, 0.02	GM114-07RA (bare soil)
USA, 2007 York, NE (clay loam)	0.250 0.250	184 184)	73 106 153 73 106 153	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	32 23 25 87 70 80	0.05, 0.03 0.08, 0.04 0.03, 0.02 0.07, 0.08 0.12, 0.13 0.06, 0.06	GM115-07RA (bare soil)
USA, 2007 Sheridan, IN (silty clay loam)	0.252 0.248	105 97	78 117 175 78 117 175	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	18 20 34 51 40 63	0.05, 0.05 0.02, 0.02 0.02, 0.03 0.10, 0.11 0.04, 0.04 0.03, 0.04	GM116-07RA (not available)
USA, 2007 Northwood, ND (loam)	0.254 0.249	142 138	100 103	forage (1st cut) hay (1st cut)	25 69	0.01, < 0.01 0.04, 0.05	GM117-07HA (bare soil)
USA, 2007 Campbell, MN (clay loam)	0.251 0.252	140 141	73 112 155 73 112 155	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	35 20 23 73 65 79	0.02, 0.02 < 0.01, < 0.01 0.01, 0.01 0.02, 0.04 0.01, 0.01 0.03, 0.03	GM118-07RA (not available)
USA, 2007 Eldridge, ND (loam)	0.259 0.241	178 176	95 130 164 95 130 164	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	32 24 26 85 79 61	0.02, 0.02 < 0.01, < 0.01 0.02, 0.02 0.05, 0.04 0.02, 0.02 0.03, 0.02	GM119-07RA (bare soil)

ALFALFA Site (soil type)	Application		PHI (days)	Commodity	%DM	Fluopyram residues (mg/kg)	Reference (Cover crop)
	kg ai/ha	L/ha					
USA, 2007 Jerome, ID (fine silty loam)	0.251 0.254	132 134	77 114 166 77 114 166	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	18 19 24 67 93 86	0.04, 0.06 0.04, 0.05 0.05, 0.06 0.19, 0.18 0.19, 0.21 0.22, 0.15	GM120-07RA (bare soil)
USA, 2007 Sanger, CA (sandy loam)	0.251 0.251	170 172	98 230 281 98 230 281	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	25 21 30 81 81 85	0.14, 0.21 0.09, 0.1 0.16, 0.19 0.68, 0.61 0.33, 0.30 0.38, 0.46	GM121-07RA (mustard)
USA, 2007 Rupert, ID (loam)	0.254 0.247	90 89	84 110 144 84 110 144	forage (1st cut) forage (2nd cut) forage (3rd cut) hay (1st cut) hay (2nd cut) hay (3rd cut)	25 21 21 86 83 91	0.39, 0.28 0.09, 0.10 0.06, 0.08 0.93, 0.92 0.36, 0.35 0.26, 0.29	GM122-07RA (bare soil)

Cotton was grown as a rotational crop after treatment with fluopyram in 11 USA trials reported by Fischer & Krolski, 2008 [Ref: RAGMY004]. In these trials, 2 spray applications of fluopyram (500 SC) were made to bare soil (at most sites), 5 days apart, at 0.24–0.26 kg ai/ha/application (0.5 to 0.51 kg ai/ha total) and cotton was planted 12–14 days after the last treatment.

Samples of mature cotton seed were collected by hand or by mechanical picker/strippers with gin trash also being collected from some of the mechanically harvested plots. The samples were frozen within 4 hours of collection and stored at or below -5 °C for up to 7 months before ginning and were returned to frozen storage until analysis about 3–4 weeks later (217 day maximum storage interval in total). The samples were extracted in acetonitrile/water and analysed for fluopyram using HPLC-MS-MS (Method GM-001-P07-01). The LOQ was 0.01 mg/kg in both matrices and mean recovery rates were 85–102% in samples spiked at 0.01, 0.05 and 0.1 mg/kg.

Residues in undelinted cottonseed from cotton planted 12–14 days after the last treatment and sampled 152–192 days later were all below the LOQ (0.01 mg/kg) and were < 0.01 to 0.02 mg/kg in cotton gin trash.

Table 62 Fluopyram residues in cottonseed and gin trash from cotton grown as a rotational crop, planted 12-14 days after 2 applications of fluopyram (500 SC), 5 days apart, totalling 0.5 kg ai/ha.

COTTONSEED Site (soil type)	Application		PHI (days)	Commodity	%DM	Fluopyram residues (mg/kg)	Reference (Cover crop)
	kg ai/ha	L/ha					
USA, 2007 Corapeake, NC (sandy loam)	0.257 0.254	93 97	192	cottonseed gin trash	91	< 0.01, < 0.01 0.02, 0.02	GM130-07RA (bare soil)
USA, 2007 Newport, AR (sandy loam)	0.250 0.249	94 93	169	cottonseed gin trash	94	< 0.01, < 0.01 < 0.01, < 0.01	GM131-07RA (not available)
USA, 2007 Proctor, AR (silty clay loam)	0.251 0.250	120 120	152	cottonseed gin trash	90	< 0.01,< 0.01 < 0.01, < 0.01	GM132-07RA (bare soil)

COTTONSEED Site (soil type)	Application		PHI (days)	Commodity	%DM	Fluopyram residues (mg/kg)	Reference (Cover crop)
	kg ai/ha	L/ha					
USA, 2007 Cheneyville, LA (silty clay loam)	0.252 0.252	138 133	177	cottonseed	89	< 0.01, < 0.01	GM133-07RA (bare soil)
USA, 2007 East Bernard, TX (sandy clay loam)	0.251 0.252	132 135	160	cottonseed gin trash	93	< 0.01, < 0.01 < 0.01, < 0.01	GM134-07RA (bare soil)
USA, 2007 Edmonson, TX (loam)	0.244 0.250	158 163	169	cottonseed gin trash	95	< 0.01, < 0.01 0.02, 0.02	GM135-07RA (bare soil)
USA, 2007 Levelland, TX (sandy loam)	0.248 0.253	138 146	163	cottonseed	99	< 0.01, < 0.01	GM137-07RA (bare soil)
USA, 2007 Larned, KS (silt loam)	0.253 0.256	174 176	169	cottonseed	98	< 0.01, < 0.01	GM138-07RA (bare soil)
USA, 2007 Porterville, CA (clay loam)	0.251 0.258	184 189	163	cottonseed	75	< 0.01, < 0.01	GM139-07RA (bare soil)
USA, 2007 Fresno, CA (sandy loam)	0.250 0.248	139 142	155	cottonseed	95	< 0.01, < 0.01	GM140-07RA (not available)
USA, 2007 Hickman, CA (sandy loam)	0.250 0.251	132 132	179	cottonseed	94	< 0.01, < 0.01	GM141-07RA (not available)

A limited rotational crop study with 8-month plant back interval was conducted in USA to measure fluopyram residues in wheat, turnip and mustard green planted as a rotational crop following bare ground or cover crop applications of fluopyram 500 SC (Murphy, 2008) [Ref: RAGMP061].

In wheat, two foliar spray applications of 0.25–0.26 kg ai/ha fluopyram 500 SC in 118–178 litres water were made to a cover crop of wheat, 5–7 days apart, the cover crops were harvested or destroyed within 14 days after the last treatment and after cultivation, wheat was sown as a rotational crop with a plant-back interval (PBI) of 238–240 days.

Samples of wheat forage, hay, grain and straw were frozen within 2 hours of sampling and stored frozen for up to 405 days before extraction in acetonitrile/water, SPE clean-up and analysed by HPLC-MS/MS (Method GM-001-P07-01). The LOQ was 0.01 mg/kg in all matrices and mean recovery rates were 86–106% in samples spiked at 0.01, 0.1 and 0.25 mg/kg.

In wheat planted 238–240 days after the last treatment, maximum fluopyram residues in forage were 0.05 mg/kg, 0.09 mg/kg in hay and 0.12 mg/kg in straw. Residues were below the LOQ (0.01 mg/kg) in grain.

Table 63 Fluopyram residues in wheat forage, hay, grain and straw from wheat grown as a rotational crop, planted 238–240 days after 2 applications of fluopyram (500 SC), 5–7 days apart, totalling 0.5 kg ai/ha.

WHEAT Site (soil type)	Application		PBI (days)	Commodity	Fluopyram residues (mg/kg)	Reference (Cover crop)
	kg ai/ha	L/ha				
USA, 2006 Molino, FL (sandy clay loam)	0.264 0.261	142 126	238	forage hay grain straw	< 0.01, < 0.01 0.019, 0.018 < 0.01, < 0.01 0.011, 0.013	GM218-06RA (wheat)
USA, 2006 Leland, MS (silt loam)	0.262 0.25	123 125	240	forage hay grain straw	0.012, < 0.01 0.032, 0.033 < 0.01, < 0.01 0.027, 0.036	GM219-06RA (wheat)
USA, 2006 Fresno, CA (sandy loam)	0.226 0.224	176 178	239	forage hay grain straw	0.034, 0.048 0.075, 0.089 < 0.01, < 0.01 0.124, 0.124	GM220-06RA (wheat)

In turnips, two foliar spray applications of 0.25–0.26 kg ai/ha fluopyram 500 SC in 124–177 litres water were made to cover crops of wheat or soya beans, 5–7 days apart, the cover crops were harvested or destroyed within 14 days after the last treatment and after cultivation, turnips were sown as a rotational crop with a plant-back interval (PBI) of 238–240 days.

Samples of turnip tops and roots were frozen within 2 hours of sampling and stored frozen for up to 400 days before extraction in acetonitrile/water, SPE clean-up and analysed by HPLC-MS/MS) (method GM-001-P07-01). The LOQ was 0.01 mg/kg in both matrices and mean recovery rates were 95–99% in samples spiked at 0.01 and 0.1 mg/kg.

In turnips planted 238–240 days after the last treatment, maximum fluopyram residues in turnip tops were 0.04 mg/kg and residues were below the LOQ (0.01 mg/kg) in turnip roots.

Table 64 Fluopyram residues in turnip tops and roots from turnips grown as a rotational crop, planted 238-240 days after 2 applications of fluopyram (500 SC), 5-7 days apart, totalling 0.5 kg ai/ha.

TURNIP Site (soil type)	Application		PBI (days)	Commodity	Fluopyram residues (mg/kg)	Reference Comments
	kg ai/ha	L/ha				
USA, 2006 Molino, FL (sandy clay loam)	0.255 0.256	138 124	238	tops roots	< 0.01, < 0.01 < 0.01, < 0.01	GM215-06RA (wheat)
USA, 2006 Leland, MS (loam)	0.247 0.246	124 123	240	tops roots	0.017, 0.019 < 0.01, < 0.01	GM216-06RA (soya beans)
USA, 2006 Fresno, CA (sandy loam)	0.252 0.25	174 177	239	tops roots	0.028, 0.041 < 0.01, < 0.01	GM217-06RA (wheat)

In mustard greens, two foliar spray applications of 0.24–0.25 kg ai/ha fluopyram 500 SC in 118–174 litres water were made to wheat or soya bean cover crops, 5–7 days apart, the cover crops were harvested or destroyed within 14 days after the last treatment and after cultivation, mustard greens were sown as a rotational crop with a plant-back interval (PBI) of 238–240 days.

Samples of fresh leaves were frozen within 2 hours of sampling and stored frozen for up to 400 days before extraction in acetonitrile/water, SPE clean-up and analysed by HPLC-MS/MS

(method GM-001-P07-01). The LOQ was 0.01 mg/kg and mean recovery rates were 80–100% in samples spiked at 0.01 and 0.1 mg/kg.

In mustard greens planted 238–240 days after the last treatment, maximum fluopyram residues in fresh leaves were 0.036 mg/kg.

Table 65 Fluopyram residues in fresh leaves from mustard greens grown as a rotational crop, planted 238–240 days after 2 applications of fluopyram (500 SC), 5–7 days apart, totalling 0.5 kg ai/ha

MUSTARD GREENS Site (soil type)	Application		PBI (days)	Commodity	Fluopyram residues (mg/kg)	Reference Comments
	kg ai/ha	L/ha				
USA, 2006 Molino, FL (sandy clay loam)	0.254 0.245	137 118	238	leaves	< 0.01, < 0.01	GM212-06RA (wheat)
USA, 2006 Leland, MS (loam)	0.251 0.242	125 121	240	leaves	0.012, 0.014	GM213-06RA (soya beans)
USA, 2006 Fresno, CA (sandy loam)	0.249 0.246	172 174	239	leaves	0.036, 0.033	GM214-06RA (wheat)

Table 66 Summary table: Highest fluopyram residues in rotational crops following one application of 0.5 kg ai/ha fluopyram (SC 500) to bare ground or to a primary crop.

Rotational crop	Matrix	Highest Fluopyram residue (mg/kg) at Plant-back Interval (days)					
		14	28–49	90–120	146–155	216–240	280–381
Alfalfa (USA)	forage	0.39					
	hay	0.93					
Carrot (Europe)	roots		0.05		0.03	0.02	0.01
	tops		0.04		0.02	0.01	0.01
Turnip (Europe, USA)	roots		0.01	< 0.01		0.02	< 0.01
	tops		0.04	0.03		0.04	0.01
Cotton (USA)	seed	< 0.01					
	gin trash	0.02					
Lettuce (Europe)	head		0.03	0.01	< 0.01	0.01	0.01
mustard greens (USA)	leaves					0.04	
Wheat (Europe, USA)	forage		0.12	0.08	0.05	0.05	0.1
	hay					0.09	
	grain		0.01	< 0.01	< 0.01	< 0.01	< 0.01
	straw		0.28	0.09	0.19	0.12	0.08

METHODS OF RESIDUE ANALYSIS

Analytical methods

The meeting received analytical method descriptions and validation data for fluopyram and major metabolites (BZM, 7-OH, PCA, PAA and the methyl-sulfoxide) in crop and animal commodities and in soil. Enforcement methods for fluopyram in soil, water, sediment and air were provided were not evaluated.

These methods summarised below are suitable for the determination of fluopyram residues (as defined for both enforcement and/or risk assessment). The principle of most methods involves extraction steps using organic solvents (predominantly acetonitrile) with varying proportions of water, followed by different matrix dependant clean up steps. The final determination is carried out mostly by HPLC-MS/MS.

Table 67 Summary of Analytical Methods for Fluopyram and metabolites

Lettuce, rape seed, orange, wheat grain and straw Diot, 2007 [Ref: MR-06/030]			
Analytes:	Fluopyram & metabolites:- -benzamide (BZM) -pyridyl-carboxylic acid (PCA) [a] -pyridyl-acetic acid (PAA) -7-hydroxy (7-OH) -methyl-sulfoxide	HPLC-MS/MS	Method 00984
LOQ:	Wheat straw 0.05 mg/kg, all other matrices 0.01 mg/kg		
Description	Samples are extracted (2X 5 min blending) with acetonitrile:water (80:20). Centrifuged, filtered extracts, generally without further clean-up, are diluted with 0.5% acetic acid for detection of the PCA and -methyl-sulfoxide metabolites and under alkaline conditions (pH 8) for detection of fluopyram and the BZM, 7-OH and the PAA metabolites. Both extracts are separated by HPLC (C8 column, water/acetonitrile) and residues detected by tandem mass spectrometry (MS/MS) using positive and negative electrospray ionisation injection and internal stable labelled standards. Analyte — Mass transition (quantitation) — Mass transition (confirmation) Fluopyram 397→173 397→208 BZM 190→102 190→170 PCA 224→162 224→198 PAA 240→159 240→194 7-OH 413→173 413→145 methyl-sulfoxide 252→146 252→208		
Apple, cabbage, grape, strawberry, rape seed, tomato and their processed commodities Schoening, 2007 [Ref: MR-06/201]			
Analytes:	Fluopyram & metabolites:- -benzamide (BZM) -pyridyl-carboxylic acid (PCA) [a] -pyridyl-acetic acid (PAA)	HPLC-MS/MS	Method 00984/M001
LOQ:	0.01 mg/kg		
Description	A modification of M-00984 involving a single extraction and an additional purification step. Samples are extracted (2X 5 min blending) with 80:20 acetonitrile:water (for rape seed oil, extraction is by partitioning with acetonitrile:n-hexane). Filtered extracts are diluted under alkaline conditions (pH 8) for detection of fluopyram and the BZM and PAA metabolites. For measuring the PCA metabolite, the filtered extracts, acidified with sulphuric acid, are cleaned-up by liquid/liquid partitioning (twice) with methyl-butyl-ether (MTBE). Both extracts are separated by HPLC (C8 column, water/acetonitrile) and residues detected by tandem mass spectrometry (MS/MS) using positive and negative electrospray ionisation injection and internal stable labelled standards. Analyte — Mass transition (quantitation) — Mass transition (confirmation) Fluopyram 397→173 397→208 BZM 190→102 190→170 PCA 224→162 224→198 PAA 240→159 240→194		
Apple, maize (incl fodder and forage), pea (incl vines), grape, orange, peach, pear, potato, tomato, wheat grain Brungardt, 2008 [Ref: GM-001-P07-01]			
Analytes:	Fluopyram	HPLC-MS/MS	GM-001-P07-01
LOQ:	0.01 mg/kg		
Description	A modification of M-00984 involving shorter blending times and an SPE clean-up step. Samples are extracted (2X 1 min blending) with 80:20 acetonitrile:water and passed through a C-18 SPE cartridge before being diluted with aqueous acetic acid and analysed for fluopyram (parent) by HPLC-electrospray ionization/tandem mass spectrometry (LC-MS/MS).		

Cattle milk & tissues, hen eggs & tissues Schoening & Billian, 2007 [Ref: MR-07/299]			
Analytes:	Fluopyram & metabolites:- -benzamide (BZM) -olefines (E & Z isomers)	HPLC-MS/MS	Method 01061
LOQ:	0.01 mg/kg (0.02 mg/kg for the calculated total residues of the two olefine isomers)		
Description	Samples are extracted with acetonitrile:water (80:20), with microwave assistance (power regulated to maintain 120°C for 15 min) for cattle liver, kidney, muscle), and passed through a C-18 SPE cartridge before being diluted with water/methanol and separated by HPLC (C18 column, methanol/water) and analysed by positive electrospray tandem mass spectrometry (MS/MS). Residues of the two olefine isomers are reported as 'total olefines' as there is internal conversion between the two isomers. Analyte — Mass transition (quantitation) — Mass transition (confirmation) Fluopyram — 397→173 — 397→208 -BZM — 190→170 — 190→150 -olefine (E-isomer) — 395→173 — 395→145 -olefine (Z-isomer) — 395→173 — 395→145		
Cattle milk & tissues, hen eggs Schoening & Willmes, 2008 [Ref: MR-07/348]			
Analytes:	Fluopyram & metabolite:- -benzamide (BZM)	HPLC-MS/MS	Method 01079
LOQ:	0.01 mg/kg		
Description	As for Method 01061 above, but only measuring fluopyram (parent) and the BZM metabolite. Analyte — Mass transition (quantitation) — Mass transition (confirmation) Fluopyram — 397→173 — 397→208 BZM — 190→170 — 190→150		
Soil Brumhard, 2006 [Ref: MR-179/05]			
Analytes:	Fluopyram	HPLC-MS/MS	Method 00973
LOQ:	0.005 mg/kg		
Description	Samples are extracted with acetonitrile:water (80:20) with microwave assistance (3 min @ 250 W), centrifuged to remove soil particles and analysed for fluopyram by HPLC (C18 column, water/acetonitrile) with MS/MS detection (electrospray ionisation) in the multiple reaction monitoring mode and internal stable labelled standards. Analyte — Mass transition (quantitation) Fluopyram — 397→173 [phenyl-$^{13}C_6$]-fluopyram — 403→179		
Soil Brumhard & Schneider, 2008 [Ref: MR-07/319]			
Analytes:	Fluopyram	HPLC-MS/MS	Method 01068
LOQ:	0.005 mg/kg		
Description	A modification of Method 00973, intended for enforcement. Samples are extracted with acetonitrile:water (80:20) with microwave assistance (3 min @ 250 W), centrifuged to remove soil particles and analysed for fluopyram by HPLC (C8 column, water/ecetonitrile) with MS/MS detection (electrospray ionisation) in the multiple reaction monitoring mode. Analyte — Mass transition (quantitation) — Mass transition (confirmation) Fluopyram — 397→173 — 397→208 [phenyl-$^{13}C_6$]-fluopyram — 403→179		
Soil Freitag & Schneider, 2007 [Ref: MR-06/187]			
Analytes:	Fluopyram & metabolites:- -benzamide (BZM) -pyridyl-carboxylic acid (PCA) -7-hydroxy (7-OH)	HPLC-MS/MS	Method 01023
LOQ:	0.001 mg/kg		
Description	Samples are extracted with acetonitrile:water (80:20) with microwave assistance (3 min @ 250 W), centrifuged, evaporated, resuspended in water/acetonitrile/acetic acid and analysed for fluopyram and metabolites by reversed phase HPLC with tandem mass spectrometry (MS/MS) in the multiple reaction monitoring mode and internal stable labelled standards. Analyte — Mass transition (quantitation) — Mass transition (confirmation)		

	Fluopyram	397→173	397→208
	BZM	190→130	190→170
	PCA	224→180	224→162
	7-OH	413→173	413→145

[a] PAA is unstable under acidic conditions. Sodium salt used as injection standard

Data collection methods

Method 00984 (plant matrices)

The analytical method 00984 (summarised above), was described and reported by Diot, 2007 [Ref: MR-06/030] as suitable for as a data-collection method to measure residues of fluopyram, the main plant metabolites (BZM, PCA and PAA) as well as the minor metabolites 7-OH and the methyl-sulfoxide. Limits of quantification were 0.05 mg/kg for wheat straw and 0.01 mg/kg for other matrices and calculated limits of detection ranged from 0.001 to 0.006 mg/kg (0.002 to 0.011 mg/kg for wheat straw).

As a measure of precision, the mean relative standard deviations (% RSD) for different sample materials and analytes (at fortification levels of 0.01 and 0.10 or 0.05 and 0.50 mg/kg) ranged from 2–16.7% (except for the methyl-sulfoxide, a minor metabolite, in rape seed and wheat straw where RSDs of 40% and 33% were reported).

As a measure of accuracy, mean overall recovery rates ranged from 72–110% over the range of fortification levels for the different matrices and analytes, except for the methyl-sulfoxide, a minor metabolite, in lettuce and rape seed, where recovery rates of 125% and 135% were reported.

Method 00984/M001 (plant matrices)

A modification of the original method (00984) was developed to analyse processed plant matrices (summarised above) and is described by Schoening, 2007 [Ref: MR-06/201]. The modifications involved the exclusion the methyl-sulfoxide and 7-OH metabolites (because these are minor metabolites, specific for rotational crops), the addition of a further purification step (SPE or methyl-butyl-ether partition) to improve the determination of PCA and the use of a single high-speed blender extraction (rather than the double extraction used in the original method.

The single and double extraction procedures were compared in different plant matrices (strawberries, grapes, rape foliage) using incurred non-radioactive residues of fluopyram and its metabolites. No significant differences were observed between the two extraction methods.

As a measure of precision, the mean relative standard deviations (% RSD) for different sample materials and analytes (at fortification levels of 0.01 and 0.10 mg/kg) ranged from 0.5–15% (except for fluopyram in grape pomace dry where a RSD of 22% was reported).

As a measure of accuracy, mean overall recovery rates ranged from 76–111% over the range of fortification levels for the different matrices and analytes, except for PCA in grape juice and must, where recovery rates of 65% and 61% were reported.

The extraction efficiency of the M001 modification was investigated in a study reported by Spiegel, 2008 [Ref: MEF-08/109] measuring aged radioactive residues from metabolism studies with grapes. Samples of the grapes from the metabolism studies performed with [phenyl-UL-^{14}C]-fluopyram and [pyridyl-2,6-^{14}C]fluopyram were extracted once with acetonitrile/water (8/2, v/v), filtered and analysed by HPLC, as described in method 00984/M001 and the results compared with those obtained in the metabolism study.

More than 97% of the TRR (total radioactive residues) measured in the metabolism study were extracted from grapes using Method 00984/M001 with > 93% of the TRR covered by the compounds measured by the method. Extraction efficiencies of 97.4% and 96.9% were calculated from these results.

Table 68 Recovery results of extraction efficiency testing of method 00984/M001 – Recoveries from representative matrices taken from the metabolism studies in grapes*

	Residues determined in metabolism study*		Residue analytical method 00984/M001	
	[mg/kg]	% of TRR	[mg/kg]	% of TRR
	Grapes, treated with [phenyl-UL-^{14}C]-fluopyram			
TRR	1.86	100.0	1.62	100.0
Fluopyram	1.82	97.6	1.50	93.1
-benzamide (BZM)	0.01	0.7	0.04	2.6
Sum TTR	1.83	98.3	1.55	95.7
extraction efficiency (based on % of TRR)	100% × 95.7/98.3 = 97.4%			
	Grapes, treated with [pyridyl-UL-^{14}C]-fluopyram			
TRR	1.70	100.0	1.03	100.0
Fluopyram	1.63	95.8	0.88	86.0
-pyridyl-carboxylic acid (PCA)	0.02	0.9	0.08	7.8
-pyridyl-acetic acid (PAA) **	-	-	-	-
Sum TTR	1.65	96.7	0.96	93.7
extraction efficiency (based on % of TRR)	100% × 93.7/96.7 = 96.9%			

TRR = total radioactive residues

* Ref: MEF-06/087 and MEF-06/086

** this metabolite was not found in the metabolism study in grapes

The efficiency of the acetonitrile/water extraction procedures used in the analytical methods 00984 and GM-001-P07-01 was reported by Klempner, 2008 [Ref: MEF-07/171]. Plant samples from the metabolism and confined rotational crop studies were used to assess the extraction efficiency of repeated acetonitrile/water (80:20) extraction, with individual extracts of a sample being radio-assayed and the radioactivity and the extraction efficiency calculated for each extract.

Extraction efficiencies were determined for the first two extraction steps for potatoes, beans, red pepper, and for the plant matrices of the rotational crop studies. The samples from the confined rotational crop studies (Swiss chard, turnip roots, turnip leaves, and wheat forage, hay, straw and grains) were repeatedly extracted (three or up to five times) with acetonitrile/water (8/2; v/v).

Over all metabolism studies, the first two extraction steps with acetonitrile/water (8/2; v/v) released > 89% of the TRR in all RACs of the target crop metabolism studies and > 91% of the TRR in turnip leaves, turnip roots, Swiss chard and wheat forage of the confined rotational crop (CRC) studies.

In case of hay and straw from the confined rotational crop studies, > 84% of the TRR was extracted, with about 5 and 15% of the residues remaining in the solids after conventional extraction. These solids were exhaustively re-extracted with acetonitrile/water (1/1, v/v) using microwave conditions. The microwave extracts showed the same metabolic profiles as the respective conventional extracts with the parent compound as the main component. No new metabolites or degradation products were detected. This supports the assumption that no metabolite is discriminated by the extraction procedure used for analytical purposes.

In case of grain, > 70% of the TRR was readily extracted, with the amount of residual radioactivity remaining in the solids after extraction increasing in the longer plant-back intervals and characterised as natural compound (e.g., starch).

As the extraction procedure in the analytical methods for plant matrices is identical to the one used in the metabolism studies and the extraction efficiency in these studies is about ≥ 89%, it is concluded that residues of fluopyram and its metabolites are readily extractable with acetonitrile/water (8:2; v/v).

Method GM-001-P07-01 (plant matrices)

This method (a modification of the original Method 00984) described by Brungardt, 2008 [Ref: GM-001-P07-01] and used in the North American crop field trials, was designed to measure only the parent compound (fluopyram) and involved a shorter blending time than the 5 minutes used in the original Method 00984 and the inclusion of an SPE clean-up step.

A comparison of the extraction performance of the two blending times in 13 different matrices showed that the one-minute blending extracted 95–123% of the residues extracted using the 5-minute blending, with an overall mean extraction efficiency of 103%, supporting the use of the shorter blending times.

Method 01061 (animal matrices)

This method was developed for measuring residues of fluopyram, the BZM metabolite and the two olefine isomers (E- and Z-) in/on animal tissues, milk and eggs (and was used in the livestock feeding study) – Schoening & Billian, 2007 [Ref: MR-07/299].

Limits of quantification are 0.01 mg/kg for fluopyram and BZM and 0.02 mg/kg for the calculated total residue of the E- and Z- olefine isomers. Limits of detection (LOD) were estimated at 0.003 mg/kg for fluopyram, 0.001 mg/kg for BZM and 0.004 mg/kg for the calculated total olefines for all matrices.

While the analytical method allows the separation by chromatography of the E- and the Z-olefine isomers, the rapid inter-conversion of the two isomers supports the reporting of these residues as the combined sum of the two isomers.

As a measure of precision, the mean relative standard deviations (% RSD) for different sample materials and analytes (at fortification levels of 0.01 and 0.10 mg/kg) ranged from 0.6–13%.

As a measure of accuracy, mean overall recovery rates ranged from 82–104% over the range of fortification levels for the different matrices and analytes.

The extraction efficiencies of the method were evaluated as part of the metabolism studies in hen and goat after application of [phenyl-UL-^{14}C]-fluopyram where the same extraction procedures were used. The extraction efficiencies for the total radioactive residue (TRR) ranged from 84–97% and a comparison of the metabolic profiles from metabolism studies and from the extracts obtained with the analytical residue method showed nearly identical metabolite pattern.

Table 69 Extraction efficiencies of fluopyram residues from hen and goat matrices

Matrix	Extraction method according to residue analytical method	Extracted amount [% TRR]
Laying hen		
Egg pool, day 1-6	1× extraction with acetonitrile/water 4/1, v/v	94.5
Egg pool, day 7-14	1× extraction with acetonitrile/water 4/1, v/v	94.4
Muscle	1× extraction with acetonitrile/water 4/1, v/v	94.1
Fat	1× extraction with acetonitrile/water 4/1, v/v	86.2
Liver	1× extraction with acetonitrile/water 4/1, v/v	90.8
Excreta	1× extraction with acetonitrile/water 4/1, v/v	84.4
Lactating goat		
Morning milk	1× extraction with acetonitrile/water 4/1, v/v	97.5
Evening milk	1× extraction with acetonitrile/water 4/1, v/v	97.0
Muscle	2× microwave extraction at 120 °C with acetonitrile/water 4/1, v/v	96.6
Fat	1× extraction with acetonitrile/water 4/1, v/v	94.8
Liver	2× microwave extraction at 120 °C with acetonitrile/water 4/1, v/v	93.6
Kidney	2× microwave extraction at 120 °C with acetonitrile/water 4/1, v/v	97.4

Method 00973 (soil matrices)

This method, described by Brumhard, 2006 [Ref: MR-179/05], was developed for data generation in the European fluopyram field dissipation trials. The limit of quantitation (LOQ) for fluopyram is 5 µg/kg and the limit of detection (LOD) is 1.5 µg/kg.

The method was validated with a silt loam soil (Höfchen) and a sandy loam soil (Laacher Hof) at levels of 5 µg/kg and 50 µg/kg, with an overall mean recovery rate of 101% (precision of the method) and a relative standard deviation (RSD, overall mean) of 2.1% (accuracy of the method).

Method 01023 (soil matrices)

This method, described by Freitag & Schneider, 2007 [Ref: MR-06/187], was developed for data generation and enforcement in North America. It is an expansion of Method 00973 (above) to include detection of the major soil metabolites (BZM, 7-OH and PCA). The limit of quantitation (LOQ) for all analytes is 1 µg/kg and the limit of detection (LOD) is 0.3 µg/kg.

Mean recovery rates in two soils spiked with 1 µg/kg and 10 µg/kg fluopyram and the three major soil metabolites were 97-105% and the relative standard deviations ranged from 2.7% to 8.1%.

An independent laboratory validation for method 01023 was conducted by Netzband, 2008 [Ref: RAGMP101] with two soils spiked with fluopyram and the three major metabolites at 1 µg/kg and 10 µg/kg. The limit of quantitation (LOQ) of the method was 1 µg/kg for fluopyram and the BZM, 7-OH and PCA metabolites and a method detection limit (MDL) was calculated as 0.2 µg/kg for fluopyram, 7-OH and BZM and as 0.3 µg/kg for the PCA metabolite.

The overall mean recovery in soil (primary ion transition) of the method ranged from 95 to 100% for both fortification levels and all analytes. For the confirmatory ion transition it ranged from 90 to 98%. The relative standard deviations ranged from 7.3% to 10.9%.

Enforcement methods

Multi-residue method DFG S19

A study by Class, 2007 [Ref: P-1304 G], investigated the suitability of the German DFG S19 multi-residue enforcement method for measuring residues of fluopyram and its BZM metabolite (the major residue components expected in plant and animal products), using acetone extraction and gas chromatography with mass spectrometric detection (GC/MS) – Module 4.

Gas chromatography with full scan mass spectrometric detection (GC/MS) of fluopyram in both plant and animal matrices gave acceptable peak shapes and sensitivity for fluopyram (parent compound) but the sensitivity of the BZM (in animal matrices) is limited to concentrations at or above 1 µg/mL.

The study concluded that the DFG S19 method used in combination with GC/MS is applicable as a multi-residue enforcement method to monitor residues of fluopyram but not applicable as a multi-residue enforcement method to monitor residues of the benzamide metabolite.

A validation study on the use of DFG S19 enforcement method for fluopyram in plant matrices was reported by Rzepka & Jungklaus, 2007 [Ref: P682077508]. Extraction of fluopyram from oranges (fruit) was performed according to extraction module E3, extraction module E2 was used for wheat (grain) and dry peas (seeds), extraction module E7 was used for oil seed rape (seeds) and extraction module E1 was used for lettuce (head) followed by clean-up procedure according to module GPC. All specimens were analysed by gas chromatography with mass selective detection (GC-MSD) with mass spectrometry detection using the ions 223, 173 and 396. Quantification was achieved with external standards (6 standard solutions from 0.02–2 µg/mL).

For fluopyram in all plant matrices tested (orange, rape seeds, wheat grain, lettuce and dry pea seeds, the limit of quantitation (LOQ) was 0.01 mg/kg and the calculated limit of detection (LOD) was 0.003 mg/kg.

As a measure of precision, the relative standard deviations (% RSD) for different sample materials at fortification levels of 0.01 and 0.10 mg/kg ranged from 2.5–15% and recovery rates (as a measure of method accuracy) ranged from 70% to 107%.

An independent validation study for the use of the DFG S19 enforcement method to measure fluopyram residues in plant matrices was reported by Class, 2007 [Ref: P 1351 G], and looked at cabbage, wheat grain, orange and (fruit) and oilseed rape seed. Mean recovery rates (as a measure of method accuracy) were 73–100% and RSD values ranged from 9% to 15%.

Fischer, 2008 [Ref: RAGMP041] reported the results of a bridging study to compare the fluopyram extraction efficiency of acetone (as used in the DGG S19 multi-residue method) with the efficiency of acetonitrile/water (as used in the data generation analytical methods). Acetone extraction of incurred (aged) fluopyram residues from tomatoes averaged 101% (range 92–106%) of the residues extracted using acetonitrile:water (80:20), indicating that for extracting fluopyram residues from plant matrices, acetone extraction efficiency is comparable with the efficiency of acetonitrile/water.

FDA PAM Multi-residue method

A report by Ballard, 2008 [Ref: RAGMP108] described the assessment of the US FDA Pesticide Analytical Manual, Volume I (PAM I - Third edition) protocols for measuring residues of fluopyram and the BZM metabolite and concluded that the FDA multi-residue methods are suitable for detection and enforcement of fluopyram in non-fatty matrices by E1 extraction and DG17 detection without Florisil ^{R} cleanup but are not suitable for detection of BZM residues.

Method 01079 (animal matrices)

Analytical method 01079 was validated by Schoening & Willmes, 2008 [Ref: MR-07/348] as an enforcement method for the determination of residues of fluopyram and its BZM metabolite in animal tissues, milk and eggs, using both matrix-matched and internal standards.

For all tested matrices, the limit of quantitation (LOQ) was 0.01 mg/kg and the estimated limit of detection (LOD) was 0.003 mg/kg for both for fluopyram and BZM.

As a measure for the precision, the mean relative standard deviation (% RSD) for different sample materials at fortification levels of 0.01 and 0.10 mg/kg ranged from 2.1% to 14.8% and mean recovery rates (as a measure of accuracy) ranged from 83% to 99%.

An independent validation study for the use of method 01079 to measure fluopyram residues in animal matrices was reported by Portet, 2008 [Ref: MR-08/053], and looked at cattle milk, muscle, liver and hen eggs. Mean recovery rates (spiked at 0.01 mg/kg and 0.1 mg/kg), as a measure of method accuracy, were 93–107% and RSD values ranged from 3.1% to 10.3%.

Analytical (concurrent) recoveries in supervised crop trials

Analytical recovery rates were measured in all the supervised crop field trials, with control samples being fortified with fluopyram at 0.01 mg/kg and at higher levels that generally reflected the range of expected residues. In the European trials, the analytical methods used were either Method 00984 or the modified Method 00984/M001 while the trials in North America used Method GM-001-P07-01.

For fluopyram, over the 166 commodities analysed, recoveries following spiking at levels from 0.01 mg/kg to more than 50 mg/kg were between 63% to 137%, with average recoveries for the different matrices of 77–106%. Average recoveries for the fluopyram metabolites over the different matrices were 78–106%.

Stability of residues in stored analytical samples

Plant matrices

The Meeting received information on the stability of residues of fluopyram and metabolites in various substrates with a high water content (lettuce, cabbage), a high starch content (wheat grain, potato), a

high protein content (dry pea seed), a high oil content (rape seed) and a high acid content (orange) stored at freezer temperatures for 6–30 months.

In a study by Cavaille & Portet, 2008 [Ref: MR-08/035], the stability of fluopyram and its BZM, PCA PAA and 7-OH metabolites was investigated in lettuce, wheat grain, dry pea seed and rape seed matrices stored under frozen conditions.

Samples of the test commodities were purchased in the market and half were fortified at 0.2 mg/kg of each analyte. The spiked and the control samples were sealed in 125 ml polypropylene bottles and stored in the dark at or below -18 °C. Samples were taken for extraction and analysis after 0, 3, 6, 13, 18 and 24 months, with the stored control samples being freshly fortified with each analyte and analysed concurrently to determine the procedural recovery. Analysis was by LC-MS/MS (Method 00984), with mean procedural recovery rates of 75–109% for all analytes and matrices (fortified at 0.01 mg/kg and 0.2 mg/kg).

After 24 months storage the measured residues of fluopyram and major metabolites in stored samples of representative plant matrices with high water, starch, protein and oil content were greater than 87% of the spiked level (fluopyram) and for the fluopyram metabolites were more than 77%.

Table 70 Stability of fluopyram residues in plant matrices spiked at 0.2 mg/kg and stored ≤ -18 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)	% Residues remaining [a]
Lettuce (head)	0	0.208; 0.193; 0.207	101	105	96
	3	0.207; 0.198; 0.206	102	107	95
	6	0.207; 0.209; 0.217	106	99	107
	13	0.177; 0.200; 0.219	100	114	87
	18	0.212, 0.191, 0.203	101	96	106
	24	0.188, 0.18, 0.191	93	93	100
Wheat (grain)	0	0.206; 0.198; 0.211	102	96	106
	3	0.197; 0.199; 0.202	100	104	96
	6	0.200; 0.190; 0.182	95	99	96
	13	0.210; 0.207; 0.205	104	114	91
	18	0.206, 0.211, 0.206	104	104	100
	24	0.208, 0.193, 0.209	102	101	101
Dry pea (seed)	0	0.185; 0.194; 0.187	95	92	103
	3	0.202; 0.199; 0.202	100	104	96
	6	0.192; 0.193; 0.191	96	98	98
	13	0.207; 0.202; 0.207	103	111	93
	18	0.205, 0.206, 0.198	102	100	102
	24	0.201, 0.199, 0.183	97	97	100
Rape (seed)	0	0.242; 0.217; 0.233	115	104	111
	3	0.207; 0.213; 0.218	106	109	97
	6	0.198; 0.195; 0.192	98	100	98
	13	0.197; 0.199; 0.195	99	108	92
	18	0.192, 0.189. 0.202	97	93	105
	24	0.19, 0.188, 0.193	95	93	102

[a] % residues remaining after correction for procedural recovery

Table 71 Stability of residues of the BZM metabolite of fluopyram in plant matrices spiked at 0.2 mg/kg and stored ≤ -18 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)	% Residues remaining [a]
Lettuce (head)	0	0.183; 0.177; 0.193	93	103	90
	3	0.199; 0.192; 0.185	96	103	93
	6	0.190; 0.192; 0.189	95	97	98
	13	0.163; 0.169; 0.172	84	109	77
	18	0.183, 0.192, 0.201	96	91	105
	24	0.177, 0.176, 0.179	89	92	96

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)	% Residues remaining [a]
Wheat (grain)	0	0.180; 0.186; 0.186	92	92	100
	3	0.203; 0.202; 0.205	101	105	96
	6	0.176; 0.191; 0.190	93	96	97
	13	0.175; 0.171; 0.168	86	110	78
	18	0.189, 0.203, 0.202	99	98	101
	24	0.183, 0.178, 0.179	90	95	95
Dry pea (seed)	0	0.182; 0.182; 0.175	90	90	100
	3	0.197; 0.188; 0.192	96	98	98
	6	0.185; 0.177; 0.183	91	94	97
	13	0.164; 0.163; 0.170	83	108	77
	18	0.179, 0.181, 0.19	92	92	100
	24	0.179, 0.176, 0.169	88	92	95
Rape (seed)	0	0.174; 0.168; 0.171	85	94	91
	3	0.252; 0.244; 0.248	124	124	100
	6	0.195; 0.198; 0.189	97	93	104
	13	0.167; 0.171; 0.177	86	107	81
	18	0.195, 0.196, 0.187	96	91	106
	24	0.168, 0.175, 0.171	86	84	102

[a] % residues remaining after correction for procedural recovery

Table 72 Stability of residues of the PAA metabolite of fluopyram in plant matrices spiked at 0.2 mg/kg and stored ≤ -18 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)	% Residues remaining [a]
Lettuce (head)	0	0.185; 0.186; 0.197	95	94	101
	3	0.190; 0.193; 0.178	94	99	95
	6	0.186; 0.187; 0.184	93	96	97
	13	0.169; 0.165; 0.163	83	108	77
	18	0.164, 0.153, 0.168	81	98	82
	24	0.157, 0.163, 0.159	80	92	87
Wheat (grain)	0	0.166; 0.162; 0.166	82	84	98
	3	0.173; 0.176; 0.170	87	91	95
	6	0.168; 0.173; 0.172	86	87	99
	13	0.185; 0.190; 0.185	93	103	91
	18	0.18, 0.192, 0.197	95	95	100
	24	0.173, 0.182, 0.172	88	87	101
Dry pea (seed)	0	0.164; 0.158; 0.170	82	80	103
	3	0.174; 0.173; 0.180	88	88	100
	6	0.171; 0.164; 0.163	83	85	98
	13	0.178; 0.167; 0.175	87	102	85
	18	0.173, 0.187, 0.179	90	85	106
	24	0.156, 0.154, 0.15	77	78	98
Rape (seed)	0	0.181; 0.180; 0.181	91	91	100
	3	0.174; 0.174; 0.173	87	91	96
	6	0.161; 0.184; 0.167	85	89	96
	13	0.192; 0.192; 0.190	96	105	91
	18	0.183, 0.171, 0.18	89	85	105
	24	0.16, 0.171, 0.158	82	80	102

[a] % residues remaining after correction for procedural recovery

Table73 Stability of residues of the PCA metabolite of fluopyram in plant matrices spiked at 0.2 mg/kg and stored ≤ -18 °C.

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)	% Residues remaining [a]
Dry pea (seed)	0	0.182; 0.188; 0.179	91	89	103
	3	0.187; 0.209; 0.212	101	100	101
	6	0.193; 0.188; 0.189	95	88	108
	13	0.172; 0.174	87	85	102
	18	0.173, 0.174	87	89	98
	24	0.191, 0.186, 0.177	93	87	107
Rape (seed)	0	0.183; 0.167; 0.178	88	99	89
	3	0.172; 0.172; 0.177	87	92	95
	6	0.187; 0.184; 0.186	93	91	102
	13	0.187; 0.193; 0.186	95	97	98
	18	0.194, 0.207	101	102	99
	24	0.135, 0.134, 0.133	67	64	104

[a] % residues remaining after correction for procedural recovery

Table 74 Stability of residues of the 7-OH metabolite of fluopyram in plant matrices spiked at 0.2 mg/kg and stored ≤ -18 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)	% Residues remaining [a]
Lettuce (head)	0	0.209; 0.197; 0.218	104	108	96
	3	0.192; 0.192; 0.202	98	104	94
	6	0.193; 0.195; 0.199	98	101	97
	13	0.195; 0.200; 0.193	98	111	88
	18	0.186, 0.184, 0.205	96	97	99
	24	0.188, 0.188, 0.194	95	97	98
Wheat (grain)	0	0.210; 0.203; 0.209	104	99	105
	3	0.200; 0.203; 0.203	101	107	94
	6	0.188; 0.187; 0.191	94	100	94
	13	0.210; 0.204; 0.208	104	116	90
	18	0.195, 0.196, 0.19	97	95	102
	24	0.199, 0.193, 0.189	97	98	99

[a] % residues remaining after correction for procedural recovery

The stability of fluopyram and its BZM, PCA and PAA was investigated in orange fruit stored under frozen conditions and reported by Cavaille & Portet, 2007 [Ref: MR-08/036].

Oranges were purchased in the market and half were fortified at 0.2 mg/kg of each analyte. The spiked and the control samples were sealed in 125 ml polypropylene bottles and stored in the dark at or below -18 °C. Samples were taken for extraction and analysis after 0, 4, 6, 12, 19 and 24 months, with the stored control samples being freshly fortified with each analyte and analysed concurrently to determine the procedural recovery efficiency. Analysis was by LC-MS/MS (Method 00984), with mean procedural recovery rates of 77-106% for all analytes and matrices (fortified at 0.01 mg/kg and 0.2 mg/kg).

After 24 months storage the measured residues of fluopyram and major metabolites in stored samples of orange (representing matrices with high acid content) were greater than 95% of the nominal level (0.2 mg/kg) except for the PAA metabolite, where levels decreased to about 70% after 24 months storage.

Table 75 Residue stability of fluopyram and major plant metabolites in orange fruit spiked at 0.2 mg/kg and stored ≤ -18 °C

Analyte	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)	% Residues remaining [a]
Fluopyram	0	0.220; 0.204; 0.220	107	103	104
	4	0.200; 0.197; 0.201	100	97	103
	6	0.206; 0.210; 0.207	104	100	104
	12	0.208, 0.193, 0.205	101	98	103
	19	0.204, 0.197, 0.182	97	93	104
	24	0.189, 0.189, 0.177	93	94	99
BZM	0	0.207; 0.205; 0.203	102	102	100
	4	0.174; 0.169; 0.172	86	93	92
	6	0.173; 0.196; 0.178	87	93	93
	12	0.189, 0.202, 0.2	98	94	105
	19	0.175, 0.191, 0.181	92	90	102
	24	0.182, 0.18, 0.185	91	90	101
PAA	0	0.184; 0.182; 0.180	91	88	103
	4	0.158; 0.166; 0.172	83	91	91
	6	0.159; 0.154; 0.152	78	81	96
	12	0.12, 0.111, 0.116	58	72	80
	19	0.125, 0.129, 0.119	62	85	73
	24	0.115, 0.116, 0.114	57	83	69
PCA	0	0.208; 0.205; 0.199	102	99	103
	4	0.190; 0.193; 0.191	96	99	97
	6	0.191; 0.188; 0.196	96	95	101
	12	0.186, 0.196, 0.185	95	101	94
	19	0.185, 0.185	93	93	99
	24	0.183, 0.183	92	96	95

[a] % residues remaining after correction for procedural recovery

The stability of the PCA metabolite (also a metabolite of fluopicolide) was investigated in wheat (grain), grapes, potato (tubers) and cabbage (leaves) stored under frozen conditions and reported by Zeitz, 2004 [Ref: C045739].

Homogenised samples were fortified with PCA in glass containers at a level of 0.1 mg/kg and then placed in deep freeze storage. At intervals of approximately 3, 6, 12, 18, 24 and 30 months after fortification, samples were removed for analysis.

After 30 months storage the measured residues in stored samples ranged were greater than 75% of the nominal level (0.1 mg/kg).

Table 76 Residue stability of the PCA metabolite of fluopyram (and fluopicolide) in plant matrices spiked at 0.1 mg/kg and stored ≤ -18 °C. Recorded residue levels are unadjusted for procedural recoveries

	Grapes		Potato tubers		Cabbage leaves		Wheat grain	
Storage interval (months)	Residues remaining (mg/kg)	%Procedural recoveries	Residues remaining (mg/kg)	%Procedural recoveries	Residues remaining (mg/kg)	%Procedural recoveries	Residues remaining (mg/kg)	%Procedural recoveries
0	0.09, 0.08, 0.1 (3)		0.07, 0.08, 0.09 (3)		0.09 (2), 0.1 (3)		0.09 (3), 0.1 (2)	
3	0.08, 0.09	78, 80	0.08, 0.09	72, 72	0.08, 0.07	74, 70	0.08, 0.08	81, 78
6	0.09, 0.09	68, 77	0.08, 0.08	79, 87	0.08, 0.08	75, 74	0.08, 0.08	73, 80
12	0.09, 0.09	87, 89	0.08, 0.09	96, 83	0.08, 0.08	77, 73	0.09, 0.11	86, 83
18	0.08, 0.08	75, 76	0.06, 0.06	71, 81	0.07, 0.07	71, 73	0.08, 0.08	75, 77
24	0.10, 0.09	75, 75	0.08, 0.08	73, 68	0.08, 0.09	72, 77	0.08, 0.08	76, 74
30	(1.0)** 0.08	92 84	0.08 0.08	86 89	0.07 0.06	78 81	0.08 0.07	78, 75

** The reported residue (of 1.0 mg/kg) was ten times higher than the initial spiked concentration (0.1 mg/kg), and was identified as an error in fortification

Billian & Schöning, 2007 [Ref: MR-178-04] studied the stability of residues of the methyl-sulfoxide, a common metabolite with fluopicolide, in laboratory fortified wheat matrices (wheat straw, grain, green material) during freezer storage for 25 months.

Homogenised aliquots were fortified with the methyl-sulfoxide metabolite in glass containers at a level of 0.1 mg/kg and then placed in deep freeze storage. The samples were analysed at nominal intervals of 0, 30, 90, 180, 360, 540 and 760 days.

Residues of the methyl-sulfoxide metabolite in samples of wheat straw, grain and green material were stable for at least 25 months under deep-freezer storage conditions.

Table 77 Residue stability of the methyl-sulfoxide metabolite of fluopyram in wheat matrices spiked at 0.1 mg/kg and stored ≤ -18 °C. Recorded residue levels are unadjusted for procedural recoveries

Storage Interval (days)	Wheat straw		Wheat green material		Wheat grain	
	Residues remaining (mg/kg)	%Procedural recoveries	Residues remaining (mg/kg)	%Procedural recoveries	Residues remaining (mg/kg)	%Procedural recoveries
0	0.07, 0.08 (3), 0.09		0.07 0.08 (4)		0.06 0.08 (3), 0.06	
28-30	0.1, 0.08, 0.09	80, 74	0.07 (3)	76, 78	0.1 (3)	74, 77
89-114	0.08, 0.1, 0.09	82, 88	0.08 (2), 0.09	95, 87	0.09, 0.08, 0.11	85, 76
168-182	0.07 (3)	85, 87	0.07 (2), 0.06	65, 67	0.06, 0.08, 0.07	88, 83
349-363	0.08 (3)	81, 82	0.08 (3)	83, 86	0.08, 0.09 (2)	82, 85
552-553	0.08 (3)	105, 104	0.09, 0.11, 0.12	117, 118	0.07, 0.07*	75, 71, 68
754-770	0.06 (3)	81, 82	0.06 (3)	67, 65	0.07, 0.08 (2)	89, 64

* One recovery sample was identified as outlier, no RSD was calculated

Soil

The storage stability of fluopyram and its BZM, 7-OH and PCA metabolites in soil under freezer storage conditions was measured by Freitag, 2008 [Ref: MR-08/003].

Untreated soil (loam and loamy sand) from two US sites was fortified separately with 20 µg/kg each of fluopyram, BZM, PCA and 7-OH and analysed after 323 days of storage in the freezer at <-18 °C using Method 01023 (HPLC-MS/MS) and with a limit of quantitation (LOQ) of 1 µg/kg and a limit of detection (LOD) of 0.3 µg/kg for all analytes.

The mean concurrent recovery during analyses of the samples was 92-102% for the four analytes and more than 70% of the initially fortified residues were recovered from the stored samples, with mean residues remaining after 323 days being 93% to 110% of the initial concentration.

Table 78 Residue stability of fluopyram and metabolites in soil spiked at 20µg/kg and stored at or below -18 °C

Substance	Soil	Day	Procedural recovery [%]	Residues remaining [µg/kg)	% Residues remaining [a]
Fluopyram	North Dakota	0	105	20.9, 19.8, 19.8, 20.8, 18.8	
		323	102	20.3, 22, 20.2, 17.5, 18.5	99
	New York	0	92.5	18.5, 17.5, 18, 18.2, 20	
		323	94.5	18.9, 20.6, 20.2, 20.1, 20	110
7-OH	North Dakota	0	101	20.1, 18.6, 22.1, 22.4, 19.5	
		323	117	23.4, 26.4, 16.1, 21.3, 23	105
	New York	0	90.5	18.1, 23.8, 16.9, 23.2, 20.2	

Substance	Soil	Day	Procedural recovery [%]	Residues remaining [µg/kg)	% Residues remaining [a]
		323	101	20.2, 16.9, 23.7, 19.1, 24	100
BZM	North Dakota	0	90	18, 19.2, 18.1, 17.4, 17.4	
		323	92.5	18.5, 18.3, 17.4, 17.5, 17	97
	New York	0	92	18.4, 18.8, 19.9, 18.2, 19.8	
		323	92.5	18.5, 19.4, 17.7, 18.6, 17.3	95
PCA	North Dakota	0	96	19.2, 19.9, 19.8, 20.3, 20.3	
		323	99	19.8, 18.8, 18.7, 18.8, 18.7	93
	New York	0	97.5	19.5, 19.8, 19.7, 19.5, 20	
		323	99	19.8, 18.5, 18.2, 18.4, 18.4	93

[a] % residues remaining in soil samples after correction for procedural recovery

Stability of residues in samples extracts

The storage stability of residues of fluopyram and metabolites in extracts was tested during development of the analytical methods. All analytes were stable in filtered raw extracts for at least 8 days when stored at 4 °C in dark, except for the PAA metabolite which degrades quickly after extraction.

USE PATTERNS

Information on GAP in China and Romania was provided to the Meeting, together with proposed uses in Europe, Latin America and North America that are currently being progressed under an OECD Joint Review exercise.

Fluopyram is intended for use as foliar applications especially in grapes, top fruits in temperate and tropical areas, berries, fruiting and leafy vegetables, root and tuber crops, brassica crops and hops. Other applications are also recommended such as application in drip-line irrigation and as a seed treatment.

Table 79 Registered uses of fluopyram (500 g ai/litre SC formulation)

Crop	Country	Application				Max/season		PHI (days)	Comments
		method	kg ai/ha	kg ai/hL	water L/ha	no	kg ai/ha		
Grapes (Table)	Romania	foliar	0.2-0.25		800-1000	2	0.5	3	12d intervals
Grapes (wine)	Romania	foliar	0.2-0.25		800-1000	2	0.5	21	12d intervals
Cucumber	China	foliar	0.0375-0.075			2-3		2	7-10d intervals

Fluopyram is currently under evaluation in Europe and North America for a range of uses on fruit and vegetable crops, cereals, pulses and other arable crops. These proposed uses, as outlined on the draft labels provided, are summarised below.

Table 80 Proposed uses of fluopyram (500 g ai/litre SC formulation)

Crop	Country	Application				Max /season		PHI (days)	Comments
		method	kg ai/ha	kg ai/hL	water L/ha	no	kg ai/ha		
Grapes	Germany	foliar	0.25		400-1600	2		28	12d interv, BBCH 75-83
Grapes	Canada	foliar	0.25		1000 (min)		0.5	7	12-14d interv
Strawberriy	Canada	foliar	0.25		1000 (min)		0.5	1	5-10d interv
Tomato (field)	Canada	foliar	0.25		300 (min)		0.5	12 hrs	3-5d interv

Crop	Country	Application				Max /season		PHI (days)	Comments
		method	kg ai/ha	kg ai/hL	water L/ha	no	kg ai/ha		
Tomato (field)	Canada	foliar	0.15		300 (min)		0.5	12 hrs	5-10d interv
Tomato (indoor)	Canada	foliar	0.5		1000-1500	2		12 hrs	
Grapes	USA	foliar	0.25		468 (min)		0.5	7	12-14d interv
Strawberry	USA	foliar	0.25		468 (min)		0.5	1	5-10d interv
Tomato	USA	foliar	0.25		94 (min)		0.5	12 hrs	3-5d interv
Tomato	USA	foliar	0.15		94 (min)		0.5	12 hrs	5-10d interv

Additional uses (maximum GAP) proposed for authorization (phase 2) in North America and Europe for which draft labels were provided, are listed below.

Table 81 Proposed uses of fluopyram (500 g ai/litre SC formulation)

Crop	Country	Application				Max /season		PHI (days)	Comments
		type	method	kg ai/ha (max)	water L/ha (min)	no	kg ai/ha		
Artichoke	Canada	F	spray	0.25	200	6	0.5	0	
Artichoke	USA	F	spray	0.25	94		0.5	0	
Brassica vegetables	Canada	F	spray	0.25	200	6	0.5	0	
Brassica vegetables	USA	F	spray	0.25	94		0.5	0	
Bulb vegetables	Canada	F	spray	0.25	200	4	0.5	0	
Bulb vegetables	USA	F	spray	0.25	94		0.5	0	
Bushberries	Canada	F	spray	0.25	500	2	0.5	0	
Bushberries	USA	F	spray	0.25	468		0.5	0	
Caneberries	Canada	F	spray	0.25	500	2	0.5	0	
Caneberries	USA	F	spray	0.25	468		0.5	0	
Carrots	Canada	F	spray	0.25	200	2	0.5	7	7d PHI for feed
Carrots (incl tops)	USA	F	spray	0.25	94		0.5	7	7d PHI for feed
Cereals grains (ex rice)	Canada	F	spray	0.25	100	2	0.5	14	14d PHI for grazing, feed
Cereals grains (ex rice)	USA	F	spray	0.25	94		0.5	14	14d PHI for grazing, feed
Citrus	USA	F	spray	0.25	468		0.5	7	
Cucumber	Canada	G	spray	0.25	500	2	0.5	1	
Cucurbit vegetables	Canada	F	spray	0.25	200	2	0.5	0	
Cucurbit vegetables	USA	F	spray	0.25	94		0.5	0	
Cucurbit vegetables	USA	F/G	drip	0.25			0.5	7	
Cucurbit vegetables	USA	G	Spray	0.25	94		0.5	3	
Fruiting vegetables	Canada	F	spray	0.25	200	2	0.5	0	
Fruiting vegetables	USA	F	spray	0.25	94		0.5	0	except tomato
Ginseng	Canada	F	spray	0.25	200	2	0.5	7	
Ginseng	USA	F	spray	0.25	94		0.5	7	
Grasses	Canada	F	spray	0.25	100	4	0.5	7	7d PHI for forage, feed
Grasses	USA	F	spray	0.25	94		0.5	7	7d PHI for forage, feed
Herbs & Spices	Canada	F	spray	0.25	200	2	0.5	0	except black pepper
Herbs & Spices	USA	F	spray	0.25	94		0.5	0	
Hops	Canada	F	spray	0.25	200	2	0.5	7	
Hops	USA	F	spray	0.25	140 – 1870		0.5	7	
Leafy vegetables	Canada	F	spray	0.25	200	2	0.5	0	
Leafy vegetables	USA	F	spray	0.25	94		0.5	0	
Leafy vegetables	USA	F	band	0.25	94		0.5	18	

Crop	Country	Application				Max /season		PHI (days)	Comments
		type	method	kg ai/ha (max)	water L/ha (min)	no	kg ai/ha		
Legume vegetables	Canada	F	spray	0.25	200	2	0.5	0	0d PHI for forage, feed
Legume vegetables	USA	F	spray	0.25	94		0.5	0	
Oil seed rape (canola)	USA	F	spray	0.25	94		0.5	14	
Oilseed rape (canola)	Canada	F	spray	0.25	100	2	0.5	14	
Other low growing berries	USA	F	spray	0.25	94		0.5	0	
Other low growing berries	USA	F	drip	0.25			0.5	0	
Other low-growing berries	Canada	F	spray	0.25	500	2	0.5	0	
Peanut	Canada	F	spray	0.25	200	4	0.5	7	
Peanut	USA	F	spray	0.25	94		0.5	7	
Peppers	Canada	G	spray	0.25	500 – 2000	2	0.5	3	
Peppers	USA	G	spray	0.25	94		0.5	3	min 10 acre greenhouses
Pome fruits	Canada	F	spray	0.15	500	3	0.5	0	
Pome fruits	USA	F	spray	0.25	468		0.5	0	
Pulses	Canada	F	spray	0.25	200	2	0.5	14	0d PHI for forage, hay
Pulses	USA	F	spray	0.25	94		0.5	14	0d PHI for forage, hay
Root vegetables	Canada	F	spray	0.25	200	2	0.5	7	7d PHI for feed
Root, tuber, corm vegetables	Canada	F	spray	0.15	200	6	0.5	7	7d PHI for feed
Root, tuber, corm vegetables	USA	F	spray	0.25	94		0.5	7	7d PHI for feed
Small vine fruits	Canada	F	spray	0.25	500	2	0.5	7	
Small vine fruits	USA	F	spray	0.25	468		0.5	7	
Soya bean	Canada	F	spray	0.25	200	2	0.5	14	7d PHI for forage, feed
Soya bean	USA	F	spray	0.25	94		0.5	14	7d PHI for forage, feed
Stone fruits	Canada	F	spray	0.25	500		0.5	0	
Stone fruits	USA	F	spray	0.25	468		0.5	0	
Strawberries	Canada	F	spray	0.25	500	2	0.5	0	
Strawberries	USA	F	drip	0.25			0.5	0	
Strawberries	USA	F	spray	0.25	94		0.5	0	
Strawberries	USA	G	drip	0.25			0.5	1	
Strawberries	USA	G	spray	0.25	94		0.5	1	
Sunflower	Canada	F	spray	0.25	100		0.5	14	
Sunflower	USA	F	spray	0.25	94		0.5	14	
Tomato	Canada	G	spray	0.25	300 – 1500	2	0.5	3	
Tomato	USA	F	spray	0.25	94		0.5	0	
Tomato	USA	G	spray	0.25	94		0.5	3	
Tree nuts	Canada	F	spray	0.25	500	4	0.5	14	
Tree nuts	USA	F	spray	0.25	468		0.5	14	

Other intended uses (maximum GAP) proposed for authorization in Latin America and Europe are listed below. Also included are intended additional uses for fluopyram co-formulated with other fungicides.

Table 82 Intended uses of fluopyram (500 g ai/L SC formulation) and additional intended uses for fluopyram co-formulations.

Crop	Country	Application				Max no /season	PHI (days)	Comments
		type	method	kg ai/ha (max)	water L/ha (min)			
Bananas	Latin America	F	spray	0.1	20	5	0	
Artichoke, Globe	EU	F	spray	100		3	7	
Asparagus	EU	F	spray	150+150	200	3		with 200 g/L tebuconazole
Asparagus	EU	F	spray	200+200	300	2		with 250 g/L trifloxystrobin
Beans	EU	F	spray	250	200	2	7	
Beans	EU	G	spray	300	300	2	7	150 g/ha/meter canopy height
Bulbs onions, garlic, shallot, spring onions	EU	F	spray	200+200	200	2	7	with 200 g/L tebuconazole
Brussels sprouts	EU	F	spray	200+200	300	2	14	with 200 g/L tebuconazole
Cauliflower, broccoli	EU	F	spray	200+200	300	2	14	with 200 g/L tebuconazole
Chinese cabbage	EU	F	spray	200+200	300	2	14	with 200 g/L tebuconazole
Cabbage (head)	EU	F	spray	200+200	300	2	7	with 200 g/L tebuconazole
Carrots	EU	F	spray	150+150	200	3	14	with 200 g/L tebuconazole
Cereals	EU	F	seed	1g/100kg seed		1	-	seed treatment
Cucumber, Courgette	EU	G	spray	300	500	2	1	150 g/ha/meter canopy height
Melon, Watermelon	EU	F	spray	250	500	2	3	
Melon, Water-melon	EU	G	spray	300	1500	2	3	
Grape (Table)	EU	F	spray	250	100	2	3	
Grape (Wine)	EU	F	spray	250	100	2	14	
Leek	EU	F	spray	200+200	200	2	14	with 200 g/L tebuconazole
Lettuce	EU	F/G	spray	250	200	2	7	
Peas	EU	F	spray	250	200	2	7	
Peppers	EU	F	spray	250	500	2	3	
Peppers	EU	G	spray	300	500	2	3	150 g/ha/meter canopy height
Pome fruits	EU	F	spray	150 + 150	300	3	14	with 200 g/L tebuconazole
Pome fruits	EU	F	spray	125	300	4	7	
Rapeseed	EU	F	spray	125+125	100	2	Up to BBCH 73	with 125 g/L prothioconazole
Apricot, Peach	EU	F	spray	125+125	500	3	3	with 200 g/L tebuconazole
Cherry, Plum	EU	F	spray	150+150	500	3	3	with 200 g/L tebuconazole
Strawberries	EU	F/G	spray	250	300	2	1	
Tomato	EU	F	spray	250	500	2	3	
Tomato	EU	G	spray	300	500	2	3	150 g/ha/meter canopy height

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials involving foliar, drip irrigation or seed treatment applications of fluopyram to the following crops.

Group	Commodities	Origin	Table No.
Citrus fruits	Orange, Mandarin, Lemon, Grapefruit	USA	Tables 83–85
Pome fruits	Apple, Pear	Canada, Europe, USA	Tables 86–89
Stone fruits	Peach, Cherry, Plum	Europe, USA	Tables 90–93
Berries and other small fruit	Grapes (wine & table), Strawberry,	Europe, USA	Tables 94–100
	Caneberries (blackberry, raspberry, boysenberry), Blueberry	USA	Tables 101–102
Banana	Bagged and unbagged	Latin America	Table 103
Bulb vegetables	Leek	Europe	Table 104
	Onion (bulb)	Europe, USA	Tables 105–106
	Welsh onion	Europe	Table 107
	Spring onion (bunching onion)	USA	Table 108
Brassica vegetables	Cabbage, Broccoli, Cauliflower	Europe, USA	Tables 109–114
	Brussels sprouts	Europe	Table 115
Fruiting vegetables, Cucurbits	Cucumber	China, USA	Tables 116–117
	Cucumber (drip irrigation)	USA	Table 118
	Cucumber (indoor)	Europe	Table 119
	Melon (foliar, drip irrigation)	USA	Tables 120–121
	Melon (indoor & outdoor)	Europe	Tables 122–123
	Watermelon	Europe	Table 124
	Summer squash (foliar, drip irrigation)	USA	Tables 125–126
Fruiting vegetables, other than Cucurbits	Tomato	USA	Table 127
	Tomato (indoor & outdoor)	Europe	Tables 128–129
	Peppers, Sweet	USA	Table 130
	Peppers, Sweet (indoor & outdoor)	Europe	Tables 131–132
	Chili pepper	USA	Table 133
	Sweetcorn	USA	Table 134

Group	Commodities	Origin	Table No.
Leafy vegetables	Lettuce (broadcast & band sprays)	USA	Tables 135–136
	Lettuce (indoor & outdoor)	Europe	Tables 137–138
	Spinach, Mustard greens	USA	Tables 139–140
	Chinese cabbage	Europe	Table 141
Legume vegetables	Beans - with pod (indoor & outdoor))	Europe, USA	Tables 142–144
	Beans (without pod)	USA	Table 145
	Peas (without pods)	Europe, USA	Tables 146–147
	Peas (with pods)	USA	Table 148
Pulses	Dry Beans, Peas & Soya bean	USA	Tables 149–151
	Bean, Pea & Soya bean forage, fodder	USA	Tables 152–154
Root & tuber vegetables	Carrot	Europe, USA	Tables 155–156
	Potato, Radish, Sugar beet (roots & tops), Turnip tops	USA	Tables 157–160
Stalk & stem vegetables	Artichoke, Globe	Europe, USA	Tables 161–162
	Celery	USA	Table 163
Cereals	Barley & Wheat (grain, forage, straw) – seed treatment	Europe	Tables 164–165
	Wheat, Sorghum & Maize (grain, forage, fodder)	USA	Tables 166–172
Grasses	Pasture grass (forage, hay)	USA	Table 173
Tree nuts	Almond, Pecan	USA	Tables 174–175
Oilseeds	Peanut,	USA	Table 176
	Oilseed rape	Europe, USA	Table 177–178
	Sunflower	USA	Table 179
Dried herbs	Hops (dry)	USA	Table 180
Herbs & spices	Basil, Chives, Dill	USA	Tables 181–183

The supervised trials were well documented with laboratory and field reports. Laboratory reports included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. In such cases, the residues found are noted as “c=nn mg/kg” in the Reference and Comments columns. Residue data are recorded unadjusted for recovery.

