Pydiflumetofen (309)

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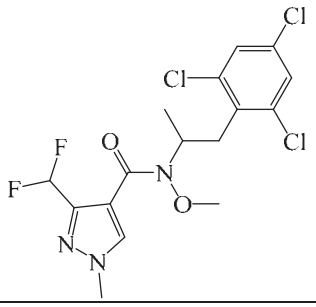
EXPLANATION

Pydiflumetofen was evaluated for the first time by the Meeting. At the Forty-ninth Session of the CCPR (2017), the compound was scheduled for evaluation as a new compound by the 2018 JMPR.

Pydiflumetofen is a broad-spectrum fungicide of the chemical group of N-methoxy-(phenyl-ethyl)-pyrazole-carboxamide and belongs to the SDHI (Succinate Dehydrogenase Inhibitors) fungicide group. It inhibits succinate dehydrogenase in complex II of fungal mitochondrial respiration.

The Meeting received information on physical and chemical properties, animal and plant metabolism, environment fate, rotational crop residues, analytical methods, storage stability, use pattern, supervised trials, and fate of residues in processing.

IDENTITY

Common name	Pydiflumetofen
Chemical name	
IUPAC:	*N*-methoxy-*N*-[(*RS*)-1-methyl-2-(2,4,6-trichlorophenyl)-ethyl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxamide
CAS:	1*H*-Pyrazole-4-carboxamide,3-(difluoromethyl)-*N*-methoxy-1-methyl-*N*-[1-methyl-2-(2,4,6-trichlorophenyl)ethyl]-
CAS Registry No:	1228284-64-7
CIPAC No:	Not available
Synonyms:	SYN545974, Adepidyn
Structural formula:	
Molecular formula:	$C_{16}H_{16}Cl_3F_2N_3O_2$
Molecular weight:	426.7

Physical and chemical properties

Pure active ingredient

Property	Results	Reference
Appearance (color, physical state, odor)	White / non free flowing powder / no odor (99.5% purity)	O'Connor, 2012 41203897
Vapour pressure	1.84×10^{-8} Pa at 20 °C 5.30×10^{-8} Pa at 25 °C (99.5% purity)	Vijayakumar, 2017 SMG11739
Boiling point	Pydiflumetofen decomposed on heating from approximately 283°C, both in air and under a nitrogen atmosphere. (99.5% purity)	O'Connor, 2012 41203899
Octanol/water partition coefficient	log Pow=3.8 at 25 °C (99.5% purity)	Halarnakar, 2012 SMG11738
Solubility in water	1.5 mg/L at 25 °C (99.5% purity)	Halarnakar, 2012 SMG11737

Property	Results	Reference
Hydrolysis	Hydrolytically stable at pH 4, 7 and 9 for up to 5 days at 50 °C corresponds to a DT_{50} of more than a year at 25 °C. ([Pyrazole-5-^{14}C]-pydiflumetofen 97.8% radiochemical purity)	Lewis, 2015 3200053
Photolysis	Stable to photolysis at pH 7 with 72.5% and 78.8% of the applied radioactivity remaining as pydiflumetofen after 30 days. ([Phenyl-U-^{14}C] and [Pyrazole-5-^{14}C]- pydiflumetofen)	Lewis, 2015 3200127
Dissociation constant	No dissociation constant at pH 2 – 12 (99.5% purity)	O'Connor, 2013 41206681

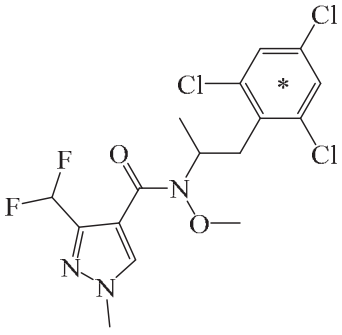
Technical material

Property	Results		Reference
Appearance (color, physical state, odor)	Off-white / non free flowing powder / no odour (98.5% purity)		O'Connor, 2012 41203900
Solubility in organic solvents (98.5% purity)	Acetone	220 g/L at 25 °C	Halarnakar, 2012 SMG11891
	Dichloroethane	>500 g/L at 25 °C	
	Ethyl acetate	130 g/L at 25 °C	
	Hexane	0.270 g/L at 25 °C	
	Methanol	26 g/L at 25 °C	
	Octanol	7.2 g/L at 25 °C	
	Toluene	67 g/L at 25 °C	
Relative density	1.55 g/cm^3 at 20.0 ± 1.0 °C (98.5% purity)		O'Connor, 2012 41203901
Thermal stability / Stability in air (98.5% purity)	Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) scans carried out on the test substance in nitrogen and air show no exothermicity or weight loss attributable to reaction/decomposition at room temperature. The test substance is stable at room temperature in nitrogen or air.		Jackson, 2012 10514986

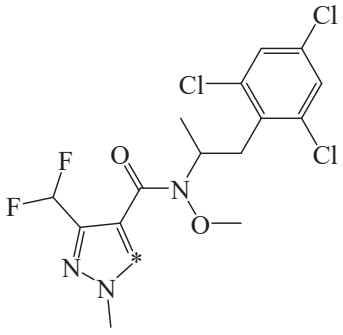
Formulations: Suspension concentrate (SC)

METABOLISM AND ENVIRONMENTAL FATE

The metabolism, distribution of pydiflumetofen has been investigated in animals and plants. The fate and behaviour of pydiflumetofen in animals, plants and the environment was investigated using the [^{14}C] labelled test materials shown in Figure 1 below.



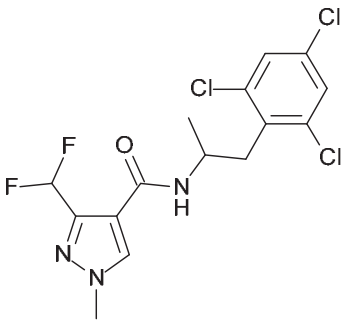
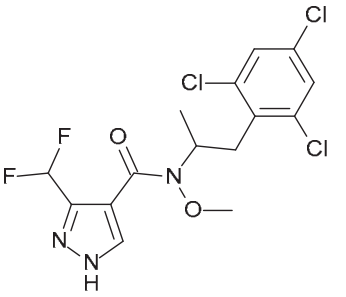
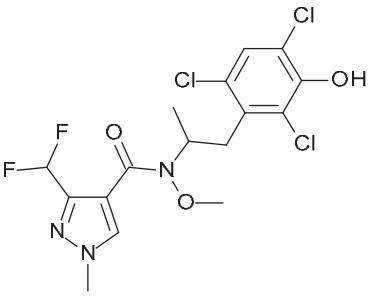
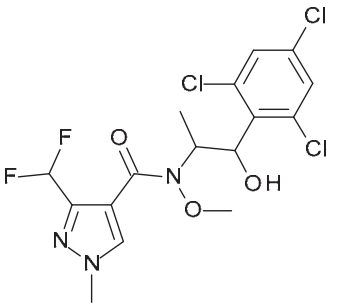
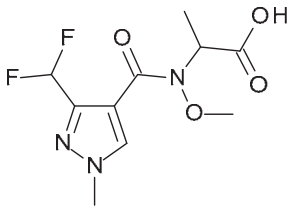
[Phenyl-U-^{14}C]-pydiflumetofen

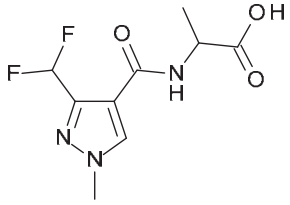
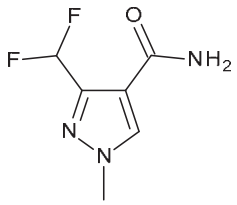
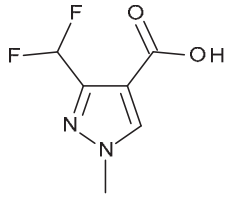
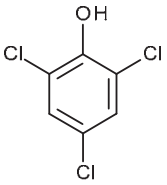
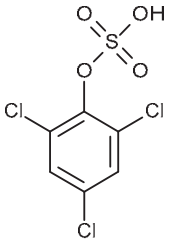
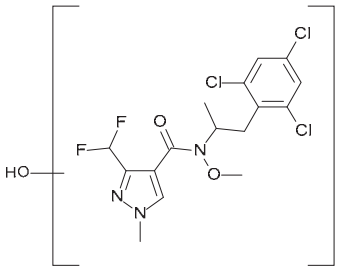


[Pyrazole-5-^{14}C]-pydiflumetofen

Figure 1 [^{14}C]-Labelled test materials used in animals, plants metabolism studies, and the environmental fate studies.

The chemical structures of the major degradation compounds from the metabolism of pydiflumetofen are provided below.

Compound name		Structure	Found in metabolism studies
SYN545547	3-(difluoromethyl)-1- methyl-N-[1-methyl-2- (2,4,6-trichlorophenyl) ethyl]pyrazole-4- carboxamide		Plants, Rat, Livestock
SYN547891	3-(difluoromethyl)-N- methoxy-N-[1-methyl-2- (2,4,6-trichlorophenyl) ethyl]-1H-pyrazole-4- carboxamide		Plants, Rat, Livestock
SYN547897	3-(difluoromethyl)-N- methoxy-1-methyl-N-[1- methyl-2-(2,4,6-trichloro- 3-hydroxy-phenyl)ethyl] pyrazole-4-carboxamide	 MW=442.7	Rat, Livestock
SYN547948	3-(difluoromethyl)-N-[2-hydroxy-1-methyl-2- (2,4,6-trichlorophenyl) ethyl]-N-methoxy-1- methyl-pyrazole-4-carboxamide		Rat, Livestock
SYN548263	2-[[3-(difluoromethyl)-1-methyl-pyrazole-4- carbonyl]-methoxy-amino]propanoic acid	 MW=277.2	Rat, Livestock

Compound name		Structure	Found in metabolism studies
SYN548264	2-[[3-(difluoromethyl)-1-methyl-pyrazole-4- carbonyl] amino]propanoic acid		Rat, Livestock
SYN508272	3-(difluoromethyl)-1- methyl-pyrazole-4- carboxamide		Rat, Livestock
NOA449410	3-(difluoromethyl)-1- methyl-pyrazole-4- carboxylic acid		Livestock
2,4,6-TCP	2,4,6-trichlorophenol	 MW=197.45	Rat, Livestock
	Sulphate conjugate of 2,4,6-trichlorophenol		Rat, Livestock
Hydroxylated pydiflumetofen	Hydroxylated N-methoxy-N-[1-methyl- 2-(2,4,6-trichlorophenyl)- ethyl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxamide		Rat, Livestock

Plant metabolism

Plant metabolism studies were performed on tomato, wheat and oilseed rape with pydiflumetofen ^{14}C-labelled at phenyl or pyrazole to track metabolites. Metabolites were identified using multiple chromatographic systems and authentic standards.

Tomato

The metabolism of pydiflumetofen in tomatoes was investigated with [^{14}C]-pydiflumetofen (Inns, 2014: 34592). Tomato plants (variety F1 Shirley), grown in a glasshouse, were treated with two foliar spray applications made at growth stage BBCH 83 and BBCH 86. Tomato plants were treated with [phenyl-U-^{14}C]-pydiflumetofen or [pyrazole-5-^{14}C]-pydiflumetofen, each at a nominal application rate of 0.200 kg ai/ha per application (total application rate 0.400 kg ai/ha). The actual application rates achieved were 0.198 kg ai/ha and 0.196 kg ai/ha for the [phenyl-U-^{14}C]-pydiflumetofen labelled treatments; and 0.227 kg ai/ha and 0.174 kg ai/ha for the [pyrazole-5-^{14}C]-pydiflumetofen applications. This gave total application rates of 0.394 kg ai/ha for [phenyl-U-^{14}C] and 0.401 kg ai/ha for [pyrazole-5-^{14}C].

A separate group of tomato plants grown in a glasshouse were treated with [phenyl-U-^{14}C]-pydiflumetofen or [pyrazole-5-^{14}C]-pydiflumetofen as a single application direct to soil containing the tomato plants. The application was made via pipette to soil surrounding 5 plants (per label) at a nominal application rate of 20 mg ai/plant, with an achieved application rate of 20.0 and 20.1 mg ai/plant for [phenyl-U-^{14}C] and [pyrazole-5-^{14}C] respectively. The radiochemicals were formulated with an SC formulation for all application methods.

Fruit and foliage were harvested from the foliar treated plants at 1 day after last application (DALA) and at 14 DALA. Mature fruit was surface rinsed by immersion in acetonitrile. Fruit and foliage were harvested from the soil treated experiment at 28 days after application and at fruit maturity (103 days after application). The TRR was measured in mature fruit samples.

Sub-samples of the crop commodities were taken for initial overall residue determination employing sample oxidation with LSC analysis. Appropriate samples were combusted to achieve a LOD of 0.001 mg/kg. Commodities with a TRR ≥0.01 mg/kg were extracted with aqueous acetonitrile solvent combinations. Extracts were analysed by HPLC to determine the nature of the residues.

Initial analysis of the foliar treated tomato fruit fractions took place 1 month after harvest. Selected original extracts were then re-analysed 4–6 months later. Comparison of the initial and final radiocomponent profiles obtained showed no significant change in the profiles had occurred during the interim period of storage.

In the foliar experiment, the overall residues were ≤0.642 md eq/kg. Residue levels were highest in fruit harvested 14 DALA, with no significant difference between the radiolabels. Extracted radioactivity was ≥97.5% TRR in all commodities, with significant radioactive residues (≥88.9% TRR) in the surface wash fractions.

Table 1 Extractability and distribution of the radioactive residues in tomato fruit

Radiolabel	Treatment	Commodity	Extracted radioactivity [b]		Unextracted radioactivity		TRR[a]
			mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Phenyl-U-^{14}C	Foliar	1 DALA Fruit	0.520	100.0	0.001	0.1	0.521
		14 DALA Fruit	0.640	99.7	0.002	0.3	0.642
	Soil	Mature fruit	NC				0.007[c]
Pyrazole-5-^{14}C	Foliar	1 DALA Fruit	0.473	98.4	0.008	1.6	0.481
		14 DALA Fruit	0.632	100.0	0.001	0.1	0.633
	Soil	Mature fruit	0.013	97.5	<0.001	2.6	0.013

NC: Not conducted, TRR <0.010 mg/kg therefore not extracted.

[a] mg/kg calculated directly from surface wash (where applicable), radioactivity extracted from washed fruit, radioactivity in the PES and specific activity

[b] Extracted radioactivity inclusive of surface wash and extracted radioactivity quantified from washed fruit.

[c] mg/kg calculated from initial combustion analysis

High levels of identification were achieved, with parent pydiflumetofen representing the major portion of the residue in all foliar treated tomato fruit samples. The highest residues were detected in 14 DALA fruit (0.592–0.611 md eq/kg, 92.2–96.6% TRR). Metabolites identified were the de-alkylated molecules, SYN545547 and SYN547891. Residues of SYN545547 and SYN547891 accounted for a maximum of 3.6% TRR and 1.6% TRR respectively.

Unidentified components were present in all foliar treated samples except 1 DALA pyrazole treated fruit and in total accounted for 1.0–2.5% TRR (0.006–0.015 md eq/kg); no individual component accounted for >0.8% TRR (0.004 md eq/kg).

In the soil treated experiment, fruit total radioactive residues were low (≤0.013 md eq/kg). Residues exceeded 0.01 md eq/kg in the pyrazole label only, so extraction of the phenyl label fruit was not conducted. The principal component detected in the pyrazole treated fruit was pydiflumetofen accounting for 4.1% TRR (0.001 md eq/kg).

Unidentified components in pyrazole labelled soil treated fruit accounted for 88.9% TRR (0.008 md eq/kg) with no individual component >11.9% TRR (0.002 md eq/kg).

Table 2 Identification and characterisation of the radioactive residues in tomato fruit

Components	Foliar treatment 1 DALA				Foliar treatment 14 DALA			
	Phenyl-U-^{14}C		Pyrazole-5-^{14}C		Phenyl-U-^{14}C		Pyrazole-5-^{14}C	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	0.520	100.0	0.473	98.3	0.641	99.9	0.633	100.2
Pydiflumetofen	0.477	91.7	0.461	95.9	0.592	92.2	0.611	96.6
SYN545547	0.019	3.6	0.009	1.8	0.021	3.3	0.009	1.4
SYN547891	0.007	1.4	0.003	0.6	0.011	1.6	0.006	1.0
Unknowns[1)]	0.010	2.1	ND	ND	0.015	2.5	0.006	1.0
Others[2)]	0.007	1.2	-	-	0.002	0.3	0.001	0.2
Unextracted[3)]	0.001	0.1	0.008	1.6	0.002	0.3	0.001	0.1
Total	0.521	100.1	0.481	99.9	0.643	100.2	0.634	100.3

Components	Soil treatment Mature fruit	
	Pyrazole-5-^{14}C	
	mg/kg eq	%TRR
Extract	0.010	98.3
Pydiflumetofen	0.001	4.1
SYN545547	<0.001	0.4
Unknowns[1)]	0.008	88.9
Others[2)]	0.001	4.9
Unextracted[3)]	<0.001	2.6
Total	0.010	100.9

ND: Not detected

[a] 1 DALA: Phenyl label: comprising at least 4 discrete components, no single one of which ≥0.8% TRR (≥0.004 mg/kg eq)

14 DALA: Phenyl label: comprising at least 9 discrete components, no single one of which ≥0.7% TRR (≥0.004 mg/kg eq);

Pyrazole label: comprising at least 6 discrete components, no single one of which ≥0.3% TRR (≥0.002 mg/kg eq)

Soil treatment: comprising at least 25 discrete components, no single one of which ≥11.9% TRR (≥0.002 mg/kg eq)

[b] Fractions produced during processing that were too low for analysis.

1 DALA: No single fraction comprised ≥0.9% TRR (≥0.005 mg/kg eq) in either radiolabelled experiment

14 DALA: No single fraction comprised ≥0.3% TRR (≥0.002 mg/kg eq) in either radiolabelled experiment

Soil treatment: No single fraction comprised ≥4.9% TRR (≥0.001 mg/kg eq)

[c] Radioactivity remaining in the PES after extraction with acetonitrile and aqueous acetonitrile. The nature of this residue was not characterised further.

Wheat

The metabolism of pydiflumetofen in wheat was investigated with [^{14}C]-pydiflumetofen (Chapleo, 2014: 33586). Spring wheat (cultivar Paragon) grown outdoors was treated twice post emergence with [phenyl-U-^{14}C]-pydiflumetofen or [pyrazole -5-^{14}C]-pydiflumetofen each at a nominal application rate of 0.125 kg ai/ha. The actual total application rate were 0.252 kg ai/ha for both radiolabelled experiments. The radiochemicals were formulated with a blank SC formulation and applied to the wheat plants as spray applications at growth stage BBCH 32–34 for 1st application and BBCH 58 for last application. Forage was harvested 10 days after 1st application (BBCH 39) and hay at 29 days after last application (BBCH 77). Straw and grain were harvested 50 days after last application, at maturity (BBCH 89). The total radioactive residue (TRR) was measured in each commodity sampled.

Sub-samples of the crop commodities were taken for initial overall residue determination employing sample oxidation with LSC analysis. Appropriate amounts of sample were combusted to achieve a limit of determination (LOD) of 0.001 mg/kg. Commodities with a TRR ≥0.01 md eq/kg were extracted with aqueous acetonitrile solvent (acetonitrile: water 80:20) combinations and the extracted residues were analysed by HPLC and TLC to determine the nature of the residues.

On the day of harvest, all samples were placed in a freezer set to maintain -20 °C and stored frozen prior to analysis. Initial analysis of the wheat fractions took place 1 month after harvest although further analysis was conducted on the extracts

after this period. The original extracts were then re-analysed 10 months later. Comparison of the initial and final radiocomponent profiles obtained showed no significant change in the profiles had occurred during the interim period of storage.

Residues increased from forage to straw to a maximum of 1.53 md eq/kg; residues in grain were the lowest of all commodities with residues ≤0.057 md eq/kg. Residues were slightly higher in the pyrazole label compared to the phenyl label for all commodities. Extractability was ≥84.9% TRR in all commodities.

Table 3 Extractability and distribution of the radioactive residues in wheat

Radiolabel	Commodity	Extracted radioactivity		Unextracted radioactivity		TRR [a]
		mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Phenyl-U-^{14}C	Forage	0.327	96.5	0.012	3.5	0.338
	Hay	0.920	94.2	0.057	5.8	0.977
	Straw [b]	1.232 (1.208 + 0.024)	95.8 (93.9 + 1.9)	0.059	4.6	1.286
	Grain	0.033	90.4	0.004	9.6	0.037
Pyrazole-5-^{14}C	Forage	0.445	95.6	0.020	4.4	0.465
	Hay	1.311	94.2	0.079	5.7	1.391
	Straw	1.443 (1.420 + 0.023)	94.5 (93.0 + 1.5)	0.093	6.1	1.527
	Grain	0.048	84.9	0.009	15.2	0.057

[a] The TRR values of all commodities were also determined by the summation of the radioactivity present in the extracts and PES

[b] The initial extraction with acetonitrile/water (80:20, v/v) + further extraction on PES with acetonitrile/water (1:1, v/v)

High levels of identification were achieved with parent representing the major portion of the residue in all commodities. The highest residues of parent were detected in straw (1.075–1.167 md eq/kg, ≥76.4% TRR), and the lowest in grain (0.030–0.046 md eq/kg, ≥81.5% TRR). Metabolites identified (SYN545547 and SYN547891) showed only minor de-alkylation of the parent compound and occurred at low levels relative to the parent residue. Residues of SYN545547 accounted for ≤3.9% TRR with the largest residue detected in straw (0.059 md eq/kg, 3.9% TRR). Residues of SYN547891 accounted for ≤8.3% TRR with the highest residue in straw (0.065 md eq/kg, 4.3% TRR).

Up to 12 unidentified components were detected in forage, hay and grain, none individually exceeding 3.3% TRR (0.036 md eq/kg). Further extraction of the straw post extraction solids (PES) with acetonitrile: water (1:1, v/v) released 0.024 and 0.023 md eq/kg for the phenyl and pyrazole labels, respectively. Unextracted residues accounted for ≤6.1% TRR (≤0.093 md eq/kg) in feed items, and ≤15.2% TRR (≤0.009 md eq/kg) in grain. Therefore no further analyses were conducted on these fractions.

Table 4 Identification and characterisation of the radioactive residues in wheat

Components	Forage				Hay			
	Phenyl-U-^{14}C		Pyrazole-5-^{14}C		Phenyl-U-^{14}C		Pyrazole-5-^{14}C	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	0.316	93.6	0.427	91.9	0.914	93.7	1.253	90.3
Pydiflumetofen	0.307	91.0	0.392	84.3	0.821	84.1	0.981	70.5
SYN545547	0.005	1.4	0.012	2.7	0.023	2.4	0.034	2.4
SYN547891	0.004	1.2	0.011	2.4	0.029	3.0	0.049	3.6
Unknown [a]	NA	NA	0.012	2.5	0.041	4.2	0.189	13.8
Unextracted [b]	0.012	3.5	0.020	4.4	0.057	5.8	0.079	5.7
Total	0.328	97.1	0.447	96.3	0.971	99.5	1.332	96.0
Components	Straw				Grain			
	Phenyl-U-^{14}C		Pyrazole-5-^{14}C		Phenyl-U-^{14}C		Pyrazole-5-^{14}C	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	1.167	90.7	1.314	86.1	0.034	92.7	0.053	95.3
Pydiflumetofen	1.075	83.6	1.167	76.4	0.030	81.5	0.046	81.6
SYN545547	0.036	2.8	0.059	3.9	0.001	2.9	0.001	2.6
SYN547891	0.032	2.4	0.065	4.3	0.003	8.3	0.004	7.8
Unknowns[1)]	0.024	1.9	0.023	1.5	NA	NA	0.002	3.3
Unextracted[2)]	0.059	4.6	0.093	6.1	0.004	9.6	0.009	15.2
Total	1.226	95.3	1.407	92.2	0.038	102.3	0.062	110.5

NA: Not applicable

[a] Forage: Pyrazole label comprising at least 3 discrete components, none >0.9% TRR, (0.004 mg/kg eq)
Hay: Phenyl label consisting at least 3 discrete components, no single one of which >2.3% TRR, (0.022 mg/kg eq)
Pyrazole label comprising at least 12 discrete components, no single one of which >2.6% TRR, (0.036 mg/kg eq)

Grain: Pyrazole label consisting of 1 discrete component, 3.3% TRR, (0.002 mg/kg eq)
Straw: Further extraction of the PES with acetonitrile: water (1:1, v/v)

[b] Radioactivity remaining in the PES after extraction with aqueous acetonitrile

Oilseed rape

The metabolism of pydiflumetofen in oilseed rape was investigated with [^{14}C]-pydiflumetofen. (Chapleo, 2015: 33587). Oilseed rape (cultivar Ability) grown outdoors was treated once post-emergence, with [phenyl-^{14}C]-pydiflumetofen or [pyrazole-^{14}C]-pydiflumetofen, at a nominal application rate of 0.150 kg ai/ha. The actual application rates achieved were 0.134 kg ai/ha for the [phenyl-^{14}C]-pydiflumetofen labelled treatment; and 0.147 kg ai/ha for the [pyrazole-^{14}C]- pydiflumetofen labelled treatment. The radiochemicals were formulated with a blank SC formulation and applied to the plants as a spray application at growth stage BBCH 65.

Foliage was harvested 21 days after application and retained deep frozen as a contingency without analysis. Seed and trash (including pods) were harvested 62 days after application at maturity (BBCH 89). The TRR was measured in each commodity sampled.

Sub-samples of the crop commodities were taken for initial overall residue determination employing sample oxidation with LSC analysis. Appropriate amounts of sample were combusted to achieve a LOD of 0.001 mg/kg. Commodities with a TRR ≥0.01 mg/kg were extracted with aqueous acetonitrile solvent combinations and the extracted residues were analysed by HPLC and TLC to determine the nature of the residues.

Initial analysis of the oilseed rape trash took place within 2 months after harvest. Re-analysis was conducted 28 months later. Comparison of the initial and final radiocomponent profiles showed no significant change during the storage period.

Residues in trash were 0.061–0.062 md eq/kg and residues in seed were 0.019–0.020 md eq/kg. There was no notable difference between the [phenyl-^{14}C] and [pyrazole-^{14}C]- pydiflumetofen labelled experiments. Extractability was ≥71.8% TRR in all commodities.

Table 5 Extractability and distribution of the radioactive residues in oilseed rape

Radiolabel	Commodity	Extracted radioactivity		Unextracted radioactivity		TRR*
		mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
[Phenyl-U-^{14}C]	Seed	0.015	74.5	0.005	25.5	0.020
	Trash	0.051	81.3	0.012	18.7	0.062
[Pyrazole-5-^{14}C]	Seed	0.014	71.8	0.005	28.2	0.019
	Trash	0.046	75.6	0.015	24.4	0.061

* The TRR values of all commodities were also determined by the summation of the radioactivity present in the extracts and PES after initial fractionation.

High levels of identification were achieved with parent representing the major portion of the extractable residue in all commodities. The highest residues of parent were detected in trash (0.018–0.032 md eq/kg, ≥30.0%TRR); lower parent residues were detected in seed (0.007–0.012 md eq/kg, ≥39.2% TRR).

Metabolites identified were the de-alkylated molecules, SYN545547 and SYN547891. Low residues of SYN545547 (≤3.7% TRR, ≤0.002 md eq/kg) and SYN547891 (≤5.1% TRR, ≤0.003 md eq/kg) were detected in trash. Low residues of SYN545547 (≤6.1% TRR, ≤0.001 md eq/kg) and SYN547891 (≤2.7% TRR, ≤0.001 md eq/kg) were detected in seed.

Up to 10 unidentified components were detected in seed and trash, none individually exceeding 8.4% (0.005 md eq/kg). Further extractions of the trash PES with 1M HCl and 1M NaOH (each 2 hours, 60 °C followed by an aqueous rinse) were conducted; no individual fraction accounted for >0.004 md eq/kg (6.8% TRR). Following these procedures, the remaining unextracted residues accounted for ≤7.1% TRR (0.004 md eq/kg) therefore no further work were conducted on these fractions. Following cold solvent extractions, unextracted residues in seed accounted for ≤28.2% TRR (0.005 md eq/kg) therefore no further analyses were conducted on these fractions.

Table 6 Identification and characterisation of the radioactive residues in oilseed rape

Components	Oilseed rape seed				Oilseed rape trash			
	Phenyl-U-^{14}C		Pyrazole-5-^{14}C		Phenyl-U-^{14}C		Pyrazole-5-^{14}C	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	0.014	71.3	0.010	53.9	0.056	89.6	0.048	84.6
Pydiflumetofen	0.012	62.6	0.007	39.2	0.032	50.9	0.018	30.0

Components	Oilseed rape seed				Oilseed rape trash			
	Phenyl-U-^{14}C		Pyrazole-5-^{14}C		Phenyl-U-^{14}C		Pyrazole-5-^{14}C	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
SYN545547	ND	ND	0.001	6.1	0.002	3.7	0.002	2.8
SYN547891	0.001	2.7	ND	ND	0.003	5.1	0.002	3.3
Unknowns [a]	0.001	6.0	0.002	8.6	0.004	6.2	0.022	34.9
Others [b]	-	-	-	-	0.015	23.7	0.008	13.6
Unextracted [c]	0.005	25.5	0.005	28.2	0.004	6.5	0.004	7.1
Total	0.019	96.8	0.015	82.1	0.060	96.1	0.056	91.7

ND: Not detected

[a] Seeds: Phenyl label comprising 1 discrete component, 6.0% TRR, (0.001 mg/kg eq);
Pyrazole label comprising at least 2 discrete components, no single one of which >4.3% TRR, (0.001 mg/kg eq)

Trash: Phenyl label comprising at least 3 discrete components, no single one of which >2.9% TRR, (0.002 mg/kg eq);
Pyrazole label comprising at least 10 discrete components, no single one of which >8.4% TRR, (0.005 mg/kg eq)

[b] Extracted residues produced during processing that were too low for analysis;

Phenyl label: 6 individual fractions, no single one of which >9.2% TRR, (0.006 mg/kg eq);
Pyrazole label: 5 individual fractions, no single one of which >6.8% TRR, (0.004 mg/kg eq)

[c] Radioactivity remaining in the PES in seed after extraction with aqueous acetonitrile, while the PES in trash after extraction with aqueous acetonitrile and further extraction with 1M HCl and 1M NaOH (each 2h, 60 °C)

Summary of plant metabolism

Metabolism studies conducted with crops representative of three different crop groups (cereal/grass: spring wheat, fruit: tomato, pulses and oilseed: oil seed rape) based on the commercially recommended use pattern, i.e. post emergence foliar treatment or a single soil application, have provided a detailed understanding of the metabolism of pydiflumetofen in food and feed commodities. The metabolic pathways in the three studies are similar and consequently the available crop metabolism studies fully support the current proposed uses of pydiflumetofen on crops. Pydiflumetofen was the principle residue present in all commodities. Metabolism was limited and the metabolites identified maintained the basic structure of parent pydiflumetofen. The principal metabolic transformations of pydiflumetofen in all commodities occurred via reduction of the parent molecule to produce SYN545547 and via demethylation of the pyrazole ring to produce SYN547891. The following metabolic pathways were proposed in the plant metabolism studies available.

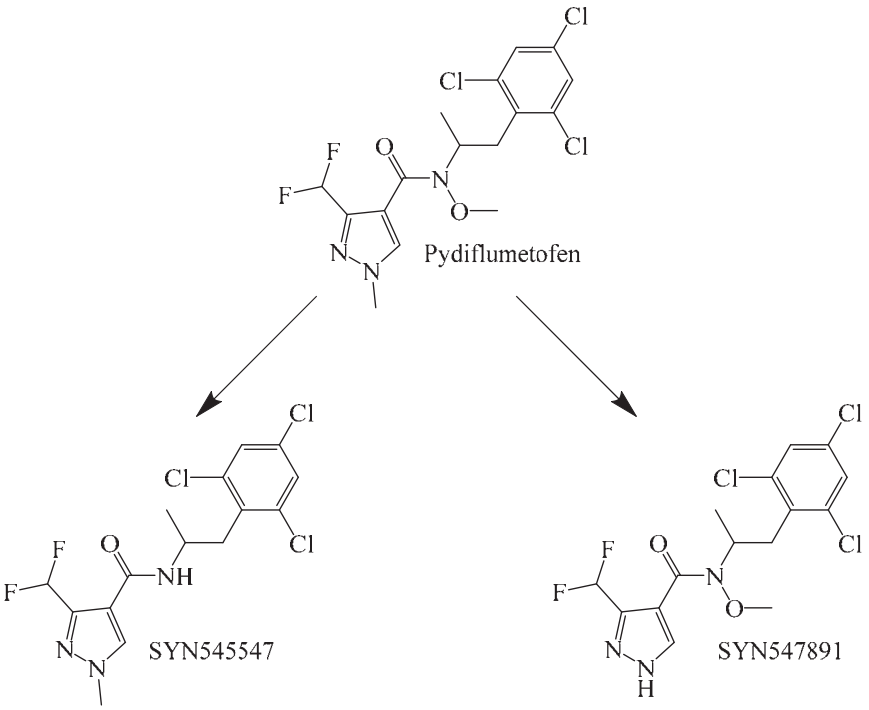


Figure 2 Metabolic Pathway of Pydiflumetofen in Plants (wheat, tomato and oilseed rape)

Farm animal metabolism

The Meeting received studies on the metabolism of pydiflumetofen in lactating goat and laying hens.

Lactating goat

The study was conducted to investigate the nature and quantity of residues in milk, liver, kidney, muscle and fat from lactating goat following oral dosing with [^{14}C]-pydiflumetofen (Wicksted, 2015: 33963). Two lactating goats, one per radiolabel, were orally dosed with [phenyl-U-^{14}C]-pydiflumetofen or [pyrazole-5-^{14}C]-pydiflumetofen for 7 days at a nominal rate of 100 ppm dietary dry matter intake. The actual dose rates achieved based on measured food consumption were approximately 144 and 205 ppm dietary dry matter intake respectively (both corresponding to 4.6 mg ai/kg bw). Milk, urine and faeces were collected daily. The goats were sacrificed approximately 11 hours after the administration of the final dose and tissues taken (liver, kidneys, peritoneal fat, perirenal fat, subcutaneous fat, flank muscle, loin muscle, Gastrointestinal (GI) tract and contents, blood, bile and carcass) *post mortem* for quantification and analysis.

All samples were radioassayed to determine the distribution of radioactivity and radioactive balance. The radioactive residue concentration present in milk samples (including the plateau residue concentration) and tissue samples (excluding carcass, bile and gastrointestinal tract) were also determined. Further analysis was undertaken to determine the nature and quantity of constituent residues, where the radioactive residue concentration was ≥0.01 md eq/kg. Samples were extracted with solvents (milk and fat: hexane and aqueous acetonitrile, liver, kidney and muscle: aqueous acetonitrile) and the extracted residues were characterised. Fractions containing significant radioactive residues were either subject to TLC/bioimage analysis or HPLC/radiodetection analysis to enable quantification and identification of residues. Where appropriate fractions were also subject to organic solvent/water partition and enzyme (β-glucuronidase) hydrolysis procedures prior to chromatographic analysis. Residues were identified by chromatographic comparison with authentic synthetic reference standards of parent pydiflumetofen and its metabolites.

Initial radiocomponent profiles of principal analytical fractions of representative samples (milk and fat) were produced within 6 months of sampling from the goat. Reanalysis of these residues after completion of nature of residue analysis demonstrated the radiocomponent profiles not to have changed significantly during the interim period of frozen storage.

The radioactive balance for both goats was greater than 94% with the majority of the radioactivity excreted in the urine and faeces as summarised in Table 7.

Table 7 Distribution of radioactivity and material balance from lactating goats dosed with [^{14}C]-pydiflumetofen

Sample	% of administered dose recovered	
	Phenyl-U-^{14}C	Pyrazole-5-^{14}C
Milk	<0.1	<0.1
Liver	0.4	0.4
Kidney	<0.1	<0.1
Flank muscle	<0.1	<0.1
Loin muscle	<0.1	<0.1
Peritoneal fat	<0.1	<0.1
Perirenal fat	<0.1	<0.1
Subcutaneous fat	<0.1	<0.1
Urine	31.5	29.9
Faeces	52.7	46.4
Bile	0.1	0.1
Cage wash	1.4	1.3
GI content	9.9	16.6
Material Balance	96.0	94.7

Residues in milk achieved a plateau concentration of approximately 0.091 md eq/kg (phenyl) and 0.126 md eq/kg (pyrazole), after 2 days (averaging residues for days: 3, 4, 5 and 6) and 6 days of dosing, respectively.

Table 8 Total radioactive residues in milk from a lactating goat dosed with [^{14}C]-pydiflumetofen

Day	Time point	Phenyl-U-^{14}C			Pyrazole-5-^{14}C		
		mg/kg eq		% of administered dose	mg/kg eq		% of administered dose
1	pm (7 h)	0.110	0.093	0.010	0.048	0.076	0.006
	am (24 h)	0.085			0.090		

Day	Time point	Phenyl-U-^{14}C			Pyrazole-5-^{14}C		
		mg/kg eq		% of administered dose	mg/kg eq		% of administered dose
2	pm (31 h)	0.151	0.110	0.011	0.089	0.101	0.009
	am (48 h)	0.092			0.108		
3	pm (55 h)	0.150	0.106	0.011	0.114	0.105	0.008
	am (72 h)	0.085			0.099		
4	pm (79 h)	0.132	0.091	0.010	0.126	0.123	0.010
	am (96 h)	0.073			0.123		
5	pm (103 h)	0.107	0.079	0.008	0.136	0.138	0.008
	am (120 h)	0.063			0.138		
6	pm (127 h)	0.111	0.087	0.008	0.140	0.126	0.010
	am (144 h)	0.075			0.118		
7	pm (151 h)	0.114	0.107	0.006	0.160	0.151	0.006
	am (156 h)	0.088			0.138		
Total				0.064			0.057

Figure 3 Residues in milk following dosing with [^{14}C]-pydiflumetofen

The 20% subsample taken following the daily pm milk collection was stored refrigerated overnight at *ca* 4 °C and then mixed with the following mornings am milk 20% subsample. This composite 24 hour sample was used to generate cream and skim milk for that time period employing physical means of separation (*i.e.* centrifugation). The cream/skimmed milk total radioactive residue ratio ranged from 4.45–5.68 and from 2.27–3.43 for phenyl and pyrazole labelled experiments, respectively.