Results from replicated field plots are presented as individual values. When residues were not detected they are shown as below the LOQ (e.g. < 0.01 mg/kg). Residues and application rates have been reported as provided in the study reports,although the results from trials used for the estimation

of maximum residue levels (double underlined) have been rounded to two significant digits (or if close to the LOQ, rounded to one significant digit) in the Appraisal.

In some trials, samples were taken just before the final application and then, again on the same day after the spray had dried. In the data tables the notation for these two sampling times is '-0' and '0' respectively.

When multiple applications were made to a crop, the application rate, spray concentration and spray volume were not always identical from one application to the next. If the variation was small, only the final values for application rate, concentration and spray volume were recorded. For larger variations all values were recorded.

In the following tables, abbreviations used for the measured fluopyram metabolites are:

- PAA = pyridyl-acetic acid metabolite, expressed as fluopyram equivalents
- BZM= benzamide metabolite expressed as fluopyram equivalents
- PCA = pyridyl-carboxylic acid metabolite expressed as fluopyram equivalents

Intervals of freezer storage between sampling and analysis were recorded for all trials and were covered by the conditions of the freezer storage stability studies in most cases.

Citrus fruits

Results from supervised trials from USA on oranges (including Satsuma mandarins), lemons and grapefruit were provided to the Meeting. In these trials, two applications of fluopyram (SC 500 formulation) were applied to mature, full-sized trees 5–8 days apart as foliar sprays using ground-based airblast equipment to apply a total of 0.5 kg ai/ha/year. At most sites, two application methods were used, one involving low volume sprays (370-670 litres/ha) and one using high volume treatments (1940–2860 litres/ha). Plot sizes in these trials ranged from 108–325 square metres and involved at least 4 trees per plot.

Duplicate samples of at least 24 fruit were taken from at least 4 trees/plot, frozen within 2.5 hours of sampling, stored at -15 °C for up to 466 days before whole fruit analysis for fluopyram using LC/MS/MS Methods 00984 or 00984/M001, with a reported LOQ of 0.01 mg/kg.

Table 83 Fluopyram residues in oranges and mandarins from supervised trials in USA involving foliar applications (400 SC or 500 SC formulations)

ORANGE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		Fluopyram	PAA	BZM	PCA	
USA, 2006 DeLeon Springs, FL (Hamlin)	2 [b]	0.249 0.255	0.056 0.063	442 407	0 3 7 9 14	0.457, 0.391 0.126, 0.184 0.136 0.146 0.126, 0.175 0.117, 0.118				RAGMP036 GM027-06DA
USA, 2006 DeLeon Springs, FL (Hamlin)	2 [a]	0.256 0.256	0.009 0.011	2835 2273	7	0.138, 0.125				RAGMP036 GM027-06DA
USA, 2006 DeLeon Springs, FL (Navel)	2 [b]	0.255 0.258	0.057 0.063	446 410	7	0.082, 0.114				RAGMP036 GM023-06HA
USA, 2006 DeLeon Springs, FL (Navel)	2 [a]	0.252 0.252	0.009 0.011	2812 2249	7	0.138, 0.116				RAGMP036 GM023-06HA
USA, 2006 Dundee, FL (Hamlin)	2 [b]	0.249 0.254	0.059 0.066	422 385	7	0.146, 0.154				RAGMP036 GM020-06HA

ORANGE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		Fluopyram	PAA	BZM	PCA	
USA, 2006 Dundee, FL (Hamlin)	2 [a]	0.254 0.254	0.012 0.012	2174 2064	7	0.156, 0.121				RAGMP036 GM020-06HA
USA, 2006 Fresno, CA (Washington Navel)	2 [b]	0.251 0.249	0.049 0.049	515 511	6	0.109, 0.126				RAGMP036 GM029-06HA
USA, 2006 Fresno, CA (Washington Navel)	2 [a]	0.251 0.249	0.011 0.011	2358 2366	6	0.130 0.132				RAGMP036 GM029-06HA
USA, 2006 Orland, CA (Mandarin - Satsuma)	2 b	0.248 0.245	0.053 0.053	468 465	7	0.139, 0.271				RAGMP036 GM030-06HA
USA, 2006 Orland, CA (Mandarin - Satsuma)	2 [a]	0.249 0.249	0.011 0.011	2348 2357	7	0.206, 0.175				RAGMP036 GM030-06HA
USA, 2006 Vero Beach, FL (Hamlin)	2 [b]	0.25 0.256	0.053 0.059	473 436	7	0.131, 0.177				RAGMP036 GM024-06HA
USA, 2006 Vero Beach, FL (Hamlin)	2 [a]	0.252 0.257	0.009 0.011	2925 2371	7	0.087, 0.103				RAGMP036 GM024-06HA
USA, 2006 Vero Beach, FL (Pineapple)	2 [b]	0.25 0.254	0.053 0.059	473 433	7	0.127, 0.187				RAGMP036 GM025-06HA
USA, 2006 Vero Beach, FL (Pineapple)	2 [a]	0.255 0.258	0.009 0.011	2922 2384	7	0.081, 0.075				RAGMP036 GM025-06HA
USA, 2007 Ft Pierce, FL (Valencia)	2 [b]	0.252 0.249	0.057 0.058	441 426	7	0.048, 0.039				RAGMP036 GM026-06HA
USA, 2007 Ft. Pierce, FL (Valencia)	2 [a]	0.249 0.25	0.007 0.007	3331 3599	7	0.093, 0.075				RAGMP036 GM026-06HA
USA, 2007 Groveland, FL (Rhode Red Valencia)	2 [b]	0.247 0.247	0.06 0.051	411 484	7	0.329, 0.374				RAGMP036 GM021-06HA
USA, 2007 Groveland, FL (Rhode Red Valencia)	2 [a]	0.249 0.248	0.012 0.011	2043 2173	7	0.278, 0.302				RAGMP036 GM021-06HA
USA, 2007 Haines City, FL (Valencia)	2 [b]	0.251 0.248	0.055 0.055	456 449	7	0.304, 0.323				RAGMP036 GM022-06HA
USA, 2007 Haines City, FL (Valencia)	2 [a]	0.25 0.25	0.01 0.01	2549 2495	7	0.177, 0.175				RAGMP036 GM022-06HA
USA, 2007 Raymondville, TX (N-33 Navels)	2 [b]	0.254 0.254	0.046 0.046	549 553	7	0.039, 0.023				RAGMP036 GM028-06HA
USA, 2007 Raymondville, TX (N-33 Navels)	2 [a]	0.255 0.256	0.011 0.011	2388 2414	7	0.077, 0.036				RAGMP036 GM028-06HA

ORANGE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		Fluopyram	PAA	BZM	PCA	
USA, 2007 Sanger, CA (Washington Navel)	2 [b]	0.248 0.251	0.047 0.047	527 535	7	0.229, 0.181				RAGMP036 GM031-06HA
USA, 2007 Sanger, CA (Washington Navel)	2 [a]	0.249 0.251	0.012 0.012	2076 2160	7	0.311, 0.284				RAGMP036 GM031-06HA

[a] = high volume airblast application (500 SC formulation) at BBCH 78-79 and 5-8 days later

[b] = low volume airblast application (500 SC formulation) at BBCH 78-79 and 5-8 days later

Table 84 Fluopyram residues in lemons from supervised trials in USA involving foliar applications (400 SC or 500 SC formulations)

LEMON Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		Fluopyram	PAA	BZM	PCA	
USA, 2006 Porterville, CA (Lisbon)	2 [b]	0.254 0.247	0.046 0.041	556 599	7	0.257, 0.280				RAGMP036 GM033-06HA
USA, 2006 Porterville, CA (Lisbon)	2 [a]	0.257 0.25	0.009 0.009	2736 2832	7	0.206, 0.403				RAGMP036 GM033-06HA
USA, 2007 Arroyo Grande, CA (Eureka)	2 [b]	0.259 0.251	0.067 0.063	389 399	7	0.313, 0.327				RAGMP036 GM035-06HA
USA, 2007 Arroyo Grande, CA (Eureka)	2 [a]	0.259 0.249	0.01 0.011	2610 2334	7	0.339, 0.311				RAGMP036 GM035-06HA
USA, 2007 Fresno, CA (Meyer)	2 [b]	0.251 0.248	0.051 0.051	495 490	0 3 7 10 14	0.499, 0.626 0.247, 0.387 0.307, 0.328 0.330, 0.224 0.351, 0.168				RAGMP036 GM036-06DA
USA, 2007 Fresno, CA (Meyer)	2 [a]	0.258 0.254	0.009 0.009	2899 2852	7	0.214, 0.205				RAGMP036 GM036-06DA
USA, 2007 Ft Pierce, FL (Bearrs)	2 [b]	0.257 0.254	0.053 0.048	485 524	7	0.210, 0.252				RAGMP036 GM032-06HA
USA, 2007 Ft Pierce, FL (Bearrs)	2 [a]	0.246 0.258	0.01 0.01	2472 2606	7	0.389, 0.353				RAGMP036 GM032-06HA
USA, 2007 Sanger, CA (Frost Lisbon)	2 [b]	0.249 0.25	0.047 0.047	532 534	7	0.184, 0.181				RAGMP036 GM034-06HA
USA, 2007 Sanger, CA (Frost Lisbon)	2 [a]	0.249 0.25	0.012 0.012	2101 2157	7	0.400, 0.439				RAGMP036 GM034-06HA

[a] = high volume airblast application (500 SC formulation) at BBCH 78-79 and 5-8 days later

[b] = low volume airblast application (500 SC formulation) at BBCH 78-79 and 5-8 days later

Table 85 Fluopyram residues in grapefruit from supervised trials in USA involving foliar applications (400 SC or 500 SC formulations)

GRAPEFRUIT Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		Fluopyram	PAA	BZM	PCA	
USA, 2006 Dundee, FL (White Marsh)	2 [b]	0.249 0.251	0.055 0.061	453 409	0 3 7 10 14	0.596, 1.366 0.161, 0.211 0.139, 0.133 0.12, 0.106 0.069, 0.061				RAGMP036 GM039-06DA
USA, 2006 Dundee, FL (White Marsh)	2 [a]	0.25 0.252	0.011 0.012	2299 2175	7	0.101, 0.195				RAGMP036 GM039-06DA
USA, 2006 Porterville, CA (Mellow Gold)	2 b	0.247 0.25	0.038 0.039	644 646	7	0.089, 0.08				RAGMP036 GM041-06HA
USA, 2006 Porterville, CA (Mellow Gold)	2 [a]	0.25 0.25	0.012 0.012	2004 2007	7	0.162, 0.149				RAGMP036 GM041-06HA
USA, 2006 Vero Beach, FL (Red)	2 [b]	0.251 0.255	0.053 0.059	476 434	7	0.089, 0.057				RAGMP036 GM038-06HA
USA, 2006 Vero Beach, FL (Red)	2 [a]	0.255 0.251	0.009 0.011	2884 2340	7	0.063, 0.046				RAGMP036 GM038-06HA
USA, 2006 Vero Beach, FL (White)	2 [b]	0.251 0.256	0.053 0.059	475 436	7	0.101, 0.078				RAGMP036 GM037-06HA
USA, 2006 Vero Beach, FL (White)	2 [a]	0.252 0.25	0.009 0.011	2861 2327	7	0.072, 0.051				RAGMP036 GM037-06HA
USA, 2007 Raymondville, TX (Rio Red)	2 [b]	0.257 0.254	0.046 0.046	554 551	7	0.052, 0.037				RAGMP036 GM040-06HA
USA, 2007 Raymondville, TX (Rio Red)	2 [a]	0.255 0.256	0.011 0.011	2395 2416	7	0.031, 0.041				RAGMP036 GM040-06HA
USA, 2007 Sanger, CA (Rio Red)	2 [b]	0.254 0.25	0.047 0.039	539 646	7	0.046, 0.051				RAGMP036 GM042-06HA
USA, 2007 Sanger, CA (Rio Red)	2 [a]	0.255 0.25	0.012 0.039	2131 646	7	0.137, 0.194				RAGMP036 GM042-06HA

[a] = high volume airblast application (500 SC formulation) at BBCH 78-79 and 5-8 days later

[b] = low volume airblast application (500 SC formulation) at BBCH 78-79 and 5-8 days later

Pome fruits

Results from supervised trials from Europe on apples and pears were provided to the Meeting. In these trials, 3–4 applications of fluopyram (SC 500 or SC 400 formulations) were applied to mature, full-sized (approximately 3 metres high) trees 6–8 days apart as foliar sprays using knapsack sprayers with single solid or hollow-cone nozzles, mini-booms (3–5 flat-fan nozzles) or single rotary atomiser nozzles, applying 0.125 or 0.15 kg ai/ha in 500–1500 L water/ha. Plot sizes in these trials ranged from 30–400 square metres and involved at least 4 trees per plot.

Unreplicated samples of at least 24 fruit (approximately 3–5 kg) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 363 days before whole

fruit analysis for fluopyram and its BZM, PCA and PAA metabolites using LC/MS/MS Methods 00984 or 00984/M001.

Table 86 Residues in apples from supervised trials in Belgium, France, Germany, Italy, Portugal, Spain and UK involving foliar applications of fluopyram (400 SC or 500 SC formulations)

APPLE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France (N), 2006 Lignieres de Touraine (Granny Smith)	4	0.125	0.0085	1500	-0 0 1 3 7 9	0.07 0.21 0.15 0.09 0.05 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2605/06 0394-06
Belgium, 2006 Fleurus (Jonagored)	4	0.125	0.0125	1000	-0 0 1 3 7 10	0.06 0.14 0.11 0.11 0.1 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2605/06 & RA-3605/06 0396-06
United Kingdom, 2006 Royston (Jonathon)	4	0.125	0.025	500	-0 0 1 3 8 10	0.09 0.25 0.24 0.21 0.13 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2605/06 & RA-3605/06 0413-06
Germany, 2007 Monheim (Roter Boskoop)	4	0.125	0.0126	1000	-0 0 3 7 14 21 28	0.29 0.35 0.29 0.31 0.28 0.2 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2579/07 0214-07 400 SC
France (N), 2007 Lignieres de Touraine (Granny Smith)	4	0.125	0.0084	1500	-0 0 3 7 14 21 28	0.13 0.22 0.16 0.15 0.11 0.09 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2579/07 0479-07
United Kingdom, 2007 Great Chishill (Jonathon)	4	0.125-0.133	0.025	500-530 (last)	-0 0 3 7 14 21 28	0.15 0.24 0.2 0.17 0.15 0.15 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2579/07 0480-07
Germany, 2007 Monheim (Roter Boskoop)	3	0.15	0.015	1000	-0 0 3 7 14 21 28	0.14 0.27 0.21 0.22 0.21 0.21 0.16	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2577/07 0177-07
France (N), 2007 Lignieres de Touraine (Granny Smith)	3	0.15	0.01	1500	-0 0 3 7 14 21 28	0.12 0.22 0.18 0.15 0.12 0.09 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2577/07 0468-07

APPLE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
United Kingdom, 2007 Royston (Jonathon)	3	0.15	0.03	500	-0 0 3 7 14 21 28	0.08 0.2 0.12 0.08 0.11 0.1 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2577/07 0469-07
Portugal, 2006 Peral-Cadaval (Fuji)	4	0.125	0.0155	800	-0 0 1 3 7 10	0.08 0.3 0.18 0.15 0.15 0.14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2606/06 0397-06
France (S), 2006 Reyniès (Granny Smith)	4	0.125	0.0085	1500	-0 0 1 3 7 10	0.06 0.13 0.13 0.08 0.07 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2606/06 & RA-3606/06 0399-06
Italy, 2006 Zevio (Golden Rainders)	4	0.125	0.01	1250	-0 0 1 3 7 10	0.09 0.2 0.1 0.11 0.06 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2606/06 RA-3606/06 0414-06
France (S), 2007 Corbarieu (Golden)	4	0.125	0.0084	1500	-0 0 3 7 14 21 28	0.11 0.19 0.19 0.18 0.15 0.14 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2580/07 0216-07
Spain, 2007 Bellmunt d'Urgell (Golden)	4	0.125	0.0084	1500	-0 0 3 7 15 21 [a] 28	0.26 0.29 0.16 0.14 0.13 0.21 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2580/07 0482-07
Portugal, 2007 Peral - Cadaval (Fuji)	4	0.125	0.0126	1000	-0 0 3 7 14 21 28	0.19 0.32 0.21 0.23 0.17 0.17 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2580/07 0483-07
France (S), 2007 Corbarieu (Golden)	3	0.15	0.01	1500	-0 0 3 7 14 21 28	0.07 0.15 0.21 0.11 0.12 0.09 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2578/07 0212-07

APPLE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Spain, 2007 Caldes de Malavella - Girona (Golden Smoothy)	3	0.15	0.01	1500	-0 0 2 7 14 21 28	0.2 0.27 0.27 0.23 0.23 0.21 0.16	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2578/07 0476-07
Portugal, 2007 Peral - Cadaval (Fuji)	3	0.15	0.015	1000	-0 0 3 7 14 21 28	0.22 0.43 0.39 0.22 0.19 0.18 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2578/07 0477-07

Trials involving the SC 400 formulation (with 200 g ai/L tebuconazole) are identified in the Reference & Comments column

[a] Results are the mean of 3 duplicate analyses

Table 87 Residues in pears from supervised trials in Germany, Greece and Italy involving foliar applications of fluopyram (400 SC or 500 SC formulations)

PEAR Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	No	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Germany, 2006 D-51399 Burscheid (Herzogin Elsa)	4	0.125	0.0125	1000	-0 0 1 3 7 10	0.3 0.44 0.45 0.39 0.31 0.26	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2605/06 0395-06
Germany, 2007 Dürrweitzschen (Conference)	4	0.125	0.025	500	-0 0 3 7 14 21 28	0.25 0.24 0.22 0.2 0.2 0.14 0.14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2579/07 0215-07
Germany, 2007 Dürrweitzschen (Conference)	3	0.15	0.03	500	-0 0 3 7 14 21 28	0.17 0.29 0.16 0.18 0.12 0.12 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2577/07 0211-07
Greece, 2006 Alexandria (Etrusca)	4	0.125	0.0165	750	-0 0 1 3 7 10	0.27 0.35 0.44 0.4 0.34 0.27	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2606/06 0398-06
Italy, 2007 Aibaro di Ronco all'Adige (Conference)	4	0.125	0.0084	1500	-0 [a] 0 [b] 3 7 14 21 28	0.14 0.13 0.13 0.14 0.12 0.1 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2580/07 0217-07

PEAR Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	No	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Italy, 2007 Bologna (Conference)	3	0.15	0.015	1000	-0 0 7 14 21 28	0.32 0.6 0.42 0.35 0.4 0.34	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2578/07 0213-07

[a] Results are the mean of 3 replicate analyses

[b] Results are the mean of 5 replicate analyses

Results from supervised trials from USA on apples and pears were provided to the Meeting. In these trials, two applications of fluopyram (SC 500 formulation,) were applied to mature, full-sized trees 5–8 days apart as foliar sprays using ground-based airblast equipment to apply a total of 0.5 kg ai/ha/year. At most sites, two application methods were used, one involving low volume sprays (370–670 litres/ha) and one using high volume treatments (1940–2860 litres/ha). Plot sizes in these trials ranged from 25–280 square metres and involved at least 4 trees per plot.

Duplicate samples of at least 2 kg fruit were taken from at least 4 trees/plot, frozen within 4 hours of sampling, stored at -15 °C from 54–484 days before whole fruit analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 88 Fluopyram residues in apples from supervised trials in USA and Canada involving foliar applications (500 SC formulation)

APPLE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	No	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Covesville, VA (Jonathan)	2 [b]	0.25 0.249	0.044 0.043	573 576	0 7	0.2, 0.207 0.236 0.247				RAGMP050 GM145-06HB
USA, 2006 Covesville, VA (Jonathan)	2 [a]	0.251 0.253	0.012 0.012	2146 2177	0 7	0.176, 0.149 0.163, 0.174				RAGMP050 GM145-06HB
USA, 2006 North Rose, NY (Ida Red)	2 [b]	0.253 0.252	0.041 0.041	617 610	0 7	0.141, 0.16 0.07, 0.067				RAGMP050 GM146-06HA
USA, 2006 North Rose, NY (Ida Red)	2 [a]	0.251 0.248	0.012 0.012	2112 2105	0 7	0.178, 0.144 0.084, 0.086				RAGMP050 GM146-06HA
USA, 2006 Hereford, Pennsylvania (Bisbee Red Delicious)	2 [b]	0.256 0.254	0.041 0.041	625 618	0 7	0.707 0.796 0.201, 0.191				RAGMP050 GM147-06HA
USA, 2006 Hereford, Pennsylvania (Bisbee Red Delicious)	2 [a]	0.251 0.252	0.011 0.011	2271 2362	0 7	0.421, 0.454 0.088, 0.113				RAGMP050 GM147-06HA
USA, 2006 Blairsville, Georgia (Empire)	2 [b]	0.248 0.249	0.061 0.068	409 368	0 7	0.162, 0.12 0.067, 0.053				RAGMP050 GM148-06HA

APPLE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	No	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Blairsville, Georgia (Empire)	2 [a]	0.253 0.258	0.01 0.009	2618 2778	0 7	0.07, 0.089 0.063, 0.062				RAGMP050 GM148-06HA
USA, 2006 Conklin, Michigan (Golden Delicious)	2 [b]	0.253 0.25	0.043 0.042	585 596	0 7	0.194, 0.116 0.113, 0.211				RAGMP050 GM149-06HA
USA, 2006 Conklin, Michigan (Golden Delicious)	2 [a]	0.249 0.248	0.011 0.01	2358 2378	0 7	0.195, 0.212 0.076, 0.076				RAGMP050 GM149-06HA
USA, 2006 Stockholm, Wisconsin (Honeycrisp)	2 [b]	0.251 0.249	0.046 0.046	542 543	0 7	0.108, 0.136 0.065, 0.073				RAGMP050 GM150-06HA
USA, 2006 Stockholm, Wisconsin (Honeycrisp)	2 [a]	0.252 0.25	0.013 0.013	1956 1941	0 7	0.16, 0.174 0.093, 0.147				RAGMP050 GM150-06HA
USA, 2006 Perry, Utah (Gala)	2 [b]	0.248 0.251	0.045 0.04	546 633	0 7	0.218, 0.2 0.2, 0.134				RAGMP050 GM151-06HA
USA, 2006 Perry, Utah (Gala)	2 [a]	0.252 0.254	0.012 0.011	2163 2225	0 7	0.202, 0.215 0.249 0.262				RAGMP050 GM151-06HA
USA, 2006 Sanger, California (Granny Smith)	2 [b]	0.249 0.25	0.051 0.053	490 476	0 7	0.054, 0.08 0.068, 0.042				RAGMP050 GM152-06HA
USA, 2006 Sanger, California (Granny Smith)	2 [a]	0.25 0.251	0.012 0.012	2082 2069	0 7	0.088, 0.076 0.059, 0.084				RAGMP050 GM152-06HA
USA, 2006 Ephrata, Washington (Red Delicious)	2 [b]	0.256 0.259	0.045 0.045	570 575	0 3 7 10 14	0.147, 0.172 0.14, 0.17 0.078, 0.135 0.066, 0.085 0.067, 0.097				RAGMP050 GM153-06DA
USA, 2006 Ephrata, Washington (Red Delicious)	2 [a]	0.251 0.25	0.012 0.012	2109 2098	0 7	0.117, 0.149 0.086, 0.087				RAGMP050 GM153-06DA
USA, 2006 Parma, Idaho (Early Spur Rome)	2 [b]	0.244 0.25	0.042 0.047	577 536	0 7	0.186, 0.164 0.088, 0.104				RAGMP050 GM154-06HA
USA, 2006 Parma, Idaho (Early Spur Rome)	2 [a]	0.248 0.248	0.01 0.01	2415 2418	0 7	0.157, 0.158 0.085, 0.087				RAGMP050 GM154-06HA
USA, 2006 Hood River, Oregon (Jonagold)	2 [b]	0.253 0.25	0.043 0.044	594 562	0 7	0.19, 0.214 0.198, 0.136				RAGMP050 GM155-06HA

APPLE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	No	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Hood River, Oregon (Jonagold)	2 [a]	0.254 0.254	0.009 0.009	2700 2860	0 7	0.137, 0.095 0.069, 0.093				RAGMP050 GM155-06HA
USA, 2006 Ephrata, Washington (Braeburn)	2 [b]	0.253 0.252	0.045 0.045	562 562	0 7	0.286, 0.173 0.076, 0.072				RAGMP050 GM156-06HA
USA, 2006 Ephrata, Washington (Braeburn)	2 [a]	0.251 0.251	0.012 0.012	2104 2113	0 7	0.215, 0.185 0.086, 0.057				RAGMP050 GM156-06HA
USA, 2006 Centralia, Illinois (Rome)	2 [b]	0.257 0.251	0.038 0.039	671 651	0 7	0.221, 0.262 0.163, 0.123				RAGMP050 GM347-06HA
USA, 2006 Beldenville, Wisconsin (Fireside)	2 [b]	0.258 0.259	0.054 0.053	482 485	0 7	0.214, 0.161 0.048, 0.081				RAGMP050 GM348-06HA
Canada, 2006 Rockwood, Ontario (Honeycrisp)	2 [b]	0.251 0.252	0.053 0.052	478 485	0 7	0.271, 0.209 0.137, 0.118				RAGMP050 GM349-06HA
Canada, 2006 Clarksburg, Ontario (MacIntosh)	2 [b]	0.251 0.24	0.044 0.042	574 565	0 3 7 10 14	0.181, 0.23 0.086, 0.106 0.04, 0.051 0.098, 0.041 0.043, 0.034				RAGMP050 GM350-06DA
Canada, 2006 Clarksburg, Ontario (MacIntosh)	4 [b]	0.139 0.133 0.133 0.129	0.025 0.028	552 480 570 530	0 3 7 10 14	0.119, 0.109 0.077, 0.083 0.061, 0.072 0.07, 0.064 0.076, 0.052				RAGMP050 GM350-06DA
USA, 2006 Conklin, Michigan (Red Delicious)	2 [b]	0.25 0.25	0.023 0.024	534 525	0 3 7 10 14	0.4, 0.374 0.286, 0.274 0.19, 0.21 0.185, 0.159 0.122, 0.106				RAGMP050 GM351-06DA
USA, 2006 Conklin, Michigan (Red Delicious)	4 [b]	0.133 0.134 0.134 0.134	0.047 0.048	511 514 511 531	0 3 7 10 14	0.174, 0.16 0.136, 0.139 0.107, 0.095 0.091, 0.088 0.066, 0.087				RAGMP050 GM351-06DA

[a] =high volume airblast application

[b] = low volume airblast application

Table 89 Fluopyram residues in pears from supervised trials in USA and Canada involving foliar applications (500 SC formulation)

PEAR Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	No	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Orefield, Pennsylvania (Pear Bartlett)	2 [b]	0.252 0.252	0.053 0.053	471 472	0 7	0.266, 0.406 0.161, 0.296				RAGMP050 GM157-06HA
USA, 2006 Orefield, Pennsylvania (Pear Bartlett)	2 [a]	0.258 0.245	0.01 0.01	2671 2565	0 7	0.156, 0.158 0.245, 0.237				RAGMP050 GM157-06HA
USA, 2006 Marysville, California (Pear Bartlett)	2 [b]	0.248 0.247	0.053 0.053	469 467	0 7	0.152, 0.166 0.163 0.099				RAGMP050 GM158-06HA
USA, 2006 Marysville, California (Pear Bartlett)	2 [a]	0.247 0.247	0.011 0.011	2338 2333	0 7	0.198, 0.204 0.155, 0.096				RAGMP050 GM158-06HA
USA, 2006 Parlier, California (Pear Honsui (Asian Pear))	2 [b]	0.251 0.25	0.047 0.046	538 539	0 6	0.095, 0.233 0.207, 0.21				RAGMP050 GM159-06HA
USA, 2006 Parlier, California (Pear Honsui (Asian Pear))	2 [a]	0.249 0.25	0.011 0.011	2222 2336	0 6	0.115, 0.166 0.205, 0.186				RAGMP050 GM159-06HA
USA, 2006 Hood River, Oregon (Pear Red Clapp)	2 [b]	0.249 0.256	0.054 0.055	462 464	0 3 7 10 14	0.157, 0.132 0.102, 0.099 0.129, 0.114 0.099, 0.081 0.059, 0.054				RAGMP050 GM160-06DA
USA, 2006 Hood River, Oregon (Pear Red Clapp)	2 [a]	0.249 0.254	0.01 0.01	2510 2487	0 7	0.26, 0.239 0.214, 0.152				RAGMP050 GM160-06DA
USA, 2006 Payette, Idaho (Pear Bartlett)	2 [b]	0.251 0.25	0.041 0.042	616 596	0 6	0.238, 0.363 0.188, 0.207				RAGMP050 GM161-06HA
USA, 2006 Payette, Idaho (Pear Bartlett)	2 [a]	0.252 0.243	0.01 0.01	2507 2453	0 6	0.417, 0.624 0.368, 0.308				RAGMP050 GM161-06HA
USA, 2006 Ephrata, Washington (Pear Concord)	2 [b]	0.251 0.25	0.044 0.044	566 566	0 7	0.511, 0.564 0.489 0.505o				RAGMP050 GM162-06HA
USA, 2006 Ephrata, Washington (Pear Concord)	2 [a]	0.251 0.252	0.012 0.012	2115 2118	0 7	0.663, 0.465 0.441, 0.372				RAGMP050 GM162-06HA

[a] =high volume airblast application

[b] = low volume airblast application

Stone fruits

Results from supervised trials from Europe on peaches were provided to the Meeting. In these trials, 3 foliar spray applications of an SC 400 formulation of 200 g ai/L fluopyram (also containing 200 g ai/L tebuconazole) were made at 7 day intervals using knapsack sprayers with single solid or hollow-cone nozzles, mini-booms (3 flat-fan nozzles) or single rotary atomiser nozzles, applying 0.125 kg ai/ha in 700–1300 litres water/ha. Plot sizes in these trials ranged from 50–141 square metres.

Unreplicated samples of at least 24 fruit (approximately 2–5 kg) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 285 days before analysis for fluopyram and its BZM, PCA and PAA metabolites using LC/MS/MS Method 00984.

Table 90 Residues in peaches from supervised trials in France, Greece, Italy, Portugal and Spain involving 3 foliar applications of fluopyram (400 SC formulation).

PEACH Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Morancé (Elegant Lady)	3	0.124	0.0124	1000	-0 0 3 8 10 14	0.1 0.16 0.15 0.13 0.11 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2581/06 R 2006 0362 2 0362-06
Italy, 2006 Bologna (Red Haven)	3	0.124	0.01	1250	-0 0 3 7 10 14	0.09 0.25 0.2 0.19 0.11 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2581/06 R 2006 0487 4 0487-06
Spain, 2006 La Fortesa (Royal Glory)	3	0.124	0.0104	1200	-0 0 3 7 10 14	0.24 0.35 0.34 0.3 0.19 0.25	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2581/06 R 2006 0488 2 0488-06
Portugal, 2006 Fazendas (Royal Gene)	3	0.124	0.0166	750	-0 0 3 7 10 14	0.08 0.13 0.11 0.06 0.08 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2581/06 R 2006 0489 0 0489-06
France, 2007 Montjoire (Redwing)	3	0.125	0.01	1250	-0 0 3 7 10 14	0.13 0.28 0.24 0.16 0.11 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/07 R 2007 0225 6 0225-07
Spain, 2007 La Fortesa (Spring lady)	3	0.125	0.0096	1300	-0 0 2 7 9 14	0.24 0.5 0.33 0.25 0.18 0.28	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/07 R 2007 0496 8 0496-07
Italy, 2007 San Martino (Elegan Lady)	3	0.125	0.0125	1000	-0 0 3 7 10 14	0.21 0.51 0.27 0.27 0.34 0.22	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/07 R 2007 0497 6 0497-07

PEACH Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Greece, 2007 Neokastro (Luande)	3	0.125	0.0178	700	-0 0 3 7 10 14	0.21 0.43 0.26 0.19 0.14 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/07 R 2007 0498 4 0498-07

Results from supervised trials from USA on peaches, cherries and plums were provided to the Meeting. In these trials, two applications of fluopyram (SC 500 formulation) were applied to mature, full-sized trees 5-8 days apart as foliar sprays using ground-based airblast equipment to apply a total of 0.5 kg ai/ha/year. At most sites, two application methods were used, one involving low volume sprays (370–625 litres/ha) and one using high volume treatments (1900–3350 litres/ha). Plot sizes in these trials ranged from 56–364 square metres and involved at least 4 trees per plot.

Duplicate samples of at least 24 fruit were taken from at least 4 trees/plot, frozen within 5 hours of sampling, stored at -15 °C for up to 561 days before analysis for fluopyram using a modification of LC/MS-MS Method 00984 (GM-001-P07-01), with a reported LOQ of 0.01 mg/kg.

Table 91 Fluopyram residues in peaches from supervised trials in USA involving two foliar applications (500 SC formulations)

PEACH Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA,2006 Orefield, Pennsylvania (Glen Glow)	2	0.249 0.245	0.053 0.053	466 460	0	0.317, 0.298				RAGMP056 GM184-06HA
USA,2006 Chula, Georgia (Hawthornes)	2	0.248 0.25	0.052 0.052	478 481	0	0.427, 0.454				RAGMP056 GM185-06HA
USA,2006 Alto, Georgia (Cara Gem)	2	0.245 0.251	0.046 0.047	537 538	0	0.27, 0.331				RAGMP056 GM186-06HA
USA,2006 Blairsville, Georgia (Cresthaven)	2	0.239 0.25	0.064 0.061	371 412	0	0.314, 0.27				RAGMP056 GM187-06HA
USA,2006 Conklin, Michigan (Bellaire)	2	0.253 0.252	0.045 0.045	557 554	0	0.457, 0.352				RAGMP056 GM188-06HA
USA,2007 Hondo, Texas (LaFeliciana)	2	0.248 0.247	0.043 0.046	577 537	0	0.179, 0.126				RAGMP056 GM189-06HB
USA,2006 Marysville, California (Stanislaus)	2	0.247 0.247	0.053 0.053	467 467	0	0.15, 0.161				RAGMP056 GM191-06HA
USA,2006 Sanger, CA (White Lady)	2	0.25 0.25	0.053 0.053	469 470	0	0.168, 0.227				RAGMP056 GM192-06HA
USA,2006 Madera, California (Angelos)	2	0.256 0.256	0.049 0.049	527 527	0 3 7 10 14	0.33, 0.397 0.428, 0.397 0.444, 0.457 0.397, 0.367 0.346, 0.363				RAGMP056 GM190-06DA
USA,2006 Orefield, Pennsylvania (Glen Glow)	2	0.261 0.251	0.011 0.011	2447 2356	0	0.319, 0.429				RAGMP056 GM184-06HA

PEACH Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA,2006 Chula, Georgia (Hawthornes)	2	0.25 0.25	0.009 0.009	2758 2753	0	0.331, 0.381				RAGMP056 GM185-06HA
USA,2006 Alto, Georgia (Cara Gem)	2	0.252 0.25	0.01 0.01	2507 2483	0	0.297, 0.317				RAGMP056 GM186-06HA
USA,2006 Blairsville, Georgia (Cresthaven)	2	0.258 0.253	0.009 0.01	2793 2613	0	0.588, 0.509				RAGMP056 GM187-06HA
USA,2006 Conklin, Michigan (Bellaire)	2	0.251 0.248	0.013 0.013	1968 1962	0	0.307, 0.345				RAGMP056 GM188-06HA
USA,2007 Hondo, Texas (LaFeliciana)	2	0.254 0.24	0.008 0.007	3132 3350	0	0.175, 0.203				RAGMP056 GM189-06HB
USA,2006 Madera, California (Angelos)	2	0.245 0.25	0.012 0.01	2030 2413	0	0.561, 0.385				RAGMP056 GM190-06DA
USA,2006 Marysville, California (Stanislaus)	2	0.249 0.248	0.011 0.011	2350 2342	0	0.313, 0.189				RAGMP056 GM191-06HA
USA, 2006 Sanger, CA (White Lady)	2	0.25 0.25	0.012 0.012	2120 2132	0	0.194, 0.271				RAGMP056 GM192-06HA

Table 92 Fluopyram residues in cherries from supervised trials in USA involving two foliar applications (500 SC formulations)

CHERRY Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Conklin, Michigan (Montmorency)	2	0.252 0.25	0.047 0.045	534 551	0	0.51, 0.597				RAGMP056 GM178-06HA (Sour cherry)
USA, 2006 Orefield, Pennsylvania (Montmorency)	2	0.257 0.259	0.053 0.053	481 485	0	0.637, 0.5				RAGMP056 GM179-06HA (Sour cherry)
USA, 2006 Shelby, Michigan (Gold)	2	0.251 0.253	0.041 0.041	611 624	0	0.641, 0.638				RAGMP056 GM180-06HA
USA, 2006 Marysville, California (Rainier)	2	0.254 0.256	0.061 0.061	419 420	0	0.0663, 0.0662				RAGMP056 GM181-06HA
USA, 2006 Mosier, Oregon (Bing)	2	0.25 0.254	0.049 0.05	506 508	0	0.194, 0.228				RAGMP056 GM183-06HA
USA, 2006 Ephrata, Washington (Bing)	2	0.251 0.25	0.041 0.041	606 603	0 3 7 10 14	0.547, 0.48 0.397, 0.432 0.426, 0.356 0.269, 0.295 0.273, 0.294				RAGMP056 GM182-06DA c=0.04 mg/kg
USA, 2006 Conklin, Michigan (Montmorency)	2	0.252 0.25	0.013 0.013	1905 1946	0	1.229, 1.12				RAGMP056 GM178-06HA (Sour cherry)

CHERRY Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Orefield, Pennsylvania (Montmorency)	2	0.249 0.252	0.011 0.011	2327 2350	0	0.656, 0.603				RAGMP056 GM179-06HA (Sour cherry)
USA, 2006 Shelby, Michigan (Gold)	2	0.245 0.259	0.012 0.012	2117 2121	0	0.583, 0.437				RAGMP056 GM180-06HA
USA, 2006 Marysville, California (Rainier)	2	0.261 0.251	0.012 0.012	2194 2115	0	0.162, 0.147				RAGMP056 GM181-06HA
USA, 2006 Ephrata, Washington (Bing)	2	0.25 0.248	0.011 0.011	2339 2318	0	0.346, 0.355				RAGMP056 GM182-06DA c=0.04 mg/kg
USA, 2006 Mosier, Oregon (Bing)	2	0.254 0.252	0.009 0.009	2889 2862	0	0.309, 0.25				RAGMP056 GM183-06HA

Table 93 Fluopyram residues in plums from supervised trials in USA involving foliar applications (500 SC formulations)

PLUM Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Conklin, Michigan (Stanley)	2	0.25 0.247	0.044 0.043	574 569	0	0.045, 0.043				RAGMP056 GM193-06HA
USA, 2006 Live Oak, California (French)	2	0.25 0.249	0.053 0.053	469 471	0	0.022, 0.024				RAGMP056 GM195-06HA
USA, 2006 Selma, California (Howard)	2	0.244 0.25	0.05 0.049	484 508	0	0.229, 0.286				RAGMP056 GM196-06HA
USA, 2006 Hickman, California (Grand Rosa)	2	0.245 0.245	0.057 0.057	433 433	0	0.079, 0.059				RAGMP056 GM197-06HA
USA, 2006 Newberg, Oregon (Italian)	2	0.242 0.239	0.06 0.059	405 405	0	0.063, 0.069				RAGMP056 GM198-06HA
USA, 2006 Madera, California (Santa Rosa)	2	0.243 0.249	0.044 0.044	553 566	0 3 7 10 14	0.03, 0.036 0.045, 0.034 0.041, 0.052 0.037, 0.033 0.021, 0.024				RAGMP056 GM194-06DA
USA, 2006 Conklin, Michigan (Stanley)	2	0.251 0.251	0.012 0.012	2074 2047	0	0.082, 0.055				RAGMP056 GM193-06HA
USA, 2006 Madera, California (Santa Rosa)	2	0.253 0.247	0.011 0.011	2403 2338	0	0.033, 0.028				RAGMP056 GM194-06DA
USA, 2006 Live Oak, California (French)	2	0.251 0.251	0.013 0.013	1923 1935	0	0.026, 0.021				RAGMP056 GM195-06HA
USA, 2006 Selma, California (Howard)	2	0.248 0.254	0.009 0.054	2817 471	0	0.292, 0.275				RAGMP056 GM196-06HA

PLUM Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Hickman, California ()	2	0.254 0.254	0.009 0.009	2812 2816	0	0.033, 0.032				RAGMP056 GM197-06HA
USA, 2006 Newberg, Oregon ()	2	0.248 0.248	0.012 0.012	2147 2133	0	0.057, 0.058				RAGMP056 GM198-06HA

Berries and small fruit

Grapes

Results from supervised trials from Europe on wine and table grapes were provided to the Meeting. In these trials, foliar spray applications of an SC 500 formulation of fluopyram were made at the start of ripening (BBCH 81) and repeated 3 days before commercial harvest, with spray intervals of 7-60 days (but mostly between 21 and 42 days). Applications were made using knapsack mistblowers, backpack sprayers with single nozzle hand lances or mini-booms (3–12 flat-fan nozzles) or a tractor-mounted tunnel-sprayer, applying 0.25 kg ai/ha in 150-1000 litres water/ha. Plot sizes in these trials ranged from 26–320 square metres.

Unreplicated samples of at least 12 bunches of grapes (2-6 kg) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 266 days before analysis for fluopyram and its BZM, PAA and PCC metabolites using either LC/MS/MS Method 00984 or Method 00984/M001. The reported LOQs were 0.01 mg/kg for each analyte.

Table 94 Residues in wine grapes (bunches) from supervised trials in France and Germany involving 2 foliar applications of fluopyram (500 SC formulation).

WINE GRAPE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Athée sur Cher (Gamay) Red	2	0.25	0.125	200	-0 0 3 7 14 21 28	0.28 0.66 <u>0.58</u> 0.5 0.4 0.5 0.56	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.02 0.02 0.02 0.02 0.03	0.02 0.02 0.02 0.02 0.02 0.03 0.03	RA-2611/06 & RA-3611/06 R 2006 0415 7 35d spray interval
Germany, 2006 Kressbronn / Berg (Müller-thurgau) White	2	0.265	0.05	530	-0 0 3 8 14 21 28	0.39 0.91 1.0 0.72 0.71 0.62 0.65	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.01 0.02 0.02 0.02 0.03 0.03	0.03 0.02 0.03 0.03 0.04 0.04 0.07	RA-2611/06 R 2006 0416 5 22d spray interval
France, 2006 Charnay les Macon (Pinot noir/PG161.49) Red	2	0.25	0.125	200	-0 0 3 7 14 21 28	0.22 0.51 <u>0.51</u> 0.48 0.41 0.36 0.3	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2611/06 & RA-3611/06 R 2006 0650 8 28d spray interval

WINE GRAPE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2007 Saint Nicolas de Bourgueil (Cabernet franc) Red	2	0.25	0.167	150	-0 0 3 7 14 21	0.17 0.72 0.57 0.57 0.57 0.46	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.01 0.02 0.02 0.03	0.04 0.03 0.05 0.05 0.07 0.08	RA-2500/07 R 2007 0001 6 32d spray interval
Germany, 2007 Duttweiler (Silvaner) White	2	0.25	0.032	800	-0 0 3 7 14 21	0.4 0.72 0.61 0.7 0.65 0.63	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2500/07 R 2007 0484 4 0484-07 60d spray interval
France, 2007 Athée sur Cher (Gamay) Red	2	0.25	0.125	200	-0 0 3 7 14 21	0.2 0.63 0.63 0.55 0.48 0.44	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.01 0.02 0.02 0.02	0.03 0.03 0.04 0.04 0.05 0.07	RA-2500/07 R 2007 0485 2 32d spray interval
Germany, 2007 Kressbronn (Müller-thurgau) White	2	0.25	0.025	1000	-0 0 3 7 13 21	0.14 0.56 0.48 0.3 0.3 0.29	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.01 0.02 0.02	0.02 0.02 0.03 0.03 0.04 0.05	RA-2500/07 R 2007 0486 0 0486-07 28d spray interval
France, 2006 Laudun (Grenache Noir Red	2	0.25	0.125	200	-0 0 3 7 14 21 28	0.17 0.44 0.36 0.29 0.23 0.27 0.35	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-2647/06 & RA-3647/06 R 2006 0622 2 42d spray interval
France, 2006 Fronton (Négrette) Red	2	0.25	0.125	200	-0 0 3 7 14 21 28	0.12 0.39 0.58 0.37 0.25 0.26 0.24	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.02 0.02 0.02 0.02 0.02	0.02 0.02 0.04 0.05 0.06 0.05 0.06	RA-2647/06 & RA-3647/06 R 2006 0623 0 23d spray interval
France, 2007 Graveson (Carignan) Red	2	0.25	0.125	200	-0 0 3 7 14 21	0.26 0.67 0.63 0.53 0.41 0.34	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.01 0.01 0.02 0.02	0.08 0.08 0.08 0.08 0.09 0.12	RA-2501/07 R 2007 0002 4 36d spray interval

Table 95 Residues in table grapes (bunches) from supervised trials in Greece, Italy, Portugal and Spain, involving 2 foliar applications of fluopyram (500 SC formulation)

TABLE GRAPE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Spain, 2006 Hondón de las Nieves (Italia) White	2	0.25	0.025	1000	-0 0 3 7 14 21 28	0.21 0.9 <u>0.55</u> 0.19 0.16 0.12 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.03 0.02 0.02 0.02 0.02	0.06 0.06 0.07 0.04 0.05 0.03 0.04	RA-2612/06 & RA-3612/06 R 2006 0417 3 31d spray interval
Portugal, 2006 Passinha-Alenquer (Cardinal) White	2	0.25	0.042	600	-0 0 3 7 14 21 28	0.06 0.35 0.32 0.22 <u>0.34</u> 0.28 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.02 0.02 0.02	RA-2612/06 & RA-3612/06 R 2006 0624 9 8d spray interval
Italy, 2006 Trinitapoli (Italia) White	2	0.25	0.025	1000	-0 0 3 7 14 21 28	0.22 0.43 <u>0.66</u> 0.56 0.43 0.33 0.41	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.02 0.02 0.02 0.02 0.02	0.02 0.01 0.02 0.02 0.03 0.03 0.04	RA-2612/06 & RA-3612/06 R 2006 0651 6 16d spray interval
Greece, 2006 Assos (Soultanina) White	2	0.25	0.025	1000	-0 0 3 7 14 21 28	0.13 0.51 <u>0.3</u> 0.18 0.27 0.22 0.22	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.01 0.01 0.01 0.01 0.01	0.01 0.01 0.01 0.01 0.02 0.01 0.02	RA-2612/06 & RA-3612/06 R 2006 0652 4 31d spray interval
Spain, 2007 Alhama de Murcia (Red globe) Red	2	0.25	0.025	1000	-0 0 3 7 15 22	0.4 0.96 0.81 <u>0.96</u> 0.68 0.61	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.02 0.02 0.02	RA-2502/07 R 2007 0003 2 21d spray interval
Portugal, 2007 Passinha-Casais Novos (Cardinal) White	2	0.25	0.032	800	-0 0 3 7 14 21	0.16 0.59 0.49 <u>0.6</u> 0.43 0.44	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 0.01 0.01 0.02	RA-2502/07 R 2007 0487 9 11d spray interval
Italy, 2007 Trinitapoli (Italia) White (berries)	2	0.25	0.025	1000	-0 0 3 7 14 21	0.3 0.97 0.62 <u>1.0</u> 0.58 0.63	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.02 0.02 0.02	0.02 0.02 0.02 0.03 0.03 0.05	RA-2502/07 R 2007 0488 7 14d spray interval

Results from supervised trials from USA on table grapes were provided to the Meeting. In these trials, unreplicated plots were treated with 2 foliar spray applications fluopyram (SC 500 formulation), 13–14 days apart, using ground-based airblast sprayers, applying 0.24–0.26 kg ai/ha in 438–627 litres water/ha. Plot sizes in these trials ranged from 53–109 square metres.

Duplicate samples of at least 1 kg grapes (berries) were taken from the inner four vines of each single-row plot, frozen within 3 hours of sampling and stored frozen for up to 401 days before

analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ was 0.01 mg/kg.

Table 96 Fluopyram residues in table grapes (berries) from supervised trials in USA, involving 2 foliar applications of fluopyram (500 SC formulation)

TABLE GRAPE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Dundee, New York (Vidal)	2	0.245 0.248	0.053 0.053	465 469	3 7	0.719, 0.662 0.41, 0.561				RAGMP043 GM106-06HA
USA, 2006 Orefield, Pennsylvania (Concord)	2	0.258 0.254	0.041 0.041	627 618	3 7	0.204, 0.116 0.136, 0.161				RAGMP043 GM107-06HA
USA, 2006 Fresno California (Thompson Seedless)	2	0.247 0.245	0.048 0.048	514 508	3 7	0.427, 0.417 0.34, 0.3				RAGMP043 GM109-06HA
USA, 2006 Sanger California (Thompson Seedless)	2	0.254 0.251	0.053 0.054	481 466	3 7	0.359, 0.272 0.238, 0.135				RAGMP043 GM110-06HA
USA, 2006 Plainview California (Crimson)	2	0.25 0.251	0.046 0.044	548 569	3 7	0.422, 0.363 0.386, 0.357				RAGMP043 GM111-06HA
USA, 2006 Fresno California (Thomson)	2	0.256 0.258	0.053 0.053	484 488	3 7	0.068, 0.085 0.101, 0.096				RAGMP043 GM112-06HA
USA, 2006 Madera California (Merlot)	2	0.248 0.252	0.044 0.045	568 564	3 7	0.224, 0.302 0.231, 0.303				RAGMP043 GM113-06HA
USA, 2006 Hickman California (Chardonnay)	2	0.25 0.243	0.046 0.04	540 615	3 7	0.802, 0.59 0.639, 0.621				RAGMP043 GM114-06HA
USA, 2006 Artois California (Centurion)	2	0.252 0.254	0.045 0.045	561 560	3 7	0.245, 0.252 0.197, 0.221				RAGMP043 GM115-06HA
USA, 2006 Hood River Oregon (Chardonnay)	2	0.251 0.252	0.057 0.049	438 516	3 7	0.136, 0.162 0.142, 0.149				RAGMP043 GM116-06HA
USA, 2006 Ephrata Washington (White Reisling)	2	0.25 0.252	0.05 0.05	498 502	3 7	0.67, 0.535 0.484, 0.463				RAGMP043 GM117-06HA
Canada, 2007 Rockwood, Ontario (Cabernet Franc)	2	0.248 0.254	0.044 0.043	558 584	4 7	0.449, 0.403 0.524, 0.328				RAGMP043 GM352-06HA
Canada, 2007 Rockwood, Ontario (Vidal)	2	0.251 0.251	0.045 0.045	560 560	4 7	0.741, 0.486 0.565, 0.471				RAGMP043 GM353-06HA
USA, 2007 Conklin, MI (Concord)	2	0.251 0.251	0.046 0.046	548 544	3 7	0.987, 0.821 0.946, 0.95				RAGMP043 GM354-06HA
USA, 2007 Carlyle, IL (Niagara)	2	0.252 0.252	0.045 0.041	560 616	3 7	0.47, 0.811 0.577, 0.572				RAGMP043 GM355-06HA
USA, 2006 Kerman, CA (Thompson Seedless)	2	0.249 0.25	0.051 0.051	487 494	0 3 7 10 14	0.879, 0.865 0.668, 0.777 0.566, 0.67 0.631, 0.862 0.542, 0.802				RAGMP043 GM108-06DA

Strawberries

Results from supervised trials from Europe on field and protected strawberries were provided to the Meeting. In these trials, 2 foliar spray applications of an SC 500 formulation of fluopyram were made at 6–7 day intervals using knapsack sprayers with single solid or hollow-cone nozzles or boom sprayers (3–12 flat-fan nozzles), applying 0.125 kg ai/ha in 300–2000 L water/ha. Plot sizes in these trials ranged from 4–70 square metres.

Unreplicated samples of at least 1 kg of fruit were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 271 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984–M001. The reported LOQs were 0.01 mg/kg for each analyte.

Table 97 Residues in protected strawberries from supervised trials in Belgium, France, Germany, Greece, Italy, Netherlands, Portugal, Spain and UK, involving 2 foliar applications of fluopyram (500 SC formulation)

STRAWBERRY Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Cremery (Darselect)	2	0.25	0.0835	300	-0 0 1 3 5 7	0.21 0.3 0.25 0.22 0.25 0.24	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/06 R 2006 0366 5 0366-06
Italy, 2006 Siracusa (Cantonga)	2	0.25	0.0415	600	-0 0 1 3 5 7	0.46 0.86 0.79 0.49 0.39 0.37	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.01 0.02 0.01 0.02 0.02	< 0.01 < 0.01 < 0.01 0.01 0.02 0.02	RA-2585/06 R 2006 0569 2 0569-06
Spain, 2006 Paterna Huelva (Ventana)	2	0.25	0.025	1000	-0 0 1 3 5 7	0.12 0.28 0.27 0.28 0.19 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/06 R 2006 0570 6 0570-06
Belgium, 2006 Ittre (Sonata)	2	0.25	0.028	900	-0 0 1 3 5 7	0.05 0.15 0.1 0.12 0.12 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/06 R 2006 0571 4 0571-06
Netherlands, 2006 Wognum (Elsanta)	2	0.25	0.025	1000	-0 0 1 3 5 7	0.07 0.21 0.15 0.2 0.2 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/06 R 2006 0572 2 0572-06
United Kingdom, 2006 Tunstead (Alsanta)	2	0.25	0.0625	400	-0 0 1 3 5 8	0.08 0.14 0.13 0.13 0.13 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/06 R 2006 0573 0 0573-06
Germany, 2006 Bornheim-Sechtem (Kimbalane)	2	0.25	0.0625	400	-0 0 1 4 6 8	0.19 0.39 0.33 0.2 0.14 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/06 R 2006 0574 9 0574-06

STRAWBERRY Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Germany, 2006 Westbevwern - Telgte (Elsanta)	2	0.25	0.025	1000	-0 0 1 3 5 7	0.19 0.76 0.71 0.55 0.63 0.49	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2585/06 R 2006 0575 7 0575-06

Table 98 Residues in field strawberries from supervised trials in Belgium, France, Germany, Greece, Italy, Netherlands, Portugal, Spain and UK, involving 2 foliar applications of fluopyram (500 SC)

STRAWBERRY Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Cremery (Darselect)	2	0.25	0.0835	300	-0 0 1 3 7	0.18 0.34 0.24 0.21 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2600/06 & RA-3600/06 R 2006 0384 3 0384-06
Germany, 2006 Muggensturm (Elsanta)	2	0.25	0.025	1000	-0 0 1 3 7	0.12 0.16 0.14 0.17 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2600/06 R 2006 0385 1 0385-06
Belgium, 2006 Ittre (Elsanta)	2	0.25	0.028	900	-0 0 1 3 7	0.16 0.38 0.36 0.3 0.21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2600/06 & RA-3600/06 R 2006 0403 3 0403-06
Netherlands, 2006 Spanbroek (Evi)	2	0.25	0.025	1000	-0 0 1 3 7	0.35 0.7 0.67 0.69 0.53	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2600/06 R 2006 0614 1 0614-06
United Kingdom, 2006 Tunstead (Alsanta)	2	0.25	0.0625	400	-0 0 1 3 7	0.1 0.19 0.13 0.15 0.14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2600/06 R 2006 0616 8 0616-06
France, 2007 Cremery (Eros)	2	0.25	0.0835	300	-0 0 1 3 7	0.15 0.4 0.35 0.28 0.23	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 < 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 0.01 0.02	RA-2503/07 R 2007 0004 0 0004-07
Germany, 2007 Bornheim (Elsanta)	2	0.25	0.0125	2000	-0 0 1 3 7	0.07 0.2 0.19 0.13 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2503/07 R 2007 0504 2 0504-07
United Kingdom, 2007 Tunstead (Alsanta)	2	0.25	0.0835	300	-0 0 1 3 7	0.05 0.16 0.12 0.17 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2503/07 R 2007 0505 0 0505-07
Netherlands, 2007 Zwaagdijk (Elsanta)	2	0.25	0.025	1000	-0 0 1 3 7	0.12 0.32 0.43 0.38 0.34	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2503/07 R 2007 0506 9 0506-07

STRAWBERRY Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Monteux (Pajaro)	2	0.25	0.025	1000	-0 0 1 3 7	0.13 0.28 0.27 0.2 0.22	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2601/06 & RA-3600/06 R 2006 0387 8 0387-06
Italy, 2006 San Martino (Alba)	2	0.25	0.0415	600	-0 0 1 3 7	0.05 0.14 0.09 0.12 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 < 0.01 < 0.01 < 0.01	RA-2601/06 R 2006 0388 6 0388-06
Spain, 2006 Paterna Huelva (Ventana)	2	0.25	0.025	1000	-0 0 1 3 7	0.13 0.24 0.25 0.23 0.17	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.02 0.02	RA-2601/06 & RA-3600/06 R 2006 0404 1 0404-06
Italy, 2006 Siracusa (Cantonga)	2	0.25	0.0415	600	-0 0 1 3 7	0.23 0.48 0.52 0.36 0.25	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.01 0.02 0.02 0.02	0.02 0.02 0.03 0.04 0.04	RA-2601/06 R 2006 0617 6 0617-06
Greece, 2006 Katerini/Pieria (Camarossa (USA / I))	2	0.25	0.0335	750	-0 0 1 3 7	0.07 0.22 0.19 0.13 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2601/06 R 2006 0618 4 0618-06
France, 2007 Monteux (Pajaro)	2	0.25	0.025	1000	-0 0 1 3 7	0.1 0.28 0.27 0.31 0.23	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2504/07 R 2007 0005 9 0005-07
Italy, 2007 San Bartolomeo in Bosco (Marmolada)	2	0.25	0.0315	800	-0 0 1 3 7	0.16 0.39 0.33 0.27 0.25	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2504/07 R 2007 0507 7 0507-07
Spain, 2007 St. Cebrià de Vallalta (Aromas)	2	0.25	0.025	1000	-0 0 1 3 7	0.13 0.32 0.34 0.32 0.27	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2504/07 R 2007 0508 5 0508-07
Portugal, 2007 Almeirim (Candonga)	2	0.25	0.0415	600	-0 0 1 3 7	0.12 0.28 0.2 0.27 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2504/07 R 2007 0509 3 0509-07

Results from supervised trials from USA on field strawberries were provided to the Meeting. In these trials, one of the plots was treated with 2 foliar spray applications of an SC 500 formulation of fluopyram were made at 5–6 day intervals using knapsack and hand lance or mini-boom sprayers, applying 0.24–0.26 kg ai/ha in 137–480 litres water/ha. In a separate plot, fluopyram (SC 500) was applied by drip irrigation, applying two treatments of 0.25 kg ai/ha in 1–2.2 litres water/hour/emitter, 5 days apart. Plot sizes in these trials ranged from 37–185 square metres.

Duplicate samples of at least 24 fruit were taken from at least 12 plants within each plot, frozen within 3 hours of sampling and stored frozen for up to 231 days before analysis for fluopyram using HPLC-MS/MS Method GM-001-P07-01. The reported LOQ was 0.01 mg/kg.

Table 99 Fluopyram residues in field strawberries from supervised trials in USA, involving two foliar applications of fluopyram (500 SC formulation)

STRAWBERRIES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 New Tripoli, PA (Earliglow)	2	0.246 0.249	0.14 0.14	176 178	0	0.183, 0.209				RAGMP084 GM072-07HA
USA, 2007 Enigma, GA (Chandler)	2	0.255 0.251	0.16 0.14	157 176	0	0.679, 0.712				RAGMP084 GM073-07HA
USA, 2007 Plant City, FL (Festival)	2	0.252 0.25	0.14 0.15	174 162	0	0.961, 1.062				RAGMP084 GM074-07HA
USA, 2007 Carroll, IA (Sea Scape)	2	0.247 0.244	0.15 0.14	168 172	0	0.207, 0.37				RAGMP084 GM075-07HA
USA, 2007 Fertile, MN (Mesabi)	2	0.244 0.25	0.18 0.14	138 178	0	0.241, 0.349				RAGMP084 GM076-07HA
USA, 2007 Ida, MI (Idea)	2	0.263 0.247	0.18 0.18	146 137	0	0.223, 0.26				RAGMP084 GM077-07HA
USA, 2007 Fresno, CA (Chandler)	2	0.243 0.25	0.05 0.05	470 480	0	0.869, 0.797				RAGMP084 GM079-07HA
USA, 2007 Sanger, CA (Camarosa)	2	0.253 0.255	0.17 0.15	149 172	0 3 7 10 14	0.41, 0.645 *0.44, 0.46, 0.43* 0.431, 0.579 0.534, 0.453 0.355, 0.371 0.293, 0.241				RAGMP084 GM078-07DA *0d PHI results incl processing study samples*
USA, 2007 Porterville, CA (Diamante)	2	0.25 0.251	0.14 0.13	177 189	0	0.743, 0.62				RAGMP084 GM080-07HA
USA, 2007 Corvallis, OR (Ozark Beauty)	2	0.26 0.259	0.15 0.15	173 173	0	0.343, 0.379				RAGMP084 GM081-07HA

Table 100 Fluopyram residues in field strawberries from supervised trials in USA, involving 2 drip irrigation treatments of fluopyram (500 SC formulation)

STRAWBERRIES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (l/hr)		parent	PAA	BZM	PCA	
USA, 2007 New Tripoli, PA (Earliglow)	2	0.25 0.25		1 1	0 6	< 0.01, < 0.01 < 0.01, < 0.01				RAGMP084 GM072-07HA
USA, 2007 Enigma, GA (Chandler)	2	0.25 0.25		2.1 2.1	0 7	0.047, 0.054 0.118, 0.09				RAGMP084 GM073-07HA
USA, 2007 Plant City, FL (Festival)	2	0.25 0.25		1.9 1.9	0 7	0.031, 0.029 0.057, 0.055				RAGMP084 GM074-07HA
USA, 2007 Carroll, IA (Sea Scape)	2	0.25 0.25		2.2 2.2	0 7	< 0.01, < 0.01 0.025, 0.02				RAGMP084 GM075-07HA

STRAWBERRIES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (l/hr)		parent	PAA	BZM	PCA	
USA, 2007 Fertile, MN (Mesabi)	2	0.248 0.248		1.9 1.8	0 7	< 0.01, < 0.01 0.015, 0.013				RAGMP084 GM076-07HA
USA, 2007 Ida, MI (Idea)	2	0.25		1	0 7	< 0.01, < 0.01 < 0.01, < 0.01				RAGMP084 GM077-07HA
USA, 2007 Sanger, CA (Camarosa)	2	0.25 0.25		1.4 1.4	0 3 7 10 14	< 0.01, < 0.01 0.013, 0.015 0.018, 0.02 *0.014 (2), 0.015* 0.025, 0.033 0.034, 0.026				RAGMP084 GM078-07DA *7d PHI results incl processing study samples*
USA, 2007 Fresno, CA (Chandler)	2	0.25 0.25		1.1 1.1	0 7	0.079, 0.112 0.219, 0.244				RAGMP084 GM079-07HA
USA, 2007 Porterville, CA (Diamante)	2	0.262 0.262		1.8 1.7	0 7	< 0.01, 0.013 0.032, 0.023				RAGMP084 GM080-07HA
USA, 2007 Corvallis, OR (Ozark Beauty)	2	0.251 0.251		1.5 1.5	0 7	0.039, < 0.01 < 0.01, < 0.01				RAGMP084 GM081-07HA

Blackberries, raspberries & boysenberries

Results from supervised trials from USA on blackberries, raspberries and boysenberries were provided to the Meeting. In these trials, 2 foliar spray applications of an SC 500 formulation of fluopyram (mixed with pyrimethanil - 600 SC formulation) were made at 7 day intervals using knapsack, boom or CO_2 pressurised backpack sprayers, applying 0.24–0.26 kg ai/ha in 337–386 litres water/ha. Plot sizes in these trials ranged from 28–102 square metres.

Duplicate samples of at least 0.5 kg (raspberries) or 1 kg (blackberries/boysenberries) were taken from at least 4 plants within each plot, frozen within 4 hours of sampling and stored frozen for up to 242 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01.

Table 101 Fluopyram residues in cane fruit (raspberries, boysenberries, blackberries) from supervised trials in USA, involving 2 foliar applications (500 SC formulation).

CANE BERRIES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Enigma, GA (Arapaho) Blackberry	2	0.251 0.252	0.067 0.07	377 360	0	0.837, 0.579				RAGMP079 GM037-07HA
USA, 2007 Hillsboro, OR (Katata) Blackberry	2	0.254 0.258	0.066 0.075	386 345	0 3 5 7 10	1.42, 1.41 1.29, 1.2 0.712, 0.709 0.569, 0.539 0.32, 0.314				RAGMP079 GM144-07DA
USA, 2007 Arkansaw, WI (Kilarney) Raspberry	2	0.242 0.257	0.067 0.067	363 385	0	2.247, 2.537				RAGMP079 GM038-07HA

CANE BERRIES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Jefferson, OR (Meeker) Raspberry	2	0.254 0.262	0.07 0.073	365 358	0	0.484, 0.369				RAGMP079 GM039-07HA
USA, 2007 Hillsboro, OR (not stated) Boysenberry	2	0.251 0.252	0.066 0.075	382 337	0	0.778, 0.885				RAGMP079 GM143-07HA

Blueberries

Results from supervised trials from USA on blueberries were provided to the Meeting. In these trials, 2 foliar spray applications of an SC 500 formulation of fluopyram (mixed with a pyrimethanil 600 SC formulation) were made at 7 day intervals using knapsack, airblast/mist blowers or CO_2 pressurised backpack sprayers, applying 0.23–0.26 kg ai/ha in 111–580 litres water/ha. Plot sizes in these trials ranged from 26–70 square metres.

Duplicate samples of at least 0.5 kg were taken from at least 4 plants within each plot, frozen within 5 hours of sampling and stored frozen for up to 266 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ was 0.01 mg/kg.

Table 102 Fluopyram residues in blueberries from supervised trials in USA, involving 2 foliar applications (500 SC formulation)

BLUEBERRIES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 New Tripoli, PA (Bluecrop)	2	0.243 0.248	0.053 0.053	462 472	0	1.171, 1.109				RAGMP037 GM001-07HA
USA, 2007 Chula, GA (Brightwell)	2	0.251 0.249	0.066 0.066	382 378	0	1.558, 1.075				RAGMP037 GM003-07HA
USA, 2007 Elizabethtown, NC (Reka)	2	0.248 0.253	0.17 0.16	146 158	0	0.554, 0.611				RAGMP037 GM004-07HA
USA, 2007 Hixton, WI (Patriot)	2	0.25 0.247	0.067 0.067	374 371	0	1.278, 1.694				RAGMP037 GM005-07HA
USA, 2007 Fennville, MI (Jersey)	2	0.25 0.25	0.159 0.155	157 161	0	0.89, 1.394				RAGMP037 GM006-07HA
USA, 2007 Covert, MI (Jersey)	2	0.234 0.261	0.047 0.045	495 580	0	4.932, 3.725				RAGMP037 GM007-07HA
USA, 2007 Hillsboro, OR (Bluecrop)	2	0.245 0.251	0.206 0.226	119 111	0	0.858, 0.892				RAGMP037 GM008-07HA

BLUEBERRIES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Ochlocknee, GA (Tifblue)	2	0.251 0.254	0.054 0.05	466 508	0 1 3 7 10	0.504, 0.509 *0.53, 0.51, 0.46* 1.163, 1.292 0.242, 0.247 0.243, 0.262 0.086, 0.114				RAGMP037 GM002-07DA 0*d PHI results incl processing study samples*

Banana

Results from supervised trials from Latin America on banana were provided to the Meeting. In these trials, 6 foliar spray applications of an SC 500 formulation of fluopyram mixed with adjuvants and crop/mineral oils were made at 5–11 day intervals using knapsack "Solo" mistblowers or helicopter-mounted airblast sprayers, applying 0.09-0.107 kg ai/ha in 21–63 litres water/ha. Plot sizes in these trials ranged from 1665–6750 square metres.

Unreplicated samples of bagged and unbagged fruit were taken from each plot, shipped fresh (chilled) and stored frozen (within 7 days of harvest), for up to 335 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984. The reported LOQs were 0.01 mg/kg for each analyte.