Table 9 Total radioactive residues in cream and skimmed milk from a lactating goat dosed with [^{14}C]-pydiflumetofen

Radiolabel	Time point (h)	Concentration (mg/kg eq)		Cream/Skimmed milk ratio
		Skimmed milk	Cream	
[Phenyl-U-^{14}C]	96	0.076	0.338	4.45
	120	0.066	0.309	4.68
	144	0.076	0.345	4.54
	156	0.087	0.494	5.68
[Pyrazole-5-^{14}C]	96	0.116	0.298	2.57
	120	0.131	0.297	2.27
	144	0.118	0.292	2.47
	156	0.138	0.474	3.43

The nature of residues in milk was investigated using the 79 hour sample from phenyl group and 127 hour sample from pyrazole group. Residues in liver, kidney, muscle and fat, which exceeded 0.01 md eq/kg were also subject to extraction and analysis. Following solvent extraction of milk, liver, kidney, muscle and fat, residue extractabilities were generally high (≥83.4% TRR) for milk, kidney, muscle and fat. Residue extractability was lower in liver (50.4 and 47.4% TRR for phenyl and pyrazole, respectively).

Table 10 Extractability and distribution of the radioactive residues in milk and tissues of lactating goats

Radiolabel	Sample	Extracted radioactivity		Unextracted radioactivity		TRR[a]
		mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
[Phenyl-U-^{14}C]	Milk (79 hours)	0.112	92.3	0.009	7.7	0.122
	Liver	3.52	50.4	3.47	49.7	6.98
	Kidney	1.44	83.4	0.287	16.6	1.73
	Muscle[b]	0.087	86.0	0.014	14.0	0.102
	Fat[c]	0.219	98.8	0.002	1.1	0.221
[Pyrazole-5-^{14}C]	Milk (127 hours)	0.124	93.9	0.008	6.1	0.132
	Liver	4.18	47.4	4.64	52.6	8.83
	Kidney	2.12	90.7	0.215	9.2	2.34
	Muscle[2)]	0.130	94.3	0.008	5.7	0.138
	Fat[3)]	0.272	97.6	0.007	2.4	0.279

[a] mg/kg calculated directly from radioactivity extracted, radioactivity in the PES and specific activity

[b] Composite muscle samples (includes flank and loin muscle)

[c] Composite fat samples (includes perirenal, subcutaneous and peritoneal fat)

Parent pydiflumetofen was identified in milk and all tissues. Parent %TRRs were greatest in fat (≤73.8% TRR), muscle (≤24.4% TRR), milk (≤15.7% TRR) and liver (≤8.2% TRR) with much smaller % TRRs being found in kidney (≤0.8% TRR). Absolute residues of parent were greatest in liver (≤0.570 md eq/kg) and fat (≤0.206 md eq/kg) with lower residues (0.011–0.025 md eq/kg) present in muscle, milk and kidney.

Table 11 Characterisation and identification of the radioactive residues in tissue and milk samples from lactating goats treated with [^{14}C]-pydiflumetofen

Compound	TRR [Phenyl-U-^{14}C]									
	Milk		Liver		Kidney		Muscle		Fat	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	0.102	83.7	2.96	42.3	1.05	60.2	0.085	81.7	0.208	93.9
Pydiflumetofen	0.019	15.7	0.570	8.2	0.014	0.8	0.025	24.4	0.149	67.2
Hydroxylated pydiflumetofen	ND	ND	ND	ND	ND	ND	ND	ND	0.019	8.6
SYN545547	ND	ND	0.239 (0.188)	3.4 (2.7)	0.128 (0.128)	7.4 (7.4)	ND	ND	ND	ND
SYN547891	ND	ND	0.100 (0.041)	1.4 (0.6)	ND	ND	ND	ND	ND	ND
SYN547897	ND	ND	0.136	1.9	0.050 (0.050)	2.9 (2.9)	0.002	1.8	ND	ND
SYN547948	0.003	2.2	0.180 (0.064)	2.6 (0.9)	0.016	0.9	0.004	3.8	0.012	5.3
2,4,6-TCP	0.052 (0.051)	43.2 (42.2)	0.037 (0.037)	0.5 (0.5)	0.021 (0.021)	1.2 (1.2)	0.009 (0.006)	9.0 (6.1)	ND	ND
Unknown[a]	0.020	16.3					0.042	39.7	0.024	10.9
in pre-enzyme hydrolysis organo soluble	-	-	0.220	3.1	0.068	3.9	-	-	-	-
in post enzyme hydrolysis organo soluble	-	-	0.696	10.0	0.331	18.9	-	-	-	-
in post enzyme	-	-	0.293	4.2	0.260	15.0	-	-	-	-

Compound	TRR [Phenyl-U-^{14}C]									
	Milk		Liver		Kidney		Muscle		Fat	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
hydrolysis water soluble										
in acetonitrile: water extract	-	-	0.489	7.0	0.109	6.3	-	-	-	-
Others[b]	0.008	6.3	-	-	0.050	2.9	0.003	3.0	0.004	1.9
Unextracted[c]	0.009	7.7	3.47	49.7	0.287	16.6	0.014	14.0	0.002	1.1
Total	0.111	91.4	6.43	92.0	1.33	76.8	0.099	95.7	0.210	95.0
Compound	TRR [Pyrazole-5-^{14}C]									
	Milk		Liver		Kidney		Muscle		Fat	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	0.113	85.9	3.45	39.3	1.61	68.7	0.113	80.2	0.274	98.5
Pydiflumetofen	0.011	8.7	0.179	2.0	0.011	0.5	0.018	13.4	0.206	73.8
Hydroxylated pydiflumetofen	ND	ND	ND	ND	ND	ND	ND	ND	0.028	10.2
SYN545547	ND	ND	0.160 (0.139)	1.8 (1.6)	ND	ND	ND	ND	ND	ND
SYN547891	ND	ND	0.038	0.4	ND	ND	ND	ND	ND	ND
SYN547897	ND	ND	0.268	3.0	0.063 (0.045)	2.7 (1.9)	0.002	1.2	ND	ND
SYN547948	0.001	0.7	0.170	1.9	0.016	0.7	0.002	1.1	0.009	3.3
SYN548263	0.019	14.2	ND	ND	0.389 (0.342)	16.6 (14.6)	0.007	4.9	0.012	4.3
SYN548264	0.038	28.7	ND	ND	0.019 (0.019)	0.8 (0.8)	0.001	0.6	ND	ND
SYN508272	0.014	11.0	ND	ND	0.036 (0.017)	1.5 (0.7)	0.024	17.7	0.003	1.0
NOA449410	0.003	2.6	0.248 (0.146)	2.9 (1.7)	0.275 (0.214)	11.7 (9.1)	0.005	3.6	ND	ND
Unknown[a]	0.025	18.3				34.2	0.052	36.4	0.009	3.4
in pre-enzyme hydrolysis organo soluble	-	-	0.678	7.9	0.128	5.5	-	-	-	-
in post enzyme hydrolysis organo soluble	-	-	0.925	10.5	0.380	16.3	-	-	-	-
in post enzyme hydrolysis water soluble	-	-	0.212	2.4	0.150	6.4	-	-	-	-
in acetonitrile: water extract	-	-	0.574	6.5	0.112	4.8	-	-	-	-
Others[b]	0.002	1.7	-	-	0.028	1.2	0.002	1.3	0.007	2.5
Unextracted[c]	0.008	6.1	4.64	52.6	0.215	9.2	0.008	5.7	0.007	2.4
Total	0.121	92.0	8.10	91.9	1.82	77.9	0.121	85.9	0.281	100.9

ND= Not detected

Values without the parentheses were the %TRR of both free and (where appropriate) conjugated forms of the residue. The proportion of the metabolite present in the conjugated (where appropriate) form is given within parentheses.

[a] Milk: Phenyl label comprising at least 7 discrete components, no single one of which >7.4% TRR (>0.009 mg/kg eq).

Pyrazole label comprising at least 7 discrete components, no single one of which >7.1% TRR (>0.009 mg/kg eq).

Liver: In pre-enzyme hydrolysis organo-soluble fraction;

Phenyl label comprising at least 3 discrete components, no single one of which >2.1% TRR (>0.147 mg/kg eq).

Pyrazole label comprising at least 14 discrete components, no single one of which >1.0% TRR (>0.085 mg/kg eq).

In post enzyme hydrolysis organo-soluble fraction;

Phenyl label comprising at least 12 discrete components, no single one of which >2.1% TRR (>0.147 mg/kg eq).

Pyrazole label comprising at least 9 discrete components, no single one of which >2.2% TRR (>0.195 mg/kg eq).

In post enzyme hydrolysis water soluble fraction;

Phenyl label comprising at least 12 discrete components, no single one of which >0.9% TRR (>0.063 mg/kg eq).

Pyrazole label comprising at least 14 discrete components, no single one of which >0.7% TRR (>0.062 mg/kg eq).

In acetonitrile:water (1:1, v/v) fraction;

Phenyl label: radioactivity located on the origin of 2D-TLC.

Pyrazole label: radioactivity located on the origin of 2D-TLC.

In both experiments the radioactivity located on the origin was characterised to be similar to that present in the liver PES

Kidney: In pre-enzyme hydrolysis organo-soluble fractions;

Phenyl label comprising at least 2 discrete components, no single one of which >1.0% TRR (>0.017 mg/kg eq).

Pyrazole label comprising at least 12 discrete components, no single one of which >0.8% TRR (>0.019 mg/kg eq).

In post enzyme hydrolysis organo-soluble fraction;

Phenyl label comprising at least 10 discrete components, no single one of which >5.6% TRR (>0.098 mg/kg eq).

Pyrazole label comprising at least 11 discrete components, no single one of which >5.4% TRR (>0.126 mg/kg eq).

In post enzyme hydrolysis water soluble fraction;

Phenyl label comprising at least 10 discrete components, no single one of which >5.0% TRR (>0.087 mg/kg eq).

Pyrazole label comprising at least 12 discrete components, no single one of which >2.0% TRR (>0.047 mg/kg eq).

In acetonitrile: water (1:1, v/v) fraction;

Phenyl label: radioactivity located on the origin of 2D-TLC.

Pyrazole label: radioactivity located on the origin of 2D-TLC.

In both experiments the radioactivity located on the origin was characterised to be similar to that present in the liver PES

Muscle: Phenyl label comprising at least 9 discrete components, no single one of which >7.4% TRR (>0.008 mg/kg eq).

Pyrazole label comprising at least 23 discrete components, no single one of which >5.6% TRR (>0.008 mg/kg eq).

Fat: Phenyl label comprising at least 4 discrete components, no single one of which >3.7% TRR (>0.008 mg/kg eq).

Pyrazole label comprising at least 2 discrete components, no single one of which >1.9% TRR (>0.005 mg/kg eq).

[b] Milk: Extracted residues in 4 fractions for phenyl and 3 fractions for pyrazole radiolabelled experiments were not analysed (associated residues too low for analysis). No single fraction comprised >3.5% TRR (>0.004 mg/kg eq) in either radiolabelled experiment.

Kidney: Extracted residues in 3 fractions (phenyl) and 2 fractions (pyrazole) that were produced during analysis and were individually too low for analysis. No single fraction comprised > 1.6% TRR (>0.028 mg/kg eq) in either radiolabelled experiment.

Muscle: Extracted residues in 1 fraction per radiolabelled experiment that were not chromatographed as the residues were too low for analysis. No single fraction comprised >3.0% TRR (>0.003 mg/kg eq) in either radiolabelled experiment.

Fat: Extracted residues in 3 fractions that were produced during processing and were too low for analysis.

Phenyl label: No single fraction comprised >1.9% TRR (>0.004 mg/kg eq).

Pyrazole label: No single fraction comprised >2.0% TRR (>0.006 mg/kg eq).

[c] Milk & Fat: Radioactivity remaining in the PES after extraction with hexane/aqueous acetonitrile. No further work was carried out on the PES (associated residues too low for analysis).

Liver & Kidney: Radioactivity remaining in the PES after extraction with aqueous acetonitrile. The nature of this residue was characterised further by SDS extraction procedure and protease digestion experiment and shown to comprise a complex mixture of unknowns.

Muscle: Radioactivity remaining in the PES after extraction with aqueous acetonitrile. The nature of phenyl residue was characterised further by SDS analysis.

Four metabolites with structures which retained only the pyrazole ring of pydiflumetofen were:

- SYN548264, an N-desmethoxy carboxylic acid, which was detected at its highest %TRR in milk (28.7% TRR, 0.038 md eq/kg, found in its free form). Much lower %TRRs were detected in kidney (0.8% TRR, 0.019 md eq/kg, conjugated form only) and in muscle (0.6% TRR, 0.001 md eq/kg, found in its free form only) and was not detected in liver or fat,
- SYN508272, an amide, which was detected at its highest %TRRs in muscle (17.7% TRR, 0.024 md eq/kg) and milk (11.0% TRR, 0.014 md eq/kg) found both in the free form only. Much lower %TRRs were detected in kidney (1.5% TRR, 0.036 md eq/kg; found in both its free and conjugated forms) and in fat (1.0% TRR, 0.003 md eq/kg, in its free form only) and was not detected in liver,
- SYN548263, a carboxylic acid, which was detected at its highest %TRRs in kidney (16.6% TRR, 0.389 md eq/kg, in predominantly its conjugated form) and milk (14.2% TRR, 0.019 md eq/kg, found in its free form only). Much lower %TRRs were detected in muscle (4.9% TRR, 0.007 md eq/kg) and in fat (4.3% TRR, 0.012 md eq/kg) found in the free form only of the metabolite, and was not detected in liver,

- NOA449410, a carboxylic acid, which was detected at its highest %TRR in kidney (11.7% TRR, 0.275 md eq/kg, in predominantly its conjugated form). Lower %TRRs were detected in liver (2.9% TRR, 0.248 md eq/kg, found in both the free and conjugated forms) and in muscle and milk (3.6% TRR and 2.6% TRR respectively, ≤0.005 md eq/kg) in only the free form, and was not detected in fat.

2,4,6-trichlorophenol, a phenolic half molecule metabolite with a structure retaining only the phenyl ring of pydiflumetofen, detected at its highest %TRRs in milk (43.2% TRR, 0.052 md eq/kg) and muscle (9.0% TRR, 0.009 md eq/kg). In both milk and muscle the residue was found predominantly in its sulphate ester conjugated form of the metabolite. Levels of this metabolite (%TRR) were much lower in kidney (1.2% TRR, 0.021 md eq/kg) and liver (0.5% TRR, 0.037 md eq/kg, found in conjugated form only in both tissues) and were not detected in fat.

Two metabolites with structures which retained both the phenyl and pyrazole rings of pydiflumetofen were:

- a hydroxylated metabolite of pydiflumetofen (position of hydroxyl functionality not established) which was detected only in fat (≤10.2% TRR, ≤0.028 md eq/kg) in its free form and
- SYN545547, an N-desmethoxy metabolite, which was detected at its highest %TRR in kidney (≤7.4% TRR, 0.128 md eq/kg, found only in its conjugated form) and in liver (≤3.4% TRR, ≤0.239 md eq/kg, found in both its free and conjugated forms), and was not detected in milk, muscle and fat.
- Other minor identified metabolites with structures which retained both the phenyl and pyrazole rings of pydiflumetofen were:
- SYN547948, a hydroxylated metabolite, detected in all tissues and milk (0.7–5.3% TRR, 0.001–0.180 md eq/kg, found in both free and conjugated form in liver and in the free form only in other tissues and milk),
- SYN547897, a phenolic metabolite detected only in liver, kidney and muscle (1.2–3.0% TRR, 0.002–0.268 md eq/kg, found in both free and conjugated forms in kidney and in the free form only in liver and muscle) and
- SYN547891, a pyrazole N-desmethyl metabolite detected only in liver (≤1.4% TRR, ≤0.100 md eq/kg, found in both the free and conjugated forms).

Radioactivity that remained unextracted by solvent in PES samples (phenyl liver: 49.7% TRR; 3.47 md eq/kg, pyrazole liver: 52.6% TRR; 4.64 md eq/kg and phenyl kidney: 16.6% TRR; 0.287 md eq/kg, pyrazole kidney: 9.2% TRR; 0.215 md eq/kg) was further characterised in two separate experiments.

In one experiment, efficient residue extraction using sodium dodecyl sulphate solution (creates a micellar suspension of protein) followed by a protein precipitation step showed that approximately two fifths to one half of the liver PES residue and approximately one half of the kidney PES residue to be associated with the protein fraction. Consequently in a second experiment, aliquots of liver and kidney PES were subject to protease enzyme hydrolysis which released significant proportions of the residues (liver: 39.6–46.3% TRR; kidney: 9.3–15.0% TRR) into the resulting hydrolysate. TLC of these protease hydrolysates showed the constituent residue to comprise a complex mixture of unknown and highly polar metabolites, individual levels of which were ≤7.4% TRR and ≤0.620 md eq/kg in liver and ≤5.3% TRR and ≤0.092 md eq/kg in kidney. The nature of these hydrolysate residues appeared similar between liver and kidney and between radiolabelled experiments. The nature of the radioactive residue present in a corresponding muscle PES sample (phenyl: 14.0% TRR; 0.014 md eq/kg) was deemed by analogy to be of a similar complex nature to that of the liver and kidney.

Laying hens

The study was conducted to investigate the nature and quantity of residues in liver, muscle, fat and eggs from the laying hen following oral dosing with [^{14}C]-pydiflumetofen (Przeorska, 2015: 33964). Six laying hens per radiolabelled treatment were dosed orally with [phenyl-U-^{14}C]-pydiflumetofen or [pyrazole-5-^{14}C]-pydiflumetofen for 14 days at a nominal rate of 30 ppm, dry matter intake. The actual dose rates achieved based on measured food consumption were approximately 56.3 and 56.9 ppm dry matter intake, respectively (3.3 and 3.6 mg ai/kg bodyweight respectively) (not including Study Day 14 as this was not a complete 24 h period). Excreta and eggs were collected daily. Eggs were separated into yolk and white. The hens were sacrificed approximately 11 hours after the administration of the final dose and tissues taken *post mortem* for quantification and analysis.

All samples were radioassayed to determine the distribution of radioactivity and radioactive balance. The radioactive residue concentration present in eggs and tissue samples (liver, muscle and fat) were also determined. Further analysis was undertaken to determine the nature and quantity of constituent residues where the radioactive residue concentration was ≥0.01 md eq/kg, samples from the individual hens were pooled by sample type. Samples were extracted with solvents (fat: hexane and aqueous acetonitrile, liver, egg yolk, egg white and muscle: aqueous acetonitrile) and the extracted residues characterised. Fractions containing significant radioactive residues were subject to 1D and/or 2D TLC/bioimage analysis and HPLC/radiodetection analysis to enable quantification and identification of residues. Where appropriate, these fractions were subject to organic solvent/water partition and enzyme (β-glucuronidase) hydrolysis procedures prior to chromatographic analysis. Residues were

identified by chromatographic comparison with authentic synthetic reference standards of parent pydiflumetofen and its metabolites.

Initial radiocomponent profiles of principal analytical fractions of representative samples (egg yolk and fat) were produced within 6 months of sampling from the hens. Reanalysis of these residues after completion of nature of residue analysis demonstrated the radiocomponent profiles not to have changed significantly during the interim period of frozen storage.

The radioactive balance for all hens was greater than 81.4% with the majority of the radioactivity recovered in excreta as summarised in Table 12.

Table 12 Distribution of radioactivity and material balance from laying hens dosed with [^{14}C]-pydiflumetofen

Sample	% of administered dose recovered (mg/kg eq): Phenyl-U-^{14}C						
	1	2	3	4	5	6	Mean
Excreta	99.3	94.3	101.8	99.9	98.3	103.8	99.1
Egg white	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Egg yolk	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Part formed eggs	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Breast muscle	<0.1 (0.013)	<0.1 (0.012)	<0.1 (0.039)	<0.1 (0.020)	<0.1 (0.021)	<0.1 (0.007)	<0.1 (0.019)
Leg and thigh muscle	<0.1 (0.022)	<0.1 (0.021)	<0.1 (0.068)	<0.1 (0.029)	<0.1 (0.038)	<0.1 (0.014)	<0.1 (0.032)
Skin and fat	<0.1 (0.088)	<0.1 (0.094)	<0.1 (0.188)	<0.1 (0.091)	<0.1 (0.125)	<0.1 (0.052)	<0.1 (0.106)
Peritoneal fat	<0.1 (0.075)	<0.1 (0.079)	<0.1 (0.131)	<0.1 (0.059)	<0.1 (0.097)	<0.1 (0.045)	<0.1 (0.081)
Liver	<0.1 (0.410)	<0.1 (0.263)	<0.1 (0.633)	<0.1 (0.347)	<0.1 (0.446)	<0.1 (0.172)	<0.1 (0.379)
GI contents	0.3	0.1	0.8	0.8	0.7	0.4	0.5
GI tract	0.2	0.1	0.5	0.1	0.3	0.1	0.2
Cage wash	3.7	6.3	2.6	2.4	3.1	3.2	3.6
Blood	NS	<0.1	<0.1	<0.1	NS	<0.1	<0.1
Material Balance	103.5	100.8	105.7	103.2	102.4	107.5	103.4
Sample	% of administered dose recovered (mg/kg eq): Pyrazole-5-^{14}C						
	7	8	9	10	11	12	Mean
Excreta	88.7	82.6	84.0	81.4	83.1	87.3	84.3
Egg white	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Egg yolk	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Part formed eggs	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Breast muscle	<0.1 (0.014)	<0.1 (0.028)	<0.1 (0.012)	<0.1 (0.031)	<0.1 (0.011)	<0.1 (0.017)	<0.1 (0.019)
Leg and thigh muscle	<0.1 (0.017)	<0.1 (0.032)	<0.1 (0.013)	<0.1 (0.041)	<0.1 (0.012)	<0.1 (0.018)	<0.1 (0.022)
Skin and fat	<0.1 (0.033)	<0.1 (0.059)	<0.1 (0.027)	<0.1 (0.068)	<0.1 (0.024)	<0.1 (0.028)	<0.1 (0.040)
Peritoneal fat	<0.1 (0.014)	<0.1 (0.035)	<0.1 (0.011)	<0.1 (0.032)	<0.1 (0.009)	<0.1 (0.020)	<0.1 (0.020)
Liver	<0.1 (0.146)	<0.1 (0.399)	<0.1 (0.089)	<0.1 (0.265)	<0.1 (0.123)	<0.1 (0.202)	<0.1 (0.204)
GI contents	0.2	0.8	0.1	0.3	0.1	0.2	0.3
GI tract	0.2	0.2	0.1	0.1	0.1	0.2	0.2
Cage wash	2.0	5.3	1.7	3.6	3.6	3.1	3.2
Blood	<0.1	<0.1	NS	<0.1	NS	<0.1	<0.1
Material Balance	91.1	88.9	85.9	85.4	86.9	90.8	88.0

NS: No sample

Mean residues in egg yolks achieved a plateau concentration of approximately 0.344 md eq/kg (phenyl) and 0.116 md eq/kg (pyrazole) after 10 and 7 days of dosing, respectively. Mean residues in egg whites achieved a plateau concentration of approximately 0.064 md eq/kg (phenyl) and 0.062 md eq/kg (pyrazole) after 6 and 7 days of dosing, respectively.

Table 13 Total radioactive residues in eggs from laying hens dosed with [^{14}C]-pydiflumetofen

Study day	Time point (h)	Phenyl-U-^{14}C, mg/kg eq			Pyrazole-5-^{14}C, mg/kg eq		
		Mean egg white	Mean egg yolk	Mean whole egg	Mean egg white	Mean egg yolk	Mean whole egg
1	24	0.015	0.035	0.021	0.014	0.005	0.011
2	48	0.058	0.034	0.051	0.055	0.015	0.043
3	72	0.088	0.100	0.092	0.061	0.033	0.052
4	96	0.072	0.161	0.098	0.055	0.051	0.054
5	120	0.087	0.218	0.127	0.071	0.073	0.072
6	144	0.064	0.269	0.126	0.092	0.094	0.093
7	168	0.060	0.284	0.123	0.062	0.116	0.078
8	192	0.052	0.311	0.128	0.053	0.085	0.063
9	216	0.063	0.322	0.139	0.062	0.114	0.077
10	240	0.060	0.344	0.140	0.064	0.105	0.077
11	264	0.061	0.355	0.147	0.058	0.119	0.077
12	288	0.061	0.359	0.150	0.055	0.108	0.071
13	312	0.049	0.331	0.130	0.073	0.106	0.084
14	324	NS	NS	NS	0.061	0.093	0.070

Figure 4 Residues in egg white following dosing with [^{14}C]-pydiflumetofen

Figure 5 Residues in egg yolk following dosing with [^{14}C]-pydiflumetofen

The nature of residues in phenyl-treated egg yolk and egg white were investigated using the Day 10–13 and Day 6–13 composite samples, respectively. The nature of residues in pyrazole-treated eggs (yolk and white) were investigated using the Day 7–13 composite samples. All composite tissues (*i.e.* liver, muscle and fat) and egg yolk and egg white samples were extracted with solvents and analysed by TLC and HPLC.

Good extractability was achieved for the eggs, muscle and fat (≥81.2% TRR). Extractability of liver was lower (≥51.7% TRR).

Table 14 Extractability and distribution of the radioactive residues in eggs and tissues of laying hens

Radiolabel	Sample	Extracted radioactivity		Unextracted radioactivity		TRR[a]
		mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
[Phenyl-U-^{14}C]	Liver	0.209	51.7	0.195[b]	48.3[b]	0.404
	Egg yolk	0.311	87.0	0.047	13.0	0.358
	Egg white	0.052	97.7	0.001	2.3	0.053
	Muscle[c]	0.023	84.2	0.004	15.8	0.027
	Skin & Fat[d]	0.096	95.8	0.004	4.3	0.101
[Pyrazole-5-^{14}C]	Liver	0.111	52.5	0.100	47.5	0.210
	Egg yolk	0.087	81.2	0.020	18.7	0.106
	Egg white	0.051	98.8	0.001	1.2	0.052
	Muscle[c]	0.018	90.1	0.002	9.9	0.021
	Skin & Fat[d]	0.029	91.5	0.003	8.4	0.032

[a] mg/kg calculated directly from radioactivity extracted, radioactivity in the PES and specific activity

[b] Following a separate extraction procedure, unextracted radioactivity (49.0%; 0.192 md eq/kg) was investigated further

[c] Composite muscle samples (includes breast and combined leg and thigh muscle)

[d] Composite fat samples (includes peritoneal fat and subcutaneous fat with skin attached)

Parent pydiflumetofen was identified in eggs and all tissues. Parent %TRRs were greatest in egg white (≤46.5% TRR) and fat (≤30.6% TRR) with much smaller %TRRs being found in muscle (≤8.7% TRR), egg yolk (≤11.0% TRR) and liver (≤5.3% TRR). Absolute residues of parent were greatest in egg white (≤0.025 md eq/kg), liver (≤0.021 md eq/kg) and fat (≤0.017 md eq/kg) with lower residues present in egg yolk (0.011–0.012 md eq/kg) and muscle (0.001–0.002 md eq/kg).

Table 15 Characterization and identification of the radioactive residues in tissues and eggs from laying hens treated with [^{14}C]-pydiflumetofen

Compound	TRR [Phenyl-U-^{14}C]									
	Liver		Egg yolk		Egg white		Muscle		Fat	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	0.187	46.8	0.302	84.5	0.050	92.7	0.021	77.2	0.088	84.9
Pydiflumetofen	0.021	5.3	0.011	3.0	0.025	46.5	0.002	8.7	0.017	16.6
SYN545547	0.005 (0.003)	1.2 (0.6)	ND	ND	ND	ND	ND	ND	ND	ND
SYN547891	0.001 (0.001)	0.2 (0.2)	ND	ND	ND	ND	ND	ND	ND	ND
SYN547897	0.009 (0.001)	2.4 (0.4)	0.008	2.3	ND	ND	ND	ND	0.002	1.7
SYN547948	0.003	0.7	ND	ND	0.004	7.1	0.001	3.4	0.003	3.0
2,4,6-TCP	ND	ND	0.242 (0.242)	67.8 (67.8)	0.008 (0.008)	14.5 (14.5)	0.013 (0.013)	48.4 (48.4)	0.030 (0.027)	29.3 (26.5)
Unknown[a]			0.024	6.6	0.013	24.6	0.004	14.3	0.015	13.8
in pre-enzyme hydrolysis organo soluble	0.078	19.4								
in post enzyme hydrolysis organo soluble	0.023	6.1								
in post enzyme hydrolysis water soluble	0.017	4.1								
in acetonitrile: water extract			0.009	2.6						
Others[b]	0.030	7.4	0.008	2.2			0.001	2.4	0.021	20.5
Unextracted[c]	0.195	48.3	0.047	13.0	0.001	2.3	0.004	15.8	0.004	4.3
Total	0.382	95.1	0.349	97.5	0.051	95.0	0.025	93.0	0.092	89.2
Compound	TRR [Pyrazole-5-^{14}C]									
	Liver		Egg yolk		Egg white		Muscle		Fat	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	0.084	40.9	0.076	70.2	0.049	91.8	0.017	82.0	0.029	85.0
Pydiflumetofen	0.001	0.5	0.012	11.0	0.014	26.6	0.001	4.7	0.010	30.6
SYN545547	0.007 (0.007)	3.3 (3.3)	0.004	3.9	ND	ND	ND	ND	ND	ND
SYN547891	ND	ND	0.003	2.5	ND	ND	ND	ND	ND	ND
SYN547897	0.002 (0.002)	0.9 (0.9)	0.007	6.7	ND	ND	<0.001	1.1	0.001	2.6
SYN547948	0.007	3.2	0.001	1.3	0.003	5.5	<0.001	1.6	0.001	4.1
SYN508272	0.005 (0.005)	2.4 (2.4)	0.008 (0.003)	7.2 (2.6)	0.018	34.3	0.010	46.3	0.003	9.6
NOA449410	ND	ND	0.007 (0.001)	6.6 (0.8)	0.008	15.4	ND	ND	0.001	3.1
Unknown[a]					0.006	10.0	0.004	21.3	0.012	32.3
in pre-enzyme hydrolysis organo soluble	0.023	11.3	0.020	18.7						
in post enzyme hydrolysis organo soluble	0.012	6.0	0.008	6.8						
in post enzyme hydrolysis water soluble	0.010	4.7								
Others[b]	0.017	8.6	0.006	5.5			0.002	7.0	0.001	2.7
Unextracted[c]	0.100	47.5	0.020	18.7	0.001	1.2	0.002	9.9	0.003	8.4
Total	0.184	88.4	0.096	88.9	0.050	93.0	0.019	91.9	0.032	93.4

ND= Not detected

Values without the parentheses were the %TRR of both free and (where appropriate) conjugated forms of the residue. The proportion of the metabolite present in the conjugated (where appropriate) form is given within parentheses.

[a] Liver: In pre-enzyme hydrolysis organo soluble fraction

Phenyl label comprising at least 27 discrete components, no single one of which >2.3% TRR (>0.009 mg/kg eq).

Pyrazole label comprising at least 9 discrete components, no single one of which >2.3% TRR (>0.005 mg/kg eq).

In post enzyme hydrolysis organo soluble fraction

Phenyl label comprising at least 16 discrete components, no single one of which >1.6% TRR (>0.006 mg/kg eq).

Pyrazole label comprising at least 8 discrete components, no single one of which >1.4% TRR (>0.003 mg/kg eq).

In post enzyme hydrolysis water soluble fraction

Phenyl label comprising at least 8 discrete components, no single one of which >1.0% TRR (>0.004 mg/kg eq).

Pyrazole label shown to consist of radioactivity located on the origin, characterised to be similar to that present in the liver PES.

Egg yolk: Phenyl label comprising at least 3 discrete components, no single one of which >3.5% TRR (>0.013 mg/kg eq).

In pre-enzyme hydrolysis organo-soluble fraction

Pyrazole label comprising at least 9 discrete components, no single one of which >5.9% TRR (>0.006 mg/kg eq).

In post enzyme hydrolysis organo-soluble fraction

Pyrazole label comprising at least 5 discrete components, no single one of which >4.3% TRR (>0.005 mg/kg eq).

In acetonitrile: water (1:1, v/v) fraction analysed by 2D-TLC

Phenyl label comprising at least 3 discrete components, no single one of which >1.7% TRR (>0.006 mg/kg eq).

Egg white: Phenyl label comprising at least 7 discrete components, no single one of which >12.3% TRR (>0.006 mg/kg eq).

Pyrazole label comprising at least 3 discrete components, no single one of which >5.0% TRR (>0.003 mg/kg eq).

Muscle: Phenyl label comprising at least 4 discrete components, no single one of which >5.0% TRR (>0.001 mg/kg eq).

Pyrazole label comprising at least 6 discrete components, no single one of which >6.0% TRR (>0.001 mg/kg eq).

Fat: Phenyl label comprising at least 5 discrete components, no single one of which >3.7% TRR (>0.004 mg/kg eq).

Pyrazole label comprising at least 9 discrete components, no single one of which >6.4% TRR (>0.002 mg/kg eq).

[b] Liver: Phenyl label consists of two fractions no individual one of which accounts for >6.7% TRR (>0.027mg/kg eq). Although this particular fraction was not chromatographed, a corresponding fraction from the initial phenyl liver analysis was analysed (acetonitrile: water (1:1, v/v) fraction: 4.7% TRR, 0.018 mg/kg eq) and shown to consist of radioactivity located on the origin, characterised to be similar to that present in the liver PES.

Pyrazole label consists of 4 fractions none of which accounts for >4.3% TRR (0.009 mg/kg eq).

Egg yolk: Extracted residue fractions which were not analysed ([^{14}C] residues too low for analysis). No single fraction comprised ≥ 4.3% TRR (≥0.005 mg/kg eq) in either radiolabelled experiment.

Muscle: Extracted residue fractions which were not analysed (associated residues too low for analysis). No single fraction comprised > 4.2% TRR (>0.001 mg/kg eq) in either radiolabelled experiment.

Fat: Extracted residue fractions which were not analysed (associated residues too low for analysis). No single fraction comprised > 6.0% TRR (>0.006 mg/kg eq) in either radiolabelled experiment.

[c] Liver: Radioactivity remaining in the PES after extraction with aqueous acetonitrile. The nature of this residue was characterised further by protease digestion and SDS experiment. For the phenyl label these experiments were performed on the re-extracted residues liver (49.0% TRR, 0.192 mg/kg eq). Following these analyses the PES residues were characterised to be similar in nature in both radiolabelled experiments and comprise a complex mixture of polar unknown radiocomponents.

Egg yolk: Radioactivity remaining in the PES after extraction with aqueous acetonitrile. The nature of this residue was characterised further by protease digestion and SDS extraction. Following these analyses the PES residues were characterised to be similar in nature in both radiolabelled experiments and comprising radiocomponent(s) deemed to be of low bioavailability.

Egg white & Muscle: Radioactivity remaining in the PES after extraction with aqueous acetonitrile. No further work was carried out on the PES (associated residues too low for analysis).

Fat: Radioactivity remaining in the PES after extraction with hexane/aqueous acetonitrile. No further work was carried out on the PES (associated residues too low for analysis).

The principal identified hen metabolites of pydiflumetofen were:

- - 2,4,6-TCP, a phenolic half molecule metabolite with a structure retaining only the phenyl ring of pydiflumetofen, was detected at its highest %TRRs in egg yolk (67.8% TRR, 0.242 md eq/kg), egg white (14.5% TRR, 0.008 md eq/kg), muscle (48.4% TRR, 0.013 md eq/kg) and fat (29.3% TRR, 0.030 md eq/kg). In eggs and all tissues the residue was found predominantly in its sulphate ester conjugated form of the metabolite.

- - SYN508272, an amide, which was detected at its highest %TRRs in muscle (46.3% TRR, 0.010 md eq/kg), egg white (34.3% TRR, 0.018 md eq/kg) and fat (9.6% TRR, 0.003 md eq/kg). SYN508272 was found in the free form only. Much lower %TRRs were detected in egg yolk (7.2% TRR, 0.008 md eq/kg) and liver (2.4% TRR, 0.005 md eq/kg); found in both its free and conjugated forms.
- - NOA449410, a carboxylic acid, which was detected at its highest %TRR in egg white (15.4% TRR, 0.008 md eq/kg, found only in the free form). Lower %TRRs were detected in egg yolk (6.6% TRR, 0.007 md eq/kg, found in both the free and conjugated forms), in fat (3.1% TRR, 0.001 md eq/kg, found in the free form only) and was not detected in liver and muscle.
- - SYN545547, an N-desmethoxy metabolite, which was detected in egg yolk (≤3.9% TRR, ≤0.004 md eq/kg, found only in the free form), in liver (≤3.3% TRR, ≤0.007 md eq/kg) found in both its free and conjugated forms and was not detected in egg white, muscle and fat.
- Other more minor identified metabolites and with structures which retained both the phenyl and pyrazole rings of pydiflumetofen were:
- - SYN547948, a hydroxylated metabolite, detected in all tissues and eggs (0.7–7.1% TRR, <0.001–0.007 md eq/kg, excluding phenyl egg yolk), found only in the free form.
- - SYN547897, a phenolic metabolite detected in liver, egg yolk, muscle and fat (0.9–6.7% TRR, <0.001–0.009 md eq/kg), found in both free and conjugated forms.
- - SYN547891, a pyrazole N-desmethyl metabolite detected only in liver (phenyl) and egg yolk (pyrazole) (0.2–2.5% TRR, 0.001–0.003 md eq/kg), found in the conjugated form for liver and free form for egg yolk.

Radioactivity that remained unextracted by solvent in PES samples (liver: 47.5–49.0% TRR, 0.100–0.192 md eq/kg and egg yolk: 13.0–18.7% TRR, 0.020–0.047 md eq/kg) was further characterised in two separate experiments:

In one experiment, efficient residue extraction using sodium dodecyl sulphate solution (creates a micellar suspension of protein) followed by a protein precipitation step showed that approximately half of the liver PES residue and egg yolk PES residue were associated with the protein fraction. Consequently in a second experiment, separate aliquots of liver and egg yolk PES were subject to protease enzyme hydrolysis which released significant proportions of the residues (liver: 32.4–45.6% TRR; egg yolk: 6.1–11.4% TRR) into the resulting hydrolysate. TLC of these protease hydrolysates from liver showed the constituent residue to comprise a complex mixture of unknown and highly polar metabolites, individual levels of which were ≤7.6%TRR (≤0.016 md eq/kg) in the pyrazole label. A similar profile was observed in the phenyl liver analysis.

Summary of animal metabolism

The metabolism and distribution of pydiflumetofen have been investigated in ruminant and poultry species (goat and hen). In both studies, the majority (≥76.3%) of the dosed radioactivity was excreted and radioactivity remaining in dietary tissues, milk and eggs was low (collectively accounting for <1%). Residues were readily extractable into solvents for milk, kidney, muscle and fat in the goat (≥83.4% TRR) and for muscle, fat, egg yolk and egg white in the hen (≥81.2% TRR). Residue extractability was lower in the liver of both species (47.4–50.4% TRR in goat and 51.7–52.5% TRR in hen). Analysis of the extracted residues confirmed the biotransformation of pydiflumetofen to be characterised by the oxidation of pydiflumetofen, N-demethylation of the pyrazole ring, N-demethoxylation of the amide nitrogen and cleavage at the benzylic methylene, N-alkyl and amide linkages between the phenyl and pyrazole rings. Monohydroxylation occurred at the benzyl methylene functional group on the trichlorophenyl ring to form a phenol and also at an unspecified location (hydroxy pydiflumetofen). The above biotransformations were also consistent with those observed in the rat metabolism of pydiflumetofen.