Table 102 Fluopyram residues in bananas from supervised trials in Colombia, Costa Rica, Ecuador, Guatamala, Honduras,and Mexico, involving 6 foliar applications (500 SC formulation)

BANANA Country, year Location (variety)	Application			PHI, (days)	Commodity	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	water (L/ha)			parent	PAA	BZM	PCA	
Costa Rica, 2007 Los Angeles (Grand Naine)	6	0.097- 0.101	22.5- 21.7	0	Bagged Unbagged	< 0.01, < 0.01 0.019, 0.018	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM149-07HA
Costa Rica, 2007 San Bosco (Grand Naine)	6	0.099- 0.105	39.9- 45	0	Bagged Unbagged	0.037, 0.04 0.258, 0.154	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM150-07HA
Costa Rica, 2007 Cartagena (Grand Naine)	6	0.099- 0.102	39.8- 45.9	0	Bagged Unbagged	0.021, 0.022 0.277, 0.215	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM151-07HA
Ecuador, 2007 El Guabo (Williams)	6	0.099- 0.102	28.5- 29.8	0	Bagged Unbagged	0.028, 0.02 0.368, 0.309	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM152-07HA
Ecuador, 2007 Machala (Williams)	6	0.099- 0.1	57.3- 62.9	0	Bagged Unbagged	0.015, < 0.01 0.194, 0.17	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM153-07HA
Ecuador, 2007 Naranjal (Williams)	6	0.099- 0.101	27.5- 29.5	0	Bagged Unbagged	0.017, 0.028 0.526, 0.494	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM154-07HA
Ecuador, 2007 Machala (Williams)	6	0.1- 0.102	55.6- 62.8	0	Bagged Unbagged	< 0.01, < 0.01 0.251, 0.196	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM155-07HA
Guatemala, 2007 Tiquisate (Grand Nain)	6	0.093- 0.107	22.7- 29.3	0	Bagged Unbagged	0.012, < 0.01 0.058, 0.05	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM156-07HA
Guatemala, 2007 Tiquisate (Gran Nain)	6	0.096- 0.107	23.6- 34.9	0	Bagged Unbagged	0.014, 0.011 0.043, 0.043	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM157-07HA

BANANA Country, year Location (variety)	Application			PHI, (days)	Commodity	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	water (L/ha)			parent	PAA	BZM	PCA	
Guatemala, 2007 San Manuel (Williams)	6	0.1- 0.109	21.2- 30.2	0 	Bagged Unbagged	0.016, 0.013 0.072, 0.052	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM158-07HA
Mexico, 2007 Tuxtepec (Gran Enano)	6	0.093- 0.102	42.9- 56.4	0 0 2 5 6	Bagged Unbagged Unbagged Unbagged Unbagged	0.021, 0.021 0.134, 0.198 0.15, 0.184 0.211, 0.144 0.134, 0.123	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	RAGPM075 GM159-07DA
Colombia, 2007 Choridó (Williams)	6	0.094- 0.1	28.7- 29.9	0 	Bagged Unbagged	< 0.01, 0.011 0.05, 0.049	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM160-07HA
Colombia, 2007 Turbo (Gran Enano)	6	0.098- 0.101	29.7- 30.2	0 	Bagged Unbagged	< 0.01, < 0.01 0.074, 0.257	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2)	RAGPM075 GM161-07HA
Costa Rica, 2007 El Humo (Grand Naine)	6	0.093- 0.106	21.7- 25.6	0 0 3 5 7	Bagged Unbagged Unbagged Unbagged Unbagged	0.033, 0.028 0.044, 0.027 0.034, 0.026 0.029, 0.028 0.01, < 0.01	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	RAGPM075 GM148-07DA

Bulb vegetables

Leek

Results from supervised trials from Europe on leeks were provided to the Meeting. In these trials, 2 applications of fluopyram + tebuconazole (SC 200+200) were applied at 13–14 day intervals as foliar sprays using knapsack or wheelbarrow sprayers with mini-booms (3–12 flat fan or hollow cone nozzles), applying fluopyram at 0.2kg ai/ha in 300–600 litres water/ha. Plot sizes in these trials ranged from 20–58 square metres.

Unreplicated samples of 12 leek plants (without roots) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 364 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQs were 0.01 mg/kg for each analyte).

Table 104 Residues in leeks from supervised field trials in France, Germany, Italy, Netherlands, Spain and United Kingdom, involving two foliar applications of fluopyram (SC formulation)

LEEK Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Faverolles (Diana)	2	0.2	0.0666	300	-0 0 7 14 21 28	0.18 2.1 1.2 0.67 0.32 0.2	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2569/06 0343-06
Germany, 2006 Langenfeld-Reusrath (Pandora)	2	0.2	0.0666	300	-0 0 7 14 21 28	0.06 0.68 0.08 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2569/06 0465-06

LEEK Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
United Kingdom, 2006 Chatteris (Shelton)	2	0.2	0.0666	300	-0 0 7 15 21 28	0.06 1.1 0.55 0.12 0.06 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2569/06 0466-06
Germany, 2006 Bornheim - Sechtem (Amundo)	2	0.2	0.0666	300	-0 0 7 15 22 28	0.27 0.97 0.67 0.23 0.17 0.14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2569/06 0468-06
Germany, 2007 Schauernheim (Nobel)	2	0.2	0.0334	600	-0 0 7 14 21 28	0.1 0.41 0.08 0.04 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2521/07 0056-07
France, 2007 Bouafle (St Vistor)	2	0.2	0.04	500	-0 0 7 14 21 28	0.05 1.6 0.22 0.15 0.09 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2521/07 0569-07
Netherlands, 2007 Zwaagdijk-Oost (Roxton)	2	0.2	0.0334	600	-0 0 7 14 21 28	0.07 0.48 0.08 0.05 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2521/07 0570-07
Germany, 2007 Langenfeld-Reusrath (Pandora)	2	0.2	0.0666	300	-0 0 7 14 21 28	0.25 1.7 0.26 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2521/07 0571-07
France, 2006 Cailloux sur Fontaines (Ahton)	2	0.2	0.0334	600	-0 0 7 14 21 29	0.12 1.1 0.37 0.28 0.17 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.02 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2570/06 0344-06
Spain, 2006 Lebrija (Shelton)	2	0.2	0.05	400	-0 0 7 14 20 28	0.02 0.32 0.18 0.07 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2570/06 0469-06
France, 2007 Castelsarrasin (Porbella)	2	0.2	0.0334	600	-0 0 7 14 21 28	0.35 2.8 0.39 0.31 0.24 0.25	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2522/07 0057-07

LEEK Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Italy, 2007 Lusia (Sabina)	2	0.2	0.0334	600	-0 0 7 14 21 28	0.06 2.1 0.15 0.16 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2522/07 0572-07

Onion, Bulb

Results from supervised trials from Europe on bulb onions were provided to the Meeting. In these trials, 2 applications of fluopyram + tebuconazole (SC 200 + 200) were applied at 6–8 day intervals as foliar sprays using knapsack or wheelbarrow sprayers with either a hand lance (single solid cone nozzle) or mini-booms (3–9 flat fan or hollow cone nozzles), applying fluopyram at 0.2kg ai/ha in 300–800 litres water/ha. Plot sizes in these trials ranged from 15–90 square metres.

Unreplicated samples of 12–30 onion bulbs were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 349 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQs were 0.01 mg/kg for each analyte).

Table105 Residues in onion bulbs from supervised field trials in France, Germany, Greece, Italy, Netherlands, Portugal, Spain and United Kingdom, involving two foliar applications of fluopyram (SC formulation)

ONION, BULB Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Bouafle (Jaunes des Cévennes)	2	0.2	0.04	500	-0 0 7 14 21	0.04 0.05 0.02 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2567/06 0340-06
Germany, 2006 Langenfeld-Reusrath (Stuttgarter Riesen)	2	0.2	0.0666	300	-0 0 7 14 21	< 0.01 0.08 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2567/06 0537-06
Germany, 2006 Kohlhof (Takstar)	2	0.2	0.04	500	-0 0 7 14 21	0.02 0.06 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2567/06 0538-06
United Kingdom, 2006 Thetford (Red Barron)	2	0.2	0.0666	300	-0 0 7 14 20	0.03 0.11 0.04 0.02 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2567/06 0539-06
Germany, 2007 Langenfeld-Reusrath (Stuttgarter Riesen)	2	0.2	0.0666	300	-0 0 7 14 21	< 0.01 0.07 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2517/07 0038-07
Germany, 2007 Vettweiß-Müddersheim (Benito)	2	0.2	0.0666	300	-0 0 7 14 20	< 0.01 0.08 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2517/07 0558-07

ONION, BULB Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2007 Luzillé (Hybing F1)	2	0.2	0.0334	600	-0 0 7 14 21	< 0.01 0.05 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2517/07 0560-07
Netherlands, 2007 Wieringerwerf (Hybell)	2	0.2	0.025	800	-0 0 7 14 21	< 0.01 0.03 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2517/07 0561-07
France, 2006 Cailloux sur Fontaines (Rebouillon)	2	0.2	0.0334	600	-0 0 7 14 21	< 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2568/06 0342-06
Spain, 2006 Gava (Dulce de Fuentes)	2	0.2120-0.2	0.0334	636-	-0 0 7 14 21	< 0.01 0.07 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2568/06 0540-06
Italy, 2006 Bologna (Density 5)	2	0.2	0.04	500	-0 0 7 14 21	0.01 0.04 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2568/06 0541-06
Greece, 2006 Thiva (Ideal)	2	0.2	0.0334	600	-0 0 7 15 21	< 0.01 0.04 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2568/06 0542-06
France, 2007 Layrac (Daytona)	2	0.2	0.0334	600	-0 0 7 14 21	0.01 0.1 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2518/07 0040-07
Italy, 2007 Poggio Renatico (Rossa di Toscana)	2	0.2	0.0334	600	-0 0 7 14 21	< 0.01 0.03 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2518/07 0563-07
Spain, 2007 Alginet (Liria)	2	0.2	0.0334	600	-0 0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2518/07 0564-07
Portugal, 2007 S. Francisco/Alcochete (Spring Star)	2	0.2	0.04	500	-0 0 7 14 21	< 0.01 0.08 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2518/07 0565-07
Greece, 2007 Aronas-Katerini (Kozanis)	2	0.2	0.04	500	-0 0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2518/07 0566-07

Results from supervised trials from USA on bulb onions were provided to the Meeting. In these trials, two applications of 0.24–0.25 kg ai/ha (SC 500 formulation) in 122–179 litres water were

applied as foliar sprays, 5 days apart, using knapsack, CO_2 backpack or plot boom sprayers. Plot sizes in these trials ranged from 42–118 square metres.

Duplicate samples (minimum 2 kg onion bulbs) were taken from each plot, frozen within 1 hour of sampling, held in frozen storage for up to 267 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 106 Fluopyram residues in onion bulbs from supervised outdoor trials in USA involving two foliar applications of fluopyram (500 SC formulations)

ONION, BULB Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Medium Red)	2	0.251 0.251	0.14 0.14	179 179	0	0.15, 0.124				RAGML001 GM043-07HA
USA, 2007 Uvalde, TX (Leona)	2	0.248 0.249	0.198 0.147	125 169	0	0.151, 0.191				RAGML001 GM044-07HA
USA, 2007 Larned, KS (Texas Supersweet)	2	0.241 0.241	0.145 0.148	166 163	0	0.15, 0.221				RAGML001 GM045-07HA
USA, 2007 Fresno, CA (Gunnison F1)	2	0.248 0.252	0.18 0.18	138 140	0	0.085, 0.113				RAGML001 GM047-07HA
USA, 2007 Parkdale, OR (Yellow Danver)	2	0.251 0.246	0.206 0.198	122 124	0	0.077, 0.043				RAGML001 GM048-07HA
USA, 2007 Richland, IA (Yellow No. 1)	2	0.248 0.246	0.16 0.156	155 158	0	0.019, 0.012				RAGML001 GM109-07HA
USA, 2007 Hillsboro, OR (Redwing)	2	0.239 0.25	0.145 0.145	165 172	0	0.118, 0.177				RAGML001 GM110-07HA
USA, 2007 Sanger, CA (Yellow sweet spanish)	2	0.251 0.247	0.155 0.147	162 168	0 1 3 7 10	0.095, 0.05 0.03, 0.023 0.039, 0.026 0.012, 0.027 0.013, 0.023				RAGML001 GM046-07DA

Onion, Welsh

Results from supervised trials from Europe on Welsh onions were provided to the Meeting. In these trials, 2 applications of fluopyram + tebuconazole (SC 200+200) were applied at 6–7 day intervals as foliar sprays using knapsack sprayers with hand-held booms (3–9 flat fan nozzles), applying fluopyram at 0.2kg ai/ha in 300-600 litres water/ha. Plot sizes in these trials ranged from 27–72 square metres.

Unreplicated 1-2 kg samples (24–84 whole plants, without roots) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 356 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQs were 0.01 mg/kg for each analyte).

Table 107 Residues in Welsh onions from supervised field trials in France, Germany, Italy and United Kingdom, involving two foliar applications of fluopram (SC formulation)

ONION, WELSH Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Tilloloy (Barletta)	2	0.2 0.188	0.04	500 470	-0 0 7 14 21	0.33 2.6 0.7 0.41 0.29	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2565/06 0337-06
Germany, 2006 Burscheid (Feast)	2	0.2	0.0666	300	-0 0 7 14 21	0.08 0.99 0.39 0.1 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.01 0.01 0.09	< 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-2565/06 0504-06
Germany, 2007 Burscheid (Vaugirard)	2	0.2	0.0666	300	-0 0 7 14 21	0.08 0.86 0.11 0.12 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2519/07 0042-07
United Kingdom, 2007 Southfleet/Gravesend (Laser)	2	0.2	0.0666	300	-0 0 7 13 21	0.96 3.3 0.61 0.23 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.03 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2519/07 0567-07
France, 2006 Saint Bonnet de Mure (Barletta)	2	0.2	0.05	400	-0 0 7 14 21	0.23 3.8 1.2 0.47 0.27	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.03 0.02 0.05 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2566/06 0339-06
Italy, 2006 Lusia (Bianco di Lisbona)	2	0.2	0.0334	600	-0 0 7 14 21	0.68 2.1 0.41 0.23 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2566/06 0505-06
France, 2007 Toulouse (Elodie,welsh onion/green onion)	2	0.2	0.0334	600	-0 0 7 14 21	0.49 2.1 0.27 0.24 0.17	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.05 0.07 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2520/07 0043-07
Italy, 2007 Lusia (Bianco di Lisbona)	2	0.2	0.0334	600	-0 0 7 14 21	0.59 2.2 0.61 0.29 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.02 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2520/07 0568-07

Spring onion

Results from supervised trials from USA on spring (green or bunching) onions were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) in 138–187 litres of water were applied as foliar sprays, 5 days apart, using a CO_2 pressurised plot sprayer, a backpack sprayer or a tractor-mounted boom sprayer. Plot sizes in these trials ranged from 28–62 square metres.

Duplicate samples (24 whole plants without roots, at least 2.5 kg) were taken from each plot, frozen within 3.7 hours of sampling, held in frozen storage for up to 205 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 108 Fluopyram residues in spring (green or bunching) onions from supervised outdoor trials in USA involving two foliar applications of fluopyram (500 SC formulations)

ONION, GREEN Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Salinas, CA (Emerald Isle)	2	0.245 0.254	0.178 0.17	138 149	0	1.173, 1.176				RAGMP081 GM040-07HA
USA, 2007 Chico, CA (Southport White Bunching)	2	0.247 0.248	0.132 0.133	187 187	0	4.527, 5.669				RAGMP081 GM041-07HA
USA, 2008 Raymondville, TX (Yellow Granex)	2	0.258 0.258	0.14 0.141	184 183	0	5.07, 7.294				RAGMP081 GM042-07HA

Brassica vegetables

Cabbages, Head

Results from supervised trials from USA on head cabbages were provided to the Meeting. In these trials, two foliar applications of 0.25–0.26 kg ai/ha fluopyram (SC 500 formulation), tank-mixed with trifloxystrobin (SC) in 92–188 litres of water were applied to cabbage plants 5 days apart using CO_2 pressurised plot sprayers or knapsack sprayers. Plot sizes in these trials ranged from 46–181 square metres.

Duplicate samples of at least 12 cabbage heads (with wrapper leaves) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 415 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg). Analytical results from cabbage heads without wrapper leaves are presented in Table 192 (Magnitude of the residue in processing).

Table 109 Fluopyram residues in cabbage heads (with wrapper leaves) from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

CABBAGE Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Blue Lagoon)	2	0.257 0.259	0.141 0.14	182 185	0 1 3 7 10	0.708, 0.626 0.867, 0.336 0.144, 0.110 0.053, 0.056 0.095, 0.139				RAGMP076 GM023-07DA
USA, 2007 Athens, GA (Bonnies Best Hybrid)	2	0.252 0.249	0.257 0.271	98 92	0	0.98, 1.054				RAGMP076 GM024-07HA
USA, 2007 Belle Glade, FL (Emblem)	2	0.253 0.253	0.143 0.135	177 188	0	1.6, 0.929				RAGMP076 GM025-07HA
USA, 2007 Richland, IA (Stonehead)	2	0.254 0.248	0.164 0.16	155 155	0	1.253, 0.751				RAGMP076 GM026-07HA
USA, 2007 Uvalde, TX (Blue Thunder)	2	0.251 0.25	0.136 0.139	184 180	0	0.254, 0.119				RAGMP076 GM027-07HA

CABBAGE Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Corning, CA (Copenhagen)	2	0.251 0.25	0.134 0.134	187 187	0	0.054, 0.064				RAGMP076 GM028-07HA

Results from supervised trials from Europe on head cabbages (round cabbage, red cabbage) were provided to the Meeting. In these trials, 2 applications of fluopyram + tebuconazole (SC 200+200) at rates 0.2kg ai/ha fluopyram in 300–600 litres water/ha of were applied at 14–15 day intervals as foliar sprays using knapsack sprayers with hand-held booms (3–12 flat fan or hollow cone nozzles). Plot sizes in these trials ranged from 30–120 square metres.

Unreplicated samples of 12 cabbage heads were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 214 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 or Method 00984/M001 (LOQs were 0.01 mg/kg for each analyte).

Table 110 Residues in cabbage heads from supervised trials in Belgium, France, Germany, Italy, Netherlands and UK involving two foliar applications of fluopyram (SC formulations)

CABBAGE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Fondettes (Eton F1) Cabbage, white	2	0.2	0.0334	600	-0 0 3 7 14 21	< 0.01 0.05 0.06 0.02 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2577/06 & RA-3577/06 0354-06
Netherlands, 2006 Noorder-koggenland (Slaudena) Cabbage, white	2	0.2	0.04	500	-0 0 3 7 14 21	0.03 < 0.01 0.02 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2577/06 0356-06
Germany, 2006 Werl-Westönnen (Novator) Cabbage, white	2	0.2	0.04	500	-0 0 3 7 14 21	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2577/06 0477-06
Germany, 2006 Leichlingen (Latima) Cabbage, white	2	0.2	0.0666	300	-0 0 3 7 14 21	0.01 0.22 0.09 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2577/06 & RA-3577/06 0478-06
Germany, 2007 Werl-Westönnen (Lennox) Cabbage, white	2	0.2	0.0666	300	-0 0 4 8 15 22	< 0.01 0.45 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2529/07 0073-07

CABBAGE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2007 Cergy (Lennox) Cabbage, white	2	0.2	0.0666	300	-0 0 3 7 14 21	0.05 0.34 0.42 0.21 0.08 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2529/07 0270-07
Germany, 2007 Leichlingen (Rodima) Cabbage, red	2	0.2	0.0666	300	-0 0 3 7 14 21	0.08 0.42 0.05 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2529/07 0589-07
United Kingdom, 2007 Saxmundham (Auralias) Cabbage, red	2	0.2	0.0666	300	-0 0 3 7 13 20	0.02 0.14 0.17 0.04 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2529/07 0590-07
Belgium, 2007 Villers-Perwin (Regina F1) Cabbage, red	2	0.2	0.0364	550	-0 0 3 7 14 21	< 0.01 0.09 0.02 0.02 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2529/07 0591-07
France, 2006 Arnas (Count) Cabbage, white	2	0.2	0.04	500	-0 0 4 7 14 21	0.02 0.19 0.04 0.04 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2578/06 & RA-3578/06 0357-06
Italy, 2006 Ladispoli (Castello) Cabbage, white	2	0.2	0.0334	600	-0 0 3 7 14 21	0.01 0.1 0.11 0.06 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.01 0.02 0.02 0.02 0.03	RA-2578/06 & RA-3578/06 0479-06
Italy, 2007 Ladispoli (Castello) Cabbage, white	2	0.2	0.0334	600	-0 0 3 7 14 21	0.01 0.12 0.11 0.03 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.01 < 0.01 0.01	0.01 0.01 0.02 0.03 0.02 0.03	RA-2530/07 0592-07

Broccoli

Results from supervised trials from USA on broccoli were provided to the Meeting. In these trials, two foliar applications of 0.24–0.26 kg ai/ha fluopyram (SC 500 formulation), tank-mixed with trifloxystrobin (SC) in 122–186 litres of water were applied to broccoli plants 5 days apart using knapsack sprayers or tractor-mounted boom sprayers. Plot sizes in these trials ranged from 53–159 square metres.

Duplicate samples of at least 12 broccoli heads and stems) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 448 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg). Analytical results from washed and/or cooked broccoli are presented in Table 194 (Magnitude of the residue in processing).

Table 111 Fluopyram residues in broccoli from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

BROCCOLI Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2008 Raymondville, TX (Green Magic)	2	0.262 0.254	0.141 0.141	186 180	0	0.882, 1.312				RAGMP076 GM017-07HA
USA, 2007 Guadalupe, CA (Heritage)	2	0.238 0.254	0.195 0.185	122 137	0	1.169, 1.189				RAGMP076 GM018-07HA
USA, 2007 Sanger, CA (Marathon)	2	0.25 0.257	0.184 0.158	136 163	0 1 3 7 10	0.981, 1.134 *0.994, 0.991, 0.951* 0.854, 1.084 0.831, 0.828 0.758, 0.523 0.701, 0.627				RAGMP076 GM019-07DA *Results from processing study*

Results from supervised trials from Europe on broccoli were provided to the Meeting. In these trials, 2 applications of fluopyram + tebuconazole (SC 200+200) at rates 0.2kg ai/ha fluopyram in 300–800 litres water/ha of were applied at 14–21 day intervals as foliar sprays using knapsack sprayers with hand-held booms (4–12 flat fan or hollow cone nozzles). Plot sizes in these trials ranged from 50–100 square metres.

Unreplicated samples of 12–20 broccoli heads were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 335 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 or Method 00984/M001 (LOQs were 0.01 mg/kg for each analyte).

Table 112. Residues in broccoli from supervised trials in France, Germany, Greece, Italy, Netherlands and Spain involving foliar applications of fluopyram (SC formulations)

BROCCOLI Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Ennemain (Chevalier)	2	0.2	0.0666	300	-0 0 3 7 14 21	< 0.01 0.74 0.41 0.19 0.09 0.14	< 0.01 < 0.01 0.01 < 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	RA-2607/06 0400-06
Netherlands, 2006 Zwaagdijk-Oos (Ironman)	2	0.2	0.0666	300	-0 0 3 7 14 21	< 0.01 0.83 0.61 0.12 0.05 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2607/06 0501-06
France, 2007 Ennemain (Chevalier)	2	0.2	0.0666	300	-0 0 3 7 14 21	< 0.01 0.64 0.05 0.02 0.01 0.02	< 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.01 < 0.01 < 0.01	RA-2525/07 0069-07

BROCCOLI Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Germany, 2007 Brühl-Schwardorf (Patinon)	2	0.2	0.0334	600	-0 0 3 8 14 22	< 0.01 0.04 < 0.01 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2525/07 0586-07
Spain, 2006 Lebrija (Partenon)	2	0.2	0.05	400	-0 0 3 7 14	< 0.01 0.48 0.04 0.03 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2608/06 0401-06
Italy, 2006 Manfredonia (Olimpia)	2	0.2	0.025	800	-0 0 3 7 14 21	0.13 0.82 0.51 0.44 0.13 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 (c) 0.03 0.03 0.04 0.03 (c) 0.04	RA-2608/06 0502-06 c=0.02 mg/kg
Greece ,2007 Aronas (Marathon)	2	0.2	0.04	500	-0 0 3 7 14 21	0.04 0.29 0.26 0.19 0.05 0.05	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	RA-2526/07 0070-07
Spain, 2007 Gavà (Megaton)	2	0.2	0.0334	600	-0 0 4 7 14 20	0.03 0.61 0.36 0.15 0.06 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2526/07 0587-07
Italy, 2007 Manfredonia (Partenon F1)	2	0.2	0.0266	750	-0 0 3 7 14 21	0.03 0.39 0.19 0.06 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	RA-2526/07 0588-07

Cauliflower

Results from supervised trials from USA on cauliflowers were provided to the Meeting. In these trials, two foliar applications of 0.25–0.27 kg ai/ha fluopyram (SC 500 formulation), tank-mixed with trifloxystrobin (SC) in 136–170 litres of water were applied to cauliflower plants 5–6 days apart using knapsack sprayers or tractor-mounted boom sprayers. Plot sizes in these trials ranged from 53–159 square metres.

Duplicate samples of at least 12 cauliflower heads and stalks) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 439 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 113 Fluopyram residues in cauliflowers from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

CAULIFLOWER Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 King City, CA (Solvang)	2	0.249 0.25	0.178 0.177	140 141	0	0.023, 0.016				RAGMP076 GM020-07HA
USA, 2007 Sanger, CA (Shasta)	2	0.25 0.268	0.184 0.158	136 170	0	0.979, 0.69				RAGMP076 GM021-07HA
USA, 2007 Corvallis, OR (Minuteman)	2	0.27 0.256	0.182 0.182	148 141	0	0.035, 0.023				RAGMP076 GM022-07HA

Results from supervised trials from Europe on cauliflower were provided to the Meeting. In these trials, 2 applications of fluopyram + tebuconazole (SC 200+200) at rates 0.2 kg ai/ha fluopyram in 300–800 litres water/ha of were applied at 13–14 day intervals as foliar sprays using knapsack sprayers with a single solid cone nozzle hand lance or hand-held booms (3–12 flat fan or hollow cone nozzles). Plot sizes in these trials ranged from 66–162 square metres.

Unreplicated samples of 12 cauliflower curds were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 334 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 or Method 00984/M001 (LOQs were 0.01 mg/kg for each analyte).

Table 114 Residues in cauliflowers from supervised trials in France, Germany, Italy, Netherlands, Spain and UK involving two foliar applications of fluopyram (SC formulations)

CAULIFLOWER Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
United Kingdom, 2006 West Row (Optamist)	2	0.212 0.2	0.0666	318 300	-0 0 3 7 14 21	0.01 0.07 0.07 0.04 0.05 0.02	< 0.01 < 0.01 < 0.01 0.01 0.02 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2571/06 0345-06
Germany, 2006 Langenfeld-Reusrath (Freedom)	2	0.2	0.0666	300	-0 0 3 7 14 21	< 0.01 0.85 0.18 0.08 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2571/06 0471-06
Germany, 2007 Langenfeld-Reusrath (Freedom)	2	0.2	0.0334	600	-0 [a] 0 [a] 3 7 14 21	0.15 2.4 0.04 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01	RA-2525/07 0067-07
Netherlands, 2007 Zwaagdijk-Oost (Speedy)	2	0.2	0.0666	300	-0 0 3 7 14 21	< 0.01 0.36 0.05 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2525/07 0583-07

CAULIFLOWER Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Graveson (Thalassa)	2	0.2	0.0334	600	-0 0 3 7 13 21	< 0.01 0.14 0.03 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2572/06 0346-06
Italy, 2006 Catania (Violetto Catanese)	2	0.2	0.025	800	-0 0 3 7 14 20	0.13 0.89 0.48 0.19 0.05 0.01	0.01 < 0.01 0.02 0.02 0.04 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.02 0.02 0.03 0.03	RA-2572/06 0472-06
France, 2007 Graveson (Thalassa)	2	0.2	0.0334	600	-0 0 3 7 14 21	< 0.01 0.08 0.06 0.02 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2526/07 0068-07
Spain, 2007 Alginet (Movidick)	2	0.2	0.04 0.025	500 800	-0 [a] 0 3 7 14 21	0.4 1.6 1.6 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.02 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2526/07 0584-07
Italy, 2007 Ladispoli (Meridien F1)	2	0.2	0.0334	600	-0 0 3 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.03 0.04 0.03 0.05	RA-2526/07 0585-07

[a] = Analysis of the whole plant, without roots

Brussels sprouts

Results from supervised trials from Europe on Brussels sprouts were provided to the Meeting. In these trials, 2 applications of fluopyram + tebuconazole (SC 200+200) at rates 0.2kg ai/ha fluopyram in 300–800 litres water/ha of were applied at 14–15 day intervals as foliar sprays using wheelbarrow or knapsack sprayers and spraying booms (3–12 flat fan or hollow cone nozzles). Plot sizes in these trials ranged from 30–200 square metres.

Unreplicated samples of sprouts (1–2.8 kg) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 253 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQs were 0.01 mg/kg for each analyte).

Table 115 Fluopyram residues in Brussels sprouts from supervised trials in France, Germany, Italy, Netherlands, Spain and UK involving foliar applications (400 SC formulation)

BRUSSELS SPROUTS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Fontaine l'Etalon (Louis)	2	0.2	0.0666	300	-0 0 7 14 21	< 0.01 0.02 0.07 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2575/06 0349-06

BRUSSELS SPROUTS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Germany, 2006 Langenfeld-Reusrath (Genius)	2	0.2	0.0666	300	-0 0 7 14 21	0.04 0.09 0.1 0.07 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2575/06 0473-06
United Kingdom, 2006 Saxmundham (Helemus)	2	0.2 0.212	0.0666	300 318	-0 0 7 14 21	0.05 0.16 0.13 0.07 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2575/06 0474-06
Germany, 2006 Meckenbeuren (Lunet)	2	0.2	0.04	500	-0 0 7 14 22	< 0.01 0.04 0.03 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2575/06 0475-06
Germany, 2007 Bornheim-Sechtem (Maximus)	2	0.212 0.2	0.05	424 400	-0 0 6 14 21	0.05 0.08 0.09 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2531/07 0076-07
France, 2007 Criquebeuf sur Seine (Abaccus)	2	0.2	0.04	500	-0 0 7 13 21	0.06 0.2 0.25 0.14 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.02 0.01	RA-2531/07 0594-07
Netherlands, 2007 Zwaagdijk-Oost (Cerius)	2	0.2	0.0334	600	-0 0 7 14 21	0.03 0.19 0.1 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2531/07 0595-07
United Kingdom, 2007 Saxmundham (Maximus)	2	0.2	0.0666	300	-0 0 7 15 21	0.02 0.26 0.06 0.03 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2531/07 0596-07
France, 2006 Castelsarrasin (Oliver)	2	0.2	0.0666	300	-0 0 7 14 21	0.05 0.34 0.12 0.05 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2576/06 0350-06
Italy, 2006 Ladispoli (Franklin)	2	0.2	0.025	800	-0 0 7 14 20	0.06 0.2 0.09 0.09 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2576/06 0476-06
France, 2007 Rillieux la Pape (Olivier)	2	0.2	0.04	500	-0 0 7 14 21	0.08 0.1 0.06 0.05 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2532/07 0077-07
Italy, 2008 Manfredonia (Lunet)	2	0.2	0.0286	700	-0 0 7 14 22	0.16 0.41 0.2 0.15 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.01 0.01	RA-2532/07 0597-07

Fruiting vegetables, Cucurbits

Cucumber

A summary report of 24 supervised trials from China on cucumbers were provided to the Meeting. In these trials, 3–4 applications of 0.075 kg ai/ha and 0.112 kg ai/ha (SC 500 formulation) were applied to cucumber plants by foliar applications, at 7-day intervals.

Samples of mature cucumbers were taken from each plot, extracted in acetonitrile and and analysed for fluopyram using GC-MS with triphenyl phosphate as an internal standard. The reported detection limit was 0.003 mg/kg and concurrent recovery rates were 102% (0.005 mg/kg), 111% (0.01 mg/kg) and 103% (0.05 mg/kg).

Table 116 Fluopyram residues in cucumbers from supervised trials in China involving foliar applications (500 SC formulations)

CUCUMBER Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
China, 2009 Guangxi province	3	0.075	0.0083	900	1 2 3 5	0.0918 0.0759 0.0624 0.0457				2008F312 Apr09
China, 2009 Guangxi province	4	0.075	0.0083	900	1 2 3 5	0.1083 0.0975 0.0793 0.0558				2008F312 Apr09
China, 2009 Guangxi province	3	0.113	0.0125	900	1 2 3 5	0.1398 0.1432 0.0983 0.0626				2008F312 Apr09
China, 2009 Guangxi province	4	0.113	0.0125	900	1 2 3 5	0.1628 0.1425 0.1256 0.0823				2008F312 Apr09
China, 2009 Beijing	3	0.075	0.0083	900	1 2 3 5	0.1111 0.1357 0.0721 0.0322				2008F312 May08
China, 2009 Beijing	4	0.075	0.0083	900	1 2 3 5	0.1076 0.0567 0.1147 0.0438				2008F312 May08
China, 2009 Beijing	3	0.113	0.0125	900	1 2 3 5	0.1787 0.1281 0.0457 0.0789				2008F312 May08
China, 2009 Beijing	4	0.113	0.0125	900	1 2 3 5	0.1522 0.136 0.0591 0.0449				2008F312 May08
China, 2009 Guangxi province	3	0.075	0.0083	900	1 2 3 5	0.0714 0.0659 0.0638 0.0566				2008F312 May09
China, 2009 Guangxi province	4	0.075	0.0083	900	1 2 3 5	0.0884 0.0725 0.0639 0.0359				2008F312 May09

CUCUMBER Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
China, 2009 Guangxi province	3	0.113	0.0125	900	1 2 3 5	0.1198 0.1445 0.0876 0.0645				2008F312 May09
China, 2009 Guangxi province	4	0.113	0.0125	900	1 2 3 5	0.1339 0.1488 0.1287 0.0914				2008F312 May09
China, 2009 Shaanxi province	3	0.075	0.0083	900	1 2 3 5	0.1825 0.1736 0.1247 0.0932				2008F312 May09
China, 2009 Shaanxi province	4	0.075	0.0083	900	1 2 3 5	0.1943 0.1697 0.1329 0.0945				2008F312 May09
China, 2009 Shaanxi province	3	0.113	0.0125	900	1 2 3 5	0.2813 0.2009 0.1739 0.1139				2008F312 May09
China, 2009 Shaanxi province	4	0.113	0.0125	900	1 2 3 5	0.3227 0.2518 0.1899 0.1358				2008F312 May09
China, 2009 Beijing	3	0.075	0.0083	900	1 2 3 5	0.0863 0.0503 0.0475 0.0304				2008F312 July08
China, 2009 Beijing	4	0.075	0.0083	900	1 2 3 5	0.0758 0.0706 0.0375 0.0212				2008F312 July08
China, 2009 Beijing	3	0.113	0.0125	900	1 2 3 5	0.116 0.0635 0.0631 0.034				2008F312 July08
China, 2009 Beijing	4	0.113	0.0125	900	1 2 3 5	0.0579 0.0544 0.0611 0.0286				2008F312 July08
China, 2009 Shaanxi province	3	0.075	0.0083	900	1 2 3 5	0.1778 0.1928 0.1096 0.0849				2008F312 Oct08
China, 2009 Shaanxi province	4	0.075	0.0083	900	1 2 3 5	0.1846 0.1785 0.1118 0.0897				2008F312 Oct08
China, 2009 Shaanxi province	3	0.113	0.0125	900	1 2 3 5	0.2602 0.2106 0.1839 0.1069				2008F312 Oct08
China, 2009 Shaanxi province	4	0.113	0.0125	900	1 2 3 5	0.3214 0.2419 0.1908 0.1367				2008F312 Oct08

Results from supervised trials from USA on cucumbers were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to cucumber plants either by foliar application or by drip line irrigation. In both plots the interval between applications was 5–6 days and the plot sizes ranged from 9–145 square metres.

In the plots treated by foliar application, CO_2 plot sprayers, backpack sprayers with hand-held booms or tractor-mounted spray booms were used to apply 146–182 litres spray mix/ha. In the drip line irrigation treated plots, fluopyram concentrations equivalent to 11–53 kg ai/100 litres were applied at rates of 0.6–2.2 litres per emitter per hour, to achieve an application rate equivalent to 0.25 kg ai/ha.

Duplicate samples of mature cucumbers (12 fruit from 12 areas within each plot) were taken from each plot, frozen within 1 hours of sampling and held in frozen storage for up to 280 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ 0.01 mg/kg).

Table 117 Fluopyram residues in cucumbers from supervised trials in USA involving two foliar applications (500 SC formulations)

CUCUMBER Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Chula, GA (Daytona)	2	0.253 0.25	0.173 0.138	146 181	0	0.065, 0.037				RAGMP082 GM050-07HA
USA, 2007 Oviedo, FL (Park's Bush Whopper)	2	0.24 0.245	0.14 0.139	172 176	0	0.092, 0.05				RAGMP082 GM051-07HA
USA, 2007 Bagley, IA (Brush Crop)	2	0.251 0.235	0.142 0.142	177 165	0	0.097, 0.092				RAGMP082 GM052-07HA c=0.09 mg/kg
USA, 2007 Richwood, OH (Straight 8)	2	0.255 0.251	0.159 0.157	160 160	0	0.064, 0.061				RAGMP082 GM053-07HA
USA, 2007 Raymondville, TX (Olympian)	2	0.257 0.252	0.141 0.142	182 178	0	0.099, 0.189				RAGMP082 GM054-07HA
USA, 2007 Seven Springs, NC (Ashley)	2	0.252 0.253	0.167 0.166	151 152	0 1 3 7 10	0.163, 0.118 0.141, 0.057 0.085, 0.065 0.054, 0.058 0.062, 0.067				RAGMP082 GM049-07DA

Table 118 Fluopyram residues in cucumbers from supervised trials in USA involving two drip irrigation treatments (500 SC formulations)

CUCUMBER Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/hr/em)		parent	PAA	BZM	PCA	
USA, 2007 Chula, GA (Daytona)	2	0.25 0.25	22.727 22.727	1.1 1.1	7	0.056, 0.058				RAGMP082 GM050-07HA
USA, 2007 Oviedo, FL (Park's Bush Whopper)	2	0.25 0.25	40.323 40.984	0.62 0.61	7	0.015, 0.012				RAGMP082 GM051-07HA
USA, 2007 Bagley, IA (Brush Crop)	2	0.25 0.25	11.364 11.364	2.2 2.2	7	< 0.01, 0.021				RAGMP082 GM052-07HA c=0.09 mg/kg
USA, 2007 Richwood, OH (Straight 8)	2	0.25 0.25	53.191 53.191	0.47 0.47	7	0.017, 0.01				RAGMP082 GM053-07HA
USA, 2007 Raymondville, TX (Olympian)	2	0.25 0.25	20.833 20.833	1.2 1.2	7	< 0.01, < 0.01				RAGMP082 GM054-07HA
USA, 2007 Seven Springs, NC (Ashley)	2	0.261 0.25	12.429 11.905	2.1 2.1	0 3 7 10 14	0.016, 0.013 0.022, 0.018 0.021, 0.025 0.017, 0.016 0.021, 0.017				RAGMP082 GM049-07DA

L/hr/em = Litres/hour/emitter

Results from supervised trials from Europe on protected cucumbers were provided to the Meeting. In 8 greenhouse trials, 2 applications of fluopyram (SC 500 formulations) were applied at 6–7 day intervals to mature plants as foliar sprays using knapsack sprayers with hand lances (1–3 fan or hollow-cone nozzles) to apply 0.3 kg ai/ha in 750–1500 litres of water/ha. Plot sizes in these trials ranged from 12–48 square metres.

Unreplicated samples of at least 12 cucumbers were taken from each plot, frozen within 24 hours of sampling and stored at -18 ° C or below for up to 338 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQ 0.01 mg/kg for each analyte).

Table 119 Fluopyram residues in cucumbers from supervised greenhouse trials in France, Germany, Greece, Italy, Netherlands and Spain involving foliar applications (500 SC formulation)

CUCUMBER Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Lucenay (Escape)	2	0.3	0.03	1000	-0 0 1 3 5 7	0.13 0.28 0.26 0.24 0.16 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2584/06 0365-06

CUCUMBER Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Italy, 2006 Zapponeta (Marinda)	2	0.3	0.04	750	-0 0 1 3 5 7	0.05 0.18 0.13 0.09 0.07 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2584/06 0561-06
Spain, 2006 Stª Mª del Aguila-El Ejido (Mondego)	2	0.3 0.3	0.03 0.025	1000 1200	-0 0 1 3 5 7	0.04 0.16 0.14 0.11 0.09 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2584/06 0562-06
France, 2006 Rognonas (Avalon)	2	0.3	0.02	1500	-0 0 1 2 5 7	0.07 0.23 0.22 0.2 0.16 0.14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2584/06 0563-06
Greece, 2006 Vasilika (Zetz 14)	2	0.3	0.04	750	-0 0 1 3 5 7	0.06 0.4 0.3 0.29 0.14 0.2	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2584/06 0564-06
Germany, 2006 Leichlingen (Curtis)	2	0.3	0.03	1000	-0 0 1 3 5 7	0.04 0.15 0.13 0.12 0.12 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2584/06 0566-06
Netherlands, 2006 Zwaagdijk-Oost (Sheila)	2	0.3	0.03	1000	-0 0 1 3 5 7	0.04 0.09 0.1 0.08 0.08 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2584/06 0567-06
Germany, 2006 Bornheim-Sechtem (Fitniss)	2	0.3	0.04	750	-0 0 1 4 5 7	0.01 0.1 0.08 0.04 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2584/06 0568-06

Melons (except Watermelon)

Results from supervised trials from USA on musk melons were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to melon plants either by foliar application (using) or by drip line irrigation. In both plots the interval between applications was 5 days and plot sizes ranged from 37–186 square metres.

In the plots treated by foliar application, CO_2 plot sprayers or backpack sprayers were used to apply 139–172 litres spray mix/ha. In the drip line irrigation treated plots, fluopyram concentrations equivalent to 16–29 kg ai/100 litres were applied at rates of 3.3–6.1 litres per emitter per hour, to achieve an application rate equivalent to 0.25 kg ai/ha.

Duplicate samples of mature melons (12 fruit from 12 areas within each plot) were taken from each plot, frozen within 3.5 hours of sampling and held in frozen storage for up to 220 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ 0.01 mg/kg).

Table 120 Fluopyram residues in musk melons from supervised outdoor trials in USA involving two foliar applications (500 SC formulations)

MELON Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Sycamore, GA (Edisto 47)	2	0.252 0.246	0.155 0.143	163 172	0	0.069, 0.075				RAGMP082 GM055-07HA
USA, 2007 Gardner, ND (Minnesota Midget)	2	0.263 0.262	0.177 0.179	149 146	0	0.243, 0.452				RAGMP082 GM056-07HA
USA, 2007 East Bernard, TX (Mainstream)	2	0.248 0.254	0.178 0.176	139 144	0	0.348, 0.529				RAGMP082 GM057-07HA
USA, 2007 Sanger, CA (Hale's Best Jumbo)	2	0.255 0.251	0.159 0.159	160 158	0	0.104, 0.168				RAGMP082 GM059-07HA
USA, 2007 Porterville, CA (Top Mark)	2	0.25 0.25	0.176 0.177	142 141	0	0.25, 0.215				RAGMP082 GM060-07HA
USA, 2007 Orland, CA (Hales Best)	2	0.248 0.251	0.177 0.178	140 141	0 1 3 7 10	0.081, 0.071 0.159, 0.101 0.135, 0.117 0.147, 0.137 0.081, 0.133				RAGMP082 GM058-07DA

Table 121 Fluopyram residues in musk melons from supervised outdoor trials in USA involving two drip irrigation treatments (500 SC formulations)

MELON Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/hr/em)		parent	PAA	BZM	PCA	
USA, 2007 Sycamore, GA (Edisto 47)	2	0.25 0.25	20.833 20.833	1.2 1.2	6	< 0.01, < 0.01				RAGMP082 GM055-07HA
USA, 2007 Gardner, ND (Minnesota Midget)	2	0.251 0.251	15.688 15.688	1.6 1.6	7	< 0.01, < 0.01				RAGMP082 GM056-07HA
USA, 2007 East Bernard, TX (Mainstream)	2	0.25 0.25	15.625 15.625	1.6 1.6	5	0.02, 0.026				RAGMP082 GM057-07HA
USA, 2007 Sanger, CA (Hale's Best Jumbo)	2	0.255 0.255	28.652 28.977	0.89 0.88	7	0.023, 0.03				RAGMP082 GM059-07HA
USA, 2007 Porterville, CA (Top Mark)	2	0.25 0.25	16.667 17.857	1.5 1.4	7	< 0.01, < 0.01				RAGMP082 GM060-07HA
USA, 2007 Orland, CA (Hales Best)	2	0.25 0.25	25 25	1 1	0 3 7 10 14	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, 0.011				RAGMP082 GM058-07DA

L/hr/em = Litres/hour/emitter

Results from supervised trials from Europe on melons were provided to the Meeting. In greenhouse trials, 2 applications of fluopyram (SC 500 formulations) were applied at 5 day intervals to mature plants as foliar sprays using knapsack or wheel barrow sprayers with 1–3 solid-cone or hollow-cone nozzles or with hand-held spray booms (2–8 flat fan nozzles) to apply 0.3 kg ai/ha in 600–1000 litres of water/ha. Plot sizes in these trials ranged from 24–180 square metres.

In field trials, 2 applications of fluopyram (SC 500 formulations) were applied at 7 day intervals to mature plants as foliar sprays using knapsack sprayers with single solid-cone nozzles or hand-held spray booms (3–10 flat-fan nozzles) to apply 0.25 kg ai/ha in 500–1000 litres of water/ha. Plot sizes in these trials ranged from 75–150 square metres.

Unreplicated samples of at least 12 melons were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 313 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQ 0.01 mg/kg for each analyte).

Table 122 Residues in melons from supervised greenhouse trials in France, Germany, Italy, Portugal and Spain involving two foliar applications of fluopyram (500 SC formulation)

MELONS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Carpentras (Lunastar)	2	0.3	0.03	1000	-0 0 1 3 7	0.11 0.21 0.17 0.19 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2587/06 0368-06
Spain, 2006 Roquetas de Mar (Vulcano)	2	0.3	0.03	1000	-0 0 1 3 7	0.01 0.03 0.03 0.02 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2587/06 0585-06
Italy, 2006 Manfredonia (Proteo)	2	0.3	0.0375	800	-0 0 1 3 7	0.07 0.11 0.06 0.11 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2587/06 0586-06
Germany, 2006 Euskirchen-DomEsch (Haon)	2	0.3	0.05	600	-0 0 1 3 7	0.01 0.25 0.12 0.17 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2587/06 0587-06
France, 2006 Castelsarrasin (Edgar)	2	0.3	0.03	1000	-0 0 1 3 7	0.09 0.12 0.21 0.16 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2587/06 0589-06
Portugal, 2006 São Bartolomeu-Lourinhã (Jaliscas)	2	0.3	0.03	1000	-0 0 1 3 7	0.06 0.12 0.08 0.09 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2587/06 0591-06 Climbing variety (1.8-2 m)
Spain, 2007 Sanlucar de Barrameda (Primal) Musk melon	2	0.3	0.03	1000	-0 0 1 7	0.06 0.14 0.09 0.08	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	RA-2680/07 0877-07

Table 123 Residues in melons from supervised outdoor trials in France, Germany, Greece, Italy, Portugal and Spain involving two foliar applications of fluopyram (500 SC formulation)

MELONS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Antogny le Tillac (Edgar)	2	0.25	0.025	1000	-0 0 1 3 7	0.08 0.23 0.18 0.12 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2588/06 0370-06
Germany, 2006 Monheim (Summer dream F1)	2	0.25	0.05	500	-0 0 1 3 7	0.04 0.15 0.15 0.11 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2588/06 0592-06
France, 2006 Scorbe clairvaux (Edgar)	2	0.25	0.0415	600	-0 0 1 3 7	0.03 0.07 0.05 0.08 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2589/06 0371-06
Greece, 2006 Epanomi (Sterlina)	2	0.25	0.05	500	-0 0 1 7	0.07 0.15 0.05 0.08	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	RA-2589/06 0593-06
Spain, 2006 Alginet (Charentais)	2	0.25	0.0315	800	-0 0 1 3 7	< 0.01 0.05 0.03 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2589/06 0594-06
Italy, 2006 Bologna (Summerdream)	2	0.25	0.0415	600	-0 0 1 7	0.05 0.13 0.07 0.05	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	RA-2589/06 0595-06
Greece, 2006 Aronas-Pieria (Velos FI)	2	0.3	0.06	500	-0 0 1 7	0.17 0.42 0.24 0.23	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	RA-2587/06 0590-06
Greece, 2007 Aronas-Katerini (Velos)	2	0.25	0.05	500	-0 0 1 3 7	0.1 0.26 0.18 0.21 0.16	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2508/07 0009-07
Portugal, 2007 Caniceira-Vale de Cavalos (Branco do Ribatejo)	2	0.25	0.05	500	-0 0 1 3 7	0.03 0.1 0.05 0.08 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2508/07 0535-07

Watermelon

Results from two supervised trials from Europe on outdoor watermelons were provided to the Meeting. In these trials, 2 applications of fluopyram (SC 500 formulations) were applied at 7 day intervals to mature plants as foliar sprays using knapsack sprayers with single solid-cone nozzles or hand-held spray booms (8 flat-fan nozzles) to apply 0.25 kg ai/ha in 500–1000 litres of water/ha. Plot sizes in these trials were 81 and 528 square metres.

Unreplicated samples of at least 12 melons were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 313 days before analysis for fluopyram and its

BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQ 0.01 mg/kg for each analyte).

Table 124 Residues in watermelons from supervised outdoor trials in Italy and Spain involving foliar applications of fluopyram (500 SC formulation)

WATERMELON Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Italy, 2007 Cervetari (Topgun F1)	2	0.25	0.0315	800	-0 0 1 3 7	0.04 0.08 0.07 0.06 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-2508/07 0010-07
Spain, 2007 Alginet (Pata negra)	2	0.25	0.025	1000	-0 0 1 3 7	0.03 0.12 0.09 0.08 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2508/07 0536-07

Squash, summer

Results from supervised trials from USA on summer squash were provided to the Meeting. In these trials, two applications of 0.25–0.26 kg ai/ha (SC 500 formulation) were applied to cucumber plants either by foliar application (using) or by drip line irrigation. In both plots the interval between applications was 5 days and plot sizes ranged from 46–112 square metres.

In the plots treated by foliar application, CO_2 plot sprayers, backpack sprayers or tractor-mounted spray booms were used to apply 131–187 litres spray mix/ha. In the drip line irrigation treated plots, fluopyram concentrations equivalent to 14–41 kg ai/100 litres were applied at rates of 0.6–1.8 litres per emitter per hour, to achieve an application rate equivalent to 0.25 kg ai/ha.

Duplicate samples of mature squash (12 fruit from 12 areas within each plot) were taken from each plot, frozen within 3.5 hours of sampling and held in frozen storage for up to 281 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ 0.01 mg/kg).

Table 125 Fluopyram residues in summer squash from supervised trials in USA involving foliar applications (500 SC formulations)

SQUASH, SUMMER Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Cougar)	2	0.248 0.249	0.14 0.141	177 177	0	0.094, 0.064				RAGMP082 GM061-07HA
USA, 2007 Sycamore, GA (Yellow Crookneck)	2	0.249 0.249	0.167 0.19	149 131	0	0.08, 0.138				RAGMP082 GM062-07HA
USA, 2007 Oviedo, FL (Park's Crookneck)	2	0.25 0.254	0.137 0.139	183 183	0	0.168, 0.179				RAGMP082 GM063-07HA
USA, 2007 Arkansaw, WI (Justice III)	2	0.257 0.254	0.143 0.144	180 177	0	0.084, 0.083				RAGMP082 GM064-07HA

SQUASH, SUMMER Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Orland, CA (Black Beauty)	2	0.251 0.25	0.134 0.134	187 186	0 1 3 7 10	0.051, 0.047 0.062, 0.075 0.054, 0.032 < 0.01, 0.025 < 0.01, < 0.01				RAGMP082 GM065-07DA

Table 126 Fluopyram residues in summer squash from supervised trials in USA involving drip irrigation treatments (500 SC formulations)

SQUASH, SUMMER Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/hr/em)		parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Cougar)	2	0.25 0.25	25 25	1 1	7	0.011, 0.01				RAGMP082 GM061-07HA
USA, 2007 Sycamore, GA (Yellow Crookneck)	2	0.25 0.25	20.8 20.8	1.2 1.2	6	0.013, 0.012				RAGMP082 GM062-07HA
USA, 2007 Oviedo, FL (Park's Crookneck)	2	0.25 0.25	39 41	0.64 0.61	7	< 0.01, 0.013				RAGMP082 GM063-07HA
USA, 2007 Arkansaw, WI (Justice III)	2	0.25 0.25	13.9 14.7	1.8 1.7	7	0.013, < 0.01				RAGMP082 GM064-07HA
USA, 2007 Orland, CA (Black Beauty)	2	0.25 0.25	25 25	1 1	0 3 7 10 14	< 0.01, < 0.01 < 0.01, 0.017 0.013, 0.017 < 0.01, < 0.01 0.016, 0.017				RAGMP082 GM065-07DA

L/hr/em = Litres/hour/emitter

Fruiting vegetables , other than Cucurbits

Tomatoes

Results from supervised trials from USA on tomatoes were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to tomato plants at 3–6 day intervals using ground-based CO_2 plot sprayers, backpack sprayers with hand-held 3-5metre booms or a tractor-mounted 5m side-boom to apply a total of 0.49–0.52 kg ai/ha/year Plot sizes in these trials ranged from 28–167 square metres.

Duplicate samples of mature fresh fruit (12 fruit from 12 areas within each plot) were taken from each plot, frozen within 3.6 hours of sampling and held in frozen storage for up to 534 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ 0.01 mg/kg).

Table 127 Fluopyram residues in tomatoes from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

TOMATOES Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Germansville, PA (Mountain Spring)	2	0.252 0.259	0.139 0.14	181 185	0	0.166, 0.152				RAGMP041 GM084-06HA
USA, 2006 Tifton, GA (Amelia 0800)	2	0.25 0.253	0.175 0.161	143 157	0	0.031, 0.01				RAGMP041 GM085-06HA
USA, 2006 Molino, FL (Amelia)	2	0.247 0.251	0.179 0.189	138 133	0	0.092, 0.059				RAGMP041 GM086-06HA
USA, 2006 Gretna, FL (Amelia)	2	0.252 0.256	0.147 0.147	172 174	0	0.127, 0.1				RAGMP041 GM087-06HA
Springfield, NE (Crista)	2	0.249 0.251	0.192 0.192	130 131	0	0.04, 0.08				RAGMP041 GM088-06HA
USA, 2006 Maxwell, CA (Heinz 9663)	2	0.247	0.175	141	0 3 7 10 14	0.092, 0.11 0.083, 0.1 0.098, 0.114 0.089, 0.066 0.051, 0.088				RAGMP041 GM089-06DA
USA, 2006 Fresno, CA (Rio Grande)	2	0.248 0.251	0.144 0.144	172	0	0.196, 0.181				RAGMP041 GM090-06HA
USA, 2006 Sanger, CA (2601)	2	0.25 0.252	0.197 0.188	127 134	0	0.162, 0.185				RAGMP041 GM091-06HA
USA, 2006 Fresno, CA (Sunbrite Fresh Mark)	2	0.252 0.249	0.15 0.147	168 169	0	0.162, 0.2				RAGMP041 GM092-06HA
USA, 2006 Corning, CA (APT410)	2	0.247	0.132 0.21	187	0	0.314, 0.369				RAGMP041 GM093-06HA
USA, 2006 Fresno, CA (Roma)	2	0.244 0.249	0.21 0.211	116 118	0	0.091, 0.081				RAGMP041 GM094-06HA
USA, 2006 Kettleman City, CA (410)	2	0.252 0.251	0.142 0.139	178 180	0	0.074, 0.06				RAGMP041 GM095-06HA

Results from supervised trials from Europe on tomatoes (including cherry tomatoes) were provided to the Meeting.

In 12 greenhouse trials, 2 applications of fluopyram (SC 500 formulations) were applied at 6–7 day intervals to mature plants as foliar sprays using knapsack sprayers with hand lances (1–3 fan or hollow-cone nozzles) to apply 0.3 kg ai/ha in 750–1500 litres of water/ha. Plot sizes in these trials ranged from 8–30 square metres.

In ten field trials, 2 applications of fluopyram (SC 500 formulations) were applied at 6–7 day intervals to mature plants as foliar sprays using knapsack sprayers with hand-held mini-booms (3–8 flat-fan nozzles) to apply 0.25 kg ai/ha in 500–1200 litres of water/ha. Plot sizes in these trials ranged from 10–120 square metres.

Unreplicated samples of 24 fruit (48–100 for cherry tomatoes) were taken from each plot, frozen within 24 hours of sampling and stored at or below -18 °C for up to 342 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Methods 00984 or 00984/M001 (LOQ 0.01 mg/kg for each analyte).

Table 128 Residues in tomatoes from supervised greenhouse trials in France, Germany, Greece, Italy, Netherlands, Portugal and Spain involving two foliar applications of fluopyram (500 SC formulation)

TOMATOES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 St Rémy de Provence (Cencara)	2	0.3	0.02	1500	-0 0 1 3 7	0.12 0.27 0.24 0.24 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2582/06 0363-06
Germany, 2006 Euskirchen-DomEsch (Milona)	2	0.3	0.04	750	-0 0 1 4 8	0.29 0.81 0.81 0.61 0.62	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2582/06 0545-06
Spain, 2006 Vilaseca (Boludo)	2	0.32	0.02		-0 0 1 3 7	0.14 0.26 0.28 0.26 0.28	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2582/06 0546-06
Italy Molfetta (Egnazia)	2	0.3	0.03	1000	-0 0 1 3 7	0.06 0.18 0.23 0.19 0.16	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-2582/06 0547-06
Portugal, 2006 São Domingos De Carmões (Blond)	2	0.3	0.025	1200	-0 0 1 3 7	0.16 0.39 0.32 0.31 0.44	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2582/06 0549-06
Greece, 2006 Katerini (Primadona)	2	0.3	0.03	1000	-0 0 1 3 7	0.07 0.15 0.1 0.15 0.14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2582/06 0550-06
Germany, 2006 Leichlingen (Bellavista)	2	0.3	0.03	1000	-0 0 1 3 7	0.09 0.16 0.17 0.15 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2582/06 0552-06
Netherlands, 2006 Wervershoof (Espero)	2	0.3	0.03	1000	-0 0 1 3 7	0.09 0.18 0.2 0.15 0.14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2582/06 0553-06
Germany, 2007 Leichlingen (Albis) Cherry tomato	2	0.3	0.03	1000	-0 0 1 3 7	0.1 0.13 0.18 0.13 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2591/07 0235-07 48 fruit/sample @ 21-25g each
Spain, 2007 El Ejido (TC-31175) Cherry tomato	2	0.3	0.02	1500	-0 0 1 3 7	0.25 0.39 0.3 0.36 0.35	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2591/07 0517-07 100 fruit/sample @18-19g each

TOMATOES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Italy, 2007 Comiso (Panarea) Cherry tomato	2	0.3	0.02	1500	-0 0 1 3 7	0.06 0.2 0.14 0.13 0.16	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2591/07 0518-07 60 fruit/sample @24-25g each
Germany, 2007 Langenfeld-Reusrath (Azizia) Cherry tomato	2	0.3	0.03	1000	-0 0 1 3 7	0.08 0.18 0.14 0.19 0.16	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2591/07 0519-07

Table 129 Residues in tomatoes from supervised field trials in France, Italy, Portugal and Spain involving two foliar applications of fluopyam (500 SC formulation)

TOMATOES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Portugal, 2006 Foros de Salvaterra-S.de Magos (H9144)	2	0.25	0.0335	750	0 3	0.13 0.19	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	RA-2599/06 0382-06
Portugal, 2006 Reguengo-Valada (H-9996)	2	0.25	0.05	500	-0 0 1 3 7	0.17 0.35 0.26 0.16 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2599/06 & RA-3599/06 0383-06
Italy, 2006 Andria (BA) (Donald)	2	0.25	0.0335	750	-0 0 1 3 7	0.11 0.4 0.35 0.24 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2599/06 & RA-3599/06 0402-06
Spain, 2006 Gava (Virena)	2	0.25	0.0315	800	-0 0 1 3 7	0.02 0.03 0.04 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2599/06 0612-05
France, 2006 Boé (Perfect Peel)	2	0.25	0.0415	600	-0 0 1 3 7	0.05 0.15 0.12 0.09 0.07	< 0.01 < 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01	0.01 < 0.01 0.01 0.01 0.01	RA-2599/06 & RA-3599/06 0613-03
Spain, 2007 Lebrija Sevilla (Juncal)	2	0.25	0.025	1000	-0 0 1 3 7	0.11 0.19 0.24 0.22 0.16	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2505/07 0006-07
Italy, 2007 S. Spirito (Bari) (Angos)	2	0.25	0.025	1000	-0 0 1 3 7	0.15 0.24 0.22 0.22 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2505/07 & RA-3505/06 0233-07
Portugal, 2007 Caniceira-Vale de Cavalos (Somma) Cherry tomato	2	0.25	0.05	500	-0 0 1 3 7	0.13 0.49 0.35 0.3 0.3	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.02	RA-2505/07 0234-07

TOMATOES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Spain, 2007 Gavà (Pera) Cherry tomato	2	0.25	0.021	1200	-0 0 1 3 7	0.09 0.35 0.32 0.23 0.24	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2505/07 0515-07 40-60 fruit@18-20g each
Italy, 2007 Ladispoli (RM) (Tomito F1) Cherry tomato	2	0.25	0.025	1000	-0 0 1 3 7	0.04 0.17 0.15 0.12 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2505/07 0516-07

Peppers, Sweet

Results from supervised trials from USA on sweet/bell peppers were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to mature plants, 3–5 days apart as foliar sprays using ground-based backpack plot sprayers with 1–3 metre hand-held booms to apply a total of 0.49–0.52 kg ai/ha/year. Plot sizes in these trials ranged from 84–264 square metres.

Duplicate samples of mature fresh peppers (12 fruit from 12 areas within each plot) were taken from each plot, frozen within 3.6 hours of sampling and held in frozen storage for up to 559 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 130. Fluopyram residues in sweet peppers from supervised outdoor trials in USA involving two foliar applications of fluopyram (500 SC formulations)

PEPPER, SWEET Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Tifton, GA (Capastrano)	2	0.25 0.254	0.174 0.162	144 157	0	0.143, 0.201				GM096-06HA
USA, 2006 Stilwell, KS (California Wonder)	2	0.25 0.244	0.179 0.178	140 137	0	0.04, 0.029				GM098-06HA
USA, 2006 East Bernard, TX (California Wonder)	2	0.249 0.247	0.183 0.189	136 131	0	0.363, 0.354				GM099-06HA
USA, 2006 Fresno, CA (Revolution)	2	0.262 0.25	0.147 0.147	178 170	0	0.129, 0.153				GM100-06HA
USA, 2006 San Ardo, CA (Choice)	2	0.251 0.25	0.144 0.145	174 173	0	0.096, 0.076				GM101-06HA
USA, 2006 Molino, FL (Comandant)	2	0.244 0.252	0.178 0.188	137 134	0 3 7 9 14	0.167, 0.097 0.04, 0.06 0.028, 0.017 0.018, 0.023 0.014, 0.011				GM097-06DA

Results from supervised trials from Europe on sweet peppers were provided to the Meeting. In nine greenhouse trials, 2 applications of fluopyram (SC 500 formulations) were applied at 7–10 day intervals to mature plants as foliar sprays using knapsack sprayers with hand held lances or mini-

booms (1–5 fan or hollow-cone nozzles) to apply 0.25 kg ai/ha in 500–1000 litres of water/ha. Plot sizes in these trials ranged from 16–73 square metres.

In eight field trials, 2 applications of fluopyram (SC 500 formulations) were applied at 7–8 day intervals to mature plants as foliar sprays using knapsack sprayers with hand-held mini-booms (3–8 flat-fan nozzles) to apply 0.25 kg ai/ha in 500–1200 litres of water/ha. Plot sizes in these trials ranged from 15–130 square metres.

Unreplicated samples of 24 fruit were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 337 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Methods 00984 or 00984/M001 (LOQ 0.01 mg/kg for each analyte).

Table 131 Residues in sweet peppers from supervised greenhouse trials in France, Germany, Greece, Italy, Netherlands, Portugal and Spain involving two foliar applications of fluopyram (500 SC formulations)

PEPPERS, SWEET Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Germany, 2006 Meckenbeuren (Confetti)	2	0.3 0.36	0.03	1000 1200	-0 0 1 3 7	0.27 0.42 0.42 0.42 0.39	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2583/06 0364-06
Netherlands, 2006 ND Zwaagdijk-Oost (Sopra)	2	0.3	0.03	1000	-0 0 1 3 7	0.14 0.24 0.22 0.16 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2583/06 0554-06
France, 2006 Pernes les Fontaines (Volga)	2	0.3	0.03	1000	-0 0 1 3 7	0.22 0.38 0.32 0.25 0.24	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2583/06 0555-06
Italy, 2006 Molfetta (BA) (Sienor)	2	0.3	0.04	750	-0 0 1 3 7	0.15 0.33 0.19 0.29 0.22	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2583/06 0556-06
Spain, 2006 Puebla de Vícar (Rony)	2	0.3	0.02	1500	-0 0 1 3 7	0.25 0.47 0.38 0.28 0.31	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2583/06 0557-06
Spain, 2006 Vilaseca (Aristocrata)	2	0.279 0.3	0.0215	1302 1400	-0 0 1 3 7	0.2 0.3 0.27 0.26 0.29	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2583/06 0558-06
Greece, 2006 Agia Marina (Raico)	2	0.3	0.04	750	-0 0 1 3 7	0.15 0.21 0.47 0.31 0.22	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2583/06 0559-06
Portugal, 2006 Frade de Baixo (Linares)	2	0.3	0.05	600	-0 0 1 3 7	0.15 0.53 0.32 0.25 0.24	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2583/06 0560-06

PEPPERS, SWEET Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Italy, 2007 Manfredonia (Eppo 3/4 giallo)	2	0.3	0.0265	1125	-0 0 1 3 7	0.39 0.72 0.57 0.51 0.58	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2594/07 0236-07

Table 132 Residues in sweet peppers from supervised outdoor trials in France, Greece, Italy and Spain involving two foliar applications of fluopyram (500 SC formulations)

PEPPERS, SWEET Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Le Burgaud (Florian)	2	0.25	0.0335	750	-0 0 1 3 7	0.06 0.25 0.07 0.06 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2590/06 0373-06
Spain, 2006 Alginet (Italico)	2	0.25	0.025	1000	-0 0 1 3 7	0.31 0.63 0.5 0.44 0.37	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2590/06 0597-06
Italy, 2006 Catania (Palio)	2	0.25	0.0315	800	-0 0 1 3 7	0.1 0.27 0.2 0.15 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.02 0.02 0.02	RA-2590/06 0598-06
Greece, 2006 Meliki (Raico)	2	0.25	0.05	500	-0 0 1 3 7	0.02 0.13 0.11 0.09 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2590/06 0599-06
France, 2007 Le Burgaud (Florian)	2	0.235 0.25	0.0315	752 800	-0 0 1 3 7	0.03 0.32 0.21 0.11 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2506/07 0007-07
Spain, 2007 Lebrija Sevilla (California)	2	0.25 0.268	0.0315	800 856	-0 0 1 3 7	0.1 0.21 0.23 0.24 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2506/07 0520-07
Italy, 2007 Ragusa (Sienor)	2	0.235 0.25	0.0315	752 800	-0 0 1 3 7	0.11 0.27 0.2 0.11 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2506/07 0521-07
Greece, 2007 Meliki (Raico)	2	0.25 0.273	0.05	500 545	-0 0 1 3 7	0.04 0.16 0.16 0.11 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2506/07 0522-07

Chili peppers

Results from supervised trials from USA on chili (non-bell) peppers were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to mature plants, 4–5 days apart as foliar sprays using ground-based backpack plot sprayers and hand-held mini-booms to apply a total of 0.49–0.52 kg ai/ha/year. Plot sizes in these trials ranged from 132–140 square metres.

Two duplicate samples of mature fresh peppers (12 fruit from 12 areas within each plot) were taken from each plot with one set of samples being allowed to air-dry for 14–17 days (to commercial dryness). All samples were frozen within 4 hours of sampling and held in frozen storage for up to 546 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 133 Residues in chili peppers from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

CHILI PEPPER Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			parent	PAA	BZM	PCA	
USA, 2006 Tifton, GA (Chili Pepper)	2	0.25 0.253	0.174 0.161	144 157	0	fresh dried	0.857, 1.319 0.05, 0.014 (c=0.52)				RAGMP041 GM102-06HA
USA, 2006 Molino, FL Pepper (Grande)	2	0.244 0.247	0.203 0.179	120 138	0	fresh dried	0.094, 0.14 0.166, 0.151				RAGMP041 GM103-06HA
USA, 2006 Fresno, CA Pepper (Cayenne)	2	0.248 0.25	0.145	171 172	0	fresh dried	1.086, 1.38 3.62, 3.811				RAGMP041 GM104-06HA

Sweetcorn

Results from supervised trials from USA on maize and sweetcorn were provided to the Meeting. In these trials, two applications of 0.243–0.267 kg ai/ha (SC 500 formulation) were applied to maize and sweet corn, 5–8 days apart as foliar sprays using knapsack sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 111–187 litres of spray mix/ha. Plot sizes in these trials ranged from 37–297 square metres.

In each maize trial, one plot was last treated over the early ripening stages (BBCH 85-87) for sampling of forage and immature cobs (simulating sweetcorn) and a second plot was last treated when the kernels were at the mature to fully ripe stage (BBCH 87-89) for sampling of kernels and fodder (i.e., leaves, stalks, husks and cobs after removal of the kernels).

Cobs (without husks) were sampled from sweetcorn and some field corn trials last treated at BBCH 75-79, when the kernels were close to mature (as sweetcorn).

Duplicate samples of at least 1 kg cobs were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 435 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 134 Fluopyram residues in sweetcorn and (immature) maize cobs+kernals (without husks) from supervised trials in USA involving two foliar applications (500 SC formulations)

SWEETCORN Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg fresh weight)					Reference & Comments
	kg ai/ha	water (L/ha)	GS – BBCH		matrix	parent	PAA	BZM	PCA	
USA, 2006 Germansville, PA (TA5750) Corn , Field	0.26 0.267	180 190	73 75	0 0	cobs forage (23%DM)	< 0.01, < 0.01				RAGMP038 GM043-06HA
USA, 2006 Tifton, GA (31N26) Corn , Field	0.25 0.25	135 125	71 75	0 0	cobs forage (29%DM)	< 0.01, < 0.01 4.97, 3.02				RAGMP038 GM044-06HA
USA, 2006 Molino, FL (Silver Queen) Corn , Sweet	0.261 0.254	117 112	75 79	0	cobs	< 0.01, < 0.01				RAGMP038 GM045-06HA
USA, 2006 New Holland, OH (Crows 7R154) Corn , Field	0.253 0.252	146 148	71 79	0 0	cobs forage (24%DM)	< 0.01, < 0.01 3.83, 3.15				RAGMP038 GM046-06HA
USA, 2006 Richland, IA (9190 LL HX) Corn , Field	0.245 0.25	129 131	73 75	0 0	cobs forage (22%DM)	< 0.01, < 0.01 2.53, 2.45				RAGMP038 GM047-06HA
USA, 2006 Stilwell, KS (Garst 8287 RR) Corn , Field	0.245 0.252	131 136	71 79	0 0	cobs forage (24%DM)	< 0.01, < 0.01 4.64, 5.41				RAGMP038 GM048-06HA
USA, 2006 Jerome, ID (Jackpot) Corn , Sweet	0.256 0.254	170 169	73 75	0	cobs	< 0.01, < 0.01				RAGMP038 GM060-06HA
USA, 2006 Cornelius, OR (Serendipity) Corn , Sweet	0.245 0.254	154 165	75 75	0	cobs	< 0.01, < 0.01				RAGMP038 GM061-06HA
USA, 2006 Fresno, CA (Silver Queen) Corn , Sweet	0.25 0.251	170 172	71 73	0 3 7 10 14 0 3 7 10 14	cobs cobs cobs cobs cobs forage (22%DM) forage (25%DM) forage (26%DM) forage (26%DM) forage (28%DM)	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 4.78, 5.52 4.06, 5.85 4.74, 5.78 4.75, 4.23 3.81, 4.07				RAGMP038 GM059-06DA

Leafy vegetables

Lettuce

Results from supervised trials from USA on outdoor lettuce were provided to the Meeting. In these trials, two foliar applications of fluopyram (SC 500 formulation) tank mixed with trifloxystrobin (SC 500) were applied to leaf lettuce or head lettuce plants at 0.24–0.26 kg ai/ha in 110–190 litres of water, 4–7 days apart, using CO_2 pressurised plot sprayers or knapsack sprayers. Plot sizes in these trials ranged from 25–124 square metres.

At each site, additional plots were treated with two narrow (3–15cm) band spray applications of 0.24–0.26 kg ai/ha in 23–180 litres of water over the top of the row, the first just after transplanting and the second being 1-5 weeks later, at the 5-leaf growth stage (BBCH 15).

Duplicate samples (lettuce leaves or lettuce heads with wrapper leaves) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 427 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg). Analytical results from lettuce heads without wrapper leaves are presented in Tables 196 and 197 (Magnitude of the residue in processing).

Table 135 Fluopyram residues in outdoor lettuce from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

LETTUCE Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Germans-ville, PA (New Red Fire) Leaf Lettuce	2	0.247 0.257	0.143 0.14	173 183	0	5.425, 4.434				RAGMP085 GM082-07HA
USA, 2008 Belle Glade, FL (SVR0071) Leaf Lettuce	2	0.254 0.246	0.148 0.14	172 176	0	1.911, 0.566				RAGMP085 GM083-07HA
USA, 2007 Corning, CA (Leaf) Leaf Lettuce	2	0.247 0.248	0.132 0.133	187 187	0	5.516, 6.396				RAGMP085 GM085-07HA
USA, 2008 Fresno, CA (Salad Bowl) Leaf Lettuce	2	0.25 0.249	0.214 0.215	117 116	0	9.505, 8.591				RAGMP085 GM086-07HA
USA, 2007 Porterville, CA (Salad Bowl) Leaf Lettuce	2	0.248 0.258	0.15 0.142	165 182	0	6.36, 7.197				RAGMP085 GM087-07HA
USA, 2007 King City, CA (Sunbelt) Leaf Lettuce	2	0.246 0.249	0.168 0.187	146 133	0 1 3 7 10	5.029, 4.375 *5.7, 5.6, 5.65* 4.332, 5.178 0.696, 0.695 0.295, 0.301 0.083, 0.114				RAGMP085 GM084-07DA *Results from processing (washing) study*

LETTUCE Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Germans-ville, PA (Ithaca) Head Lettuce	2	0.26 0.253	0.141 0.138	185 184	0	1.874, 3.063				RAGMP085 GM088-07HA
USA, 2008 Belle Glade, FL (Gator) Head Lettuce	2	0.254 0.248	0.148 0.139	172 178	0	1.762, 0.794				RAGMP085 GM089-07HA
USA, 2007 Corning, CA (Great Lakes) Head Lettuce	2	0.247 0.247	0.132 0.132	187 187	0	2.375, 2.6				RAGMP085 GM091-07HA
USA, 2008 Fresno, CA (Great Lakes) Head Lettuce	2	0.251 0.249	0.215 0.215	117 116	0	4.706, 4.021				RAGMP085 GM092-07HA
USA, 2007 Porterville, CA (Vandenburg) Head Lettuce	2	0.25 0.251	0.227 0.226	110 111	0	0.354, 0.812				RAGMP085 GM093-07HA
USA, 2007 Sanger, CA (Great Lakes) Head Lettuce	2	0.236 0.26	0.183 0.157	129 166	0 1 3 7 10	5.185, 5.386 2.116, 3.811 2.365, 2.06 1.131, 1.333 0.892, 0.702				RAGMP085 GM090-07DA

Table 136 Fluopyram residues in outdoor lettuce from supervised trials in USA involving two banded applications of fluopyram (500 SC formulations)

LETTUCE Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Ithaca) Head Lettuce	2	0.257 0.25	0.273 0.148	94 169	18	0.042, 0.028				RAGMP085 GM088-07HA 3-leaf (transplant)+ 5-leaf (30d interv)
USA, 2008 Belle Glade, FL (Gator) Head Lettuce	2	0.034 0.092	0.052 0.135	65 68	40	< 0.01, < 0.01				RAGMP085 GM089-07HA 3-leaf (transplant)+ 5-leaf (7d interv)
USA, 2007 Sanger, CA (Great Lakes) Head Lettuce	2	0.249 0.25	1.083 1.087	23 23	41	< 0.01, < 0.01				RAGMP085 GM090-07DA 10d postplant+5-leaf (27d interv)

LETTUCE Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Corning, CA (Great Lakes) Head Lettuce	2	0.247 0.259	0.176 0.185	140 140	33	< 0.01, < 0.01				RAGMP085 GM091-07HA Planting+5-leaf (29d interv)
USA, 2008 Fresno, CA (Great Lakes) Head Lettuce	2	0.243 0.25	1.057 1.042	23 24	120	0.04, 0.049				RAGMP085 GM092-07HA Planting+5-leaf (35d interv)
USA, 2007 Porterville, CA (Vanden-burg) Head Lettuce	2	0.25 0.25	0.176 0.176	142 142	41	0.027, 0.016				RAGMP085 GM093-07HA 2-leaf (transplant)+5-leaf (20d interv)
USA, 2007 Germansville, PA (New Red) Leaf Lettuce	2	0.252 0.259	0.205 0.142	123 182	20	0.19, 0.103				RAGMP085 GM082-07HA 3 -leaf (transplant)+5-leaf (20d interv)
USA, 2008 Belle Glade, FL (SVR0071) Leaf Lettuce	2	0.042 0.084	0.053 0.135	80 62	40	< 0.01, < 0.01				RAGMP085 GM083-07HA 3 -leaf (transplant)+5-leaf (7d interv)
USA, 2007 King City, CA (Sunbelt) Leaf Lettuce	2	0.251 0.254	0.163 0.165	154 154	43	0.027, 0.019 *0.017, 0.018 (2)*				RAGMP085 GM084-07DA 1d postplant+5-leaf (23d interv) *Results from processing (washing) study*
USA, 2007 Corning, CA (Leaf) Leaf Lettuce		0.247 0.242	0.176 0.172	140 141	48	0.012, < 0.01				RAGMP085 GM085-07HA 2-leaf (transplant)+5-leaf (14d interv)
USA, 2008 Fresno, CA (Salad Bowl) Leaf Lettuce	2	0.249 0.245	1.038 1.065	24 23	120	0.118, 0.152				RAGMP085 GM086-07HA Planting+5-leaf (35d interv)
USA, 2007 Porterville, CA (Salad Bowl) Leaf Lettuce	2	0.25 0.25	0.174 0.176	144 142	35	0.238, 0.303				RAGMP085 GM087-07HA Planting+5-leaf (33d interv)

Results from supervised trials from Europe on field and protected lettuce were provided to the Meeting. In these trials, 2 foliar applications of fluopyram (SC 500 formulations) were applied to head and leaf lettuce at a rate of 0.25 kg ai/ha in 300–1000 litres of water and 7 days apart in the greenhouse trials and in 200–800 litres water and 7–10 days apart in the field trials. Application was by knapsack sprayers and hand-held booms with 3–12 flat fan or hollow-cone nozzles. Plot sizes in these trials ranged from 16–105 square metres.

Unreplicated samples of at least 12 lettuce heads were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 342 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Methods 00984 (LOQs of 0.01 mg/kg for each analyte).

Table 137 Residues in lettuce from supervised greenhouse trials in France, Germany, Greece, Italy, Portugal and Spain involving two foliar applications of fluopyram (500 SC formulations)

LETTUCE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Ouzilly (Arcadia) Butterhead	2	0.25	0.0415	600	-0 0 3 7 10 14	5.2 11 1.7 1.2 0.86 0.51	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.02 0.01 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2586/06 0367-06
France, 2006 Fondettes (Sestice) Butterhead	2	0.25	0.0415	600	-0 0 3 7 10 14	0.51 7.3 0.66 0.16 0.12 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.02 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2586/06 0576-06
Germany, 2006 Leichlingen (Alexandria) Head lettuce	2	0.25	0.0835	300	-0 0 3 7 10 14	3.2 7.2 1.8 1.9 1.8 1.4	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2586/06 0577-06
Germany, 2006 Meckenbeuren (Natalie,) Butterhead	2	0.25	0.0835	300	-0 0 3 7 10 14	0.89 4.3 0.99 0.81 0.35 0.33	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2586/06 0578-06
Spain, 2006 Bigues i Riells (Trocadero,) Butterhead	2	0.25	0.05 0.0355	500 700	-0 0 3 7 10 14	3.4 8.7 6.2 3.8 2.8 1.6	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.02 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2586/06 0580-06
Italy, 2006 Monopoli (Brest) Head lettuce (Romaine)	2	0.25	0.0415	600	-0 0 3 7 10 14	2.7 9.1 6 4.9 7.7 5.2	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.02 0.02 0.04 0.03	< 0.01 0.01 0.01 0.01 0.02 0.02	RA-2586/06 0581-06 24 small immature heads/1.5-2kg sample
Portugal, 2006 Olho Marinho (Gisela,) Butterhead	2	0.25	0.05	500	-0 0 3 7 10 14	4.6 8.6 9.1 4.4 3.5 1.9	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.04 0.04 0.04 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2586/06 0583-06
Greece, 2006 Marathonas (Manita) Butterhead	2	0.25	0.05	500	-0 0 3 7 10 14	6 22 11 8.4 7 6.5	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.03 0.04 0.03 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2586/06 0584-06 13 small (120g) heads/1.2-1.8kg sample

LETTUCE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2007 St Rémy de Provence (Zandria) Head lettuce	2	0.25	0.025	1000	-0 0 3 7 10 14	1.4 5.1 4 2.5 2.2 1.5	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.04 0.04 0.05 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2604/07 0247-07
Germany, 2007 Leichlingen (Locarno) Leaf lettuce	2	0.25	0.0415	600	-0 0 3 7 10 14	2.0 9.4 7.8 2.7 2.1 1.4	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 0.02 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2604/07 0542-07
Italy, 2007 Manfredonia (Canasta rossa-Holter) Leaf lettuce	2	0.25	0.028	900	-0 0 3 7 10 14	1.9 5.5 2.4 0.83 2.3 1.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 < 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2604/07 0543-07
Spain, 2008 Bigues i Riells (Batavia) Leaf lettuce	2	0.25	0.05 0.0415	500 600	-0 0 3 7 10	6.6 11 8.3 7.2 5.5	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2604/07 0544-07

Table 138 Residues in lettuce from supervised field trials in Belgium, France, Germany, Greece, Italy, Portugal, Spain and UK involving two foliar applications of fluopyram (500 SC formulations)

LETTUCE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Cergy (Estelle) Head lettuce	2	0.25	0.05	500	-0 0 3 7 10 13	1.9 5.9 0.87 0.62 0.46 0.34	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.02 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2592/06 0375-06
Germany, 2006 Meckenbeuren (Jiska) Head lettuce	2	0.25	0.05	500	-0 0 3 7 11 14	0.08 8.2 1.9 0.18 0.05 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2592/06 0604-06 10d spray interval
Germany, 2006 Langenfeld-Reusrath (Gisela) Head lettuce	2	0.25	0.0415	600	-0 0 3 7 10 14	0.2 4.3 1.3 0.93 0.47 0.3	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2592/06 0605-06
Netherlands, 2006 Zwaagdijk-Oost (Namia) Head lettuce	2	0.25	0.0315	800	-0 0 3 7 10 14	0.37 1.7 0.71 0.26 0.1 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2592/06 0606-06

LETTUCE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
United Kingdom, 2006 Ely (Iceburg) Head lettuce	2	0.25 0.27	0.125	200 216	-0 0 3 7 10 14	0.75 7.6 1.2 0.13 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2592/06 0607-06
United Kingdom, 2007 Barway-Near Ely (Elenar) Head lettuce	2	0.25	0.0835	300	-0 0 3 6 9 13	0.36 6.1 0.82 0.18 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2509/07 0011-07
Germany, 2007 Meckenbeuren (Nobilan) Butterhead	2	0.2675 0.25	0.0835	321 300	-0 0 2 7 10 13	0.41 4.5 3.6 0.61 0.44 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2509/07 0244-07
Germany, 2007 Langenfeld-Reusrath (Torpedo) Head lettuce	2	0.25	0.0415	600	-0 0 3 7 10 14	0.85 6.1 1.1 0.57 0.43 0.33	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.03 0.02 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2509/07 0537-07
France, 2007 Puzeaux (Madras) Head lettuce	2	0.25	0.0835	300	-0 0 4 7 11 15	< 0.01 6 0.12 0.12 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2509/07 0538-07 large, 700g units
Belgium, 2007 Villers-Perwin (Appia) Butterhead	2	0.25	0.0625	400	-0 0 3 7 10 14	0.09 5.9 2.1 0.63 0.42 0.26	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2509/07 0539-07
Netherlands, 2007 Zwaagdijk-Oost (Lolo Rosso) Leaf lettuce	2	0.25	0.0335	750	-0 0 3 7 10 14	0.48 6.5 1.3 0.53 0.16 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.02 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2509/07 0540-07
France, 2006 St Jory (Toucan) Head lettuce	2	0.25	0.0415	600	-0 0 3 7 10 14	0.47 2.7 0.61 0.25 0.17 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.02 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2593/06 0376-06
Spain, 2006 Vilanova del Vallés (Rumina) Head lettuce	2	0.25	0.0625 0.0415	400 600	-0 0 4 7 10 14	0.27 3.5 1.5 0.29 0.07 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.02 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2593/06 0608-06

LETTUCE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Italy, 2006 Andria (Brest) Head lettuce	2	0.25	0.0355	700	-0 0 4 7 10 14	0.09 0.62 0.02 0.02 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2593/06 0609-06
Italy, 2006 Catania (Trocadero) Head lettuce	2	0.25	0.0415	600	-0 0 3 8 10 14	0.59 1.8 0.92 0.46 0.23 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2593/06 0610-06
Greece, 2006 Katerini/ Paralia (White Boston) Head lettuce	2	0.25	0.05	500	-0 0 3 8 10 14	2.1 11 6.4 1.7 0.39 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.04 0.05 0.04 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2593/06 0611-06 12-40 small heads/0.5-1kg sample
Portugal, 2007 Olho Marinho (Faustinas) Butterhead	2	0.25	0.0625	400	-0 0 3 7 10 14	0.31 4.5 1.3 0.48 0.24 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2510/07 0012-07
Greece, 2007 Agia Marina (Atraction) Leaf lettuce	2	0.25	0.0625	400	-0 0 3 7 10 14	0.11 7 1.2 0.29 0.1 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.02 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2510/07 0245-07
France, 2007 Ouzilly (Santoro) Butterhead	2	0.25	0.0415	600	-0 0 3 7 10 14	0.17 6 0.56 0.27 0.14 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.02 0.02 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2510/07 0246-07
Italy, 2007 Manfredonia (Antony) Leaf lettuce	2	0.25	0.0355	700	-0 0 3 7 10 14	0.8 7.4 4.8 5.3 0.87 0.98	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 0.02 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2510/07 0541-07

Spinach

Results from supervised trials from USA on outdoor spinach were provided to the Meeting. In these trials, two foliar applications of fluopyram (SC 500 formulation) tank mixed with trifloxystrobin (SC 500) were applied to spinach plants at 0.25–0.26 kg ai/ha in 117–183 litres of water, 5–7 days apart, using CO_2 pressurised plot sprayers, knapsack sprayers or tractor-mounted booms. Plot sizes in these trials ranged from 33–124 square metres.

Duplicate samples (spinach leaves and stalks) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 211 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg). Analytical results from washed and cooked spinach are presented in Table 198 (Magnitude of the residue in processing).

Table 139 Fluopyram residues in spinach from supervised outdoor trials in USA involving two foliar applications of fluopyram (500 SC formulations)

SPINACH Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Tyee)	2	0.257 0.257	0.14 0.14	183 183	0	8.52, 11.02				RAGMP085 GM100-07HA
USA, 2007 Suffolk, VA (Tyee)	2	0.254 0.253	0.207 0.211	123 120	0	22.5, 21.55				RAGMP085 GM101-07HA
USA, 2007 Hinton, OK (Bloomsdale)	2	0.248 0.252	0.2 0.208	124 121	0	16.26, 18.4				RAGMP085 GM102-07HA
USA, 2007 Jerome, ID (Unipack 151)	2	0.252 0.249	0.145 0.146	174 170	0	7.994, 8.434				RAGMP085 GM103-07HA
USA, 2007 Fresno, CA (Shasta)	2	0.247 0.247	0.211 0.211	117 117	0	14.65, 15.04				RAGMP085 GM105-07HA
USA, 2007 King City, CA (Interceptor/Avenger)	2	0.251 0.249	0.179 0.184	140 135	0 1 3 7 10	8.437, 9.241 *9.76, 9.26, 9.8* 8.684, 8.067 7.481, 6.954 1.126, 1.192 0.365, 0.62				RAGMP085 GM104-07DA *Results from processing study*

Mustard greens

Results from supervised trials from USA on mustard greens were provided to the Meeting. In these trials, two foliar applications of 0.24–0.26 kg ai/ha fluopyram (SC 500 formulation), tank-mixed with trifloxystrobin (SC) in 92–188 litres of water were applied to mustard green plants 4–5 days apart using CO_2 pressurised plot sprayers, knapsack sprayers with hand-held booms or tractor-mounted spray booms. Plot sizes in these trials ranged from 67–212 square metres.

Duplicate samples of at least 1 kg mustard green leaves were taken from each plot, frozen within 1 hour of sampling, held in frozen storage for up to 567 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg). Analytical results from washed mustard greens are presented in Table 199 (Magnitude of the residue in processing).

Table 140 Fluopyram residues in mustard greens from supervised trials in USA involving foliar applications (500 SC formulations)

MUSTARD GREENS Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Bumpass, Virginia (Unknown)	2	0.249 0.251	0.18 0.181	138 139	0	8.56, 9.42				RAGMP063 GM231-06HA
USA, 2006 Molino, Florida (Giant Southern Curl)	2	0.247 0.249	0.242 0.239	102 104	0	17.24, 17.23				RAGMP063 GM232-06HA

MUSTARD GREENS Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Leland, Mississippi (Florida Broadleaf)	2	0.249 0.248	0.201 0.2	124 124	0	13.89, 13.19				RAGMP063 GM233-06HA
USA, 2006 Seymour, Illinois (Southern Giant Curl)	2	0.251 0.249	0.177 0.179	142 139	0	15.4, 16.99				RAGMP063 GM234-06HA
USA, 2006 East Bernard, Texas (Florida Broadleaf)	2	0.25 0.259	0.181 0.186	138 139	0	23.56, 23.97				RAGMP063 GM235-06HA
USA, 2006 Fresno, California (Florida Broadleaf)	2	0.252 0.25	0.147 0.149	172 168	0	24.73, 26.51				RAGMP063 GM236-06HA
USA, 2007 Fresno, California (Green Wave)	2	0.244 0.245	0.145 0.145	168 169	0	15.47, 14.51				RAGMP063 GM237-06HA
USA, 2007 Sycamore, Georgia (Florida Broadleaf)	2	0.252 0.253	0.217 0.201	116 126	0 3 7 10 14	13.74, 9.97 *12.2, 12.7, 13.1* 12.45, 12.39 4.16, 4.2 4.56, 3.48 1.53, 1.65				RAGMP063 GM230-06DA *Results from processing (washing) study*

Chinese cabbage, (type Pe-tsai)

Results from supervised trials from Europe on chinese cabbage were provided to the Meeting. In these trials, 2 applications of fluopyram + tebuconazole (SC 200+200) at rates 0.2 kg ai/ha fluopyram in 300–600 litres water/ha of were applied at 14–16 day intervals as foliar sprays using knapsack sprayers with spraying booms (5–10 flat fan nozzles). Plot sizes in these trials ranged from 30–80 square metres.

Unreplicated samples of at least 8–12 cabbage heads or plants (without roots) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 315 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQs were 0.01 mg/kg for each analyte).

Table 141 Fluopyram residues in chinese cabbage from supervised trials in France, Germany, Italy, Spain and UK involving two foliar applications (400 SC formulation)

CHINESE CABBAGE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Bouafle (Chorus F1) closed head	2	0.2	0.04	500	-0 0 3 7 14 21	0.26 1.5 0.43 0.22 0.16 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2573/06 0347-06
United Kingdom, 2006 Little Shelford (F1 The Blues) closed head	2	0.2 0.212	0.0666 0.0664	300 319	-0 0 3 7 14 21	0.13 1.6 1.3 0.84 0.15 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2573/06 0543-06

CHINESE CABBAGE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
United Kingdom, 2007 Broad Fen Drove/Wissington (Onekilo) closed head	2	0.2	0.0666	300	-0 0 7 14 21	0.01 4.4 0.42 0.03 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2533/07 0078-07
Germany, 2007 Meckenbeuren (Orient Express) closed head	2	0.2	0.0666	300	-0 0 7 14 21	0.06 3.8 0.29 0.23 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2533/07 0599-07
France, 2006 Ouzilly (Kaboko) closed head	2	0.2	0.0334	600	-0 0 3 7 14 21	0.12 1.5 0.44 0.21 0.17 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2574/06 0348-06
Italy, 2006 Andria (Taranko) open head (“Bok Choy”	2	0.2	0.0334	600	-0 0 3 7 14 21	0.02 0.57 0.13 0.1 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.03 0.03 0.03 0.02	< 0.01 < 0.01 0.02 0.02 0.03 0.03	RA-2574/06 0544-06
France, 2007 Ouzilly (Kaboko) closed head	2	0.2	0.0334	600	-0 0 7 14 21	0.19 5.4 0.88 0.49 0.42	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2534/07 0079-07
Spain, 2007 Brenes (Sumiko) closed head	2	0.2	0.05	400	-0 0 7 14 21	0.22 1.7 0.44 0.21 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 < 0.01 < 0.01	< 0.01 0.01 < 0.01 < 0.01 < 0.01	RA-2534/07 0600-07

Legume vegetables

Common Bean

Results from supervised trials from USA on common beans (seeds and pods) were provided to the Meeting. In these trials, two applications of 0.22–0.24 kg ai/ha (SC 500 formulation) were applied to snap beans, 5–7 days apart as foliar sprays using CO_2 plot sprayers or knapsack sprayers with hand-held spray booms to apply 127–177 litres of spray mix/ha. Plot sizes in these trials ranged from 70–233 square metres.

Duplicate samples of at least 1 kg pods (with seeds) were taken from each plot, frozen within 2 hours of sampling, held in frozen storage for up to 538 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 142 Fluopyram residues in common beans (young pods and immature seeds) from supervised trials in USA involving two foliar applications (500 SC formulations)

BEANS Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Germansville, PA (Bush Blue Lake 274)	2	0.25 0.249	0.141 0.141	177 177	0	0.653, 0.743				RAGMP067 GM303-06HA
USA, 2006 Tifton, GA (Bronco)	2	0.25 0.25	0.174 0.157	144 159	0	0.27, 0.555				RAGMP067 GM304-06HA
USA, 2006 Molino, FL (Bush Blue Lake)	2	0.253 0.256	0.189 0.187	134 137	0	0.313, 0.18				RAGMP067 GM305-06HA
USA, 2006 Springfield, NE (Bush Blue Lake)	2	0.248 0.249	0.195 0.192	127 130	0	0.137, 0.119				RAGMP067 GM307-06HA
USA, 2006 Parkdale, OR (Blue Lake 274)	2	0.258 0.254	0.174 0.149	148 170	0	0.151, 0.193				RAGMP067 GM308-06HA
USA, 2006 Stilwell, KS (Top Crop)	2	0.248 0.25	0.18 0.179	138 140	0 3 7 10 14	0.165, 0.137 0.134, 0.159 0.078, 0.09 0.067, 0.073 0.057, 0.032				RAGMP067 GM306-06DA

Results from European supervised trials on outdoor kidney beans and protected French climbing beans were provided to the Meeting. In these trials, 2 applications of fluopyram (SC 500 formulations) were applied 7–8 days apart as foliar sprays using knapsack or wheel barrow sprayers with 1–4 flat-fan, solid or hollow-cone nozzles or hand-held-booms (3–12 flat-fan nozzles), applying 0.3 kg ai/ha in 600–1500 litres water/ha to the protected crops and 0.25 kg ai/ha in 300–1000 L water/ha to the outdoor crops. Plot sizes in these trials ranged from 8–108 square metres.

Unreplicated samples of 1-3 kg of pods (including seeds) and in the outdoor trials at least 12–18 kidney bean plants (minimum of 1 kg green material, including pods and seeds) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 342 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Methods 00984 or 00984/M001. The reported LOQs were 0.01 mg/kg for each analyte.

Table 143 Residues in protected common beans (seeds and pods) from supervised trials in Belgium, France, Germany, Netherlands and Spain involving foliar applications of fluopyram (500 SC)

BEANS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
France, 2006 Rognonas (Nadal) Bean, French	2	0.3	0.03	1000	-0 0 3 7 10 14	pod	0.24 1.2 0.63 0.43 0.33 0.24	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2596/06 0379-06
France, 2006 Noves (Donna) Bean, French	2	0.3	0.03	1000	-0 0 3 7 10 14	pod	0.43 1.2 0.78 0.69 0.49 0.36	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-2596/06 0752-06

BEANS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Spain, 2006 Almerimar (Donna) Bean, French	2	0.3	0.02	1500	-0 0 3 7 10 14	pod	0.14 0.83 0.63 0.2 0.16 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2596/06 0753-06
Spain, 2006 St M del Aguila (Festival) Bean, French	2	0.3	0.02	1500	-0 0 3 7 10 14	pod	0.27 0.99 0.68 0.22 0.15 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2596/06 0754-06
Germany, 2006 Langenfeld (Markant) Bean, French	2	0.3	0.05	600	-0 0 3 7 10 14	pod	0.23 0.39 0.28 0.16 0.09 0.1	< 0.01 < 0.01 < 0.01 0.01 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2596/06 0755-06
Germany, 2006 Meckenbeuren (Eva) Bean, French	2	0.3	0.02	1500	-0 0 3 7 10 13	pod	0.21 0.55 0.49 0.12 0.16 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2596/06 0756-06
Netherlands, 2006 Andijk (Overvloed) Bean, French	2	0.3	0.03	1000	-0 0 3 7 10 14	pod	0.16 0.33 0.11 0.06 0.07 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2596/06 0757-06
Belgium, 2006 Villers-Perwin (Grappes de Malines) Bean, French	2	0.3 0.327	0.03	1000 1090	-0 0 3 7 10 14	pod	0.1 0.27 0.22 0.14 0.15 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2596/06 0759-06
Spain, 2007 Puebla de Vicar (Donna) Bean, French	2	0.3	0.02	1500	-0 0 3 7 10 14	pod	0.2 0.73 0.59 0.22 0.09 0.06	< 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2607/07 0248-07

Table 144 Residues in outdoor common beans (seeds and pods) from supervised trials in Belgium, France, Germany, Italy, Netherlands and Spain involving foliar applications of fluopyram (500 SC)

BEANS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Germany, 2006 Lampertheim (Albani) Bean, Kidney	2	0.25	0.05	500	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.09 0.57 0.53 0.24 0.18 0.15 0.24 3.2 4.1 0.71 0.4 0.26	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.02 0.03 0.04 0.05 0.02 0.03 0.05 0.06 0.06 0.06	< 0.01 < 0.01 < 0.01 0.01 0.02 0.02 < 0.01 0.02 0.01 0.01 0.01 0.01	RA-2594/06 0377-06
Germany, 2006 Langenfeld-Reusrath (Classic) Bean, Kidney	2	0.25	0.0835	300	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.05 0.2 0.1 0.07 0.06 0.06 0.25 7.8 0.42 0.24 0.17 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02 0.02 0.03 0.03 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2594/06 0654-06
Netherlands, 2006 Zwaagdijk-Oost (Unknown) Bean, Kidney	2	0.25 0.23	0.05	500 460	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.05 0.22 0.21 0.2 0.19 0.13 0.33 5.7 2.6 0.88 0.55 0.37	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.01 0.02 0.02 0.03 0.03 0.06 0.08 0.08 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2594/06 0655-06
Belgium, 2006 Villers-Perwin (Polder) Bean, Kidney	2	0.25	0.0385	650	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.14 0.47 0.41 0.21 0.18 0.12 0.98 14 8 1.3 0.99 0.99	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.01 0.02 0.03 0.03 0.03 0.02 0.03 0.05 0.03 0.02 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01	RA-2594/06 0656-06

BEANS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Belgium, 2007 Villers-Perwin (Cadillac) Bean, Kidney	2	0.25	0.025	1000	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.06 0.34 0.3 0.12 0.09 0.08 0.37 7 4.2 0.42 0.33 0.19	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02 0.02 0.03 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 	RA-2511/07 0014-07
Germany, 2007 Langenfeld-Reusrath (Classic) Bean, Kidney	2	0.25	0.0415	600	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.09 0.39 0.23 0.18 0.14 0.13 0.14 3 1.4 0.57 0.37 0.2	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 0.01 0.01 0.02 0.02 0.02 0.03 0.05 0.04 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2511/07 0546-07
France, 2007 Fresnoy les Roye (Lugos,french bean variety) Bean, Kidney	2	0.25	0.05	500	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.13 0.45 0.39 0.26 0.18 0.13 0.37 6.2 3.4 0.68 0.34 0.17	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 0.02 0.02 0.02 0.02 0.03 0.04 0.05 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2511/07 0547-07
Netherlands, 2007 Biddinghuizen (Cadillac,french bean variety) Bean, Kidney	2	0.25	0.05	500	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.07 0.27 0.19 0.19 0.17 0.15 0.56 5.4 1.8 0.72 0.65 0.46	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01 0.01 0.02 0.02 0.03 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2511/07 0548-07

BEANS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Germany, 2007 Swisttal-Heimerzheim (Sonesta,french bean variety) Bean, Kidney	2	0.25	0.0415	600	-0 0 3 6 10 13 -0 0 3 6 10 13	pod plant 	0.08 0.41 0.35 0.17 0.1 0.08 0.2 5.9 5.4 0.31 0.18 0.09	< 0.01 < 0.01 0.01 0.02 0.02 0.02 0.01 0.01 0.02 0.02 0.02 0.02	< 0.01 < 0.01 0.01 0.01 0.01 0.01 0.04 0.04 0.06 0.05 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2511/07 0549-07
Spain, 2006 Alginet (Cleo) Bean, Kidney	2	0.25	0.0415	600	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.03 0.4 0.17 0.24 0.13 0.14 0.25 3.0 2.9 1.6 1.3 1.2	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02	< 0.01 < 0.01 0.01 0.03 0.03 0.06 0.01 0.02 0.05 0.07 0.09 0.13	< 0.01 < 0.01 < 0.01 0.01 0.03 0.04 < 0.01 < 0.01 0.01 0.01 0.02 0.03	RA-2595/06 0378-06
Italy, 2006 Pradelle di Nogarole Rocca (Jamaica) Bean, Kidney	2	0.25	0.05	500	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.08 0.6 0.15 0.1 0.08 0.06 0.22 7.7 0.42 0.34 0.26 0.2	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02 0.02 0.03 0.03 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2595/06 0620-06
Spain, 2006 Malgrat de Mar (Nasao) Bean, Kidney	2	0.25	0.05 0.0415	500 600	-0 0 2 7 10 14 -0 0 2 7 10 14	pod plant 	0.03 0.42 0.1 0.05 0.05 0.03 0.18 3.9 0.81 0.25 0.19 0.13	< 0.01 < 0.01 < 0.01 0.01 0.02 0.02 < 0.01 < 0.01 < 0.01 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 0.02 0.03 0.03 0.03 0.03 0.05 0.06 0.08 0.1	< 0.01 < 0.01 < 0.01 0.01 0.02 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-2595/06 0657-06

BEANS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Italy, 2006 Ladispoli (Bronco) Bean, Kidney	2	0.25	0.0315	800	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.06 0.33 0.18 0.11 0.08 0.1 0.51 5.8 4.1 0.84 0.86 0.39	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 0.02 0.02 0.04 0.03 0.04 0.06 0.08 0.09 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2595/06 0658-06
France, 2007 Chazay d'azergues (Contender) Bean, French	2	0.25	0.025	1000	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.07 0.45 0.25 0.11 0.1 0.08 0.52 8.2 5.7 0.61 0.69 0.8	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 0.02 0.02 0.02 0.03 0.05 0.04 0.09 0.06 0.08 0.08	< 0.01 < 0.01 0.01 0.02 0.02 0.02 < 0.01 < 0.01 0.02 0.01 < 0.01 0.01	RA-2512/07 0035-07
Italy, 2007 Ladispoli (Bronco) Bean, Kidney	2	0.25	0.0315	800	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.02 0.47 0.05 0.03 0.02 0.02 0.28 8.9 0.58 0.26 0.19 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02 0.02 0.03 0.03 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2512/07 0550-07
Spain, 2007 Alginet (Cleo) Bean, Kidney	2	0.25	0.025	1000	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.12 0.17 0.26 0.25 0.19 0.16 2.6 5.9 3.7 4.3 2.4 1.2	< 0.01 < 0.01 0.01 0.03 0.04 0.06 < 0.01 < 0.01 0.01 0.02 0.03 0.03	< 0.01 0.01 0.02 0.04 0.05 0.06 0.05 0.05 0.06 0.13 0.13 0.14	< 0.01 < 0.01 < 0.01 0.01 0.01 0.02 < 0.01 < 0.01 < 0.01 0.01 0.01 0.01	RA-2512/07 0551-07

BEANS Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Portugal, 2007 Ribafria Peniche (Tradicional) Bean, Kidney	2	0.25	0.025	1000	-0 0 3 7 10 14 -0 0 3 7 10 14	pod plant 	0.3 0.52 0.45 0.43 0.27 0.32 1.7 7.8 3.4 2.2 2.8 2.0	0.01 < 0.01 0.02 0.05 0.07 0.11 0.02 0.02 0.02 0.05 0.08 0.09	0.02 0.01 0.02 0.05 0.05 0.07 0.05 0.06 0.07 0.11 0.13 0.15	< 0.01 < 0.01 < 0.01 0.01 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02	RA-2512/07 0552-07

Beans, shelled

Results from supervised trials from USA on Lima beans (without pods) were provided to the Meeting. In these trials, two applications of 0.25 kg ai/ha (SC 500 formulation) were applied to Lima beans, 5–7 days apart as foliar sprays using knapsack sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 143–197 litres of spray mix/ha. Plot sizes in these trials ranged from 93–446 square metres.

Duplicate samples of at least 1 kg seeds were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 557 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 145 Fluopyram residues in shelled lima beans (immature seeds) from supervised trials in USA involving foliar applications (500 SC formulations).

BEANS (SHELLED) Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Chula, GA (Bridgeton)	2	0.253 0.25	0.147 0.145	172 172	0	0.019, 0.02				RAGMP068 GM313-06HB
USA, 2006 Springfield, NE (Ford Hook)	2	0.249 0.251	0.192 0.192	130 131	0	0.015, 0.013				RAGMP068 GM315-06HA
USA, 2006 Sanger, CA (Henderson Bush)	2	0.25 0.249	0.197 0.196	127 127	0	0.051, 0.038				RAGMP068 GM316-06HA
USA, 2006 Jerome, ID (Fordhook 242)	2	0.248 0.25	0.153 0.151	162 166	0	0.011, 0.01				RAGMP068 GM317-06HA
USA, 2006 Seven Springs, NC (Early Thorogreen Bus)	2	0.252 0.249	0.143 0.141	176 177	0 3 7 10 14	0.041, 0.04 0.01, 0.004 0.063, 0.064 0.066, 0.074 0.051, 0.045				RAGMP068 GM312-06DA

Peas, shelled

Results from supervised trials from USA on garden peas (without pods) were provided to the Meeting. In these trials, two applications of 0.25–0.26 kg ai/ha (SC 500 formulation) were applied to garden

peas, 5–7 days apart as foliar sprays using knapsack or pressurised sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 83–182 litres of spray mix/ha. Plot sizes in these trials ranged from 60–186 square metres.

Duplicate samples of at least 1 kg seeds were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 557 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 146 Fluopyram residues in shelled garden peas (succulent seeds) from supervised trials in USA involving foliar applications (500 SC formulations)

PEAS (SHELLED) Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Wando)	2	0.253 0.256	0.14 0.141	181 182	0	< 0.01, < 0.01				RAGMP068 GM318-06HB
USA, 2006 Stilwell, KA (Little Marvel)	2	0.249 0.251	0.195 0.189	128 133	0	0.033, 0.035				RAGMP068 GM320-06HA
USA, 2006 Seymour, IL (Early Frosty)	2	0.258 0.253	0.198 0.178	130 142	0	0.076, 0.068				RAGMP068 GM321-06HA
USA, 2006 Rupert, ID (Little Marvel)	2	0.244 0.249	0.294 0.28	83 89	0	0.050, 0.067				RAGMP068 GM322-06HA
USA, 2006 Hillsboro, OR (Majorettes)	2	0.247 0.25	0.142 0.142	174 176	0	0.089, 0.038				RAGMP068 GM323-06HA
USA, 2006 Springfield, NE (Lincoln)	2	0.25 0.248	0.197 0.197	127 126	0 3 7 10	0.081, 0.064 0.036, 0.031 0.041, 0.036 0.04, 0.038				RAGMP068 GM319-06DA

Results from supervised trials on outdoor garden peas in Europe were provided to the Meeting. In these trials, 2 applications of fluopyram (SC 500 formulation) were applied to peas 7–8 days apart as foliar sprays using knapsack or wheel barrow sprayers with hand-held spray booms (3–12 flat-fan or hollow cone nozzles), applying 0.25 kg ai/ha in 300–600 litres water/ha. Plot sizes in these trials ranged from 24–101 square metres.

Unreplicated samples of at 0.5–4 kg succulent seeds, pods and/or whole plants (including pods and seeds but without roots) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 329 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984. The reported LOQs were 0.01 mg/kg for each analyte

Table 147 Residues in garden peas (seeds, pods and whole plants) from supervised trials in Belgium, France, Germany, Italy, Netherlands, Spain and UK involving two foliar applications of fluopyram (500 SC)

GARDEN PEA Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Germany, 2006 Machern (Harnaß)	2	0.25	0.079 0.0745	317 336	-0 0 3 7 10 14 3 7 10 14	plant pod seed 	1.2 3.4 3.4 3.5 1.8 2.1 0.2 0.03 0.02 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.03 0.05 0.04 0.06 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 0.02	RA-2597/06 0380-06
United Kingdom, 2006 Needham (Hawk)	2	0.25	0.0835	300	-0 0 3 7 10 14 3 7 10 14	plant pod seed 	0.91 3.9 4.1 3.3 2.7 1.7 0.57 0.05 0.03 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.02 0.03 0.04 0.06 0.07 < 0.01 < 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2597/06 0722-06
Germany, 2006 Meckenbeuren (Rondo)	2	0.25	0.0835	300	-0 0 3 7 10 13 3 7 10 13	plant pod seed 	0.05 4.2 3.8 0.46 0.37 0.24 0.4 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.02 0.04 0.04 0.04 0.04 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-2597/06 0723-06
Netherlands, 2007 Kopstukken (unknown)	2	0.25	0.0415	600	-0 0 3 7 10 14 7 10 14	plant seed 	0.29 5.7 4.9 1.9 1.8 0.66 0.05 0.04 0.03	< 0.01 < 0.01 < 0.01 0.01 0.01 < 0.01 0.01 0.01 0.01	0.01 0.01 0.05 0.06 0.05 0.02 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01 < 0.01	RA-2513/07 0036-07
Germany, 2007 Burscheid (Wunder von Kelvedon)	2	0.25	0.0835	300	-0 0 3 7 10 14 7 10 14	plant seed 	2.1 6.6 1.6 1.1 0.7 0.51 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.03 0.04 0.02 0.02 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2513/07 0553-07

GARDEN PEA Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
France, 2007 Goyencourt (Arabelle)	2	0.25	0.0835	300	-0 0 3 7 10 14 7 10 14	plant seed 	0.97 7.6 3.8 1.3 1.3 2.3 0.03 0.03 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02 < 0.01 < 0.01 0.01	0.02 0.03 0.03 0.04 0.04 0.06 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 0.01 0.02	RA-2513/07 0554-07
Belgium, 2007 Landenne-Sur-Meuse (Tristar)	2	0.25	0.0625	400	-0 0 3 7 10 14 7 10 14	plant seed 	0.31 2.5 2.4 0.81 0.7 0.45 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	0.01 0.01 0.03 0.04 0.05 0.04 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-2513/07 0555-07
Germany, 2007 Swisttal-Heimerzheim (Spring)	2	0.25	0.0835	300	-0 0 3 7 10 13 7 10 13	plant seed 	0.58 6.7 7.1 4.3 2.5 1.1 0.02 0.01 < 0.01	< 0.01 < 0.01 0.01 0.02 0.01 0.01 0.02 0.01 < 0.01	< 0.01 0.06 0.11 0.17 0.06 0.06 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 0.01 0.01 < 0.01	RA-2513/07 0556-07
Spain, 2006 Brenes (Rondo)	2	0.25	0.0835	300	-0 0 3 7 9 14 3 7 9 14	plant pod seed 	4.5 8.8 8 6.6 4.6 5.7 0.94 0.1 0.05 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.08 0.09 0.1 0.18 0.13 0.28 0.02 0.01 0.01 0.02	0.02 0.02 0.03 0.04 0.02 0.03 < 0.01 0.01 0.02 0.03	RA-2598/06 0381-06
Italy, 2006 Migliarino (Agami)	2	0.25	0.0625	400	-0 0 3 7 9 14 3 7 9 14	plant pod seed 	0.21 5.8 0.24 0.2 0.15 0.12 0.06 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.02 0.03 0.03 0.03 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2598/06 0724-06

GARDEN PEA Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
France, 2007 Chazay d'Azergues (Douce de provence)	2	0.25	0.0625	400	-0 0 3 7 10 14 7 10 14	plant seed 	0.75 7.9 6 0.93 0.51 0.35 0.03 0.03 0.02	< 0.01 < 0.01 0.01 0.02 0.01 0.01 0.02 0.03 0.02	0.09 0.11 0.2 0.23 0.14 0.1 0.03 0.04 0.03	< 0.01 < 0.01 0.01 0.02 0.01 0.01 0.03 0.05 0.06	RA-2514/07 0037-07
Spain, 2007 Brenes (Rondo)	2	0.25	0.0625	400	-0 0 3 7 10 14 7 10 14	plant seed 	0.3 3.4 2.2 0.45 0.39 0.24 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.04 0.04 0.07 0.05 0.06 0.05 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 0.02 0.02	RA-2514/07 0557-07

Peas (pods and succulent = immature seeds)

Results from supervised trials from USA on snow peas (with pods) were provided to the Meeting. In these trials, two applications of 0.22–0.24 kg ai/ha (SC 500 formulation) were applied to snow peas, 5–7 days apart as foliar sprays using CO_2 or pressurised plot sprayers with hand-held spray booms or a tractor-mounted boom sprayer to apply 133–176 litres of spray mix/ha. Plot sizes in these trials ranged from 77–186 square metres.

Duplicate samples of at least 1 kg pods (with seeds) were taken from each plot, frozen within 30 minutes of sampling, held in frozen storage for up to 538 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 148 Fluopyram residues in snow peas (pods and succulent seeds) from supervised trials in USA involving two foliar applications (500 SC formulations)

PEA (WITH POD) Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Seymour, IL (Snowflake)	2	0.261 0.267	0.196 0.194	133 134	0	1.086, 1.197				RAGMP067 GM309-06HA
USA, 2006 Arkansaw, WI (Oregon Sugar Pod II)	2	0.249 0.251	0.143 0.142	174 176	0	1.318, 1.162				RAGMP067 GM310-06HA
USA, 2006 Jerome, ID (Ho Lan Dow Snow Pea)	2	0.256 0.249	0.175 0.16	146 156	0	0.756, 0.812				RAGMP067 GM311-06HA

Pulses

Beans (dry)

Results from supervised trials from USA on dry, shelled beans were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to beans, 5–8 days apart as foliar sprays using knapsack or pressurised sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 91–183 litres of spray mix/ha. Plot sizes in these trials ranged from 84–372 square metres. In each trial, one plot was last treated just before flowering (for sampling of forage) and a second plot was last treated after the end of flowering (for sampling of hay and seed). Hay and seeds from this second plot were allowed to dry to commercial dryness before sampling (0–18 days after cutting).

Duplicate samples of at least 1 kg seeds were taken from each plot, frozen within 2 hours of sampling, held in frozen storage for up to 694 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 149 Fluopyram residues in lima beans (dry) from supervised trials in USA involving two foliar applications (500 SC formulations)

BEANS (DRY) Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Earlham, IA (Maverick Pinto Beans)	2	0.25 0.253	0.197 0.232	127 109	14	0.012, 0.016				RAGMP069 GM325-06HA
USA, 2006 Springfield, NE (Pinto)	2	0.249 0.25	0.192 0.195	130 128	13+1 [a]	0.031, 0.022				RAGMP069 GM326-06HA
USA, 2006 Sabin, MN (Navigator)	2	0.257 0.25	0.161 0.147	160 170	14	0.012, 0.0098				RAGMP069 GM327-06HA
USA, 2006 Velva, ND (Maverick)	2	0.251 0.249	0.209 0.204	120 122	14	0.0066, 0.0071				RAGMP069 GM328-06HA
USA, 2006 Levelland, TX (Vision)	2	0.249 0.249	0.179 0.179	139 139	13	0.059, 0.076				RAGMP069 GM329-06HA 81% DM
USA, 2006 Jerome, ID (410 Pintos)	2	0.25 0.244	0.157 0.157	159 155	14+17[a]	0.0073, 0.0053				RAGMP069 GM330-06HA
USA, 2007 Fresno, CA (Lima Beans)	2	0.248 0.25	0.212 0.212	117 118	0+14 [a]	0.049, 0.055				RAGMP069 GM331-06HA
USA, 2006 Rupert, ID (Bill Z)	2	0.251 0.25	0.27 0.275	93 91	14+10 [a]	0.0049, 0.0067				RAGMP069 GM332-06HA
USA, 2006 Seymour, IL (Sanilac navy beans)	2	0.25 0.252	0.137 0.138	182 183	0 7 14 17 22	0.036, 0.041 0.015, 0.013 0.018, 0.011 0.0081, 0.011 0.024, 0.01				RAGMP069 GM324-06DA

[a] Drying interval between cutting and sampling

Peas (dry)

Results from supervised trials from USA on dry, shelled peas were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to peas, 5–7 days apart as foliar sprays using CO_2 plot or knapsack sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 115–190 litres of spray mix/ha. Plot sizes in these trials ranged from 24–74 square metres. In each trial, one plot (for vine and hay sampling) was last treated between flowering and when the first pods had reached their final length (BBCH 70) and a second plot (for seed sampling) was last treated between the end of flowering and early ripening (BBCH 80). Seeds from these second plots were allowed to dry to commercial dryness before sampling (0–6 days after cutting).

Duplicate samples of at least 1 kg seeds were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 624 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 150 Fluopyram residues in peas (dry) from supervised trials in USA involving two foliar applications (500 SC formulations)

PEAS (DRY) Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Parkdale, OR (Green Arrow)	2	0.251 0.258	0.173 0.151	145 171	14+6[a]	0.037, 0.03				RAGMP069 GM334-06HA
USA, 2006 Hermiston, OR (Majorettes)	2	0.25 0.244	0.134 0.13	186 188	14	0.057, 0.06				RAGMP069 GM335-06HA
USA, 2006 Payette, ID (Austrian winter pea)	2	0.254 0.247	0.134 0.135	190 183	14+5[a]	0.353, 0.345				RAGMP069 GM336-06HA 91% DM
USA, 2006 Madras, OR (Maples)	2	0.251 0.249	0.216 0.217	116 115	14	0.106, 0.214				RAGMP069 GM337-06HA
USA, 2006 Ephrata, WA (Cruiser)	2	0.253 0.252	0.181 0.18	140 140	0 7 14 18 24	0.032, 0.04 0.023, 0.037 0.044, 0.039 0.049, 0.03 0.033, 0.018				RAGMP069 GM333-06DA

[a] Drying interval between cutting and sampling

Soya bean (dry)

Results from supervised trials from USA on soya beans were provided to the Meeting. In these trials, two applications of 0.24–0.27 kg ai/ha (SC 500 formulation) were applied to soya beans, 5–7 days apart (14 days in one plot) as foliar sprays using knapsack or pressurised sprayers with hand-held spray booms, tractor-mounted or motorised boom sprayers to apply 113–197 litres of spray mix/ha. Plot sizes in these trials ranged from 62–298 square metres. In each trial, one plot was treated over the flowering period (for sampling of forage and hay) and a second plot was treated when pods were close to maturity (for sampling of seed). Hay from the first plot was allowed to dry to commercial dryness before sampling, 0–6 days after cutting (22 days in one plot).

Duplicate samples of at least 1 kg seeds were taken from at least 12 areas within each plot, frozen within 4 hours of sampling, held in frozen storage for up to 518 days before measuring dry matter content and analysing for fluopyram using LC/MS/MS Method GM-001-P07-01 (with a reported LOQ of 0.01 mg/kg).

Table 151 Fluopyram residues in soya bean (dry) from supervised trials in USA involving foliar applications (500 SC formulations)

SOYA BEAN (DRY) Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Tifton, GA (DP 4546 RR)	2	0.25 0.25	0.177 0.174	144 141	14	0.03, 0.05				RAGMP039 GM062-06HA
USA, 2006 Molino, FL (Pioneer 97B52)	2	0.238 0.248	0.208 0.183	113 119	14	< 0.01, < 0.01				RAGMP039 GM063-06HA
USA, 2006 Leland, MS (Asgrow STS 4404)	2	0.248 0.26	0.206 0.211	124 126	12	< 0.01, < 0.01				RAGMP039 GM064-06HA
USA, 2006 Proctor, AR (AG4403RR)	2	0.249 0.249	0.169 0.168	147 147	14	0.01, 0.02				RAGMP039 GM065-06HA
USA, 2006 Cheneyville, LA (DP 5634 RR)	2	0.26 0.256	0.154 0.152	169 166	14	0.06, 0.06				RAGMP039 GM066-06HA
USA, 2006 Springfield, NE (NKS28 G1)	2	0.249 0.251	0.195 0.198	129 129	12	< 0.01, < 0.01				RAGMP039 GM069-06HA
USA, 2006 Earlham, IA (S2783-4)	2	0.252 0.256	0.225 0.197	126 114	14	0.04, 0.04				RAGMP039 GM070-06HA
USA, 2006 Sabin, MN (RG 200)	2	0.25 0.269	0.137 0.161	167 197	13	< 0.01, < 0.01				RAGMP039 GM071-06HA
USA, 2006 Percival, IA (Sucrosco 935-01RNX)	2	0.25 0.251	0.192 0.195	128 131	12	< 0.01, < 0.01				RAGMP039 GM072-06HA
USA, 2006 Stilwell, KS (NSQ49-Q9)	2	0.25 0.248	0.178 0.181	138 139	14	< 0.01, < 0.01				RAGMP039 GM073-06HA
USA, 2006 Stilwell, KS (NKS49-Q9)	2	0.251 0.254	0.179 0.18	140 142	13	< 0.01, < 0.01				RAGMP039 GM074-06HA
USA, 2006 Campbell, MN (Dekalb 009-51 RR)	2	0.25 0.25	0.134 0.134	187 187	14	< 0.01, < 0.01				RAGMP039 GM075-06HA
USA, 2006 Bernie, MO (Hutchison)	2	0.251 0.251	0.17 0.169	148 148	13	0.14, 0.18				RAGMP039 GM076-06HA
USA, 2006 Sheridan, IN (RT3253)	2	0.248 0.249	0.193 0.211	137 129	14	0.01, < 0.01				RAGMP039 GM077-06HA
USA, 2006 Geneva, MN (Pioneer 91M90)	2	0.251 0.252	0.152 0.158	162 166	14	< 0.01, < 0.01				RAGMP039 GM078-06HA
USA, 2006 Richland, IA (Pioneer 93B82)	2	0.256 0.252	0.147 0.149	174 172	17	0.02, 0.02				RAGMP039 GM079-06HA
USA, 2006 Washington C.H., OH (SC 9384RR)	2	0.25 0.253	0.17 0.171	147 149	12	< 0.01, < 0.01				RAGMP039 GM080-06HA

SOYA BEAN (DRY) Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Arkansaw, WI (91M91)	2	0.252 0.251	0.143 0.143	177 176	14	0.01, < 0.01				RAGMP039 GM081-06HA 14d spray interval
USA, 2006 York, NE (Garst 2834 RR)	2	0.251 0.25	0.134 0.138	188 186	0 6 13 19 28	0.09, 0.09 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01				RAGMP039 GM067-06DA
USA, 2006 Seymour, IL (Agripro 3212 RR/N)	2	0.256 0.253	0.141 0.177	185 180	0 7 14 21 28	0.16, 0.19 0.05, 0.06 0.01, 0.01 < 0.01, < 0.01 < 0.01, < 0.01				RAGMP039 GM068-06DA

Bean fodder and forage

Results from supervised trials from USA on bean forage and hay were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to beans, 5–8 days apart as foliar sprays using knapsack or pressurised sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 91–183 L of spray mix/ha. Plot sizes in these trials ranged from 84–372 square metres. In each trial, one plot was last treated just before flowering (for sampling of forage) and a second plot was last treated after the end of flowering (for sampling of hay and seed). Hay and seeds from this second plot were allowed to dry to commercial dryness before sampling (0–18 days after cutting).

Duplicate samples of at least 1 kg forage (green plant material) and at least 0.45 kg hay were taken from each plot, frozen within 2 hours of sampling, held in frozen storage for up to 624 days (forage) or 567 days (hay) before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 152 Fluopyram residues in bean forage and fodder (hay) from supervised trials in USA involving two foliar applications (500 SC formulations)

BEAN FORAGE / HAY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Earlham, IA (Maverick Pinto Beans)	2	0.248 0.251	0.184 0.187	135 134	0	forage	12.91, 16.32				RAGMP069 GM325-06HA
USA, 2006 Springfield, NE (Pinto)	2	0.25 0.253	0.195 0.199	128 127	0	forage	15.45, 12.2				RAGMP069 GM326-06HA
USA, 2006 Sabin, MN (Navigator)	2	0.248 0.252	0.152 0.153	163 165	0	forage	12.27, 13.4				RAGMP069 GM327-06HA
USA, 2006 Velva, ND (Maverick)	2	0.252 0.25	0.205 0.203	123 123	0	forage	25.43, 25.18				RAGMP069 GM328-06HA 16% DM

BEAN FORAGE / HAY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Levelland, TX (Vision)	2	0.25 0.252	0.179 0.177	140 142	0	forage	14.14, 13.51				RAGMP069 GM329-06HA
USA, 2006 Jerome, ID (Pintos)	2	0.25 0.254	0.158 0.16	158 159	0	forage	13.19, 13.2				RAGMP069 GM330-06HA
USA, 2007 Fresno, CA (Lima Beans)	2	0.251 0.251	0.132 0.132	190 190	0	forage	20.09, 22.52				RAGMP069 GM331-06HA
USA, 2006 Rupert, ID (Bill Z)	2	0.252 0.252	0.311 0.3	81 84	0	forage	9.79, 10.41				RAGMP069 GM332-06HA
USA, 2006 Seymour, IL (Sanilac navy beans)	2	0.261 0.253	0.199 0.178	131 142	0 3 7 10 14	forage forage forage forage forage	13.13, 13.66 5.79, 5 3.46, 3.32 1.56, 1.57 1.18, 1.03				RAGMP069 GM324-06DA
USA, 2006 Earlham, IO (Maverick Pinto Beans)	2	0.25 0.253	0.197 0.232	127 109	0+3[a]	hay	11.46, 21.86				RAGMP069 GM325-06HA
USA, 2006 Springfield, NE (Pinto)	2	0.249 0.25	0.192 0.195	130 128	0+3 [a]	hay	37.71, 15.39				RAGMP069 GM326-06HA 61% DM
USA, 2006 Sabin, MN (Navigator)	2	0.257 0.25	0.161 0.147	160 170	0+18 [a]	hay	26.45, 26.65				RAGMP069 GM327-06HA
USA, 2006 Velva, ND (Maverick)	2	0.251 0.249	0.209 0.204	120 122	0+3 [a]	hay	7.94, 9.83				RAGMP069 GM328-06HA
USA, 2006 Levelland, TX (Vision)	2	0.249 0.249	0.179 0.179	139 139	0+6 [a]	hay	11.33, 13.29				RAGMP069 GM329-06HA
USA, 2006 Jerome, ID (410 Pintos)	2	0.25 0.244	0.157 0.157	159 155	0+13 [a]	hay	30.13, 27.26				RAGMP069 GM330-06HA
USA, 2007 Fresno, CA (Lima Beans)	2	0.248 0.25	0.212 0.212	117 118	0	hay	15.64, 22.51				RAGMP069 GM331-06HA
USA, 2006 Rupert, ID (Bill Z)	2	0.251 0.25	0.27 0.275	93 91	0+13 [a]	hay	16.74, 29.64				RAGMP069 GM332-06HA
USA, 2006 Seymour, IL (Sanilac navy beans)	2	0.25 0.252	0.137 0.137	182 183	0 3 7 10 14	hay hay hay hay hay	3.91, 4.25 2.78, 2.88 0.77, 0.591 0.598, 0.588 0.525, 0.558				RAGMP069 GM324-06DA

[a] Drying interval between cutting and sampling

Pea vines and hay

Results from supervised trials from USA on peas forage and hay were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to peas, 5–7 days apart as foliar sprays using CO_2 plot or knapsack sprayers with hand-held spray booms or

tractor-mounted boom sprayers to apply 116–190 L of spray mix/ha. Plot sizes in these trials ranged from 24–74 square metres. In each trial, one plot (for vine and hay sampling) was last treated between flowering and when the first pods had reached their final length (BBCH 70) and a second plot (for seed sampling) was last treated between the end of flowering and early ripening (BBCH 80). Hay from the first plots was allowed to dry to commercial dryness before sampling (0–10 days after cutting).

Duplicate samples of at least 1 kg vines and at least 0.45 kg hay were taken from each plot,, frozen within 4 hours of sampling, held in frozen storage for up to 397 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 153 Fluopyram residues in pea vines and hay from supervised trials in USA involving two foliar applications (500 SC formulations)

PEA VINES / HAY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Parkdale, OR (Green Arrow)	2	0.252 0.251	0.166 0.156	152 161	0 0+2 [a]	vines hay	5.72, 6.04 29.05, 31.24				RAGMP069 GM334-06HA
USA, 2006 Hermiston, OR (Majorettes)	2	0.247 0.253	0.14 0.135	177 188	0 0+7 [a]	vines hay	5.1, 4.01 20.3, 15.36				RAGMP069 GM335-06HA
USA, 2006 Payette, ID (Austrian winter pea)	2	0.253 0.247	0.133 0.134	190 185	0 0+4 [a]	vines hay	9.08, 11.15 49.44, 46.9				RAGMP069 GM336-06HA 16% vine DM 83% hay DM
USA, 2006 Madras, OR (Maples)	2	0.253 0.251	0.216 0.216	117 116	0 0+10 [a]	vines hay	5.4, 5.85 15.42, 18.16				RAGMP069 GM337-06HA
USA, 2006 Ephrata, WA (Cruiser)	2	0.252 0.252	0.18 0.179	140 141	0 0+3 [a] 3 3+4 [a] 7 7+3 [a] 10 10+4 [a] 14 14+3 [a]	vines hay vines hay vines hay vines hay vines hay	2.66, 2.71 14.95, 13.18 0.764, 0.838 3.15, 2.95 0.589, 0.468 2.36, 1.59 0.461, 0.358 1.37, 1.59 0.32, 0.204 0.833, 0.85				RAGMP069 GM333-06DA

[a] Drying interval between cutting and sampling

Soya bean forage (green) and fodder

Results from supervised trials from USA on soya beans were provided to the Meeting. In these trials, two applications of 0.24–0.27 kg ai/ha (SC 500 formulation) were applied to soya beans, 5–7 days apart (14 days in one plot) as foliar sprays using knapsack or pressurised sprayers with hand-held spray booms, tractor-mounted or motorised boom sprayers to apply 113–197 litres of spray mix/ha. Plot sizes in these trials ranged from 62–298 square metres. In each trial, one plot was treated over the flowering period (for sampling of forage and hay) and a second plot was treated when pods were close to maturity (for sampling of seed). Hay from the first plot was allowed to dry to commercial dryness before sampling, 0-6 days after cutting (22 days in one plot).

Duplicate samples of at least 1 kg forage and 0.3 kg hay were taken from at least 12 areas within each plot, frozen within 4 hours of sampling, held in frozen storage for up to 585 days (hay) and 581 days (forage) before measuring dry matter content and analysing for fluopyram using LC/MS/MS Method GM-001-P07-01 (with a reported LOQ of 0.01 mg/kg).

Table 154 Fluopyram residues in soya bean forage and hay from supervised trials in USA involving foliar applications (500 SC formulations)

SOYA BEAN VINES / HAY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Tifton, GA (DP 4546 RR)	2	0.25 0.25	0.156 0.211	144 160	7 7+6 [a]	forage (28%DM) hay (70%DM)	2.37, 1.42 6.17, 6.22				RAGMP039 GM062-06HA
USA, 2006 Molino, FL (Pioneer 97B52)	2	0.251 0.243	0.188 0.2	137 129	6 6+6 [a]	forage (21%DM) hay (91%DM)	2.34, 2.8 9.71, 8.44				RAGMP039 GM063-06HA
USA, 2006 Leland, MS (Asgrow STS 4404)	2	0.258 0.252	0.2 0.169	122 126	7 7+3 [a]	forage (23%DM) hay (63%DM)	1.39, 2.88 3.51, 3.64				RAGMP039 GM064-06HA
USA, 2006 Proctor, AR (AG4403RR)	2	0.25 0.251	0.17 0.154	149 148	7 7+1 [a]	forage (22%DM) hay (81%DM)	4.98, 6.19 10.47, 11.69				RAGMP039 GM065-06HA
USA, 2006 Cheneyville, LA (DP 5634 RR)	2	0.252 0.247	0.152 0.134	166 163	7 7+3 [a]	forage (27%DM) hay (81%DM)	4.05, 3.33 10.79, 11.04				RAGMP039 GM066-06HA
USA, 2006 Springfield, NE (NKS28 G1)	2	0.253 0.25	0.195 0.2	128 128	6 6+2 [a]	forage (16%DM) hay (46%DM)	1.21, 1.08 3, 3.3				RAGMP039 GM069-06HA
USA, 2006 Earlham, IA (S2783-4)	2	0.254 0.259	0.216 0.15	129 120	7 7+5 [a]	forage (19%DM) hay (56%DM)	1.06, 1.23 3.25, 2.69				RAGMP039 GM070-06HA
USA, 2006 Sabin, MN (RG 200)	2	0.257 0.239	0.147 0.195	160 163	7 7+22 [a]	forage (63%DM) hay (87%DM)	0.32, 0.4 1.21, 2.37				RAGMP039 GM071-06HA
USA, 2006 Percival, IA (Sucrosco 935-01RNX)	2	0.251 0.248	0.195 0.181	129 127	6 6+1 [a]	forage (21%DM) hay (44%DM)	2.52, 2.78 7.83, 4.02				RAGMP039 GM072-06HA
USA, 2006 Stilwell, KS (NSQ49-Q9)	2	0.25 0.248	0.18 0.179	138 138	7 7	forage (19%DM) hay (77%DM)	5.56, 5.85 19.5, 20.9				RAGMP039 GM073-06HA
USA, 2006 Stilwell, KS (NKS49-Q9)	2	0.252 0.248	0.18 0.134	140 138	7 7	forage (19%DM) hay (71%DM)	2.54, 2.92 9.91, 9.58				RAGMP039 GM074-06HA
USA, 2006 Campbell, MN (Dekalb 009-51 RR)	2	0.251 0.251	0.134 0.17	188 188	7 7+2 [a]	forage (19%DM) hay (58%DM)	3.29, 3.21 5.96, 8.91				RAGMP039 GM075-06HA
USA, 2006 Bernie, MO (Hutchison)	2	0.252 0.25	0.168 0.181	149 149	7 7+2 [a]	forage (19%DM) hay (61%DM)	2.64, 1.96 6.52, 6.44				RAGMP039 GM076-06HA
USA, 2006 Sheridan, IN (RT3253)	2	0.251 0.252	0.208 0.155	119 121	7 7+2 [a]	forage (19%DM) hay (39%DM)	1.21, 1.21 2.33, 2.11				RAGMP039 GM077-06HA

SOYA BEAN VINES / HAY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Geneva, MN (Pioneer 91M90)	2	0.256 0.252	0.158 0.147	162 160	7 7+4 [a]	forage (15%DM) hay (49%DM)	2.4, 2.23 6.93, 5.23				RAGMP039 GM078-06HA
USA, 2006 Richland, IA (Pioneer 93B82)	2	0.249 0.25	0.168 0.17	167 149	7 7+2 [a]	forage (16%DM) hay (55%DM)	1.18, 1.05 3.32, 3.43				RAGMP039 GM079-06HA
USA, 2006 Washington, OH (SC 9384RR)	2	0.25 0.254	0.173 0.142	146 147	6 6+3 [a]	forage (20%DM) hay (39%DM)	3.09, 3.6 5.46, 5.83				RAGMP039 GM080-06HA
USA, 2006 Arkansaw, WI (91M91)	2	0.251 0.251	0.143 7	176 176	7 7+3 [a]	forage (19%DM) hay (77%DM)	2.98, 3.54 11.48, 15.74				RAGMP039 GM081-06HA
USA, 2006 York, NE (Garst 2834 RR)	2	0.249 0.256	0.138 0.138	180 185	0 0+3 [a] 3 3+5 [a] 7 7+3 [a] 10 10+4 [a] 14 14+1 [a]	forage (16%DM) hay (72%DM) forage (16%DM) hay (66%DM) forage (19%DM) hay (58%DM) forage (17%DM) hay (59%DM) forage (18%DM) hay (45%DM)	14.98, 12.69 66.95, 67.88 11.07, 9.75 43.3, 34.2 4.62, 4.65 17.53, 14.44 2.05, 1.73 7.11, 7.12 1.32, 1.39 3.75, 3.77				RAGMP039 GM067-06DA
USA, 2006 Seymour, IL (Agripro 3212 RR/N)	2	0.259 0.248	0.191 0.193	146 130	0 0+4 [a] 3 3+5 [a] 7 7+4 [a] 9 9+9 [a] 14 14+5 [a]	forage (20%DM) hay (56%DM) forage (17%DM) hay (ND%DM) forage (18%DM) hay (56%DM) forage (19%DM) hay (56%DM) forage (19%DM) hay (66%DM)	17.86, 18.42 59.28, 61.8 2.08, 2.02 7.71, 9.35 1.29, 1.37 3.89, 5.37 1.15, 1.14 4.06, 3.68 0.92, 0.7 3, 2.52				RAGMP039 GM068-06DA c=0.014 mg/kg (day 7 forage)

[a] Drying interval between cutting and sampling

Root and tuber vegetables

Carrots

Results from supervised trials from USA on carrots were provided to the Meeting. In these trials, two applications of 0.24–0.27 kg ai/ha (SC 500 formulation) in 125–187 litres water were applied as foliar sprays, 5–7 days apart, using knapsack or plot sprayers with hand-held mini-booms (3–6 nozzles) or tractor-mounted 4m boom. Plot sizes in these trials ranged from 31–150 square metres.