The following metabolic pathways was proposed:

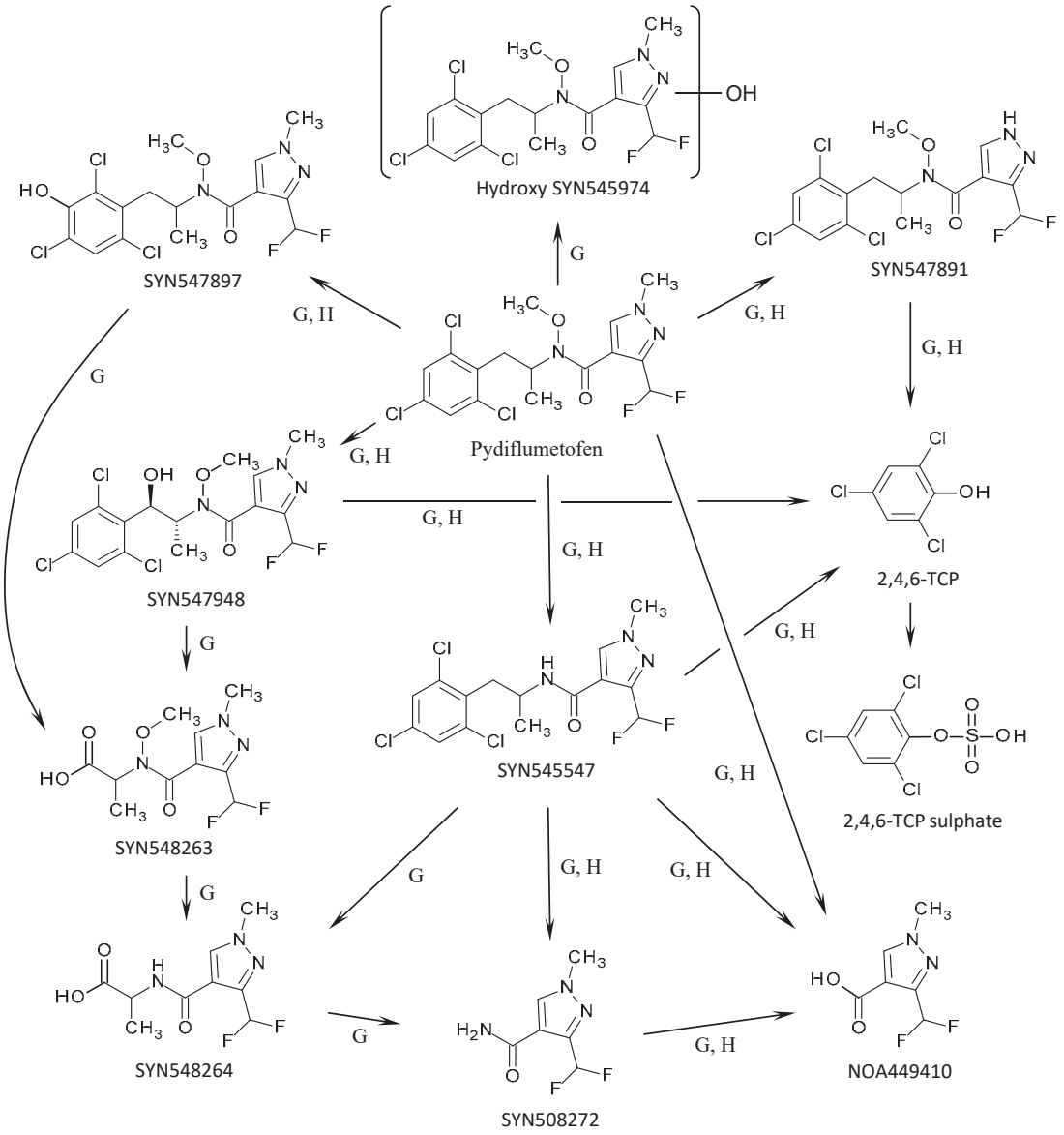


Figure 6 Metabolic Pathway of Pydiflumetofen in Animals (lactating goat and laying hen)

Rotational crop studies

Confined rotational crop study

The metabolism in representative succeeding crops (lettuce, wheat and turnip) were investigated with [^{14}C]-pydiflumetofen (Chapleo, 2015: 34316). The crops were sown 30, 120 and 270 days after application (DAA) of the test substance at a nominal rate of 0.400 kg ai/ha to a sandy loam soil. The actual application rates achieved were 0.388 kg ai/ha for the [Phenyl-U-^{14}C]-pydiflumetofen labelled treatment and 0.409 kg ai/ha for the [Pyrazole-5-^{14}C]-pydiflumetofen labelled treatment. The radiochemicals were formulated as an SC formulation and applied to the soil as a single spray application. The soil containers were maintained outdoors until 28 days after application when they were moved into a glasshouse for the remainder of the crop growth phase.

At each rotational interval, seeds of representative cereal (spring wheat), leafy vegetable (lettuce) and root vegetable (turnip) crops were sown. Crops were harvested at appropriate immature and mature growth stages and separated into

commodities representative of food and feed items (wheat: forage, hay, straw and grain; lettuce: immature and mature foliage; turnip: foliage and roots). Homogenised samples of all the commodities were prepared for analysis. Sub-samples of the crop commodities were taken for initial overall residue determination employing sample oxidation with LSC analysis. Appropriate amounts of sample were combusted to achieve a limit of determination (LOD) of 0.001 mg/kg. Commodities with a TRR ≥0.01 mg/kg were extracted with aqueous acetonitrile solvent combinations and where possible the extracted residue was analysed by HPLC and TLC to determine the nature of the residues.

The TRRs were highest in the wheat foliage derived commodities; wheat forage (≤0.031 md eq/kg), wheat hay (≤0.108 md eq/kg) and wheat straw (≤0.219 md eq/kg), with residues increasing as the crop matured. Highest residues were detected in wheat straw. Generally, the highest residues were observed in the 30 DAA rotational commodities declining in the 120 DAA and 270 DAA rotational commodities (wheat hay – phenyl: 0.064 md eq/kg decreasing to 0.039 md eq/kg, wheat straw – phenyl: 0.174 md eq/kg decreasing to 0.114 md eq/kg). Two exceptions were observed in the pyrazole label where residues were highest at 120 DAA in wheat hay and straw, with levels decreasing significantly in the 270 DAA commodities (wheat hay – pyrazole: 0.108 md eq/kg decreasing to 0.035 md eq/kg, wheat straw – pyrazole: 0.219 md eq/kg decreasing to 0.164 md eq/kg). Much lower TRRs were observed at all plantback intervals in wheat grain (≤0.008 md eq/kg), immature lettuce (≤0.019 md eq/kg), mature lettuce (≤0.007 md eq/kg), turnip foliage (≤0.012 md eq/kg) and turnip tubers (≤0.008 md eq/kg). All commodities showed a decline in residues at the final 270 DAA plant back interval.

Table 16 Total radioactive residues (TRRs) found in crops for confined rotational crop study

Sample			TRR (mg/kg eq)[a]		
			30 DAA	120 DAA	270 DAA
Wheat	Forage	[Phenyl-U-^{14}C]	0.031	0.010	0.013
		[Pyrazole-5-^{14}C]	0.028	0.027	0.014
	Hay	[Phenyl-U-^{14}C]	0.064	0.059	0.039
		[Pyrazole-5-^{14}C]	0.092	0.108	0.035
	Straw	[Phenyl-U-^{14}C]	0.174	0.153	0.114
		[Pyrazole-5-^{14}C]	0.211	0.219	0.164
	Grain	[Phenyl-U-^{14}C]	0.004	0.005	0.003
		[Pyrazole-5-^{14}C]	0.008	0.007	0.002
Lettuce	Immature	[Phenyl-U-^{14}C]	0.012	0.005	0.001
		[Pyrazole-5-^{14}C]	0.019	0.004	0.006
	Mature	[Phenyl-U-^{14}C]	0.001	0.005	0.001
		[Pyrazole-5-^{14}C]	0.007	0.004	0.002
Turnip	Foliage	[Phenyl-U-^{14}C]	0.010	0.004	0.004
		[Pyrazole-5-^{14}C]	0.012	0.007	0.007
	Tubers	[Phenyl-U-^{14}C]	0.007	0.002	0.002
		[Pyrazole-5-^{14}C]	0.008	0.003	0.002

[a] Where extracted the TRR values of commodities were also determined by the summation of the radioactivity present in the extracts and PES after initial fractionation.

All commodities with TRRs ≥0.01 md eq/kg were extracted with solvents and analysed. High solvent extractability (83.2–96.8% TRR) was achieved for all analysed commodities. The following table displays the extractability and distribution of radioactive residues in analysed rotational commodities.

Table 17 Extractability and distribution of radioactive residues in analysed crop rotation commodities

Rotational interval	Crop commodity	Label	Extracted radioactivity		Unextracted radioactivity		TRR[a]
			mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
30 DAA	Wheat Forage	[Phenyl-U-^{14}C]	0.030	96.8	0.001	3.2	0.031
		[Pyrazole-5-^{14}C]	0.027	96.8	0.001	3.2	0.028
	Wheat Hay	[Phenyl-U-^{14}C]	0.060	91.9	0.005	8.1	0.064
		[Pyrazole-5-^{14}C]	0.082	90.0	0.009	9.9	0.092
	Wheat Straw	[Phenyl-U-^{14}C]	0.151	86.9	0.021	12.2	0.174
		[Pyrazole-5-^{14}C]	0.187	88.2	0.024	11.3	0.211
	Lettuce Immature	[Phenyl-U-^{14}C]	0.010	85.5	0.002	14.5	0.012
		[Pyrazole-5-^{14}C]	0.016	86.1	0.003	13.9	0.019
	Turnip Foliage	[Phenyl-U-^{14}C]	0.010	93.6	0.001	6.5	0.010
		[Pyrazole-5-^{14}C]	0.011	89.6	0.001	10.4	0.012

Rotational interval	Crop commodity	Label	Extracted radioactivity		Unextracted radioactivity		TRR[a]
			mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
120 DAA	Wheat Forage	[Phenyl-U-^{14}C]	0.010	89.2	0.001	10.7	0.010
		[Pyrazole-5-^{14}C]	0.025	91.1	0.002	8.9	0.027
	Wheat Hay	[Phenyl-U-^{14}C]	0.050	85.2	0.009	14.8	0.059
		[Pyrazole-5-^{14}C]	0.091	84.5	0.017	15.5	0.108
	Wheat Straw	[Phenyl-U-^{14}C]	0.131	85.9	0.022	14.1	0.153
		[Pyrazole-5-^{14}C]	0.188	85.9	0.031	14.1	0.219
270 DAA	Wheat Forage	[Phenyl-U-^{14}C]	0.013	96.3	<0.001	3.7	0.013
		[Pyrazole-5-^{14}C]	0.013	95.5	0.001	4.5	0.014
	Wheat Hay	[Phenyl-U-^{14}C]	0.037	94.7	0.002	5.3	0.039
		[Pyrazole-5-^{14}C]	0.033	93.5	0.002	6.5	0.035
	Wheat Straw	[Phenyl-U-^{14}C]	0.100	87.3	0.014	12.7	0.114
		[Pyrazole-5-^{14}C]	0.138	83.2	0.028	16.8	0.164

[a] Total radioactive residue of commodity determined by summation of the initial aqueous acetonitrile extracts.

High levels of identification were achieved with parent representing the major portion of the residue in all commodities and declining between the 30 DAA and 270 DAA intervals. The highest absolute residue of parent was detected in 120 DAA pyrazole wheat straw of 0.063 md eq/kg, (28.7% TRR), declining to 0.030 md eq/kg (18.6% TRR) by 270 DAA. Parent pydiflumetofen again represented the major component in both immature lettuce and turnip foliage at 30 DAA (maximum of 76.7% and 77.2% TRR, respectively) but only equated to maximum residue levels of 0.015 md eq/kg and 0.008 md eq/kg respectively.

Metabolites identified were the de-alkylated molecules, SYN5457891 and SYN545547 detected in all commodities. Residues of SYN545547 accounted for ≤5.6% TRR with the largest residue detected in 30 DAA and 120 DAA pyrazole labelled straw (0.005 md eq/kg, 2.2–2.3% TRR). Residues of SYN547891 accounted for ≤13.3% TRR with the highest residue detected in 30 DAA pyrazole labelled straw (0.012 md eq/kg, 5.5% TRR). There was no firm evidence for conjugation of metabolites in rotational crops. Multiple polar unidentified metabolites were detected in wheat with the highest individual residue accounting for 0.014 md eq/kg (6.7% TRR; 30 DAA straw). These were characterised by solvent partitioning and by acid, base and pectinase-mediated hydrolyses. The enantiomer fraction was 0.5 for the applied radiochemical and 0.55–0.57 for 120 DAA and 270 DAA straw extracts.

Table 18 Characterization and identification of the radioactive residues in rotational crop wheat forage treated with [^{14}C]-pydiflumetofen

Components	TRR [Phenyl-U-^{14}C]					
	30 DAA		120 DAA		270 DAA	
	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR
Extract	0.034	102.6	0.005	78.3	0.010	87.1
Pydiflumetofen	0.024	77.8	0.004	37.3	0.008	59.7
SYN547891	0.004	12.0	<0.001	4.9	0.001	7.5
SYN545547	0.001	2.2	<0.001	2.4	<0.001	3.1
Unknown[a]	0.005	10.6	0.001	33.7	0.001	16.8
Others[b]	-	-	<LOD	<LOD	<LOD	<LOD
Unextracted[c]	0.001	3.2	0.001	10.7	<0.001	3.7
Total	0.035	105.8	0.006	89.0	0.010	90.8
Components	TRR [Pyrazole-5-^{14}C]					
	30 DAA		120 DAA		270 DAA	
	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR
Extract	0.025	90.2	0.022	93.3	0.013	88.9
Pydiflumetofen	0.016	59.1	0.006	22.5	0.003	21.9
SYN547891	0.004	13.3	0.001	3.0	0.001	4.6
SYN545547	0.001	3.5	0.001	2.3	ND	ND
Unknown[a]	0.004	14.3	0.013	63.0	0.009	62.4
Others[b]	-	-	0.001	2.5	<LOD	<LOD
Unextracted[c]	0.001	3.2	0.002	8.9	0.001	4.5
Total	0.026	93.4	0.024	102.2	0.014	93.4

ND= Not detected

[a] 30 DAA:

Phenyl label comprising at least 5 individual components none individually exceeding >2.9% TRR (>0.001 mg/kg eq)

Pyrazole label comprising at least 6 individual components none individually exceeding >3.5% TRR (>0.001 mg/kg eq)

120 DAA:

Phenyl label comprising at least 12 individual components none individually exceeding >5.3% TRR (>0.001 mg/kg eq)

Pyrazole label comprising at least 29 individual components none individually exceeding >9.2% TRR (>0.002 mg/kg eq)

270 DAA:

Phenyl label comprising at least 6 individual components none individually exceeding >4.1% TRR (>0.001 mg/kg eq)

Pyrazole label comprising at least 15 individual components none individually exceeding >7.0% TRR (>0.001 mg/kg eq)

[b] 120 DAA:

Extracted residues in 1 fraction from each radiolabelled experiment that were not analysed; levels of radioactivity were too low for analysis. No single fraction comprised >2.5% TRR (0.001 mg/kg eq) in either radiolabelled experiment.

270 DAA:

Extracted residues in 2 fractions from each radiolabelled experiment that were not analysed; levels of radioactivity were too low for analysis. No single fraction was in excess of the limit of detection (LOD)

[c] Radioactivity remaining in the PES after extraction with acetonitrile and aqueous acetonitrile. The nature of this residue was not characterised further

Table 19 Characterization and identification of the radioactive residues in rotational crop wheat hay treated with [^{14}C]-pydiflumetofen

Components	TRR [Phenyl-U-^{14}C]					
	30 DAA		120 DAA		270 DAA	
	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR
Extract	0.065	100.1	0.050	81.1	0.039	99.8
Pydiflumetofen	0.032	50.1	0.025	42.9	0.030	76.1
SYN547891	0.004	6.2	0.003	4.5	0.004	9.7
SYN545547	0.002	2.5	0.001	1.5	0.001	3.6
Unknown [a]	0.025	38.9	0.019	28.0	0.003	7.3
Others [b]	0.002	2.4	0.002	4.2	0.001	3.1
Unextracted [c]	0.005	8.1	0.009	14.8	0.002	5.3
Total	0.070	108.2	0.059	95.9	0.041	105.1
Components	TRR [Pyrazole-5-^{14}C]					
	30 DAA		120 DAA		270 DAA	
	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR
Extract	0.080	85.0	0.099	92.6	0.034	98.5
Pydiflumetofen	0.022	23.8	0.056	52.2	0.024	67.9
SYN547891	0.003	3.1	0.006	5.5	0.004	12.2
SYN545547	0.002	1.7	0.002	2.3	0.002	5.6
Unknown[a]	0.051	53.8	0.029	27.3	0.003	9.3
Others[b]	0.002	2.6	0.006	5.3	0.001	3.5
Unextracted[c]	0.009	9.9	0.017	15.5	0.002	6.5
Total	0.089	94.9	0.116	108.1	0.036	105.0

[a] 30 DAA:

Phenyl label comprising at least 21 individual components none individually exceeding >5.0% TRR (>0.003 mg/kg eq)

Pyrazole label comprising at least 30 individual components none individually exceeding >7.3% TRR (>0.007 mg/kg eq)

120 DAA:

Phenyl label comprising at least 20 individual components none individually exceeding >4.7% TRR (>0.003 mg/kg eq)

Pyrazole label comprising at least 17 individual components none individually exceeding >4.3% TRR (>0.005 mg/kg eq)

270 DAA:

Phenyl label comprising at least 2 individual components none individually exceeding >3.9% TRR (>0.002 mg/kg eq)

Pyrazole label comprising at least 2 individual components none individually exceeding >5.2% TRR (>0.002 mg/kg eq)

[b] Extracted residues in 1 fraction from each radiolabelled experiment that were not analysed; levels of radioactivity were too low for analysis.

30 DAA:

No single fraction comprised >2.6% TRR (>0.002 mg/kg eq) in either radiolabelled experiment.

120 DAA:

No single fraction comprised >5.3% TRR (>0.006 mg/kg eq) in either radiolabelled experiment.

270 DAA:

No single fraction comprised >3.5% TRR (>0.001 mg/kg eq) in either radiolabelled experiment.

[c] Radioactivity remaining in the PES after extraction with acetonitrile and aqueous acetonitrile. The nature of this residue was not characterised further

Table 20 Characterization and identification of the radioactive residues in rotational crop wheat straw treated with [^{14}C]-pydiflumetofen

Components	TRR [Phenyl-U-^{14}C]					
	30 DAA		120 DAA		270 DAA	
	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR
Extract	0.148	84.6	0.129	83.2	0.103	90.1
Pydiflumetofen	0.052	30.0	0.051	33.7	0.037	32.2
SYN547891	0.011	6.1	0.008	5.3	0.006	5.5
SYN545547	0.003	1.8	0.002	1.4	0.002	2.0
Unknown[a]	0.076	43.1	0.068	42.8	0.050	44.2
Others[b]	0.006	3.6	-	-	0.008	6.2
Unextracted[c]	0.021	12.2	0.022	14.1	0.004	3.2
Total	0.169	96.8	0.151	97.3	0.107	93.3
Components	TRR [Pyrazole-5-^{14}C]					
	30 DAA		120 DAA		270 DAA	
	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR
Extract	0.193	90.4	0.181	81.7	0.173	108.6
Pydiflumetofen	0.055	26.0	0.063	28.7	0.030	18.6
SYN547891	0.012	5.5	0.010	4.4	0.008	4.6
SYN545547	0.005	2.3	0.005	2.2	0.002	1.5
Unknown[a]	0.116	54.3	0.103	46.4	0.107	67.0
Others[b]	0.005	2.3	-	-	0.026	16.9
Unextracted[c]	0.024	11.3	0.031	14.1	0.005	2.9
Total	0.217	101.7	0.212	95.8	0.178	111.5

[a] 30 DAA:

Phenyl label comprising at least 22 individual components none individually exceeding >6.9% TRR (>0.012 mg/kg eq)

Pyrazole label comprising at least 38 individual components none individually exceeding >6.7% TRR (>0.014 mg/kg eq)

120 DAA:

Phenyl label comprising at least 27 discrete components, no single one of which >6.0% TRR (>0.009 mg/kg eq)

Pyrazole label comprising at least 33 discrete components, no single one of which >4.6% TRR (>0.010 mg/kg eq)

270 DAA:

Phenyl label comprising at least 17 discrete components, no single one of which >4.6% TRR (>0.005 mg/kg eq

Pyrazole label comprising at least 19 discrete components, no single one of which >5.8% TRR (>0.009 mg/kg eq)

[b] 30 DAA:

Extracted residues in 1 fraction from each radiolabelled experiment that were not analysed; levels of radioactivity were too low for analysis. No single fraction comprised >3.6% TRR (>0.006 mg/kg eq) in either radiolabelled experiment.

270 DAA:

Extracted residues in 4 fractions (phenyl label) and 3 fractions (pyrazole label) that were not analysed; levels of radioactivity were too low for analysis. No single fraction comprised >3.9% TRR (>0.006 mg/kg eq) in either radiolabelled experiment.

[c] Radioactivity remaining in the PES after extraction with acetonitrile and aqueous acetonitrile (30 DAA and 120 DAA) or with acetonitrile and aqueous acetonitrile, 1M HCl and clean fractionation (270 DAA). The nature of this residue was not characterised further

Table 21 Characterization and identification of the radioactive residues in rotational crop immature lettuce and mature turnip foliage treated with [^{14}C]-pydiflumetofen

Components	TRR [Phenyl-U-^{14}C]			
	30 DAA Immature lettuce		30 DAA Mature turnip foliage	
	mg/kg eq	% TRR	mg/kg eq	% TRR
Extract	0.010	88.3	0.009	91.3
Pydiflumetofen	0.009	69.3	0.008	77.2
SYN547891	0.001	11.6	<0.001	3.9
SYN545547	<0.001	4.0	ND	ND

Components	TRR [Phenyl-U-^{14}C]			
	30 DAA Immature lettuce		30 DAA Mature turnip foliage	
	mg/kg eq	% TRR	mg/kg eq	% TRR
Unknown[a]	<0.001	3.4	0.001	10.2
Others[b]	<LOD	<LOD	<LOD	<LOD
Unextracted[c]	0.002	14.5	0.001	6.5
Total	0.012	102.8	0.010	97.8
Components	TRR [Pyrazole-5-^{14}C]			
	30 DAA Immature lettuce		30 DAA Mature turnip foliage	
	mg/kg eq	% TRR	mg/kg eq	% TRR
Extract	0.018	99.2	0.011	105.4
Pydiflumetofen	0.015	76.7	0.005	44.4
SYN547891	0.001	6.8	0.001	4.8
SYN545547	<0.001	2.3	<0.001	3.8
Unknown[a]	<0.001	2.1	0.002	24.8
Others[b]	0.002	11.3	0.003	27.6
Unextracted[c]	0.003	13.9	0.001	10.4
Total	0.021	113.1	0.012	115.8

ND= Not detected

[a] Lettuce:

Phenyl label comprising at least 1 individual component not exceeding 3.4% TRR (0.001 mg/kg eq)

Pyrazole label comprising at least 1 individual component not exceeding 2.1% TRR (0.001 mg/kg eq)

Turnip:

Phenyl label comprising at least 1 individual component not exceeding 10.2% TRR (0.001 mg/kg eq)

Pyrazole label comprising at least 5 individual components none individually exceeding >10.2% TRR (>0.001 mg/kg eq)

[b] Lettuce:

Extracted residues in 1 fraction (pyrazole label) that was not analysed; levels of radioactivity were too low for analysis. No single fraction comprised >11.3% TRR (>0.002 mg/kg eq) in either radiolabelled experiment.

Turnip:

Extracted residues in 1 fraction (pyrazole label) that was not analysed; levels of radioactivity were too low for analysis. No single fraction comprised >27.6% TRR (>0.003 mg/kg eq) in either radiolabelled experiment.

[c] Radioactivity remaining in the PES after extraction with acetonitrile and aqueous acetonitrile. The nature of this residue was not characterised further

Parent pydiflumetofen represents the major portion of the residue taken up into rotated wheat, lettuce and turnip commodities up to a maximum of 77.8% TRR. Maximum absolute residues of parent were detected in 120 DAA wheat straw (pyrazole-0.063 md eq/kg). Residues declined between the 30 DAA and 270 DAA planting intervals across all crop commodities. Uptake of radioactive residues into grain and turnip tubers was low accounting for ≤0.008 md eq/kg. SYN545547 was detected in all extracted commodities at ≤0.005 md eq/kg. SYN547891 was detected in all extracted commodities at ≤0.012 md eq/kg.

Summary of metabolism in succeeding crops

The results show that the metabolism of pydiflumetofen in rotated crops was similar for all crop types. Comparison with primary crop metabolism studies shows that the pathway in rotational crops is consistent with that in primary crops, though the magnitude of residues in rotational crops is lower. The following metabolic pathways were speculated in the rotational crop metabolism studies available.

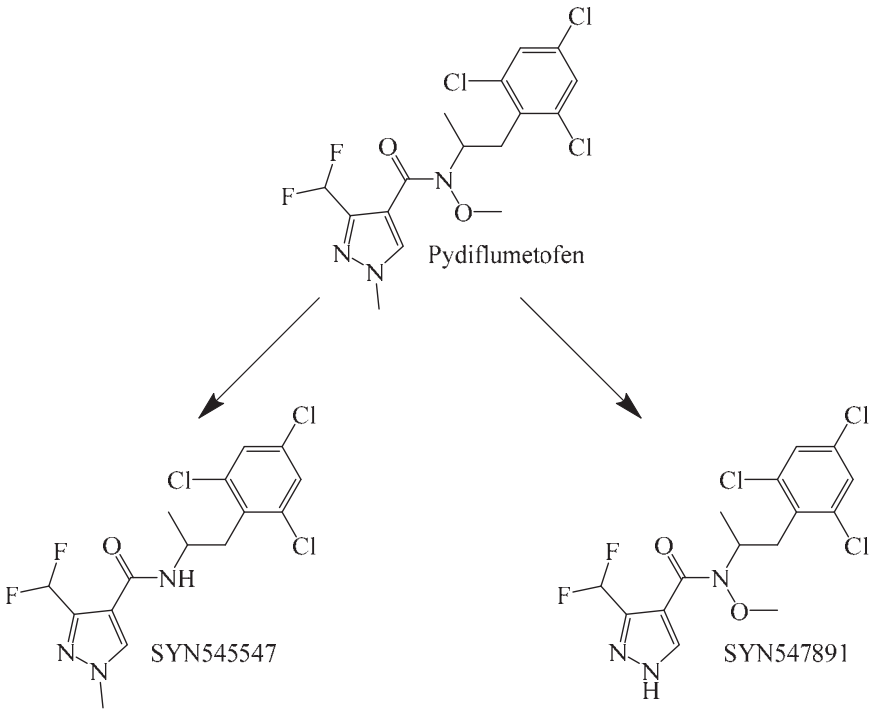


Figure 7 Metabolic pathway of pydiflumetofen in succeeding crops

Field rotational crop studies

Studies in the USA

Thirty-six rotational crop field trials were conducted in the USA to investigate the magnitude of residues in succeeding crops (Mäyer, 2015: TK0103846). Three trials were established for each of three rotational crop types (leafy vegetable, root crop, and small grain crop) at each of four rotational crop plantback intervals (30, 60, 90, and 150 days). Pydiflumetofen SC (a 200 g/L SC) formulation was applied to bare ground by broadcast spray applications. Two applications, each at the target rate of 0.20 kg ai/ha, were made at seven-day intervals for a total of 0.40 kg ai/ha.

Country	State	City	Soil type
USA	NC	Seven Springs	Loamy Sand
USA	CA	Porterville	Sandy Loam
USA	TX	Raymondville	Clay Loam

Radish (root crop), spinach or lettuce (leafy vegetable crop) and wheat (small grain crop) were planted at 30, 60, 90, and 150 days after the last (second) application. All trial locations established a non-treated control plot of the same crop planted at the same time as the treated plot. Crops were grown according to normal practices for the growing area.

Crop samples were collected at normal maturity. The radishes produced leaf (tops) and root samples, the spinach and lettuce produced leaf samples, and the wheat produced forage, hay, grain, and straw samples. Duplicate samples were collected from the treated plot and a single sample was collected from the non-treated control plot.

Samples were analyzed for pydiflumetofen residues by Method GRM061.03A using LC-MS/MS. The LOQ was 0.01 mg/kg for all commodities. The treated samples were analyzed for pydiflumetofen within 18 months after sample collection. Residues have been shown to be stable for at least 18 months in a range of crops.

The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70-120% and the relative standard deviations (RSD) of <20% indicating acceptable performance of the analytical method during the conduct of this study. Residues above the LOQ were not found in any of the non-treated control samples of any rotational crop matrix.

No pydiflumetofen residues greater than the LOQ were found in wheat grain, radish root, radish top, spinach, or lettuce at any plantback interval from any trial. The LOQ level residues of pydiflumetofen were found in wheat forage at only one trial for the 30-day plantback and at only one trial for 90-day plantback interval. No pydiflumetofen residues greater than the LOQ were found in wheat forage at the 60-day and 150-day plantback intervals. Low level residues of pydiflumetofen were found in wheat hay and straw at all intervals at all three trials. For both matrices, a peak in residues is apparent at the 90 day plantback interval with a subsequent decline in residues at the 150 day plantback interval.

Table 22 Pydiflumetofen residues in rotational crops

Crop	Variety	State	Residues, mg/kg			
			30-day PBI	60-day PBL	90-day PBI	150-day PBI
Spinach	Baker	NC	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
	Bloomsdale Long Standing	TX	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
Leaf lettuce	Waldmann's Green	CA	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
Radish tops	Cherriette	NC	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
	Crimson Giant	CA	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
	Champion	TX	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
Radish roots	Cherriette	NC	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
	Crimson Giant	CA	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
	Champion	TX	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
Wheat forage	Pioneer 26R20	NC	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
	Syngenta Uitra	CA	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
	TAM 304	TX	<0.01, 0.011	<0.01, <0.01	0.010, 0.012	<0.01, <0.01
Wheat hay	Pioneer 26R20	NC	0.016, 0.029	0.035, 0.041	0.011, 0.012	<0.01, 0.011
	Syngenta Uitra	CA	0.017, 0.018	0.014, 0.022	0.036, 0.039	0.013, 0.015
	TAM 304	TX	0.032, 0.033	0.027, 0.028	0.043, 0.047	0.028, 0.029
Wheat grain	Pioneer 26R20	NC	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
	Syngenta Uitra	CA	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
	TAM 304	TX	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
Wheat straw	Pioneer 26R20	NC	0.034, 0.053	0.017, 0.020	0.016, 0.018	0.015, 0.016
	Syngenta Uitra	CA	0.031, 0.031	0.057, 0.057	0.108, 0.119	0.047, 0.067
	TAM 304	TX	0.029, 0.037	0.028, 0.030	0.043, 0.044	0.022, 0.024

Studies in Northern Europe

This study consisted of two field crop rotation trials carried out in northern Europe (UK and Germany) during 2013 and 2014 to determine any residues of pydiflumetofen in crops grown in an area previously treated with pydiflumetofen. In each trial, pydiflumetofen was applied as an SC formulation containing 200 g ai/L. To all treated subplots, one application was made to bare soil at a rate of 500 g ai/ha (Lakaschus, 2016: S13-01022).

Three representative rotated crops were employed (spinach, short cycle carrots and spring barley) and were planted onto the treated and untreated subplots in spring 2014 at nominal intervals of 30, 60 and 365 days. The rotational crops were maintained according to normal agricultural practices. From the spinach crops, immature leaves were collected at BBCH 43 and mature leaves collected at BBCH 49 (from German trial only). From carrot crops, mature roots and tops were collected at BBCH 49. From the barley crops, immature whole plants were collected at BBCH 41 and mature grain and straw was collected at BBCH 89.

Specimens of spinach (immature and mature leaves), carrot (roots and tops with leaves) and barley (immature whole plant) were taken by hand. Specimens of barley (mature grain and straw) were threshed mechanically. All specimens were stored deep frozen at <-18 °C at the test site before being shipped on to the test facility for residue analysis, where the specimens were also stored at <- 18 °C in freezer. The crop samples were prepared by chopping in a bowl chopper with solid CO_2.

Specimens were stored frozen for a maximum period of 13 months from sampling to analysis. Extract solutions were stored for a maximum of 1 day in the refrigerator before analysis. The stability of the analytes in the specimen extracts was proven by the corresponding procedural recovery specimens, which were stored under the same conditions together with the sample extracts.

Country	State/Province	Town/Village	Soil type
UK	Derbyshire	Hemington	Clay Loam
Germany	Niedersachsen	Ohrensen	Loamy Sand

In the crop samples, pydiflumetofen residues were analysed using method GRM061.03A. Crop samples were extracted twice by homogenisation with acetonitrile/ultra-pure water (80/20, v/v) and then centrifuged at 15 °C for 5 min. The supernatants

were combined. For pydiflumetofen determination, aliquots were diluted with water followed by a solid phase extraction (SPE) clean-up. Pydiflumetofen was analysed by LC-MS/MS. The LOQ was 0.01 mg/kg.

The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70-120% and the relative standard deviations (RSD) of <20% indicating acceptable performance of the analytical method during the conduct of this study. Residues above the LOQ were not found in any of the non-treated control samples of any rotational crop matrix.

Residues of pydiflumetofen were all below the limit of quantification of the method (0.01 mg/kg) in all the following crops tested except for residues in spring barley straw samples in UK trial which were 0.02 mg/kg at 30 day plant back interval (PBI), 0.03 mg/kg at 60 day PBI and 0.01 mg/kg at 365 day PBI.

Table 23 Pydiflumetofen residues in rotational crops

Crop part	Variety	Trial site	Sampling (BBCH)	Residues, mg/kg		
				30 day PBI*	60 day PBI*	365 day PBI*
Spinach immature leaves	Renegade	UK	14-18	<0.01	<0.01	<0.01
	Molokai	Germany	43	<0.01	<0.01	<0.01
Spinach leaves	Molokai	Germany	49	<0.01	<0.01	<0.01
Carrot top	Chantenay	UK	49	<0.01	<0.01	<0.01
	Laguna F1	Germany	49	<0.01	<0.01	<0.01
Carrot root	Chantenay	UK	49	<0.01	<0.01	<0.01
	Laguna F1	Germany	49	<0.01	<0.01	<0.01
Barley whole plant	Optic	UK	39-42	<0.01	<0.01	<0.01
	Simba	Germany	41	<0.01	<0.01	<0.01
Barley grain	Optic	UK	89	<0.01	<0.01	<0.01
	Simba	Germany	89	<0.01	<0.01	<0.01
Barley straw	Optic	UK	89	0.02	0.03	0.01
	Simba	Germany	89	<0.01	<0.01	<0.01

* Actual PBI

UK: 63, 94, 400 days (spinach and carrot), 28, 59, 365 days (barley)

Germany: 29, 58, 383 days

Study in Southern Europe

This study consisted of two field crop rotation trials carried out in southern Europe (southern France and Italy) during 2013, 2014 and 2015 to determine any residues of pydiflumetofen in crops grown in an area previously treated with pydiflumetofen. In each trial, pydiflumetofen was applied as an SC formulation containing 200 g ai/L. To all treated subplots, one application was made to bare soil at a rate of 500 g ai/ha (Lakaschus, 2016: S13-01023).

Three representative rotated crops were employed (spinach, carrot and spring barley) and were planted onto the treated subplots in spring 2014 at nominal intervals of 30, 60 and 365 days. From the spinach crops, immature leaves were collected at BBCH 43 and mature leaves collected at BBCH 49. From the carrot crops, mature roots and tops were collected at BBCH 49. From the barley crops, immature whole plants were collected at BBCH 41 and mature grain and straw was collected at BBCH 89.

All specimens were stored deep frozen at <-18 °C at the test site before being shipped to the test facility for residue analysis, where the specimens were also stored at <- 18 °C in a freezer. The crop samples were prepared by chopping in a bowl chopper with solid CO_2.

Specimens were stored frozen for a maximum period of 16 months from sampling to analysis. Extract solutions were stored for a maximum of 8 days in the refrigerator before analysis. The stability of the analytes in the specimen extracts was proven by the corresponding procedural recovery specimens, which were stored under the same conditions together with the sample extracts.

Country	State/Province	Town/Village	Soil type
France	Tarn et Garonne	Meauzac	Loam
Italy	Bologna	San Marino di Bentivolio	Sandy Loam

In the crop samples, pydiflumetofen residues were analysed using method GRM061.03A. Crop samples were extracted twice by homogenisation with acetonitrile/ultra-pure water (80/20, v/v) and then centrifuged at 15 °C for 5 min. The supernatants were combined. For pydiflumetofen determination, aliquots were diluted with water followed by a SPE clean-up. Pydiflumetofen was analysed by LC-MS/MS. The LOQ was 0.01 mg/kg.

The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70-120% and the relative standard deviations (RSD) of <20% indicating acceptable performance of the analytical method during the conduct of this study. Residues above the LOQ were not found in any of the non-treated control samples of any rotational crop matrix.

Residues of pydiflumetofen found in spinach immature leave and mature leave specimens at the 30, 60 and 365 day PBIs (nominal) were all below the LOQ, except for immature leave specimens taken at BBCH 43 in French trial, where residues of 0.01 mg/kg at the 30 day PBI and 0.02 mg/kg at the 60 day PBI were found.

Residues of pydiflumetofen found in carrot (top) specimens from the 30 and 365 day PBI (nominal) were below the LOQ in both trials. Carrot (top) specimens from the 60 day PBI (nominal) had a residue of 0.01 mg/kg in French trial and <0.01 mg/kg in Italian trial. Residues of pydiflumetofen found in carrot (root) specimens from the 30 day PBI (nominal) were 0.02 mg/kg in French trial and <0.01 mg/kg in Italian trial. Carrot (root) specimens from the 60 day PBI (nominal) had a residue of 0.02 mg/kg in both trials. At the 365 day PBI (nominal) the residues in carrot (root) specimens taken in both trials were below the LOQ.

Residues of pydiflumetofen found in spring barley whole plant, grain and straw specimens at the 30, 60 and 365 day PBIs (nominal) were all below the LOQ in all the following crops tested, except for residues in spring barley (straw) specimens from French and Italian trial which were 0.06 mg/kg and 0.02 mg/kg at the 30 day PBI, 0.09 mg/kg and 0.02 mg/kg at the 60 day PBI and 0.01 mg/kg at the 365 day PBI, respectively. Furthermore, a residue of 0.02 mg/kg was found in the spring barley (whole plant) specimen from the 30 day PBI (nominal) from Italian trial.