Duplicate samples of carrot roots (at least 24 unitsequalling 2 kg) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 640 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 155 Fluopyram residues in carrot roots from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations).

CARROT Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Oviedo, FL (Danvers 126 Improved)	2	0.245 0.242	0.14 0.139	175 174	0	0.049, 0.074				RAGMP066 GM290-06HA
USA, 2006 Seymore, IL (Scarlet Nantes)	2	0.258 0.259	0.174 0.173	148 150	0	0.052, 0.036				RAGMP066 GM291-06HA
USA, 2006 Uvalde, TX (Danvers)	2	0.249 0.25	0.199 0.175	125 143	0	0.016, 0.018				RAGMP066 GM292-06HA
USA, 2006 Madera, CA (Danvers 126)	2	0.251 0.253	0.147 0.147	171 172	0 3 7 10 14	0.029, 0.055 0.05, 0.076 0.076, 0.048 0.058, 0.085 0.083, 0.079				RAGMP066 GM293-06DA
USA, 2006 Sanger, CA (Baby New F1)	2	0.252 0.25	0.205 0.207	123 121	0	0.076, 0.1				RAGMP066 GM294-06HA
USA, 2006 San Ardo, CA (Maverick)	2	0.252 0.265	0.14 0.142	180 187	0	0.065, 0.062				RAGMP066 GM295-06HA

Results from supervised trials from Europe on carrots were provided to the Meeting. In these trials, 3 applications of an SC 400 formulation of fluopyram (co-formulated with tebuconazole) were applied at 13–15 day intervals as foliar sprays using knapsack sprayers with hand-held mini booms (3–9 hollow cone or flat fan nozzles), applying 0.15 kg ai/ha in 300–800 litres water/ha. Plot sizes in these trials ranged from 15–80 square metres.

Unreplicated samples of at least 24 carrot roots were taken from each plot, some being lightly rinsed or wiped to remove soil, frozen within 24 hours of sampling and stored at -18 °C or below for up to 367 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQs of 0.01 mg/kg for each analyte).

Table 156 Residues in carrot roots from supervised field trials in France, Germany, Greece, Italy, Netherlands, Portugal, Spain, and United Kingdom, involving three foliar applications of fluopyram (400 SC formulations)

CARROT Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
France, 2006 Cergy (Nandrin F1)	3	0.15	0.03	500	-0 0 7 14 21 28	0.04 0.11 0.11 0.09 0.12 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2579/06 0358-06
Germany, 2006 Meckenbeuren (Eva)	3	0.15	0.03	500	0 14	0.05 0.08	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	RA-2579/06 0359-06

CARROT Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Netherlands, 2006 Zwaagdijk-Oost (Bangor)	3	0.15	0.0188	800	-0 0 7 14 21 28	0.05 0.06 0.11 0.12 0.07 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2579/06 0480-06
United Kingdom, 2006 Thetford (Narobie)	3	0.15	0.05	300	-0 0 7 13 21 28	0.03 0.02 0.04 0.04 0.04 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2579/06 0481-06
Germany, 2006 Brüggen (Mokum F1)	3	0.15	0.05	300	-0 0 7 14 21 28	0.14 0.18 0.22 0.18 0.16 0.19	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2579/06 0482-06
France, 2007 Cergy (Chambord)	3	0.15	0.05	300	-0 0 7 14 21 28	0.11 0.11 0.13 0.13 0.1 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2523/07 0062-07
Germany, 2007 Langenfeld-Reusrath (Nantes Fancy)	3	0.15	0.05	300	-0 0 7 14 21 28	0.07 0.07 0.1 0.07 0.09 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2523/07 0577-07
United Kingdom, 2007 Norfolk (Niarobi)	3	0.15	0.05	300	-0 0 7 14 21 27	0.02 0.02 0.02 0.03 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2523/07 0578-07
Germany, 2007 Ruchheim (Merida)	3	0.15	0.025	600	-0 0 7 14 21 28	0.09 0.09 0.12 0.1 0.1 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2523/07 0579-07
France, 2006 Drace (Damco)	3	0.15	0.025	600	-0 0 7 14 21 28	0.05 0.07 0.05 0.07 0.05 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2580/06 0360-06
France, 2006 Velleron (Presto)	3	0.15	0.0376	400	0 14	0.07 0.08	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	RA-2580/06 0361-06
Spain, 2006 Alginet (Ceres)	3	0.15	0.0376	400	-0 0 7 14 21 28	0.13 0.14 0.18 0.12 0.13 0.2	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2580/06 0483-06

CARROT Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Italy, 2006 Maccarese Fiumicino (Napoli)	3	0.15	0.03	500	-0 0 7 14 21 28	0.03 0.03 0.05 0.08 0.04 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2580/06 0485-06
Portugal, 2006 Tapada- Almeirim (Maestro)	3	0.15	0.0376	400	-0 0 7 14 21 28	0.1 0.09 0.14 0.12 0.12 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2580/06 0486-06
Spain, 2007 Lebrija Sevilla (Saturno F1)	3	0.15	0.0376	400	-0 0 7 14 21 28	0.07 0.07 0.07 0.06 0.08 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2524/07 0066-07
France, 2007 Scorbe Clairvaux (Chambor F1)	3	0.15	0.025	600	-0 0 7 14 21 28	0.05 0.06 0.08 0.08 0.09 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2524/07 0580-07
Italy, 2007 Maccarese - Fiumicino (Napoli F1)	3	0.15	0.03	500	-0 0 7 14 21 28	0.04 0.03 0.04 0.05 0.04 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2524/07 0581-07
Greece, 2007 Aronas-Katerini-Pieria (Mirim)	3	0.15	0.03	500	-0 0 7 14 21 29	0.04 0.04 0.06 0.06 0.04 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2524/07 0582-07

Potatoes

Results from supervised trials from USA on potatoes were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied 3–5 days apart as foliar sprays using knapsack or CO_2 plot sprayers with hand-held mini-booms or tractor-mounted side booms (8–9 nozzles). Plot sizes in these trials ranged from 28–233 square metres.

Duplicate samples (12–24 tubers) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 623 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg)

Table 157 Fluopyram residues in potato tubers from supervised outdoor trials in USA involving two foliar applications of fluopyram (500 SC formulations)

POTATO Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Germansville, Pennsylvania (Katahdin)	2	0.253 0.25	0.141 0.14	180 178	7	< 0.01, < 0.01				RAGMP065 GM274-06HA
USA, 2006 North Rose, New York (Sebago)	2	0.253 0.251	0.15 0.149	169 168	7	< 0.01, < 0.01				RAGMP065 GM275-06HA
USA, 2006 Suffolk, Virginia (Kennebec)	2	0.251 0.255	0.254 0.271	99 94	7	0.017, 0.016				RAGMP065 GM276-06HA
USA, 2006 Molino, Florida (Red La Soda)	2	0.247 0.245	0.204 0.233	121 105	7	< 0.01, < 0.01				RAGMP065 GM277-06HA
USA, 2006 Springfield, Nebraska (Norland Red)	2	0.249 0.251	0.195 0.192	128 131	6	< 0.01, < 0.01				RAGMP065 GM279-06HA
USA, 2006 Seymour, Illinois (Red Pontiac)	2	0.244 0.252	0.183 0.185	133 136	7	< 0.01, < 0.01				RAGMP065 GM280-06HA
USA, 2006 Sabin, Minnesota (Red Norland)	2	0.263 0.236	0.154 0.151	171 156	6	< 0.01, < 0.01				RAGMP065 GM281-06HA
USA, 2006 Kimberly, Idaho (Russet Burbank)	2	0.254 0.247	0.163 0.159	156 155	7	< 0.01, < 0.01				RAGMP065 GM282-06HA
USA, 2006 Fresno, California (Red La Soda)	2	0.244 0.245	0.145 0.145	168 169	7	< 0.01, < 0.01				RAGMP065 GM283-06HA
USA, 2006 Rupert, Idaho (Russet Burbank)	2	0.25 0.251	0.269 0.256	93 98	6	< 0.01, < 0.01				RAGMP065 GM285-06HA
USA, 2006 Ephrata, Washington (Ranger Russet)	2	0.249 0.25	0.178 0.179	140 140	6	< 0.01, < 0.01				RAGMP065 GM286-06HA
USA, 2006 Hermiston, Oregon (Russet Burbank)	2	0.247 0.249	0.147 0.152	168 164	7	< 0.01, < 0.01				RAGMP065 GM287-06HA
USA, 2006 Payette, Idaho (Russet Burbank)	2	0.245 0.251	0.14 0.14	175 179	7	< 0.01, < 0.01				RAGMP065 GM288-06HA
USA, 2006 Madras, Oregon (Russet Burbank)	2	0.259 0.25	0.216 0.208	120 120	7	< 0.01, < 0.01				RAGMP065 GM289-06HA
USA, 2006 Stilwell, Kansas (Kennebec)	2	0.246 0.248	0.185 0.18	133 138	0 3 7 14 21	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 0.012, 0.013				RAGMP065 GM278-06DA

POTATO Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Jerome, Idaho (Russet Burbank)	2	0.25 0.25	0.143 0.14	175 178	0 3 7 14 21	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01				RAGMP065 GM284-06DA

Radish

Results from supervised trials from USA on radish roots and tops were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) in 103–187 litres water were applied as foliar sprays, 5–7 days apart, using knapsack or plot sprayers with hand-held mini-booms (3–6 nozzles). Plot sizes in these trials ranged from 45–134 square metres.

Duplicate samples of radish roots and radish tops (at least 24 units or 2 kg) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 629 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 158 Fluopyram residues in radish roots and tops from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

RADISH Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			parent	PAA	BZM	PCA	
USA, 2006 Germansville, PA (Crimson Giant)	2	0.261 0.249	0.14 0.14	187 178	0	roots tops	0.086, 0.109 9.57, 11.48				RAGMP066 GM296-06HA
USA, 2006 Monticello, FL (Sparkler White Tip)	2	0.252 0.252	0.2 0.214	126 118	0	roots tops	0.062, 0.075 16.75, 18.7				RAGMP066 GM298-06HA
USA, 2006 Stilwell, KS (Cherry Belle)	2	0.254 0.249	0.179 0.183	142 136	0	roots tops	0.123, 0.108 21.45, 19.69				RAGMP066 GM299-06HA
USA, 2006 Fresno, CA (Champion)	2	0.251 0.25	0.143 0.142	175 176	0	roots tops	0.051, 0.042 24.31, 26.3				RAGMP066 GM300-06HA
USA, 2006 Molino, FL (French Breakfast)	2	0.25 0.245	0.243 0.243	103 101	0 0 3 3 7 7 10 10 14 14	roots tops roots tops roots tops roots tops roots tops	0.148, 0.115 16.09, 16.41 0.172, 0.148 10.1, 16.47 0.123, 0.15 11.78, 11.02 0.062, 0.066 6.59, 7.71 0.029, 0.041 5.13, 5.29				RAGMP066 GM297-06DA

Sugar beet (roots and tops)

Results from supervised trials from USA on sugar beet roots and tops were provided to the Meeting. In these trials, two applications of 0.22–0.23 kg ai/ha (SC 500 formulation) were applied 5–7 days apart as foliar sprays using knapsack or CO_2 plot sprayers and hand-held 4-nozzle mini-booms or tractor-mounted boom sprayers. Plot sizes in these trials ranged from 56–124 square metres.

Duplicate samples (12 sugar beet roots and 1 kg sugar beet tops) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 573 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg). Residues were reported on a fresh weight basis.

Table 159 Fluopyram residues in sugar beet roots and tops from supervised outdoor trials in USA involving two foliar applications of fluopyram (500 SC formulations)

SUGAR BEET Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		commodity	parent	PAA	BZM	PCA	
USA, 2006 Seymour, Illinois (VDH 66283)	2	0.253 0.258	0.138 0.143	183 181	7 7	roots tops (12%DM)	0.024, 0.024 4.72, 4.65				RAGMP053 GM166-06HA
USA, 2006 Springfield, Nebraska (VDH 66556)	2	0.253 0.248	0.192 0.192	132 129	7 7	roots tops (19%DM)	0.034, 0.045 3.83, 2.6				RAGMP053 GM167-06HA
USA, 2006 Sabin, Minnesota (Beta 4797R)	2	0.253 0.242	0.153 0.148	165 163	7 7	roots tops (12%DM)	0.025, 0.026 1.01, 0.761				RAGMP053 GM168-06HA
USA, 2006 Arkansaw, Wisconsin (VDH 55665)	2	0.254 0.252	0.143 0.142	178 177	7 7	roots tops (18%DM)	0.037, 0.033 0.768, 0.58				RAGMP053 GM169-06HA
USA, 2006 New Rockford, North Dakota (Beta)	2	0.252 0.248	0.197 0.197	128 126	7 7	roots tops (21%DM)	0.034, 0.021 1.73, 1.82				RAGMP053 GM170-06HA
USA, 2006 Levelland, Texas (Phoenix)	2	0.25 0.253	0.176 0.183	142 138	6 6	roots tops (15%DM)	0.039, 0.035 0.786, 0.703				RAGMP053 GM171-06HA
USA, 2006 Jerome, Idaho (Beta 4490 R)	2	0.244 0.249	0.153 0.152	159 164	5 5	roots tops (15%DM)	0.023, 0.021 0.273, 0.285				RAGMP053 GM172-06HA
USA, 2006 Porterville, California (Beta 4430R)	2	0.249 0.25	0.138 0.137	180 183	7 7	roots tops (20%DM)	0.048,0.0178 18.7, 14.3				RAGMP053 GM173-06HA
USA, 2006 Fresno, California (SSNB7R)	2	0.247 0.245	0.146 0.145	169 169	7 7	roots tops (15%DM)	0.013, 0.03 8.36, 10.45				RAGMP053 GM174-06HA
USA, 2006 Rupert, Idaho (2992 Rz)	2	0.251 0.249	0.314 0.28	80 89	7 7	roots tops (14%DM)	0.023, 0.015 0.301, 0.456				RAGMP053 GM175-06HA
USA, 2006 Hermiston, Oregon (BXX-22)	2	0.25 0.25	0.147 0.15	170 167	7 7	roots tops (14%DM)	0.041, 0.05 0.413, 0.289				RAGMP053 GM176-06HA
USA, 2006 Gardner, North Dakota (VDH 46519)	2	0.249 0.25	0.164 0.124	152 201	0 0 6 6 13 13 19 19 27 27	roots tops (16%DM) roots tops (19%DM) roots tops (19%DM) roots tops (19%DM) roots tops (20%DM)	0.079, 0.068 7.87, 11.1 0.013, 0.019 0.82, 0.56 0.015, 0.02 0.492, 0.452 0.01, 0.01 0.217, 0.133 0.01, 0.009 0.056, 0.042				RAGMP053 GM165-06DA

Turnip leaves or tops

Results from supervised trials from USA on turnips were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied 5–7 days apart as foliar sprays using knapsack or CO_2 plot sprayers and hand-held 4-nozzle mini-booms or tractor-mounted boom sprayers. Plot sizes in these trials ranged from 56–124 square metres.

Duplicate samples of turnip tops (at least 1 kg of leaves and stems) were taken from each plot, frozen within 1.5 hours of sampling, held in frozen storage for up to 293 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg). Residues were reported on a fresh weight basis.

Table 160 Fluopyram residues in turnip leaves and tops from supervised outdoor trials in USA involving two foliar applications of fluopyram (500 SC formulations)

TURNIP (TOPS) Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		commodity	parent	PAA	BZM	PCA	
USA, 2007 Chula, Georgia (Purple Top)	2	0.253 0.252	0.158 0.145	160 174	7	Tops (8%DM)	3.262, 3.646				RAGMP053 GM357-06HA
USA, 2007 Proctor Arkansas (Purple Top)	2	0.251 0.249	0.19 0.189	132 132	7	Tops (11%DM)	2.504, 5.127				RAGMP053 GM358-06HA
USA, 2007 Hudson, Kansas (Purple Top)	2	0.259 0.243	0.152 0.146	170 167	7	Tops (11%DM)	1.343, 1.390				RAGMP053 GM359-06HA
USA, 2007 Bernard, Texas (Purple Top)	2	0.247 0.252	0.189 0.185	131 136	7	Tops (14%DM)	1.871, 1.470				RAGMP053 GM360-06HA
USA, 2007 Suffolk, Virginia (Purple Top)	2	0.253 0.257	0.213 0.211	119 122	0 3 7 14 21	Tops (13%DM) Tops (14%DM) Tops (10%DM) Tops (11%DM) Tops (12%DM)	24.03, 31.03 1.874, 1.683 0.451, 0.539 0.272, 0.267 0.305, 0.292				RAGMP053 GM356-06DA c=0.03 mg/kg (day 7)

Stalk and stem vegetables

Artichoke, Globe

Results from supervised trials from Europe on Globe artichokes were provided to the Meeting. In these trials, 3 applications of fluopyram (SC 500) were applied 6–7 days apart as foliar sprays using knapsack sprayers with single solid or hollow-cone nozzles or mini-booms (3 flat-fan or solid cone nozzles), applying 0.1kg ai/ha in 800–1500 litres water/ha. Plot sizes in these trials ranged from 78–270 square metres.

Unreplicated flowerhead samples of 12–24 heads were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 194 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 (LOQs were 0.01 mg/kg for each analyte).

Table 161 Residues in Globe artichokes from supervised field trials in France, Germany, Italy, Netherlands, Spain and United Kingdom, involving 3 foliar applications of fluopyram (500 SC formulation)

ARTICHOKE, GLOBE Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
Italy, 2006 Stornarella (Violetto di Provenza)	3	0.1	0.01	1000	-0 0 4 7 15	0.1 0.44 0.3 0.18 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.02 0.02	RA-2602/06 0389-06
Spain, 2006 Albuixech (Blanca de Tudela)	3	0.1	0.01	1000	-0 0 3 7 14	0.15 0.43 0.23 0.16 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.01 0.02	RA-2602/06 0619-06
Spain, 2007 Albuixech (Blanca de Tudela)	3	0.1	0.01	1000	-0 0 3 7 14	0.2 0.47 0.32 0.21 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.02	RA-2516/07 0060-07
Italy, 2006 Cerignola (Violetto di Provenza)	3	0.1	0.0125	800	-0 0 3 7 14	0.15 0.35 0.14 0.05 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.05 0.05 0.07	RA-2516/07 0575-07

Results from supervised trials from USA on Globe artichokes were provided to the Meeting. In these trials, two applications of 0.24–0.25 kg ai/ha (SC 500 formulation) were applied as foliar sprays, 7 days apart as foliar sprays using knapsack or pressurised plot sprayers with hand lances or 2 m hand-held mini-booms. Plot sizes in these trials ranged from 44–93 square metres. Trifloxystrobin (500 SC) was included as a tank mix in these trials.

Duplicate samples (12 flowerheads/sample) were taken from each plot, frozen within 1 hour of sampling, held in frozen storage for up to 324 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 162 Fluopyram residues in Globe artichokes from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

ARTICHOKE, GLOBE Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Salinas, CA (Green globe)	2	0.249 0.246	0.23 0.225	108 109	0	1.59, 1.148				RAGMY003 GM145-07HA
USA, 2007 Castroville, CA (Green globe)	2	0.254 0.252	0.179 0.179	142 141	0	1.276, 1.259				RAGMY003 GM146-07HB
USA, 2007 Fresno, CA (Imperial)	2	0.251 0.25	0.215 0.214	117 117	0	0.983, 1.046				RAGMY003 GM147-07HA

Celery

Results from supervised trials from USA on outdoor celery were provided to the Meeting. In these trials, two foliar applications of fluopyram (SC 500 formulation) tank mixed with trifloxystrobin (SC

500) were applied to celery plants at 0.24–0.26 kg ai/ha in 140–190 litres of water, 5 days apart, using CO_2 pressurised plot sprayers, knapsack sprayers or tractor-mounted or self-propelled boom sprayers. Plot sizes in these trials ranged from 22–177 square metres.

Duplicate samples (celery stalks and leaves) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 241 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg). Analytical results from washed, trimmed and/or cooked celery are presented in Table 200 (Magnitude of the residue in processing).

Table 163 Fluopyram residues in celery (stalks and leaves) from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

CELERY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Belle Glade, FL (M9)	2	0.257 0.255	0.144 0.134	178 190	0	4.507, 6.38				GM094-07HA
USA, 2007 Arkansaw, WI (Dutchess)	2	0.255 0.256	0.142 0.143	179 179	0	0.038, < 0.01				GM095-07HA
USA, 2007 Guadalupe, CA (Conquistador)	2	0.247 0.252	0.163 0.158	152 160	0	3.25, 4.384				GM097-07HA
USA, 2007 Santa Maria, CA (Mission)	2	0.251 0.254	0.179 0.179	140 142	0	1.561, 1.593				GM098-07HA
USA, 2007 King City, CA (Hills Special)	2	0.253 0.263	0.145 0.144	174 183	0	2.379, 2.093				GM099-07HA
USA, 2007 Sanger, CA (Command)	2	0.244 0.248	0.139 0.143	175 173	0 1 3 7 10	10.6, 10.56 *7.96, 8.13, 8.1* 8.632, 8.959 1.319, 1.363 1.428, 1.328 1.382, 1.207				GM096-07DA *Results from processing study*

Cereal grains

Barley

Results from supervised seed treatment trials on barley in Europe were provided to the Meeting. In these trials, winter and spring barley seed was treated with fluopyram at a nominal rate of 1 g ai/100 kg seed, using either a 500 SC formulation or a 285 FS formulation containing fluopyram (20 g ai/Litre) in combination with fluoxastrobin, prothioconazole and tebuconazole (In the 2006 trials, a higher treatment rate of 1.2 g ai/100 kg seed was used). Actual treatment rates, based on measured (dislodgeable) residues in the treated seed ranged from 0.64–0.98 g ai/100 kg seed. Plot sizes ranged from 70–230 square metres and sowing (at 200 kg seed/ha) occurred the day of treatment.

Unreplicated samples of treated seed (minimum of 0.2 kg), plants (minimum of 2 kg green material), grain (minimum of 1 kg) and straw (minimum of 0.5 kg) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 401 days (seeds), 267 days (plants), 209 days (straw) and 209 days (grain) before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Methods 00984 or 00984/M001. Reported LOQs for Method 00984 were 0.01 mg/kg for seeds, grain and green plant material and 0.05 mg/kg in straw (2006 trials)

and for Method 00984/M001 (2007 trials) the LOQs were 0.1 mg/kg for seeds and 0.01 mg/kg for plants and grain.

Table 164 Residues in barley from supervised trials in France, Germany, Italy and UK, involving seed treatment applications of fluopyram

BARLEY Country, year Location (variety)	Application		PHI, (days)	Residues (mg/kg)					Reference & Comments
	form	g ai/100kg seed		matrix	parent	PAA	BZM	PCA	
France, 2006 Chambourg sur Indre (Lomerit) Barley, winter	500 SC	1.2 (0.83)[a]	0 179 262 262	seed plant grain straw	8.3 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	0.03 < 0.01 < 0.01 < 0.05	RA-2657/06 0894-06
Germany, 2006 Burscheid (Lomerit) Barley, winter	500 SC	1.2	196 284 284	plant grain straw	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	RA-2657/06 0896-06
Germany, 2007 Burscheid (Belana) Barley, spring	285 FS	1 (0.98)[a]	0 62 145 145	seed plant grain straw	9.8 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	RA-2618/07 0259-07
United Kingdom, 2007 Little Shelford (Belana) Barley, spring	285 FS	1 (0.92)[a]	0 44 136 136	seed plant grain straw	9.2 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	RA-2618/07 0260-07
France, 2006 Gargas (Platine) Barley, winter	500 SC	1.2 (0.77)[a]	0 148 241 241	seed plant grain straw	7.7 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	0.06 < 0.01 < 0.01 < 0.05	RA-2658/06 0897-06
Italy, 2006 Bologna (Platine) Barley, winter	500 SC	1.2	160 230 230	plant grain straw	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	RA-2658/06 0898-06
France, 2007 Villeneuve les Bouloc (Prestige) Barley, spring	285 FS	1 (0.66)[a]	0 76 129 129	seed plant grain straw	6.6 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	RA-2619/07 0207-07
Italy, 2007 Bologna (Prestige) Barley, spring	285 FS	1 (0.64)[a]	0 63 121 121	seed plant grain straw	6.4 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	RA-2619/07 0208-07

[a] Dislodgeable residues measured on treated grain at time of sowing

Wheat

Results from supervised seed treatment trials on wheat in Europe were provided to the Meeting. In these trials, winter and spring wheat seed was treated with fluopyram at a nominal rate of 1 g ai/100 kg seed, using either a 500 SC formulation or a 285 FS formulation containing fluopyram (20 g ai/L) in combination with fluoxastrobin, prothioconazole and tebuconazole (In some of the 2006

trials, a higher treatment rate of 1.2 g ai/100 kg seed was used). Actual treatment rates, based on measured (dislodgeable) residues in the treated seed ranged from 0.86–0.91 g ai/100 kg seed. Plot sizes ranged from 69–200 square metres and sowing (at 240 kg seed/ha) occurred the day of treatment.

Unreplicated samples of treated seed (minimum 0.2 kg), plants (minimum 1.8 kg green material), grain (minimum 1 kg) and straw (minimum 0.5 kg) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 393 days (seeds), 267 days (plants), 208 days (straw) and 208 days (grain) before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Methods 00984 or 00984/M001. Reported LOQs for Method 00984 were 0.01 mg/kg for seeds, grain and green plant material and 0.05 mg/kg in straw (2006 trials) and for Method 00984/M001 (2007 trials) the LOQs were 0.1 mg/kg for seeds and 0.01 mg/kg for plants and grain.

Table 165 Residues in wheat from supervised trials in France, Germany, Italy, Spain and UK, involving seed treatment applications of fluopyram

WHEAT Country, year Location (variety)	Application		PHI, (days)	Residues (mg/kg)					Reference & Comments
	form	g ai/100kg seed		matrix	parent	PAA	BZM	PCA	
France, 2006 Chambourg sur Indre (Limes) Wheat, winter	500 SC	1.2 (0.91)[a]	0 162 268 268	seed plant grain straw	9.1 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	RA-2659/06 0895-06
Germany, 2006 Burscheid (Limes) Wheat, winter	500 SC	1.2	190 293 293	plant grain straw	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	RA-2659/06 0899-06
Germany, 2007 Burscheid (Melissos) Wheat, spring	285 FS	1 (0.91)[a]	0 62 153 153	seed plant grain straw	9.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	RA-2622/07 0261-07
United Kingdom, 2007 Little Shelford (Melissos) Wheat, spring	285 FS	1 (0.95)[a]	0 52 147 147	seed plant grain straw	9.5 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	RA-2622/07 0262-07
France, 2006 Gargas (Quality) Wheat, winter	500 SC	1 (0.97)[a]	0 148 247 247	seed plant grain straw	9.7 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.01 < 0.05	RA-2660/06 0900-06
Spain, 2006 Brenes Sevilla (Quality) Wheat, winter	500 SC	1	113 180 180	plant grain straw	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	< 0.01 < 0.01 < 0.05	RA-2660/06 0901-06
France, 2007 Villeneuve les Bouloc (Panifor) Wheat, spring	285 FS	1 (0.86)[a]	0 76 126 126	seed plant grain straw	8.6 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	RA-2623/07 0209-07

WHEAT Country, year Location (variety)	Application		PHI, (days)	Residues (mg/kg)					Reference & Comments
	form	g ai/100kg seed		matrix	parent	PAA	BZM	PCA	
Italy, 2007 Bologna (Panifor) Wheat, spring	285 FS	1 (0.91)[a]	0 63 122 122	seed plant grain straw	9.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	< 0.1 < 0.01 < 0.01 < 0.01	RA-2623/07 0210-07

[a] Dislodgeable residues measured on treated grain at time of sowing

Results from supervised trials from USA on wheat were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to wheat, 12–17 days apart as foliar sprays using knapsack sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 93–189 litres of spray mix/ha. Plot sizes in these trials ranged from 50–230 square metres. In each trial, one plot was last treated over the tillering period (up to BBCH 41) for sampling of forage, a second plot was last treated before the start of grain ripening (up to BBCH 83) for sampling of hay and a third plot was last treated over the grain ripening period (up to BBCH 89) for sampling of grain and straw. Hay from the second plots was allowed to dry to commercial dryness before sampling (0–9 days after cutting).

Duplicate samples of at least 1 kg forage and grain and at least 0.5 kg hay and straw were taken from each plot, frozen within 2 hours of sampling, held in frozen storage for up to 374 days (grain), 388 days (forage), 340 days (hay) and 391 days (straw) before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg (forage, hay, grain) and 0.1 mg/kg (straw).

Table 166 Fluopyram residues in wheat grain and straw from supervised trials in USA involving foliar applications (500 SC formulations)

WHEAT GRAIN, STRAW Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	water (L/ha)	GS (BBCH)		matrix	parent	PAA	BZM	PCA	
USA, 2007 Sufflok, VA (Pioneer 26R24) Wheat, Winter	2	0.254 0.252	121 123	83 87	14 14	grain (84%DM) straw (84%DM)	0.21, 0.21 7.06, 6.82				RAGMP064 GM238-06HA
USA, 2007 Proctor, AR (Delta King 9410) Wheat, Winter	2	0.251 0.249	132 133	87 89	13 13	grain (91%DM) straw (91%DM)	0.68, 0.76 7.33, 10.56				RAGMP064 GM239-06HA
USA, 2007 Richland, IA (Variety Unknown) Wheat, Winter	2	0.249 0.243	152 144	61 75	13 13	grain (86%DM) straw (72%DM)	0.2, 0.11 0.89, 0.81				RAGMP064 GM241-06HB
USA, 2007 Carlyle, IL (BT-Branson) Wheat, Winter	2	0.252 0.247	119 109	77 85	14 14	grain (82%DM) straw (75%DM)	0.31, 0.29 4.62, 4.97				RAGMP064 GM242-06HA

WHEAT GRAIN, STRAW Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	water (L/ha)	GS (BBCH)		matrix	parent	PAA	BZM	PCA	
USA, 2007 East Bernard, TX (Fannin) Wheat, Winter	2	0.249 0.256	97 98	83 87	12 12	grain (89%DM) straw (87%DM	0.19, 0.15 12.26, 10.79				RAGMP064 GM243-06HA
USA, 2007 Grand Island, NE (Wahoo HRW Wheat) Wheat, Winter	2	0.26 0.25	140 189	73 77	14 14	grain (90%DM) straw (84%DM	0.13, 0.13 2.93, 3.56				RAGMP064 GM244-06HA
USA, 2006 New Rockford, ND (Alsen) Wheat, Spring	2	0.248 0.249	138 138	83 87	14 14	grain (89%DM) straw (81%DM	0.26, 0.24 3.99, 4				RAGMP064 GM245-06HA
USA, 2006 Eldridge, ND (Knudson) Wheat, Spring	2	0.248 0.252	137 144	73 83	13 13	grain (89%DM) straw (68%DM	0.23, 0.22 4.66, 4.69				RAGMP064 GM246-06HA
USA, 2006 Velva, ND (Alsen) Wheat, Spring	2	0.251 0.252	122 121	75 85	15 15	grain (90%DM) straw (87%DM	0.16, 0.14 4.6, 5.97				RAGMP064 GM247-06HA
USA, 2007 Larned, KS (Jagalene) Wheat, Winter	2	0.245 0.262	172 172	71 85	13 13	grain (72%DM) straw (52%DM	0.14, 0.12 1.04, 0.94				RAGMP064 GM248-06HA
USA, 2007 Belpre, KS (Overley) Wheat, Winter	2	0.251 0.252	175 173	73 85	13 13	grain (77%DM) straw (59%DM	0.037, 0.038 0.89, 0.85				RAGMP064 GM249-06HA
USA, 2008 Plainview, TX (Dumas) Wheat, Winter	2	0.246 0.253	155 161	69 87	14 14	grain (92%DM) straw (83%DM	0.19, 0.19 7.32, 7.35				RAGMP064 GM250-06HB
USA, 2007 Plainview, TX (TAM 111) Wheat, Winter	2	0.248 0.256	181 187	73 87	14 14	grain (89%DM) straw (69%DM	0.19, 0.19 3.15, 3.21				RAGMP064 GM251-06HA
USA, 2006 Ephrata, WA (Sunstar 50-30) Wheat, Spring	2	0.249 0.252	140 141	85 87	14 14	grain (92%DM) straw (85%DM	0.2, 0.19 5.27, 6.08				RAGMP064 GM252-06HA

WHEAT GRAIN, STRAW Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	water (L/ha)	GS (BBCH)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Sabin, MN	2	0.261 0.253	156 179	85 89	0 7 14 21 28 0 7 14 21 28	grain (90%DM) grain (84%DM) grain (89%DM) grain (90%DM) grain (89%DM) straw (89%DM straw (89%DM straw (89%DM straw (91%DM straw (88%DM	0.35, 0.33 0.19, 0.2 0.22, 0.23 0.23, 0.26 0.24, 0.25 26.82, 30.29 5.3, 5.26 5.5, 4.89 4.44, 4.29 1.48, 1.82				RAGMP064 GM240-06DA

Table 167 Fluopyram residues in wheat forage from supervised trials in USA involving foliar applications (500 SC formulations)

WHEAT FORAGE Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	water (L/ha)	GS (BBCH)		matrix	parent	PAA	BZM	PCA	
USA, 2007 Sufflok, VA (Delta King 9410) Wheat, Winter	2	0.253 0.25	125 130	29 37	12	forage (27%DM)	0.52, 0.49				RAGMP064 GM238-06HA
USA, 2007 Proctor, AR (Variety Unknown) Wheat, Winter	2	0.25 0.25	133 133	21 26	14	forage (27%DM)	0.92, 0.75				RAGMP064 GM239-06HA
USA, 2007 Richland, IA (BT-Branson) Wheat, Winter	2	0.25 0.25	161 167	15 23	14	forage (24%DM)	0.14, 0.13				RAGMP064 GM241-06HB
USA, 2007 Carlyle, IL (Fannin) Wheat, Winter	2	0.247 0.25	180 146	24 29	13	forage (19%DM)	0.53, 0.52				RAGMP064 GM242-06HA
USA, 2007 East Bernard, TX (Wahoo HRW Wheat) Wheat, Winter	2	0.245 0.25	93 94	26 27	13	forage (22%DM)	0.39, 0.32				RAGMP064 GM243-06HA
USA, 2007 Grand Island, NE (Alsen) Wheat, Spring	2	0.245 0.25	186 183	23 24	14	forage (18%DM)	0.49, 0.58				RAGMP064 GM244-06HA
USA, 2006 New Rockford, ND (Knudson) Wheat, Spring	2	0.254 0.25	143 140	11 21	12	forage (16%DM)	0.17, 0.12				RAGMP064 GM245-06HA

WHEAT FORAGE Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	water (L/ha)	GS (BBCH)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Eldridge, ND (Alsen) Wheat, Spring	2	0.249 0.257	140 144	22 32	13	forage (22%DM)	1.43, 1.12				RAGMP064 GM246-06HA
USA, 2006 Velva, ND (Jagalene) Wheat, Winter	2	0.243 0.249	130 122	12 22	14	forage (19%DM)	0.067, 0.052				RAGMP064 GM247-06HA
USA, 2007 Larned, KS (Overley) Wheat, Winter	2	0.249 0.257	171 173	23 30	14	forage (23%DM)	1.2, 1.13				RAGMP064 GM248-06HA
USA, 2007 Belpre, KS (Dumas) Wheat, Winter	2	0.256 0.256	176 173	23 30	14	forage (22%DM)	1.08, 1.08				RAGMP064 GM249-06HA
USA, 2007 Plainview, TX (Dumas) Wheat, Winter	2	0.241 0.242	152 155	24 30	14	forage (17%DM)	0.88, 0.99				RAGMP064 GM250-06HA
USA, 2008 Plainview, TX (Sunstar 50-30) Wheat, Spring	2	0.246 0.245	157 155	14 28	14	forage (21%DM)	0.79, 0.79 (c=0.013)				RAGMP064 GM250-06HB
USA, 2006 Ephrata, WA (Steele) Wheat, Spring	2	0.25 0.251	138 141	16 30	14	forage (23%DM)	0.64, 0.48				RAGMP064 GM252-06HA
USA, 2006 Sabin, MN (Steele) Wheat, Spring	2	0.251 0.257	165 159	22 41	0 6 13 20 29	forage (16%DM) forage (18%DM) forage (25%DM) forage (32%DM) forage (43%DM)	9.31, 10.76 4.13, 3.64 3.03, 2.78 1.55, 1.34 1.31, 1.27				RAGMP064 GM240-06DA

Table 168 Fluopyram residues in wheat hay from supervised trials in USA involving foliar applications (500 SC formulations)

WHEAT HAY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	water (L/ha)	GS (BBCH)		matrix	parent	PAA	BZM	PCA	
USA, 2007 Sufflok, VA (Delta King 9410) Wheat, Winter	2	0.252 0.252	111 110	57 73	12	hay (69%DM)	3.41, 3.4				RAGMP064 GM238-06HA
USA, 2007 Proctor, AR (Variety Unknown) Wheat, Winter	2	0.249 0.249	133 133	59 61	14	hay (51%DM)	2.38, 1.79				RAGMP064 GM239-06HA
USA, 2007 Richland, IA (BT-Branson) Wheat, Winter	2	0.25 0.252	174 173	24 41	14	hay (70%DM)	1.11, 0.9				RAGMP064 GM241-06HB
USA, 2007 Carlyle, Illinois (Fannin) Wheat, Winter	2	0.252 0.252	147 123	29 30	17	hay (39%DM)	0.55, 0.84				RAGMP064 GM242-06HA
USA, 2007 East Bernard, TX (Wahoo HRW Wheat) Wheat, Winter	2	0.253 0.248	97 103	61 65	13	hay (70%DM)	0.99, 0.92				RAGMP064 GM243-06HA
USA, 2007 Grand Island, NE (Alsen) Wheat, Spring	2	0.244 0.245	185 186	43 55	13	hay (87%DM)	0.51, 0.41				RAGMP064 GM244-06HA
USA, 2006 New Rockford, ND (Knudson) Wheat, Spring	2	0.249 0.251	139 141	58 71	14	hay (81%DM)	2.67, 2.44				RAGMP064 GM245-06HA
USA, 2006 Eldridge, ND (Alsen) Wheat, Spring	2	0.253 0.254	143 150	32 51	12	hay (69%DM)	4.73, 5.12				RAGMP064 GM246-06HA
USA, 2006 Velva, ND (Jagalene) Wheat, Winter	2	0.249 0.251	122 123	30 59	12	hay (65%DM)	5.06, 4.97				RAGMP064 GM247-06HA
USA, 2007 Larned, KS (Overley) Wheat, Winter	2	0.258 0.247	179 172	32 47	12	hay (59%DM)	0.29, 0.35				RAGMP064 GM248-06HA

WHEAT HAY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	water (L/ha)	GS (BBCH)		matrix	parent	PAA	BZM	PCA	
USA, 2007 Belpre, KS (Dumas) Wheat, Winter	2	0.263 0.263	178 178	32 53	12	hay (68%DM)	0.28, 0.3				RAGMP064 GM249- 06HA
USA, 2007 Plainview, TX (Dumas) Wheat, Winter	2	0.239 0.24	153 153	30 41	14	hay (61%DM)	1.54, 1.53				RAGMP064 GM250- 06HA
USA, 2008 Plainview, TX (TAM 111) Wheat, Winter	2	0.249 0.252	157 159	30 36	14	hay (52%DM)	3.5, 3.54				RAGMP064 GM250- 06HB
USA, 2007 Plainview, TX (Sunstar 50-30) Wheat, Spring	2	0.253 0.249	185 181	37 61	15	hay (73%DM)	2.27, 2.26				RAGMP064 GM251- 06HA
USA, 2006 Ephrata, WA (Steele) Wheat, Spring	2	0.247 0.25	138 140	65 83	14	hay (86%DM)	0.58, 0.66				RAGMP064 GM252- 06HA
USA, 2006 Sabin, MN (Steele) Wheat, Spring	2	0.257 0.254	159 148	49 53	0 7 14 21 28	hay (84%DM) hay (86%DM) hay (86%DM) hay (92%DM) hay (88%DM)	36.5, 36.67 13.52, 14.82 5.31, 5.51 4.95, 4.67 2.01, 2.08				RAGMP064 GM240- 06DA

Sorghum

Results from supervised trials from USA on sorghum (grain, forage and straw/stover) were provided to the Meeting.

In these trials, two applications of 0.244–0.26 kg ai/ha (SC 500 formulation) were applied to sorghum, 13–15 days apart as foliar sprays using knapsack sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 99–189 litres of spray mix/ha. Plot sizes in these trials ranged from 50–264 square metres. In each trial, one plot was last treated over the grain development to early ripening stages (up to BBCH 87) for sampling of forage and a second plot was last treated over the grain ripening period (up to BBCH 89) for sampling of grain and straw (stover).

Duplicate samples of at least 1 kg forage and grain and at least 0.5 kg straw (stover) were taken from each plot, frozen within 2 hours of sampling, held in frozen storage for up to 374 days (grain), 385 days (forage) and 391 days (straw) before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg (forage, hay, grain) and 0.1 mg/kg (straw).

Table 169 Fluopyram residues in sorghum grain and straw from supervised trials in USA involving two foliar applications (500 SC formulations)

SORGHUM GRAIN, STRAW Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	kg ai/ha	water (L/ha)	GS (BBCH)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Sufflok, VA (Pioneer 85G46)	0.256 0.253	107 101	87 89	14 14	fodder (38%DM) grain (88%DM)	1.1, 1.17 0.76, 0.61				RAGMP064 GM253-06HA
USA, 2006 Leland, MS (Pioneer 83G66-N271)	0.257 0.261	154 150	85 89	12 12	fodder (42%DM) grain (86%DM)	1.41, 0.96 0.25, 0.27				RAGMP064 GM254-06HA
USA, 2006 Earlham, IA (NC+371)	0.251 0.244	116 113	77 85	14 14	fodder (23%DM) grain (74%DM)	0.34, 0.31 0.23, 0.26				RAGMP064 GM256-06HA
USA, 2006 Seymour, IL (Garst 5440)	0.245 0.258	167 175	87 87	12 12	fodder (40%DM) grain (82%DM)	0.6, 0.58 0.4, 0.49				RAGMP064 GM257-06HA
USA, 2006 Arkansaw, WI (86G71)	0.253 0.252	178 177	87 87	14 14	fodder (37%DM) grain (85%DM)	1.34, 0.9 0.67, 0.61				RAGMP064 GM258-06HA
USA, 2006 Raymondville, TX (NK KS310)	0.258 0.258	145 145	85 87	14 14	fodder (54%DM) grain (90%DM)	1.49, 1.32 0.68, 0.73				RAGMP064 GM259-06HA
USA, 2006 East Bernard, TX (DK52)	0.253 0.253	103 99	75 87	13 13	fodder (51%DM) grain (85%DM)	0.61, 0.59 0.24, 0.23				RAGMP064 GM260-06HA
USA, 2006 Grand Island, NE (NC+ 6B50)	0.25 0.248	189 188	87 87	14 14	fodder (28%DM) grain (87%DM)	0.49, 0.57 0.24, 0.25				RAGMP064 GM261-06HA
USA, 2006 Larned, KS (Pioneer 94G62)	0.249 0.247	171 170	85 87	13 13	fodder (35%DM) grain (87%DM)	3.0, 2.91 0.54, 0.45				RAGMP064 GM262-06HA
USA, 2006 Levelland, TX (Y363)	0.252 0.245	141 138	81 85	12 12	fodder (41%DM) grain (80%DM)	12.15, 5.1 (c= 0.02) 3.24, 2.82				RAGMP064 GM263-06HA
USA, 2006 Plainview, TX (Pioneer 84G62)	0.254 0.249	144 139	85 89	14 14	fodder (26%DM) grain (86%DM)	0.22, 0.19 0.28, 0.24				RAGMP064 GM264-06HA
USA, 2006 Stilwell, KS (Garst 5401)	0.254 0.253	141 141	85 85	0 7 14 21 27 0 14 7 21 27	fodder (34%DM) fodder (30%DM) fodder (24%DM) fodder (26%DM) fodder (26%DM) grain (75%DM) grain (76%DM) grain (79%DM) grain (86%DM) grain (88%DM)	10.41, 11.43 1.76, 2.05 1.05, 1.22 1.13, 1.09 1.31, 1.01 1.39, 1.42 0.58, 0.55 0.23, 0.23 0.22, 0.21 0.19, 0.16 (c=0.027)				RAGMP064 GM255-06DA

Table 170 Fluopyram residues in sorghum forage from supervised trials in USA involving two foliar applications (500 SC formulations)

SORGHUM FORAGE Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	kg ai/ha	water (L/ha)	GS (BBCH)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Sufflok, VA (Pioneer 85G46)	0.254 0.254	97 107	85 87	14	forage (38%DM)	0.94, 0.99				RAGMP064 GM253-06HA
USA, 2006 Leland, MS (Pioneer 83G66-N271)	0.259 0.26	155 156	75 85	12	forage (29%DM)	1.44, 3.49				RAGMP064 GM254-06HA
USA, 2006 Earlham, IO (NC+371)	0.247 0.258	114 119	71 77	14	forage (30%DM)	0.18, 0.28				RAGMP064 GM256-06HA
USA, 2006 Seymour, IL (Garst 5440)	0.248 0.249	138 171	85 85	13	forage (32%DM)	0.71, 0.73				RAGMP064 GM257-06HA
USA, 2006 Arkansaw, WI (86G71)	0.252 0.247	177 172	81 85	14	forage (30%DM)	0.36, 0.54				RAGMP064 GM258-06HA
USA, 2006 Raymondville, TX (NK KS310)	0.251 0.256	141 143	67 85	14	forage (40%DM)	1.04, 0.9				RAGMP064 GM259-06HA
USA, 2006 East Bernard, TX (DK52)	0.258 0.253	104 98	69 85	13	forage (39%DM)	0.92, 1.11				RAGMP064 GM260-06HA
USA, 2006 Grand Island, NE (NC+ 6B50)	0.251 0.25	182 183	71 85	14	forage (28%DM)	0.44, 0.33				RAGMP064 GM261-06HA
USA, 2006 Larned, KS (Pioneer 94G62)	0.25 0.252	169 170	81 85	14	forage (32%DM)	0.47, 0.44				RAGMP064 GM262-06HA
USA, 2006 Levelland, TX (Y363)	0.258 0.247	140 138	81 85	14	forage (47%DM)	4.06, 4.1				RAGMP064 GM263-06HA
USA, 2006 Plainview, TX (Pioneer 84G62)	0.253 0.252	138 143	75 85	14	forage (35%DM)	0.81, 1.05				RAGMP064 GM264-06HA
USA, 2006 Stilwell, KS (Garst 5401)	0.245 0.251	138 139	67 75	0 7 14 20 28	forage (25%DM) forage (27%DM) forage (28%DM) forage (31%DM) forage (33%DM)	4.09, 4.71 0.97, 0.97 1.36, 0.54 0.7, 0.61 0.48, 0.44				RAGMP064 GM255-06DA

Maize & Sweetcorn

Results from supervised trials from USA on maize and sweetcorn were provided to the Meeting. In these trials, two applications of 0.243–0.267 kg ai/ha (SC 500 formulation) were applied to maize and sweet corn, 5–8 days apart as foliar sprays using knapsack sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 111–187 litres of spray mix/ha. Plot sizes in these trials ranged from 37–297 square metres.

In each maize trial, one plot was last treated over the early ripening stages (BBCH 85–87) for sampling of forage and a second plot was last treated when the kernels were at the mature to fully ripe stage (BBCH 87–89) for sampling of kernels and fodder (i.e., leaves, stalks, husks and cobs after removal of the kernels).

Cobs (without husks) and forage (including husks) were sampled from sweetcorn and some field corn trials last treated when the kernels were close to mature (BBCH 75–79).

Duplicate samples of at least 1 kg cobs, grain and forage and at least 0.5 kg fodder (stover) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 340 days (grain), 435 days (cobs), 409 days (forage) and 366 days (fodder) before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg (kernels, cobs, forage and fodder. All residues were reported on a fresh weight basis.

Table 171 Fluopyram residues in maize kernals and fodder (stover) from supervised trials in USA involving two foliar applications (500 SC formulations)

MAIZE Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg fresh weight)					Reference & Comments
	kg ai/ha	water (L/ha)	GS – BBCH		matrix	parent	PAA	BZM	PCA	
USA, 2006 Germansville, PA (TA5750) Corn , Field	0.262 0.258	187 184	87 89	14 14	fodder (32%DM) kernels (70%DM)	1.64, 1.31 < 0.01, < 0.01				RAGMP038 GM043-06HA
USA, 2006 Tifton, GA (31N26) Corn , Field	0.25 0.25	141 138	85 87	13 13	fodder (80%DM) kernels (84%DM)	2.38, 2.13 < 0.01, < 0.01				RAGMP038 GM044-06HA
USA, 2006 New Holland, OH (Crows 7R154) Corn , Field	0.257 0.25	151 147	87 87	14 14	fodder (40%DM) kernels (82%DM)	0.698, 0.871 < 0.01, < 0.01				RAGMP038 GM046-06HA
USA, 2006 Richland, IA (9190 LL HX) Corn , Field	0.247 0.251	118 183	87 87	13 13	fodder (34%DM) kernels (80%DM)	1.51, 2.12 < 0.01, < 0.01				RAGMP038 GM047-06HA
USA, 2006 Stilwell, KS (Garst 8287 RR) Corn , Field	0.248 0.253	133 136	85 87	14 14	fodder (60%DM) kernels (83%DM)	1.99, 1.74 < 0.01, < 0.01				RAGMP038 GM048-06HA
USA, 2006 Sabin, MN (Pioneer 39H85) Corn , Field	0.259 0.251	174 183	85 85	14 14	fodder (79%DM) kernels (85%DM)	2.98, 2.57 0.016, 0.02				RAGMP038 GM051-06HA
USA, 2006 Earlham, IA (35P17 LL) Corn , Field	0.245 0.249	111 116	85 85	12 12	fodder (37%DM) kernels (80%DM)	0.782, 1.56 < 0.01, < 0.01				RAGMP038 GM052-06HA
USA, 2006 Springfield, NE (NK38B4) Corn , Field	0.25 0.251	130 131	87 87	11	fodder (50%DM) kernels (83%DM)	4.36, 3.89 < 0.01, < 0.01				RAGMP038 GM053-06HA

MAIZE Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg fresh weight)					Reference & Comments
	kg ai/ha	water (L/ha)	GS – BBCH		matrix	parent	PAA	BZM	PCA	
USA, 2006 Percival, IA (NK 65C5) Corn , Field	0.249 0.251	130 129	87 87	12 12	fodder (33%DM) kernels (78%DM)	1.28, 0.962 < 0.01, < 0.01				RAGMP038 GM054-06HA
USA, 2006 Gardner, KS (Garst 8881 RR) Corn , Field	0.244 0.248	131 132	85 87	14 14	fodder (55%DM) kernels (87%DM)	1.4, 0.999 0.011, < 0.01				RAGMP038 GM055-06HA
USA, 2006 New Holland, OH (NK-N69-P9) Corn , Field	0.25 0.256	147 150	87 87	14 14	fodder (40%DM) kernels (82%DM)	0.967, 1.06 < 0.01, < 0.01				RAGMP038 GM056-06HA
USA, 2006 Arkansaw, WI (38B85) Corn , Field	0.253 0.256	177 179	87 87	14 14	fodder (44%DM) kernels (81%DM)	1.9, 2.24 < 0.01, < 0.01				RAGMP038 GM057-06HA
USA, 2006 Uvalde, TX (Pioneer 32R25) Corn , Field	0.245 0.248	164 171	89 89	12 12	fodder (53%DM) kernels (82%DM)	14.69, 12.12 < 0.01, < 0.01				RAGMP038 GM058-06HA
USA, 2006 Seymour, IL (Garst 8568 CB/LL) Corn , Field	0.252 0.257	173 174	87 89	0 6 13 19 26 0 6 13 19 26	fodder (35%DM) fodder (44%DM) fodder (47%DM) fodder (49%DM) fodder (52%DM) kernels (78%DM) kernels (80%DM) kernels (85%DM) kernels (87%DM) kernels (87%DM)	6.9, 7.36 3.55, 3.42 3.19, 2.79 2.8, 2.18 2.74, 2.26 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01				RAGMP038 GM049-06DA
USA, 2006 York, NE (NK N70-F1 LL/YG) Corn , Field	0.248 0.244	185 187	87 87	1 8 12 21 26 1 8 12 21 26	fodder (38%DM) fodder (38%DM) fodder (40%DM) fodder (60%DM) fodder (83%DM) kernels (70%DM) kernels (75%DM) kernels (77%DM) kernels (80%DM) kernels (83%DM)	9.21, 8.13 1.18, 1.55 1.39, 0.827 1.13, 0.913 1.08, 0.689 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01				RAGMP038 GM050-06DA

Table 172 Fluopyram residues in maize/sweetcorn forage from supervised trials in USA involving foliar applications (500 SC formulations)

MAIZE FORAGE Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg fresh weight)					Reference & Comments
	kg ai/ha	water (L/ha)	GS – BBCH		matrix	parent	PAA	BZM	PCA	
USA, 2006 Germansville, PA (TA5750) Corn , Field	0.26 0.267	180 190	73 75	0	forage (23%DM)	2.98, 2.93				RAGMP038 GM043-06HA
USA, 2006 Tifton, GA (31N26) Corn , Field	0.25 0.25	135 125	71 75	0	forage (29%DM)	4.97, 3.02				RAGMP038 GM044-06HA
USA, 2006 New Holland, OH (Crows 7R154) Corn , Field	0.253 0.252	146 148	71 79	0	forage (24%DM)	3.83, 3.15				RAGMP038 GM046-06HA
USA, 2006 Richland, IA (9190 LL HX) Corn , Field	0.245 0.25	129 131	73 75	0	forage (22%DM)	2.53, 2.45				RAGMP038 GM047-06HA
USA, 2006 Stilwell, KS (Garst 8287 RR) Corn , Field	0.245 0.252	131 136	71 79	0	forage (24%DM)	4.64, 5.41				RAGMP038 GM048-06HA
USA, 2006 Fresno, CA (Silver Queen) Corn , Sweet	0.25 0.251	170 172	71 73	0 3 7 10 14	forage (22%DM) forage (25%DM) forage (26%DM) forage (26%DM) forage (28%DM)	4.78, 5.52 4.06, 5.85 4.74, 5.78 4.75, 4.23 3.81, 4.07				RAGMP038 GM059-06DA
USA, 2006 Sabin, MN (Pioneer 39H85) Corn , Field	0.253 0.243	158 159	79 85	0	forage (47%DM)	3.39, 4.34				RAGMP038 GM051-06HA 16d spray interval
USA, 2006 Earlham, IO (35P17 LL) Corn , Field	0.258 0.25	118 118	85 85	0	forage (51%DM)	2.21, 3.7				RAGMP038 GM052-06HA
USA, 2006 Springfield, NE (NK38B4) Corn , Field	0.251 0.25	129 131	83 85	0	forage (25%DM)	2.54, 3.54				RAGMP038 GM053-06HA
USA, 2006 Percival, IO (NK 65C5) Corn , Field	0.251 0.252	131 132	85 85	0	forage (33%DM)	1.98, 3.1				RAGMP038 GM054-06HA
USA, 2006 Gardner, KS (Garst 8881 RR) Corn , Field	0.253 0.245	136 132	79 85	0	forage (48%DM)	5.01, 4.42				RAGMP038 GM055-06HA

MAIZE FORAGE Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg fresh weight)					Reference & Comments
	kg ai/ha	water (L/ha)	GS – BBCH		matrix	parent	PAA	BZM	PCA	
USA, 2006 New Holland, OH (NK-N69-P9) Corn , Field	0.258 0.252	153 148	85 85	0	forage (28%DM)	5.05, 3.97				RAGMP038 GM056-06HA
USA, 2006 Arkansaw, WI (38B85) Corn , Field	0.254 0.249	178 174	83 87	0	forage (36%DM)	1.56, 2.11				RAGMP038 GM057-06HA
USA, 2006 Uvalde, TX (Pioneer 32R25) Corn , Field	0.251 0.255	179 188	85 85	0	forage (24%DM)	2.55, 3.19				RAGMP038 GM058-06HA
USA, 2006 Seymour, IL (Garst 8568 CB/LL) Corn , Field	0.26 0.247	174 170	85 85	0 3 7 9 13	forage (38%DM) forage (46%DM) forage (46%DM) forage (46%DM) forage (47%DM)	4.13, 5.08 5.79, 5.08 1.82, 2.06 1.48, 2.55 1.75, 1.26				RAGMP038 GM049-06DA
USA, 2006 York, NE (NK N70-F1 LL/YG) Corn , Field	0.251 0.258	181 186	85 87	1 3 7 10 13	forage (33%DM) forage (40%DM) forage (40%DM) forage (36%DM) forage (47%DM)	2.53, 2.04 1.82, 2.78 1.71, 2.91 1.75, 1.69 1.92, 2.07				RAGMP038 GM050-06DA

Straw, fodder, forage of cereal grains and grasses

Grasses

Results from supervised trials from USA on pasture grasses were provided to the Meeting. In these trials, two applications of 0.24–0.262 kg ai/ha (SC 500 formulation) were applied to a range of pasture grasses, 14–15 days apart as foliar sprays using knapsack sprayers with hand-held spray booms, plot or tractor-mounted boom sprayers to apply 120–186 litres of spray mix/ha. Plot sizes in these trials ranged from 28–278 square metres and the last treatments were applied at BBCH 41 (early boot stage).

Duplicate samples of at least 0.9 kg forage and 0.5 kg hay (cut and allowed to dry for 1–6 days to commercial dryness) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 282 days (forage) and 307 days (hay) before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg for forage and hay. All residues were reported on a fresh weight basis.

Table 173 Fluopyram residues in grass forage and hay from supervised trials in USA involving two foliar applications (500 SC formulations)

GRASS FORAGE, HAY Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2007 Hudson, KS (Ozark) Bermuda grass	2	0.249 0.239	171 162	0 7	forage (33%DM) hay (68%DM)	13.375, 15.955 3.966, 5.476				RAGMP044 GM009-07HA
USA, 2007 Pepin, WI (Native) Bluegrass	2	0.249 0.248	174 174	0 7	forage (31%DM) hay (91%DM)	34.132, 26.592 10.029, 7.296				RAGMP044 GM010-07HA
USA, 2007 Springfield, NE (Kentucky 31) Fescue	2	0.251 0.249	131 134	0 7	forage (16%DM) hay (33%DM)	14.124, 13.425 10.609, 13.101 (c=0.06)				RAGMP044 GM011-07HA (hay results mean of 3 analyses)
USA, 2007 Hermiston, OR (Penn 91) Rye Grass	2	0.253 0.252	178 174	0 7	forage (19%DM) hay (19%DM)	6.426, 8.352 1.816, 1.892 (c=0.33)				RAGMP044 GM012-07HA (hay results mean of 3 analyses)
USA, 2007 Ephrata, WA (Germains Covermate) Fescue	2	0.253 0.251	142 141	0 7	forage (23%DM) hay (88%DM)	6.641, 5.688 1.954, 1.957				RAGMP044 GM013-07HA
USA, 2007 Cornelius, OR (Orchard grass) Pasture Grass	2	0.254 0.262	165 175	0 7	forage (19%DM) hay (54%DM)	6.469, 7.808 15.378, 16.639				RAGMP044 GM014-07HA
USA, 2007 Albany, OR (Bravo) Fescue	2	0.259 0.262	120 121	0 7	forage (20%DM) hay (73%DM)	5.759, 5.492 14.583, 13.519 (c=16)				RAGMP044 GM016-07HA
USA, 2007 Larned, KS (Unknown) Bermuda grass	2	0.255 0.241	173 166	0 6	forage (41%DM) hay (78%DM)	21.398, 24.44 17.427, 17.254				RAGMP044 GM124-07HA
USA, 2007 Marion, AR (Tif 419) Bermuda grass	2	0.25 0.25	154 132	0 7	forage (38%DM) hay (68%DM)	29.025, 31.899 11.127, 9.966 (c=0.02)				RAGMP044 GM125-07HA
USA, 2007 Theilman, MN (Native) Bluegrass	2	0.251 0.254	175 178	0 7	forage (34%DM) hay (60%DM)	56.335, 55.501 6.183, 6.064 (c=0.11)				RAGMP044 GM126-07HA

GRASS FORAGE, HAY Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	no	kg ai/ha	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2007 Velva, ND (York) Bluegrass	2	0.25 0.247	131 130	0 6	forage (34%DM) hay (61%DM)	21.351, 25.532 6.271, 6.061				RAGMP044 GM127-07HA
USA, 2007 York, NE (Kentucky) Bluegrass	2	0.245 0.245	185 186	0 7	forage (23%DM) hay (69%DM)	29.562, 31.81 12.054, 12.767 (c=0.04)				RAGMP044 GM128-07HA
USA, 2007 Porterville, CA (Unknown) Fescue	2	0.251 0.251	171 173	0 7	forage (26%DM) hay (93%DM)	13.02, 10.626 3.042, 3.188 (c=0.27)				RAGMP044 GM129-07HA
USA, 2007 Athens, GA (Coastal) Bermuda grass	2	0.252 0.251	152 132	0 3 7 10 14 0 3 7 10 14	forage (41%DM) forage (38%DM) forage (38%DM) forage (38%DM) forage (50%DM) hay (71%DM) hay (84%DM) hay (82%DM) hay (88%DM) hay (76%DM)	29.743, 28.936 12.917, 12.15 9.251, 8.726 8.244, 8.385 8.775, 6.901 44.017, 45.405 22.575, 27.174 17.165, 17.174 16.39, 14.794 11.283, 11.041				RAGMP044 GM123-07DA
USA, 2007 Forest Grove, OR (Timothy) Pasture Grass	2	0.249 0.247	162 165	0 3 7 10 14 0 3 7 10 14	forage (21%DM) forage (20%DM) forage (18%DM) forage (22%DM) forage (23%DM) hay (43%DM) hay (39%DM) hay (53%DM) hay (81%DM) hay (69%DM)	8.609, 12.386 5.337, 8.242 2.969, 1.941 2.083, 2.292 1.683, 1.662 27.822, 25.755 19.248, 19.292 10.153, 8.162 7.758, 7.327 6.62, 5.264				RAGMP044 GM015-07DA

For hay, the reported PHIs were the intervals between the last application and cutting. Field samples were taken when hay had reached commercial dryness, 1-6 days after cutting. (c=from treated plot)

Tree nuts

Almonds

Results from supervised trials from USA on almonds were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to mature trees, 6–7 days apart as foliar sprays using ground-based airblast equipment to apply a total of 0.49–0.52 kg ai/ha/year. At each site, two application methods were used, one involving low volume sprays (468–561 L/ha) and one using high volume treatments (1956–2800 L/ha). Plot sizes in these trials ranged from 196–268 square metres.

Duplicate samples (nutmeat + shells and hulls) were taken from each plot, frozen within 3.5 hours of sampling, held in frozen storage for up to 654 days before the nut meat and hulls were analysed for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 174 Fluopyram residues in almond nutmeat and hulls from supervised trials in USA involving two foliar applications (500 SC formulations)

ALMOND Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		Nutmeat	Hulls	
USA, 2006 Hickman, CA (Carmel)	2 [b]	0.25 0.25	0.048 0.045	524 560	14	< 0.01, < 0.01	3.96, 4.09	RAGMP059 GM205-06HA Hulls DM = 90%
USA, 2006 Hickman, CA (Carmel)	2 [a]	0.244 0.247	0.01 0.01	2343 2372	14	< 0.01, < 0.01	4.75, 6.12	RAGMP059 GM205-06HA Hulls DM = 81%
USA, 2006 Kerman, CA (Non-pareil)	2 [b]	0.245 0.245	0.051 0.051	485 485	0 7 14 21 28	< 0.01, < 0.01 0.014, < 0.01 0.019, 0.019 0.016, 0.012 0.014, 0.013	1.96, 2.05 1.82, 1.9 1.93, 2.02 2.56, 3.12 3.09, 3.22	RAGMP059 GM201-06DA Hulls DM 24% @14d
USA, 2006 Kerman, CA (Non-pareil)	2 [a]	0.247 0.246	0.009 0.009	2799 2781	14	0.016, 0.014	1.22, 1.28	RAGMP059 GM201-06DA Hulls DM = 23%
USA, 2006 Madera, CA (Non-pareil)	2 [b]	0.251 0.252	0.045 0.045	558 561	14	< 0.01, < 0.01	4.24, 4.26	RAGMP059 GM202-06HA Hulls DM = 57%
USA, 2006 Madera, CA (Non-pareil)	2 [a]	0.259 0.259	0.013 0.013	1962 1964	14	< 0.01, 0.011	2.25, 1.94	RAGMP059 GM202-06HA Hulls DM = 46%
USA, 2006 Sanger, CA (Carmel)	2 [b]	0.25 0.249	0.053 0.053	470 472	14	< 0.01, < 0.01	1.97, 2.35	RAGMP059 GM203-06HA Hulls DM = 88%
USA, 2006 Sanger, CA (Carmel)	2 [a]	0.251 0.25	0.012 0.012	2044 2057	14	< 0.01, < 0.01	2.3, 2.58	RAGMP059 GM203-06HA Hulls DM = 91%
USA, 2006 Sutter, CA (Non-pareil)	2 [b]	0.25 0.249	0.053 0.053	468 468	14	< 0.01, < 0.01	2.57, 4.45	RAGMP059 GM204-06HA Hulls DM = 74%
USA, 2006 Sutter, CA (Non-pareil)	2 [a]	0.25 0.251	0.013 0.013	1962 1956	14	< 0.01, < 0.01	3.81, 3.46	RAGMP059 GM204-06HA Hulls DM = 65%

[a] = high volume airblast application

[b] = low volume airblast application

Pecans

Results from supervised trials from USA on pecans were provided to the Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to mature trees, 13–14 days apart as foliar sprays using ground-based airblast equipment to apply a total of 0.5–0.51 kg ai/ha/year. At each site, two application methods were used, one involving low volume sprays (385–647 L/ha) and one using high volume treatments (1914–2880 L/ha). Plot sizes in these trials ranged from 878–1021 square metres.

Duplicate samples were taken from each plot, frozen within 2.5 hours of sampling, held in frozen storage for up to 562 days before the nut meat was analysed for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg

Table 175 Fluopyram residues in pecan nutmeat from supervised trials in USA involving two foliar applications (500 SC formulations)

PECAN Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		Nutmeat	
USA, 2006 Albany, GA (Sumner)	2 [b]	0.254 0.249	0.059 0.065	431 385	14	< 0.01, 0.01	RAGMP059 GM207-06HA
USA, 2006 Albany, GA (Sumner)	2 [a]	0.253 0.253	0.011 0.013	2324 1914	14	< 0.01, < 0.01	RAGMP059 GM207-06HA
USA, 2006 Anton, TX (Western Schley)	2 [b]	0.253 0.249	0.047 0.047	539 529	14	0.021, 0.015	RAGMP059 GM210-06HA
USA, 2006 Anton, TX (Western Schley)	2 [a]	0.254 0.25	0.012 0.012	2167 2080	12	0.016, 0.045	RAGMP059 GM210-06HA
USA, 2006 Chula, GA (Sumner)	2 [b]	0.255 0.252	0.04 0.039	638 647	14	< 0.01, < 0.01	RAGMP059 GM206-06HA
USA, 2006 Chula, GA (Sumner)	2 [a]	0.256 0.254	0.009 0.009	2859 2879	14	< 0.01, < 0.01	RAGMP059 GM206-06DA
USA, 2006 Marked Tree, AR (Stuart)	2 [b]	0.253 0.26	0.051 0.05	497 520	0 7 14 21 28	0.044, 0.046 0.012, 0.011 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	RAGMP059 GM208-06DA
USA, 2006 Marked Tree, AR (Stuart)	2 [a]	0.253 0.246	0.011 0.01	2334 2413	14	< 0.01, < 0.01	RAGMP059 GM208-06DA
USA, 2006 Waller, TX (Kiowa)	2 [b]	0.251 0.252	0.055 0.056	456 454	13	< 0.01, < 0.01	RAGMP059 GM204-06HA
USA, 2006 Waller, TX (Kiowa)	2 [a]	0.251 0.25	0.012 0.012	2163 2155	13	< 0.01, < 0.01	RAGMP059 GM209-06HA

[a] = high volume airblast application

[b] = low volume airblast application

Oilseeds

Peanut

Results from supervised trials from USA on peanut were provided to the Meeting. In these trials, two applications of 0.242–0.256 kg ai/ha (SC 500 formulation) were applied to peanuts, 12–14 days apart as foliar sprays using CO_2 plot sprayers or knapsack sprayers with hand-held spray booms or tractor-mounted spray booms to apply 92–184 litres of spray mix/ha. Plot sizes in these trials ranged from 67–335 square metres.

At the specified PHIs, peanuts were dug and the hay was cut (same day), with both commodities allowed to dry to commercial dryness in the field or under cover for 2–17 days before duplicate samples (of at least 1 kg nutmeat and 0.5 kg hay) were taken, frozen within 4 hours of

sampling and held in frozen storage for up to 593 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with reported LOQs of 0.01 mg/kg (nutmeat) and 1.0 mg/kg (peanut hay).

Table 176 Fluopyram residues in peanut (nutmeat and hay) from supervised trials in USA involving two foliar applications (500 SC formulations).

PEANUT Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg, fresh weight)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Molino, FL (Georgia Green)	0.242 0.249	0.183 0.224	132 111	7 7	nutmeat hay (82%DM)	< 0.01, < 0.01 8.94, 9.2				RAGMP048 GM133-06HA
USA, 2006 Tifton, GA (C99R)	0.25 0.25	0.174 0.181	144 138	6 14	nutmeat hay (80%DM)	< 0.01, < 0.01 17.44, 18.28				RAGMP048 GM134-06HA
USA, 2006 Sufflok, VA (VA 98 R)	0.255 0.254	0.268 0.247	95 103	7 7	nutmeat hay (79%DM)	< 0.01, < 0.01 6.69, 6.71				RAGMP048 GM135-06HA
USA, 2006 Chula, GA (Georgia 02-C)	0.249 0.249	0.151 0.152	165 164	7 7	nutmeat hay (65%DM)	< 0.01, < 0.01 2.69, 2.6				RAGMP048 GM136-06HA
USA, 2006 Athens, GA (Georgia Green)	0.246 0.251	0.178 0.186	138 135	7 7	nutmeat hay (75%DM)	< 0.01, < 0.01 18.37, 19.43				RAGMP048 GM137-06HA
USA, 2006 Sycamore, GA (Georgia Greens)	0.251 0.253	0.16 0.154	157 164	7 7	nutmeat hay (74%DM)	< 0.01, < 0.01 5.68, 5.05				RAGMP048 GM138-06HA
USA, 2006 Chula, GA (Georgia Green)	0.25 0.253	0.159 0.153	157 165	7 7	nutmeat hay (75%DM)	< 0.01, < 0.01 12.40, 10.47				RAGMP048 GM139-06HA
USA, 2006 Oviedo, FL (Valencia A 1)	0.256 0.252	0.139 0.14	184 180	7 7	nutmeat hay (75%DM)	0.017, 0.018 2.94, 2.93				RAGMP048 GM140-06HA
USA, 2006 Pearsall, TX (Florunner)	0.244 0.252	0.145 0.145	168 174	7 7	nutmeat hay (83%DM)	0.012, 0.01 3.64, 3.73				RAGMP048 GM141-06HA
USA, 2006 East Bernard, TX (Tamspan 90)	0.251 0.252	0.189 0.274	133 92	7 7	nutmeat hay (82%DM)	< 0.01, < 0.01 4.48, 3.85				RAGMP048 GM142-06HA
USA, 2006 Levelland, TX (Tamspan 90)	0.248 0.249	0.175 0.182	142 137	7 7	nutmeat hay (58%DM)	< 0.01, < 0.01 1.34, 1.08				RAGMP048 GM143-06HA
USA, 2006 Seven Springs, NC (Perry)	0.252 0.25	0.171 0.162	147 154	0 2 6 9 13 0 2 6 9 13	nutmeat nutmeat nutmeat nutmeat nutmeat hay (48%DM) hay (73%DM) hay (59%DM) hay (64%DM) hay (68%DM)	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 23.94, 29.13 33.25, 31.37 19.44, 21.88 8.97, 12.03 10.24, 12.35				RAGMP048 GM132-06DA

Rape seed

Results from supervised trials on winter and summer rape in Europe were provided to the Meeting. In these trials, 2 applications of fluopyram (SC 500 formulation or as a 250 SC formulation, in combination with prothioconazole) were applied to rape plants at BBCH 63 (30% flowers open) and again, 14–39 days later, at BBCH 73 (30% pods at full size), as foliar sprays using knapsack sprayers with spray booms (3–12 flat fan nozzles), applying 0.125 kg ai/ha in 200–400 litres water/ha. Plot sizes in these trials ranged from 48–563 square metres.

Unreplicated samples of mature seeds (minimum 1 kg), pods and seeds (minimum 0.7 kg) and/or plants (minimum 2 kg green material, including pods and seeds) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 376 days (423 days for plants) before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984-M001. The reported LOQs were 0.01 mg/kg for each analyte

Table 177 Residues in oilseeed rape from supervised trials in France, Germany, Italy and UK, involving two foliar applications of fluopyram

RAPE SEED Country, year Location (variety)	Application					PHI, (days)	Residues (mg/kg)					Reference & Comments
	form	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Germany, 2006 Werl-Westönnen (Smart) Rape, winter	500 SC	2	0.125	0.0415	300	-0 0 12 41 50	plant plant plant plant seed	0.06 1.6 0.41 0.1 0.11	0.01 0.01 0.02 < 0.01 0.01	0.01 0.01 0.05 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2609/06 0406-06
France (N), 2006 Beuvraignes (Salomon) Rape, winter	500 SC	2	0.125	0.05	250	-0 0 29 54 66	plant plant plant plant seed	0.06 1 0.12 0.04 0.02	0.01 0.01 0.01 < 0.01 < 0.01	0.01 0.02 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2609/06 0408-06
Germany, 2006 Burscheid (Talent) Rape, winter	500 SC	2	0.125	0.0415	300	-0 0 57	plant plant seed	0.07 1.6 0.04	< 0.01 < 0.01 < 0.01	0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2609/06 0409-06
United Kingdom, 2006 Royston (Canberra) Rape, winter	500 SC	2	0.125	0.0625	200	-0 0 43	plant plant seed	0.04 1.4 0.1	< 0.01 < 0.01 0.01	< 0.01 < 0.01 0.04	< 0.01 < 0.01 < 0.01	RA-2609/06 0410-06
France (N), 2007 Braslou (Grizzly) Rape, winter	250 SC	2	0.125	0.0416	300	-0 0 14 36 50	plant plant plant pod seed	0.27 0.98 0.45 0.3 0.09	0.02 0.02 0.01 0.01 0.01	0.04 0.06 0.05 0.07 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2616/07 0238-07
Germany, 2007 Burscheid (Elektra) Rape, winter	250 SC	2	0.125	0.0416	300	-0 0 14 49 69	plant plant plant pod seed	0.45 0.91 0.36 0.07 0.08	< 0.01 0.01 0.01 < 0.01 < 0.01	0.03 0.03 0.03 0.03 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2616/07 0808-07

RAPE SEED Country, year Location (variety)	Application					PHI, (days)	Residues (mg/kg)					Reference & Comments
	form	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Germany, 2007 Werl-Westönnen (Oase) Rape, winter	250 SC	2	0.125	0.0416	300	-0 0 17 44 61	plant plant plant pod seed	0.05 0.75 0.29 0.37 0.11 (c=0.02	0.02 0.02 0.01 0.03 0.01	0.03 0.04 0.03 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2616/07 0809-07
United Kingdom, 2007 Near Eye (Es Astrid) Rape, winter	250 SC	2	0.125	0.0416	300	-0 0 25 46 57	plant plant plant pod seed	0.18 0.91 0.31 0.13 0.19 (c=0.01	0.04 0.02 0.03 0.02 0.02	0.06 0.05 0.07 0.02 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2616/07 0810-07
France (S), 2006 Latille (Savana) Rape, winter	500 SC	2	0.125	0.0415	300	-0 0 11 35 50	plant plant plant plant seed	0.23 1.1 0.2 0.09 0.06	0.03 0.04 0.02 0.02 0.01	0.03 0.05 0.03 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2610/06 0625-06
Italy, 2006 Bologna (Molino) Rape, summer	500 SC	2	0.125	0.038	330	-0 0 20 27 34	plant plant plant plant seed	0.25 1.1 0.1 0.35 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2610/06 0626-06
France (S), 2007 Gargas (Corail) Rape, winter	250 SC	2	0.125	0.0416	300	-0 0 10 59	plant plant plant seed	1.1 0.95 0.31 0.1	0.01 < 0.01 < 0.01 < 0.01	0.07 0.04 0.04 0.01	< 0.01 < 0.01 < 0.01 < 0.01	RA-2617/07 0239-07
Italy, 2007 Conselice (Belcanto) Rape, winter	250 SC	2	0.125	0.0313	400	-0 0 14 30 40	plant plant plant pod seed	0.26 0.87 0.71 0.71 0.14	0.02 0.02 0.02 0.01 0.01	0.06 0.07 0.1 0.11 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2617/07 0811-07

Results from supervised trials from USA on oilseed rape (canola) were provided to the Meeting. In these trials, two applications of 0.242–0.256 kg ai/ha (SC 500 formulation) were applied to canola, 13–14 days apart from flowering as foliar sprays using CO_2 plot sprayers or knapsack sprayers with hand-held spray booms, field sprayers or tractor-mounted spray booms to apply 92–184 litres of spray mix/ha. Plot sizes in these trials ranged from 70–210 square metres.

Duplicate samples of rape seed (minimum 1 kg) were taken from at least 12 areas within each plot, frozen within 3 hours of sampling and held in frozen storage for up to 516 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 178 Fluopyram residues in rape seed from supervised trials in USA involving two foliar applications (500 SC formulations)

RAPE SEED Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Sycamore, GA (Hyola)	2	0.25 0.242	0.184 0.161	136 150	13	0.122, 0.14				RAGMP035 GM012-06HA
USA, 2006 Sabin, MN (5630)	2	0.256 0.252	0.16 0.144	160 175	14	0.093, 0.099				RAGMP035 GM014-06HA
USA, 2006 Eledridge, ND (Hyola357RR)	2	0.252 0.252	0.187 0.177	135 142	14	0.105, 0.112				RAGMP035 GM015-06HA
USA, 2006 Velva, ND (Hyola 357RR Magnum)	2	0.253 0.248	0.209 0.203	121 122	14	0.219, 0.174				RAGMP035 GM016-06HA
USA, 2006 Jerome, ID (Phoenix)	2	0.247 0.249	0.143 0.15	173 166	12	2.771, 3.003				RAGMP035 GM017-06HA
USA, 2006 Madras, OR (CrackerJack)	2	0.252 0.25	0.215 0.216	117 116	14	0.436, 0.414				RAGMP035 GM018-06HA
USA, 2006 Ephrata, WA (47755 / 65037)	2	0.248 0.248	0.14 0.139	177 178	14	0.089, 0.135				RAGMP035 GM019-06HA
USA, 2006 Springfield, NE (Invigor 4870 LL)	2	0.249 0.248	0.196 0.195	127 127	0 6 12 19 26	1.62, 1.598 0.203, 0.188 0.139, 0.146 0.123, 0.157 0.2, 0.175				RAGMP035 GM013-06DA

Sunflower seed

Results from supervised trials from USA on sunflowers were provided to the Meeting. In these trials, two applications of 0.235–0.253 kg ai/ha (SC 500 formulation) were applied to sunflower plants, 12–14 days apart as foliar sprays using knapsack or CO_2 plot sprayers with hand-held or tractor-mounted spray booms to apply a 121–190 litres spray mix/ha. Plot sizes in these trials ranged from 46–149 square metres.

Duplicate samples (minimum 1 kg seed) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 579 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 179 Fluopyram residues in sunflower seed (dried) from supervised trials in USA involving two foliar applications (500 SC formulations)

SUNFLOWER SEED Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Sabin, MN (Dekalb DKF38-80CL)	2	0.242 0.25	0.14 0.149	173 168	13	0.073, 0.078				RAGMP070 GM339-06HA

SUNFLOWER SEED Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Springfield, NE (Mycogen 8N429CL)	2	0.251 0.253	0.192 0.192	131 132	12	0.014, 0.008				RAGMP070 GM340-06HA
USA, 2006 Velva, ND (DKF38-80CL)	2	0.249 0.248	0.204 0.205	122 121	14	0.227, 0.214				RAGMP070 GM341-06HA
USA, 2006 Grand Island, NE (Garst 4704 NS)	2	0.251 0.248	0.132 0.132	190 188	14	0.251, 0.246				RAGMP070 GM342-06HA
USA, 2006 Eldridge, ND (DKF29-90)	2	0.25 0.249	0.16 0.154	156 162	14	0.02, 0.02				RAGMP070 GM343-06HA
USA, 2006 New Rockford, ND (Pioneer 63M80)	2	0.237 0.249	0.156 0.154	152 162	14	0.058, 0.055				RAGMP070 GM344-06HA
USA, 2006 Larned, KS (Pioneer 63M91-N402)	2	0.25 0.253	0.148 0.148	169 171	13	0.029, 0.077				RAGMP070 GM345-06HA
USA, 2007 Carlyle, IL (Hybrid Lot 1)	2	0.25 0.253	0.169 0.169	148 150	1 7 14 22 27	0.775, 0.583 0.674, 0.998 0.479, 0.284 0.292, 0.185 0.108, 0.133				RAGMP070 GM338-06DB

Hops, dry

Results from supervised trials from USA on hops were provided to the Meeting. In these trials, two applications of 0.246–0.26 kg ai/ha (SC 500 formulation) were applied to mature hop vines, 13–14 days apart as foliar sprays using ground-based airblast equipment to apply 468–525 litres spray mix/ha. Plot sizes in these trials ranged from 65–91 square metres.

Duplicate samples of ripe cones (minimum 0.5 kg) were harvested, allowed to dry to commercial dryness (oven-dried for 12 hours, hot-air dried for 3.5 hours or air-dried for 24 hours), collected, frozen within 4 hours of collection and held in frozen storage for up to 545 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.05 mg/kg.

Table 180 Fluopyram residues in hops (dried) from supervised trials in USA involving two foliar applications (500 SC formulations)

HOPS, DRY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Greenleaf, ID (Zeus)	2	0.254 0.26	0.053 0.053	478 487	7	6.45, 6.97				RAGMP045 GM127-06HA
USA, 2006 Yakim, WA (Warrior)	2	0.249 0.25	0.053 0.053	468 470	7	4.32, 7.28				RAGMP045 GM128-06HA

HOPS, DRY Country, year Location (variety)	Application				PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2006 Hillsboro, OR (Glacier)	2	0.247 0.248	0.047 0.049	525 508	7	25.7, 25.03				RAGMP045 GM129-06HA

Herbs

Basil

Results from supervised trials from USA on basil were provided to the Meeting. In these trials, unreplicated plots were treated with 2 foliar sprays of an SC 500 formulation of fluopyram (tank mixed with trifloxystrobin), using knapsack sprayers to apply 0.246–0.262 kg ai/ha in 140–204 L water/ha, with a treatment interval of 7 days. Plot sizes ranged from 59–116 square metres.

Duplicate samples of at least 0.45 kg fresh leaves and at least 0.2 kg dried leaves (after 5–12 days drying) were taken from 12 plants in each treated plot, frozen within 2 hours of sampling and stored frozen for up to 395 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with reported LOQs of 0.1 mg/kg for fresh basil leaves and 0.25 mg/kg for dried basil leaves.

Table 181 Fluopyram residues in fresh and dried basil leaves from supervised trials in USA, involving two foliar applications (500 SC formulation).

BASIL Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2007 Suffolk, VA (Ceasar)	2	0.253 0.253	0.204 0.202	124 125	0 0	fresh dried	19.13, 19.58 92.49, 99.22				RAGMP083 GM067-07HA
USA, 2007 Sanger, CA (Italian Large Leaf)	2	0.246 0.252	0.156 0.158	158 159	0 0	fresh dried	32.09, 27.91 175, 187				RAGMP083 GM068-07HA
USA, 2007 Germansville, PA (Unknown)	2	0.257 0.262	0.140 0.144	183 182	0 1 3 7 10 0 1 3 7 10	dried dried dried dried dried fresh fresh fresh fresh fresh	90.88, 90.4 64.19, 63.75 9.17, 8.96 4.38, 4.23 3.69, 3.59 15.808, 21.747 14.017, 16.229 1.366, 1.344 0.864, 0.889 0.624, 0.639				RAGMP083 GM066-07DA

Chives

Results from supervised trials from USA on chives were provided to the Meeting. In these trials, unreplicated plots were treated with 2 foliar sprays of an SC 500 formulation of fluopyram (tank mixed with trifloxystrobin), using knapsack or plot sprayers to apply 0.251–0.263 kg ai/ha in 141–187 litres water/ha, with a treatment interval of 7 days.

Duplicate samples of at least 1 kg chive plants (without roots) were taken from 24 plants in each treated plot, frozen within 2 hours of sampling and stored frozen for up to 395 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 182 Fluopyram residues in chives from supervised trials in USA, involving two foliar applications (500 SC formulation)

CHIVES Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Unknown)	2	0.263 0.254	0.141 0.145	187 175	0	5.866, 6.235				RAGMP083 GM069-07HA
USA, 2007 Fresno, CA (Staro)	2	0.255 0.253	0.178 0.179	143 141	0	18.7, 20.9				RAGMP083 GM071-07HA
USA, 2007 Hudson, KS (Earl May)	2	0.251 0.252	0.146 0.146	172 173	0	7.754, 7.906				RAGMP083 GM070-07HA

Dill

Results from supervised trials from USA on dill (seed) were provided to the Meeting. In these trials, unreplicated plots were treated with 2 foliar sprays of an SC 500 formulation of fluopyram (tank mixed with trifloxystrobin), using knapsack sprayers to apply 0.243–0.261 kg ai/ha in 125–186 litres water/ha, with a treatment interval of 7 days. Plot sizes ranged from 74–118 square metres.

Dill seed heads were harvested and allowed to dry in the field for up to 9 days before duplicate samples of at least 0.2 kg dill seed were taken, frozen within 1 hour of sampling and stored frozen for up to 223 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.1 mg/kg.

Table 183 Fluopyram residues in dried dill seed from supervised trials in USA, involving two foliar applications (500 SC formulation).

DILL SEED Country, year Location (variety)	Application				PHI, (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 Oviedo, FL (Unknown)	2	0.261 0.252	0.14 0.141	186 179	14	8.862, 9.462				RAGMP106 GM106-07HA
USA, 2007 Sanger, CA (Mammoth)	2	0.243 0.249	0.14 0.147	174 169	14	29.29, 25.47				RAGMP106 GM107-07HA
USA, 2007 Parkdale, OR (Long Island Mammoth)	2	0.256 0.247	0.205 0.166	125 149	13	31.2, 27.98				RAGMP106 GM108-07HA

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

In storage

No information was provided on the fate of fluopyram residues under commercial storage conditions

In processing

Nature of the Residue in Processing

The hydrolytic behaviour of fluopyram and major metabolites under simulated processing conditions was investigated in a series of five aqueous hydrolysis studies with radio-labelled fluopyram and metabolites.

Samples of standard buffer solutions at pH values of 4, 5 and 6 were fortified with [phenyl-UL-^{14}C] or [pyridyl-2,6-^{14}C] fluopyram and metabolites. The fortified samples (1.0 mg ai/L) were subjected to hydrolysis conditions reflecting pasteurisation, baking/brewing/boiling and sterilization.

No degradation of fluopyram, BZM, 7-OH or PCA was observed in these studies. Between 2% and 6% of the applied PAA radio-label was found in the head space of the test vials.

Analysis of the PAA samples (after vigorous shaking with acetonitrile to capture the volatile degradates) identified the transformation -picoline breakdown product as the major residue, this being up to 96.7% AR (pasteurisation scenario), up to 94.5% AR (baking, brewing, boiling scenario) and up to 76% AR (sterilization scenario). Residues of the PAA metabolite were only found (18.8%) in the sterilization scenario samples.

Table 184 Stability of fluopyram and metabolites under hydrolysis conditions simulating pasteurisation; baking, brewing, boiling; and sterilisation

Test item [Reference]	Hydrolysis conditions	Initial conc	Post-processing conc		Degradates
		mg ai/Litre	mg ai/Litre	%AR	
Fluopyram (phenyl-label) [Koenig, 2006 Ref: MEF-06/170]	pH 4–90 ˚C–20 min	1.0	1.0	100.4	
	pH 5–100 ˚C–60 min	1.0	1.1	100.9	
	pH 6–120 ˚C–20 min	1.0	1.0	102.2	
Fluopyram (pyridyl-label) [Koenig, 2006 Ref: MEF-06/170]	pH 4–90 ˚C–20 min	1.1	1.1	99.4	
	pH 5–100 ˚C–60 min	1.1	1.1	99.2	
	pH 6–120 ˚C–20 min	1.1	1.1	102.3	
-benzamide (BZM) [Menke, 2006, Ref: MEF-06/273]	pH 4–90 ˚C–20 min	1.09	1.09	99.8	
	pH 5–100 ˚C–60 min	1.10	1.08	97.3	
	pH 6–120 ˚C–20 min	1.07	1.15	107.8	
-7-hydroxy (7-OH) [Heinemann, 2006, Ref: MEF-06/349]	pH 4–90 ˚C –20 min	1.0	1.1	100.7	
	pH 5–100 ˚C –60 min	1.1	1.1	102.0	
	pH 6–120 ˚C–20 min	1.0	1.1	102.1	
-pyridyl-carboxylic acid (PCA) [Menke, 2006, Ref: MEF-06/358]	pH 4–90 ˚C–20 min	1.19	1.11	93.1	
	pH 5–100 ˚C–60 min	1.06	1.12	106.1	
	pH 6–120 ˚C–60 min	1.08	1.07	99.5	
-pyridyl-acetic acid (PAA) [Heinemann, 2006, Ref: MEF-06/354].	pH 4–90 ˚C–20 min	0.99	0.90	91.1	picoline (96.7% AR)
	pH 5–100 ˚C–60 min	1.00	0.90	90.2	picoline (94.5% AR)
	pH 6–120 ˚C–20 min	0.99	0.92	93.1	picoline (76% AR)

Fluopyram and its 7-OH, BZM and PCA metabolites are stable under the three simulated processing conditions. Under acidic test conditions the PAA metabolite can be transformed to fluopyram-picoline.

Magnitude of the residue in processing

Information was provided to the Meeting on the residue distribution of fluopyram in peel and pulp (melons), on the effects of trimming, washing and cooking of strawberries, blueberries, cabbage, broccoli, summer squash, lettuce, spinach, Mustard greens, celery and on the effects of simulated commercial processing on residues of fluopyram and metabolites in oranges, apples, plums, grapes,

tomatoes, potatoes, sugar beet, soya beans, wheat, maize, peanut, rape seed, cotton seed and sunflower seed.

Peel and pulp residue distribution

Melon and watermelon

In a series of field trials conducted in Europe on melons and water melons, residues in whole fruit, pulp and in peel were analysed separately to investigate the distribution of residues of fluopyram and metabolites.

Table 185 Residues in melon fruit, peel and pulp from supervised greenhouse trials in France, Germany, Italy, Portugal and Spain involving foliar applications of fluopyram (500 SC formulation)

MELONS Country, year Location (variety)	Application			PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL		matrix	parent	PAA	BZM	PCA	
France, 2006 Carpentras (Lunastar)	2	0.3	0.03	3 3 3	fruit pulp peel	0.19 0.01 0.38	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2587/06 0368-06
Spain, 2006 Roquetas de Mar (Vulcano)	2	0.3	0.03	3 3 3	fruit pulp peel	0.02 < 0.01 0.05	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2587/06 0585-06
Italy, 2006 Manfredonia (Proteo)	2	0.3	0.0375	3 3 3	fruit pulp peel	0.11 < 0.01 0.21	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2587/06 0586-06
Germany, 2006 Euskirchen-DomEsch (Haon)	2	0.3	0.05	3 3 3	fruit pulp peel	0.17 < 0.01 0.26	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2587/06 0587-06
France, 2006 Castelsarrasin (Edgar)	2	0.3	0.03	3 3 3	fruit pulp peel	0.16 0.02 0.76	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2587/06 0589-06
Portugal, 2006 São Bartolomeu-Lourinhã (Jaliscas)	2	0.3	0.03	3 3 3	fruit pulp peel	0.09 < 0.01 0.16	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2587/06 0591-06
Spain, 2007 Sanlucar de Barrameda (Primal) Musk melon	2	0.3	0.03	3 3 3	fruit pulp peel	- 0.01 0.23	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2680/07 0877-07

Table 186 Residues in melon fruit, peel and pulp from supervised outdoor trials in France, Germany, Greece, Italy, Portugal and Spain involving foliar applications of fluopyram (500 SC formulation)

MELONS Country, year Location (variety)	Application			PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL		matrix	parent	PAA	BZM	PCA	
France, 2006 Antogny le Tillac (Edgar)	2	0.25	0.025	3 3 3	fruit pulp peel	0.12 < 0.01 0.21	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2588/06 0370-06
Germany, 2006 Monheim (Summer dream F1)	2	0.25	0.05	3 3 3	fruit pulp peel	0.11 0.01 0.31	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2588/06 0592-06
France, 2006 Scorbe clairvaux (Edgar)	2	0.25	0.0415	3 3 3	fruit pulp peel	0.08 < 0.01 0.22	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2589/06 0371-06

MELONS Country, year Location (variety)	Application			PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL		matrix	parent	PAA	BZM	PCA	
Greece, 2006 Epanomi (Sterlina)	2	0.25	0.05	3 3 3	fruit pulp peel	- 0.02 0.14	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2589/06 0593-06
Spain, 2006 Alginet (Charentais)	2	0.25	0.0315	3 3 3	fruit pulp peel	0.05 < 0.01 0.06	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2589/06 0594-06
Italy, 2006 Bologna (Summerdream)	2	0.25	0.0415	3 3 3	fruit pulp peel	- < 0.01 0.07	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2589/06 0595-06
Greece, 2006 Aronas-Pieria (Velos FI)	2	0.3	0.06	3 3 3	fruit pulp peel	- < 0.01 0.5	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2587/06 0590-06
Greece, 2007 Aronas-Katerini (Velos)	2	0.25	0.05	3 3 3 7 7 7	fruit pulp peel fruit pulp peel	0.21 < 0.01 0.3 0.16 < 0.01 0.26	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2508/07 0009-07
Portugal, 2007 Caniceira-Vale de Cavalos (Branco do Ribatejo)	2	0.25	0.05	3 3 3 7 7 7	fruit pulp peel fruit pulp peel	0.08 < 0.01 0.15 0.05 < 0.01 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2508/07 0535-07

Table 187 Residues in watermelon fruit, pulp and peel from supervised outdoor trials in Italy and Spain involving foliar applications of fluopyram (500 SC formulation).

WATERMELON Country, year Location (variety)	Application			PHI, (days)	Residues (mg/kg)					Reference & Comments
	no	kg ai/ha	kg ai/hL		matrix	parent	PAA	BZM	PCA	
Italy, 2007 Lazio (Topgun F1)	2	0.25	0.0315	3 3 3 7 7 7	fruit pulp peel fruit pulp peel	0.06 < 0.01 0.08 0.06 < 0.01 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.01 < 0.01 0.02	RA-2508/07 0010-07
Spain, 2007 Negra (Pata)	2	0.25	0.025	3 3 3 7 7 7	fruit pulp peel fruit pulp peel	0.08 < 0.01 0.16 0.08 0.01 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02	RA-2508/07 0536-07

Similar studies were conducted in USA, where muskmelons from supervised field trials were peeled (muskmelon), simulating household practices and fluopyram residues were measured in the peeled fruit.

Table 188 Fluopyram residues in peeled musk melons from a supervised outdoor trial in USA involving two applications of fluopyram (500 SC formulation).

MELON Country, year Location (variety)	Application			PHI, (days)		Fluopyram Residues (mg/kg)				Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)			parent	PAA	BZM	PCA	
USA, 2007 Orland, CA (Hales Best) Foliar spray	0.248 0.251	0.177 0.178	140 141	0	whole fruit peeled fruit	0.163, 0.15, 0.14 < 0.01 (3)				RAGMP082 GM058-07DA
USA, 2007 Orland, CA (Hales Best) Drip irrigation	0.25 0.25	25 25	1 [a] 1 [a]	7	whole fruit peeled fruit	< 0.01 (3) < 0.01 (3)				RAGMP082 GM058-07DA

[a] = Litres/hour/emitter

Household washing, trimming and cooking

Strawberries

Strawberries from four European field trials were processed into preserve and jam using simulated household practices. In these trials, foliar spray applications of an SC 500 formulation of fluopyram were made at the start of ripening (BBCH 87) and repeated 3 days before commercial harvest, with spray intervals of 7 days. Applications were made using backpack sprayers with single nozzle hand lances or mini-booms (8–12 flat-fan nozzles, applying 0.25 kg ai/ha in 300–1000 L water/ha. Plot sizes in these trials ranged from 25–60 square metres.

Unreplicated samples of 4–5 kg fruit were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 221 days (fruit) and 46 days (processed matrices) before analysis for fluopyram and its BZM, PCA and PAA metabolites using Method 00984/M001. The reported LOQs were 0.01 mg/kg for each analyte.

Deep frozen strawberries (without sepals) were washed in lukewarm standing water. A part of the washed fruits was minced in a mixer and the other portion was cut into small pieces. Both portions were mixed and cooked together with jell-sugar.

Table 189 Residues in field strawberries, preserve and jam from supervised trials in Belgium, France and Spain, involving 2 foliar applications of fluopyram (500 SC formulation) - 1 day PHI

STRAWBERRY Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
France, 2006 Cremery (Darselect)	0.25	0.0835	300	fruit washed fruit preserve jam washings	0.24 0.2 0.08 0.14 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3600/06 R 2006 0384 3 0384-06
Belgium, 2006 Ittre (Elsanta)	0.25	0.028	900	fruit washed fruit preserve jam washings	0.36 0.22 0.09 0.1 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3600/06 R 2006 0403 3 0403-06

STRAWBERRY Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
France, 2006 Monteux (Pajaro)	0.25	0.025	1000	fruit washed fruit preserve jam washings	0.27 0.22 0.08 0.17 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3601/06 R 2006 0387 8 0387-06
Spain, 2006 Paterna Huelva (Ventana)	0.25	0.025	1000	fruit washed fruit preserve jam washings	0.25 0.23 0.1 0.16 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 < 0.01 < 0.01 < 0.01	RA-3601/06 R 2006 0404 1 0404-06

Strawberries from one USA field trial involving plots treated by foliar application and via drip irrigation were collected to determine possible residue reduction from washing strawberries and from cooking strawberries. In this trial, either 2 foliar spray applications or two drip irrigation treatments of an SC 500 formulation of fluopyram were made 5 days apart, to plots of about 94 square metres.

Bulk samples of at least 3.6 kg fruit were taken from each plot and delivered chilled to the laboratory for processing the next day. The washing and cooking procedures were performed in a manner similar to normal household practice.

For the washed sub-sample, the stems and leaves were removed by hand, the strawberries were rinsed under running tap water for about 30 seconds, drained sealed in a plastic bag and stored frozen until analysed. Washed strawberries were sliced and then crushed using a potato masher before boiling for 1 minute with pectin, canned and immersed in a boiling water bath for 5 minutes. The sealed canning jars were cooled and stored frozen until analysis. Fluopyram residues were analysed using LC/MS/MS Method GM-001-P07-01. The reported LOQ was 0.01 mg/kg.

Table 190 Fluopyram residues in fresh, washed and cooked strawberries from a supervised field trial in USA, involving 2 foliar or drip irrigation applications of fluopyram (500 SC formulation)

STRAWBERRIES Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	Water (L/ha)	fruit	parent	PAA	BZM	PCA	
USA, 2007 Sanger, CA (Camarosa) Foliar application	0.253 0.255	0.17 0.15	149 L/ha 172 L/ha	unwashed washed cooked [a]	0.44, 0.46, 0.43 0.32, 0.33, 0.34 0.31, 0.35, 0.33				RAGMP084 GM078-07DA 0 day PHI
USA, 2007 Sanger, CA (Camarosa) via drip irrigation	0.25 0.25		1.4 L/hr 1.4 L/hr	unwashed cooked [a]	0.014 (2), 0.015 0.014 (2), 0.015				RAGMP084 GM078-07DA 7 day PHI

[a] washed, cooked strawberries

Blueberries

Blueberries from one USA trial were washed and cooked to simulate household practices. In this trial, 2 applications of fluopyram + pyrimethanil (SC 300 + 300) at a rate 0.25kg ai/ha fluopyram in 460–500 L water/ha of were applied at 7 day intervals as a simulated airblast treatment (knapsack).

Duplicate samples of 4 kg fruit were taken from each plot and subsamples were either rinsed in warm water for 30 seconds and drained or were rinsed, simmered for 30 seconds in water, drained

and cooked in sugar syrup for 15 minutes and sealed. The processed samples were stored at -18 °C for about 210 days before HPLC-MS/MS analysis for fluopyram using method GM-001-P07-01.

Table 191 Residues in washed and cooked blueberries from a supervised trial in USA, involving two foliar applications of fluopyram (SC formulations)- 0 day PHI

BLUEBERRY Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2007 Ochlocknee, GA (Tifblue)	0.25 0.25	0.054 0.05	466 508	berries washed berries cooked berries	0.533, 0.507, 0.463 0.324, 0.325, 0.325 0.21, 0.21, 0.201				RAGMP037 GM002-07DA

Cabbage

Washed and cooked cabbage heads were produced from cabbages obtained from four European field trials. In these trials, 2 applications of fluopyram + tebuconazole (SC 200+200) at rates 0.2kg ai/ha fluopyram in 300–600 litres water/ha of were applied at 14–15 day intervals as foliar sprays using knapsack sprayers with hand-held booms (3–12 flat fan or hollow cone nozzles). Plot sizes in these trials ranged from 50–80 square metres.

Unreplicated samples of 12 cabbage heads, divided into quarters, were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 214 days before being defrosted, washed and cooked in salted water for about 40 minutes.

Raw, washed and cooked cabbages as well as the washing and cooking water were analysed for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984/M001. LOQs were 0.01 mg/kg for each analyte in all matrices).

Table 192 Residues in washed and cooked cabbage heads from supervised trials in France, Germany and Italy, involving two foliar applications of fluopyram (SC formulations)- 7 day PHI

CABBAGE Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
France, 2006 Fondettes (Eton F1) Cabbage, white	0.2	0.0334	600	heads washed heads cooked heads washing water cooking water	0.02 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3577/06 0354-06
Germany, 2006 Leichlingen (Latima) Cabbage, white	0.2	0.0666	300	heads washed heads cooked heads washing water cooking water	0.02 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3577/06 0478-06
France, 2006 Arnas (Count) Cabbage, white	0.2	0.04	500	heads washed heads cooked heads washing water cooking water	0.04 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3578/06 0357-06
Italy, 2006 Ladispoli (Castello) Cabbage, white	0.2	0.0334	600	heads washed heads cooked heads washing water cooking water	0.06 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.01 < 0.01 < 0.01 < 0.01	RA-3578/06 0479-06

Cabbages were collected from six USA field trials to estimate the residue distribution of fluopyram in cabbage heads with and without wrapper leaves. In these trials, two foliar applications of 0.25–0.26 kg ai/ha fluopyram (SC 500 formulation), tank-mixed with trifloxystrobin (SC) in 92–188 litres of water were applied to cabbage plants 5 days apart using CO_2 pressurised plot sprayers or knapsack sprayers. Plot sizes in these trials ranged from 46–181 square metres.

Duplicate samples of at least 12 cabbage heads (with and without wrapper leaves) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 415 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 193 Fluopyram residues in cabbage heads (with and without wrapper leaves) from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations) – 0 day PHI

CABBAGE Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Blue Lagoon)	0.257 0.259	0.141 0.14	182 185	with wrapper leaves no wrapper leaves	0.708, 0.626 0.02, < 0.01				RAGMP076 GM023-07DA
USA, 2007 Athens, GA (Bonnies Best Hybrid)	0.252 0.249	0.257 0.271	98 92	with wrapper leaves no wrapper leaves	0.980 1.054 0.04, 0.02				RAGMP076 GM024-07HA
USA, 2007 Belle Glade, FL (Emblem)	0.253 0.253	0.143 0.135	177 188	with wrapper leaves no wrapper leaves	1.6, 0.929 0.079, 0.061				RAGMP076 GM025-07HA
USA, 2007 Richland, IA (Stonehead)	0.254 0.248	0.164 0.16	155 155	with wrapper leaves no wrapper leaves	1.253, 0.751 0.024, < 0.01				RAGMP076 GM026-07HA
USA, 2007 Uvalde, TX (Blue Thunder)	0.251 0.25	0.136 0.139	184 180	with wrapper leaves no wrapper leaves	0.254, 0.119 0.015, 0.019				RAGMP076 GM027-07HA
USA, 2007 Corning, CA (Copenhagen)	0.251 0.25	0.134 0.134	187 187	with wrapper leaves no wrapper leaves	0.054, 0.064 0.018, 0.022				RAGMP076 GM028-07HA

Broccoli

Broccoli heads were collected from one USA field trial to estimate the effect of washing and cooking on the residue distribution of fluopyram in broccoli. In this trial, two foliar applications of fluopyram (SC 500 formulation) tank mixed with trifloxystrobin (SC 500) were applied to celery plants at 0.25 and 0.26 kg ai/ha in 136 and 163 litres of water, 5 days apart, using a knapsack sprayer and with a plot size of 159 square metres.

Bulk samples of broccoli (15 units) were collected once the last spray application had dried, chilled and delivered to the laboratory where sub-samples (after removal of leaves) were processed the next day (tap-washed three times, shaken, drained, quartered and cooked in boiling water for 8 minutes) and the samples frozen for up to 427 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 194 Fluopyram residues in fresh, washed and cooked broccoli from a supervised trial in USA involving two foliar applications of fluopyram (500 SC formulations) – 0 day PHI

BROCCOLI Country, year Location (variety)	Application				Fluopyram Residues (mg/kg)				Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2007 Sanger, CA (Marathon)	0.25 0.257	0.184 0.158	136 163	fresh washed washed & cooked	0.994, 0.991, 0.951 0.765, 0.782, 0.791 0.581, 0.621, 0.608				RAGMP076 GM019-07DA

Summer squash

In two of the field trials conducted in USA on summer squash, samples were washed and cooked, simulating household practices and fluopyram residues were measured in the washed and cooked squash.

Table 195 Fluopyram residues in washed and/or cooked summer squash from a supervised outdoor trial in USA involving two applications of fluopyram (500 SC formulation)

SUMMER SQUASH Country, year Location (variety)	Application			PHI, (days)		Fluopyram Residues (mg/kg)				Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)			parent				
USA, 2007 Orland, CA (Black Beauty) Foliar spray	0.251 0.25	0.134 0.134	187 186	0	whole fruit washed fruit cooked fruit	0.074, 0.068, 0.063 0.046, 0.03, 0.04 0.033, 0.032, 0.03				RAGMP082 GM065-07HA
USA, 2007 Orland, CA (Black Beauty) Drip irrigation	0.25 0.25	25 25	1 [a] 1 [a]	7	whole fruit cooked fruit	0.011, 0.011, 0.012 0.011, 0.011, 0.01				RAGMP082 GM065-07HA

[a] = Litres/hour/emitter

Lettuce

Leaf and head lettuces were collected from six USA field trials to estimate the residue distribution of fluopyram in lettuce heads with and without wrapper leaves and in washed and unwashed lettuce leaves.

Wrapper leaves from the 1 kg head lettuce samples were removed in the field and both sets of samples were frozen within 4 hours of sampling and held in frozen storage for up to 427 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 196 Fluopyram residues in outdoor head lettuce with and without wrapper leaves from supervised trials in USA involving two foliar applications of fluopyram (500 SC) – 0 day PHI

LETTUCE Country, year Location (variety)	Application				Fluopyram Residues (mg/kg)				Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2007 Germansville, PA (Ithaca)	0.26 0.253	0.141 0.138	185 184	with wrapper leaves no wrapper leaves	1.874, 3.063 0.062, 0.032				RAGMP085 GM088-07HA

LETTUCE Country, year Location (variety)	Application				Fluopyram Residues (mg/kg)				Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2008 Belle Glade, FL (Gator)	0.254 0.248	0.148 0.139	172 178	with wrapper leaves no wrapper leaves	1.762, 0.794 0.037, < 0.01				RAGMP085 GM089-07HA
USA, 2007 Corning, CA (Great Lakes)	0.247 0.247	0.132 0.132	187 187	with wrapper leaves no wrapper leaves	2.375, 2.6 0.168, 0.094				RAGMP085 GM091-07HA
USA, 2008 Fresno, CA (Great Lakes)	0.251 0.249	0.215 0.215	117 116	with wrapper leaves no wrapper leaves	4.706, 4.021 0.978, 0.712				RAGMP085 GM092-07HA
USA, 2007 Porterville, CA (Vandenburg)	0.25 0.251	0.227 0.226	110 111	with wrapper leaves no wrapper leaves	0.354, 0.812 0.014, 0.026				RAGMP085 GM093-07HA
USA, 2007 Sanger, CA (Great Lakes)	0.236 0.26	0.183 0.157	129 166	with wrapper leaves no wrapper leaves	5.185, 5.386 0.62, 1.493				RAGMP085 GM090-07DA

Bulk (2.5 kg) samples of leaf lettuce from one outdoor field trial site in USA were taken from foliar-treated and the band sprayed plots and chilled overnight until delivered to the laboratory where they were washed and stored frozen for up to 240 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 197 Fluopyram residues in outdoor washed and unwashed leaf lettuce from supervised trials in USA involving two foliar sprays or banded applications of fluopyram (500 SC formulations)

LETTUCE Country, year Location (variety)	Application			PHI, (days)	Fluopyram Residues (mg/kg)				Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)		parent	PAA	BZM	PCA	
USA, 2007 King City, CA (Sunbelt)	0.251 0.254	0.163 0.165	154 154	43 (fresh) 43 (washed)	0.017, 0.018, 0.018 0.014, 0.014, 0.013				RAGMP085 GM084-07DA Band applications, 1d postplant and 23 d later, at 5-leaf stage
USA, 2007 King City, CA (Sunbelt)	0.246 0.249	0.168 0.187	146 133	0 (fresh) 0 (washed)	5.699, 5.631, 5.652 1.46, 1.318, 1.367				RAGMP085 GM084-07DA 2 foliar sprays, 5 days apart

Spinach

Spinach leaves were collected from one USA field trial to estimate the effect of washing and cooking on the residue distribution of fluopyram in spinach. In this trials, two foliar applications of fluopyram (SC 500 formulation) tank mixed with trifloxystrobin (SC 500) were applied to spinach plants at 0.25 kg ai/ha in 135 and 140 litres of water, 5 days apart, using a self-propelled boom sprayer and with a plot size of 177 square metres.

Bulk (4.5 kg minimum) samples of spinach leaves were chilled and delivered to the laboratory where they were processed the next day (tap-washed three times, shaken, drained and cooked in boiling water) and the samples frozen for up to 240 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 198 Fluopyram residues in fresh, washed and cooked spinach from a supervised field trial in USA involving two foliar applications of fluopyram (500 SC formulation) – 0 day PHI

SPINACH Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2007 King City, CA (Interceptor/Avenger)	0.251 0.249	0.179 0.184	140 135	fresh leaves washed leaves cooked leaves	9.76, 9.26, 9.8 0.786, 0.657, 0.657 0.609, 0.617, 0.587				RAGMP085 GM104-07DA

Mustard greens

Mustard greens were collected from one USA field trial to estimate the effect of washing on the residue distribution of fluopyram. In this trial, two foliar applications of fluopyram (SC 500 formulation) tank mixed with trifloxystrobin (SC 500) were applied to mustard green plants at 0.25 kg ai/ha in 116 and 126 litres of water, 4 days apart, using a knapsack sprayer with a hand boom and with a plot size of 112 square metres.

Bulk samples (3 kg) were collected once the last spray application had dried, chilled and delivered to the laboratory where sub-samples were processed the next day (sink-washed three times, shaken and drained) and the samples frozen for up to 567 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 199 Fluopyram residues in fresh and washed mustard greens from a supervised trial in USA involving foliar applications (500 SC formulations) – 0 day PHI

MUSTARD GREENS Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2007 Sycamore, Georgia (Florida Broadleaf)	0.252 0.253	0.217 0.201	116 126	fresh washed	12.2, 12.7, 13.1 1.87, 1.82, 1.89				RAGMP063 GM230-06DA

Celery

Celery tops were collected from one USA field trial to estimate the effect of trimming, washing and cooking on the residue distribution of fluopyram in celery. In this trials, two foliar applications of fluopyram (SC 500 formulation) tank mixed with trifloxystrobin (SC 500) were applied to celery plants at 0.24 and 0.25 kg ai/ha in 175 and 173 litres of water, 5 days apart, using a self-propelled boom sprayer and with a plot size of 177 square metres.

Bulk samples of celery tops were collected once the last spray application had dried, chilled and delivered to the laboratory where sub-samples were processed the next day (sink-washed three times, shaken, drained, trimmed (by removing the woody base, the outer petioles and trimming the top 70-80% of the leaves), sliced and cooked in boiling water for 6 minutes) and the samples frozen for up to 171 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 200 Fluopyram residues in fresh, washed, trimmed and cooked celery from a supervised trial in USA involving two foliar applications of fluopyram (500 SC formulations) – 0 day PHI

CELERY Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2007 Sanger, CA (Command)	0.244 0.248	0.139 0.143	175 173	fresh washed [a] trimmed [b] cooked [c]	7.96, 8.13, 8.1 1.42, 1.49, 1.47 0.904, 0.831, 0.87 0.173, 0.152, 0.164				GM096-07DA

[a] untrimmed

[b] unwashed

[c] washed and trimmed

Commercial processing

Orange

An orange processing trial was conducted in USA to measure the fluopyram residues in orange peel, dried pulp, juice and oil following exaggerated (5×) field treatment of oranges [Coopersmith, 2008, Ref: RAGMP046]. One unreplicated 0.125 ha plot of orange trees was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 6 days apart, using an orchard airblast sprayer to apply 1.28 and 1.26 kg ai/ha in 627 and 584 litres water/ha.

Bulk samples of oranges (227 kg min) were chilled and shipped overnight to the laboratory for processing the next day. Processing was performed using procedures that simulated commercial processing practices.

Peel and pulp samples were prepared by hand-peeling unwashed oranges. The remaining oranges were tub-washed and scarified to recover the oil. The resulting oil-water emulsion was screened to remove any flavedo fragments (which were subsequently added to the wet peel-pulp fraction). The oil was separated from the screened oil-water emulsion by centrifugation (before and after overnight freezing). Juice was also recovered from an aliquot of the scarified oranges and screened (1.19mm) to remove the rag and seeds (these also being added to the wet peel-pulp fraction). The combined wet peel-pulp fractions from the oil and juice extractions was mixed with lime and pressed before being air-dried to less than 10% moisture content and milled.

The oranges and orange processed commodities analysed in this study were held in frozen storage for a maximum of 35 days prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ for all matrices was 0.01 mg/kg.

Table 201 Fluopyram residues in oranges and orange peel, dried pulp, juice and oil from a supervised trial in USA involving two foliar applications (500 SC formulation) – 6 day PHI

ORANGE Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	Fluopyram	PAA	BZM	PCA	
USA, 2007 Sanger, CA (Campbell) Valencia	1.276 1.256	0.202 0.215	627 584	whole fruit peel pulp dried pulp juice oil	0.899, 0.956, 0.875 1.45, 1.67, 1.68 0.143, 0.144, 0.147 0.856, 0.853, 0.844 < 0.01, < 0.01, 0.012 14.7, 14.4, 14.2				RAGMP046 GM130-06PA

Apples

Apples from four European field trials were processed into sauce, juice, pomace (wet and dried), fruit (dried), peel and pulp, simulating household processing or commercial practices.

In these trials, 4 applications of fluopyram (SC 500 formulations) were applied to mature, full-sized trees (approximately 3 metres high) 7–8 days apart as foliar sprays using knapsack sprayers with single solid or hollow-cone nozzles or 5 flat-fan nozzles, applying 0.125 kg ai/ha in 500–1500 litres water/ha. Plot sizes in these trials ranged from 33 – 240 square metres and involved at least 4 trees per plot.

Unreplicated samples of at least 8 kg were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below until processing using procedures which simulated commercial practices.

To prepare apple sauce, apples (without stalks) were tub-washed in lukewarm water, drained, cut and blanched in water to 98–100 °C for 40 minutes before being sieved, separating raw sauce and pulp. The raw sauce was mixed with sugar (16.5%), canned and pasteurized in an autoclave. Juice was prepared by tub-washing and crushing deep-frozen apples, warming up the mash to 40 °C for 30–40 minutes and pressing out the raw juice. The juice was briefly heated to 80–90°C, cooled to 45–50 °C and enzyme added (Pectinex XXL and Amylase AG300L). After 20 hours storage at 4 °C, the juice was centrifuged, ultra-filtered and pasteurised in a plate heat-exchanger. The remaining wet pomace was dried in a fan-assisted oven at about 100 °C. Lightly defrosted apples were also peeled, cored and sliced (5–7 mm thick), treated with sulphite and citric acid to prevent enzymatic reactions, tub-washed in lukewarm water and dried in a fan-assisted oven at 80 °C.

Analytical samples were stored for up to 255 days (36 days for processed fractions) before extraction and analysis for fluopyram and its metabolites AE F148815 (benzamide), AE C657188 (pyridyl-carboxylic acid) and BCS-AA10189 (pyridyl-acetic acid) using LC/MS/MS Method 00984/M001. The reported LOQ for all matrices was 0.01 mg/kg.

Table 202 Residues in apples and processed apple fractions from supervised trials in Belgium, France, Italy and UK involving four foliar applications of fluopyram (500 SC) – 3 day PHI

APPLE Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
Belgium, 2006 Fleurus (Jonagored)	0.125	0.0125	1000	fresh fruit washed fruit dried fruit peeled fruit raw sauce sauce raw juice juice wet pomace dry pomace washings strain rest peel rest	0.11 0.06 0.07 0.02 0.06 0.04 0.02 < 0.01 0.19 0.6 0.01 0.76 0.6	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3605/06 0396-06

APPLE Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
United Kingdom, 2006 Royston (Jonathon)	0.125	0.025	500	fresh fruit washed fruit dried fruit peeled fruit raw sauce sauce raw juice juice wet pomace dry pomace washings strain rest peel rest	0.21 0.09 0.13 0.05 0.08 0.05 0.03 < 0.01 0.26 1.2 0.04 1.0 1.6	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3605/06 0413-06
France (S), 2006 Reyniès (Granny Smith)	0.125	0.0085	1500	fresh fruit washed fruit dried fruit peeled fruit raw sauce sauce raw juice juice wet pomace dry pomace washings strain rest peel rest	0.08 0.11 0.07 0.02 0.07 0.05 0.03 < 0.01 0.33 0.95 < 0.01 0.75 0.64	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.03 0.02 0.01 0.02 0.02 0.02 0.01 0.02 0.09 < 0.01 0.03 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3606/06 0399-06
Italy, 2006 Zevio (Golden Rainders)	0.125	0.01	1250	fresh fruit washed fruit dried fruit peeled fruit raw sauce sauce raw juice juice wet pomace dry pomace washings strain rest peel rest	0.11 0.04 0.1 0.03 0.05 0.04 0.03 < 0.01 0.27 0.84 0.03 0.75 0.63	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-3606/06 0414-06

An apple processing trial was conducted in USA to measure the fluopyram residues in washed, peeled or dried apples and in sauce, juice and wet following exaggerated (5×) field treatment of apples [Mackie, 2007, Ref: RAGMP033].

One unreplicated 334 square metre plot of apple trees was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 6 days apart, using an orchard airblast sprayer to apply 1.26 and 1.25 kg ai/ha in 610 litres water/ha.

Bulk apple samples (136 kg) were collected 5 days after the last application and shipped overnight to the laboratory for processing the next day, using procedures which simulated commercial practices.

Representative samples of fruit were washed and pulped in a hammermill before being heated with low pressure steam (in a steam-jacketed kettle) to 40–50°C, treated with pectin enzyme and allowed to react for about 2 hours. The enzyme-treated pulp was then pressed and the resulting juice, after filtration (40-mesh screen), was heated to 93 °C for 15–30 seconds to deactivate the pectic enzymes. The juice was then cooled and allowed to settle overnight, with the clear juice being racked off, vacuum filtered, heated to 88–91 °C, canned and stored frozen until analysis.

The apple fresh fruit and processed commodities were held in frozen storage for a maximum of 245 days prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ for all matrices was 0.01 mg/kg.

Table 203 Fluopyram residues in apples (washed, peeled, dried) and apple products (pomace, juice, sauce) from a supervised trial in USA involving two foliar applications (500 SC) – 6 day PHI

APPLE Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	Fluopyram	PAA	BZM	PCA	
USA, 2006 North Rose, NY (Rome)	1.26 1.25	0.207 0.205	610 610	whole fruit washed fruit peeled fruit dried fruit juice sauce wet pomace	0.95, 0.92, 0.92 0.69, 0.63, 0.62 0.03, 0.02, 0.03 0.03, 0.03, 0.03 0.43, 0.37, 0.44 0.01, 0.01, 0.01 2.22, 2.11, 2.09				RAGMP033 GM010-06P

Plums

A trial was conducted in USA to measure the fluopyram residues in washed and dried plums (prunes) following exaggerated (5×) field treatment of plums [Mackie, 2008, Ref: RAGMP049].

One unreplicated 0.02 ha plot of plum trees was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 7 days apart, using an orchard airblast sprayer to apply 1.25 kg ai/ha in 470 litres water/ha. Bulk plum samples (41 kg) were collected the day of the last application (once the spray had dried) and shipped overnight to the laboratory where they were cool-stored for 10 days before processing using procedures which simulated commercial practices.

Plums were washed in water at 52–57 °C for 3–5 minutes and dehydrated for just over 26 hours at 67–68 °F. Analytical samples were held in frozen storage for a maximum of 295 days prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ for all matrices was 0.01 mg/kg.

Table 204 Fluopyram residues in washed and dried plums (prunes) from a supervised trial in USA involving two foliar applications (500 SC formulation) – 6 day PHI

PLUMS Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	Fluopyram	PAA	BZM	PCA	
USA, 2006 Live Oak, CA (French)	1.25 1.25		470 469	whole fruit washed fruit dried fruit	0.17, 0.19, 0.2, 0.18, 0.18 0.09, 0.08, 0.1 0.2, 0.2, 0.22				RAGMP049 GM144-06PA

Grapes

Wine was produced from red grapes obtained from four different field trials located in Southern and Northern France. Following the simulated commercial processing of fluopyram-treated grapes, residues were measured in the various processing fractions. Flow charts describing the grape juice and wine production processes are presented below. The fining agent used in these studies was bentonite and the storage interval (at 12 °C) between filtration and taste testing was 132 days.

Table 205 Residues in wine grapes (bunches) and red wine processing fractions from supervised trials in France, involving 2 foliar applications of fluopyram (500 SC formulation) – 3 day PHI

WINE GRAPE Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	Matrix	parent	PAA	BZM	PCA	
France, 2006 Athée sur Cher (Gamay)	0.25	0.125	200	grape bunch washed berries washings raw juice pomace, wet pomace, dry retentate juice pomace must wine (young) wine @1st taste	0.58 0.38 0.06 0.06 1.3 2.8 0.05 < 0.01 1.6 0.12 0.06 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 < 0.01 0.01 0.02 0.05 0.01 0.01 0.02 0.02 0.01 0.02	0.02 0.01 < 0.01 < 0.01 0.02 0.04 0.01 < 0.01 0.02 0.02 0.01 0.02	RA-3611/06 R 2006 0415 7 35d spray interval
France, 2006 Charnay les Macon (Pinot noir PG161.49)	0.25	0.125	200	grape bunch washed berries washings raw juice pomace, wet pomace, dry retentate juice pomace must wine (young) wine @1st taste	0.51 0.3 0.05 0.06 1.6 3.0 0.05 < 0.01 2.0 0.11 0.08 0.10	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3611/06 R 2006 0650 8 28d spray interval
France, 2006 Laudun (Grenache Noir	0.25	0.125	200	grape bunch washed berries washings raw juice pomace, wet pomace, dry retentate juice pomace must wine (young) wine @1st taste	0.36 0.18 0.03 0.05 1.4 2.7 0.03 < 0.01 1.2 0.11 0.08 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3647/06 R 2006 0622 2 42d spray interval
France, 2006 Fronton (Négrette)	0.25	0.125	200	grape bunch washed berries washings raw juice pomace, wet pomace, dry retentate juice pomace must wine (young) wine @1st taste	0.58 0.43 0.02 0.09 2.1 4.2 0.08 < 0.01 2.1 0.13 0.08 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 < 0.01 0.02 0.03 0.06 0.01 0.01 0.03 0.02 0.01 0.02	0.04 0.02 < 0.01 0.02 0.04 0.08 0.02 0.01 0.04 0.03 0.02 0.04	RA-3647/06 R 2006 0623 0 23d spray interval

Wine at 1st taste test = after 132 days bottled storage at 12 °C

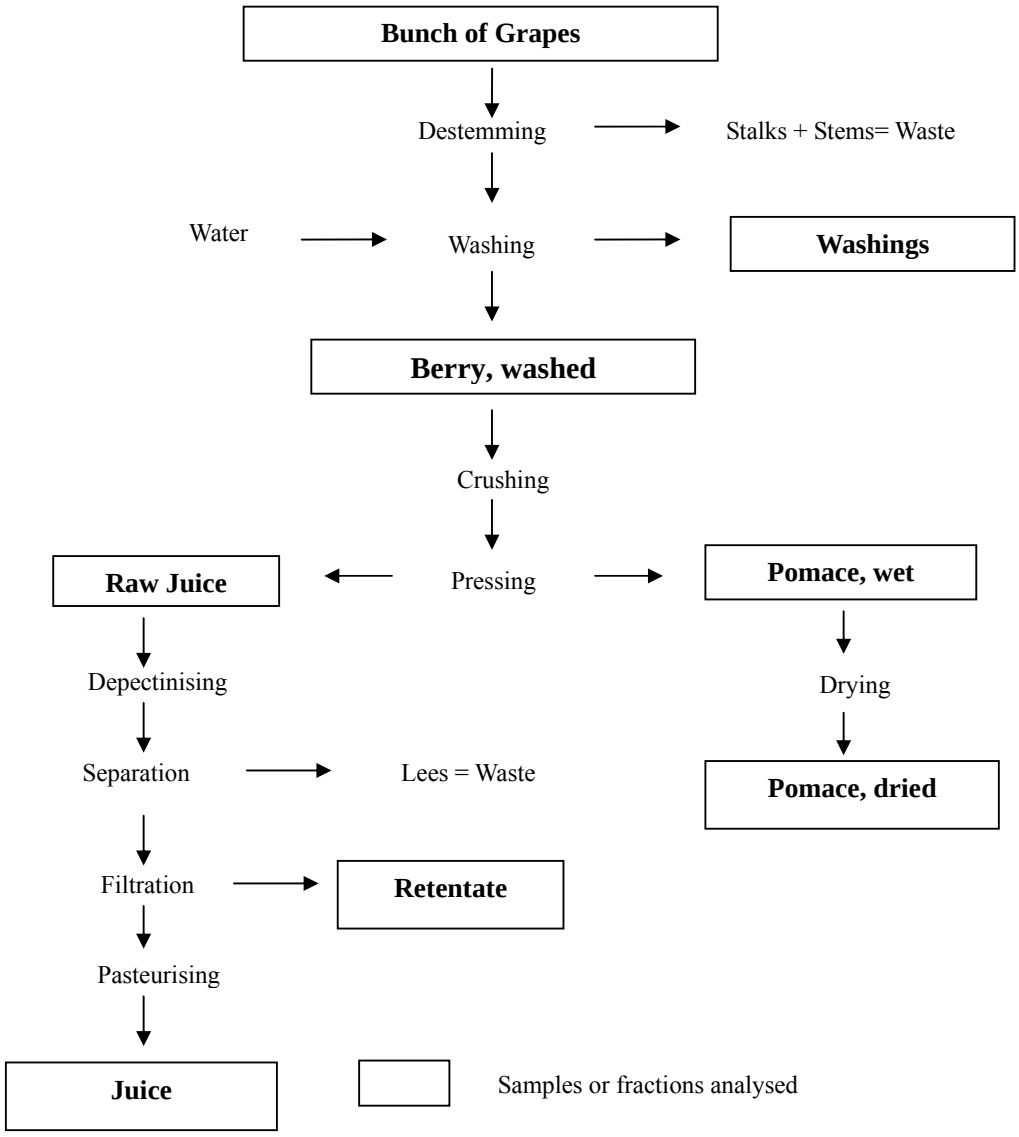


Figure 5 Flow chart for the preparation of grape bunches into berries, washed; washing water; retentate; pomace, wet; pomace, dried; raw juice and juice

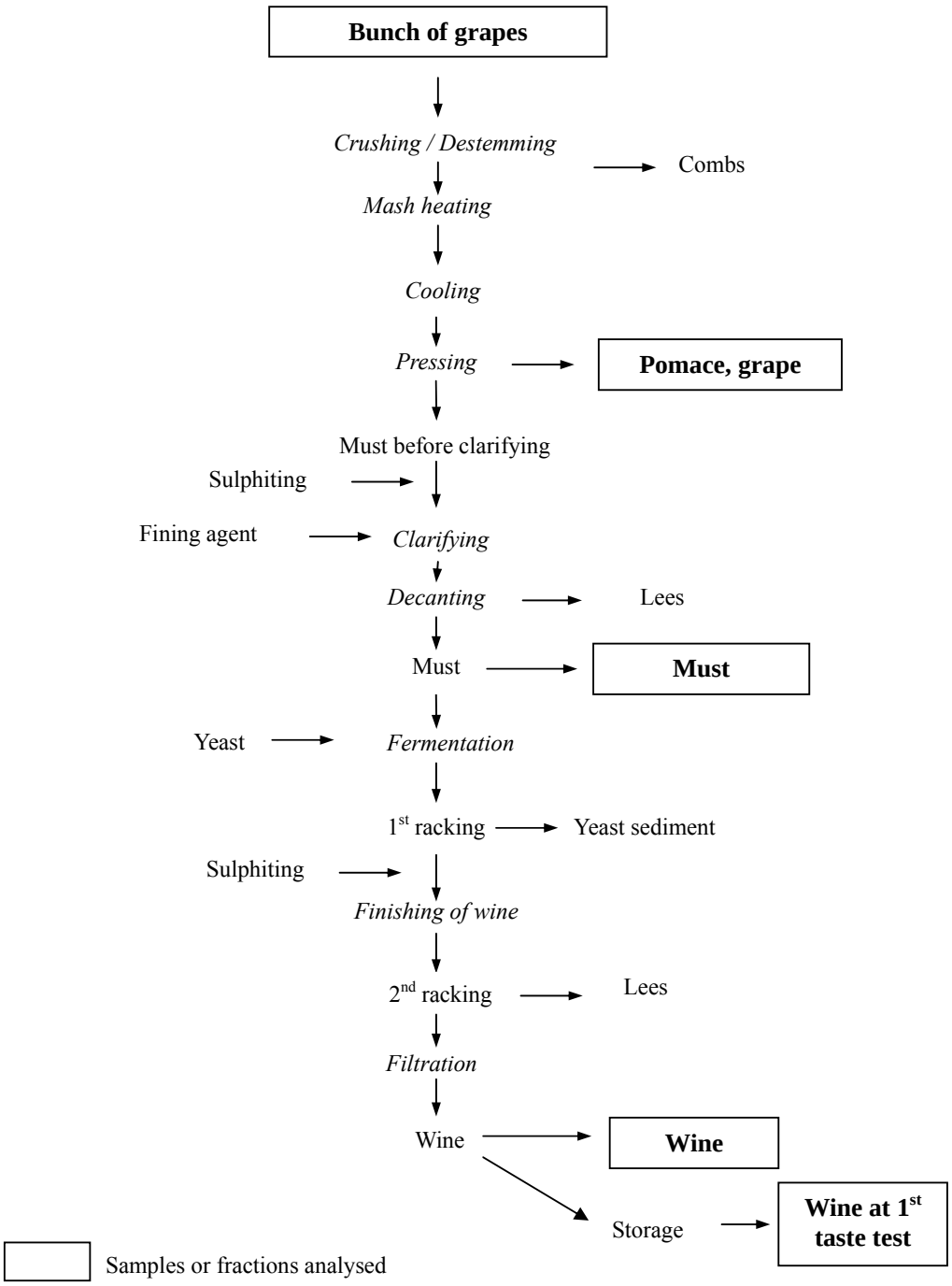


Figure 6 Flow chart of the preparation of bunches of grapes into pomace, grape; must; wine and wine at 1st taste test

Raisins were produced from white grapes obtained from four field trials located in Southern Europe (Spain, Portugal, Italy and Greece). The processing of bunch of table grapes into raisin washed; raisin waste; and washings simulated the industrial practice at a laboratory scale. Bunches of grapes were de-stemmed before drying. Berries and stakes/stems were dried at 60–65 °C to a water content of approximately 12%. After drying berries were washed in water. The process is described in the flow chart below.

Table 206 Residues in white table grapes (bunches) and raisins from supervised trials in Greece, Italy, Portugal and Spain, involving 2 foliar applications of fluopyram (500 SC) – 3 day PHI

TABLE GRAPE Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
Spain, 2006 Hondón de las Nieves (Italia)	0.25	0.025	1000	grape bunch raisin waste washings raisin	0.55 4.6 0.02 1.1	< 0.01 < 0.01 < 0.01 < 0.01	0.03 0.29 < 0.01 0.07	0.07 1.9 0.01 0.17	RA-3612/06 R 2006 0417 3 31d spray interval
Portugal, 2006 Passinha-Alenquer (Cardinal)	0.25	0.042	600	grape bunch raisin waste washings raisin	0.32 5.3 0.03 2.1	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.06 < 0.01 0.03	< 0.01 0.1 < 0.01 0.02	RA-3612/06 R 2006 0624 9 8d spray interval
Italy, 2006 Trinitapoli (Italia)	0.25	0.025	1000	grape bunch raisin waste washings raisin	0.66 6.1 < 0.01 1.9	< 0.01 < 0.01 < 0.01 < 0.01	0.02 0.28 < 0.01 0.07	0.02 0.59 < 0.01 0.07	RA-3612/06 R 2006 0651 6 16d spray interval
Greece, 2006 Assos (Soultanina)	0.25	0.025	1000	grape bunch raisin waste washings raisin	0.3 3.3 0.02 0.96	< 0.01 < 0.01 < 0.01 < 0.01	0.01 0.11 < 0.01 0.04	0.01 0.39 < 0.01 0.03	RA-3612/06 R 2006 0652 4 31d spray interval

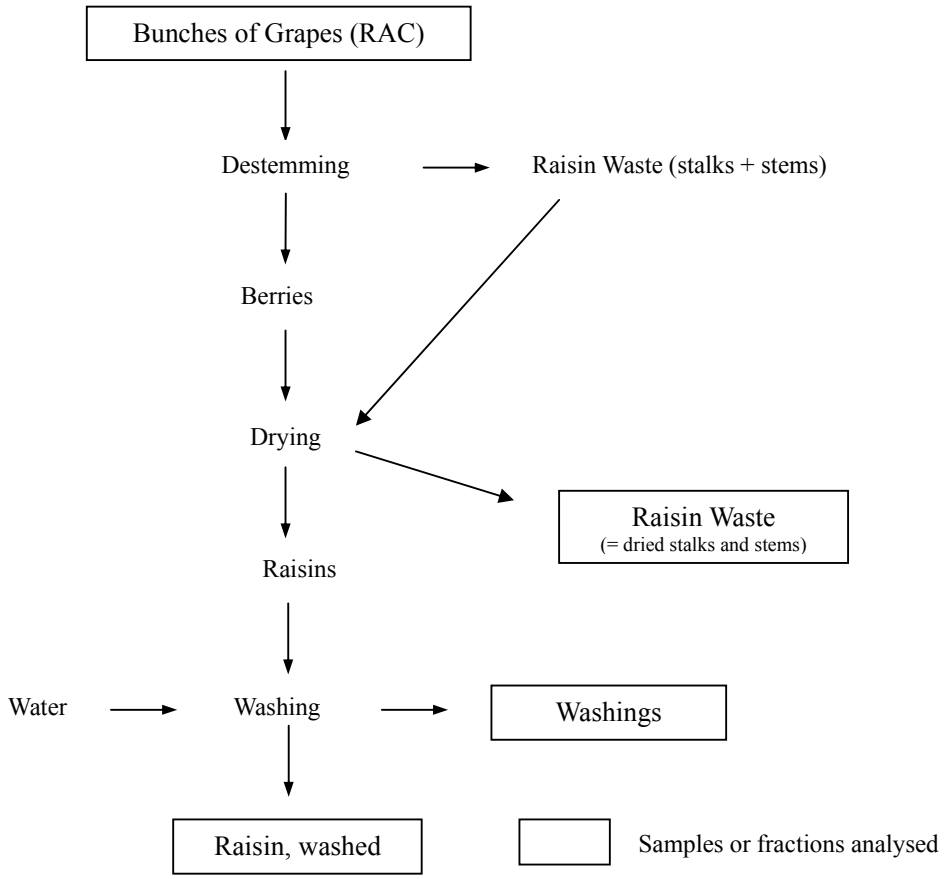


Figure 7 Flow Chart for the preparation of bunches of grapes into raisin, washed; raisin waste; and washings

A grape processing trial was conducted in USA to measure the fluopyram residues in grapes and in washed berries, grape juice, raisins and grape jelly following exaggerated (5×) field treatment of grapes [Mackie, 2008, Ref: RAGMP042].