There were no recorded residues above the LOQ found in the untreated specimens analysed.

Table 24 Pydiflumetofen residues in rotational crops

Crop part	Variety	Trial site	Sampling (BBCH)	Residues, mg/kg		
				30 day PBI*	60 day PBI*	365 day PBI*
Spinach immature leaves	Falcon	France	43	0.01	0.02	<0.01
	Carmen	Italy	43	<0.01	<0.01	<0.01
Spinach leaves	Falcon	France	49	<0.01	<0.01	<0.01
	Carmen	Italy	49	<0.01	<0.01	<0.01
Carrot top	Mondibel F1	France	49	<0.01	0.01	<0.01
	Bolero	Italy	49	<0.01	<0.01	<0.01
Carrot root	Mondibel F1	France	49	0.02	0.02	<0.01
	Bolero	Italy	49	<0.01	0.02	<0.01
Barley whole plant	Explorer	France	41	<0.01	<0.01	<0.01
	Tunika	Italy	41	0.02	<0.01	<0.01
Barley grain	Explorer	France	89	<0.01	<0.01	<0.01
	Tunika	Italy	89	<0.01	<0.01	<0.01
Barley straw	Explorer	France	89	0.06	0.09	0.01
	Tunika	Italy	89	0.02	0.02	0.01

* Actual PBI

France: 27, 66, 364 days

Italy: 27, 55, 338 days

Environmental fate in water and soil

The Meeting received information on hydrolytic degradation, soil photolysis and soil dissipation study. Because pydiflumetofen is intend for use as foliar treatment, hydrolytic degradation study relevant to the current evaluations were reported below (FAO Manual Third edition, 2016).

Hydrolysis

The hydrolysis of [^{14}C]-pydiflumetofen at 0.6 μg/mL was studied in the dark in sterile aqueous buffered solutions containing acetonitrile (0.3%) at pH 4, 7 and 9 at 50 °C for 5 days (Lewis, 2015: 3200053). Duplicate samples from each pH were analysed at zero time and after 3 and 5 days incubation at 50 °C. Acetonitrile (300 μL) was added to the aqueous solutions prior to analysis by LSC and HPLC with radio-detection. Selected samples were analysed by TLC.

The temperatures remained constant throughout the incubation period (50 ± 0.5 °C) and there was no significant variation in the pH values of the buffered solutions. The samples also remained sterile throughout the study.

The mean radioactivity balance was 98.7% (range 95.4–100.3%), indicating that no losses from the test system had taken place.

Table 25 Recovery and quantification of ^{14}C-pydiflumetofen in buffer solutions incubated at 50 °C

Fractions		% of applied radioactivity								
		pH 4			pH 7			pH 9		
		0 days	3 days	5 days	0 days	3 days	5 days	0 days	3 days	5 days
Pydiflumetofen	A	96.3	94.9	96.9	96.2	97.4	93.6	92.0	94.4	95.8
	B	95.1	97.8	94.8	96.5	96.3	95.6	94.9	96.8	96.0
	Mean	95.7	96.3	95.9	96.4	96.9	94.6	93.5	95.6	95.9
Others*	A	2.9	4.4	2.5	3.4	2.7	4.1	3.4	4.0	3.5
	B	4.2	2.5	3.1	2.2	2.2	2.8	2.7	2.8	2.2
	Mean	3.6	3.5	2.8	2.8	2.5	3.5	3.0	3.4	2.9
Total	A	99.2	99.3	99.4	99.6	100.1	97.7	95.4	98.4	99.3
	B	99.3	100.3	97.9	98.7	98.5	98.4	97.6	99.6	98.2
	Mean	99.3	99.8	98.7	99.2	99.3	98.1	96.5	99.0	98.8

* Includes small unknown components on chromatograms and any unresolved background. No single component exceeded 1.71%.

Pydiflumetofen was found to be hydrolytically stable at pH 4, 7 and 9 for up to 5 days at 50 ºC. At five days, pydiflumetofen accounted for ≥92.0% of applied radioactivity (AR) in all samples analysed.

Soil photolysis

The photolysis of ^{14}C-phenyl ring labelled and ^{14}C-pyrazole ring labelled pydiflumetofen was investigated on both dry and moist soil surfaces (Hurst, 2014: 3200128). Pydiflumetofen was applied, at rates equivalent to *ca* 0.250 kg ai/ha, to thin layers ((*ca* 1 mm for dry soil, *ca* 3 mm for moist soil) of 18 Acres soil (sandy clay loam, UK) in individual photolysis vessels. The treated soils were continuously irradiated using light from a xenon arc lamp. The emitted light was filtered to give a spectral distribution close to that of natural sunlight at a light intensity in the range of 46–52 W/m^2. The samples were maintained at 20 ± 2 ºC and were irradiated for periods up to the equivalent of *ca* 30 days summer sunlight at latitudes 30-50ºN.

In each test, duplicate samples were taken for analysis at up to six intervals during irradiation. Dark control samples were also prepared and maintained at *ca* 20 ºC. Dark control samples were taken for analysis at intervals equivalent to or exceeding that of the irradiation test. Any volatile radioactivity was continuously flushed from the vessels and collected in liquid traps. A mass balance was determined for each sample. Samples were analysed directly by reverse phase HPLC. Selected samples were analysed by chiral HPLC to check for any isomer change during incubation and selected samples were analysed by TLC to confirm the presence of pydiflumetofen and its metabolite SYN545547, which had been identified by HPLC. Selected samples were analysed by LC-MS to confirm the presence of pydiflumetofen.

The mass balance from the irradiated samples was in the range of 91.0–99.4% applied radioactivity (AR) and from the dark controls was 87.0–100.6% AR, mean of duplicate samples quoted.

The metabolite SYN545547 was observed in the majority of samples, although it was also characterised at levels of 1.2–1.3% in the applications solutions. Its maximum level was 2.3% AR, which was noted in the irradiated dry soil taken 12 DAT (with the pyrazole radiolabel). In addition, a number of discrete unknown degradates were also observed throughout all of the samples, none of which exceeded 4% AR.

Carbon dioxide was a product of photolysis in the dry irradiated soils reaching a maximum of 4.2% AR by the end of the irradiation period. The dry dark control samples showed mineralisation to be below the limit of detection in all units. In the moist soils (both irradiated and dark control test units) little degradation to carbon dioxide was observed, with mean levels not exceeding 0.4% of the applied dose.

Unextracted residues remained similar in the moist soils during the incubation, reaching a maximum of 3.2% AR by the end of the study. The dry soils contained lower levels of unextracted residues, with a maximum of 1.7% reached at the end of the study.

Based on confidence intervals of <0, it can be assumed that pydiflumetofen did not degrade in the dark controls (neither in dry or moist conditions). Irradiated moist soils treated with pyrazole labelled pydiflumetofen also showed negative confidence intervals thus this data is not discussed further. Once this data was combined with units treated with phenyl-labelled pydiflumetofen, results were well described.

Table 26 DT_{50} and DT_{90} values for pydiflumetofen under irradiated conditions

		Dry soil		Moist soil	
		DT_{50} (days)	DT_{90} (days)	DT_{50} (days)	DT_{90} (days)
Pyrazole label	Irradiated	89	297	387	1284
	Dark	236	785	747	2481
	Summer Sunlight 30-50 ºN	171	568	713	2370
Phenyl label	Irradiated	65	215	134	444
	Dark	>1000	>1000	245	813
	Summer Sunlight 30-50 ºN	135	448	243	808
Pyrazole and phenyl combined	Irradiated	77	254	197	654
	Dark	>1000	>1000	369	1227
	Summer Sunlight 30-50 ºN	154	507	361	1198

Results from the dark controls were not calculated accurately by the kinetics programme CAKE due to large fluctuations in the recovery of applied radioactivity throughout the duration of the study.

The DT_{50} and DT_{90} values were calculated using non-linear regression and first-order kinetics (SFO). Degradation was extrapolated well beyond the 16 day study period (equivalent to 30 summer days), with DT_{50} reached in approximately one third of the time in irradiated dry soil than in irradiated moist soil. Enantiomer ratios of pydiflumetofen in both the pyrazole label and the phenyl label stock solutions used to treat the samples and in units removed at the final sampling interval did not change significantly.

Degradation of pydiflumetofen was relatively slow in samples treated with [pyrazole-5-^{14}C]-pydiflumetofen or [phenyl-U-^{14}C]-pydiflumetofen, regardless of experimental conditions. Half-lives were shortest in dry irradiated soils (77 experimental days), with longer half-lives calculated in moist irradiated soils (197 experimental days). Both results were based on data combined from both labels. Consequently, degradation in the dark controls was negligible over the study period with data unable to be accurately determined due to negative confidence intervals.

Table 27 Distribution and recovery of radioactivity (dry soil photolysis)

		Sampling times (actual experimental days after treatment)						
		0	3	5	8	10	12	16
Pyrazole-^{14}C Irradiated	Extract	97.1	95.5	98.6	97.5	94.6	94.8	89.5
	Unextracted	ND	0.5	0.7	1.1	1.0	1.3	1.4
	CO_2*	NA	0.1	0.1	0.3	0.7	0.5	0.2
	Total	97.1	96.1	99.4	98.9	96.2	96.6	91.0
Pyrazole-^{14}C Dark	Extract	97.1	95.2	89.2	92.5	96.5	87.8	91.8
	Unextracted	ND	0.2	0.2	0.2	0.2	0.2	0.4
	CO_2*	ND	ND	ND	ND	ND	ND	ND
	Total	97.1	95.4	89.4	92.7	96.7	88.0	92.2
Phenyl-^{14}C Irradiated	Extract	97.1	96.2	96.8	90.4	89.9	94.6	90.0
	Unextracted	0.2	0.8	0.9	1.3	1.6	1.4	1.7
	CO_2*	NA	0.3	0.6	2.3	4.2	2.2	1.5
	Total	97.3	97.3	98.3	93.9	95.6	98.1	93.1
Phenyl-^{14}C Dark	Extract	97.1	86.6	87.1	89.0	97.8	98.3	100.1
	Unextracted	0.2	0.4	0.6	0.5	0.5	0.5	0.6
	CO_2*	NA	ND	ND	ND	ND	ND	ND
	Total	97.3	87.0	87.6	89.5	98.3	98.8	100.6

*The nature of the volatiles was assumed to be CO_2 but was not determined due to low levels of mineralization.

ND: Not detected, or <0.1% AR

NA: Not applicable

Table 28 Distribution and recovery of radioactivity (moist soil photolysis)

		Sampling times (actual experimental days after treatment)						
		0	3	5	8	10	12	16
Pyrazole-^{14}C Irradiated	Extract	98.2	95.2	96.0	94.8	95.8	96.0	92.5
	Unextracted	0.1	1.4	1.7	1.8	2.5	2.2	2.6
	CO_2*	NA	ND	0.1	0.1	0.2	0.2	0.2
	Total	98.3	96.6	97.8	96.6	98.4	98.3	95.1

		Sampling times (actual experimental days after treatment)						
		0	3	5	8	10	12	16
Pyrazole-^{14}C Dark	Extract	98.2	93.2	95.8	96.7	94.3	93.4	94.8
	Unextracted	0.1	1.2	1.4	1.8	1.8	1.9	2.0
	CO_2*	NA	ND	ND	ND	ND	ND	ND
	Total	98.3	94.3	97.2	98.4	96.1	95.3	96.8
Phenyl-^{14}C Irradiated	Extract	98.0	97.6	94.5	95.0	94.4	89.6	93.0
	Unextracted	0.2	1.6	1.9	2.3	1.3	3.0	3.2
	CO_2*	NA	0.2	0.2	0.4	0.4	0.4	0.4
	Total	98.2	99.4	96.6	97.6	96.1	92.9	96.6
Phenyl-^{14}C Dark	Extract	98.0	96.4	97.4	91.1	94.2	94.3	93.5
	Unextracted	0.2	1.5	1.7	2.0	1.8	2.1	2.1
	CO_2*	NA	0.1	0.1	0.1	0.2	0.2	0.2
	Total	98.2	97.9	99.2	93.1	96.1	96.6	95.8

*The nature of the volatiles was assumed to be CO_2 but was not determined due to low levels of mineralization.

ND: Not detected, or <0.1% AR

NA: Not applicable

SYN545547 was observed in the majority of samples, although it was also characterised at levels of 1.2–1.3% in the applications solutions. No single metabolite was observed at >3.5% AR. Ultimately, pydiflumetofen mineralised to CO_2 (<5% AR) and negligible levels of unextracted radioactivity (up to 2% and 4% AR for the moist and dry samples respectively, for both radiolabels).

The mass balance ranged from 93.1–99.2% AR for the moist soil samples and from 87.0-100.6% AR for the dry soil samples. All irradiated samples were ≥91.0% AR. The enantiomeric composition of pydiflumetofen did not change significantly through the duration of the study.

Table 29 Quantification of pydiflumetofen and its photodegradates (dry soil photolysis)

		Sampling times (actual experimental days after treatment)						
		0	3	5	8	10	12	16
Pyrazole-^{14}C Irradiated	Pydiflumetofen	93.8	90.7	94.3	91.5	87.6	86.1	82.6
	SYN545547	1.4	1.5	1.9	1.9	1.5	2.3	1.6
	Unidentified regions	1.1	2.2	1.4	2.7	4.1	6.2	4.3
	Unresolved background	0.8	1.1	0.9	1.5	1.3	0.2	0.9
	CO_2*	NA	0.1	0.1	0.3	0.7	0.5	0.2
Pyrazole-^{14}C Dark	Pydiflumetofen	93.8	91.9	85.9	89.3	93.1	85.0	89.3
	SYN545547	1.4	1.2	1.2	1.3	1.1	1.0	0.6
	Unidentified regions	1.1	1.2	0.9	1.2	1.5	1.4	1.1
	Unresolved background	0.8	0.9	1.1	0.7	0.8	0.4	0.8
	CO_2*	NA	ND	ND	ND	ND	ND	ND
Phenyl-^{14}C Irradiated	Pydiflumetofen	94.0	93.2	93.0	84.6	82.0	88.8	80.3
	SYN545547	1.5	1.3	1.0	1.4	1.7	1.9	1.3
	Unidentified regions	0.5	1.0	1.6	3.1	4.7	3.0	7.4
	Unresolved background	1.1	0.6	1.3	1.3	1.5	0.9	1.0
	CO_2*	NA	0.3	0.6	2.3	4.2	2.2	1.5
Phenyl-^{14}C Dark	Pydiflumetofen	94.0	84.6	84.9	86.9	95.6	95.6	97.9
	SYN545547	1.5	0.8	1.0	0.5	1.1	1.0	0.9
	Unidentified regions	0.5	0.7	0.4	1.1	0.2	1.2	0.4
	Unresolved background	1.1	0.6	0.7	0.5	0.9	0.5	0.8
	CO_2*	NA	ND	ND	ND	ND	ND	ND

*The nature of the volatiles was assumed to be CO_2 but was not determined due to low levels of mineralization.

ND: Not detected, or <0.1% AR

NA: Not applicable

Table 30 Quantification of pydiflumetofen and its photodegradates (moist soil photolysis)

		Sampling times (actual experimental days after treatment)						
		0	3	5	8	10	12	16
Pyrazole-^{14}C Irradiated	Pydiflumetofen	94.2	92.2	93.3	92.1	91.7	93.8	89.3
	SYN545547	0.9	1.7	1.5	0.8	1.7	1.4	1.2
	Unidentified regions	1.7	0.9	0.4	0.6	1.6	0.7	1.3
	Unresolved background	1.3	0.4	0.8	1.3	0.9	0.1	0.7
	CO_2*	NA	ND	0.1	0.1	0.2	0.2	0.2
Pyrazole-^{14}C Dark	Pydiflumetofen	94.2	90.0	93.4	92.7	91.4	91.0	92.3
	SYN545547	0.9	1.0	1.1	0.8	1.0	0.4	1.1
	Unidentified regions	1.7	1.3	1.2	2.3	0.9	1.1	0.5
	Unresolved background	1.3	0.9	0.2	0.9	1.1	0.9	0.9
	CO_2*	NA	ND	ND	ND	ND	ND	ND
Phenyl-^{14}C Irradiated	Pydiflumetofen	95.7	95.5	91.9	92.0	91.7	86.8	90.2
	SYN545547	1.3	1.2	1.1	1.6	1.2	1.2	1.3
	Unidentified regions	0.4	ND	0.5	0.5	0.4	0.8	0.9
	Unresolved background	0.6	0.9	1.0	0.8	1.0	0.8	0.6
	CO_2*	NA	0.2	0.2	0.4	0.4	0.4	0.4
Phenyl-^{14}C Dark	Pydiflumetofen	95.7	95.5	94.3	89.7	92.1	92.9	91.8
	SYN545547	1.3	0.7	1.8	1.1	1.4	0.7	0.4
	Unidentified regions	0.4	ND	ND	ND	ND	0.5	0.4
	Unresolved background	0.6	0.2	1.3	0.3	0.7	0.2	0.9
	CO_2*	NA	0.1	0.1	0.1	0.2	0.2	0.2

*The nature of the volatiles was assumed to be CO_2 but was not determined due to low levels of mineralization.

ND: Not detected, or <0.1% AR

NA: Not applicable

Degradation in soil

Study 1

The rate of degradation of pydiflumetofen has been studied in the laboratory in five soils (Hurst, 2015: SYN/48/01-KIN01). The original data from this study was used to calculate the rate of degradation of pydiflumetofen in soil, following the guidance in FOCUS Kinetics (2006, 2011) and using the analysis software CAKE v3.1 (2015).

Data were generated according to the data handling recommendations made in the FOCUS guidance for degradation kinetics (FOCUS, 2006, 2011) and kinetic models were fitted to the levels of readily extractable pydiflumetofen in each soil. True replicates were included individually in the optimisations. Levels of pydiflumetofen remained above the LOD throughout the study in all soils. Correction of values below the LOD was, therefore, not required for these data. Initial pydiflumetofen levels in the model input data were set to the total extractable radioactivity measured in the time zero samples.

Confidence in the resulting parameters has been assessed visually and from the confidence intervals for the α and β parameters of the first order multi compartment (FOMC) model or probability values for a t-test of the rate parameters for the single first order (SFO), dual first order in parallel (DFOP) and hockey stick (HS) models. Where the parameters for a particular model are not significantly different from zero at the 95th or 90th significance level, it has been concluded that the model is not appropriate to represent the degradation behaviour of pydiflumetofen in that soil. The χ^2 error % parameter has been used to determine goodness of fit and where two models are an appropriate to fit the data, the choice of best fit has been based on the lowest value of this parameter.

The degradation study was conducted at 20 °C and at a soil moisture content equal to pF2. Normalisation of endpoints was, therefore, not required.

Table 31 Detailed trigger DT_{50} and DT_{90} values for pydiflumetofen

Soil name	Soil texture (USDA)	pH	Study endpoints (days)		Kinetic model
			DT_{50}	DT_{90}	
Gartenacker	Loam	7.4	398	1320	SFO
18 Acres	Sandy clay loam	6.5	2380	7640	DFOP
Sarpy	Silty clay loam	6.8	567	2970	DFOP
East Anglia	Sandy loam	7.8	1300	4870	DFOP

Soil name	Soil texture (USDA)	pH	Study endpoints (days)		Kinetic model
			DT_{50}	DT_{90}	
Capay	Clay loam	8.4	410	2540	DFOP
Geometric mean			779	3264	

Table 32 Detailed modeling DT_{50} values for pydiflumetofen

Soil name	Soil texture (USDA)	pH	Measured DT_{50} at 20 °C and pF2 (days)	Kinetic model
Gartenacker	Loam	7.4	398	SFO
18 Acres	Sandy clay loam	6.5	1690	SFO
Sarpy	Silty clay loam	6.8	1036*	DFOP
East Anglia	Sandy loam	7.8	1534*	DFOP
Capay	Clay loam	8.4	917*	DFOP
Geometric mean			940	

* DT_{50} calculated from slow phase rate constant (ln (2)/k2)

Study 2

Soil dissipation field trials were carried out in France, Germany, Italy, Spain and the UK during 2013–2015 to investigate the field dissipation behaviour of pydiflumetofen (Finger, 2015: S13-02236, 02237, 02238, 02239, 02240 and 02241). A single application of pydiflumetofen (a 200 g/L SC formulation) was applied at a target rate of 1.0 L product/ha (equivalent to 0.204 kg ai/ha based on analysed content) as a broadcast application to the bare soil surface. Immediately after application but before the 0 days after application (DAA) soil residue sampling, the treated plot was covered with approximately 3–10 mm of untreated sand. No control plot was established.

A sample for soil characterisation (12 cores, 0–100 cm depth) was taken 0–21 days before application (DBA) from the plot corners within the plot boundaries. Soil samples for taxonomic classification and bulk density analysis (0–100 cm cores) were taken from the plot corners in the plot boundaries. Soil samples for water holding capacity analysis were taken from the plot boundaries. A bulk soil sample for biomass analysis (0–20 cm) was taken from the plot boundary using a spade.

Soil samples were analysed for residues of pydiflumetofen using residue analytical method GRM061.04A using LC-MS/MS, with a LOQ of 0.5 µg/kg. For the 0 and 539 DAA (0–10 cm depth) soil samples, the isomer separation of pydiflumetofen was conducted and the isomer ratio determined.

Pydiflumetofen was found to remain mostly in the top soil layer (0–10 cm) with initial concentrations in the range of 116–189 g ai/ha, gradually declining to a range of 46–118 g ai/ha at the end of the study period. At lower layers, levels of parent were in the range of 17 g ai/ha to not detected at 10–20 cm, dropping to 13 g ai/ha to not detected at 20–30 cm. At one site (Germany) no parent was detected below 10 cm depth.

Table 33 Residues of pydiflumetofen in the soil layers (0–10 cm)

Sampling interval (DAA)	Pydiflumetofen residue (g ai/ha)					
	Germany	Italy	North France	South France	Spain	UK
Control	ND	ND	ND	ND	ND	ND
0	145	153	116	217	189	125
3	157	106	120	179	231	132
7	131	148	157	182	210	148
13-15	142	155	133	93	65	111
27-29	117	126	123	111	81	105
58-62	105	132	132	95	110	132
118-121	109	93	137	100	106	108
172-182	131	119	144	85	146	154
358-372	111	112	142	84	144	132
533-546	144	116	91	63	80	119
714-721	112	69	118	46	91	85

ND: not detected (residues are below the LOD, 0.15 µg/kg wet soil).

The enantiomer elution order was confirmed to be (S)-pydiflumetofen (SYN546968) before (R)-pydiflumetofen (SYN546969). No significant change in the enantiomer ratio between the two time points was observed for all sites, with initial ratios in the range of 0.95–1.03 and final ratios in the range of 0.97–1.08.

Table 34 Enantiomer ratio of 0–10 cm depth soil samples

	Sampling interval	Enantiomer ratio (calculated as S-isomer/R-isomer)
Germany	0 DAA	0.99, 0.96, 1.01
	533 DAA	1.00, 0.97, 1.00
Italy	0 DAA	0.97, 0.97, 1.03
	542 DAA	0.98, 1.00, 1.00
Northern France	0 DAA	0.99, 0.97, 0.98
	546 DAA	0.97, 0.98, 1.01
Southern France	0 DAA	0.99, 0.99, 1.02
	533 DAA	1.05, 1.08, 1.03
Spain	0 DAA	0.99, 0.97, 0.95
	538 DAA	1.05, 1.05, 1.05
UK	0 DAA	0.96, 1.02, 1.01
	539 DAA	1.00, 1.01, 0.99

RESIDUE ANALYSIS

Analytical methods

Descriptions of analytical methods together with validation data for residues of pydiflumetofen in plant and animal matrices were submitted to the Meeting. The methods rely on an initial extraction, usually with acetonitrile/water. After column clean-up, pydiflumetofen and its metabolites are prepared for LC analysis. Their residues can be measured by mass spectrometric detector (MS/MS), to an LOQ of 0.01 mg/kg. Since the methods use standard extraction solvents and standard detection techniques, they have the potential to be incorporated into existing multi-residue methods.

Detailed descriptions of all these analytical methods are presented below.

Plant matrices

High water content and dry crop categories			
Analyte:	Pydiflumetofen (m/z 426→193 for quantification, 428→195 for confirmation)	LC-MS/MS	GRM061.03A
LOQ:	0.01 mg/kg		
Description	The representative sample is extracted twice with 100 mL of acetonitrile/water (80:20 v/v, HPLC grade) using Polytron homogenizer at high speed. The extracts are combined after removal of solids by centrifugation. An aliquot of sample extract is diluted and processed via SPE procedures prior to final reconstitution and subjected to residue determination. Alternatively, an aliquot of sample extract is filtered through a PTFE syringe filter or treated with BONDESIL C18 via dispersive SPE procedure and diluted prior to final analysis if instrument sensitivity allows with minimum matrix interferences. Residue determination is achieved by LC-MS/MS using electrospray ionization techniques.		
Lettuce, Oilseed rape, Dried broad beans, Wheat grain, Orange fruit and Coffee bean			
Analyte:	Pydiflumetofen (m/z 426→193 for quantification, 426→171 for confirmation)	LC-MS/MS	QuEChERS
LOQ:	0.01 mg/kg		
Description	The residues of pydiflumetofen are extracted by agitation with acetonitrile in presence of water. After addition of a buffer salt mixture, containing magnesium sulphate, sodium chloride and sodium citrate, the extract is shaken. After centrifugation, an aliquot of the acetonitrile phase is cleaned by dispersive solid phase extraction, using primary secondary amine (PSA). After centrifugation, an aliquot is diluted in ultra-pure water prior to quantification by LC-MS/MS.		

The capability of the FDA multiresidue Protocols A, C, D, E and F were evaluated (Perez, 2014: ADPEN-2K14-TK0163712-001). The natural fluorescence of pydiflumetofen was extremely weak and was not sufficient for practical work. Protocol A was inappropriate for the analysis of pydiflumetofen. Pydiflumetofen produced broad tailing double-peak signal with possible thermal degradation under Protocol C, Module DG1 and Module DG10. Protocol D was inappropriate for the analysis of pydiflumetofen due to inadequate extraction routine. The C1 cleanup method produced 100% recovery, while C5 produced almost no recovery. Protocols E and F were inappropriate for the analysis of pydiflumetofen because the recovery yields from CI and C2 cleanup tests were zero.

Validation data for methods on plant matrices are summarized in Table 35.

Table 35 Summary of Recovery Data for pydiflumetofen fortified into plant matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Apple (MV)	426→193	0.01	5	79 – 108	91	12	GRM061.03A
	Quantification	0.20	5	90 – 96	93	2.6	
	428→195	0.01	5	82 – 97	92	6.6	TK013794
	Confirmation	0.20	5	93 – 96	93	2.3	Hung, 2015
Lettuce (MV)	426→193	0.01	5	73 – 97	82	11	
	Quantification	10	5	88 – 101	93	5.9	
	428→195	0.01	5	78 – 92	83	7.1	
	Confirmation	10	5	84 – 96	90	6.0	
Tomato (MV)	426→193	0.01	5	78 – 111	96	12	
	Quantification	1.4	5	90 – 96	93	2.8	
	428→195	0.01	5	79 – 116	98	15	
	Confirmation	1.4	5	97 – 103	99	2.7	
Cabbage (MV)	426→193	0.01	5	91 – 103	94	5.2	
	Quantification	10	5	102 - 108	105	2.7	
	428→195	0.01	5	82 – 102	93	8.3	
	Confirmation	10	5	96 – 99	97	1.2	
Fresh Peas (MV)	426→193	0.01	4	95 – 101	98	3.3	
	Quantification	0.20	5	90 – 119	108	10	
	428→195	0.01	4	81 – 91	86	6.2	
	Confirmation	0.20	5	93 – 119	110	9.4	
Dry Beans (MV)	426→193	0.01	5	87 – 107	96	8.1	
	Quantification	0.20	5	65 – 87	76	13	
	428→195	0.01	5	95 – 104	99	4.2	
	Confirmation	0.20	5	70 – 88	80	9.6	
Wheat Grain (MV)	426→193	0.01	5	90 – 108	97	7.6	
	Quantification	0.20	5	92 – 101	96	4.2	
	428→195	0.01	5	82 – 103	90	9.7	
	Confirmation	0.20	5	89 – 98	93	4.2	
Wheat Straw (MV)	426→193	0.01	5	100 – 123	110	8.2	
	Quantification	10	5	82 – 113	102	14	
	428→195	0.01	5	88 – 117	105	10	
	Confirmation	10	5	81 – 121	106	16	
Wheat Forage (MV)	426→193	0.01	5	93 – 111	101	7.0	
	Quantification	10	5	92 – 118	108	11	
	428→195	0.1	5	88 – 120	103	12	
	Confirmation	10	5	93 – 120	110	11	
Potato (MV)	426→193	0.01	5	81 – 101	92	8.2	
	Quantification	10	5	89 – 103	97	5.3	
	428→195	0.01	5	73 – 109	90	16	
	Confirmation	10	5	93 – 108	100	6.0	
Rape Seed (MV)	426→193	0.01	5	82 – 110	92	12	
	Quantification	0.20	5	92 – 100	95	4.6	
	428→195	0.01	5	86 – 99	93	5.3	
	Confirmation	0.20	5	86 – 102	95	6.1	
Grape (MV)	426→193	0.01	5	74 – 104	88	18	
	Quantification	1.4	5	87 – 95	88	6.6	
	428→195	0.01	5	75 – 112	92	19	
	Confirmation	1.4	5	86 – 97	93	4.5	
Lettuce leaves (MV)	426→193	0.01	3	84, 87, 88	86	2.9	GRM061.03A
	Quantification	0.10	3	88, 88, 89	88	0.9	
	428→195	0.01	3	78, 83, 84	82	3.8	TK0103846
	Confirmation	0.10	3	85, 87, 88	86	1.8	Mäyer, 2015
Radish leaves (MV)	426→193	0.01	3	82, 84, 87	84	2.7	
	Quantification	0.10	3	85, 85, 87	86	1.2	

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
	428→195 Confirmation	0.01 0.10	3 3	79, 82, 83 84, 87, 87	81 86	2.8 2.1	
Radish root (MV)	426→193 Quantification	0.01 0.10	3 3	81, 83, 86 88, 89, 90	84 89	2.7 1.1	
	428→195 Confirmation	0.01 0.10	3 3	84, 86, 86 89, 90, 91	85 90	1.3 1.0	
Wheat whole plant (MV)	426→193 Quantification	0.01 0.10	3 3	87, 95, 99 97, 99, 99	94 98	6.2 0.8	
	428→195 Confirmation	0.01 0.10	3 3	88, 89, 93 93, 95, 98	90 95	2.9 2.3	
Wheat hay	426→193 Quantification	0.01 0.10	3 3	99, 104, 107 115, 117, 118	103 117	3.8 1.2	
	428→195 Confirmation	0.01 0.10	3 3	99, 100, 104 112, 112, 115	101 113	2.3 1.5	
Wheat grain	426→193 Quantification	0.01 0.10	3 3	83, 85, 86 86, 90, 93	85 89	1.7 3.7	
	428→195 Confirmation	0.01 0.10	3 3	86, 86, 87 86, 90, 92	87 90	0.8 3.4	
Raisin Dry (MV)	426→193 Quantification	0.01 0.1	5 5	80 – 125 92 – 104	104 98	16 5	GRM061.03A S13-03422 TK0178744 Tessier, 2015
	428→186 Confirmation	0.01 0.1	5 5	81 – 103 83 – 102	93 95	9 8	
Must (Raisin) (MV)	426→193 Quantification	0.01 0.1	5 5	77 – 104 88 – 101	94 93	11 5	
	428→186 Confirmation	0.01 0.1	5 5	75 – 112 89 – 105	95 94	16 7	
Red wine (MV)	426→193 Quantification	0.01 0.1	5 5	73 – 100 83 – 88	82 86	14 2	
	428→186 Confirmation	0.01 0.1	5 5	75 – 116 78 – 102	96 88	20 11	
Grape Seed Oil (MV)	426→193 Quantification	0.01 0.1	5 5	73 – 100 82 – 88	86 84	12 3	
	428→186 Confirmation	0.01 0.1	5 5	87 – 128 80 – 91	106 88	18 5	
Grape Dry Pomace (MV)	426→193 Quantification	0.01 0.1	3 3	96 – 104 96 – 104	99 99	4 4	
	428→186 Confirmation	0.01 0.1	3 3	76 – 90 98 – 103	81 100	9 3	
Grape Seed Cake (MV)	426→193 Quantification	0.01 0.1	3 3	71 – 108 86 – 91	89 89	21 3	
	428→186 Confirmation	0.01 0.1	3 3	73 – 78 88 – 93	75 90	3 3	
Tomato (MV)	426→193 Quantification	0.01 5.0	3 3	102 – 123 103 – 122	114 112	9.8 8.7	GRM061.03A TK0163552 Salzman, 2015
	428→195 Confirmation	0.01 5.0	3 3	124 – 126 106 – 125	125 114	0.6 8.4	
Tomato Juice (MV)	426→193 Quantification	0.01 0.5	3 3	107 – 115 91 – 110	112 100	4.1 9.5	
	428→195 Confirmation	0.01 0.5	3 3	89 – 114 104 – 106	103 105	13 1.0	
Tomato Paste (MV)	426→193 Quantification	0.01 5.0	3 3	95 – 106 102 – 116	99 111	6.5 6.7	
	428→195 Confirmation	0.01 5.0	3 3	90 – 113 110 – 113	105 111	12 1.5	

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Lettuce (MV)	426→193	0.01	5	81 – 101	94	9	QuEChERS
	Quantification	5	5	99 – 111	103	5	
	426→171	0.01	5	88 – 112	101	10	S14-05402
	Confirmation	5	5	101 – 110	105	3	TK0253228
Oilseed Rape (MV)	426→193	0.01	5	76 – 84	80	4	Meseguer, 2015
	Quantification	0.2	5	71 – 86	81	8	
	426→171	0.01	5	74 – 82	78	5	
	Confirmation	0.2	5	71 – 88	82	8	
Wheat Grain (MV)	426→193	0.01	5	86 – 99	94	6	
	Quantification	1	5	70 – 92	85	10	
	426→171	0.01	5	92 – 107	98	6	
	Confirmation	1	5	69 – 95	86	12	
Dried Broad Brans (MV)	426→193	0.01	5	83 – 90	87	3	
	Quantification	0.2	5	80 – 87	83	3	
	426→171	0.01	5	66 – 90	78	13	
	Confirmation	0.2	5	77 – 84	81	3	
Oranges (MV)	426→193	0.01	5	70 – 101	87	13	
	Quantification	1	5	67 – 81	74	7	
	426→171	0.01	5	71 – 101	85	15	
	Confirmation	1	5	70 – 80	74	6	
Coffee Bean (MV)	426→193	0.01	5	81 – 117	95	14	
	Quantification	0.2	5	61 – 78	70	10	
	426→171	0.01	5	58 – 97	77	19	
	Confirmation	0.2	5	102 – 108	105	2	
Lettuce (ILV)	426→193	0.01	5	68 – 75	73	4.2	QuEChERS
	Quantification	5.0	5	76 – 79	77	1.8	
	426→171	0.01	5	67 – 76	73	5.1	S14-05729
	Confirmation	5.0	5	76 - 80	77	2.0	TK0103788
Wheat Grain (ILV)	426→193	0.01	5	77 – 83	81	3.1	Merdian, 2015
	Quantification	1.0	4	82 – 88	86	2.9	
	426→171	0.01	5	81 – 84	82	1.6	
	Confirmation	1.0	4	83 – 87	85	2.1	
Oil Seed Rape (ILV)*	426→193	0.01	5	63 – 74	70	5.8	
	Quantification	0.20	5	86 – 92	90	2.8	
	426→171	0.01	5	66 – 82	75	8.2	
	Confirmation	0.20	5	86 – 92	90	2.5	
Coffee Bean (ILV)	426→193	0.01	5	78 – 87	81	4.6	
	Quantification	0.20	5	84 – 89	86	2.2	
	426→171	0.01	5	78 – 86	83	4.7	
	Confirmation	0.20	5	84 – 87	86	1.5	

MV: Method Validation, ILV: Independent Laboratory Validation

* Data after minor modifications: To keep samples cool throughout the procedure to avoid any fat re-dissolving into extract and reduce the sample weight from 5.0 g to 2.0 g.

Animal matrices

Bovine muscle, liver, kidney, milk, blood and hen eggs			
Analyte:	Pydiflumetofen (m/z 426→193 for quantification, 426→166 for confirmation)	LC-MS/MS	GRM061.06A
LOQ:	0.01 mg/kg		

Description	A 10 g sample is homogenized with acetonitrile: ultra-pure water (80:20, v/v) for 3-5 minutes. For the extraction of bovine fat, a 10 g sample is dissolved into 60 mL of hexane before liquid-liquid partitioning into 2 × 50 mL acetonitrile: ultra-pure water (80:20, v/v). Samples are centrifuged and a 1.0 mL aliquot is directly diluted with 0.1% ultra-pure water for final determination by LC-MS/MS. If there is insufficient instrument sensitivity to analyse samples by direct dilution, or additional clean-up is required, a 10 mL aliquot is evaporated to 2-3 mL (only aqueous remaining). Samples are buffered with 0.4 M sodium acetate adjusted to pH 5 and cleaned up using solid phase extraction (SPE) (Oasis HLB 3 mL, 60 mg). Final determination is performed by LC-MS/MS.		
Bovine muscle, liver, kidney, milk, blood and hen eggs			
Analyte:	2,4,6-trichlorophenol (m/z 195→159 for quantification, 197→161 for confirmation)	LC-MS/MS	GRM061.07A
LOQ:	0.01 mg/kg		
Description	A 10 g sample is homogenized or shaken with acetonitrile: ultra-pure water (80:20, v/v) for 2-5 minutes. For the extraction of bovine fat, a 10 g sample is dissolved into 50 mL n-hexane before liquid-liquid partitioning into 2 × 50 mL acetonitrile: ultra-pure water (80:20, v/v). Samples are centrifuged (except fat samples) and a 10 mL aliquot is evaporated to approximately 2-3 mL (only aqueous remaining). Samples are buffered with 0.4 M sodium acetate containing β-glucuronidase and diluted with water. 2,4,6-trichlorophenol residues are released from conjugates by hydrolysis, incubating at 37°C for 18 hrs. After hydrolysis, samples are chilled at < -10 °C in a freezer, diluted with water and an additional clean-up using solid phase extraction (SPE) (Oasis HLB 3 mL, 60mg) carried out. Final determination is performed by LC-MS/MS.		
Bovine milk			
Analyte:	SYN548264 (m/z 246→131 for quantification, 246→111 for confirmation) SYN508272 (m/z 176→136 for quantification, 176→156 for confirmation)	LC-MS/MS	GRM061.08A
LOQ:	0.01 mg/kg		
Description	A 10 g sample is shaken with 40 mL of acetonitrile. A 50 µL aliquot is diluted with ultra-pure water and acetonitrile. Final determination is by LC-MS/MS using matrix-matched calibration.		
Bovine kidney and liver			
Analyte:	SYN547897 (m/z 442→210 for quantification, 442→360 for confirmation) SYN548263 (m/z 276→131 for quantification, 276→200 for confirmation)	LC-MS/MS	GRM061.09A
LOQ:	0.01 mg/kg		
Description	A 10 g sample of liver or kidney is homogenized with acetonitrile: ultra-pure water (80:20, v/v) for 2-3 minutes. Samples are centrifuged, a 10 mL aliquot is filtered through a Sep-Pak cartridge (C18 3mL, 200 mg) then evaporated to approximately 2 mL (until only aqueous remaining). Samples are buffered to pH 5 with 0.4M sodium acetate containing β-glucuronidase and diluted with water. Conjugates of SYN547897 and SYN548263 are hydrolyzed by incubation at 37°C for 18 hrs. After hydrolysis, samples are diluted to 10 mL with 50 mM KH_2PO_4 and sonicated for 15 min. Samples are cleaned-up by solid phase extraction (SPE) (Oasis MAX 6 mL, 150 mg). Final determination is by LC-MS/MS using matrix-matched calibration.		
Bovine liver, milk, fat, blood and hen eggs			
Analyte:	Pydiflumetofen (m/z 426→193 for quantification, 428→195 for confirmation)	LC-MS/MS	QuEChERS
LOQ:	0.01 mg/kg		
Description	The residues of pydiflumetofen are extracted by agitation with acetonitrile in presence of water. After addition of a buffer salt mixture, containing $MgSO_4$ (to bind water), NaCl (to salt-out the analyte), sodium citrate tribasic dihydrate and sodium citrate dibasic sesquihydrate (to obtain a slightly acidic buffer), the extract is shaken. After centrifugation, an aliquot of the acetonitrile phase is cleaned by dispersive solid phase extraction, using primary secondary amine (PSA). After centrifugation, an aliquot is diluted with acetonitrile: water (20:80 v/v) prior to quantification by LC-MS/MS.		