One unreplicated 296 square metre plot of table grapes was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 14 days apart, using a ground-based airblast sprayer, applying 0.25 kg ai/ha in 520 litres water/ha. A bulk sample of about 90 kg grapes was taken from the inner halves of the two-row plot, 7 days after the last application and cool-stored within 6 hours, before processing the next day. The grapes were sub-sampled for analysis and the remaining fruit processed into raisins, juice and jelly, using simulated commercial practices.

Prior to processing, random sub-samples of the control and treated bulk grape samples were collected for analysis, and the remainder of the grape samples were used to generate the required processed commodities of raisins and juice and jelly. Processing was performed using procedures which simulated commercial processing practices.

Grapes for processing into grape juice were crushed, separated from the stems and transferred to a steam-jacketed kettle for depectinization where the enzyme-inoculated crush was heated to about 57–60 °C for about 2 hours before being passed through a screw press to produce the unclarified juice and the wet pomace.

The unclarified juice was then heated in a steam-jacketed kettle to 32 °C to inactivate the pectinase enzyme and then allowed to settle under refrigeration for 25 days before filtering, canning or further processing into jelly. A portion of the filtered juice was heated to canning temperature (92 °C) in a steam-jacketed kettle and then poured into plastic jars, sealed and cooled. A further portion of the filtered juice was mixed in a steam-jacketed kettle with citric acid (to adjust the pH to about 3), sugar and pectin and then heated to boiling before being placed in plastic jars and cooled.

Raisins were prepared by spreading the grapes out on stainless steel drying trays and sun-dried for 2 months, achieving a moisture content of 12–16%, before being stored in plastic bags at about 20 °C for 2 days (moisture equilibration). The dried grapes were then frozen to facilitate the manual destemming and cap-removal and then washed and rehydrated to 18–19% moisture content by dipping in fresh water for 1-2 seconds.

Analytical samples were stored frozen for up to 300 days before analysis for fluopyram using HPLC-MS/MS Method GM-001-P07-01. The reported LOQ was 0.01 mg/kg.

Table 207 Fluopyram residues in grapes and processed grape commodites from a supervised trial in USA, involving 2 foliar applications of fluopyram (500 SC formulation)

TABLE GRAPE Country, year Location (variety)	Application			PHI, (days)	Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
USA, 2006 Fresno, CA (Thompson Seedless)	1.25 1.25	0.053 0.053	523 519	7	grapes washed berries juice raisins jelly	1.25, 1.27, 1.22 0.94, 0.97, 0.97 0.6, 0.71, 0.69 3.01, 2.91, 3.22 0.18, 0.18, 0.18				RAGMP042

Tomatoes

Tomatoes from four European field trials were processed into juice, preserve and purées, with washing and peeling procedures that reflected household practice and the preparation of juice, preserves, and puree that simulated commercial practice.

In these trials, 2 applications of fluopyram (SC 500) were applied at 7 day intervals to mature plants as foliar sprays using knapsack sprayers with hand-held mini-booms (3–8 flat-fan nozzles) to apply 0.25 kg ai/ha in 500–1200 litres of water/ha. Plot sizes in these trials ranged from 22–120

square metres. Unreplicated samples of 8–20 kg fruit were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 313 days before processing.

After washing, the tomatoes were chopped and blanched in water (98–100 °C) for 15–30 minutes before being strained to separate the raw juice and the pomace ("strain rest"). Sodium chloride (0.5–0.7% w/w) was added and the sample of raw juice was split, one part for processing into preserves and the other portion for pasteurization.

Another portion of the washed tomatoes were peeled and the peeling water was sieved, raw juice (see above) was added and the sample was pasteurized before hand mincing to produce the tomato preserve.

Chopped, washed tomatoes were also blanched in water (98–100 °C) for 25–35 minutes to prevent enzymatic reactions and strained to separate raw juice and pomace. The raw juice was mixed with sodium chloride (with exception of R 2006 0383/5) and raw puree (dry weight: 8–17%) was prepared by distillation (100–200 mbar, 75 °C, while stirring) or centrifugation to produce raw puree and distillate. The processes are described in detail in the flow charts below.

Processed commodities were stored frozen for up to 22 days before analysis. Residues of fluopyram and its BZM, PAA and PCA metabolites were measured using LC/MS/MS 00984/M001 (LOQ 0.01 mg/kg for each analyte).

Table 208 Residues in tomatoes and processed tomato matrices from supervised field trials in France, Italy and Portugal involving two foliar applications of fluopyram (500 SC formulation) – 3 day PHI

TOMATOES Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
Portugal, 2006 Reguengo-Valada (H-9996)	0.25	0.05	500	fruit	0.16	< 0.01	< 0.01	< 0.01	RA-3599/06 0383-06
				washed fruit	0.15	< 0.01	< 0.01	< 0.01	
				raw juice	0.08	< 0.01	< 0.01	< 0.01	
				juice	0.07	< 0.01	< 0.01	< 0.01	
				peel	0.93	< 0.01	< 0.01	< 0.01	
				pulp	0.02	< 0.01	< 0.01	< 0.01	
				preserve	0.04	< 0.01	< 0.01	< 0.01	
				raw puree [a]	0.21	< 0.01	< 0.01	< 0.01	
				raw puree [b]	0.16	< 0.01	< 0.01	< 0.01	
				puree	0.15	< 0.01	< 0.01	< 0.01	
				fruit washings	0.06	< 0.01	< 0.01	< 0.01	
				peeling water	0.04	< 0.01	< 0.01	< 0.01	
				strain rest	0.76	< 0.01	< 0.01	< 0.01	
				separation water	0.05	< 0.01	< 0.01	< 0.01	
Italy, 2006 Andria (BA) (Donald)	0.25	0.0335	750	fruit	0.24	< 0.01	< 0.01	< 0.01	RA-3599/06 0402-06
				washed fruit	0.22	< 0.01	< 0.01	< 0.01	
				raw juice	0.1	< 0.01	< 0.01	< 0.01	
				juice	0.1	< 0.01	< 0.01	< 0.01	
				peel	0.72	< 0.01	< 0.01	< 0.01	
				pulp	0.02	< 0.01	< 0.01	< 0.01	
				preserve	0.05	< 0.01	< 0.01	< 0.01	
				raw puree [a]	0.13	< 0.01	< 0.01	< 0.01	
				puree	0.11	< 0.01	< 0.01	< 0.01	
				fruit washings	0.04	< 0.01	< 0.01	< 0.01	
				peeling water	0.04	< 0.01	< 0.01	< 0.01	
				strain rest	1.2	< 0.01	< 0.01	< 0.01	

TOMATOES Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
France, 2006 Boé (Perfect Peel)	0.25	0.0415	600	fruit	0.09	< 0.01	< 0.01	0.01	RA-3599/06 0613-03
				washed fruit	0.06	< 0.01	< 0.01	< 0.01	
				raw juice	0.05	< 0.01	< 0.01	< 0.01	
				juice	0.05	< 0.01	< 0.01	< 0.01	
				peel	0.51	< 0.01	0.01	0.01	
				pulp	0.01	< 0.01	< 0.01	< 0.01	
				preserve	0.03	< 0.01	0.01	< 0.01	
				raw puree [a]	0.16	< 0.01	0.02	0.02	
				puree	0.2	< 0.01	0.02	0.02	
				fruit washings	0.01	< 0.01	< 0.01	< 0.01	
				peeling water	0.02	< 0.01	< 0.01	< 0.01	
				strain rest	0.49	< 0.01	< 0.01	< 0.01	
Italy, 2007 S. Spirito (Bari) (Angos)	0.25	0.025	1000	fruit	0.22	< 0.01	< 0.01	< 0.01	RA-3505/07 0233-07
				washed fruit	0.07	< 0.01	< 0.01	< 0.01	
				raw juice	0.03	< 0.01	< 0.01	< 0.01	
				juice	0.06	< 0.01	< 0.01	< 0.01	
				peel	0.54	< 0.01	< 0.01	< 0.01	
				pulp	0.02	< 0.01	< 0.01	< 0.01	
				preserve	0.04	< 0.01	< 0.01	< 0.01	
				raw puree [a]	0.17	< 0.01	< 0.01	< 0.01	
				puree	0.16	< 0.01	< 0.01	0.02	
				fruit washings	< 0.01	< 0.01	< 0.01	< 0.01	
				peeling water	0.02	< 0.01	< 0.01	< 0.01	
				strain rest	0.5	< 0.01	< 0.01	< 0.01	

[a] raw puree prepared by distillation

[b] raw puree prepared by centrifugation

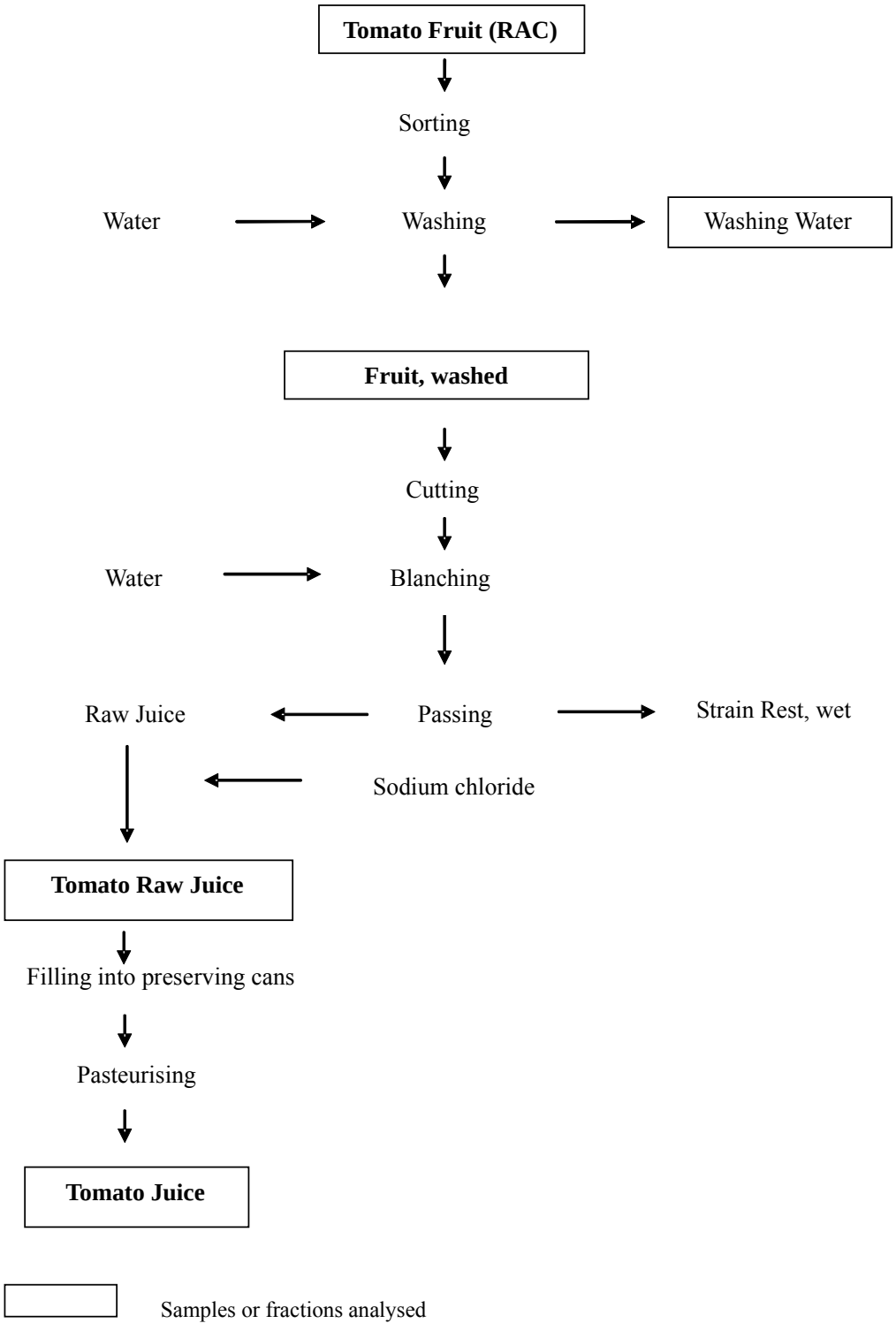


Figure 8 Flow Chart for the preparation of tomato fruits into fruit, washed; washing water; strain rest; raw juice and juice

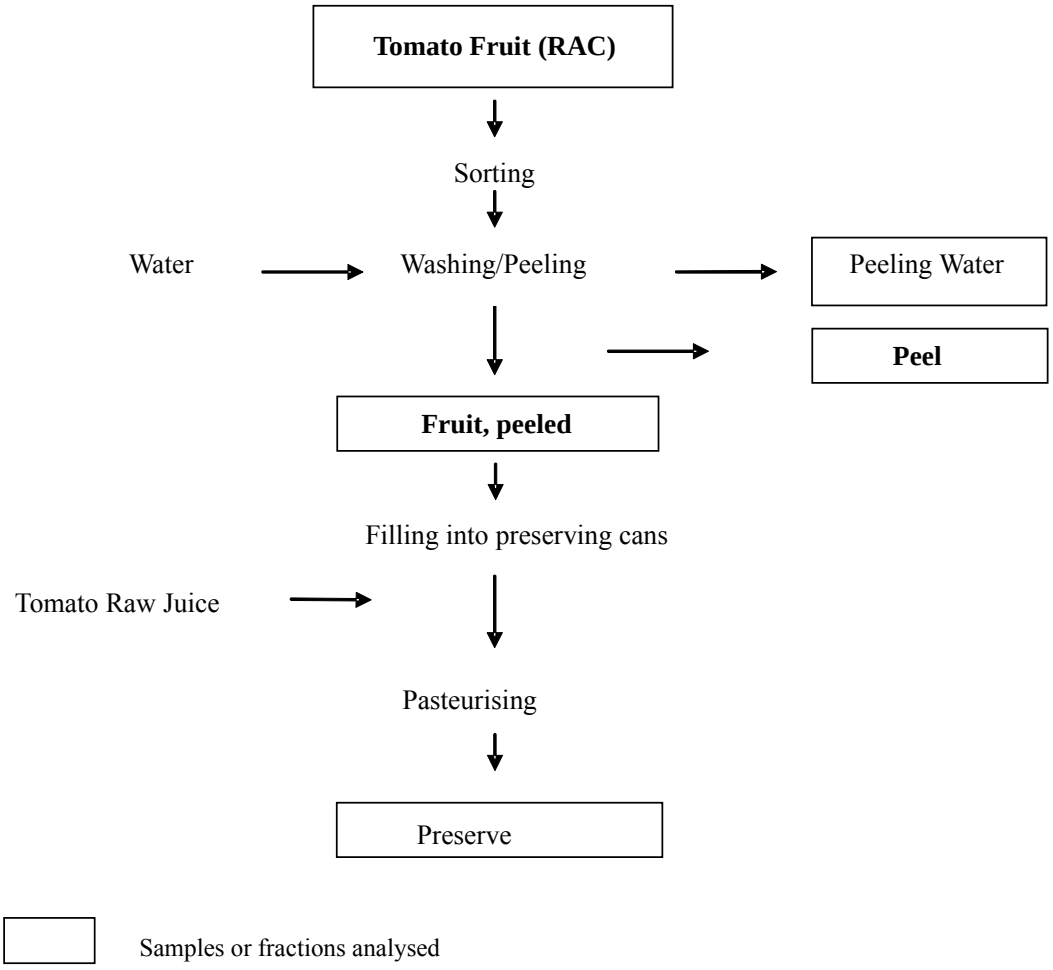


Figure 9 Flow Chart for the preparation of tomato fruits into fruit, peeled; peeling water; peel and preserve

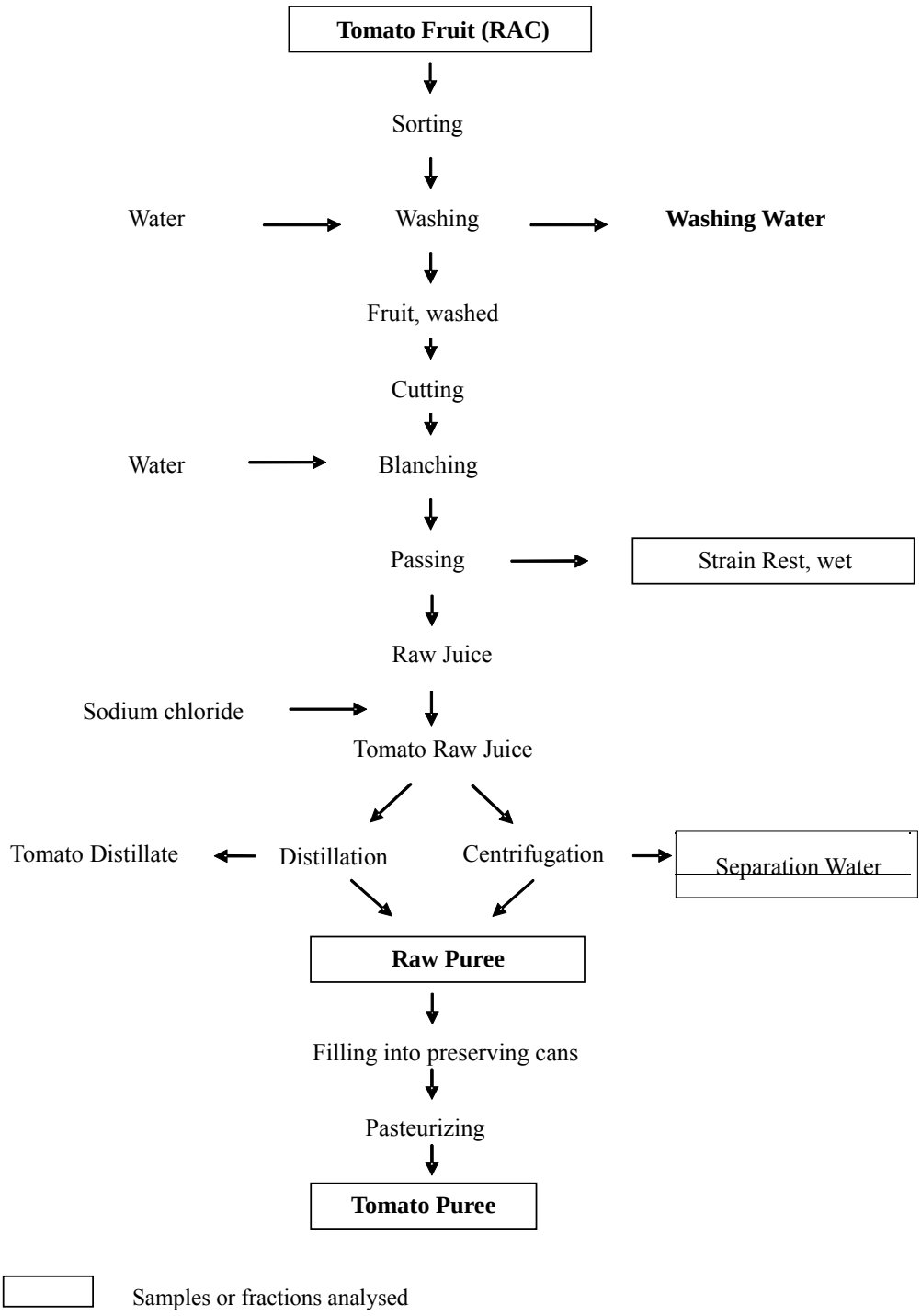


Figure 10 Flow Chart for the preparation of tomato fruits into raw puree, strain rest, wet, separation water and puree.

A tomato processing trial was conducted in USA to measure the fluopyram residues in tomatoes and in washed fruit and a range of processed tomato products, following exaggerated (5×) field treatment of field tomatoes [Lenz & Harbin, 2008, Ref: RAGMP058].

One unreplicated 1400 square metre plot of field tomatoes was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 4 days apart, using a self-propelled boom sprayer, applying 2.5 kg ai/ha in 170 litres water/ha.

A single bulk sample of about 90 kg of fresh tomatoes was collected from each plot after the spray had dried (0-day PHI). Subsamples of the tomatoes were removed for analysis, and the remaining tomatoes were processed into paste, puree, juice and were also cooked, canned and dried using procedures that simulated commercial processing practices.

The washed tomatoes were chopped, and passed through a 2.4mm screen before heating to 91–97 °C and the hot break material was further sieved (0.8mm) to produce juice and the retained pulp. The juice was heated to 85–90 °C for 3 minutes before being canned, pressure cooked, cooled and stored frozen until analysis. Subsamples of the juice were evaporated under heat and vacuum to produce puree (8–24% solids) and paste (24–30% solids), both of which were heated to 85–90 °C before being canned, cooled and stored frozen until analysis. Canned tomatoes were prepared by steam blanching the washed tomatoes, submerging them in water and removing the skins and placing the peeled tomatoes in cans with heated juice. The cans were sealed and pressure-cooked 120–124 °C for about 30 minutes before cooling and being stored frozen until analysis. A further sub-sample of the washed tomatoes were also sliced (6–10mm) and dehydrated at 49–55 °C until the moisture content was 7.6–8.2%.

The tomato fresh fruit and the tomato processed commodities analysed in this study were held in frozen storage for a maximum of 332 days prior to extraction and analysis for fluopyram using HPLC-MS/MS Method GM-001-P07-01. The reported LOQ was 0.01 mg/kg.

Table 209 Fluopyram residues in tomatoes and processed tomato matrices from a supervised field trial in USA involving two foliar applications of fluopyram (500 SC) – 0 day PHI

TOMATOES Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2006 Fresno, CA (AB 2)	1.26 1.24	0.746 0.721	169 172	fruit washed fruit juice cooked fruit canned fruit dried fruit paste puree	0.981, 0.983, 0.857 0.455, 0.465, 0.53 0.087, 0.079, 0.083 0.196, 0.199, 0.199 0.068, 0.064, 0.064 4.45, 3.93, 3.81 0.427, 0.452, 0.422 0.187, 0.171, 0.162				RAGMP058 GM200-06PA

Potatoes

A potato processing trial was conducted in USA to measure the fluopyram residues in potatoes and in washed, peeled, cooked tubers, chips and flakes, following exaggerated (5×) field treatment of potatoes [Lenz, 2008, Ref: RAGMP051]. One unreplicated 148 square metre plot of potatoes was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 4 days apart, using a self-propelled boom sprayer, applying 1.27 kg ai/ha in 160 litres water/ha.

Bulk samples of about 77 kg of fresh tubers were collected from each plot 6 days after the last application of fluopyram and shipped unfrozen to the laboratory for processing the next day. The potatoes were washed, peeled, cooked and further processed into flakes and chips using procedures that simulated commercial processing practices.

Tubers were washed, steam peeled (45 seconds at 6.9–8.3 bar) and scrubbed (20 seconds) before being cut into slabs and washed to remove free starch. The washed slabs were pre-cooked at 72–75 °C for 20 minutes in a steam-jacketed kettle, cooled and then cooked in a steam cabinet for 40 minutes at 94–100 °C. The cooked slabs were mashed, mixed with pre-weighed food additives and fed into a drum dryer to produce a thin sheet of dry mash which was then hammer milled to produce the flakes. Washed tubers were also batch peeled in a restaurant style peeler and cut into 15mm slices before being immersed in hot water to remove free starch, drained, fried at 158–192 °C for about 2 minutes, drained and salted.

The tubers and the processed potato commodities analysed in this study were held in frozen storage for a maximum of 312 days prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ for all matrices was 0.01 mg/kg.

Table 210 Fluopyram residues in potatoes and processed potato matrices from a supervised field trial in USA involving two foliar applications of fluopyram (500 SC formulation) – 6 day PHI

POTATOES Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2006 Sabin, MN (Red Norland)	1.27 1.27	0.789 0.789	161 161	tubers washed tuber peeled tubers cooked tubers chips flakes wet peel	0.016, 0.017, 0.014 0.011, 0.011, 0.012 < 0.01 (3) < 0.01 (3) < 0.01 (3) 0.016, 0.016, 0.016 0.065, 0.07, 0.069				RAGMP051 GM163-06PA

Sugarbeet

A sugarbeet processing trial was conducted in USA to measure the fluopyram residues in sugarbeet roots, pulp, molasses and sugar, following exaggerated (5×) field treatment of sugarbeet [Lenz, 2008, Ref: RAGMP057]. One unreplicated 158 square metre plot of sugarbeet was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 6 days apart, using a self-propelled boom sprayer, applying 1.2–1.3 kg ai/ha in 160–170 litres water/ha.

Bulk samples of about 79 kg of sugarbeet roots were collected from each plot 7 days after the last application of fluopyram and frozen within 1 hour and stored frozen for up to 337 days before processing using procedures that simulated commercial processing practices.

The sugarbeet roots were cleaned and washed, sliced and exposed to hot water (88–92 °C water for 30–45 seconds) before diffusion in water-bath kettles (68–74 °C for at least 9 minutes). The beet slices (cossettes), after diffusion, were dewatered and oven-dried (55–70 °C) to produce dried pulp with a final moisture content of 15% or less. The juice from the dewatering, after filtration, was added to the juice from the diffusion process and the combined juice fraction was processed into molasses and sugar. The juice fraction was purified and concentrated through a series of pH-adjusted precipitation, centrifugation, vacuum filtration and evaporation steps to produce refined sugar and molasses.

The sugarbeet roots and the processed commodities analysed in this study were held in frozen storage for a maximum of 312 days (roots) and 98 days (pulp, sugar, molasses) prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ for all matrices was 0.01 mg/kg.

Table 211 Fluopyram residues in sugarbeet roots, pulp, sugar and molasses from a supervised field trial in USA involving two foliar applications of fluopyram (500 SC) – 7 day PHI

SUGAR BEET Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2006 Sabin, MN (Beta 4797R)	1.3 1.19	0.765 0.739	170 161	roots refined sugar molasses dried pulp	0.251, 0.276, 0.251 0.329, 0.311, 0.357 0.225, 0.257, 0.229 0.332, 0.356, 0.308				RAGMP057 GM199-06PA

Soya bean

A soya bean processing trial was conducted in USA to measure the fluopyram residues in seeds, aspirated grain fractions and processed commodities following exaggerated (5×) field treatment of soya beans [Dallstream & Beadle, 2008, Ref: RAGMP054]. One unreplicated 0.5 ha plot of soya beans was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 7 days apart, using a 3 metre hand boom to apply 1.25 kg ai/ha in 138 litres water/ha.

Bulk samples of 725–997 kg soya beans were harvested by combine harvester 13 days after the last application of fluopyram, frozen within 4 hours and stored frozen for up to 350 days before processing. Subsamples of the soya bean seed were removed and the remaining seed was used to generate aspirated grain fractions and for processing into meal, hulls, oil (refined, bleached and deodorized), defatted flour and soymilk using procedures that simulated commercial processing practices.

After drying to 10–13% moisture content and screening to remove whole pods, a subsample of seeds were cycled through two bucket conveyors and one screw conveyor for 120 minutes, with aspirated grain fractions being collected at several locations. A further subsample, after aspiration and screening was milled and after separation, the kernels were heated to 71–80 °C and flaked. A sample of the flaked kernels was extruded by direct steam injection and compression, oven-dried for 30–40 minutes at 54–71 °C and triple-extracted (49–60 °C for 30 minutes) and double-washed (15 minutes) with hexane to produce the meal. A sample of the flaked kernels was also solvent-extracted using the same procedure and the defatted flakes were ground to produce flour. The extracted crude oil was separated from the hexane solvent by heating to 72–73 °C and alkali refined to produce soapstock and refined oil which was then bleached and deodorized. Soymilk was produced by soaking whole clean soya beans in reverse osmosis water for at least 12 hours, grinding and filtering the soaked beans to collect the soymilk filtrate (after heating to 90–96 °C for about 10 minutes).

The soya beans and processed commodities analysed in this study were held in frozen storage for a maximum of 354 days (seeds) and 65 days (processed fractions) prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ for the aspirated grain fraction was 0.1 mg/kg and the LOQ for all other matrices was 0.01 mg/kg.

Table 212 Fluopyram residues in soya bean seeds and processing fractions from a supervised trial in USA involving two foliar applications (500 SC formulations) – 13 day PHI

SOYA BEAN (DRY) Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2006 Stilwell, KS (NKS49-Q9)	1.25 1.25	0.9 0.9	138 138	seed asp grain fraction meal hulls refined oil flour soymilk	0.499, 0.492, 0.489 113, 115, 102 0.025, 0.022, 0.021 0.676, 0.596, 0.634 < 0.01, 0.017, < 0.01 0.021, 0.019, 0.026 < 0.01, < 0.01, < 0.01				RAGMP054 GM177-06PA

Wheat

A trial was conducted in USA to measure the fluopyram residues in wheat bran, flour, shorts, middlings, wheat germ and in the aspirated wheat grain fraction following exaggerated (5X) field treatment of wheat [Dallstream & Beedle, 2008, Ref: RAGMP060].

One unreplicated 0.22 ha plot of wheat was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 13 days apart, using a mechanised crop boom sprayer to apply 1.3 kg ai/ha in 166 L water/ha. Bulk grain samples (about 320 kg) were harvested by combine 14 days after the last treatment, frozen within about 1 hour of sampling and stored frozen for up to 11 months before processing using procedures which simulated commercial practices.

Grain samples were dried to a moisture content of 10–13%, aspirated in a dust generation room (with bucket and screw conveyors) for 2 hours to collect the aspirated grain dust and impurities and screened to separate out the wheat. For flour processing, the cleaned wheat was moisture-adjusted to 17.5% moisture content, milled and separated using a series of screening operations involving mesh sizes of 140, 160 and 800 microns to produce the flour, shorts, middlings and bran fractions respectively. Wheat germ recovery involved the milling of moisture-adjusted (16%) cleaned grain, sieve-separation (34 mesh) to isolate the germ fraction and the subsequent aspiration to remove additional bran.

Analytical samples were held in frozen storage for a maximum of 346 days (grain) and up to 17 days for the processed fractions, prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQs were 0.01 mg/kg in wheat grain, 0.10 mg/kg in wheat bran, flour, middlings, shorts, and germ, and 0.20 mg/kg in aspirated grain fractions.

Table 213 Fluopyram residues in wheat grain and processed grain fractions from a supervised wheat trial in USA involving two foliar applications (500 SC formulation) – 14 day PHI

WHEAT Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	Fluopyram	PAA	BZM	PCA	
USA, 2006 Sabine, MN (Steele)	1.3 1.31	0.793 0.784	164 167	grain bran flour germ asp grain fract shorts middlings	1.588, 1.598, 1.522 3.975, 4.378, 4.326 0.187, 0.191, 0.191 3.727, 3.841, 3.834 110, 110, 108 1.161, 1.161, 1.228 0.523, 0.528, 0.526				RAGMP060 GM211-06PA

Maize

A trial was conducted in USA to measure the fluopyram residues in maize grain, meal, bran, grits, starch, flour, oil and in the aspirated grain fractions following exaggerated (5×) field treatment of maize [Dallstream & Krolski, 2008, Ref: RAGMP040].

One unreplicated 750 square metre plot of maize was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 7 days apart, using a mechanised crop boom sprayer to apply 1.22–1.24 kg ai/ha in 167–170 litres water/ha. Bulk samples (about 320 kg) of peanuts (with shells) were taken 12 days after the last treatment before being frozen and stored for about 13 months prior to processing using procedures which simulated commercial practices.

Grain samples were dried to a moisture content of 10–13%, aspirated in a dust generation room (with bucket and screw conveyors) for 2 hours to collect the aspirated grain dust and impurities and screened to separate out the maize grain. Representative samples of the cleaned grain were further processed by dry milling or wet milling.

Dry milling involved conditioning the grain to a 21% moisture content, milling to crack the kernels and the subsequent oven-drying (54–71 °C for 30 mins) and sieve separation of the bran, germ and large grits from the grits, meal and flour. These two fractions were further milled and sieved in a series of steps to separate out these fractions.

The wet milling procedure involved steeping the clean grain in warm sulphur dioxide/water for 1–2 days before milling and hydrocloning (water centrifuging) to remove most of the non-cornstock material which, after drying, was separated into the germ material and hulls by aspiration and screening. The retained cornstock was milled and screened to separate the bran from the process water, the latter being separated into starch and gluten by batch centrifugation. The starch fraction was dried to a moisture content of 15% or less.

Processing the germ material from both the wet and dry milling processes involved a similar process of heating, flaking and triple-washing the germ material in hexane to extract the crude oil. The crude oil was separated from the miscella by vacuum evaporation and refined by mixing with sodium hydroxide and decanting the centrifuged oil. This was then bleached at 85–100 °C with activated bleaching earth, filtered and heated under vacuum at 220–230 °C for 10–15 minutes with citric acid being added during the cooling period to produce deodorized oil.

Analytical samples were held in frozen storage for a maximum of 386 days (grain) and 56 days (processed fractions), prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQs were 2.0 mg/kg for the aspirated grain fraction and 0.01 mg/kg for grain and the other processed fractions.

Table 214 Fluopyram residues in maize grain, meal, bran, grits, starch, flour, oil and in the aspirated grain fractions from a supervised trial on maize involving 2 foliar applications (500 SC) – 12 day PHI

MAIZE Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	Fluopyram	PAA	BZM	PCA	
USA, 2006 Seymour IL (Garst 8568 CB/LL))	1.22 1.24	0.73 0.726	167 170	grain grits meal flour bran starch oil (wet milled) oil (dry milled) asp grain fract	0.0287, 0.0259, 0.0291 0.016, 0.015, 0.012 0.022, 0.023, 0.023 0.024, 0.023, 0.024 0.07, 0.077, 0.075 0.01, < 0.01 (2) 0.015, 0.017, 0.017 < 0.01 (3) 4.76, 4.17, 4.52				RAGMP040 GM083-06PA

Peanut

A trial was conducted in USA to measure the fluopyram residues in raw and roasted peanuts, peanut meal, butter and oil following exaggerated (5×) field treatment of peanuts [Fischer, 2008, Ref: RAGMP047].

One unreplicated 335 square metre plot of peanuts was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 13 days apart, using a mechanised crop boom sprayer to apply 1.25 kg ai/ha in 138–144 litres water/ha. Bulk samples (about 320 kg) of peanuts (with shells) were lifted 6 days after the last treatment and allowed to dry in the field for 8 days before being frozen and stored for about 7 months before processing using procedures which simulated commercial practices.

Whole peanuts were oven-dried to a moisture content of 7–12%, cleaned by aspiration and screening and then cracked and aspirated to separate the hull material from the kernels (nutmeat). A representative sample of the nutmeat was oven-dried to a moisture content of 7–10% before being salted and dry-roasted. Peanut butter was produced by blending a sub-sample of the dry roasted peanuts with peanut oil and salt. Oil was also prepared by pressing heated nutmeat (conditioned to 12% moisture) to extract crude oil, with the residual crude oil being solvent-extracted (hexane) from the presscake. The crude oil was refined by mixing with sodium hydroxide and decanting the centrifuged oil. This was then bleached at 85–100 °C with activated bleaching earth, filtered and heated under vacuum at 220–230 °C for 10–15 minutes with citric acid being added during the cooling period to produce deodorized oil.

Analytical samples were held in frozen storage for a maximum of 358 days (nutmeat), 111 days (oil) and 28 days for the remaining processed fractions, prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQs were 0.01 mg/kg for nutmeat and all processed peanut matrices.

Table 215 Fluopyram residues in peanut meat, meal, butter, oil and roasted nuts from a supervised trial in USA on peanuts involving two foliar applications (500 SC) – 6 day PHI

PEANUT Country, year Location (variety)	Application			Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	Fluopyram	PAA	BZM	PCA	
USA, 2006 Tifton, GA (C99R)	1.25 1.25	0.868 0.906	144 138	nutmeat meal butter oil nuts (dry roast)	0.086, 0.099, 0.095 0.021, 0.019, 0.014 0.02, 0.021, 0.021 0.027, 0.01, 0.032 0.024 (3)				RAGMP047 GM131-06PA

Rape seed

Seeds from four European oil seed rape field trials were processed into oil, meal and press cake (pomace), simulating commercial practices. In these trials, 2 applications of fluopyram (SC 500 formulation) were applied to rape plants at BBCH 63 (30% flowers open) and again, 14–39 days later, at BBCH 73 (30% pods at full size), as foliar sprays using knapsack sprayers with spray booms (3–12 flat fan nozzles), applying 0.125 kg ai/ha in 300-330 litres water/ha. Plot sizes in these trials ranged from 100–563 square metres. Bulk samples of seeds were frozen within 24 hours of sampling and stored at -18° C or below for about 7 months before processing.

Rape seeds were cleaned, oven-dried (80 °C for 30 minutes), conditioned to a water content of about 8% and screw-pressed to produce the pressed oil and press cake. An aliquot of the press cake was pulverized to meal in a cutter with dry ice and the remaining oil was extracted with n-hexane in a soxhlet apparatus resulting in miscella (a mixture of n-hexane and oil) and extracted press cake meal. The miscella (n-hexane+oil) was distilled in a vacuum rotary evaporator and the extracted oil was combined with the pressed oil to form the crude oil fraction. An aliquot of the solvent extracted press cake meal was steam-distilled (toasting) in order to remove the n-hexane and hot air-dried (80 °C for 30 minutes) to produce the press cake fraction (minimum 86% dry matter).

The crude oil was pre-clarified by heating in the presence of water and citric acid solution (15–20 minutes at 80–90 °C), the precipitated colloidal compounds were removed by centrifugation. The pre-clarified oil was heated to 90 °C and sodium hydroxide was added to convert the free fatty acids to their sodium soaps which were removed as soap stock. The neutralised oil was bleached by mixing with Fullers Earth for 30 minutes at 80–90 °C and after filtering, the oil was refined further by vacuum-water-steam-distillation to remove constituents that are volatile in water vapour. The processes are described in detail in the following flow charts.

Analytical samples were stored at -18 °C or below for about to 227 days (seeds) and 28 days (processed fractions) before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984-M001. The reported LOQs were 0.01 mg/kg for each analyte

Table 216 Residues in seed, oil, meal and press cake from supervised trials on oilseed rape in France, Germany and Italy, involving two foliar applications of fluopyram (500 SC)

RAPE SEED Country, year Location (variety)	Application			PHI, (days)	Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)		matrix	parent	PAA	BZM	PCA	
Germany, 2006 Werl-Westönnen (Smart) Rape, winter	0.125	0.0415	300	50	seed oil (pressed) oil (extracted) oil (crude) oil (refined) press cake meal (extracted)	0.11 0.13 0.15 0.14 0.07 0.14 0.15	0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01 < 0.01 < 0.01 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3609/06 0406-06
Germany, 2006 Burscheid (Talent) Rape, winter	0.125	0.0415	300	57	seed oil (pressed) oil (extracted) oil (crude) oil (refined) press cake meal (extracted)	0.04 0.04 0.05 0.05 0.04 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-3609/06 0409-06
France (S), 2006 Latille (Savana) Rape, winter	0.125	0.0415	300	50	seed oil (pressed) oil (extracted) oil (crude) oil (refined) press cake meal (extracted)	0.06 0.05 0.06 0.06 0.05 0.04 0.04	0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	RA-3610/06 0625-06
Italy, 2006 Bologna (Molino) Rape, summer	0.125	0.038	330	34	seed oil (pressed) oil (extracted) oil (crude) oil (refined) press cake meal (extracted)	0.07 0.18 0.13 0.15 0.12 0.05 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2610/06 0626-06

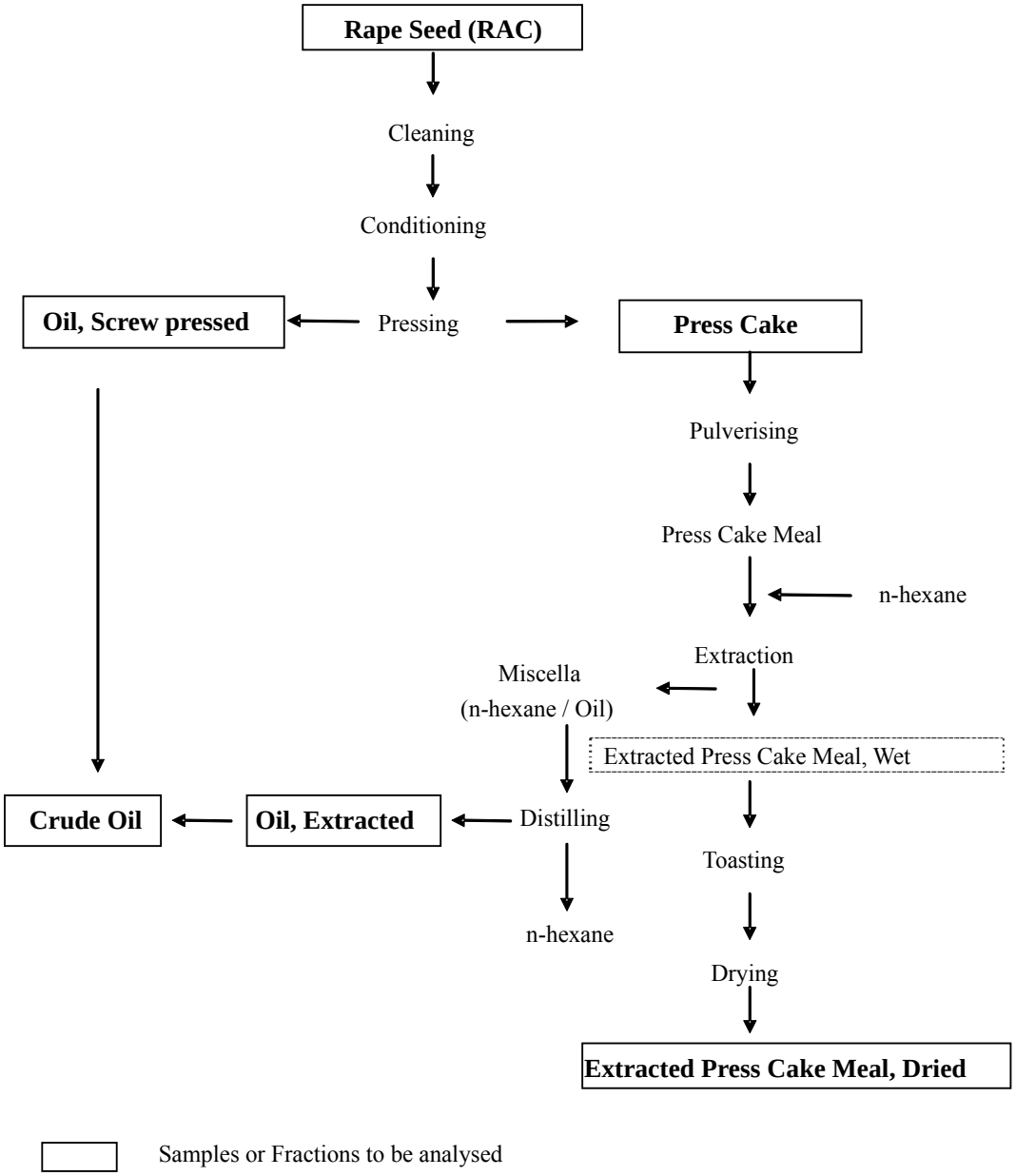


Figure 11 Processing of rape seeds into screw pressed oil, press cake, extracted oil, crude oil and extracted press cake meal, dried

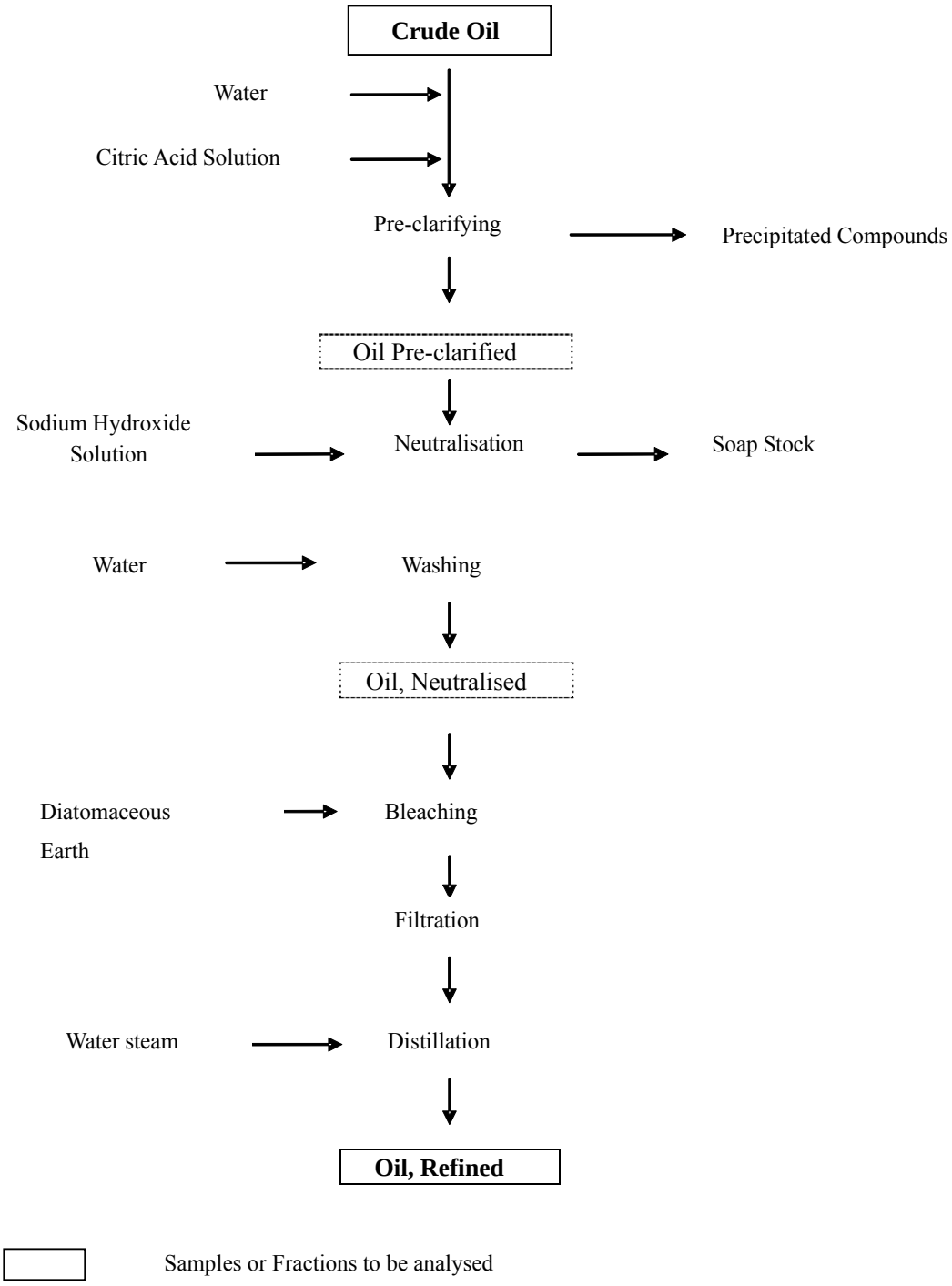


Figure 12 Processing of rape crude oil into refined oil

An oil seed rape (canola) processing trial was conducted in USA to measure the fluopyram residues in rape seed, meal and refined oil and following exaggerated (5×) field treatment of oil seed rape [Lenz, 2008, Ref: RAGMP034]. One unreplicated 613 square metre plot of oil seed rape was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 13 days apart, using a mechanised crop boom sprayer to apply 1.26–1.32 kg ai/ha in 161–180 litres water/ha.

Bulk samples of about 27 kg seed were harvested by combine harvester 14 days after the last application of fluopyram, frozen within 1 hour and stored frozen for up to 360 days before processing using procedures that simulated commercial processing practices.

After aspiration and screening, seeds were flaked, heated to 82–100 °C for 10–15 minutes and mechanically pressed to extract crude oil. The press cake was triple-extracted (49–60 °C for 30 minutes) and double-washed (15 minutes) with hexane. The extracted crude oil was separated from the hexane solvent by heating to 90–95 °C and alkali refined to produce soapstock and refined oil which was then bleached (by mixing with activated bleaching earth), filtered and deodorized by adding citric acid.

The samples analysed in this study were held in frozen storage for a maximum of 360 days (seeds), 153 days (oil) and 12 days (meal) prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ for all matrices was 0.01 mg/kg.

Table 217 Fluopyram residues in rape seed, meal and refined oil from a supervised oil seed rape trial in USA involving two foliar applications (500 SC) – 14 day PHI

RAPE SEED Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2006 Sabin, MN (5630 Invigor)	1.32 1.26	0.82 0.7	161 180	seed meal refined oil	0.844, 0.803, 0.843 0.323, 0.268, 0.252 0.012, < 0.01, < 0.01				RAGMP034 GM011-06PA

Cotton seed

A cotton seed processing trial was conducted in USA to measure the fluopyram residues in cotton seed, meal, hulls and refined oil following bare ground application prior to planting cotton [Dallstream & Fischer, 2008, Ref: RAGMY005]. One unreplicated 470 square metre bare ground plot was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 5 days apart, using a knapsack sprayer to apply 1.25 kg ai/ha in about 94 litres water/ha (2.5 kg ai/ha/season), 12 days before planting cotton.

Bulk samples of about 45 kg seed were harvested at maturity, about 5 months after the last treatment/planting and stored frozen for up to 7 months before processing using procedures that simulated commercial processing practices.

After cleaning (to remove gin trash) and ginning (to remove most of the lint), a representative sample of cottonseed was delinted, cracked using a roller mill and screened to separate the hull material from the kernels. The kernel material was heated to 80–90 °C for 10-15 minutes, flaked and steam extruded to produce collets. Crude oil was obtained from the oven-dried collets by triple-extraction (49–60 °C for 30 minutes) and double-washing (15 minutes) with hexane. The extracted crude oil was separated from the hexane solvent by heating to 90–96 °C and alkali refined to produce soapstock and refined oil which was then bleached (by mixing with activated bleaching earth), filtered and deodorized by adding citric acid.

The samples analysed in this study were held in frozen storage for a maximum of 205 days (seeds) and 34 days (meal, hulls, oil) prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ for all matrices was 0.01 mg/kg.

Table 218 Fluopyram residues in cotton seed, meal, hulls and refined oil from a supervised trial in USA involving two bare ground applications (500 SC), 12 days before planting cotton

COTTON SEED Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2008 Newport, AR (American Upland)	1.25 1.25	1.34 1.33	93 94	seed meal refined oil hulls	< 0.01 (3) < 0.01 (3) < 0.01 (3) < 0.01 (3)				RAGMY005 GM142-07PA

Sunflower seed

A sunflower seed processing trial was conducted in USA to measure the fluopyram residues in sunflower seed, meal and refined oil following exaggerated (5×) field treatment on sunflowers [Lenz, 2008, Ref: RAGMP071]. One unreplicated 279 square metre plot was treated with 2 foliar spray applications of fluopyram (SC 500 formulation), 13 days apart, using a knapsack sprayer to apply 1.26–1.27 kg ai/ha in 180 L water/ha.

Bulk sunflower seed heads (39 kg) were harvested by hand, 13 days after the last application and placed into a mechanical combine to thresh the seeds. The samples were frozen within 4 hours, and placed in frozen storage for about 14 months before processed using procedures that simulated commercial practices.

After aspiration and cleaning (to remove trash), a representative seed sample was cracked using a roller mill and aspirated to separate the hull material from the kernels. The kernel material was moisture conditioned to 12%, heated to 88–104 °C and extruded to liberate the crude oil. Residual oil in the presscake was triple-extracted (49–60 °C for 30 minutes) and double-washing (15 minutes) with hexane. The extracted crude oil was separated from the hexane solvent by heating to 88–96 °C and alkali refined to produce soapstock and refined oil which was then bleached (by mixing with activated bleaching earth), filtered and deodorized by adding citric acid.

The samples analysed in this study were held in frozen storage for a maximum of 337 days (seeds) and 21 days (meal, oil) prior to extraction and analysis for fluopyram using LC/MS/MS Method GM-001-P07-01. The reported LOQ for all matrices was 0.01 mg/kg.

Table 219 Fluopyram residues in sunflower seed, meal and refined oil from a supervised trial in USA involving two foliar applications (500 SC formulation) – 13 day PHI

SUNFLOWER SEED Country, year Location (variety)	Application			Fluopyram Residues (mg/kg)					Reference & Comments
	kg ai/ha	kg ai/hL	water (L/ha)	matrix	parent	PAA	BZM	PCA	
USA, 2006 St John, KS (Pioneer 63M91-N402)	1.27 1.26	0.7 0.7	180 180	seed meal refined oil	0.916, 0.918, 0.849 0.024, 0.016, 0.017 < 0.01 (3)				RAGMP071 GM346-06PA

Table 220 Summary of selected processing factors for fluopyram – Household processing

Raw agricultural commodity	Processed commodity	Calculated processing factors [a]	Processing factor (mean or median)
Melons & Watermelon	Pulp	< 0.02, < 0.05, 0.05, < 0.06, < 0.06, < 0.07, < 0.08, < 0.09, 0.09, < 0.11, < 0.13, < 0.13, < 0.13, 0.13, 0.13, < 0.17, < 0.17, < 0.2, < 0.2	< 0.11
Summer squash	Washed squash	0.57	0.57
	Cooked squash	0.46, 1.0	0.73

Raw agricultural commodity	Processed commodity	Calculated processing factors [a]	Processing factor (mean or median)
Strawberry	Washed fruit	0.61, 0.74, 0.81, 0.83, 0.92	0.78
	Preserve	0.25, 0.27, 0.33, 0.4	0.31
	Jam	0.28, 0.58, 0.63, 0.64, 0.74, 1.0	0.65
Blueberries	Washed berries	0.65	0.65
	Cooked berries	0.42	0.42
Cabbage	Washed heads	0.17, 0.25, < 0.5, < 0.5	< 0.36
	Cooked washed heads	< 0.17, < 0.25, < 0.5, < 0.5	< 0.36
	Trimmed heads [b]	0.01, 0.02, 0.02, 0.03, 0.09, 0.34	0.03 (median)
Lettuce	Trimmed heads [b]	0.02, 0.02, 0.03, 0.05, 0.19, 0.2	0.09
	Washed leaves	0.24, 0.85	0.55
Spinach	Washed leaves	0.07	0.07
	Cooked washed leaves	0.06	0.06
Celery	Washed stalks	0.18	0.18
	Trimmed stalks	0.11	0.11
	Trimmed, washed, cooked	0.02	0.02
Broccoli	Washed heads	0.8	0.8
	Cooked washed heads	0.62	0.62
Mustard greens	Washed leaves	0.15	0.15
Plums	Washed fruit	0.49	0.49
	Dried fruit	1.1	1.1

[a] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of the total residue in the processed item divided by the total residue in the RAC.

[b] Wrapper leaves removed

Table 221 Summary of selected processing factors for fluopyram – Commercial processing

Raw agricultural commodity	Processed commodity	Calculated processing factors [a]	Processing factor (mean or median)
Grape	Washed berries	0.5, 0.59, 0.66, 0.74	0.62
	Juice	< 0.02, < 0.02, < 0.02, < 0.03, 0.53	< 0.02 (median)
	Red wine (young)	0.1, 0.14, 0.16, 0.22	0.16
	Red wine [b]	0.14, 0.17, 0.2, 0.2	0.18
	Pomace (wet)	2.2, 3.1, 3.6, 3.9	3.2
	Raisins	2.0, 2.4, 2.9, 3.2, 6.6	2.9 (median)
	Jelly	0.14	0.14
Tomato	Washed fruit	0.32, 0.51, 0.67, 0.92, 0.94	0.67
	Juice	0.09, 0.27, 0.42, 0.44, 0.56	0.36
	Pulp	0.08, 0.09, 0.11, 0.13	0.1
	Preserve	0.07, 0.18, 0.21, 0.25, 0.33	0.21
	Puree	0.18, 0.46, 0.73, 0.94, 2.2	0.73 (median)
	Paste	0.46	0.46
Potato	Washed tubers	0.7	0.7
	Peeled tubers	< 0.64	< 0.64
	Chips	< 0.64	< 0.64
	Flakes	1	1
	Wet peel	4.3	4.3

Raw agricultural commodity	Processed commodity	Calculated processing factors [a]	Processing factor (mean or median)
Sugar beet	Sugar	1.3	1.3
	Molasses	0.92	0.92
	Dried pulp	1.3	1.3
Soya bean	Meal	0.05	0.05
	Refined oil	0.02	0.02
	Flour	0.04	0.04
	Soymilk	< 0.02	< 0.02
	Asp grain fraction	223	223
Orange	Peel	1.8	1.8
	Pulp	0.16	0.16
	Juice	0.01	0.01
	Oil	16	16
Apples	Washed fruit	0.36, 0.43, 0.55, 0.7, 1.4	0.55 (median)
	Dried fruit	0.03, 0.62, 0.64, 0.88, 0.91	0.64 (median)
	Peeled fruit	0.03, 0.18, 0.24, 0.25, 0.27	0.24 (median)
	Sauce	0.01, 0.24, 0.36, 0.36, 0.63	0.36 (median)
	Juice	< 0.05, < 0.09, < 0.09, < 0.13, 0.44	< 0.09 (median)
	Wet pomace	1.2, 1.7, 2.3, 2.5, 4.1	2.3 (median)
Plums	Washed fruit	0.49	0.49
	Dried fruit	1.1	1.1
Wheat	Wheat bran	2.7	2.7
	Middlings	0.34	0.34
	Shorts	0.75	0.75
	Flour	0.12	0.12
	Asp grain fraction	70	70
	Wheat germ	2.4	2.4
Peanut	Nuts (roasted)	0.26	0.26
	Meal	0.19	0.19
	Butter	0.22	0.22
	Oil	0.01	0.01
Maize	Grits	0.51	0.51
	Meal	0.81	0.81
	Flour	0.85	0.85
	Bran	2.7	2.7
	Starch	0.36	0.36
	Oil (wet milled)	0.58	0.58
	Oil (dry milled)	< 0.36	< 0.36
	Asp grain fraction	161	161
Rape seed	Oil (crude)	1.0, 1.3, 1.3, 2.1	1.4
	Oil (refined)	0.01, 0.64, 0.83, 1.0, 1.7	0.71
	Meal	0.29, 0.34, 0.67, 0.75, 1.4	0.69
Cotton seed	Oil (refined)	< 0.01	< 0.01
	Meal	0.022	0.022

Raw agricultural commodity	Processed commodity	Calculated processing factors [a]	Processing factor (mean or median)
Sunflower seed	Oil (refined)	< 0.01	< 0.01
	Meal	0.02	0.02

[a] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of the total residue in the processed item divided by the total residue in the RAC.

[b] Wine at first taste (approx 132 days bottled storage)

LIVESTOCK FEEDING STUDIES

Poultry

A poultry feeding study was conducted by Billian, 2008 [Ref: MR07/243] to measure expected residues of fluopyram and its metabolites BZM total -olefines in poultry exposed to fluopyram residues in feed.

Laying hens (20 weeks old and weighing approximately 1.5 kg) were divided into a control group of 9 animals, a depuration study group of 15 animals (in 3 sub-groups) and four groups of 12 animals (for the dose-response investigation). Each group was dosed (in commercially prepared laying hen feed) for 28 consecutive days with fluopyram at dose rates of 0, 0.05, 0.49, 1.6 or 4.8 ppm in feed, approximating 3.4, 35, 110 and 320 µg/kg bw/day (330 µg/kg bw/day for the depuration study group) and designated as the 0×-control, 0.1×, 1×, 3×, 10× and 10×-depuration dose groups respectively.

Eggs were collected at intervals during the 28-day dosing period and the hens were sacrificed on day 28, with liver, fat, muscle, and overlaying skin together with any associated fat being collected for analysis. Eggs were also collected from the 10×-depuration dose group at regular intervals from 7 days before the end of the dosing period until 21 days after the last dose. Hens from this group were also sacrificed 8, 13 and 21 days after the last dose and samples of liver, fat, muscle, and skin (with fat) were collected for analysis

Tissues and egg (without shells) were analysed within 30 days for fluopyram, its BZM metabolite and the sum of the two -olefine isomers by HPLC-MS/MS using Method 01061. Mean recovery rates ranged 84–123% in egg and tissue samples fortified with 0.01, 0.1, 0.5, 1.0 and 2.0 mg/kg fluopyram or BZM or with 0.02, 0.2, 1.0, 2.0 and 4.0 mg/kg total-olefines (1:1 ratio for the two isomers). The limit of quantitation (LOQ) was 0.01 mg/kg for fluopyram and BZM (expressed as fluopyram equivalents). In case of the total-olefines, residues were calculated as sum of the two individual olefine isomers (LOQ of 0.02 mg/kg for all matrices). The reported LODs were 0.003 mg/kg for fluopyram, 0.001 mg/kg for benzamide and 0.004 mg/kg for the sum of the two olefine isomers.

Residues of fluopyram and its metabolites in eggs reached a plateau within 21 days after the first dose. Residues of fluopyram in eggs were not detectable in the three lower dose groups and were either not detectable of were < 0.01 (LOQ) in eggs from the highest (10×) dose group. The plateau level for BZM in eggs was 0.71 mg/kg for the 10× dose group and below the LOQ of 0.01 mg/kg for the 0.1× dose group (0.08 mg/kg for the 1× dose group). The plateau level for the total-olefines in eggs was 0.02 mg/kg for the 10× dose group and below the LOQ of 0.02 mg/kg for the 0.1× dose group.

Table 222 Residues of fluopyram and its metabolites (BZM, total-olefines) in eggs

Day	Residues (mg parent equivalents/kg) in eggs					
	0.05 ppm dose group (0.1×)			0.49 ppm dose group (1×)		
	Fluopyram	BZM	Total-olefines	Fluopyram	BZM	Total-olefines
0	nd	nd	nd	nd	nd	nd
1	nd	nd	nd	nd	< 0.01	nd
2	nd	nd	nd	nd	0.01	nd
5	nd	< 0.01	nd	nd	0.04	nd
7	nd	< 0.01	nd	nd	0.05	nd
9	nd	< 0.01	nd	nd	0.06	nd
12	nd	< 0.01	nd	nd	0.07	nd
14	nd	< 0.01	nd	nd	0.06	nd
16	nd	< 0.01	nd	nd	0.06	nd
21	nd	< 0.01	nd	nd	0.08	nd
23	nd	< 0.01	nd	nd	0.07	nd
26	nd	< 0.01	nd	nd	0.07	nd
28	nd	< 0.01	nd	nd	0.08	nd
Day	1.6 ppm dose group (3×)			4.8 ppm dose group (10×)		
	Fluopyram	BZM	Total-olefines	Fluopyram	BZM	Total-olefines
0	nd	nd	nd	nd	nd	nd
1	nd	0.01	nd	nd	0.02	nd
2	nd	0.03	nd	nd	0.1	nd
5	nd	0.1	nd	nd	0.36	nd
7	nd	0.13	nd	nd	0.45	< 0.02
9	nd	0.15	nd	nd	0.5	< 0.02
12	nd	0.17	nd	< 0.01	0.59	< 0.02
14	nd	0.17	nd	< 0.01	0.56	< 0.02
16	nd	0.18	nd	< 0.01	0.63	< 0.02
21	nd	0.22	nd	nd	0.72	< 0.02
23	nd	0.2	nd	nd	0.7	< 0.02
26	nd	0.2	nd	nd	0.7	< 0.02
28	nd	0.21	< 0.01	nd	0.72	0.02

Results are the mean residues from 3 sub-groups in each dose group

Residues of fluopyram in tissues were either not detectable or in single animals were found occasionally at the LOQ of 0.01 mg/kg.

The BZM metabolite was found in most of the tissue samples. In skin with fat and muscle samples from the 0.1× group, residues were below the LOQ level of 0.01 mg/kg. In skin with fat and muscle samples from the 1× group residues of 0.04 mg/kg and 0.03 mg/kg (muscle) were determined (mean value of the three subgroups each). In skin with fat and muscle samples from the 10× group residues of 0.41 mg/kg and 0.29 mg/kg (muscle) were determined (mean value of the three subgroups each). Residues in liver were between 0.01 mg/kg (0.1× dose group) and 1.4 mg/kg (10× dose group), showing a clear dose-response.

Residues of total-olefines in tissues were found mainly in skin/fat. In liver, residues were below the LOQ for the 10× dose group and at the LOQ (0.02 mg/kg) in muscle for the 10× dose

group. In skin/fat, residues were below the LOQ in the 0.1× and 1× dose groups and 0.02 mg/kg and 0.05 mg/kg for the 3× and 10× dose groups respectively.

Table 223 Residues of fluopyram and metabolites in hen eggs and tissues

Feeding level	0.05 ppm (0.1× group)		0.49 ppm (1× group)		1.6 ppm (3× group)		4.8 ppm (10× group)	
Fluopyram residues (mg/kg)								
Commodity	mean	max	mean	max	mean	max	mean	max
eggs	nd	nd	nd	nd	nd	nd	nd	nd
Skin with Fat	nd	nd	nd	nd	nd	nd	nd	< 0.01
Liver	nd	nd	nd	nd	nd	nd	nd	nd
Muscle	nd	nd	nd	nd	nd	nd	nd	nd
BZM residues (mg/kg)								
Commodity	mean	max	mean	max	mean	max	mean	max
eggs	nd	< 0.01	0.08	0.09	0.22	0.23	0.72	0.92
Skin with Fat	< 0.01	< 0.01	0.04	0.04	0.1	0.11	0.41	0.63
Liver	0.01	0.02	0.16	0.16	0.41	0.43	1.4	1.6
Muscle	< 0.01	< 0.01	0.03	0.04	0.09	0.1	0.29	0.33
Total-olefines residues (mg/kg)								
Commodity	mean	max	mean	max	mean	max	mean	max
eggs	nd	nd	nd	nd	nd	< 0.02	0.02	0.03
Skin with Fat	nd	nd	< 0.02	< 0.02	0.02	0.03	0.05	0.08
Liver	nd	nd	nd	nd	< 0.02	< 0.02	< 0.02	0.02
Muscle	nd	nd	nd	nd	nd	nd	0.02	0.06

Results are the mean residues from 3 sub-groups (of 3 animals each) in each dose group

In the depuration study, where 5 hens/group were fed at the 10× feeding level for 28 consecutive days, with eggs being collected at intervals during the last 7 days of dosing and during the subsequent 21 days and the subgroups sacrificed on days 36, 41 and 49, residues of BZM and total-olefines decreased steadily with time.

In eggs, average BZM residues were 0.83 mg/kg on day 28 and decreased steadily to 0.03 mg/kg on day 49. The total-olefine residues averaged 0.03 mg/kg on day 28 and decreased steadily to < 0.02 on day 49. In skin/fat, liver and muscle samples, BZM residues were 0.41 mg/kg, 1.4 mg/kg, and 0.29 mg/kg on day 28, respectively, and decreased to 0.05 mg/kg in liver, 0.02 mg/kg (skin with fat) and 0.01 (muscle) on day 49.

Table 224 Residue depuration of fluopyram and its BZM and total-olefine metabolites in eggs and tissues from hens dosed daily for 28 days with 4.8 ppm fluopyram in the diet

Sampling time (days)	Eggs (mg/kg)	Liver (mg/kg)	Skin/fat (mg/kg)	Muscle (mg/kg)
Fluopyram				
21	nd			
23	nd			
26	nd			
28	nd	nd	nd	nd
30	nd			

Sampling time (days)	Eggs (mg/kg)	Liver (mg/kg)	Skin/fat (mg/kg)	Muscle (mg/kg)
33	nd			
36	nd	nd	nd	nd
37	nd			
40	nd			
41	nd	nd	nd	nd
44	nd			
48	nd			
49	nd	nd	nd	nd
BZM				
21	0.73			
23	0.68			
26	0.7			
28	0.83	1.4	0.41	0.29
30	0.67			
33	0.47			
36	0.3	0.49	0.12	0.21
37	0.23			
40	0.13			
41	0.11	0.19	0.05	0.08
44	0.07			
48	0.04			
49	0.03	0.05	0.02	0.01
Total-olefines				
21	< 0.02			
23	< 0.02			
26	< 0.02			
28	0.03	< 0.02	0.06	nd
30	< 0.02			
33	< 0.02			
36	< 0.02			
37	< 0.02			
40	< 0.02			
41	< 0.02	< 0.02	0.03	nd
44	< 0.02			
48	< 0.02			
49	< 0.02	nd	nd	nd

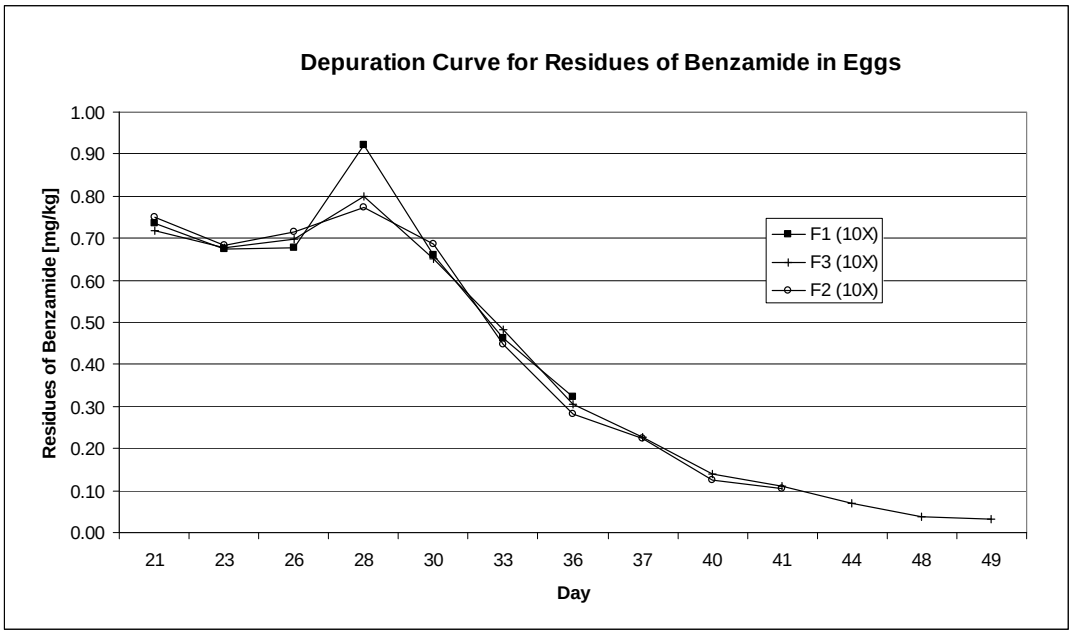


Figure 13 Depuration curve of residue of BZM in eggs.