Validation data for methods on animal matrices are summarised in Tables 36–39.

Table 36 Summary of recovery data for pydiflumetofen fortified into animal matrices

Commodity	Mass transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine Meat (MV)	426→193	0.01	5	107 – 116	111	3.9	GRM061.06A Direct Dilution Method TK0103792 Mayer, 2015
	Quantification	0.1	5	78 – 119	108	16	
	426→166	0.01	5	110 – 120	115	3.1	
	Confirmation	0.1	5	77 – 116	106	16	
Bovine Liver (MV)	426→193	0.01	5	105 – 112	108	2.6	
	Quantification	0.1	5	111 – 118	115	2.6	
	426→166	0.01	5	93 – 112	105	7.1	
	Confirmation	0.1	5	110 – 122	116	4.3	
Bovine Kidney (MV)	426→193	0.01	5	101 – 110	105	3.5	
	Quantification	0.1	5	100 – 107	104	2.6	
	426→166	0.01	Not validated due to significant interference				
	Confirmation	0.1					
Bovine Fat (MV)	426→193	0.01	5	84 – 95	88	5.3	
	Quantification	0.1	5	94 – 109	102	6.9	
	426→166	0.01	5	84 – 103	91	8.0	
	Confirmation	0.1	5	95 – 107	102	5.5	
Milk (MV)	426→193	0.01	5	99 – 109	104	4.9	
	Quantification	0.1	5	105 – 113	109	3.0	
	426→166	0.01	5	95 – 111	104	6.0	
	Confirmation	0.1	5	106 – 112	110	2.2	
Bovine Blood (MV)	426→193	0.01	5	101 – 125	110	8.2	
	Quantification	0.1	5	102 – 108	105	2.2	
	426→166	0.01	5	97 – 124	105	11	
	Confirmation	0.1	5	103 – 109	105	2.4	
Eggs (MV)	426→193	0.01	4	99 – 105	101	2.7	
	Quantification	0.1	5	101 – 108	104	2.6	
	426→166	0.01	4	93 – 112	102	8.3	
	Confirmation	0.1	5	105 – 109	107	1.7	
Bovine Meat (MV)	426→193	0.01	5	86 – 103	93	7.6	GRM061.06A SPE Clean-up Method TK0103792 Mayer, 2015
	Quantification	0.1	5	70 - 99	87	12	
	426→166	0.01	5	89 – 100	96	4.6	
	Confirmation	0.1	5	73 – 98	88	11	
Bovine Liver (MV)	426→193	0.01	5	92 – 106	97	5.4	
	Quantification	0.1	5	100 – 105	103	1.9	
	426→166	0.01	5	94 – 103	97	3.5	
	Confirmation	0.1	5	99 – 105	103	2.8	
Bovine Kidney (MV)	426→193	0.01	5	84 – 104	92	9.7	
	Quantification	0.1	5	84 – 100	91	7.0	
	426→166	0.01	5	86 – 117	98	13	
	Confirmation	0.1	5	86 – 104	92	7.7	
Bovine Fat (MV)	426→193	0.01	5	77 – 89	81	5.4	
	Quantification	0.1	5	86 – 91	89	2.3	
	426→166	0.01	5	78 – 85	81	3.6	
	Confirmation	0.1	5	85 – 91	88	2.3	
Milk (MV)	426→193	0.01	5	84 – 109	93	11	
	Quantification	0.1	5	82 – 99	90	8.0	
	426→166	0.01	5	88 – 105	94	7.0	
	Confirmation	0.1	5	82 – 95	89	6.4	
Bovine Blood (MV)	426→193	0.01	5	92 – 99	96	2.8	
	Quantification	0.1	5	96 – 100	98	1.5	
	426→166	0.01	5	89 – 100	94	4.5	
	Confirmation	0.1	5	97 – 104	99	2.7	
Eggs (MV)	426→193	0.01	5	91 – 103	95	5.9	
	Quantification	0.1	5	94 – 100	97	2.8	
	426→166	0.01	5	92 – 107	97	6.0	
	Confirmation	0.1	5	96 – 100	98	1.7	

Commodity	Mass transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine Meat (MV)	426→193	0.01	5	86 – 103	93	7.6	GRM061.06A
	Quantification	0.1	5	70 – 99	87	12	SPE Clean-up
	426→166	0.01	5	89 – 100	96	4.6	
	Confirmation	0.1	5	73 – 98	88	11	Solvent Calibration
Bovine Liver (MV)	426→193	0.01	5	92 – 106	97	5.4	
	Quantification	0.1	5	100 – 105	103	1.9	36383
	426→166	0.01	5	94 – 103	97	3.5	TK0103783
	Confirmation	0.1	5	99 – 105	103	2.8	Harris, 2015
Bovine Kidney (MV)	426→193	0.01	5	84 – 104	92	9.7	
	Quantification	0.1	5	84 – 100	91	7.0	
	426→166	0.01	5	86 – 117	98	13	
	Confirmation	0.1	5	86 – 104	92	7.7	
Bovine Fat (MV)	426→193	0.01	5	77 – 89	81	5.4	
	Quantification	0.1	5	86 – 91	89	2.3	
	426→166	0.01	5	78 – 85	81	3.6	
	Confirmation	0.1	5	85 – 91	88	2.3	
Milk (MV)	426→193	0.01	5	84 – 109	93	11	
	Quantification	0.1	5	82 – 99	90	8.0	
	426→166	0.01	5	88 – 105	94	7.0	
	Confirmation	0.1	5	82 – 95	89	6.4	
Bovine Blood (MV)	426→193	0.01	5	92 – 99	96	2.8	
	Quantification	0.1	5	96 – 100	98	1.5	
	426→166	0.01	5	89 – 100	94	4.5	
	Confirmation	0.1	5	97 – 104	99	2.7	
Eggs (MV)	426→193	0.01	5	91 – 103	95	5.9	
	Quantification	0.1	5	94 – 100	97	2.8	
	426→166	0.01	5	92 – 107	97	6.0	
	Confirmation	0.1	5	96 – 100	98	1.7	
Bovine Meat (MV)	426→193	0.01	5	85 – 102	92	7.8	GRM061.06A
	Quantification	0.1	5	72 – 101	90	12	SPE Clean-up
	426→166	0.01	5	84 – 96	92	5.1	
	Confirmation	0.1	5	76 – 102	91	11	Matrix Matched Calibration
Bovine Liver (MV)	426→193	0.01	5	88 – 101	93	5.2	
	Quantification	0.1	5	97 – 102	100	2.0	36383
	426→166	0.01	5	89 – 97	92	3.5	TK0103783
	Confirmation	0.1	5	98 – 104	101	2.8	Harris, 2015
Bovine Kidney (MV)	426→193	0.01	5	82 – 102	90	10	
	Quantification	0.1	5	85 – 101	92	7.1	
	426→166	0.01	5	77 – 108	89	14	
	Confirmation	0.1	5	86 – 103	92	7.5	
Bovine Fat (MV)	426→193	0.01	5	75 – 87	80	5.6	
	Quantification	0.1	5	87 – 92	89	2.3	
	426→166	0.01	5	78 – 85	81	3.6	
	Confirmation	0.1	5	85 – 91	88	2.3	
Milk (MV)	426→193	0.01	5	78 – 103	87	12	
	Quantification	0.1	5	83 – 100	91	8.0	
	426→166	0.01	5	86 – 103	91	7.4	
	Confirmation	0.1	5	83 – 97	90	6.5	
Bovine Blood (MV)	426→193	0.01	5	89 – 97	93	2.9	
	Quantification	0.1	5	96 – 99	97	1.5	
	426→166	0.01	5	87 – 99	92	4.7	
	Confirmation	0.1	5	95 – 102	97	3.0	
Eggs (MV)	426→193	0.01	5	84 – 96	89	5.9	
	Quantification	0.1	5	88 – 93	90	2.8	

Commodity	Mass transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
	426→166 Confirmation	0.01 0.1	5 5	83 – 97 89 – 92	88 91	6.1 1.8	
Bovine Meat (MV)	426→193 Quantification	0.01 0.1	5 5	107 – 116 78 – 119	111 108	3.9 16	GRM061.06A Direct Analysis Solvent Calibration 36383 TK0103783 Harris, 2015
	426→166 Confirmation	0.01 0.1	5 5	110 – 120 77 – 116	115 106	3.1 16	
Bovine Liver (MV)	426→193 Quantification	0.01 0.1	5 5	105 – 112 111 – 118	108 115	2.6 2.6	
	426→166 Confirmation	0.01 0.1	5 5	93 – 112 110 – 122	105 116	7.1 4.3	
Bovine Kidney (MV)	426→193 Quantification	0.01 0.1	5 5	101 – 110 100 – 107	105 104	3.5 2.6	
	426→166 Confirmation	0.01 0.1	Not validated due to significant interference				
Bovine Fat (MV)	426→193 Quantification	0.01 0.1	5 5	84 – 95 94 – 109	88 102	5.3 6.9	
	426→166 Confirmation	0.01 0.1	5 5	84 – 103 95 – 107	91 102	8.0 5.5	
Milk (MV)	426→193 Quantification	0.01 0.1	5 5	99 – 109 105 – 113	104 109	4.9 3.0	
	426→166 Confirmation	0.01 0.1	5 5	95 – 111 106 – 112	104 110	6.0 2.2	
Bovine Blood (MV)	426→193 Quantification	0.01 0.1	5 5	101 – 125 102 – 108	110 105	8.2 2.2	
	426→166 Confirmation	0.01 0.1	5 5	97 – 124 103 – 109	105 105	11 2.4	
Eggs (MV)	426→193 Quantification	0.01 0.1	5 5	99 – 105 101 – 108	101 104	2.7 2.6	
	426→166 Confirmation	0.01 0.1	5 5	93 – 112 105 – 109	102 107	8.3 1.7	
Bovine Meat (MV)	426→193 Quantification	0.01 0.1	5 5	100 – 108 73 - 111	104 101	3.8 16	GRM061.06A Direct Analysis Matrix Matched Calibration 36383 TK0103783 Harris, 2015
	426→166 Confirmation	0.01 0.1	5 5	103 – 112 72 – 109	107 100	3.0 16	
Bovine Liver (MV)	426→193 Quantification	0.01 0.1	5 5	99 – 106 101 – 107	102 104	2.7 2.5	
	426→166 Confirmation	0.01 0.1	5 5	90 – 107 100 – 110	101 105	6.6 4.3	
Bovine Kidney (MV)	426→193 Quantification	0.01 0.1	5 5	102 – 112 104 – 112	107 109	3.7 2.7	
	426→166 Confirmation	0.01 0.1	5 5	Quantification not possible 100 – 126	 109	 9.5	
Bovine Fat (MV)	426→193 Quantification	0.01 0.1	5 5	82 – 93 94 – 110	86 103	5.4 7.0	
	426→166 Confirmation	0.01 0.1	5 5	82 – 102 96 – 108	90 103	8.3 5.3	
Milk (MV)	426→193 Quantification	0.01 0.1	5 5	92 – 102 97 – 104	97 101	5.0 2.8	
	426→166 Confirmation	0.01 0.1	5 5	88 – 103 99 – 104	97 102	6.0 2.2	
Bovine Blood (MV)	426→193 Quantification	0.01 0.1	5 5	100 – 124 98 – 105	109 101	8.2 2.4	
	426→166 Confirmation	0.01 0.1	5 5	98 – 126 103 – 110	106 106	11 2.7	

Commodity	Mass transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Eggs (MV)	426→193	0.01	5	100 – 106	102	2.6	
	Quantification	0.1	5	99 – 105	102	2.5	
	426→166	0.01	5	90 – 108	98	8.0	
	Confirmation	0.1	5	99 – 102	101	1.7	
Bovine Liver (ILV)	426→193	0.01	5	103 – 109	105	2.2	GRM061.06A
	Quantification	0.1	5	90 – 101	95	4.8	
	426→166	0.01	5	103 – 118	109	5.1	1781.7103
	Confirmation	0.1	5	96 – 115	103	6.7	TK0272121
	428→195	0.01	5	104 – 110	106	2.0	Smith, 2015
	Confirmation	0.1	5	90 – 110	97	8.1	
Bovine Liver (MV)	426→193	0.01	5	92 – 107	101	6	QuEChERS
	Quantification	0.1	5	97 – 107	101	4	
	428→195	0.01	5	91 – 107	101	7	PTRL Europe ID P 3592 G
	Confirmation	0.1	5	97 – 108	101	4	TK0208383
Bovine Fat (MV)	426→193	0.01	5	87 – 90	88	1	Richter, 2015
	Quantification	0.1	5	90 – 96	92	2	
	428→195	0.01	5	85 – 90	87	2	
	Confirmation	0.1	5	89 – 96	92	3	
Milk (MV)	426→193	0.01	5	87 – 113	100	9	
	Quantification	0.1	5	93 – 106	100	5	
	428→195	0.01	5	87 – 114	100	9	
	Confirmation	0.1	5	93 – 107	100	5	
Bovine Blood (MV)	426→193	0.01	5	95 – 98	97	1	
	Quantification	0.1	5	95 – 98	96	1	
	428→195	0.01	5	95 – 100	98	2	
	Confirmation	0.1	5	95 – 99	97	2	
Eggs (MV)	426→193	0.01	5	87 – 90	88	1	
	Quantification	0.1	5	90 – 96	92	2	
	428→195	0.01	5	91 – 98	95	3	
	Confirmation	0.1	5	94 – 100	98	2	
Bovine Liver (ILV)	426→193	0.01	5	74 – 84	80	4.6	QuEChERS
	Quantification	0.1	5	87 – 95	92	3.8	
	428→195	0.01	5	75 – 78	77	1.7	CEMR-7055
	Confirmation	0.1	5	87 – 94	91	3.2	TK0259229
Milk (ILV)	426→193	0.01	5	87 – 91	88	2.0	Bradford, 2015
	Quantification	0.1	5	90 – 95	92	2.2	
	428→195	0.01	5	81 – 91	87	4.5	
	Confirmation	0.1	5	91 – 99	95	3.2	
Bovine Muscle (ILV)	426→193	0.01	5	83 – 88	85	2.8	QuEChERS
	Quantification	0.1	5	78 – 85	80	3.6	
	428→195	0.01	5	79 – 89	85	5.1	PASC-REP-1467
	Confirmation	0.1	5	78 – 85	80	3.5	TK0374048
Eggs (ILV)	426→193	0.01	5	90 – 97	95	3.0	Xu, 2017
	Quantification	0.1	5	77 – 86	81	4.5	
	428→195	0.01	5	85 – 97	93	5.5	
	Confirmation	0.1	5	79 – 84	81	3.1	

MV: Method Validation, ILV: Independent Laboratory Validation

Table 37 Summary of recovery data for 2,4,6-trichlorophenol fortified into animal matrices

Commodity	Mass transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine Muscle (MV)	195→159	0.01	5	80 – 94	87	6	GRM061.07A
	Quantification	0.1	5	82 – 91	85	4	
	197→161	0.01	5	81 – 90	85	4	TK0103792 Mayer, 2015
	Confirmation	0.1	5	81 – 88	85	4	
Bovine Liver (MV)	195→159	0.01	5	78 – 86	81	4	
	Quantification	0.1	5	85 – 88	86	2	
	197→161	0.01	5	81 – 89	84	4	
	Confirmation	0.1	5	82 – 88	85	3	
Bovine Kidney (MV)	195→159	0.01	5	83 – 89	85	3	
	Quantification	0.1	5	83 – 89	86	3	
	197→161	0.01	5	82 – 88	85	3	
	Confirmation	0.1	5	80 – 89	85	4	
Bovine Fat (MV)	195→159	0.01	5	85 – 88	86	1	
	Quantification	0.1	5	85 – 95	89	4	
	197→161	0.01	5	84 – 88	85	2	
	Confirmation	0.1	5	87 – 94	90	3	
Milk (MV)	195→159	0.01	5	82 – 91	86	4	
	Quantification	0.1	5	86 – 94	90	3	
	197→161	0.01	5	82 – 90	85	4	
	Confirmation	0.1	5	86 – 92	89	3	
Bovine Blood (MV)	195→159	0.01	5	83 – 90	90	3	
	Quantification	0.1	5	87 – 93	88	3	
	197→161	0.01	5	79 – 92	85	6	
	Confirmation	0.1	5	85 – 92	89	3	
Egg (MV)	195→159	0.01	5	79 – 82	80	2	
	Quantification	0.1	5	82 – 85	83	2	
	197→161	0.01	5	78 – 84	80	3	
	Confirmation	0.1	5	81 – 84	82	1	
Bovine Muscle (MV)	195→159	0.01	5	80 – 94	87	6	GRM061.07A
	Quantification	0.1	5	82 – 91	85	4	
	197→161	0.01	5	81 – 90	85	4	PTRL Europe ID P 3613 G TK0257521 Senciuc, 2015
	Confirmation	0.1	5	81 – 88	85	4	
Bovine Liver (MV)	195→159	0.01	5	78 – 86	81	4	
	Quantification	0.1	5	85 – 88	86	2	
	197→161	0.01	5	81 – 89	84	4	
	Confirmation	0.1	5	82 – 88	85	3	
Bovine Kidney (MV)	195→159	0.01	5	83 – 89	85	3	
	Quantification	0.1	5	83 – 89	86	3	
	197→161	0.01	5	82 – 88	85	3	
	Confirmation	0.1	5	80 – 89	85	4	
Bovine Fat (MV)	195→159	0.01	5	85 – 88	86	1	
	Quantification	0.1	5	85 – 95	89	4	
	197→161	0.01	5	84 – 88	85	2	
	Confirmation	0.1	5	87 – 94	90	3	
Milk (MV)	195→159	0.01	5	82 – 91	86	4	
	Quantification	0.1	5	86 – 94	90	3	
	197→161	0.01	5	82 – 90	85	4	
	Confirmation	0.1	5	85 – 92	89	3	
Bovine Blood (MV)	195→159	0.01	5	83 – 90	87	3	
	Quantification	0.1	5	87 – 93	90	3	
	197→161	0.01	5	79 – 92	85	6	
	Confirmation	0.1	5	85 – 92	89	3	
Egg (MV)	195→159	0.01	5	79 – 82	80	2	
	Quantification	0.1	5	82 – 85	83	2	
	197→161	0.01	5	78 – 84	80	3	
	Confirmation	0.1	5	81 – 84	82	1	

Commodity	Mass transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine Muscle (ILV)	195→159	0.01	5	94 – 99	96	2	GRM061.07A
	Quantification	0.1	5	97 – 100	98	1	
	197→161	0.01	5	95 – 100	97	2	R B5134
	Confirmation	0.1	5	98 – 101	99	1	TK0259228
Bovine Liver (ILV)	195→159	0.01	5	70 – 78	75	4	Sclewitz, 2015
	Quantification	0.1	5	82 – 90	84	4	
	197→161	0.01	5	64 – 76	71	6	
	Confirmation	0.1	5	81 – 91	84	5	
Bovine Kidney (ILV)	195→159	0.01	5	94 – 100	98	2	
	Quantification	0.1	5	99 – 103	101	1	
	197→161	0.01	5	83 – 89	88	3	
	Confirmation	0.1	5	97 – 101	99	1	
Bovine Fat (ILV)	195→159	0.01	5	70 – 74	72	2	
	Quantification	0.1	5	72 – 79	74	4	
	197→161	0.01	5	70 – 77	73	4	
	Confirmation	0.1	5	73 – 79	75	3	
Milk (ILV)	195→159	0.01	5	93 – 106	98	5	
	Quantification	0.1	5	94 – 96	95	1	
	197→161	0.01	5	92 – 101	97	4	
	Confirmation	0.1	5	93 – 97	96	3	
Bovine Blood (ILV)	195→159	0.01	5	76 – 80	78	2	
	Quantification	0.1	5	71 – 82	74	6	
	197→161	0.01	5	79 – 83	81	3	
	Confirmation	0.1	5	71 – 82	74	6	
Eggs (ILV)	195→159	0.01	5	92 – 100	95	4	
	Quantification	0.1	5	96 – 101	98	2	
	197→161	0.01	5	98 – 102	100	2	
	Confirmation	0.1	5	97 – 101	98	2	

MV: Method Validation, ILV: Independent Laboratory Validation

Table 38 Summary of recovery data for SYN548264 and SYN508272 fortified into bovine milk

Analyte	Mass transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
SYN548264	246→131	0.01	5	82 – 97	89	6.1	GRM061.08A
	Quantification	0.1	5	92 – 102	95	4.5	
Solvent Calibration	246→111	0.01	5	88 – 93	92	2.2	TK0103792
	Confirmation	0.1	5	95 – 105	97	4.6	Mayer, 2015
SYN548264	246→131	0.01	5	78 – 91	84	6.0	
	Quantification	0.1	5	85 – 94	87	4.6	
Matrix Matched Calibration	246→111	0.01	5	82 – 87	85	2.2	
	Confirmation	0.1	5	88 – 97	90	4.4	
SYN508272	176→136	0.01	5	75 – 94	85	9.1	
	Quantification	0.1	5	78 – 92	85	7.7	
Solvent Calibration	176→156	0.01	5	81 – 92	87	4.6	
	Confirmation	0.1	5	77 – 92	85	7.4	
SYN508272	176→136	0.01	5	75 – 94	85	9.1	
	Quantification	0.1	5	78 – 92	85	7.7	
Matrix Matched Calibration	176→156	0.01	5	81 – 92	87	4.6	
	Confirmation	0.1	5	77 – 92	85	7.4	

Table 39 Summary of recovery data for SYN547897 and SYN548263 fortified into bovine liver and kidney

Commodity	Mass transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
SYN547897							GRM061.09A
Bovine Liver	442→210	0.01	5	86 – 125	108	16	
	Quantification	0.1	5	88 – 99	94	4.5	TK0103792
	442→360	0.01	5	81 – 125	106	18	Mayer, 2015
	Confirmation	0.1	5	83 – 96	92	5.6	
Bovine Kidney	442→210	0.01	5	94 – 115	106	6.7	
	Quantification	0.1	5	109 – 119	113	3.5	
	442→360	0.01	5	98 – 113	105	5.6	
	Confirmation	0.1	5	107 – 119	113	3.9	
SYN548263							
Bovine Liver	276→131	0.01	5	83 – 88	85	2.3	
	Quantification	0.1	5	88 – 92	91	2.2	
	276→200	0.01	5	78 -86	82	4.2	
	Confirmation	0.1	5	84 – 95	90	4.7	
Bovine Kidney	276→131	0.01	5	75 – 85	78	5.2	
	Quantification	0.1	5	77 – 85	80	4.5	
	276→200	0.01	5	74 – 83	80	4.8	
	Confirmation	0.1	5	79 – 85	81	3.2	

Stability of pesticide residues in stored analytical samples

The Meeting received data on the storage stability of pydiflumetofen and its metabolites in samples for plant and animal commodities stored frozen.

Sample extracts

The stability of pydiflumetofen was assessed by storing the final extracts refrigerated at 5 ± 4 °C. The samples were then re-analysed by a second measurement after 8–21 days storage using freshly prepared calibration standards. The overall mean recoveries in the stored fortified samples were within the acceptable range of 70–120% and within ±10% of the initial values for all matrices.

Table 40 Summary of Recovery Data for pydiflumetofen in sample final extracts after 8–21 days of storage at 5 ± 4 °C (m/z 426→193)

Commodity	Storage interval (days)	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Apple	0	0.01	5	79 – 108	91	12	GRM061.03A
	14	0.01	5	83 – 102	92	8.4	
Lettuce	0	0.01	5	73 – 97	82	11	TK013794
	13	0.01	5	82 – 104	89	9.9	Hung, 2015
Tomato	0	0.01	5	78 – 111	97	12	
	14	0.01	5	83 – 107	94	9.5	
Cabbage	0	0.01	5	92 – 103	94	5.2	
	19	0.01	5	85 – 104	95	8.6	
Fresh Peas	0	0.01	4	95 – 100	98	3.3	
	13	0.01	4	95 – 107	102	7.4	
Dry Beans	0	0.01	5	87 – 107	96	8.1	
	11	0.01	5	97 – 102	100	5.0	
Wheat Grain	0	0.20	5	92 – 101	96	4.2	
	8	0.20	5	83 – 93	87	4.6	
Wheat Straw	0	0.01	5	100 – 123	110	8.2	
	11	0.01	5	93 – 115	103	7.8	
Wheat Forage	0	0.01	5	93 – 111	101	7.0	
	21	0.01	5	96 - 107	103	4.1	
Potato	0	0.01	5	81 – 101	92	8.2	
	15	0001	5	81 – 96	88	6.5	

Commodity	Storage interval (days)	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Rape Seed	0	0.01	5	82 – 110	92	12	
	12	0.01	5	79 – 115	98	18	
Grape	0	0.01	5	74 – 104	88	17	
	11	0.01	5	73 – 100	89	13	
Lettuce	0	0.01	5	81 – 101	94	9	QuEChERS
	8	0.01	5	104 – 114	107	4	
Oilseed Rape	1	0.01	5	76 – 84	80	4	S14-05402
	8	0.01	5	67 – 83	73	9	TK0253228
Dried Broad Beans	1	0.01	5	83 – 90	87	3	Meseguer, 2015
	8	0.01	5	82 – 87	84	2	
Wheat Grain	1	0.01	5	86 – 99	94	6	
	8	0.01	5	79 – 95	86	8	
Oranges	1	0.01	5	70 – 101	87	13	
	8	0.01	5	79 – 105	92	10	
Coffee Bean	1	0.01	5	81 – 117	95	14	
	9	0.01	5	81 – 101	93	8	
Lettuce	0	0.01	5	68 – 75	73	4.2	QuEChERS
	7	0.01	5	72 – 79	76	3.5	
Wheat Grain	0	0.01	5	77 – 83	81	3.1	S14-05729
	7	0.01	5	81 – 94	90	5.7	TK0103788
Oil Seed Rape	0	0.01	5	63 – 74	70	5.8	Merdian, 2015
	7	0.01	5	57 – 79	68	13	
Coffee Bean	0	0.01	5	78 – 87	81	4.6	
	7	0.01	5	70 – 85	77	7.2	
Bovine Meat	0	0.01	5	100 – 108	104	3.8	GRM061.06A
	7	0.01	5	103 – 112	107	3.3	
Bovine Liver	0	0.01	5	99 – 106	102	2.7	TK0103792
	7	0.01	5	99 – 109	104	3.6	Mayer, 2015
Bovine Kidney	0	0.01	5	101 – 110	105	3.5	
	7	0.01	5	106 – 115	109	3.4	
Bovine Fat	0	0.01	5	84 – 95	88	5.3	
	7	0.01	5	85 – 95	89	4.5	
Milk	0	0.01	5	84 – 109	93	11	
	7	0.01	5	84 – 98	90	6.1	
Bovine Blood	0	0.01	5	94 – 108	102	5.7	
	7	0.01	5	87 – 96	93	4.1	
Eggs	0	0.01	5	91 – 103	95	5.9	
	6	0.01	5	92 – 99	95	3.2	
Bovine Liver	0	0.01	5	103 – 109	105	2.3	GRM061.06A
	8	0.01	5	89 – 99	93	3.9	1781.7103
	0	0.1	5	90 – 101	95	4.9	TK0272121
	8	0.1	5	97 – 109	105	4.5	Smith, 2015
Bovine Liver	0	0.01	5		101		QuEChERS
	13	0.01	5	110 – 115	112	2	
Bovine Fat	0	0.01	5		88		PTRL Europe ID P
	8	0.01	5	82 – 88	85	3	3592 G
Milk	0	0.01	5		100		TK0208383
	11	0.01	5	82 – 111	91	13	Richter, 2015
Bovine Blood	0	0.01	5		97		
	12	0.01	5	107 – 111	109	1	
Eggs	0	0.01	5		96		
	8	0.01	5	97 – 108	99	5	
Bovine Liver	0	0.01	5	74 – 84	80	4.6	QuEChERS
	7	0.01	5	89 – 104	96	7.6	CEMR-7055
Milk	0	0.01	5	87 – 91	88	2.0	TK0259229
	7	0.01	5	85 – 96	90	4.9	Bradford, 2015

The stability of sample extracts fortified with 2,4,6-trichlorophenol was checked after a storage period of 10–26 days in a refrigerator at 3–8 °C against freshly prepared calibration standards. The mean recovery values of selected samples at the LOQ level (0.01 mg/kg) and higher level (0.1 mg/kg) were between 70% and 110%, and within ±10% of the initial values when re-analysed.

Table 41 Summary of recovery data for 2,4,6-trichlorophenol in sample final extracts 10–26 days of storage at 3–8 °C (m/z 195→159)

Commodity	Storage interval (days)	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine Muscle	26	0.01	2	87, 93	90	-	GRM061.07A
	26	0.1	2	82, 84	83	-	
Bovine Liver	19	0.01	2	79, 82	81	-	TK0103792 Mayer, 2015
	19	0.1	2	83, 88	86	-	
Bovine Kidney	19	0.01	2	85, 85	85	-	
	19	0.1	2	86, 88	87	-	
Bovine Fat	10	0.01	2	81, 85	83	-	
	10	0.1	2	90, 91	91	-	
Milk	21	0.01	2	83, 90	87	-	
	21	0.1	2	91, 98	95	-	
Bovine Blood	26	0.01	2	86, 91	89	-	
	26	0.1	2	89, 95	92	-	
Eggs	19	0.01	2	73, 76	75	-	
	19	0.1	2	84, 91	88	-	
Bovine Muscle	0	0.01	5	80 – 94	87	6	GRM061.07A
	26	0.01	2	85, 97	91	-	
	0	0.1	5	82 – 91	85	4	PTRL Europe ID P 3613 G TK0257521 Senciuc, 2015
	26	0.1	2	82, 84	83	-	
Bovine Liver	0	0.01	5	78 – 86	81	4	
	19	0.01	2	79, 82	81	-	
	0	0.1	5	85 – 88	86	2	
	19	0.1	2	83, 88	86	-	
Bovine Kidney	0	0.01	5	83 – 89	85	3	
	19	0.01	2	85, 85	85	-	
	0	0.1	5	83 – 89	86	3	
	19	0.1	2	86, 88	87	-	
Bovine Fat	0	0.01	5	85 – 88	86	1	
	10	0.01	2	81, 85	83	-	
	0	0.1	5	85 – 95	89	4	
	10	0.1	2	90, 91	91	-	
Milk	0	0.01	5	82 – 91	86	4	
	21	0.01	2	83, 90	87	-	
	0	0.1	5	86 – 94	90	3	
	21	0.1	2	91, 98	95	-	
Bovine Blood	0	0.01	5	83 – 90	87	3	
	26	0.01	2	86, 91	89	-	
	0	0.1	5	87 – 93	90	3	
	26	0.1	2	89, 95	92	-	
Eggs	0	0.01	5	79 – 82	80	2	
	19	0.01	2	73, 76	75	-	
	0	0.1	5	82 – 85	83	2	
	19	0.1	2	84, 91	88	-	

The stability of sample extracts fortified with SYN548264 and SYN508272 was checked after a storage period of 11 days in a refrigerator at 4 °C against freshly prepared calibration standards. Extracts at only the LOQ concentration (0.01 mg/kg) were then reanalysed using only the quantification mass transition for each analyte. Sample extracts were determined to be stable if the assay accuracy was 70–110% of the theoretical value and the assay precision was ≤20%.

Table 42 Summary of recovery data for SYN548264 (m/z 246→131) and SYN508272 (m/z 176→136) in final extracts from milk sample 11 days of storage at 4 °C

Analyte	Storage interval (hours)	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
SYN548264	0	0.01	5	82 – 97	89	6.1	GRM061.08A
(m/z 246→131)	11	0.01	5	84 – 94	89	4.5	
SYN508272	0	0.01	5	75 – 94	85	9.1	TK0103792
(m/z 176→136)	11	0.01	5	78 – 93	85	7.5	Mayer, 2015

The stability of sample extracts fortified with SYN547897 and SYN548263 was checked after a storage period of between 7 and 8 days in a refrigerator at 4 °C against freshly prepared calibration standards. Extracts at only the LOQ concentration (0.01 mg/kg) were then reanalysed using only the quantification mass transition for each analyte. Sample extracts were determined to be stable the assay accuracy was 70–110% of the theoretical value and the assay precision was ≤20%.

Table 43 Summary of recovery data for SYN547897 (m/z 442→210) and SYN548263 (m/z 276→131) in sample final extracts 7–8 days of storage at 4 °C

Commodity	Storage interval (days)	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
SYN547897 (m/z 442→210)							GRM061.09A
Bovine Liver	0	0.01	5	86 – 125	108	16	
	8	0.01	5	88 – 106	95	7.3	TK0103792
Bovine Kidney	0	0.01	5	95 – 115	106	6.7	Mayer, 2015
	7	0.01	5	90 – 117	104	9.9	
SYN548263 (m/z 276→131)							
Bovine Liver	0	0.01	5	83 – 88	85	2.3	
	7	0.01	5	77 – 90	86	5.7	
Bovine Kidney	0	0.01	5	75 – 85	78	5.2	
	7	0.01	5	78 – 88	81	5.7	

Stored analytical samples

The storage stability of pydiflumetofen was tested in seven different plant matrices (lettuce, orange, oilseed rape seed, potato, beans without pods, wheat grain and straw) (Yozgatli, 2015: S13-02224). Homogenised samples (10 g) of the plant materials destined for frozen storage were fortified with a known amount of pydiflumetofen in acetonitrile at a rate of 0.2 mg/kg. The solution was left to soak, and the samples were sealed and stored in a freezer at a nominal temperature of ≤-18 °C. Triplicate samples were analysed at zero-time, and duplicate samples were analysed after 1, 3, 6, 12, 18 and 23 months of storage. Pydiflumetofen was analysed using method GRM061.03A by LC-MS/MS. The LOQ was 0.01 mg/kg.

Table 44 Recovery of pydiflumetofen from stored fortified samples of plant matrices

Storage interval (Actual)	Recovery (%) [0.2 mg/kg fortification]		
	Procedural	% remaining	Mean
Lettuce head (High water content)			
Day 0	-	96, 97, 106	100
3 months (92 days)	103	92, 96	94
6 months (190 days)	99	99, 101	100
12 months (370 days)	97	111, 115	113
18 months (552 days)	73	77, 83	80
23 months (715 days)	80	81, 88	85
Orange (High acid content)			
Day 0	-	90, 101, 107	100
3 months (92 days)	93	78, 84	81
6 months (190 days)	88	101, 103	102
12 months (370 days)	81	81, 109	95
18 months (561 days)	73	81, 86	84
23 months (708 days)	110	89, 118	103
Oilseed rape seed (High oil content)			

Storage interval (Actual)	Recovery (%) [0.2 mg/kg fortification]		
	Procedural	% remaining	Mean
Day 0	-	99, 102, 110	103
3 months (92 days)	103	80, 82	81
6 months (190 days)	110	89, 98	94
12 months (370 days)	77	69, 73	71
18 months (552 days)	105	81, 85	83
23 months (715 days)	75	70, 76	73
Potato tuber (High starch content)			
Day 0	-	79, 85, 94	86
3 months (97 days)	97	88, 92	90
6 months (186 days)	106	102, 102	102
12 months (364 days)	93	84, 90	87
18 months (551 days)	78	89, 93	91
23 months (697 days)	78	80, 91	86
Adzuki beans without pods (High protein content)			
Day 0	-	99, 105, 108	104
3 months (116 days)	85	85, 102	93
6 months (186 days)	98	108, 110	109
12 months (364 days)	109	107, 111	109
18 months (551 days)	84	90, 99	94
23 months (697 days)	115	99, 108	103
Wheat grain (High starch content)			
Day 0	-	95, 110, 113	106
3 months (91 days)	84	69, 83	76
6 months (186 days)	88	89, 95	92
12 months (379 days)	99	86, 96	91
18 months (551 days)	92	87, 95	91
23 months (697 days)	92	85, 90	88
Wheat straw			
Day 0	-	89, 90, 99	93
3 months (91 days)	108	99, 112	106
6 months (186 days)	108	102, 105	104
12 months (379 days)	83	91, 94	92
18 months (560 days)	91	90, 90	90
23 months (705 days)	79	93, 95	94

The recoveries of pydiflumetofen showed no significant decrease (>30%) in any of the plant matrices analysed after deep-frozen storage for up to 23 months. The analyses of the control samples showed that no residues of pydiflumetofen were present above 30% of the LOQ.

The stability of pydiflumetofen was conducted in processed commodities of corn (flour, meal and oil), soya bean (flour, milk, oil), grape (raisin) and apple fractions (dried fruit and juice) under freezer storage conditions (*ca* -20 ºC) over a period of 24 months (Sharp, 2015: 110G850). Fortifications of pydiflumetofen were performed in corn, soybean, grape and apple processed commodities at 0.20 mg/kg (which is equivalent to 20 times the method limit of quantitation). After fortification, samples were immediately stored frozen except for the Day 0 samples. Stability recovery samples were extracted and analysed at time intervals of day 0, month 3, 6, 9 and 12. Samples were analyzed using method GRM061.03A with LC-MS/MS.