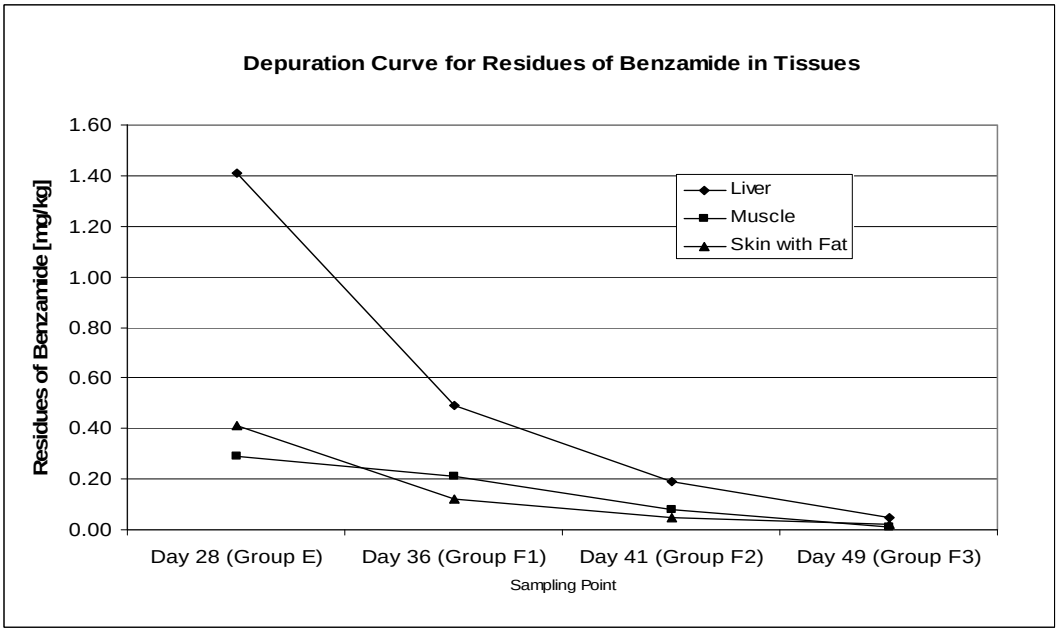


Figure 14 Depuration curve of residue of BZM in hen tissues.

Lactating dairy cows

A feeding study with fluopyram on lactating dairy cows was reported by Schoening & Wolters, 2008 [Ref: MR-07/367]. In this study, dairy cows (3–7 years old and weighing 484–635 kg) were dosed orally, via double-coated gelatine capsules, for 29 consecutive days with fluopyram at levels of 23 mg/capsule, 232 mg/capsule, 696 mg/capsule and 2323 mg/capsule corresponding to feed concentrations of 1.5 ppm (0.04 mg/kg bw/day), 14.4 ppm (0.44 mg/kg bw/day), 44 ppm (1.21 mg/kg bw/day) and 133 ppm (4.38 mg/kg bw/day). These were designated as the 0.1×, 1×, 3×

and 10× dose groups respectively. A depuration study was conducted with a separate group of three cows dosed with 146 ppm dry feed/day (dose group 10×-depuration) for 29 consecutive days, with milk being collected at intervals during the last 9 days of dosing and over the following 21 days, with animals being sacrificed 7, 14 and 21 days after the last dose was administered. Average feed consumption (55% corn silage, 31% grass silage and 13% corn, soy and rape seed), was about 43 kg (fresh weight)/cow/day and milk production during the dosing phase ranged from 10.3–26.2 kg/cow/day. Milk fat content in the centrifuged 'cream' ranged from 71–78%.

Duplicate milk samples from the animals of the groups 0×, 0.1×, 1×, 3×, and 10× were taken before the 1st dosing (day -7) and at intervals during the dosing period and stored frozen for less than 30 days before analysis. On day 30, less than 24 hours after the final dosing, the animals were sacrificed and liver, muscle, kidney and fat (perirenal, subcutaneous and mesenteric) were sampled and stored frozen for less than 30 days before analysis.

Tissue and milk samples were analysed for fluopyram and its metabolites by HPLC-MS/MS (Method 01061) using isotopically labelled internal standards. Mean recovery rates in the animal matrices fortified at 0.01 mg/kg and 0.10 mg/kg (fluopyram and BZM and at 0.02 mg/kg and 0.20 mg/kg for the total-olefines (1:1 isomer ratio) ranged from 71–135%. The limit of quantitation (LOQ) was 0.01 mg/kg for fluopyram and the BZM metabolite and 0.02 mg/kg for the sum of the two individual -olefine isomers. The limit of detection LOD was set to 0.003 mg/kg for fluopyram, 0.001 mg/kg for BZM and 0.004 mg/kg for the total-olefines in all matrices.

Residues of fluopyram were not detectable in milk from the 0.1× dose group and were at or below the LOQ (0.01 mg/kg) in the 1× dose group. Residues in the 3× dose group were between 0.02 and 0.05 mg/kg and reached a maximum of 0.16 mg/kg at day-4 in the 10× dose group.

Mean residues of BZM (expressed as fluopyram equivalents) in milk reached a maximum of 0.04 mg/kg at day 21 in the 0.1× dose group, 0.25 mg/kg at day 24 in dose group 1×, 0.65 mg/kg at day 26 in the 3× dose group and 1.7 mg/kg at day 17 in the 10× group.

The mean residues of total-olefines in milk (expressed as fluopyram-equivalents) were not detectable in the 0.1× dose group, below the LOQ (0.02 mg/kg) in the 1× group and were between 0.02 and 0.03 mg/kg from day 8 in the 3× dose group. Residues in the 10× group reached 0.1–0.12 mg/kg during the last 7 days of the dosing period.

Table 225 Residues of fluopyram and its metabolites (BZM and total-olefines) in milk

Day	Residues (mg parent equivalents/kg) in milk					
	1.5 ppm dose group (0.1×)			14.4 ppm dose group (1×)		
	Fluopyram	BZM	Total-olefines	Fluopyram	BZM	Total-olefines
-7	nd	nd	nd	nd	nd	nd
1	nd	< 0.01	nd	< 0.01	0.01	nd
2	nd	< 0.01	nd	0.01	0.05	nd
4	nd	0.01	nd	0.01	0.12	< 0.02
8	nd	0.02	nd	0.01	0.19	< 0.02
10	nd	0.02	nd	0.01	0.19	< 0.02
13	nd	0.02	nd	0.01	0.21	< 0.02
17	nd	0.02	nd	< 0.01	0.22	< 0.02
21	nd	0.04	nd	0.01	0.24	< 0.02
24	nd	0.02	nd	0.01	0.25	< 0.02
26	nd	0.02	nd	< 0.01	0.23	< 0.02
29	nd	0.02	nd	< 0.01	0.25	< 0.02

Day	Residues (mg parent equivalents/kg) in milk					
Day	44 ppm dose group (3×)			133 ppm dose group (10×)		
	Fluopyram	BZM	Total-olefines	Fluopyram	BZM	Total-olefines
-7	nd	nd	nd	nd	nd	nd
1	0.02	0.03	nd	0.09	0.08	< 0.02
2	0.05	0.13	< 0.02	0.13	0.34	0.03
4	0.05	0.33	< 0.02	0.16	0.81	0.05
8	0.03	0.55	0.02	0.11	1.8	0.09
10	0.03	0.54	0.02	0.09	1.5	0.08
13	0.03	0.53	0.02	0.08	1.6	0.09
17	0.02	0.53	0.02	0.08	1.7	0.08
21	0.02	0.45	0.02	0.1	1.2	0.1
24	0.05	0.52	0.03	0.12	1.3	0.1
26	0.03	0.65	0.03	0.11	1.3	0.12
29	0.03	0.56	0.02	0.1	1.4	0.1

Milk taken from the 10× dose group was separated by centrifugation into skim milk and 'cream' (measured at 71–78% milk fat). The mean residues of fluopyram in skim milk and cream were 0.02 mg/kg and 1.3 mg/kg, respectively. The mean residues of the BZM metabolite in skim milk and cream were 1.4 mg/kg and 0.85 mg/kg, respectively. The mean value of the total-olefines in skim milk and cream were below the LOQ (0.02 mg/kg) and 1.0 mg/kg, respectively.

In tissues, fluopyram residues were found mainly in liver, where residues between 0.25 mg/kg (0.1× dose group) and 4.0 mg/kg (10× dose group) were measured. Residues of fluopyram in fat were 0.04–0.69 mg/kg (mesenteric fat), 0.04–0.57 mg/kg (subcutaneous fat) and 0.04–0.49 mg/kg (perirenal fat). The highest residues of fluopyram found in kidney were 0.07 mg/kg (10× dose group) and in muscle, maximum residues were 0.03 mg/kg (10× dose group). The fluopyram residues showed a clear dose-response.

The BZM metabolite was also found mainly in liver, where residues (fluopyram-equivalents) ranged from 0.1 mg/kg (0.1× dose group) to 6.9 mg/kg (10× dose group). Maximum BZM residues (10× dose group) in fat were 0.72 mg/kg (mesenteric fat), 1.0 mg/kg (subcutaneous fat) and 0.85 mg/kg (perirenal fat). Residues in kidney and muscle were up to 1.6 mg/kg and 1.4 mg/kg respectively, in the 10× dose group. BZM residues showed a clear dose-response.

Residues of total-olefines in tissues were mainly found in fat, with residues (fluopyram-equivalents) up to 0.85 mg/kg (perirenal fat), 0.9 mg/kg (mesenteric fat) and 0.55 mg/kg (subcutaneous fat) in the 10× dose group. Residues of total-olefines ranged from 0.04 to 0.5 mg/kg in liver and up to 0.13 mg/kg in kidney and up to 0.04 mg/kg in muscle. The total-olefines showed a clear dose-response.

Table 226 Residues of fluopyram and its BZM and total-olefine metabolites in cow tissues

Feeding level	1.5 ppm (0.1×)		14.4 ppm (1×)		44 ppm (3×)		133 ppm (10×)	
Fluopyram residues (mg/kg)								
Commodity	mean	max	mean	max	mean	max	mean	max
Milk skim milk cream	nd	nd	0.01	0.02	0.05	0.09	0.12 0.02 1.3	0.15 0.02 1.4
Perirenal Fat	< 0.01	< 0.01	0.04	0.06	0.25	0.33	0.49	0.6

Feeding level	1.5 ppm (0.1×)		14.4 ppm (1×)		44 ppm (3×)		133 ppm (10×)	
Mesenteric Fat	< 0.01	< 0.01	0.04	0.07	0.25	0.33	0.69	0.71
Subcutaneous Fat	< 0.01	< 0.01	0.04	0.07	0.25	0.33	0.57	0.65
Liver	0.25	0.26	0.71	0.98	2.07	2.8	4.0	4.0
Muscle	nd	nd	< 0.01	< 0.01	0.02	0.04	0.03	0.03
Kidney	nd	nd	< 0.01	< 0.01	0.03	0.05	0.07	0.08
BZM residues (mg/kg)								
Commodity	mean	max	mean	max	mean	max	mean	max
Milk skim milk cream	0.02	0.02	0.24	0.37	0.57	0.77	1.3 1.4 0.85	1.4 1.5 0.98
Perirenal Fat	< 0.01	< 0.01	0.18	0.33	0.27	0.34	0.85	0.94
Mesenteric Fat	< 0.01	< 0.01	0.16	0.29	0.26	0.31	0.72	0.8
Subcutaneous Fat	0.01	0.01	0.18	0.31	0.37	0.45	1	1.1
Liver	0.1	0.1	1.21	1.9	2.8	3.2	6.9	7.0
Muscle	0.02	0.02	0.29	0.44	0.6	0.79	1.4	1.5
Kidney	0.03	0.03	0.28	0.38	0.72	0.88	1.6	1.6
Total-olefines residues (mg/kg)								
Commodity	mean	max	mean	max	mean	max	mean	max
Milk skim milk cream	nd	nd	< 0.02	0.02	0.03	0.05	0.12 nd 1.0	0.14 nd 1.3
Perirenal Fat	< 0.02	< 0.02	0.09	0.12	0.28	0.29	0.85	0.86
Mesenteric Fat	< 0.02	< 0.02	0.07	0.09	0.29	0.32	0.9	0.94
Subcutaneous Fat	< 0.02	< 0.02	0.06	0.08	0.18	0.23	0.55	0.57
Liver	nd	< 0.02	0.04	0.06	0.12	0.13	0.5	0.58
Muscle	nd	nd	< 0.02	< 0.02	0.02	0.03	0.04	0.04
Kidney	nd	nd	< 0.02	< 0.02	0.04	0.04	0.13	0.15

In the depuration study, where three dairy cows/group were fed at the 10×-depuration feeding level (145.9 ppm) for 29 consecutive days with milk being collected at intervals during the last 9 days of dosing and during the subsequent 21 days and cows sacrificed on days 36, 43 and 50, residues of BZM and total-olefines decreased steadily with time.

The mean residue of fluopyram in milk was 0.12 mg/kg at the end of the dosing period (day 29), decreasing to < 0.01 mg/kg by day 31 (i.e., within 2 days after the last dose). In tissues, initial residues in animals sacrificed 7 days after the last dose, < 0.01 mg/kg in muscle, 0.08 mg/kg in liver, 0.03 mg/kg in kidney and 0.04–0.12 mg/kg in the fat components decreased to < 0.01 mg/kg within 7 days.

Average residues of BZM in milk were 2.7 mg/kg at the end of the dosing period (day 29) and decreased steadily to 0.02 mg/kg on day 43 and to < 0.01 mg/kg at the end of the study (day 50). In fat, liver, kidney and muscle sampled 7 days after the last dose, BZM residues were 0.45–0.58 mg/kg (fat), 2.8 mg/kg (liver), 0.86 mg/kg (kidney) and 0.77 mg/kg (muscle) and these levels decreased to 0.42 mg/kg in liver, 0.05 mg/kg in kidney, 0.19 mg/kg (muscle) on day 50 and were < 0.01 mg/kg in fat after 43 days.

The total-olefine residues in milk were 0.13 mg/kg at the end of the dosing period (day 29) and decreased to 0.03–0.04 mg/kg (days 36–43) and to < 0.02 mg/kg at the end of the study. Seven days after the last dose, total-olefine residues in fat, liver, kidney and muscle, were 0.04–0.13 mg/kg, 0.08 mg/kg, 0.03 mg/kg and < 0.02 mg/kg respectively, decreasing to 0.11–0.28 mg/kg in the fat components, 0.04 mg/kg in liver and < 0.02 mg/kg in kidney and muscle) on day 50.

Table 227 Residue depuration of fluopyram and its BZM and total-olefines in milk and tissues from cows fed daily for 29 days with 146 ppm fluopyram in the diet

Sampling time (days)	Milk [a] (mg/kg)	Liver (mg/kg)	Kidney (mg/kg)	Muscle (mg/kg)	Fat (mg/kg)		
					perirenal	mesenteric	subcutaneous
Fluopyram							
21	0.06						
24	0.1						
26	0.12						
29	0.12						
31	< 0.01						
34	nd						
36	nd	0.08	0.03	< 0.01	0.04	0.13	0.12
38	nd						
41	nd						
43	nd	< 0.01	nd	nd	nd	nd	nd
50		nd	nd	nd	nd	nd	nd
BZM							
21	2.1						
24	2.4						
26	2.5						
29	2.7						
31	2.2						
34	0.82						
36	0.39	2.8		0.77	0.47	0.45	0.58
38	0.15		0.86				
41	0.04						
43	0.02	0.59	0.07	0.15	< 0.01	< 0.01	< 0.01
50		0.42	0.05	0.19	< 0.01	nd	< 0.01
Total-olefines							
21	0.09						
24	0.13						
26	0.18						
29	0.13						
31	0.07						
34	0.05						
36	0.04	0.08	0.03	< 0.02	0.12	0.13	0.04
38	0.04						
41	0.03						
43	0.03	0.08	0.04	< 0.02	0.21	0.26	0.13
50		0.04	< 0.02	< 0.02	0.11	0.11	0.28

[a] mean residues in milk from 3 cows

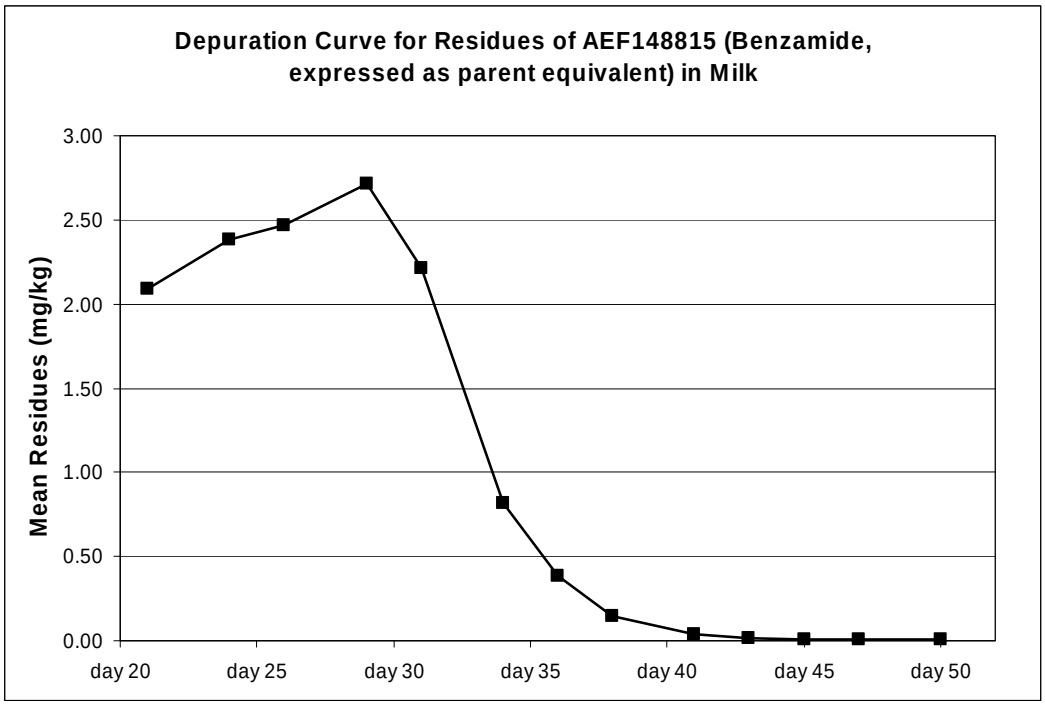


Figure 15 Depuration curve of residue of AE C656948-benzamide in milk

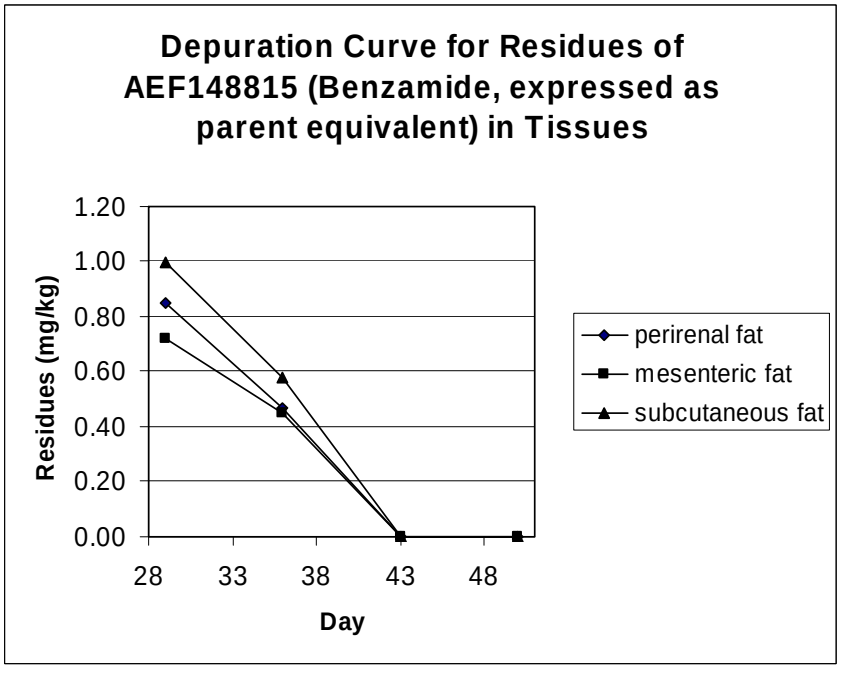


Figure 16 Depuration curve of residue of AE C656948-benzamide in cow tissues

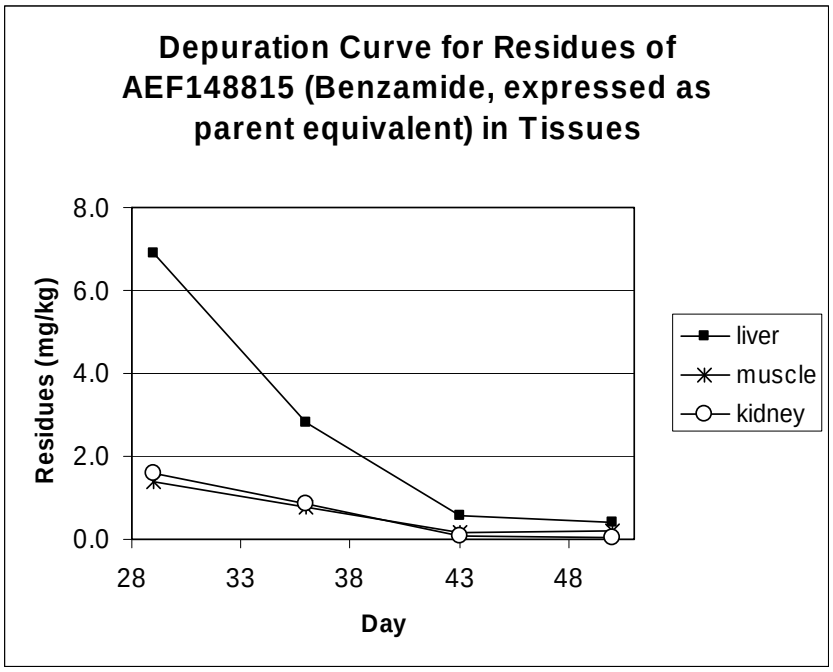


Figure 17 Depuration curve of residue of AE C656948-benzamide in cow tissues

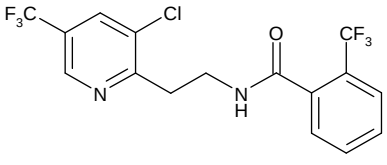
APPRAISAL

Fluopyram, a pyridylethylamide broad spectrum fungicide, is being developed for protection against a range of Ascomycete and Deuteromycete diseases in many horticultural and arable crops. Fungicidal action is by the inhibition of succinate dehydrogenase (complex II) within the fungal mitochondrial respiratory chain, thus blocking electron transport. Fluopyram inhibits spore germination, germ tube elongation, mycelium growth and sporulation. Within plants, fluopyram shows translaminar activity and some upwards movement within the xylem.

Authorisations exist for the use of fluopyram (SC formulation) in China and registration has recently been granted in Romania for use on grapes. Other uses are currently being progressed in Europe and North America.

Residue and analytical aspects of fluopyram were considered for the first time by the present meeting. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability, environmental fate in soil and rotational crop residues.

Fluopyram is N-{2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethyl}-2-(trifluoromethyl)benzamide. It is relatively insoluble in water (15 mg/L), stable to hydrolysis, of low volatility (1.2 x 10^{-6} Pa at 20 °C), has a log P_{OW} of 3.3 and is soluble (> 250 g/L) in methanol, dichloromethane, acetone, ethyl acetate and dimethyl sulfoxide.



Fluopyram (AE C656948)

The following abbreviations are used for the metabolites discussed below:

BZM	-benzamide	2-(trifluoromethyl)benzamide
PAA	-pyridyl-acetic acid	[3-chloro-5-(trifluoromethyl)pyridin-2-yl]acetic acid
PCA [a]	-pyridyl-carboxylic acid	3-chloro-5-(trifluoromethyl)pyridine-2-carboxylic acid
7-OH	-7-hydroxy	N-{2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-2-hydroxyethyl}-2-trifluoromethyl) benzamide
8-OH	-8-hydroxy	N-{2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]-1-hydroxyethyl}-2-trifluoromethyl) benzamide
E-olefine		N-{(E)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl}-2-trifluoromethyl) benzamide
Z-olefine		N-{(Z)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl) benzamide
GA	glucuronic acid	
glyc	glycoside	
glc	glucoside	

[a] Also a metabolite of fluopicolide (M05)

Animal metabolism

The Meeting received information on the metabolism of radio-labelled fluopyram (separately ^{14}C-labelled at the [pyridyl-2,6-^{14}C]- and [phenyl-U-^{14}C]-rings) in rats, lactating goats and laying hens.

In rats, absorption was rapid and residues in tissues at 168 h accounted for < 0.5% (pyridyl ring-label) or 3–5% (phenyl ring-label) of the administered dose, with liver and kidney containing the highest concentrations of residues. Elimination of the radiolabel was via the faeces (39–64%) and the urine (35–60%).

The metabolism of fluopyram in rats was principally oxidative and took place mainly at the ethylene bridge of the molecule. Also, hydrolytic cleavage of the molecule and subsequent oxidation was observed, as was conjugation of several hydroxylated metabolites with glucuronic acid and to a lesser extent with sulphate. Somewhat higher levels of unchanged parent compound in tissues of female rats compared to male rats indicated that the metabolic transformation of the parent compound was generally more pronounced in males.

Lactating goats were orally dosed with [pyridyl-2,6-^{14}C]- or [phenyl-U-^{14}C]-fluopyram at doses equivalent to approximately 45 ppm in the feed for 5 consecutive days and sacrificed 24 hours after the last dose.

The majority of the administered dose was recovered in excreta (29–36% in faeces, 52–53% in urine) with an estimated 7–18% assumed to have remained in the gastro-intestinal tract. Radioactivity retained in tissues or secreted in milk accounted for about 5% (phenyl-label) and less than 1% (pyridyl-label) of the administered dose. Overall 82–93% of administered radioactivity was accounted for.

Radio-labelled residues (fluopyram equivalents) in the phenyl-label study were highest in liver (8.4 mg/kg) and kidney (2.3 mg/kg) and were 0.74 mg/kg in muscle, 0.4 mg/kg in fat and 0.25 mg/kg in milk. Except for fat, much lower residues (but a similar distribution) occurred in the pyridyl-label study, with highest residues in liver (1.4 mg/kg) and kidney (0.4 mg/kg) and were 0.04 mg/kg in muscle and 0.05 mg/kg in milk. In fat, residues reported in the two studies were comparable (0.37–0.4 mg/kg).

In the phenyl-label study, the TRR-values in milk increased continuously over the 5 day dosing period without reaching a plateau, suggesting ongoing absorption, rapid distribution and delayed excretion. The equivalent concentrations in milk samples ranged from 0.05 (8 hours after the first dose) to 0.45 mg/kg (24 hours after the last of 5 doses), with a cumulative total representing

0.56% of the total applied dose. However, in the pyridyl-label study, TRR-values increased during the 8 hour period after each dosing and then decreased (suggesting rapid absorption, distribution and elimination) and reached plateau-levels within 24 hours. The highest TRR-value in milk (0.063 mg/kg) was detected 32 hours after the first dose, with a cumulative total TRR of 0.08% of the total applied dose.

The two metabolism studies were combined using appropriate scaling to obtain an overall picture of the metabolism and the scaled results indicated that residues of BZM represented 98% of the TRR in muscle, 89% TRR in milk, 81% TRR in liver, 73% TRR in kidney and 48% in fat. The Z- and E-olefine isomers made up about 13% and 8% TRR in fat respectively. Residues of the parent compound were about 18% TRR in fat and were less than 1% TRR in all other matrices. Other metabolites were present at less than 10% in any matrix.

Laying hens were orally dosed with [pyridyl-2,6-^{14}C]- or [phenyl-U-^{14}C]-fluopyram at doses equivalent to approximately 26 ppm in the feed for 14 consecutive days and sacrificed 24 hours after the last dose.

The majority of the administered dose was recovered in excreta (83–95%) with an estimated 4–5% assumed to have remained in the gastro-intestinal tract. Radioactivity retained in tissues or found in eggs accounted for about 12% (phenyl-label) and less than 1% (pyridyl-label) of the administered dose. Overall 95–96% of administered radioactivity was accounted for.

Radio-labelled residues in the pyridyl-label study were considerably lower than those in the phenyl-label study (consistent with the results of the goat metabolism studies). Total residues were highest in liver (9.5 mg/kg in the phenyl-label study, 0.54 mg/kg in the pyridyl-label study). In kidney, the phenyl-label residues were 5.8mg/kg and the pyridyl-label residues were 0.24 mg/kg. In muscle, total radioactive residues were 3.3 mg/kg (phenyl-label) and 0.05 mg/kg (pyridyl-label) and were 2.5 mg/kg (phenyl-label) and 0.15 mg/kg (pyridyl-label) in skin. Residues in fat were 1.7 mg/kg in the phenyl-label study and 0.5 mg/kg in the pyridyl-label study.

In eggs, after 8 days, the TRR values in eggs increased to 3.2 mg/kg (phenyl-label study) and to 0.32 mg/kg (pyridyl-label study), reaching a plateau after 8–10 days. The residue-level in the eggs collected from the ovary and oviduct were about 1.5× (phenyl-label) and about 3× (pyridyl-label) higher than those in the laid eggs at the test end, suggesting that the egg yolk was probably the preferred site for the secretion.

The two metabolism studies were combined using appropriate scaling to obtain an overall picture of the metabolism and the scaled results indicated that the BZM metabolite was the major component of the extracted radioactivity identified in muscle (98% TRR), liver (89% TRR), eggs (90% TRR) and fat (68% TRR). The only other metabolite found above 10% TRR was the Z-olefine isomer, found in fat at about 26% TRR. Fluopyram was found in fat at about 2.5% TRR and made up less than 1% TRR in other matrices.

In summary, BZM was the main residue component in edible livestock matrices, making up more than about 90% of the residues in milk, eggs and muscle, about 80–90% of the residues in liver and about 73% of the residues in kidney. The predominant residues in fat were the BZM metabolite (48–68%), the E-/Z-olefine isomers (21–26%) and to a lesser extent fluopyram (18% of the residues in ruminant fat).

The proposed metabolic pathway in livestock (hen and goat) includes hydroxylation of the parent compound to the 7-OH and 8-OH metabolites, the formation of the Z/E-olefines, cleavage of the fluopyram molecule to produce BZM (from the phenyl-label) and PAA (from the pyridyl-label) and conjugation of the hydroxylated parent compound, mainly with glucuronic acid.

These main metabolic reactions are also observed in the rat metabolism studies.

Plant metabolism

The Meeting received information on the metabolism of radio-labelled fluopyram (separately ^{14}C-labelled at the [pyridyl-2,6-^{14}C]- and [phenyl-U-^{14}C]-rings) in grapes, potatoes, and beans after two or

three spray applications at a maximum annual application rate of 0.5 kg ai/ha and on red bell pepper applying 5 mg ai/plant through the drip irrigation system. To facilitate metabolite identification in bell pepper a 4× overdose experiment was also performed at an application rate of 20 mg ai/plant.

In grapes treated three times at 42–49 day intervals up to the start of ripening (total 0.5 kg ai/ha) and sampled 18–19 days later, 77–80% (1.3–1.5 mg/kg fluopyram equivalents) of the radioactive residue in grapes was found in the acetonitrile surface wash and a further 19–20% (0.34 mg/kg) was obtained by triple-extraction in acetonitrile/water. Residues in leaves sampled immediately after the second application were 28–64 mg/kg and were 43–48 mg/kg at harvest 19 days after the third application.

Between 94% and 99% of the TRR was identified, mostly as the unchanged fluopyram (96–98% in grapes and 91–92% in leaves) with minor components being the BZM, the 7-OH (and its glycoside), the 8-OH and the PCA metabolites, all present in grapes or leaves at 1% TRR or less.

In potatoes foliar treated three times at 11–16 day intervals up to berry development (total 0.5 kg ai/ha) and sampled 51 days later, total radioactive residues in tubers were very low (0.008–0.012 mg/kg) and were 22–48 mg/kg in leaves.

Between 74% and 99% of the TRR was identified, mostly as the unchanged fluopyram (23–69% in tubers and 98% in leaves) with the PCA metabolite (pyridyl-label) also present in tubers at about 50% TRR (0.006 mg/kg) and 0.5% (0.11 mg/kg) in leaves. Minor residue components were the BZM and the 7-OH metabolites, present in tubers or leaves at 7% TRR or less.

In beans treated twice at the equivalent of 0.25–0.27 kg ai/ha, 28 days apart (total 0.5 kg ai/ha) and sampled as green beans and foliage (4 days after the last application), as succulent beans (without pods) and straw (29 days after the last application) and as dry beans (29 days after the last application and a further 11 days drying), total radioactive residues were 1.4–3.9 mg/kg (green beans), 0.07–0.17 mg/kg (succulent beans), 0.12–0.31 mg/kg (dry beans), 37–39 mg/kg (foliage) and 17–19 mg/kg in straw (vines and empty pods).

Between 76% and 99% of the TRR was identified, mostly as the unchanged fluopyram (92–99% in green beans and foliage and 87–90% in straw), as the BZM metabolite (52% in succulent beans and 64% in dry beans) or as the PAA and PCA metabolites (30–31% in succulent beans and 23–33% in dry beans). Glycoside-glucuronic acid conjugates of the hydroxylated fluopyram were also found at up to 10% TRR in dry and succulent beans.

In sweet peppers treated with a single drip irrigation application of 5 mg ai/plant and harvested at fruit maturity, 55–96 days later, total radioactive residues were 0.04–0.06 mg/kg (fruit) and 2.3–3.5 mg/kg (plants without fruit). In plants treated at 20 mg ai/plant (4×), TRRs were 6.2–18 mg/kg in plants sampled 33 days after treatment.

Between 78% and 98% of the TRR was identified, mostly as the unchanged fluopyram (16–49% in fruit, 64–70% in plants (without fruits) and 87–88% in the 4× plants), as the BZM metabolite (16% TRR in fruit and 10% in plants or as the PCA metabolite (20–44% TRR) and PAA-glycoside isomers (individually ranging from 13–24% TRR).

In summary, residues of unchanged fluopyram account for the major part of the residues in grapes and green beans and also occur at lower levels in potatoes, drip-irrigated red peppers, dry and succulent beans (without pods), where the commodities are not directly exposed to fluopyram spray applications. In these latter commodities, significant levels of the cleavage products BZM and to a lesser extent the PCA and PAA metabolites also occur, generally at concentrations close to the combined fluopyram plus BZM levels. The PCA metabolite residues were up to 0.1 mg/kg in dry beans, less than 0.05 mg/kg in succulent beans (without pods) and peppers (drip irrigated) and less than 0.01 mg/kg in potatoes. Residues of the PAA metabolite in beans were 0.05–0.07 mg/kg in both the fresh and dry beans (without pods).

The proposed metabolic pathway in plants involves hydroxylation of the parent compound to the 7-OH and 8-OH metabolites and subsequent conjugation (mainly with sugars), cleavage of the parent molecule to produce the BZM, PAA and PCA metabolites.

With the exception of PCA and its methyl-sulfoxide derivative, the main metabolites in plants are also observed in the rat, goat and poultry metabolism studies.

Environmental fate in soil

The Meeting received information the environmental fate and behaviour of fluopyram, including hydrolytic stability, aerobic degradation in soil, photolysis on the soil surface, field dissipation and confined and field rotational crop studies. Radio-labelled fluopyram (separately ^{14}C-labelled at the [pyridyl-2,6-^{14}C]- and [phenyl-U-^{14}C]-rings) were used in the confined soil degradation and rotational crop studies.

Hydrolysis

Fluopyram was stable in sterile aqueous buffered solutions at pH 4, 7 and 9 when stored at 50 °C in the dark for 5 days. One minor degradate was measured, at up to 1.6% applied radiolabel, in the pH 7 and pH 9 solutions at the end of the test period.

Photolysis

After exposure to continuous artificial sunlight for 13 days, radio-labelled fluopyram residues in sterile buffer solutions decreased to 64–72% of the applied phenyl and pyridyl ring radiolabels. In both studies, the one major degradation product was the lactame degradate, comprising 12–13% of the applied radiolabel. A number of minor degradation products were reported, all at less than 4.2% of the applied amount. Based on simple first order degradation kinetics, experimental DT_{50} values of 21–25 days and DT_{90} values of 70–83 days were calculated.

In soil treated with the equivalent of 0.25 kg ai/ha and exposed to artificial irradiation for 23 days, phenyl ring radio-labelled fluopyram was stable, with no organic volatiles or degradation products being detected, about 0.2% of the applied radio-label being mineralised to $^{14}CO_2$ and non-extractable residues increasing to a maximum of 2.3%.

Aerobic soil metabolism

Studies were conducted in four European soils (2 loam soils, a silt-loam and a sandy-loam) with radio-labelled (phenyl ring) fluopyram at a target rate of 0.67 mg/kg soil (equivalent to 250 g/ha, mixed to 2.5 cm depth). In these studies, soils were incubated in the dark for 121–128 days at 20 °C and samples were collected at intervals up to 128 days after application.

Fluopyram was slowly degraded with an estimated single first order (SFO) half-lives of 165–339 day (mean 239 days). The identified soil metabolites were BZM (maximum 1.1%TARafter 30 days), 7-OH (maximum 3.3–4.2%TARafter 30–60 days), PCA (maximum 0.7%TARafter 30 days) and the methyl sulfoxide (maximum 1%TARat 128 days). The estimated single first order half-lives for the 7-OH metabolite ranged from 7.5–16 days..

In two US soils (silty clay loam and sandy loam), radio-labelled fluopyram was applied to soil at 0.11–0.13 mg ai/kg (equivalent to 250 g/ha mixed to 15 cm depth), the soils were incubated in the dark at 25 °C for 365 days and sampled at 12 regular intervals during the 1 year study period.

Fluopyram (parent) residues decreased to 60–71% in the two soils and the SFO model half-lives were 922 days and 484 days. Apart from CO_2, no metabolite of significance was detected in either soil and volatile residues were insignificant. No intermediate metabolites accumulated in the aerobic soil system.

The proposed metabolic pathway of fluopyram in soil includes hydroxylation to form the 7-OH metabolite, cleavage of the parent molecule and the formation of the BZM and the PCA metabolites, the latter being further metabolized to the methyl-sulfoxide. Microbial breakdown leads to the formation of carbon dioxide and soil bound residues and all degradation products were either further transformed to their metabolic downstream products, mineralized (5–24% AR), or substantially integrated into the soil matrix as non-extractable residues (9–15%) after 128 days incubation.

Soil dissipation

In six European field studies involving bare soil (pre-emergent - grass) treatments equivalent to 0.25 kg ai/ha, about 50% of the total applied fluopyram disappearing within 21 to 347 days after treatment in the different soils. Dissipation then proceeded more slowly until the end of the 2-year study when residues remaining were 3–27% of the total applied amount. The time required for dissipation of 90% of the initial concentration of fluopyram ranged from 497 to more than 1000 days.

Interim results of a 4-year soil dissipation study in Europe (two sites) involving annual applications of 0.25 kg ai/ha report an increase in fluopyram residues at the end of each year, from 23–47% of the annual application rate just before the 2nd application to 46–66% of the annual application rate just before the 3rd application.

Five field dissipation studies were conducted in USA with fluopyram (0.5 kg ai/ha) being applied to bare soil. Fluopyram showed biphasic degradation behaviour (double first order in parallel (DFOP) kinetics model) with estimated DT_{50} values of 24 days, 87 days and 166–537 days with 21–44% of the applied residue remaining after 18–22 months. DT_{75} values in these studies ranged from 500 to more than 1000 days.

The observed transformation products were 7-OH, BZM, and PCA metabolites, only found above the LOQ (1.0 µg/kg) in the top 15 cm segment. The highest concentrations found were 3.0 µg/kg (7-OH), 9.7 µg/kg (BZM) and 10 µg/kg (PCA). Residues of BZM and PCA were below the LOQ (1 µg/kg) at all sites within 30 days after treatment. In four of the sites, residues of 7-OH were below the LOQ after 9 months and at one site, were present at about 1.6 µg/kg (mostly in the top 15 cm segment) at the end of the 22-month study period.

Residues in succeeding crops

In rotational crop metabolism studies involving wheat, Swiss chard and turnips as representative crops (small grains, leafy crops and root crops respectively), 1st, 2nd and 3rd rotation crops were planted 30, 139 and 280 days after a single bare-soil spray treatment at a nominal rate of 0.5 kg ai/ha radio-labelled fluopyram (separately ^{14}C-labelled at the [pyridyl-2,6-^{14}C]- and [phenyl-U-^{14}C]-rings).

Total radioactive residues (fluopyram equivalents) were found in all matrices from the 1st rotation crops, at levels ranging from 0.04–0.07 mg/kg (turnip roots) to 6.1–6.7 mg/kg (wheat straw). Total residues decreased in the 2nd rotation crops (0.01–3.5 mg/kg) and decreased further to < 0.01–1.6 mg/kg in the 3rd rotation crops.

Fluopyram was the major residue in most commodities, accounting for 20–95% of the TRR in wheat matrices, Swiss chard, turnip leaves and roots.

The 7-OH metabolite was a significant residue in Swiss chard (21–39% TRR in all rotations), with the BZM metabolite occurring at close to 10% TRR in Swiss chard and turnip leaves of the first rotation. In all other commodities the BZM metabolite was only a minor component. The PCA metabolite was the main residue component in wheat grains from the first rotation, accounting for about half the TRR and the methyl-sulfoxide metabolite, a degradation product of PCA, was a significant residue in wheat grains in the second rotation (49% TRR).

Rotational crop field studies were conducted in Europe where wheat, turnip/carrot and lettuce were planted as representative crops in bare soil treated with 0.5 kg ai/ha or following harvest of lettuce (primary crop) treated with 0.5 kg ai/ha. Plant-back intervals in these trials were 28–49 days (1st rotation), 90–240 days (2nd rotation) and 286–320 days (3rd rotation).

Fluopyram was found in all commodities with highest residues present in wheat straw and forage (0.28 mg/kg and 0.12 mg/kg respectively). Residues were also present in wheat grain, lettuce, turnip and carrot tops and roots at levels of 0.01–0.05 mg/kg.

Residues of the 7-OH metabolite were only found in wheat straw at levels of 0.11–0.08 mg/kg with residues of the BZM and the methyl-sulfoxide metabolites also present at 0.14 mg/kg and 0.07 mg/kg respectively.

In rotational crop studies in North America where alfalfa and cotton were planted 12–14 days after the last of two applications of fluopyram (total 0.5 kg ai/ha), fluopyram residues were not found

in cotton seed, were less than 0.02 mg/kg in cotton gin trash and were generally less than 0.1 mg/kg in alfalfa forage (except in 2 of the 12 sites where residues in 1st cut forage were up to 0.39 mg/kg and up to 0.19 mg/kg in 3rd cut forage). Residues in alfalfa hay, sampled at the same times as the forage showed a similar pattern (taking into account the increased dry matter content).

In a limited rotational crop study in North America where wheat, turnip and mustard greens were planted in rotation with cover crops treated with 2× 0.25 kg ai/ha fluopyram (plant-back interval of about 240 days), residues of fluopyram were found in all commodities except wheat grain and turnip roots. Maximum residues found were 0.12 mg/kg in wheat straw, 0.09 mg/kg in wheat hay, 0.05 mg/kg in wheat forage and 0.04 mg/kg in turnip tops and in fresh mustard leaves.

In summary, at plant-back intervals of more than 28 days, residues of fluopyram can be expected at low levels (0.01–0.05 mg/kg) in root crops, cereal grain crops and in leafy vegetables grown as rotational crops, with higher levels likely to occur in cereal forage (up to 0.12 mg/kg) and feed commodities (up to 0.28 mg/kg in straw). Levels of up to 0.11 mg/kg of the 7-OH metabolite and up to 0.14 mg/kg of the BZM metabolite could occur in cereal straw and residues of the methyl-sulfoxide metabolite may also occur in cereal grain (up to 0.09 mg/kg), cereal forage (up to 0.06 mg/kg) and cereal straw (up to 0.07 mg/kg).

Methods of analysis

Several analytical methods have been reported for the analysis of fluopyram and for selected metabolites and in animal commodities. The basic approach employs extraction by homogenisation with acetonitrile/water and in some methods, additional clean-up using SPE or liquid/liquid partition. Residues are determined by liquid chromatography with mass spectrometric detection.

The methods for fluopyram and selected metabolites have been validated for a range of substrates with LOQs of 0.01 mg/kg for each analyte (0.05 mg/kg for wheat straw). Studies on extraction efficiency indicated that in most matrices, greater than 80% of the residue can be extracted with acetonitrile/water with single and double extraction and that comparable results can be achieved for fluopyram using acetone/water extraction (as used in the DFG S19 multi-residue method).

In the methods used to measure fluopyram metabolites in plant commodities, dilution under acid conditions allowed the determination of the PCA metabolite and its methyl-sulfoxide derivative while a parallel dilution under basic conditions allowed the determination of fluopyram and the BZM, 7-OH and PAA metabolites.

For analysis of animal commodities, extracts are cleaned-up on a C18 cartridge and further diluted with methanol/water (containing the corresponding internal standards) before analysis for fluopyram, the BZM metabolite and the sum of the two olefine isomers (because of internal inter-conversion between the E-isomer and the Z-isomer).

Based on the results of validation studies and the concurrent recovery rates achieved in the supervised field trials, the available analytical methods fulfilled the following criteria:-

- adequate limit of quantification
- mean recovery 70–110%
- relative standard deviation of recovery rates < 20%
- interfering blanks lower than 30% of the limit of quantification.

The European multi-residue method DFG S19 method used in combination with GC/MS was validated as a multi-residue enforcement method to monitor residues of fluopyram in both plant and animal matrices, but is not applicable as an enforcement method for the BZM metabolite. The US-FDA PAM 1 multi-residue methods (E1 extraction and DG17 detection without Florisil cleanup) are also suitable for detection and enforcement of fluopyram in non-fatty matrices but are also not suitable for measuring the BZM residues. For animal tissues, milk and eggs, a modification of the data collection method was validated as an enforcement method for measuring fluopyram and its BZM metabolite.

Stability of residues in stored analytical samples

Freezer storage stability was tested for a range of representative substrates covering those with a high water content (lettuce), a high starch content (wheat grain), a high protein content (dry pea seed), a high oil content (rape seed) and a high acid content (orange).

Fluopyram and major metabolites (BZM, PAA and PCA) are stable in these representative substrates for at least 24 months in frozen storage. Residues of the PCA metabolite are stable in grapes potato tubers, cabbage leaves and wheat grain for at least 30 months in frozen storage and residues of the methyl-sulfoxide metabolite in wheat forage, straw and grain are stable for at least 25 months in frozen storage.

Definition of the residue

In animal commodities, BZM is the main residue in edible animal tissues, milk and eggs, with the combined E/Z olefine isomers and the parent compound being major components only in fat of ruminants and poultry.

Although a suitable multi-residue method was not available to measure these components, an HPLC-MS/MS method measuring fluopyram and the BZM metabolite has been validated for MRL-compliance and the Meeting recommended that for MRL-compliance, the residue definition should be fluopyram and its BZM metabolite.

For animal commodity dietary intake estimation, in addition to the parent compound and the BZM metabolite, the E/Z olefine isomers contributes about 30% to the final residue in ruminant and poultry fat.

Both BZM (2-(trifluoromethyl)benzamide) and to a much lesser extent the E/Z olefine isomers (N-{(E)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl) benzamide and N-{(Z)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl) benzamide) also occur in rats and are adequately covered in the derived toxicological reference doses and the Meeting agreed that these metabolites should be included in the residue definition.

The compound fluopyram has a log K_{ow} of 3.3, suggesting that it is fat-soluble, and this is supported by the preferential partitioning into milk fat (cream) and fat reported in a cow feeding study. Analysis of skim milk and heavy cream in the cow feeding study also indicated that the combined E/Z olefine isomers also show preferential partitioning into fat (0.12:1). However the BZM metabolite (the major component of the residue in eggs, milk and animal tissues) is not fat-soluble. The Meeting therefore concluded that fluopyram (as defined in the residue definitions for animal commodities) is not fat-soluble.

Fluopyram is the major residue in treated plant commodities and where residues occur in rotational crops, fluopyram is also the major residue. The Meeting also noted that multi-residue analytical methods exist to measure the parent residues and agreed that for MRL-compliance, the residue definition for plant commodities should be fluopyram.

Metabolites identified in the plant metabolism studies at more than 10% TRR are BZM, PCA and PAA. While these metabolites were generally present at not more than 1% TRR and below 0.02 mg/kg in most edible commodities, higher percentages (up to 60% TRR but at relatively low concentrations up to 0.1 mg/kg) were found in commodities not directly exposed to spray applications (drip irrigated peppers, potato tubers and beans without pods). In wheat grown as a rotational crop, PCA was found in grain at up to 56% TRR and 0.23 mg/kg.

In supervised crop field trials, residues of BZM, PCA and to a lesser extent PAA were sometimes detected in a number of commodities, mostly at longer PHIs of 10–21 days and generally at levels below 0.02 mg/kg. Higher levels of BZM and less frequently, PCA and its methyl sulfoxide (up to about 0.1 mg/kg) and PAA (rarely more than 0.05 mg/kg) were found occasionally in some legumes and brassicas, rape seed, grapes, lettuce and strawberries. The related parent residues are usually more than twice the metabolite levels.

Two of the main metabolites in plants (BZM and PAA) are also observed in the animal metabolism studies and the toxicology of these metabolites are addressed in the rat studies and are covered by the derived reference doses. Sufficient toxicology information is available to confirm that the PCA metabolite and its methyl sulfoxide, common metabolites with fluopicolide, are significantly less toxic than fluopyram

The Meeting therefore agreed that the BZM, PCA and PAA metabolites need not be included in the plant commodity residue definitions for MRL enforcement or estimation of dietary intake.

Proposed definition of the residue (for compliance with the MRL and for estimation of dietary intake for plant commodities): *fluopyram.*

Proposed definition of the residue (for compliance with the MRL for animal commodities): *sum of fluopyram and 2-(trifluoromethyl) benzamide, expressed as fluopyram.*

Proposed definition of the residue (for estimation of dietary intake for animal commodities: *sum of fluopyram, 2-(trifluoromethyl)benzamide and the combined residues of N-{(E)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl) benzamide and N-{(Z)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl) benzamide, all expressed as fluopyram.*

The residue is not fat-soluble.

Results of supervised trials on crops

The Meeting received supervised trial data for foliar applications of fluopyram (SC formulations) on a wide range of fruit, vegetable, cereal, tree nut, oilseed, hops and herb crops and for drip-line irrigation treatments to strawberries and cucurbits and for seed treatment to cereals. These trials were conducted mainly in Europe and/or North America.

The NAFTA calculator was used as a tool in the estimation the maximum residue level from the selected residue data set obtained from trials conducted according to GAP. As a first step, the Meeting reviewed all relevant factors related to each data set in arriving at a best estimate of the maximum residue level with the calculator using expert judgement. Then, the NAFTA calculator was employed. If the statistical calculation spreadsheet suggested a different value than that recommended by the JMPR, a brief explanation of the deviation was supplied. Some common factors that may lead to rejection of the statistical estimate include when the number of data points in a data set is < 15 or when there are a large number of values < LOQ.

The Meeting noted that GAP has been authorised for the use of fluopyram (500 SC) on table and wine grapes in Romania and on cucumbers in China.

Grapes

GAP for fluopyram in Romania is 2× 0.25 kg ai/ha (max), PHI 21 days for wine grapes and 3 days for table grapes. Residues in trials in South Europe matching the Romanian wine grape GAP (i.e., PHI 21 days) in wine grapes were: 0.26, 0.34, 0.35, 0.36, 0.44, 0.46 and 0.56 mg/kg (n = 7) and in table grapes were: 0.13, 0.22, 0.28, 0.41, 0.44, 0.61 and 0.63 mg/kg (n = 7). As these data sets were not from different populations, the Meeting agreed to combine the results, giving a data set of: 0.13, 0.22, 0.26, 0.28, 0.34, 0.35, 0.36, 0.41, 0.44, 0.44, 0.46, 0.56, 0.61 and 0.63 mg/kg (n = 14).

In trials matching the GAP for table grapes in Romania (i.e., PHI of 3 days), residues in trials from Europe (on table grapes) were: 0.3, 0.34, 0.55, 0.6, 0.66, 0.96 and 1 mg/kg (n = 7) and on wine grapes were: 0.36, 0.51, 0.57, 0.58, 0.58, 0.63 and 0.63 mg/kg (n = 7). As these data sets were not from different populations, the Meeting agreed to combine the results, giving a data set of: 0.3, 0.34, 0.36, 0.51, 0.55, 0.57, 0.58, 0.58, 0.6, 0.63, 0.63, 0.66, 0.96 and 1 mg/kg (n = 14).

The Meeting agreed to use the data from the European trials on grapes matching the Romanian GAP for table grapes (PHI 3 days) and estimated a maximum residue level of 2 mg/kg, an STMR of 0.58 mg/kg and an HR of 1 mg/kg for fluopyram on grapes. The value derived from use of the NAFTA Calculator was 1.3 mg/kg (unrounded).

Cucumber

Supervised residue trial data and GAP information on the use of fluopyram as a foliar spray on cucumber in China were provided to the Meeting. The GAP for use in China is for up to 3 foliar applications of 0.075 kg ai/ha at 7–10 day intervals and the PHI is 2 days.

In six trials matching this GAP, fluopyram residues were 0.05, 0.07, 0.08, 0.14, 0.17 and 0.19 mg/kg.

The Meeting estimated an STMR of 0.11 mg/kg, an HR of 0.19 mg/kg and recommended a maximum residue level of 0.5 mg/kg. The value derived from use of the NAFTA Calculator was 0.36 mg/kg (unrounded).

Estimation of residues in plant commodities grown as potential succeeding crops

Although the results of the rotational crop studies indicate that potential residues could occur in root crops, cereals and leafy vegetables planted in rotation with cucumbers, the Meeting noted that interim soil accumulation studies suggested that residues could build up following repeat applications and concluded there was insufficient information to estimate a residue plateau level for fluopyram in soil. As this is a prerequisite for estimating possible residues in rotational crops (according to the principles outlined in the JMPR Report 2008, General consideration 2.9), the Meeting was not able to recommend maximum residue levels for fluopyram in rotational crops.

Fate of residues during processing

The effect of processing on the nature of residues was investigated in buffer solutions under conditions simulating pasteurisation, boiling and sterilisation. Fluopyram was shown to be stable under these conditions.

The fate of fluopyram residues has been examined in a number of studies reflecting household washing, peeling and cooking practices and also simulated commercial processing. Estimated processing factors and STMR-Ps for the commodities considered at this Meeting are summarised below.

Summary of relevant processing factors and STMR-P values for fluopyram residues.

Raw agricultural commodity	Processed commodity	Calculated processing factors [a]	Processing factor (mean or median)	RAC STMR (mg/kg)	STMR-P (mg/kg)
Grape	Juice	< 0.02, < 0.02, < 0.02, < 0.03,	< 0.02 (median)	0.58	< 0.012
	Red wine (young)	0.1, 0.14, 0.16, 0.22	0.16	0.58	0.093
	Red wine [b]	0.14, 0.17, 0.2, 0.2	0.18	0.58	0.104
	Pomace (wet)	2.2, 3.1, 3.6, 3.9	3.2	0.58	1.86
	Raisins	2.0, 2.4, 2.9, 3.2, 6.6	2.9 (median)	0.58	1.68

[a] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of the total residue in the processed item divided by the total residue in the RAC.

[b] Wine at first taste (approximately 132 days bottled storage)

In four grape processing studies conducted in Europe, fluopyram residues decreased in juice median processing factor of < 0.02) and in wine (mean processing factor of 0.18) with residues increasing about 3-fold in raisins and wet pomace.

The Meeting agreed to estimate a maximum residue level for dried grapes of 5 mg/kg based on a highest residue for grapes of 1 mg/kg and a median processing factor of 2.9 (1 mg/kg × 2.9 PF). The STMR-P for residues of fluopyram in dried grapes is 1.68 mg/kg and the HR-P is 2.9 mg/kg.

The STMR-P for grape pomace (wet) is 1.86 mg/kg and assuming a default 15% dry matter content, the STMR-P for grape pomace (dry) is 12.4 mg/kg.

The STMR-P for grape juice is 0.012 mg/kg and is 0.1 mg/kg for wine.

The Meeting agreed that for commodities not being considered for maximum residue levels at this meeting, the relevant processing studies would not be reviewed and processing factors would not be estimated at this meeting.

Residues in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of fluopyram in farm animals on the basis of the diets listed in Annex 6 of the 2009 JMPR Report (OECD Feedstuffs Derived from Field Crops). Based on the estimated STMR-P of 12.4 mg/kg for grape pomace (dry) and the 20% contribution to the Australian dairy and beef cattle diets, the maximum and mean residue contribution is 2.48 mg/kg. Grape pomace is not a significant component of poultry diets.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef and dairy cattle, calculated using the animal diets from US/CAN, EU and Australia in the OECD Table (Annex 6 of the 2006 JMPR Report) are summarised below.

		Animal dietary burden, fluopyram, ppm of dry matter diet		
		US/CAN	EU	Australia
Beef cattle	max	0.0	0.0	2.48 [a]
	mean	0.0	0.0	2.48 [c]
Dairy cattle	max	0.0	0.0	2.48 [b]
	mean	0.0	0.0	2.48 [d]
Poultry – broiler	max	0.0	0.0 [e]	0.0
	mean	0.0	0.0 [f]	0.0
Poultry – layer	max	0.0	0.0 [g]	0.0
	mean	0.0	0.0 [h]	0.0

[a] Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

[b] Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

[c] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

[d] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues.

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues.

[g] Highest maximum poultry dietary burden suitable for MRL estimates for poultry eggs.

[h] Highest mean poultry dietary burden suitable for STMR estimates for poultry eggs.

The fluopyram dietary burdens for animal commodity MRL and STMR estimation (residue levels in animal feeds expressed on dry weight) are: 2.48 ppm for beef and dairy cattle and 0.0 ppm for poultry.

Farm animal feeding studies

The Meeting received information on the residue levels arising in animal tissues and milk when dairy cows were dosed with fluopyram for 29 days at the equivalent of 1.5, 14.4, 44 and 133 ppm in the diet. A separate dose group (146 ppm) was used to estimate residue depuration of fluopyram and its major metabolites.

In milk, average residues of fluopyram were not detectable in the 1.5 ppm dose group and increased to 0.12 mg/kg in the highest dose group (133 ppm). In skim milk, fluopyram residues were 0.02 mg/kg and 1.3 mg/kg in centrifuged 'cream' (71–78% milk fat). Residues of BZM, the predominant residue component in milk, increased from 0.04–1.7 mg/kg over the four dose groups while residues of the total-olefine metabolites were above the LOQ only in the two higher dose groups (0.03 mg/kg and 0.12 mg/kg respectively).

In muscle, residues of fluopyram did not exceed 0.03 mg/kg at any dose level and the predominant residue was the BZM metabolite, found at up to 1.4 mg/kg. Residues of the total-olefine metabolites were found above the LOQ (0.02 mg/kg) only in the two higher dose groups, up to 0.04 mg/kg.

In fat, residues of fluopyram were < LOQ in the lowest dose group and increased to 0.58 mg/kg in the 133 ppm dose group and a similar pattern was observed with BZM (up to 0.86 mg/kg). Total-olefine residues were found at up to 0.77 mg/kg.

In liver, fluopyram residues were found in all dose groups, up to 4.0 mg/kg with higher residues of the BZM metabolite also being found in the top three dose groups (1.2–6.9 mg/kg). Residues of the total-olefine isomers were found in the top three dose groups at levels of 0.04–0.5 mg/kg.

In kidney, fluopyram residues were above the LOQ in the two higher dose groups (44 ppm and 133 ppm) at levels of 0.03 mg/kg and 0.07 mg/kg respectively. The predominant residue was the BZM metabolite, present at 0.03–1.6 mg/kg over the four dose groups and residues of the total-olefine metabolites above the LOQ were found in the two higher dose groups at 0.04 mg/kg and 0.13 mg/kg respectively.

Residue depletion was studied in cows dosed orally for 29 days with the equivalent of 146 ppm fluopyram. Fluopyram residues in milk had depleted to < 0.01 mg/kg within 2 days after the last dose and were not detectable after 5 days with residues of BZM and the total-olefine metabolites being < LOQ within 21 days. In tissues, fluopyram residues decreased to less than the LOQ within 7 days (muscle) and within 14 days (fat, liver and kidney). Tissue residues of BZM decreased to less than the LOQ within 14 days in fat and within 21 days in milk with residues of 0.42 mg/kg (liver), 0.19 mg/kg (muscle) and 0.05 mg/kg (kidney) remaining at the end of the study period (21 days after the last dose). Residues of the total-olefine metabolites also remained in fat (0.17 mg/kg) and liver (0.04 mg/kg) at the end of the study period and were < 0.02 mg/kg (LOQ) in muscle within 7 days and within 21 days in kidney.

The Meeting also received information on the residues in tissues and eggs when laying hens were dosed with fluopyram for 28 days at levels equivalent to 0.05, 0.49, 1.6 and 4.8 ppm in the diet.

Fluopyram residues were not detected in eggs or any tissues from any dose groups and residues of the total-olefine metabolites were either not detectable or were at or below 0.02 mg/kg (LOQ) except in skin/fat from the highest (4.8ppm) dose group. The BZM metabolite was the predominant residue and was found in eggs and all tissues up to 1.4 mg/kg in liver, 0.72 mg/kg in eggs, 0.41 mg/kg in skin/fat and 0.29 mg/kg in muscle from the highest dose group.

In the residue depuration dose group (4.8 ppm), residues of the total-olefine metabolites in skin/fat were not detectable within 21 days after the last dose and residues of the BZM metabolite decreased to 0.01 mg/kg in muscle, 0.02 mg/kg in skin/fat, 0.03 mg/kg in eggs and 0.05 mg/kg in liver at the end of the study (21 days after the last dose).

Animal commodity maximum residue levels

The maximum and the mean dietary burden for beef and dairy cattle is 2.48 ppm and residue levels in tissues were obtained by interpolation between the 1.5 ppm and the 14.4 ppm feeding levels.

Dietary burden (mg/kg)[a]		Combined Fluopyram, BZM and Total olefine residues, mg/kg (fluopyram equivalents)[c]								
Feeding level [ppm][b]		Milk	Fat		Muscle		Liver		Kidney	
		mean	high	mean	high	mean	high	mean	high	mean
MRL beef	2.48		*< 0.049*		*< 0.053*		*0.551*		*< 0.057*	
	[1.5:14.4] F [1.5:14.4] B		< 0.01:0.07 0.01:0.33		0:< 0.01 0.02:0.44		0.26:0.98 0.1:1.9		0:< 0.01 0.03:0.38	
MRL dairy	2.48	*0.037*								
	[1.5:14.4] F [1.5:14.4] B	0:0.01 0.02:0.24								

Hi-res cattle			*< 0.076*		*< 0.054*		*< 0.574*		*< 0.059*	
	[1.5:14.4] F [1.5:14.4] B [1.5:14.4] O		< 0.01:0.07 0.01:0.33 < 0.02:0.12		0:< 0.01 0.02:0.44 0:< 0.02		0.26:0.98 0.1:1.9 < 0.02:0.06		0:< 0.01 0.03:0.38 0:< 0.02	
STMR beef	2.48			*< 0.061*		*< 0.043*		*0.472*		*< 0.051*
	[1.5:14.4] F [1.5:14.4] B [1.5:14.4] O			< 0.01:0.04 0.01:0.18 < 0.02:0.09		0:< 0.01 0.02:0.29 0:< 0.02		0.25:0.71 0.1:1.21 0:0.04		0:< 0.01 0.03:0.28 0:< 0.02
STMR dairy	2.48	*< 0.039*								
	[1.5:14.4] F [1.5:14.4] B [1.5:14.4] O	0:0.01 0.02:0.24 0:< 0.02								

[a] Values in parentheses are the estimated dietary burdens

[b] Values in square brackets are the actual feeding levels in the transfer study

[c] Residue values in italics are interpolated from the dietary burden, feeding levels and the residues found in the transfer studies (F = fluopyram, B = BZM, O = total olefins)

High is the highest individual animal tissue residue in the relevant feeding group.

Mean is mean animal tissue residue in the relevant feeding group.

Combined residues of fluopyram and BZM (expressed as fluopyram equivalents) expected in cattle milk and tissues for use in estimating maximum residue levels are: < 0.049 mg/kg (fat), < 0.053 mg/kg (muscle), 0.551 mg/kg (liver) and < 0.057 mg/kg (kidney) and the mean residue for milk is 0.037 mg/kg.

The Meeting estimated maximum residue levels of 0.1 mg/kg for fluopyram in meat (from mammals other than marine mammals), 0.7 mg/kg for edible offal (mammalian), 0.1 mg/kg for fat and 0.07 mg/kg for milks.

Estimated HRs for dietary intake estimation for fluopyram (and including residues of BZM and total olefins) are 0.076 mg/kg for mammalian fat, 0.054 mg/kg for mammalian muscle, 0.574 mg/kg for liver and 0.059 mg/kg for kidney.

Estimated STMRs for dietary intake estimation for fluopyram (and including residues of BZM and total olefins) are 0.061 mg/kg for mammalian fat, 0.043 mg/kg for mammalian muscle, 0.472 mg/kg for mammalian liver, 0.051 mg/kg for mammalian kidney and 0.039 mg/kg for milks.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue for plant commodities (for compliance with MRL and estimation of dietary intake): *fluopyram.*

Definition of the residue for animal commodities (for compliance with MRL): *Sum of fluopyram and 2-(trifluoromethyl) benzamide, expressed as fluopyram.*

Definition of the residue for animal commodities (for estimation of dietary intake): *Sum of fluopyram, 2-(trifluoromethyl)benzamide and the combined residues N-{(E)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl) benzamide and N-{(Z)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl) benzamide, all expressed as fluopyram.*

The residue is not fat soluble.

CCN	Commodity	Maximum residue level recommendation (mg/kg)	HR or HR-P (mg/kg)	STMR or STMR-P (mg/kg)
VB 0402	Cucumber	0.5	0.19	0.11
FB 0269	Grape	2	1	0.58
DF 0269	Dried Grapes	5	2.9	1.68
MO 0105	Edible offal (mammalian)	0.7	0.574 (liver) 0.059 (kidney)	0.472 (liver) 0.051 (kidney)
MM 0095	Meat (from mammals other than marine mammals)	0.1	0.054 (muscle) 0.076 (fat)	0.043 (muscle) 0.061 (fat)
ML 0106	Milks	0.07		0.039
AB 0269	Grape pomace (dry)			12.4
	Wine			0.1
JF 0269	Grape juice			0.012

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for fluopyram was calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available. The results are shown in Annex 3.

The International Estimated Daily Intakes of fluopyram for the 13 GEMS/Food regional diets, based on estimated STMRs were 1–6% of the maximum ADI of 0.01 mg/kg bw (see Annex 3 of the 2010 JMPR Report). The Meeting concluded that the long-term intake of residues of fluopyram from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-term Intake (IESTI) for fluopyram was calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available (see Annex 4 of the 2010 JMPR Report).

For fluopyram the IESTI varied from 0–4% of the ARfD (0.5 mg/kg bw) for the general population and 0–10% for children. The Meeting concluded that the short-term intake of residues of fluopyram from uses considered by the Meeting is unlikely to present a public health concern.

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