Table 45 Recovery of pydiflumetofen from stored fortified samples of processed commodities

Storage interval	Recovery (%) [0.2 mg/kg fortification]		
	Procedural	% remaining	Mean
Corn flour			
Day 0	115, 115	114, 116	115
3 months	115, 115	114, 116	115
6 months	105, 112	104, 105	104
9 months	157, 161	151, 157	154
12 months	114, 118	106, 113	109
Corn meal			
Day 0	116, 118	93, 122	107
3 months	100, 101	96, 98	97

Storage interval	Recovery (%) [0.2 mg/kg fortification]		
	Procedural	% remaining	Mean
6 months	104, 105	108, 112	110
9 months	164, 165	147, 154	151
12 months	106, 109	97, 97	97
Corn oil			
Day 0	112, 112	89, 114	102
3 months	69, 95	87, 90	89
6 months	81, 91	87, 91	89
9 months	99, 103	93, 96	94
12 months	89, 100	82, 87	84
Soybean flour			
Day 0	100, 108	103, 105	104
3 months	88, 93	93, 94	94
6 months	37, 49	51, 52	52
9 months	104, 129	91, 92	91
12 months	92, 93	80, 83	81
Soybean milk			
Day 0	110, 111	110, 112	111
3 months	104, 106	108, 109	108
6 months	74, 82	87, 93	90
9 months	85, 88	82, 85	83
12 months	83, 85	76, 77	76
Soybean oil			
Day 0	107, 110	104, 128	116
3 months	108, 117	76, 100	88
6 months	83, 90	84, 175	130
9 months	56, 100	90, 100	95
12 months	93, 101	77, 83	80
Grape raisin			
Day 0	98, 99	104, 109	106
3 months	117, 129	124, 125	124
6 months	108, 112	99, 100	100
9 months	111, 115	106, 115	110
12 months	113, 121	113, 114	113
Apple dried fruit			
Day 0	124, 128	127, 127	127
3 months	145, 151	136, 149	143
6 months	128, 132	128, 132	130
9 months	157, 159	144, 159	151
12 months	111, 115	107, 108	108
Apple juice			
Day 0	56, 105	96, 96	96
3 months	111, 113	99, 107	103
6 months	87, 91	81, 84	82
9 months	90, 91	87, 90	88
12 months	84, 84	77, 79	78

The stability of pydiflumetofen was tested in bovine muscle, liver, fat, milk and chicken eggs following deep frozen storage for up to 2 years (Harris, 2017: 36552). Individual samples of bovine muscle, liver, fat, milk and chicken eggs were fortified with a known amount of standard solution containing pydiflumetofen in acetonitrile: water (80:20, v/v) at a rate of 0.10 mg/kg. Samples were either extracted or sealed and transferred to the freezer to simulate conditions under which actual field samples were stored prior to their analysis. Triplicate samples of the matrices were analysed for pydiflumetofen at zero-time. Duplicate samples were removed after approximately 1, 3, 6, 9, 12, 18 and 24 months of frozen storage at typically -20 °C. The samples were analysed for pydiflumetofen using method GRM061.06A with LC-MS/MS.

Table 46 Recovery of pydiflumetofen from stored fortified samples of animal matrices

Storage interval (Actual)	Fortification level (mg/kg)	Recovery (%)		
		Procedural	% remaining	Mean
Bovine muscle				
Day 0	0.1	64, 91	92, 94, 97	94
1 month (28 days)	0.01	93, 100	-	-
	0.1	98, 103	97, 97	97
3 months (91 days)	0.01	71, 75	-	-
	0.1	70, 72	68, 73	71
6 months (181 days)	0.01	108, 111	-	-
	0.1	102, 105	107, 110	109
9 months (273 days)	0.01	104, 105	-	-
	0.1	95, 99	101, 104	103
12 months (365 days)	0.01	102, 103	-	-
	0.1	97, 97	101, 110	106
18 months (547 days)	0.01	96, 104	-	-
	0.1	96, 99	105, 107	106
24 months (730 days)	0.01	105, 109	-	-
	0.1	108, 109	106, 108	107
Bovine liver				
Day 0	0.1	84, 87	74, 78, 95	82
1 month (35 days)	0.01	111, 113	-	-
	0.1	113, 113	116, 118	117
3 months (92 days)	0.01	61, 75	-	-
	0.1	71, 72	63, 82	73
6 months (180 days)	0.01	88, 95	-	-
	0.1	96, 98	98, 102	100
9 months (272 days)	0.01	105, 111	-	-
	0.1	107, 109	111, 111	111
12 months (364 days)	0.01	98, 100	-	-
	0.1	105, 110	108, 111	110
18 months (547 days)	0.01	105, 113	-	-
	0.1	94, 96	89, 123	106
24 months (729 days)	0.01	104, 109	-	-
	0.1	102, 109	106, 109	108
Bovine milk				
Day 0	0.1	80, 86	87, 89, 98	91
1 month (34 days)	0.1	103, 110	108, 113	111
3 months (98 days)	0.01	80, 84	-	-
	0.1	90, 94	96, 98	97
6 months (181 days)	0.01	86, 98	-	-
	0.1	92, 95	95, 99	97
9 months (273 days)	0.01	106, 131	-	-
	0.1	103, 104	105, 106	106
12 months (365 days)	0.01	98, 110	-	-
	0.1	98, 106	101, 113	107
18 months (547 days)	0.01	77, 98	-	-
	0.1	93, 98	98, 98	98
24 months (737 days)	0.01	106, 113	-	-
	0.1	107, 110	100, 101	101
Bovine fat				
Day 0	0.1	81, 92	87, 88, 95	90
1 month (30 days)	0.01	95, 100	-	-
	0.1	108, 113	105, 111	108
3 months (78 days)	0.01	72, 82	-	-
	0.1	76, 78	82, 85	84
6 months (182 days)	0.01	70, 87	-	-
	0.1	85, 88	94, 98	96
9 months (273 days)	0.01	79, 105	-	-
	0.1	91, 94	96, 98	97
12 months (365 days)	0.01	81, 85	-	-
	0.1	78, 80	93, 98	95
18 months (546 days)	0.01	98, 102	-	-

Storage interval (Actual)	Fortification level (mg/kg)	Recovery (%)		
		Procedural	% remaining	Mean
	0.1	89, 89	92, 93	92
24 months (724 days)	0.01	91, 101	-	-
	0.1	96, 98	99, 105	102
Chicken egg				
Day 0	0.1	99, 107	99, 101, 102	101
1 month (33 days)	0.01	105, 106	-	-
	0.1	106, 112	106, 108	107
3 months (90 days)	0.01	75, 76	-	-
	0.1	73, 75	75, 79	77
6 months (180 days)	0.01	86, 93	-	-
	0.1	92, 93	93, 98	95
9 months (272 days)	0.01	97, 102	-	-
	0.1	102, 103	103, 108	106
12 months (365 days)	0.01	98, 105	-	-
	0.1	99, 101	100, 103	101
18 months (547 days)	0.01	88, 92	-	-
	0.1	81, 83	88, 92	90
24 months (736 days)	0.01	98, 101	-	-
	0.1	104, 106	102, 103	103

There was no significant decrease (>30% as compared to the zero-time value) in the observed residue levels of pydiflumetofen in any of the animal matrices studied after deep frozen storage for up to 2 years. Interference in non-fortified aliquots of blank matrix were <30% of the LOQ response.

The stability of SYN508272 and SYN548264 in milk, SYN547897 in liver, SYN547897 and SYN548263 in kidney were tested following frozen storage for up to 12 months (Langridge, 2016: CEMR-7064). Individual samples of milk were fortified with a known amount of standard solution containing SYN548264 or SYN508272 in acetonitrile at a rate of 0.20 mg/kg. Individual samples of liver were fortified with a known amount of standard solution containing SYN547897 in acetonitrile at a rate of 0.20 mg/kg. Individual samples of kidney were fortified with a known amount of standard solution containing SYN547897 or SYN548263 in acetonitrile at a rate of 0.20 mg/kg. Each sample was left to stand for at least five minutes after fortification to allow the spiking solution to soak into the matrix before proceeding with the extraction or sealed and transferred to the freezer to simulate conditions under which actual field samples are stored prior to their analysis. Triplicate samples of the animal matrices were analysed for SYN548264, SYN508272, SYN547897 and SYN548263 at zero-time. Duplicate samples were removed after 1, 2, 3, 6, 9 11 and 12 months of frozen storage and at 5 months of frozen storage for SYN547897 in liver at <-18 °C. A spare set of liver and kidney samples were analysed for SYN547897 as a second 9 month sample set to confirm the trend of the analytical data seen in the original 9 month analysis. The samples were analysed for SYN548264 and SYN508272 using method GRM061.08A and for SYN547897 and SYN548263 using method GRM061.09A.

Table 47 Recovery of SYN508272, SYN548264, SYN547897 and SYN548263 from stored fortified samples of animal matrices

Analyte	Storage interval (Actual)	Recovery (%) [0.2 mg/kg fortification]		
		Procedural	% remaining	Mean
Milk				
SYN508272	Day 0	117, 119	111, 114, 115	113
	1 month (29 days)	108, 111	108, 108	108
	2 months (58 days)	103, 104	102, 104	103
	3 months (101 days)	102, 102	99, 102	100
	6 months (186 days)	103, 103	99, 103	101
	9 months (281 days)	95, 97	100, 101	100
	11 months (338 days)	109, 110	107, 107	107
	12 months (367 days)	104, 104	97, 100	99
SYN548264	Day 0	106, 108	108, 108, 109	108
	1 month (29 days)	101, 102	100, 106	103
	2 months (58 days)	106, 107	107, 109	108
	3 months (101 days)	107, 109	103, 106	105
	6 months (190 days)	103, 105	109, 110	109
	9 months (281 days)	104, 105	112, 112	112
	11 months (338 days)	101, 102	106, 107	107
	12 months (367 days)	115, 115	121, 122	121

Analyte	Storage interval (Actual)	Recovery (%) [0.2 mg/kg fortification]		
		Procedural	% remaining	Mean
Liver				
SYN547897	Day 0	88, 94	96, 98, 99	98
	1 month (27 days)	110, 116	104, 106	105
	2 months (60 days)	92, 94	94, 95	94
	3 months (102 days)	98, 104	80, 84	82
	5 months (158 days)	107, 108	95, 98	96
	6 months (195 days)	101, 117	89, 92	91
	9 months (285 days)	105, 108	72, 81	76
	9 months (299 days)	85, 87	55, 58	57
	11 months (335 days)	91, 92	68, 69	68
	12 months (369 days)	104, 109	58, 66	62
Kidney				
SYN547897	Day 0	95, 98	97, 100, 102	100
	1 month (27 days)	105, 114	97, 107	102
	2 months (60 days)	90, 101	93, 94	94
	3 months (102 days)	90, 97	90, 96	93
	6 months (195 days)	94, 101	88, 89	89
	9 months (285 days)	90, 94	72, 74	73
	9 months (299 days)	97, 97	68, 77	73
	11 months (335 days)	97, 99	71, 81	76
	12 months (369 days)	109, 115	67, 73	70
SYN548263	Day 0	117, 117	116, 119, 122	119
	1 month (27 days)	113, 116	119, 122	120
	2 months (60 days)	118, 120	118, 119	119
	3 months (102 days)	120, 120	113, 116	114
	6 months (195 days)	113, 118	131, 133	132
	9 months (285 days)	104, 120	113, 115	114
	11 months (343 days)	113, 116	126, 127	126
	12 months (369 days)	116, 117	124, 127	125

There was no significant decrease (>30% as compared to the zero-time value) in the observed residue levels of SYN548264 and SYN508272 in milk and of SYN548263 in kidney when stored frozen at <-18 °C for a period of 12 months. Residues of SYN547897 in liver and kidney show a decreasing trend when stored frozen at <-18 °C for a period of 12 months.

The stability of conjugated 2,4,6-trichlorophenol was tested in animal matrices following deep frozen storage for up to 12 months (Senciuc, 2016: PTRL Europe ID P 3669 G). Individual samples of bovine muscle, liver, kidney, fat, whole milk and egg were fortified with an amount of standard solution containing conjugated 2,4,6-trichlorophenol in acetonitrile: water (80:20, v/v) at a rate of 0.62 mg/kg equivalent to 0.50 mg/kg of free 2,4,6-trichlorophenol. Triplicate samples of the animal matrices were fortified with conjugated 2,4,6-trichlorophenol and were analysed for deconjugated 2,4,6-trichlorophenol at zero-time. Duplicate samples fortified with conjugated 2,4,6-trichlorophenol were extracted after 1 month, 2, 3, 6, 11 and 12 months of frozen storage at typically ≤-18 °C and analysed for deconjugated 2,4,6-trichlorophenol. The samples were analysed for deconjugated 2,4,6-trichlorophenol using method GRM061.07A.

Table 48 Recovery of 2,4,6- trichlorophenol from stored fortified samples of animal matrices

Storage interval	Recovery (%) [0.5 mg/kg fortification]		
	Procedural	% remaining	Mean
Bovine muscle			
Day 0	96, 100	94, 94, 97	95
1 month (28 days)	84, 98	90, 94	92
2 months (56 days)	85, 88	85, 91	88
3 months (92 days)	84, 86	83, 89	86
6 months (181 days)	83, 84	91, 94	92
11 months (330 days)	99, 99	94, 95	95
12 months (358 days)	82, 85	81, 89	85
Whole milk			
Day 0	91, 94	95, 98, 99	98
1 month (28 days)	87, 93	92, 94	93
2 months (56 days)	89, 94	101, 102	102

Storage interval	Recovery (%) [0.5 mg/kg fortification]		
	Procedural	% remaining	Mean
3 months (92 days)	85, 98	94, 94	94
6 months (181 days)	73, 89	88, 94	91
9 months (330 days)	98, 101	87, 95	91
12 months (358 days)	83, 89	90, 90	90
Egg			
Day 0	94, 97	92, 93, 96	94
1 month (28 days)	77, 80	80, 83	82
2 months (56 days)	84, 89	90, 91	91
3 months (92 days)	92, 106	83, 90	87
6 months (181 days)	81, 86	84, 90	87
9 months (330 days)	97, 98	85, 88	86
12 months (358 days)	73, 75	79, 80	79
Bovine liver			
Day 0	101, 101	98, 100, 100	99
1 month (28 days)	90, 102	73, 81	77
2 months (56 days)	84, 95	81, 87	84
3 months (92 days)	83, 99	87, 94	91
6 months (181 days)	88, 88	93, 95	94
9 months (330 days)	98, 98	94, 95	95
12 months (358 days)	70, 74	78, 80	79
Bovine kidney			
Day 0	90, 93	93, 95, 97	95
1 month (28 days)	83, 89	88, 90	89
2 months (56 days)	88, 90	86, 87	87
3 months (92 days)	82, 84	74, 75	75
6 months (181 days)	84, 85	84, 84	84
9 months (330 days)	95, 97	85, 86	86
12 months (358 days)	70, 73	79, 79	79
Bovine fat			
Day 0	91, 91	75, 76, 85	79
1 month (28 days)	85, 89	73, 84	78
2 months (56 days)	90, 92	87, 90	89
3 months (92 days)	73, 78	75, 78	76
6 months (181 days)	81, 86	77, 77	77
9 months (330 days)	94, 96	77, 79	78
12 months (358 days)	85, 86	83, 85	84

Stored samples were dosed with 0.62 mg/kg free TCP equivalent. The dose consisted of 74% conjugated TCP (about 0.5 mg/kg) and 12% free TCP (about 0.1 mg/kg). Procedural recoveries were dosed with 0.50 mg/kg free TCP.

The residues of conjugated 2,4,6-trichlorophenol determined after enzymatic deconjugation as deconjugated 2,4,6-trichlorophenol showed no significant decrease (>30% as compared to the zero time value) in any of the animal commodities studied after deep frozen storage for at least 12 months. The analysis of the control samples showed that no residues of deconjugated 2,4,6-trichlorophenol were present above 25% of the LOQ.

USE PATTERN

Pydiflumetofen is a new broad spectrum fungicide of the chemical group of N-methoxy- (phenyl-ethyl)-pyrazole-carboxamide. It delivers efficacy against leaf spots (such as *Venturia sp. and Alternaria sp.*), powdery mildews and Botrytis. The Meeting received labels in Argentina, Canada and the USA. The information available to Meeting on registered uses of pydiflumetofen is summarised in table below.

Table 49 Registered uses of pydiflumetofen for crops

Crop	Country	Formulation		Application					PHI, days and/or Application timing
		Type	Conc.	Method	Rate kg ai/ha	Water L/ha	No. max	Interval, days	
Small fruit vine climbing Crops (except fuzzy kiwifruit)	Canada	SC	150 g/L	Spray	0.12-0.15 max 0.30 /season	≥500[a] ≥150[b]	2	21	PHI 14

Crop	Country	Formulation		Application					PHI, days and/or Application timing
		Type	Conc.	Method	Rate kg ai/ha	Water L/ha	No. max	Interval, days	
Grape and Small Fruit Vine Climbing Subgroup (except Fuzzy Kiwifruit), Crop Subgroup 13-07F	USA	SC	200 g/L	Spray	0.050-0.20 max 0.40 /year	≥93[a] ≥45[b]	2	14	PHI 14
	USA	SC	75 g/L	Spray	0.049-0.077 max 0.31 /year	≥140[a] ≥93[b]	4	14	PHI 14
	USA	SC	150 g/L	Spray	0.075-0.15 max 0.40 /year	≥93[ab]	2	21	PHI 14
Cucumber (greenhouse)	Canada	SC	200 g/L	Spray	0.01 kg ai/hL	400-500	2	7-14	PHI 0
Cucurbit vegetables Crops	Canada	SC	75 g/L	Spray	0.075 max 0.15 /season	≥150[a]	2	14	PHI 0
Cucurbit Vegetables, Crop Group 9	USA	SC	200 g/L	Spray	0.073-0.13 max 0.25 /year	≥93[a] ≥45[b]	2	7	PHI 0
	USA	SC	75 g/L	Spray	0.049-0.077 max 0.25 /year	≥93[a] ≥45[b]	3	7	PHI 0
	USA	SC	150 g/L	Spray	0.072-0.12 max 0.25 /year	≥93[ab]	2	7	PHI 1
Fruiting vegetables Crops	Canada	SC	75 g/L	Spray	0.075 max 0.15 /season	≥150[a]	2	7-14	PHI 0
Fruiting Vegetables, Crop Group 8-10	USA	SC	200 g/L	Spray	0.073-0.13 max 0.25 /year	≥93[a] ≥45[b]	2	7	PHI 0
	USA	SC	75 g/L	Spray	0.049-0.077 max 0.25 /year	≥140[a] ≥45[b]	3	7	PHI 0
	USA	SC	150 g/L	Spray	0.072-0.12 max 0.25 /year	≥93[ab]	2	7	PHI 0
Tomato	USA	SC	75 g/L	Spray	0.049-0.077 max 0.25 /year	≥140[a]	3	7	PHI 0
Leafy Greens Crops	Canada	SC	150 g/L	Spray	0.12-0.15 max 0.30 /season	≥150[a]	2	7-10	PHI 3
Leafy Greens, Crop Subgroup 4-16A	USA	SC	200 g/L	Spray	0.073-0.20 max 0.40 /year	≥93[a] ≥45[b]	2	7	PHI 0
Specific Leafy Greens	USA	SC	150 g/L	Spray	0.075-0.15 max 0.40 /year	≥93[ab]	2	7	PHI 0
Dried shelled peas and beans (pulses) (Crop Subgroup 6C) Crops (does not include soybeans)	Canada	SC	200 g/L	Spray	0.10-0.20 max 0.40 /season	≥100[a] ≥50[b]	2	14	PHI 14
	Canada	SC	75 g/L	Spray	0.075-0.094 max 0.19 /season	≥100[a] ≥50[b]	2	14	PHI 30[d]
Dried Shelled Peas and Beans (except Soybean), Crop Subgroup 6C	USA	SC	200 g/L	Spray	0.073-0.20 max 0.40 /year	≥93[a] ≥45[b]	2	14	PHI 14 vines and hay: PHI 0

Crop	Country	Formulation		Application					PHI, days and/or Application timing
		Type	Conc.	Method	Rate kg ai/ha	Water L/ha	No. max	Interval, days	
	USA	SC	75 g/L	Spray	0.049-0.077 max 0.31 /year max 0.16 /year for pea vines and hay	≥140[a] ≥93[b]	4	14	PHI 14 Do not feed or harvest cowpeas forage and hay.
Specific Dried Shelled Beans	USA	SC	150 g/L	Spray	0.072-0.15 max 0.40 /year	≥93[a] ≥45[b]	2	14	PHI 14 Do not apply to cowpea.
	USA	SE	75 g/L	Spray	0.055-0.075 max 0.16 /year	≥93[a] ≥45[b]	2	14	PHI 14 Not for use on cowpea cultivars intended for livestock feeding only.
Soya bean	Argentina	SC	75 g/L	Spray	0.038-0.045	≥100[a] ≥15[b]	2	15	PHI 30
Soya bean[e]	Canada	SC	200 g/L	Spray	0.10-0.20 max 0.40 /season	≥100[a] ≥50[b]	2	7-14	PHI 14
	Canada	SC	75 g/L	Spray	0.056-0.094 max 0.19 /season	≥100[a] ≥50[b]	2	14	PHI 30
Soybean[e]	USA	SC	200 g/L	Spray	0.050-0.20 max 0.40 /year	≥93[a] ≥45[b]	2	7	PHI 14
	USA	SC	75 g/L	Spray	0.049-0.075 max 0.16 /year	≥93[a] ≥45[b]	2	7	PHI 14
	USA	SE	75 g/L	Spray	0.055-0.11 max 0.24 /season	≥93[a] ≥45[b]	2	14	PHI 14 Do not apply after R6.
Potato	Canada	SC	75 g/L	Spray	0.075 max 0.225 /season	≥150[a] ≥50[b]	2	7-14	PHI 14
Potato[f]	USA	SC	200 g/L	Spray	0.050-0.13 max 0.38 /year	≥93[a] ≥45[b]	3	7	PHI 7
	USA	SC	75 g/L	Spray	0.049-0.077 max 0.31 /year	≥140[a] ≥45[b]	4	7	PHI 14
	USA	SC	150 g/L	Spray	0.075-0.12 max 0.37 /year	≥93[a] ≥45[b]	3	7	PHI 14
Tuberous and corm vegetables	Canada	SC	75 g/L	Spray	0.075 max 0.225 /season	≥150[a]	2	7-14	PHI 14
Tuberous and Corm Vegetables (except Potato), Crop Subgroup 1C[f]	USA	SC	200 g/L	Spray	0.073-0.13 max 0.38 /year	≥93[a] ≥45[b]	3	7	PHI 7
	USA	SC	75 g/L	Spray	0.049-0.077 max 0.31 /year	≥93[a] ≥45[b]	4	7	PHI 14
	USA	SC	150 g/L	Spray	0.12 max 0.37 /season	≥93[a] ≥45[b]	3	7	PHI 14
Leaf Petioles Vegetables (Crop group 22B) Crops	Canada	SC	150 g/L	Spray	0.12-0.15 max 0.30 /season	≥150[a]	2	7-10	PHI 3
Leaf Petioles Vegetables, Crop Subgroup 22B	USA	SC	200 g/L	Spray	0.073-0.20 max 0.40 /year	≥93[a] ≥45[b]	2	7	PHI 0

Crop	Country	Formulation		Application					PHI, days and/or Application timing
		Type	Conc.	Method	Rate kg ai/ha	Water L/ha	No. max	Interval, days	
Specific Leaf Petioles	USA	SC	150 g/L	Spray	0.075-0.15 max 0.40 /year	≥93[ab]	2	7	PHI 0
Barley Wheat (spring, winter, and durum)	Canada	SC	200 g/L	Spray	0.060-0.20	≥150[a] ≥50[b]	1	14	Do not apply after Feekes 10.54 (flowering completed, kernel watery ripe) or BBCH 71 [g]
Cereal grains Crops	Canada	SC	75 g/L	Spray	0.056	≥100[1)] ≥50[2)]	2	14	PHI 45 No later than BBCH 55[h]
Cereal Grains	USA	SC	200 g/L	Spray	0.055-0.20 max 0.35 /year	≥93[a] ≥45[b]	1[c]	14	Do not apply after full head emergence (Feekes 10.54)[g]
	USA	SE	75 g/L	Spray	0.038-0.075 max 0.15 /year	≥93[a] ≥45[b]	2	14	Do not apply after full head emergence (Feekes 10.54)[g] Do not apply within 14 days prior grazing.
Quinoa[i]	USA	SC	200 g/L	Spray	0.10-0.20 max 0.35 /year	≥93[a] ≥45[b]	1	14	PHI 30
	USA	SE	75 g/L	Spray	0.058-0.075 max 0.16 /year	≥93[a] ≥45[b]	2	14	PHI 30
Corn (field, pop, specialty, sweet and seed)	Canada	SC	200 g/L	Spray	0.10 max 0.20 /season	≥200[a] ≥50[b]	2	-	PHI 30 for grain and stover[g] PHI 7 for sweet corn
	Canada	SC	75 g/L	Spray	0.056-0.094 max 0.15 /season	≥200[a] ≥50[b]	2	14	PHI 30 for grain and stover[j] PHI 14 for sweet corn
Corn (field corn, popcorn (including for seed production))	USA	SC	200 g/L	Spray	0.050-0.20 max 0.25 /year	≥93[a] ≥45[b]	1[c]	7	PHI 30[g]
	USA	SE	75 g/L	Spray	0.055-0.075 max 0.25 /year	≥93[a] ≥45[b]	3	7	PHI 30
Sweet corn (including for seed production	USA	SC	200 g/L	Spray	0.050-0.13 max 0.25 /year	≥93[a] ≥45[b]	2	7	PHI 7
	USA	SE	75 g/L	Spray	0.055-0.075 max 0.25 /year	≥93[a] ≥45[b]	3	7	PHI 14
Canola (Crop subgroup 20A) Crops	Canada	SC	200 g/L	Spray	0.10-0.20 max 0.33 /season	≥100[a] ≥50[b]	2	-	PHI 30
Canola (Rapeseed, Crop Subgroup 20A)	USA	SC	200 g/L	Spray	0.10-0.20 max 0.33 /year	≥93[a] ≥45[b]	1[c]	14	PHI 30
	USA	SC	75 g/L	Spray	0.049-0.077 max 0.077 /year	≥93[a] ≥45[b]	1	-	PHI 30
	USA	SE	75 g/L	Spray	0.057-0.075 max 0.075 /year	≥93[a] ≥45[b]	1	-	PHI 30
Peanut	Argentina	SC	75 g/L	Spray	0.045-0.053	≥100[a] ≥15[b]	2	15	PHI 20
Peanut	Canada	SC	200 g/L	Spray	0.025-0.050 max 0.20 /season	≥100[a] ≥50[b]	4	14-21 or 21-28[k]	PHI 14

Crop	Country	Formulation		Application					PHI, days and/or Application timing
		Type	Conc.	Method	Rate kg ai/ha	Water L/ha	No. max	Interval, days	
Peanut	USA	SC	200 g/L	Spray	0.025-0.050 max 0.20 /year	≥93[a] ≥45[b]	4	14-21 or 21-28[k]	PHI 14
	USA	SE	75 g/L	Spray	0.050 max 0.20 /year	≥93[a] ≥45[b]	4	21	PHI 14

[a] ground application,

[b] aerial application

[c] Do not make more than 1 application at the maximum application rate per year.

[d] Dry pea hay and vines may be fed or harvested 14 days after last application. Do not make more than one application to dry pea hay. Do not feed dried pea vines to livestock.

[e] Do not feed soybean forage, hay and silage to livestock.

[f] Do not harvest tops for feed or food.

[g] For harvest of forage and hay, one application with a minimum PHI of 7days is required.

[h] Do not apply within 30 days of harvest for forage and hay. Do not make more than one application for harvest of forage and hay.

[i] Do not feed treated quinoa forage or hay to livestock. Do not graze livestock on treated quinoa.

[j] Do not apply within 30 days of harvest for forage.

[k] Apply 0.025 kg ai/ha on a 14-21 day interval. Application at the 0.050 kg ai/ha may be made on a 21-28 day interval.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on pydiflumetofen supervised field trials for the following crops.

Group	Commodity	Table
Berries and other small fruits	Grapes	Table 50
Fruiting vegetables, Cucurbits	Cucumber	Table 51
	Squash, summer	Table 52
	Cantaloupe	Table 53
Fruiting vegetables, other than Cucurbits	Tomato	Table 54
	Pepper	Table 55
Leafy vegetables	Lettuce, Head	Table 56
(including Brassica leafy vegetables)	Lettuce, Leaf	Table 57
	Spinach	Table 58
Pulses	Beans (dry)	Table 59
	Soya bean (dry)	Table 60
	Peas (dry)	Table 61
Root and tuber vegetables	Potato	Table 62
Stalk and stem vegetables	Celery	Table 63
Cereal grains	Wheat	Table 64
	Barley	Table 65
	Oats	Table 66
	Maize	Table 67
	Sweet corn	Table 68
Oilseed	Rape seed	Table 69
	Peanut	Table 70
Legume Animal feeds	Pea vines and hay	Table 71
	Peanut hay	Table 72
	Soya bean forage and hay	Table 73

Group	Commodity	Table
Straw, fodder and forage of cereal grains	Barley straw	Table 74
	Barley hay	Table 75
	Maize forage	Table 76, 77
	Maize fodder	Table 78, 79
	Oats forage and hay	Table 80
	Oats straw	Table 81
	Wheat forage and hay	Table 82
	Wheat straw	Table 83

Pydiflumetofen formulation was applied as a foliar treatment. Each of the field trial sites generally consisted of an untreated control plot and a treated plot. Application rates and residue concentrations have generally been rounded to two significant figures.

Residue values from the trials, which have been used for the estimation of maximum residue levels, STMRs and HRs, are underlined.

Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Date of analyses and duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except when residues were found in samples from control plots. Residue data are not corrected for percent recovery.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most field reports provided data on the applicators used, plot size, field sample size and sampling date.

Berries and other small fruits

Small fruit vine climbing

Grapes

The Meeting received 12 trials (10 at harvest trials, two decline trials) on grapes which were conducted in the USA (Salzman, 2015: TK0103853). In each of these trials, a SC formulations (200 g ai/L) was applied twice to grapes as a foliar treatment at a nominal rate of 0.20 kg ai/ha. The first application was made 14 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, Non-Ionic Surfactant (NIS) or Crop Oil Concentrate (COC). At each trial, fruits were taken 14 ± 1 days after last application (DALA). In the decline trials additional samples were collected at 7, 10, 18 and 21 ± 1 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from grapes as 97% at 0.01 mg/kg fortified and 106% at 5.0 mg/kg fortified. Grape samples were stored at -10 °C or below for a maximum of 11 months between sampling and analysis.

Table 50 Residues of pydiflumetofen on grapes from supervised trials in the USA

Grapes country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.20 0.40 /year	[b] 93 [c] 45		2	14		
USA, 2013 Dundee, NY (Concord) Outdoor	SC	0.20 0.20	94 94	81-81 81-83	2	14	0.35, 0.39 (<u>0.37</u>)	TK0103853 Mean recovery for pydiflumetofen: 97% (n=6) at 0.01 mg/kg 106% (n=6) at 5.0 mg/kg
USA, 2013 Breinigsville, PA (Corot Noir) Outdoor	SC	0.21 0.21	727 727	83 85	2	7 9 14 19 21	0.48 0.30 0.32, 0.45 (<u>0.39</u>) 0.31 0.22	
USA, 2013 Paso Robles, CA (Petite Sirah) Outdoor	SC	0.22 0.21	75 56	85 85	2	14	<0.01, <0.01 (<0.01)	Sampling to

Grapes country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[a]	Ref
USA, 2013 Porterville, CA (Thompson) Outdoor	SC	0.20 0.20	477 589	83-85 83-85	2	14	0.15, 0.16 (0.16)	analysis: 245-322 days
USA, 2013 Madera, CA[d] (Merlot) Outdoor	SC	0.20 0.20	757 748	88-89 88-89	2	14	0.57, 0.65 (0.61)	
USA, 2013 Madera, CA[d] (Thompson Seedless) Outdoor	SC	0.20 0.20	748 729	88-89 88-89	2	14	0.33, 0.42 (0.37)	
USA, 2013 Kingsburg, CA (Thompsons) Outdoor	SC	0.20 0.20	542 542	83-85 85-89	2	14	0.045, 0.060 (0.053)	
USA, 2013 Templeton, CA (Roussanne) Outdoor	SC	0.20 0.20	664 617	79-81 81-85	2	14	0.46, 0.55 (0.50)	
USA, 2013 Lindsay, CA (Redglobe) Outdoor	SC	0.21 0.20	571 552	81-83 85-89	2	7 10 14 18 21	0.22 0.21 0.19, 0.21 (0.20) 0.15 0.16	
USA, 2013 Kerman, CA (Thompson Seedless) Outdoor	SC	0.21 0.20	851 842	85-85 85-85	2	14	0.21, 0.38 (0.29)	
USA, 2013 Ephrata, WA (White Riesling) Outdoor	SC	0.21 0.21	926 935	84-85 86-87	2	14	0.69, 0.85 (0.77)	
USA, 2013 Underwood, WA (Sauvignon Blanc) Outdoor	SC	0.21 0.20	851 842	83-83 85-85	2	13	0.13, 0.19 (0.16)	

Portion analysed: fruit

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

[d] The trial sites were located adjacent to each other, and treated and harvested on the same day.

Address: CIMIS Madera II station 188 ~2.7 miles

Fruiting vegetables, Cucurbits

Fruiting vegetables, Cucurbits – Cucumber and Summer squashes

Cucumber

The Meeting received 10 trials (nine at harvest trials, one decline trial) on field/greenhouse grown cucumber which were conducted in the USA (Homa and Leonard, 2015: TK0174260). In each of these trials, a SC formulations (200 g ai/L) was applied twice to cucumber as a foliar treatment at a nominal rate of 0.13 kg ai/ha. The first application was made 6 or 7 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS. At each trial, fruits were taken 0 DALA. In the decline trials additional samples were collected at 1, 3, 6 and 9 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from cucumber fruits as 82% at 0.01 mg/kg fortified, 98% at 0.1 mg/kg fortified and 100% at 0.5 mg/kg fortified. Cucumber fruit samples were stored at ca -20 °C for a maximum of 14 months between sampling and analysis.

Table 51 Residues of pydiflumetofen on cucumber from supervised trials in the USA

Cucumber country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	no.			
GAP, USA	SC	0.13 0.25 / year	[b] 93 [c] 45		2	0		
Pickling cucumber								TK0174260
USA, 2013 Holt, MI (Expedition) Outdoor	SC	0.12 0.13	561 573	Fruiting Fruiting	2	0	0.14, 0.15 (0.14)	Mean recovery for pydiflumetofen: 82% (n=5) at 0.01 mg/kg 98% (n=3) at 0.1 mg/kg 100% (n=3) at 0.5 mg/kg Sampling to analysis: 55-431 days
USA, 2013 Salisbury, MD (Little Leaf) Outdoor	SC	0.13 0.13	427 429	First mature fruits Mature fruits	2	0	0.11, 0.11 (0.11)	
USA, 2013 Citra, FL (Supremo) Outdoor	SC	0.13 0.13	655 671	Fruiting Fruiting	2	0 1 3 6 9	0.11, 0.12 (0.12) 0.10, 0.11 (0.11) 0.094, 0.096 (0.095) 0.090, 0.098 (0.094) 0.067, 0.075 (0.071)	
Slicing cucumber								
USA, 2013 Tifton, GA (Straight Eight) Outdoor	SC	0.13 0.13	408 401	Fruiting Fruiting	2	0	0.11, 0.12 (0.11)	
USA, 2014 Uvalde, TX (Stone Wall) Outdoor	SC	0.13 0.13	379 379	Fruits mostly 3 cm 1 mature fruit per plant	2	0	0.18, 0.20 (0.19)	
USA, 2013 Clinton, NC (Poinsett 76) Outdoor	SC	0.13 0.13	411 403	Fruiting Fruiting	2	0	0.16, 0.16 (0.16)	
USA, 2013 Arlington, WI (Marketmore 76) Outdoor	SC	0.13 0.13	452 474	Fruiting Fruiting	2	0	0.11, 0.11 (0.11)	
English cucumber								
USA, 2013 Raleigh, NC (Cumlaude) Indoor (greenhouse)	SC	0.12 0.13	621 626	Fruiting Fruiting	2	0	0.11, 0.12 (0.11)	
USA, 2013 Parlier, CA (Manar) Indoor (greenhouse)	SC	0.13 0.13	568 580	Blooming/ Fruiting Blooming/ Fruiting	2	0	0.26, 0.27 (0.26)	
USA, 2013 Fayetteville, AR (Sweet Success) Indoor (greenhouse)	SC	0.14 0.13	462 449	Fruiting Fruiting	2	0	0.20, 0.26 (0.23)	

Portion analysed: fruit

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

Squash, summer

The Meeting received six trials (five at harvest trials, one decline trial) on summer squash which were conducted in the USA (Homa and Leonard, 2015: TK0174261). In each of these trials, a SC formulations (200 g ai/L) was applied twice to summer squash as a foliar treatment at a nominal rate of 0.13 kg ai/ha. The first application was made 6 or 7 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, fruits were taken 0 DALA. In the decline trials additional samples were collected at 1, 4, 6 and 9 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from summer squash fruits as 100% at 0.01 mg/kg fortified, 98% at 0.1 mg/kg fortified and 101% at 0.25 mg/kg fortified. Summer squash fruit samples were stored at ca -20 °C for a maximum of 14 months between sampling and analysis.

Table 52 Residues of pydiflumetofen on summer squash from supervised trials in the USA

Summer squash country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	no.			
GAP, USA	SC	0.13 0.25 / year	[b] 93 [c] 45		2	0		
USA, 2013 Arlington, WI (Lioness) Outdoor	SC	0.12 0.13	450 459	Fruiting Fruiting	2	0	0.13, 0.18 (0.16)	TK0174261 Mean recovery for pydiflumetofen: 100% (n=6) at 0.01 mg/kg 98% (n=6) at 0.1 mg/kg 101% (n=3) at 0.25 mg/kg Sampling to analysis: 49-426 days
USA, 2013 Tifton, GA (Early Prolific Straightneck Squash) Outdoor	SC	0.13 0.12	402 390	Fruiting Fruiting	2	0	0.052, 0.070 (0.061)	
USA, 2013 Citra, FL (Gentry) Outdoor	SC	0.13 0.13	661 670	Blooming/ Fruiting Fruiting	2	0	0.028, 0.18 (0.10)	
USA, 2013 Clinton, NC[d] (Fortune) Outdoor	SC	0.13 0.12	471 462	Fruiting (9 days) Fruit and flowers (70 to 80% maturity)	2	0	0.21, 0.21 (0.21)	
		0.13 0.12 0.13	471 462 475	Fruiting (9 days) Fruit and Flowers (6 days) Mature fruit	3	0	0.16, 0.18 (0.17)	
USA, 2013 Freeville, NY (Multipik) Outdoor	SC	0.13 0.12	645 656	Bloom and fruiting Bloom and fruiting	2	0	0.18, 0.18 (0.18)	
USA, 2013 Davis, CA (Zucchini, Black Beauty) Outdoor	SC	0.12 0.12	410 410	Fruiting Fruiting	2	0 1 4 6 9	0.049, 0.062 (0.056) 0.037, 0.051 (0.044) 0.035, 0.039 (0.037) 0.019, 0.023 (0.021) 0.027, 0.034 (0.031)	

Portion analysed: fruit

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

[d] Summer squash samples developed slowly due to cool weather, resulting in 70 to 80% maturity at the first harvest of samples. An additional

application and additional samples were added in order to collect samples at the proper maturity.

Fruiting vegetables, Cucurbits – Melons, Pumpkins and Winter squashes

Cantaloupe

The Meeting received six trials (five at harvest trials, one decline trial) on cantaloupe which were conducted in the USA (Homa and Leonard, 2015: TK0174258). In each of these trials, a SC formulations (200 g ai/L) was applied twice to cantaloupe as a foliar treatment at a nominal rate of 0.13 kg ai/ha. The first application was made 6 to 8 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS. At each trial, fruits were taken 0 DALA. In the decline trials additional samples were collected at 1, 2, 6 and 9 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from cantaloupe fruits as 103% at 0.01 mg/kg fortified, 119% at 0.1 mg/kg fortified and 107% at 0.5 mg/kg fortified. Cantaloupe fruit samples were stored at ca -20 °C for a maximum of 12 months between sampling and analysis.

Table 53 Residues of pydiflumetofen on cantaloupe from supervised trials in the USA

Cantaloupe country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	no.			
GAP, USA	SC	0.13 0.25 / year	[b] 93 [c] 45		2	0		
USA, 2013 Salisbury, MD (Athena) Outdoor	SC	0.12 0.13	463 468	Fully sized green fruits First ripe fruits	2	0	0.087, 0.14 (0.11)	TK0174258 Mean recovery for pydiflumetofen: 103% (n=3) at 0.01 mg/kg 119% (n=3) at 0.1 mg/kg 107% (n=3) at 0.5 mg/kg Sampling to analysis: 28-359 days
USA, 2013 Holt, MI (Athena) Outdoor	SC	0.13 0.12	571 540	Fruiting Fruiting	2	0	0.14, 0.18 (0.16)	
USA, 2013 Davis, CA (Hales Best Jumbo) Outdoor	SC	0.13 0.12	415 412	Fruiting Fruiting	2	0	0.036, 0.098 (0.067)	
USA, 2013 Holtville, CA (XP6328) Outdoor	SC	0.13 0.13	586 575	Bud, bloom, fruit Fruiting	2	0	0.10, 0.19 (0.15)	
USA, 2013 Parlier, CA (Durango) Outdoor	SC	0.14 0.13	437 441	Fruiting Fruiting	2	0	0.074, 0.082 (0.078)	
USA, 2014 Uvalde, TX (Primo) Outdoor	SC	0.13 0.13	379 382	1 ripe melon per 100 ft^2 Mature melons	2	0 1 2 6 9	0.14, 0.20 (0.17) 0.045, 0.064 (0.055) 0.044, 0.060 (0.052) 0.047, 0.057 (0.052) 0.045, 0.046 (0.046)	

Portion analysed: whole fruit

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

Fruiting vegetables, other than Cucurbits

Tomato

The Meeting received 12 trials (10 at harvest trials, two decline trials) on tomato which were conducted in the USA (Salzman, 2015: TK0163552). In each of these trials, a SC formulations (200 g ai/L) was applied twice to tomato as a foliar treatment at a nominal rate of 0.13 kg ai/ha. The first application was made 7 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, fruits were taken 0 DALA. In the decline trials additional samples were collected at 3, 7, 10 and 14 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from tomato fruit as 110% at 0.01 mg/kg fortified and 114% at 5.0 mg/kg fortified. Tomato fruit samples were stored at -10 °C or below for a maximum of 17 months between sampling and analysis.

Table 54 Residues of pydiflumetofen on tomato from supervised trials in the USA

Tomato country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[a]	Ref
GAP, USA	SC	0.13 0.25 / year	[b] 93 [c] 45		2	0		
Tomato								TK0163552
USA, 2013 North Rose, NY (Mountain Spring) Outdoor	SC	0.12 0.13	274 384	84-85 85-86	2	0	0.067, 0.097 (0.082)	Mean recovery for pydiflumetofen: 110% (n=6) at 0.01 mg/kg 114% (n=6) at 5.0 mg/kg Sampling to analysis: 221-511 days
USA, 2013 Athens, GA (Amelia) Outdoor	SC	0.12 0.12	224 234	81-82 85-86	2	0	0.11, 0.12 (0.11)	
USA, 2013 Oviedo, FL (Better Boy) Outdoor	SC	0.12 0.12	47 47	75 81	2	0	0.026, 0.033 (0.030)	
USA, 2013 Verona, WI (Mountain Fresh) Outdoor	SC	0.13 0.13	271 253	81-84 81-85	2	0	0.074, 0.077 (0.075)	
USA, 2013 Madera, CA (Quality) Outdoor	SC	0.12 0.13	281 29	88-89 88-89	2	0	0.066, 0.089 (0.077)	
USA, 2013 Porterville, CA[d] (7883) Outdoor	SC	0.12 0.12	309 318	87-89 87-89	2	0	0.15, 0.16 (0.16)	
USA, 2013 Paso Robles, CA (Galilea) Outdoor	SC	0.13 0.12	196 187	86 87-89	2	0 3 7 10 14	0.19, 0.19 (0.19) 0.23 0.14 0.066 0.055	
USA, 2013 King City, CA (Q47) Outdoor	SC	0.12 0.12	290 299	84-89 84-89	2	0 3 7 10 14	0.069, 0.082 (0.076) 0.052 0.038 0.083 0.050	
USA, 2013 Kettleman City, CA (Galilea) Outdoor	SC	0.13 0.12	393 374	83-87 83-87	2	0	0.035, 0.050 (0.043)	
USA, 2013 Porterville, CA[e] (AB2, Roma type) Outdoor	SC	0.12 0.12	318 318	87-89 86-89	2	0	0.11, 0.15 (0.13)	
Cherry tomato								

Tomato country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
USA, 2013 Oviedo, FL (Sweet 100) Outdoor	SC	0.12 0.12	281 271	81 86	2	0	0.22, 0.31 (0.27)	
USA, 2013 San Ardo, CA (Naomi) Outdoor	SC	0.12 0.13	209 299	85-89 87-89	2	0	0.18, 0.22 (0.20)	

Portion analysed: fruit

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

[d] Address: Porterville CIMIS Weather Station #169 ~6.2 miles, Application dates (1st): 30 Oct 2013

[e] Address: Porterville CIMIS Weather Station #169 ~5.4 miles, Application dates (1st): 21 Aug 2013

Pepper and pepper-like commodities

Peppers

The Meeting received nine trials (eight at harvest trials, one decline trial) on pepper which were conducted in the USA (Salzman, 2015: TK0163552). In each of these trials, a SC formulations (200 g ai/L) was applied twice to pepper as a foliar treatment at a nominal rate of 0.13 kg ai/ha. The first application was made 7 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, fruits were taken 0 DALA. In the decline trials additional samples were collected at 3, 7, 11 and 14 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from bell pepper fruit as 118% at 0.01 mg/kg fortified and 118% at 5.0 mg/kg fortified. Pepper fruit samples were stored at -10 °C or below for a maximum of 12 months between sampling and analysis.

Table 55 Residues of pydiflumetofen on pepper from supervised trials in the USA

Pepper country, year (variety)	Application					DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.13 0.25 / year	2) 93 3) 45		2	0		
Bell pepper								TK0163552
USA, 2013 Athens, GA (Aristotle) Outdoor	SC	0.12 0.12	187 196	78-79 78-79	2	0	0.080, 0.081 (0.081)	Mean recovery for pydiflumetofen: 118% (n=3) at 0.01 mg/kg 118% (n=3) at 5.0 mg/kg Sampling to analysis: 238-361 days
USA, 2013 Oviedo, FL (Orange Bell) Outdoor	SC	0.12 0.12	281 281	71-71 81-81	2	0	0.30, 0.43 (0.37)	
USA, 2013 Verona, WI (Excursion) Outdoor	SC	0.12 0.12	253 253	73-77 73-75	2	0	0.14, 0.19 (0.17)	
USA, 2013 Raymondville, TX (Camelot) Outdoor	SC	0.13 0.12	47 37	88-89 88-89	2	0	0.058, 0.065 (0.062)	
USA, 2013 Madera, CA (Cypress) Outdoor	SC	0.13 0.12	281 281	88-89 88-89	2	0	0.25, 0.28 (0.26)	
USA, 2013 Arroyo Grande, CA (Crusader) Outdoor	SC	0.13 0.13	243 243	75 76	2	0 3 7 11 14	0.063, 0.090 (0.076) 0.057 0.065 0.030 0.046	

Pepper country, year (variety)	Application					DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
Non-bell pepper								
USA, 2013 Larned, KS (Tam Hot Jalapeno) Outdoor	SC	0.12 0.13	206 206	71-72 88-89	2	0	0.087, 0.088 (0.088)	
USA, 2013 San Angelo, TX (Grande Hybrid Jalapeno) Outdoor	SC	0.13 0.12	187 187	85-87 89-89	2	0	0.11, 0.16 (0.14)	
USA, 2013 King City, CA (Serrano) Outdoor	SC	0.12 0.12	299 309	84-88 87-89	2	0	0.23, 0.28 (0.26)	

Portion analysed: fruit

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

Leafy vegetables (including Brassica leafy vegetables)

Leafy greens

Lettuce, Head

The Meeting received eight trials (all at harvest trials) on head lettuce which were conducted in the USA (Oakes, 2015: TK0163598). In each of these trials, a SC formulations (200 g ai/L) was applied twice to head lettuce as a foliar treatment at a nominal rate of 0.20 kg ai/ha. The first application was made 7 to 9 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, head lettuce without wrapper leaves and head lettuce with wrapper leaves samples were taken 0 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from head lettuce without wapper leaves as 96% and from head lettuce with wapper leaves as 95% at 0.01–10 mg/kg fortified. Head lettuce samples were stored at ca -18 °C for a maximum of 16 months between sampling and analysis.

Table 56 Residues of pydiflumetofen on head lettuce from supervised trials in the USA

Head lettuce country, year (variety)	Application					DALA Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.		Without wrapper leaves With wrapper leaves	
GAP, USA	SC	0.20 0.40 / year	[b] 93 [c] 45		2	0		
USA, 2013 Lyons, NY (Crispino) Outdoor	SC	0.20 0.20	281 281	47-48 49	2	0	0.31, 0.67 (0.49) 0.68, 0.89 (0.78)	TK0163598 Mean recovery for pydiflumetofen: 95% (n=3) at 0.01 mg/kg 92% (n=2) at 0.1 mg/kg 100% (n=3) at 1.0-10 mg/kg for head lettuce without wrapper leaves, 99% (n=3) at 0.01 mg/kg 79% (n=2) at 0.1 mg/kg
USA, 2013 Paso Robles, CA (Vandenberg) Outdoor	SC	0.20 0.21	234 234	48 49	2	0	0.018, 0.058 (0.038) 2.2, 2.5 (2.4)	
USA, 2013 King City, CA [d] (Declaration) Outdoor	SC	0.21 0.20	187 196	48 48	2	0	<0.010, <0.010 (<0.010) 0.44, 0.59 (0.51)	
USA, 2013 Madera, CA (Vandenberg) Outdoor	SC	0.20 0.20	234 234	46-47 48-49	2	0	0.082, 0.085 (0.084) 2.3, 2.8 (2.6)	
USA, 2013 Kerman, CA	SC	0.21 0.21	187 187	47-49 48-49	2	0	0.25, 0.30 (0.28) 4.2, 4.8 (4.5)	

Head lettuce country, year (variety)	Application					DALA Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.		Without wrapper leaves With wrapper leaves	
(Vail) Outdoor								102% (n=3) at 1.0-10 mg/kg for head lettuce with wrapper leaves Sampling to analysis: 219-498 days for head lettuce without wrapper leaves, 218-493 days for head lettuce with wrapper leaves
USA, 2013 King City, CA[e] (Vandenberg) Outdoor	SC	0.20 0.20	262 262	45-47 47-49	2	0	0.015, 0.062 (0.039) 1.2, 1.2 (1.2)	
USA, 2013 Visalia, CA* (Regency) Outdoor	SC	0.20 0.20	224 224	45-47 48-49	2	0	0.11, 0.17 (0.14)* 3.0, 3.0 (3.0)*	
USA, 2014 Oviedo, FL (Great Lakes) Outdoor	SC	0.20 0.20	262 262	48-48 49-49	2	0	0.050, 0.053 (0.052) 2.1, 2.4 (2.3)	

Portion analysed: Without wrapper leaves (up), With wrapper leaves (down)

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

[d] Address: King City CIMIS Weather Station#113 ~20 miles,, Application dates (1st): 13 Jun 2013

[e] Address: King City CIMIS Weather Station#113 ~5.9 miles,, Application dates (1st): 23 May 2013

* Samples from this trial were inadvertently switched at some point in the process. However based on data trends in the study, data in this study report residue values consistent with data trends. Values are based on the average of original and confirmation results.

Lettuce, Leaf

The Meeting received eight trials (seven at harvest trials, one decline trial) on leaf lettuce which were conducted in the USA (Oakes, 2015: TK0163598). In each of these trials, a SC formulations (200 g ai/L) was applied twice to head lettuce as a foliar treatment at a nominal rate of 0.20 kg ai/ha. The first application was made 7 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, leaves were taken 0 DALA. In the decline trials additional samples were collected at 1, 3, 7 and 10 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from leaf lettuce as 105% at 0.01 mg/kg fortified, 87% at 0.1 mg/kg fortified and 85% at 1.0–20 mg/kg fortified. Leaf lettuce samples were stored at ca -18 °C for a maximum of 15 months between sampling and analysis.

Table 57 Residues of pydiflumetofen on leaf lettuce from supervised trials in the USA

Leaf lettuce country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.20 0.40 / year	[b] 93 [c] 45		2	0		
USA, 2013 Lyons, NY (Tropicana) Outdoor	SC	0.20 0.20	281 281	47-48 49	2	0	3.8, 5.0 (4.4)	TK0163598
USA, 2013 Hobe Sound, FL (Sanibel) Outdoor	SC	0.21 0.21	243 234	46-47 47-49	2	0	5.4, 5.7 (5.5)	Mean recovery for pydiflumetofen: 105% (n=2) at 0.01 mg/kg 87% (n=2) at 0.1 mg/kg 85% (n=3) at 1.0-20 mg/kg
USA, 2013 King City, CA[d] (Sunbelt) Outdoor	SC	0.21 0.21	243 253	46-48 48-49	2	0	1.6, 1.8 (1.7)	
USA, 2013 Madera, CA[e] (Green Star)	SC	0.20 0.20	234 234	46-47 47-48	2	0	7.0, 8.5 (7.7)	Sampling to

Leaf lettuce country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
Outdoor								analysis: 160-470 days
USA, 2013 Paso Robles, CA (Green Thunder) Outdoor	SC	0.20 0.20	187 187	45-48 46-49	2	0	3.1, 3.8 (3.5)	
USA, 2013 Madera, CA[f] (Green Star) Outdoor	SC	0.20 0.20	234 234	46-47 47-48	2	0 1 3 7 10	9.1, 10 (9.7) 7.6 5.7 2.4 1.7	
USA, 2013 Porterville, CA (Red Sails) Outdoor	SC	0.20 0.20	224 224	47-49 47-49	2	0	9.6, 12 (11)	
USA, 2013 King City, CA[g] (Red Sails) Outdoor	SC	0.20 0.20	253 253	46-48 47-49	2	0	11, 13 (12)	

Portion analysed: leaves

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

[d] Address: King City CIMIS Weather Station#113 ~20 miles, Application dates (1st): 29 Jun 2013

[e] Address: CIMIS Madera II station 188 ~2.7 miles, Application dates (1st): 08 May 2013

[f] Address: CIMIS Madera II station 188 ~2.7 miles, Application dates (1st): 08 May 2013

[g] Address: King City CIMIS Weather Station#113 ~9.9 miles, Application dates (1st): 03 Jul 2013

Spinach

The Meeting received eight trials (seven at harvest trials, one decline trial) on spinach which were conducted in the USA (Oakes, 2015: TK0163598). In each of these trials, a SC formulations (200 g ai/L) was applied twice to spinach as a foliar treatment at a nominal rate of 0.20 kg ai/ha. The first application was made 7 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, leaves were taken 0 DALA. In the decline trials additional samples were collected at 1, 4, 7 and 10 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from spinach leaves as 90% at 0.01 mg/kg fortified, 102% at 0.1 mg/kg fortified and 94% at 1.0–20 mg/kg fortified. Spinach samples were stored at ca -18 °C for a maximum of 18 months between sampling and analysis.

Table 58 Residues of pydiflumetofen on spinach from supervised trials in the USA

Spinach country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.20 0.40 / year	[b] 93 [c] 45		2	0		
USA, 2013 Lyons, NY (Space F1) Outdoor	SC	0.20 0.20	281 281	47-48 48-49	2	0	8.7, 9.7 (9.2)	TK0163598 Mean recovery for pydiflumetofen: 90% (n=3) at 0.01 mg/kg 102% (n=2) at 0.1 mg/kg 94% (n=4) at 1.0-20 mg/kg
USA, 2014 Athens, GA (Bloomsdale) Outdoor	SC	0.20 0.20	262 262	45-46 46-47	2	0	13, 13 (13)	
USA, 2013 Elko, SC (Bloomsdale) Outdoor	SC	0.21 0.21	140 140	45-47 45-49	2	0	14, 17 (16)	

Spinach country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
USA, 2013 Uvalde, TX (DMC 66-07) Outdoor	SC	0.20 0.20	150 140	47- 4748- 49	2	0	12, 15 (13)	Sampling to analysis: 86-541 days
USA, 2013 San Angelo, TX (Bloomsdale Longstanding) Outdoor	SC	0.20 0.20	131 131	47-47 49-49	2	0	14, 15 (14)	
USA, 2013 Jerome, ID (Unipack 151) Outdoor	SC	0.20 0.20	178 178	47-48 48-49	2	0	12, 13 (12)	
USA, 2013 King City, CA[d] (Violin F1) Outdoor	SC	0.21 0.20	47 47	47-49 47-49	2	0	7.1, 7.9 (7.5)	
USA, 2013 King City, CA[e] (Interceptor) Outdoor	SC	0.20 0.20	187 187	43-45 47	2	0 1 4 7 10	9.7, 9.8 (9.7) 6.3 1.6 0.79 0.54	

Portion analysed: leaves

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

[d] Address: King City CIMIS Weather Station#113 ~6.5 miles, Application dates (1st): 31 May 2013

[e] Address: King City CIMIS Weather Station#113 ~20 miles, Application dates (1st): 29 Jun 2013

Pulses

Dry beans

Beans (dry)

The Meeting received 10 trials (eight at harvest trials, two decline trials) on beans (dry) which were conducted in Canada (Sagan, 2015: TK0103849) and the USA (Mäyer, 2015: TK0163595). In each of these trials, pydiflumetofen was applied to beans as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. Two applications at a nominal rate of 0.20 kg ai/ha were made. The first application was made 14 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, seed samples were taken 14 ± 3 DALA. In the decline trials additional seed samples were collected at 7, 10, 17 and 21 ± 1 DALA. Some samples were allowed to dry for 3–5 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from dry bean seed was 99% at 0.01–1.0 mg/kg fortified (Canada) and 92% at 0.01–0.1 mg/kg fortified (USA). Dry bean seed samples were stored at -10 °C or below (Canada) or at -18 °C or below (USA) for a maximum of 13 months between sampling and analysis.

Table 59 Residues of pydiflumetofen on dry bean from supervised trials in Canada and the USA

Beans (dry) country, year (variety)	Application					DALA Days	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.20 0.40 / year	[c] 93 [d] 45		2	14		
USA, 2013 Campbell, MN (HMS Medalist Navy Bean) Outdoor	SC	0.20 0.20	187 187	77-77 80-80	2	7 10 14 17 21	<0.01 <0.01 <0.01, <0.01 (<0.01) <0.01 <0.01	TK0163595 Mean recovery for pydiflumetofen: 89% (n=2) at

Beans (dry) country, year (variety)	Application					DALA	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.	Days		
	EC	0.20 0.20	187 187	77-77 80-80	2	14	<0.01, <0.01 (<0.01)	0.01 mg/kg 96% (n=2) at 0.1 mg/kg
USA, 2013 Eaton, CO (Bill Z) Outdoor	SC	0.20 0.21	103 112	75-78 85-88	2	14	0.22, 0.26 (0.24) c 0.022	
	EC	0.21 0.21	103 112	75-78 85-88	2	14	0.21, 0.22 (0.21) c 0.022	Sampling to analysis: 301-390 days
USA, 2013 Smithfield, UT (Remington) Outdoor	SC	0.22 0.21	178 168	82-82 87-87	2	15	0.031, 0.090 (0.060)	
	EC	0.21 0.21	178 168	82-82 87-87	2	15	<0.01, 0.058 (0.034)	
USA, 2013 Porterville, CA (Ford Hook 242) Outdoor	SC	0.21 0.20	47 47	80-81 85-89	2	11	<0.01, <0.01 (<0.01)	
	EC	0.20 0.20	47 47	80-81 85-89	2	11	0.017, 0.019 (0.018)	
USA, 2013 Payette, ID (Eclipse Black Bean) Outdoor	SC	0.21 0.20	243 234	67-79 78-81	2	15	<0.01, <0.01 (<0.01)	
	EC	0.20 0.20	243 234	67-79 78-81	2	15	<0.01, <0.01 (<0.01)	
Canada, 2013 Branchton, ON (Etna) Outdoor	SC	0.21 0.21	200	77-78 79-81	2	14	<0.010, <0.010 (<0.010)	TK0103849
	EC	0.20 0.21	200	77-78 79-81	2	7 10 14 17 21	<0.010 <0.010 <0.010, <0.010 (<0.010) <0.010 <0.010	Mean recovery for pydiflumetofen: 105% (n=3) at 0.01 mg/kg 92% (n=3) at 1.0 mg/kg
Canada, 2013 St-Mar-sur-Richelieu, QC (Etna) Outdoor	SC	0.20 0.20	200	72-75 82-83	2	15	<0.010, <0.010 (<0.010)	
	EC	0.20 0.20	200	72-75 82-83	2	15	<0.010, <0.010 (<0.010)	Sampling to analysis: 139-184 days
Canada, 2013 Plattsville, ON (T9903) Outdoor	SC	0.20 0.20	200	69-75 75-91	2	14	<0.010, <0.010 (<0.010)	
	EC	0.21 0.20	200	69-75 75-91	2	14	<0.010, <0.010 (<0.010)	
Canada, 2013 Taber, AB[e] (AC Polaris Great Northern) Outdoor	SC	0.21 0.20	200	67-73 74-75	2	15	0.012, 0.015 (0.014)	
	EC	0.21 0.20	200	67-73 74-75	2	15	<0.010, <0.010 (<0.010)	
Canada, 2013 Taber, AB[f] (AC Polaris Great Northern) Outdoor	SC	0.20 0.21	200	71-75 79	2	15	0.074, 0.10 (0.087)	
	EC	0.20 0.20	200	71-75 79	2	15	0.096, 0.11 (0.10)	

Portion analysed: seed (dry)

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

[e] Address: PO Box 4833 (Drive east for 8.5 km along Red Trail (Twp 10-0) from Taber, AB; drive 1.6 km south at RR15-5; site is on the right, about 235 m west), Application dates (1st): 31 Jul 2013

[f] Address: PO Box 4833 Taber, AB T1G 2E1 (From Taber, AB head east on Red Trail (Twp Rd. 10-0) to Rge Rd 15-5, turn right; turn right into the third driveway, marked by Ag-Quest signs to access the site), Application dates (1st): 31 Jul 2013

Soya bean (dry)

The Meeting received 21 trials (17 at harvest trials, four decline trials) on soya bean (dry) which were conducted in the USA (Oakes, 2015: TK0103860). In each of these trials, pydiflumetofen was applied to soya beans as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. Two applications at a nominal rate of 0.20 kg ai/ha were made. The first application was made 7 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, seed samples were taken 14 ± 1 DALA. In the decline trials additional seed samples were collected at 7, 10, 18 and 21 ± 1 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from soya bean seed as 91% at 0.01 mg/kg fortified, 89% at 0.1 mg/kg fortified and 103% at 0.5–1.0 mg/kg fortified. Soya bean seed samples were stored at -18 °C or below for a maximum of 19 months between sampling and analysis.

Table 60 Residues of pydiflumetofen on soya bean from supervised trials in the USA

Soya bean (dry) country, year (variety)	Application					DALA	Residues, mg/kg [ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.	Days		
GAP, USA	SC	0.20 0.40 / year	[c] 93 [d] 45		2	14		
USA, 2013 Chula, GA (Pioneer 97M50) Outdoor	SC	0.21 0.21	178 178	84-85 87-88	2	15	<0.01, <0.01 (<u><0.01</u>)	TK0103860 Mean recovery for pydiflumetofen: 91% (n=8) at 0.01 mg/kg 89% (n=9) at 0.1 mg/kg 103% (n=5) at 0.5-1.0 mg/kg Sampling to analysis: 226-548 days
	EC	0.20 0.21	178 178	84-85 87-88	2	15	<0.01, <0.01 (<0.01)	
USA, 2013 Seven Springs, NC (Asgrow 6931) Outdoor	SC	0.20 0.20	178 178	79-81 82-83	2	14	0.015, 0.017 (<u>0.016</u>)	
	EC	0.20 0.20	178 178	79-81 82-83	2	14	<0.01, <0.01 (<0.01)	
USA, 2013 Pollard, AR (5N451R2) Outdoor	SC	0.20 0.20	187 187	83-85 87-89	2	14	<0.01, <0.01 (<u><0.01</u>)	
	EC	0.20 0.20	187 187	83-85 87-89	2	14	<0.01, <0.01 (<0.01)	
USA, 2013 Fisk, MO (5N451R2) Outdoor	SC	0.20 0.20	187 187	83-85 85-87	2	14	<0.01, <0.01 (<u><0.01</u>)	
	EC	0.20 0.20	187 187	83-85 85-87	2	7 9 14 19 21	ND <0.01 ND, <0.01 (<0.01) <0.01 ND	
USA, 2013 Proctor, AR (HBK LL4950) Outdoor	SC	0.20 0.20	178 178	87-88 88-89	2	12	0.020, 0.033 (0.027)	
	EC	0.20 0.20	178 178	87-88 88-89	2	12	<0.01, 0.014 (0.012)	
USA, 2013 Jefferson, IA (93Y13) Outdoor	SC	0.20 0.19	234 224	82-82 83-84	2	7 10 14 18 21	0.35 0.39 0.069, 0.34 (0.21) 0.19 0.37 c0.033	
	EC	0.20 0.19	224 224	82-82 83-84	2	14	0.14, 0.20 (0.17) c0.033	
USA, 2013 Gardner, KS (Willcross RR2428N) Outdoor	SC	0.20 0.20	37 37	79-80 81-83	2	13	0.25, 0.32 (0.29)	
	EC	0.20 0.21	37 37	79-80 81-83	2	13	0.13, 0.13 (0.13)	
USA, 2013 Geneva, MN (Pioneer 91Y41) Outdoor	SC	0.20 0.20	168 187	79-80 84-85	2	14	0.011, 0.013 (0.012)	
	EC	0.20 0.20	159 187	79-80 84-85	2	14	<0.01, <0.01 (<0.01)	
USA, 2013 Lawrence, KS	SC	0.20 0.21	140 150	79-81 81-83	2	12	0.086, 0.090 (0.088)	

Soya bean (dry) country, year (variety)	Application					DALA	Residues, mg/kg [ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.	Days		
(MG3981-NR2) Outdoor	EC	0.20 0.20	150 150	79-81 81-83	2	12	0.055, 0.060 (0.058)	
USA, 2013 Perry, IA (93Y13) Outdoor	SC	0.20 0.21	19 19	81-83 85-85	2	13	0.044, 0.075 (0.060)	
	EC	0.19 0.21	19 19	81-83 85-85	2	13	0.043, 0.046 (0.045)	
USA, 2013 York, NE (S28-U7) Outdoor	SC	0.20 0.20	187 178	83-84 86-87	2	12	<0.01, 0.016 (0.014)	
	EC	0.21 0.20	187 178	83-84 86-87	2	12	0.031, 0.046 (0.039)	
USA, 2013 Stafford, KS (93Y72) Outdoor	SC	0.20 0.20	206 206	77-79 81-81	2	14	0.023, 0.033 (0.028)	
	EC	0.20 0.20	196 206	77-79 81-81	2	14	0.014, 0.014 (0.014)	
USA, 2013 Royalton, MN (PB 0954RR) Outdoor	SC	0.20 0.20	187 187	77-77 88-93	2	13	0.017, 0.041 (0.029)	
	EC	0.20 0.20	187 187	77-77 88-93	2	13	0.012, 0.027 (0.020)	
USA, 2013 Northwood, ND (11R02 RR2Y) Outdoor	SC	0.20 0.20	187 187	94-95 95-96	2	14	0.013, 0.013 (0.013)	
	EC	0.20 0.20	187 187	94-95 95-96	2	14	<0.01, <0.01 (<0.01)	
USA, 2013 Springfield, NE (NK28U7) Outdoor	SC	0.20 0.20	131 131	79-80 83-84	2	12	0.039, 0.043 (0.041)	
	EC	0.20 0.20	131 131	79-80 83-84	2	12	0.020, 0.028 (0.024)	
USA, 2013 Verona, WI (NK S20-Y2) Outdoor	SC	0.21 0.20	252 234	94-95 95-96	2	14	0.031, 0.033 (0.032)	
	EC	0.20 0.20	243 234	94-95 95-96	2	14	0.011, 0.012 (0.012)	
USA, 2013 Hedrick, IA (93Y15-PA35) Outdoor	SC	0.20 0.20	187 196	83 85	2	7 9 14 17 20	0.016 <0.01 0.010, 0.010 (0.010) 0.012 0.015	
	EC	0.21 0.21	196 196	83 85	2	14	<0.01, 0.022 (0.016)	
USA, 2013 Bloomfield, MS (R2 46X29) Outdoor	SC	0.20 0.20	187 187	83-84 87-89	2	14	<0.01, <0.01 (<0.01)	
	EC	0.20 0.20	187 187	83-84 87-89	2	8 9 14 19 21	0.024 <0.01 <0.01, <0.01 (<0.01) <0.01 <0.01	
USA, 2013 Campbell, MN (Asgrow 0832) Outdoor	SC	0.20 0.20	187 187	79-79 79-79	2	14	0.011, 0.011 (0.011)	
	EC	0.20 0.20	187 187	79-79 79-79	2	14	<0.01, <0.01 (<0.01)	
USA, 2013 Richland, IA (93Y60) Outdoor	SC	0.20 0.20	196 224	83 85-86	2	13	0.027, 0.035 (0.031)	
	EC	0.21 0.20	206 224	83 85-86	2	13	0.015, 0.036 (0.026)	
USA, 2013 Cooper, IA (93Y13) Outdoor	SC	0.21 0.21	243 224	81-83 83-85	2	16	0.016, 0.037 (0.027)	
	EC	0.21 0.20	234 215	81-83 83-85	2	16	0.026, 0.046 (0.036)	

Portion analysed: seed (dry)

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

Dry peas

Peas (dry)

The Meeting received 10 trials (eight at harvest trials, two decline trials) on peas (dry) which were conducted in Canada (Sagan, 2015: TK0103849) and the USA (Mäyer, 2015: TK0163595). In each of these trials, pydiflumetofen was applied to peas as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. Two applications at a nominal rate of 0.20 kg ai/ha were made. The first application was made 14 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, seed samples were taken 14 ± 3 DALA. In the decline trials additional seed samples were collected at 7, 10, 17 and 21 ± 1 DALA. Some samples were allowed to dry for 2–11 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from dry pea seed was 90% at 0.01–1.0 mg/kg fortified (Canada) and 92% at 0.01–0.1 mg/kg fortified (USA). Dry pea seed samples were stored at -10 °C or below (Canada) or at -18 °C or below (USA) for a maximum of 14 months between sampling and analysis.

Table 61 Residues of pydiflumetofen on dry pea from supervised trials in Canada and the USA

Peas (dry) country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.20 0.40 / year	[b] 93 [c] 45		2	14		
USA, 2013 Mount Hood, OR (Columbia Green Pea) Outdoor	SC	0.20 0.19	168 187	69-73 79-79	2	13	0.048, 0.066 (0.057)	TK0163595 Mean recovery for pydiflumetofen: 90% (n=2) at 0.01 mg/kg 93% (n=2) at 0.1 mg/kg Sampling to analysis: 339-423 days
	EC	0.20 0.19	168 187	69-73 79-79	2	13	0.039, 0.050 (0.045)	
USA, 2013 Jerome, ID (Latah Yellow) Outdoor	SC	0.20 0.21	168 187	72-74 79-82	2	14	0.035, 0.036 (0.035)	
	EC	0.20 0.20	168 178	72-74 79-82	2	14	0.029, 0.041 (0.035)	
USA, 2013 Payette, ID (Austrian Winter Pea) Outdoor	SC	0.20 0.20	234 234	67-74 69-81	2	14	0.041, 0.054 (0.048)	
	EC	0.21 0.20	234 234	67-74 69-81	2	14	0.060, 0.068 (0.064)	
USA, 2013 Acequia, ID (Cascadia) Outdoor	SC	0.21 0.20	122 131	67-74 77-79	2	7 10 14 17 21	0.035 0.050 0.073, 0.10 (0.088) 0.016 0.019	
	EC	0.20 0.20	122 122	67-74 77-79	2	14	0.093, 0.10 (0.096)	
USA, 2013 American Falls, ID (Progress #9) Outdoor	SC	0.21 0.21	28 28	77-79 79-82	2	14	0.057, 0.060 (0.059)	
	EC	0.21 0.20	28 28	77-79 79-82	2	14	0.012, 0.025 (0.019)	
Canada, 2013 Pike Lake, SK (Golden) Outdoor	SC	0.19 0.19	200	76-79 81-83	2	14	0.048, 0.057 (0.053)	TK0103849 Mean recovery for pydiflumetofen: 93% (n=3) at 0.01 mg/kg 88% (n=3) at 1.0 mg/kg Sampling to
	EC	0.19 0.19	200	76-79 81-83	2	14	0.025, 0.029 (0.027)	
Canada, 2013 Delisle, SK (Golden) Outdoor	SC	0.19 0.20	200	73-75 79-80	2	15	0.062, 0.064 (0.063)	
	EC	0.19 0.20	200	73-75 79-80	2	15	0.059, 0.065 (0.062)	
Canada, 2013 Hanley, SK (Golden) Outdoor	SC	0.20 0.20	200	79-81 84-85	2	15	0.021, 0.034 (0.028)	
	EC	0.20 0.20	200	79-81 84-85	2	15	<0.010, 0.011 (0.011)	

Peas (dry) country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[a]	Ref
Canada, 2013 Carberry, MB (CDC Meadow) Outdoor	SC	0.20 0.20	200	73-75 81-83	2	15	0.021, 0.024 (0.023)	analysis: 156-205 days
	EC	0.20 0.20	200	73-75 81-83	2	15	0.016, 0.025 (0.021)	
Canada, 2013 Minto, MB (CDC Meadow) Outdoor	SC	0.20 0.20	200	65-72 67-75	2	14	0.016, 0.029 (0.023)	
	EC	0.20 0.19	200	65- 66/73 67-76	2	7 10 14 17 21	0.018 0.016 0.014, 0.014 (0.014) 0.017 0.018	

Portion analysed: seed (dry)

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

Root and tuber vegetables

Tuberous and corm vegetables

Potato

The Meeting received 26 trials (22 at harvest trials, four decline trials) on potato which were conducted in Canada (Sagan, 2015: TK0103851) and the USA (Smith, 2015: TK0163562). In each of these trials, a SC formulations (200 g ai/L) was applied in three applications to potato as a foliar treatment at a nominal rate of 0.13 kg ai/ha. Applications occurred at 7 ± 1 day intervals. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, tubers were taken 7 ± 1 DALA. In the decline trials additional tuber samples were collected at 0, 3, 10 and 14 ± 1 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from potato was 110% at 0.01–0.1 mg/kg fortified (Canada) and 114% at 0.01–1.0 mg/kg fortified (USA). Potato tuber samples were stored at -10 °C or below for a maximum of 15 months between sampling and analysis.

Table 62 Residues of pydiflumetofen on potato from supervised trials in Canada and the USA

Potato country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[a]	Ref
GAP, USA	SC	0.13 0.38 /year	[b] 93 [c] 45		3	7		
USA, 2013 Lyons, NY (Gold Rush) Outdoor	SC	0.12 0.12 0.12	281 281 281	47 48 49	3	7	<0.01, <0.01 (<0.01)	TK0163562 Mean recovery for pydiflumetofen: 109% (n=8) at 0.01 mg/kg 118% (n=8) at 1.0 mg/kg Sampling to analysis: 135-447 days
USA, 2013 North Rose, NY (NY118) Outdoor	SC	0.13 0.13 0.12	383 383 374	45-47 45-47 47-48	3	7	<0.01, <0.01 (<0.01)	
USA, 2013 Seven Springs, NC (Red Pontiac) Outdoor	SC	0.12 0.12 0.13	271 290 337	42 44 47	3	6	<0.01, 0.018 (0.014)	
USA, 2013 Oviedo, FL (Red La Soda) Outdoor	SC	0.13 0.13 0.12	281 281 281	47 47 47	3	7	<0.01, <0.01 (<0.01)	
USA, 2013 Northwood, ND (Red Pontiac) Outdoor	SC	0.13 0.13 0.12	187 196 187	43-44 45-46 47-48	3	7	<0.01, <0.01 (<0.01)	

Potato country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[a]	Ref
USA, 2013 Geneva, MN (Cascade) Outdoor	SC	0.12 0.12 0.13	168 168 168	44-46 45-47 46-48	3	0 3 7 10 14	<0.01 0.022 <0.01, <0.01 (<0.01) <0.01 <0.01	
USA, 2013 Richland, IA (Dark Red Norland) Outdoor	SC	0.12 0.13 0.12	37 37 37	47 48 12	3	7	<0.01, <0.01 (<0.01)	
USA, 2013 Rice, MN (Snowden) Outdoor	SC	0.12 0.12 0.12	187 187 187	75 46 84	3	7	<0.01, <0.01 (<0.01)	
USA, 2013 Kimberly, ID (Russet Burbank) Outdoor	SC	0.12 0.12 0.13	290 327 271	46-47 47-48 47-48	3	7	<0.01, <0.01 (<0.01)	
USA, 2013 San Ardo, CA (FL2048) Outdoor	SC	0.13 0.13 0.13	346 327 346	43 45-47 47	3	6	<0.01, <0.01 (<0.01)	
USA, 2013 Aberdeen, ID (Russet Burbank) Outdoor	SC	0.12 0.12 0.12	243 243 234	46-47 47-48 48	3	6	<0.01, <0.01 (<0.01)	
USA, 2013 Ephrata, WA (Umatilla) Outdoor	SC	0.13 0.12 0.12	281 281 281	45-46 46-47 48-49	3	7	<0.01, <0.01 (<0.01)	
USA, 2013 Jerome, ID (Ranger Russet) Outdoor	SC	0.12 0.12 0.12	178 178 187	47-48 47-48 48-49	3	6	<0.01, <0.01 (<0.01)	
USA, 2013 Rupert, ID (Russet Burbank) Outdoor	SC	0.12 0.12 0.12	37 37 37	47-48 47-48 48-49	3	7	<0.01, <0.01 (<0.01)	
USA, 2013 Payette, ID (Russet Norkotah) Outdoor	SC	0.12 0.12 0.13	234 234 243	47-48 47-48 48	3	0 3 8 11 14	<0.01 <0.01 <0.01, <0.01 (<0.01) <0.01 <0.01	
USA, 2013 American Falls, ID (Russet Burbank) Outdoor	SC	0.12 0.12 0.12	234 243 234	46-47 47-48 48	3	6	<0.01, <0.01 (<0.01)	
Canada, 2013 New Glasgow, PE[a)] (Goldrush) Outdoor	SC	0.12 0.12 0.12	200 200 200	69-70 69-70 79-92	3	6	<0.010, <0.010 (<0.010)	TK0103851 Mean recovery for pydiflumetofen: 110% (n=4) at 0.01 mg/kg 120% (n=4) at 0.1 mg/kg Sampling to analysis: 323-425 days
Canada, 2013 New Glasgow, PE[b)] (Shepody) Outdoor	SC	0.13 0.12 0.13	45 45 45	69-70 69-70 79-92	3	6	<0.010, <0.010 (<0.010)	
Canada, 2013 New Glasgow, PE[c)] (Russet Burbank) Outdoor	SC	0.13 0.12 0.13	200 200 200	85-92 92-93 93-95	3	0 3 7 10 14	<0.010 <0.010 <0.010, <0.010 (<0.010) <0.010 <0.010	

Potato country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[a]	Ref
Canada, 2013 Nictaux East, NS (Superior) Outdoor	SC	0.12 0.12 0.13	200 200 200	68-70 69-91 81-91	3	7	<0.010, <0.010 (<0.010)	
Canada, 2013 Upper Dyke Village, NS (Dakota Pearl) Outdoor	SC	0.12 0.13 0.13	200 200 200	65-69 47-48 69-91	3	7	<0.010, <0.010 (<0.010)	
Canada, 2013 Branchton, ON (Norland) Outdoor	SC	0.13 0.12 0.13	200 200 200	44-45 46-47 48-49	3	0 3 7 11 13	<0.010 <0.010 <0.010, <0.010 (<0.010) <0.010 <0.010	
Canada, 2013 Taber, AB[d)] (Sangre) Outdoor	SC	0.13 0.13 0.13	45 45 45	45-48 46-48 46-48	3	7	<0.010, <0.010 (<0.010)	
Canada, 2013 Taber, AB[e)] (Yukon Gold) Outdoor	SC	0.13 0.13 0.13	200 200 200	46-48 47-48 47-48	3	7	<0.010, <0.010 (<0.010)	
Canada, 2013 Minto, MB[f)] (Norland) Outdoor	SC	0.13 0.13 0.13	200 200 200	81(50%)/ 91- 93(50%), 81- 93/47 91- 95/47- 48	3	7	<0.010, <0.010 (<0.010)	
Canada, 2013 Minto, MB[g)] (Russet Burbank) Outdoor	SC	0.13 0.13 0.14	45 45 45	39-47 48-91 91-48	3	7	<0.010, <0.010 (<0.010)	

Portion analysed: tuber

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

[d] and [e] Address: 265 Route 258 New Glasgow, PE, Application dates (1st): 12 Aug 2013

[f] Address: 265 Route 258 New Glasgow, PE, Application dates (1st): 06 Sep 2013

[g] and [h] Address: PO Box 4833, Application dates (1st): 13 Aug 2013

[i] Address: 210 South Railway St Minto, MB R0K 1M0, Application dates (1st): 06 Aug 2013

[j] Address: 210 South Railway St Minto, MB R0K 1M0, Application dates (1st): 28 Aug 2013

Stalk and stem vegetables

Stalk and stem vegetables - Stems and Petioles

Celery

The Meeting received 8 trials (at harvest trials) on celery which were conducted in the USA (Oakes, 2015: TK0163598). In each of these trials, a SC formulations (200 g ai/L) was applied twice to celery as a foliar treatment at a nominal rate of 0.20 kg ai/ha. The first application was made 7 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, whole plants were taken 0 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from celery as 106% at 0.01 mg/kg fortified, 104% at 0.1 mg/kg fortified and 94% at 1.0–20 mg/kg fortified. Celery samples were stored at ca -18 °C for a maximum of 18 months between sampling and analysis.

Table 63 Residues of pydiflumetofen on celery from supervised trials in the USA

Celery country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.20 0.40 / year	100[b] 50[c]		2	0		
USA, 2014 Oviedo, FL (Utah) Outdoor	SC	0.20 0.20	262 262	49-49 49-49	2	0	4.4, 5.1 (4.8)	TK0163598 Mean recovery for pydiflumetofen: 106% (n=3) at 0.01 mg/kg 104% (n=2) at 0.1 mg/kg 94% (n=4) at 1.0-20 mg/kg Sampling to analysis: 202-549 days
USA, 2013 Oviedo, FL (Starlet) Outdoor	SC	0.20 0.20	262 262	49-49 49-49	2	0	5.4, 5.5 (5.4)	
USA, 2013 Verona, WI (Tango) Outdoor	SC	0.20 0.21	215 224	47-48 48-49	2	0	3.4, 4.4 (3.9)	
USA, 2013 King City, CA[d] (Conquistador) Outdoor	SC	0.20 0.20	243 262	47-49 47-49	2	0	3.8, 4.8 (4.3)	
USA, 2013 Porterville, CA (Sonora) Outdoor	SC	0.20 0.20	206 224	44-47 47-49	2	0	4.1, 4.8 (4.5)	
USA, 2013 King City, CA[e] (SSC1) Outdoor	SC	0.21 0.21	234 243	46 47	2	0	2.4, 2.7 (2.6)	
USA, 2013 Nipomo, CA (Conquistador) Outdoor	SC	0.20 0.21	281 290	48 49	2	0	2.4, 3.0 (2.7)	
USA, 2013 Madera, CA (Sonora) Outdoor	SC	0.20 0.21	234 234	48-49 48-49	2	0	7.0, 9.3 (8.1)	

Portion analysed: whole plant

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

[d] Address: King City CIMIS Weather Station#113 ~8.0 miles, Application dates (1st): 21 Jun 2013

[e] Address: King City CIMIS Weather Station#113 ~20 miles, Application dates (1st): 01 Jul 2013

Cereal grains

Wheat, similar grains, and Pseudocereals without husks

Wheat

The Meeting received 33 trials (29 at harvest trials, four decline trials) on wheat which were conducted in Canada (Sagan, 2015: TK0103847) and the USA (Oakes, 2015: TK0163574). In each of these trials, pydiflumetofen was applied to wheat as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. One application at 0.15 kg ai/ha was followed by a second application at 0.20 kg ai/ha. The first application was made 12–21 days prior to the second application, and the second application was made at BBCH 71. All applications were made in tank-mix with an adjuvant, NIS, COC or Methylated Seed Oil (MSO). At each trial, grain samples were taken at normal grain harvest maturity which occurred at 16–74 days after the second application. In the decline trials additional grain samples were collected at 10 and 5 ± 1 day before normal harvest, and 5 and 10 ± 1 day after normal harvest.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from wheat grain was 102% at 0.01–0.49 mg/kg fortified (Canada) and 92% at 0.01–10 mg/kg fortified (USA). Wheat grain samples were stored at -10 °C or below (Canada) or at -18 °C or below (USA) for a maximum of 19 months between sampling and analysis.

Table 64 Residues of pydiflumetofen on wheat grains from supervised trials in Canada and the USA

Wheat grains country, year (variety)	Application					DALA	Residues, mg/kg [bc]	Ref
	Form	kg ai/ha	L/ha	BBCH [a]	no.	Days		
GAP, USA	SC	0.20 0.35 / year	93[d] 45[e]	(14 days)	2	no apply after BBCH 71		
USA, 2014 Seven Springs, NC (Pioneer 26R20) Outdoor	SC	0.15 0.20	243 234	66-67 71-73 (14 days)	2	20	0.066, 0.073 (0.070)	TK0163574 Mean recovery for pydiflumetofen: 89% (n=7) at 0.01 mg/kg 96% (n=6) at 0.1 mg/kg 90% (n=3) at 0.5-10 mg/kg Sampling to analysis: 93-574 days
	EC	0.15 0.20	234 234	66-67 71-73 (14 days)	2	20	0.16, 0.18 (0.17)	
USA, 2014 Proctor, AR (Progeny 357) Outdoor	SC	0.15 0.20	112 150	68-69 71-71 (14 days)	2	23	0.11, 0.12 (0.12)	
	EC	0.15 0.20	112 150	68-69 71-71 (14 days)	2	23	0.11, 0.11 (0.11)	
USA, 2014 St. Cloud, MN (Arapahoe) Outdoor	SC	0.15 0.20	187 187	48-51 69-71 (14 days)	2	45	0.012, 0.031 (0.022)	
	EC	0.15 0.20	187 187	49-51 69-71 (14 days)	2	45	0.038, 0.061 (0.050)	
USA, 2013 Richland, IA (748) Outdoor	SC	0.15 0.20	215 168	40 71 (14 days)	2	44 48 53 58 62	0.056 0.057 0.054, 0.060 (0.057) 0.047 0.041	
	EC	0.15 0.20	215 168	40 71 (14 days)	2	53	0.045, 0.053 (0.049)	
USA, 2014 Stafford, KS (Armour) Outdoor	SC	0.14 0.20	206 206	50-53 71-75 (14 days)	2	29	0.028, 0.052 (0.040)	
	EC	0.14 0.20	206 206	50-53 71-75 (14 days)	2	29	0.027, 0.036 (0.032)	
USA, 2014 Bagley, IA (BW 442) Outdoor	SC	0.16 0.21	243 196	69-69 71-71 (15 days)	2	33	0.062, 0.074 (0.068)	
	EC	0.16 0.21	243 196	69-69 71-71 (15 days)	2	33	0.098, 0.12 (0.11)	
USA, 2014 Bloomfield, MO (McAlister) Outdoor	SC	0.15 0.20	187 187	43-45 71-71 (13 days)	2	32	0.042, 0.054 (0.048)	
	EC	0.14 0.20	187 187	43-45 71-71 (13 days)	2	32	0.028, 0.060 (0.044)	
USA, 2014 Uvalde, TX (Greer) Outdoor	SC	0.15 0.20	224 234	49-51 71-71 (16 days)	2	33	0.14, 0.23 (0.19)	
	EC	0.15 0.20	224 234	49-51 71-71 (16 days)	2	33	0.11, 0.13 (0.12)	
USA, 2013 Cleveland, ND [f] (Divide)	SC	0.15 0.20	187 140	70-71 81-82 (13 days)	2	16	0.10, 0.13 (0.12)	

Wheat grains country, year (variety)	Application					DALA	Residues, mg/kg [bc]	Ref
	Form	kg ai/ha	L/ha	BBCH [a]	no.	Days		
Outdoor	EC	0.15 0.20	187 140	70-71 81-82 (13 days)	2	16	0.14, 0.15 (0.15)	
USA, 2013 Carrington, ND (Faller) Outdoor	SC	0.16 0.20	150 140	44-45 71-71 (14 days)	2	52	0.057, 0.076 (0.067)	
	EC	0.15 0.20	140 140	44-45 71-71 (14 days)	2	52	0.043, 0.044 (0.044)	
USA, 2013 Grand Island, NE (Taverse) Outdoor	SC	0.15 0.20	150 187	45-45 73-73 (12 days)	2	22	0.081, 0.099 (0.090)	
	EC	0.15 0.20	150 187	45-45 73-73 (12 days)	2	22	0.071, 0.080 (0.076)	
USA, 2013 Prosser, NE (Barlow) Outdoor	SC	0.15 0.21	47 47	61-65 71-71 (14 days)	2	29	0.050, 0.052 (0.051)	
	EC	0.15 0.20	47 47	61-65 71-71 (14 days)	2	29	0.098, 0.10 (0.10)	
USA, 2013 Cleveland, ND [g] (Glenn) Outdoor	SC	0.15 0.21	187 150	70-71 81-82 (13 days)	2	16	0.15, 0.17 (0.16)	
	EC	0.15 0.20	187 140	70-71 81-82 (13 days)	2	16	0.12, 0.24 (0.18)	
USA, 2014 Jetmore, KS (TAM 112) Outdoor	SC	0.15 0.22	37 47	50-53 71-71 (14 days)	2	47	0.029, 0.051 (0.040)	
	EC	0.15 0.21	37 47	50-53 71-71 (14 days)	2	47	0.057, 0.066 (0.062)	
USA, 2014 San Angelo, TX (WB-Grainfield) Outdoor	SC	0.14 0.20	140 140	49-56 71-73 (16 days)	2	21 27 32 36 42	0.099 0.068 0.051, 0.051 (0.051) 0.090 0.13	
	EC	0.15 0.20	140 140	49-56 71-73 (16 days)	2	32	0.090, 0.097 (0.094)	
USA, 2014 Levelland, TX (Weathermaster) Outdoor	SC	0.15 0.20	140 140	59-59 71-83 (14 days)	2	19	0.18, 0.25 (0.22)	
	EC	0.15 0.20	140 140	59-59 71-83 (14 days)	2	19	0.23, 0.24 (0.23)	
USA, 2014 Larned, KS (T-154) Outdoor	SC	0.15 0.20	215 206	50-53 71-71 (12 days)	2	40	0.050, 0.073 (0.062)	
	EC	0.15 0.20	206 206	50-53 71-71 (12 days)	2	40	0.058, 0.065 (0.062)	
USA, 2014 Dill City, OK (Duster) Outdoor	SC	0.15 0.21	122 131	59-60 71-73 (14 days)	2	28	0.095, 0.10 (0.099)	
	EC	0.15 0.20	112 131	59-60 71-73 (14 days)	2	28	0.12, 0.12 (0.12)	
USA, 2014 Hinton, OK	SC	0.14 0.20	122 122	61-69 81-83	2	33	0.063, 0.096 (0.080)	

Wheat grains country, year (variety)	Application					DALA	Residues, mg/kg [bc]	Ref
	Form	kg ai/ha	L/ha	BBCH [a]	no.	Days		
(Duster) Outdoor				(13 days)				
	EC	0.15 0.20	131 122	61-69 81-83 (13 days)	2	33	0.15, 0.16 (0.16)	
USA, 2014 Jerome, ID (Legacy) Outdoor	SC	0.14 0.20	187 196	45-49 65-69 (15 days)	2	74	<0.010, 0.020 (0.015)	
	EC	0.15 0.21	196 196	45-49 65-69 (15 days)	2	74	<0.010, <0.010 (<0.010)	
Canada, 2013 Pike Lake, SK (Shaw) Outdoor	SC	0.15 0.20	45 45	59-65 71-75 (14 days)	2	41	0.11, 0.12 (0.12)	TK0103847
	EC	0.15 0.20	45 45	59-65 71-75 (14 days)	2	29 36 41 47 51	0.20 0.19 0.073, 0.079 (0.076) 0.23 0.18	Mean recovery for pydiflumetofen: 100% (n=4) at 0.01 mg/kg 104% (n=4) at 0.1 mg/kg 103% (n=3) at 0.49 mg/kg
Canada, 2014 Vanscoy, SK (CDC Utmost) Outdoor	SC	0.15 0.20	200 200	58-61 69-71 (14 days)	2	39	0.046, 0.079 (0.063)	
	EC	0.15 0.20	200 200	58-61 69-71 (14 days)	2	39	0.037, 0.053 (0.045)	Sampling to analysis: 138-506 days
Canada, 2013 Taber, AB (Bentley) Outdoor	SC	0.15 0.20	200 200	61-65 69-73 (13 days)	2	38	0.056, 0.057 (0.057)	
	EC	0.15 0.20	200 200	61-65 69-73 (13 days)	2	38	0.037, 0.038 (0.038)	
Canada, 2013 Boissevain, MB (Harvest) Outdoor	SC	0.15 0.21	45 45	51-59 71-73 (12 days)	2	48	0.066, 0.068 (0.067)	
	EC	0.15 0.20	45 45	51-59 71-73 (12 days)	2	48	0.051, 0.064 (0.058)	
Canada, 2013 Elgin A, MB [h] (Vesper) Outdoor	SC	0.16 0.20	45 45	55-59 71-73 (13 days)	2	44	0.054, 0.060 (0.057)	
	EC	0.16 0.20	45 45	55-59 71-73 (13 days)	2	44	0.043, 0.044 (0.044)	
Canada, 2013 Elgin B, MB [i] (Unity) Outdoor	SC	0.15 0.21	200 200	55-59 71-73 (13 days)	2	44	0.036, 0.044 (0.040)	
	EC	0.16 0.21	200 200	55-59 71-73 (13 days)	2	44	0.023, 0.025 (0.024)	
Canada, 2013 Rosthern, SK (Shaw) Outdoor	SC	0.14 0.21	200 200	59-61 69-71 (14 days)	2	41	0.086, 0.12 (0.10)	
	EC	0.14 0.21	200 200	59-61 69-71 (14 days)	2	41	0.091, 0.10 (0.096)	
Canada, 2013 Coalhurst, AB (AC Carberry) Outdoor	SC	0.15 0.20	200 200	45-61 71-73 (14 days)	2	47	0.019, 0.031 (0.025)	
	EC	0.15 0.20	200 200	45-61 71-73 (14 days)	2	47	0.014, 0.020 (0.017)	
Canada, 2013	SC	0.15	200	55-56	2	40	0.030, 0.037	

Wheat grains country, year (variety)	Application					DALA Days	Residues, mg/kg [bc]	Ref
	Form	kg ai/ha	L/ha	BBCH [a]	no.			
Carberry, MB (Harvest) Outdoor		0.20	200	71 (15 days)			(0.034)	
	EC	0.15 0.20	200 200	55-56 71 (15 days)	2	40	0.034, 0.036 (0.035)	
Canada, 2013 Alvena, SK (Unity) Outdoor	EC	0.16 0.20	200 200	51-53 71-72 (13 days)	2	56	0.11, 0.12 (0.12) c 0.012	
Canada, 2013 Lamont, AB (Harvest) Outdoor	SC	0.16 0.21	200 200	54-59 69-71 (15 days)	2	46	0.10, 0.13 (0.12)	
	EC	0.15 0.21	200 200	54-59 69-71 (15 days)	2	46	0.035, 0.037 (0.036)	
Canada, 2014 Josephburg, AB (Harvest) Outdoor	SC	0.16 0.21	200 200	51-57 69-71 (21 days)	2	48	0.057, 0.069 (0.063)	
	EC	0.15 0.21	200 200	51-57 69-71 (21 days)	2	48	0.051, 0.069 (0.060)	
Canada, 2013 Minto, MB (Unity) Outdoor	SC	0.15 0.20	200 200	51-58 71-73 (14 days)	2	45	0.026, 0.050 (0.038)	
	EC	0.15 0.20	200 200	51-58 71-73 (14 days)	2	35 40 45 49 54	0.047 0.019 0.022, 0.031 (0.027) 0.030 0.025	

Portion analysed: grain

[a] Re-treatment interval is given in parenthesis.

[b] Mean of replicate field samples is given in parenthesis.

[c] c: sample from control plot

[d] ground application,

[e] aerial application

[f] and [g] Address: Jamestown Municipal Airport ~27 miles, Application dates (1st): 15 Aug 2013

[h] and [i] Address: 210 South Railway St, Minto, MB R0K 1M0, Application dates (1st): 17 Jul 2013

Barley, similar grains, and pseudocereals with husks

Barley

The Meeting received 21 trials (17 at harvest trials, four decline trials) on barley which were conducted in Canada (Sagan, 2015: TK0163517) and the USA (Oakes, 2015: TK0163584). In each of these trials, pydiflumetofen was applied to barley as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. One application at 0.15 kg ai/ha was followed by a second application at 0.20 kg ai/ha. The first application was made 9–15 days prior to the second application, and the second application was made at BBCH 71. All applications were made in tank-mix with an adjuvant, NIS, COC or MSO. At each trial, grain samples were taken at normal grain harvest maturity which occurred at 16–68 days after the second application. In the decline trials additional grain samples were collected at 10 and 5 ± 1 day before normal harvest, and 5 and 10 ± 1 days after normal harvest.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from barley grain was 93% at 0.01–1.0 mg/kg fortified (Canada) and 99% at 0.01–25 mg/kg fortified (USA). Barley grain samples were stored at -10 °C or below (Canada) or at -18 °C or below (USA) for a maximum of 18 months between sampling and analysis.

Table 65 Residues of pydiflumetofen on barley from supervised trials in Canada and the USA

Barley country, year (variety)	Application					DALA Days	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
GAP, USA	SC	0.20 0.35 / year	93[c] 45[d]	(14 days)	2	not apply after BBCH 71		
USA, 2013 Suffolk, VA (Atlantic) Outdoor	SC	0.15 0.21	122 112	55-56 71-71 (15 days)	2	36	0.81, 0.83 (0.82)	TK0163584 Mean recovery for pydiflumetofen: 101% (n=6) at 0.01 mg/kg 104% (n=5) at 0.1 mg/kg 92% (n=5) at 2-25 mg/kg Sampling to analysis: 100-558 days
	EC	0.15 0.21	122 112	55-56 71-71 (15 days)	2	36	0.70, 0.83 (0.76)	
USA, 2013 Richland, IA (Robust) Outdoor	SC	0.15 0.20	168 262	50 73 (13 days)	2	49 54 59 63 68	0.52 0.23 0.20, 0.23 (0.22) 0.31 0.29	
	EC	0.15 0.20	59	50 73 (13 days)	2	59	0.17, 0.18 (0.18)	
USA, 2013 York, NE (Lacey) Outdoor	SC	0.15 0.21	187 150	45-45 73-73 (14 days)	2	21	1.0, 1.1 (1.1)	
	EC	0.14 0.20	187 150	45-45 73-73 (14 days)	2	21	0.95, 1.0 (0.98)	
USA, 2013 Bagley, IA (Robust) Outdoor	SC	0.14 0.21	94 112	61-61 71-71 (13 days)	2	28	1.6, 1.7 (1.7)	
	EC	0.16 0.20	103 103	61-61 71-71 (13 days)	2	28	3.0, 3.0 (3.0)	
USA, 2013 Cleveland, ND (Tradition) Outdoor	SC	0.16 0.21	47 47	70-71 81-82 (13 days)	2	16	1.4, 2.4 (1.9)	
	EC	0.15 0.21	56 47	70-71 81-82 (13 days)	2	16	1.9, 2.0 (1.9)	
USA, 2013 Grand Island, NE (Lacey) Outdoor	SC	0.15 0.20	150 196	45-45 73-73 (14 days)	2	27	0.35, 0.51 (0.43)	
	EC	0.15 0.20	150 196	45-45 73-73 (14 days)	2	27	0.17, 0.26 (0.22)	
USA, 2013 Carrington, ND (Robust) Outdoor	SC	0.15 0.20	140 140	45-45 71-71 (14 days)	2	52	0.076, 0.085 (0.081)	
	EC	0.15 0.20	140 140	45-45 71-71 (14 days)	2	52	0.073, 0.076 (0.075)	
USA, 2013 Prosser, NE (Lacey) Outdoor	SC	0.15 0.20	168 168	61-65 71-71 (14 days)	2	24 29 34 39 45	0.31 0.30 0.25, 0.28 (0.26) 0.14 0.25	
	EC	0.15 0.20	168 168	61-65 71-71 (14 days)	2	34	0.21, 0.42 (0.31)	
USA, 2013 Jerome, ID (Moravian 69) Outdoor	SC	0.15 0.20	187 159	61-65 71-73 (14 days)	2	45	0.043, 0.044 (0.044)	
	EC	0.15 0.20	187 159	61-65 71-73 (14 days)	2	45	0.080, 0.081 (0.081)	

Barley country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH[a]	no.	DALA Days	Residues, mg/kg[b]	Ref
USA, 2013 Porterville, CA (UC937) Outdoor	SC	0.15 0.20	290 281	69-71 73-77 (13 days)	2	21	2.5, 2.6 (2.6)	
	EC	0.15 0.20	290 281	69-71 73-77 (13 days)	2	21	1.8, 2.5 (2.2)	
USA, 2013 Parkdale, OR (Haybet) Outdoor	SC	0.15 0.20	37 47	69-69 85-85 (13 days)	2	26	0.52, 0.66 (0.59)	
	EC	0.15 0.19	37 47	69-69 85-85 (13 days)	2	26	0.71, 0.73 (0.72)	
USA, 2013 Payette, ID (Westbred 501) Outdoor	SC	0.15 0.20	234 234	41-45 71-71 (14 days)	2	44	0.19, 0.19 (<u>0.19</u>)	
	EC	0.15 0.21	234 243	41-45 71-71 (14 days)	2	44	0.068, 0.14 (0.10)	
Canada, 2013 Taber, AB (CDC Austenson) Outdoor	SC	0.16 0.21	200	58-65 69-73 (13 days)	2	36	0.51, 0.58 (<u>0.55</u>)	TK0163517
	EC	0.16 0.21	200	58-65 69-73 (13 days)	2	36	0.40, 0.51 (0.46)	Mean recovery for pydiflumetofen: 98% (n=6) at 0.01 mg/kg 88% (n=6) at 1.0 mg/kg
Canada, 2013 Boissevain, MB (Legacy) Outdoor	SC	0.16 0.22	45	39-41 71-73 (13 days)	2	47	0.20, 0.20 (<u>0.20</u>)	
	EC	0.16 0.21	45	39-41 71-73 (13 days)	2	47	0.095, 0.13 (0.11)	Sampling to analysis: 124-165 days
Canada, 2013 Elgin, MB (Bentley) Outdoor	SC	0.15 0.21	45	43-49 68-71 (9 days)	2	42	0.075, 0.10 (0.088)	
	EC	0.15 0.22	45	43-49 68-71 (9 days)	2	42	0.20, 0.20 (0.20)	
Canada, 2013 Rosthern, SK (AC Metcalf) Outdoor	SC	0.14 0.20	200	59-61 69-71 (14 days)	2	41	0.38, 0.54 (0.46)	
	EC	0.14 0.20	200	59-61 69-71 (14 days)	2	41	0.57, 0.75 (<u>0.66</u>)	
Canada, 2013 Waldheim, SK (AC Metcalf) Outdoor	SC	0.16 0.20	200	51-61 71-75 (14 days)	2	50	0.21, 0.24 (<u>0.23</u>)	
	EC	0.16 0.20	200	51-61 71-75 (14 days)	2	50	0.11, 0.19 (0.15)	
Canada, 2013 Carberry, MB (Conlon) Outdoor	SC	0.15 0.20	200	53-54 71 (15 days)	2	40	0.14, 0.15 (<u>0.15</u>)	
	EC	0.15 0.20	200	53-54 71 (15 days)	2	40	0.13, 0.14 (0.14)	
Canada, 2013 Josephburg, AB (Coalition) Outdoor	SC	0.16 0.22	45	47-52 71-73 (15 days)	2	48	0.51, 0.62 (<u>0.57</u>)	
	EC	0.15 0.21	45	47-52 71-73 (15 days)	2	48	0.30, 0.30 (0.30)	
Canada, 2013	SC	0.15	200	41-45	2	42	0.066, 0.070	

Barley country, year (variety)	Application					DALA Days	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
Minto, MB[e] (Bentley) Outdoor		0.20		71,77 (13 days)			(0.068)	
	EC	0.15 0.20	200	41-45 71,77 (13 days)	2	31 38 42 46 51	0.053 0.045 0.056, 0.068 (0.062) 0.053 0.055	
Canada, 2013 Minto, MB[f] (AC Metcalf) Outdoor	SC	0.16 0.21	200	39-41 69-73 (11 days)	2	48	0.089, 0.14 (0.11)	
	EC	0.16 0.21	200	39-41 69-73 (11 days)	2	38 43 48 52 57	0.050 0.048 0.035, 0.052 (0.044) 0.071 0.047	

Portion analysed: grain

[a] Re-treatment interval is given in parenthesis.

[b] Mean of replicate field samples is given in parenthesis.

[c] ground application,

[d] aerial application

[e] Address: 210 South Railway St, Minto, MB R0K 1M0 (From Minto, drive 2.4 km north on Hwy #10, turning east on gravel road 29N at Double Diamond Farm Supply. Travel east for 3.5 km. The trials are located immediately north of this road.), Application dates (1st): 03 Jul 2013

[f] Address: 210 South Railway St, Minto, MB R0K 1M0 (From Minto, travel north on Hwy #10 for 0.8 km, turn east on gravel road at Manitoba Hydro station. Travel 0.8 km to farm entrance, turn south and travel ~ 300 m. Turn west and travel ~ 400 m to the trial site.), Application dates (1st): 05 Jul 2013

Oats

The Meeting received 28 trials (24 at harvest trials, four decline trials) on oats which were conducted in Canada (Sagan, 2015: TK0163519) and the USA (McDonald, 2015: TK0163581). In each of these trials, pydiflumetofen was applied to oats as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. One application at 0.15 kg ai/ha was followed by a second application at 0.20 kg ai/ha. The first application was made 7–15 days prior to the second application, and the second application was made at BBCH 71. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, grain samples were taken at normal grain harvest maturity which occurred at 16–61 days after the second application. In the decline trials additional grain samples were collected at 10 and 5 ± 1 day before normal harvest, and 5 and 10 ± 1 days after normal harvest.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from oat grain was 100% at 0.01–5.1 mg/kg fortified (Canada) and 98% at 0.01–5 mg/kg fortified (USA). Oat grain samples were stored at -10 °C or below (Canada) or at ca -18 °C (USA) for a maximum of 18 months between sampling and analysis.

Table 66 Residues of pydiflumetofen on oats from supervised trials in Canada and the USA

Oats country, year (variety)	Application					DALA Days	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
GAP, USA	SC	0.20 0.35 / year	93[c] 45[d]	(14 days)	2	not apply after BBCH 71		
USA, 2013 North Rose, NY (Rodeo) Outdoor	SC	0.15 0.20	253 243	59-61 71-73 (14 days)	2	30	0.068, 0.086 (0.077)	TK0163581
	EC	0.15 0.20	253 243	59-61 71-73 (14 days)	2	30	0.11, 0.14 (0.12)	Mean recovery for pydiflumetofen: 97% (n=5) at

Oats country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH[a]	no.	DALA Days	Residues, mg/kg[b]	Ref
USA, 2014 Athens, GA (SS 76-50) Outdoor	SC	0.15 0.20	206 224	61-63 71 (13 days)	2	43	0.33, 0.44 (0.39)	0.01 mg/kg 97% (n=8) at 0.1 mg/kg 99% (n=4) at 1.0-5.0 mg/kg Sampling to analysis: 104-408 days
	EC	0.15 0.20	206 215	61-63 71 (13 days)	2	43	0.48, 0.53 (0.51)	
USA, 2013 Richland, IA (NC Jerry Oats) Outdoor	SC	0.15 0.20	196 178	60 71 (14 days)	2	18	0.065, 0.092 (0.079)	
	EC	0.15 0.20	196 178	60 71 (14 days)	2	18	0.068, 0.087 (0.078)	
USA, 2013 Gardner, ND (Horsepower) Outdoor	SC	0.15 0.21	140 196	67-68 81-83 (12 days)	2	28	0.12, 0.15 (0.14)	
	EC	0.14 0.21	140 187	68-69 80-81 (12 days)	2	28	0.12, 0.13 (0.12)	
USA, 2013 Bagley, IA (Reeves) Outdoor	SC	0.15 0.20	19 19	59-61 71 (12 days)	2	22	0.12, 0.30 (0.21)	
	EC	0.15 0.18	19 19	59-61 71 (12 days)	2	22	0.12, 0.20 (0.16)	
USA, 2013 Campbell, MN (Souris Oats) Outdoor	SC	0.15 0.20	187 187	39 71 (14 days)	2	24 29 34 39 44	0.045 0.051 0.042, 0.047 (0.044) 0.051 0.048	
	EC	0.15 0.20	187 187	39 71 (14 days)	2	34	0.046, 0.066 (0.056)	
USA, 2013 Atlantic, IA (Souris) Outdoor	SC	0.16 0.20	234 271	55-56 71-73 (14 days)	2	7 11 16 21 25	0.19 0.30 0.17, 0.24 (0.21) 0.12 0.093	
	EC	0.15 0.20	224 280	55-56 71-73 (14 days)	2	16	0.27, 0.27 (0.27)	
USA, 2013 Northwood, ND (Jerry) Outdoor	SC	0.15 0.20	140 140	45-47 71 (15 days)	2	46	0.085, 0.11 (0.098)	
	EC	0.14 0.20	140 140	45-47 71 (15 days)	2	46	0.14, 0.14 (0.14)	
USA, 2013 Kirksville, MO (NC Jerry Oats) Outdoor	SC	0.15 0.20	168 178	60 71 (14 days)	2	23	1.4, 2.8 (2.1)	
	EC	0.15 0.20	168 178	60 71 (14 days)	2	23	1.5, 1.5 (1.5)	
USA, 2013 St. Cloud, MN (Morton) Outdoor	SC	0.15 0.20	47 47	39-41 71-72 (14 days)	2	33	<0.010, <0.010 (<0.010)	
	EC	0.14 0.20	47 47	39-41 71-72 (14 days)	2	33	0.40, 0.41 (0.41)	
USA, 2014 Raymondville, TX (VNS)	SC	0.15 0.21	327 337	65-69 71 (14 days)	2	19	0.68, 0.79 (0.73)	

Oats country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH[a]	no.	DALA Days	Residues, mg/kg[b]	Ref
Outdoor	EC	0.15 0.21	337 337	65-69 71 (14 days)	2	19	0.85, 1.0 (0.94)	
USA, 2013 Cleveland, ND (Horsepower) Outdoor	SC	0.14 0.19	187 140	70-71 81-82 (13 days)	2	16	1.2, 1.3 (1.3)	
	EC	0.15 0.21	187 140	70-71 81-82 (13 days)	2	16	1.5, 1.5 (1.5)	
USA, 2013 Carrington, ND (Rockford) Outdoor	SC	0.16 0.20	150 140	44-45 71 (14 days)	2	52	0.22, 0.25 (0.24) c 0.017	
	EC	0.15 0.20	140 140	44-45 71 (14 days)	2	52	0.19, 0.29 (0.24) c 0.017	
USA, 2013 Prosser, NE (Horsepower Oats) Outdoor	SC	0.15 0.21	47 47	61-65 71 (14 days)	2	29	0.38, 0.39 (0.39)	
	EC	0.15 0.20	47 47	61-65 71 (14 days)	2	29	0.44, 0.52 (0.48)	
USA, 2014 San Angelo, TX (TAM 606) Outdoor	SC	0.15 0.20	131 140	39-49 71 (13 days)	2	27	0.36, 0.48 (0.42)	
	EC	0.15 0.20	131 131	39-49 71 (13 days)	2	27	0.46, 0.62 (0.54)	
USA, 2014 Brooklyn, WI (Badger) Outdoor	SC	0.15 0.20	234 243	59-61 71-73 (14 days)	2	28	0.20, 0.20 (0.20)	
	EC	0.15 0.20	234 243	59-61 71-73 (14 days)	2	28	0.36, 0.36 (0.36)	
Canada, 2013 Branchton, ON (Bradley) Outdoor	SC	0.16 0.20	200 200	41-45 71 (15 days)	2	40	0.17, 0.17 (0.17)	TK0163519
	EC	0.15 0.19	200 200	41-45 71 (15 days)	2	40	0.21, 0.24 (0.23)	Mean recovery for pydiflumetofen: 100% (n=5) at 0.01 mg/kg 100% (n=5) at 0.1 mg/kg 97% (n=3) at 5.1 mg/kg
Canada, 2013 St-Marc-sur-Richelieu, QC (RC Amaze) Outdoor	SC	0.16 0.21	200 200	56-57 71-73 (10 days)	2	28	0.63, 0.72 (0.68)	
	EC	0.15 0.20	200 200	56-57 71-73 (10 days)	2	28	0.65, 0.66 (0.66)	
Canada, 2014 Pike Lake, SK (Leggett) Outdoor	SC	0.15 0.19	200 200	51-56 69-71 (14 days)	2	51	0.11, 0.14 (0.13)	Sampling to analysis: 121-536 days
	EC	0.15 0.19	200 200	51-56 69-71 (14 days)	2	51	0.12, 0.17 (0.15)	
Canada, 2013 Hanley, SK (Souris) Outdoor	SC	0.15 0.21	200 200	51-52 71-73 (15 days)	2	37	0.55, 0.63 (0.59)	
	EC	0.15 0.21	200 200	51-52 71-73 (15 days)	2	37	0.63, 0.69 (0.66)	
Canada, 2013 Elgin, MB (Summit) Outdoor	SC	0.15 0.20	45 45	55-59 71-73 (7 days)	2	54	0.084, 0.088 (0.086)	
	EC	0.15	45	55-59	2	54	0.076, 0.095	

Oats country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH[a]	no.	DALA Days	Residues, mg/kg[b]	Ref
		0.20	45	71-73 (7 days)			(0.086)	
Canada, 2013 Rosthern, SK (Leggett) Outdoor	SC	0.14 0.20	45 45	59-61 71-73 (14 days)	2	39	0.43, 0.45 (0.44)	
	EC	0.15 0.20	45 45	59-61 71-73 (14 days)	2	39	0.26, 0.26 (0.26)	
Canada, 2013 Waldheim, SK (Leggett) Outdoor	SC	0.16 0.20	200 200	53-56 71-73 (14 days)	2	52	0.15, 0.15 (0.15)	
	EC	0.16 0.20	200 200	53-56 71-73 (14 days)	2	52	0.19, 0.20 (0.20)	
Canada, 2014 Minto, MB (Pinnacle) Outdoor	SC	0.15 0.20	200 200	51-55 69-71 (14 days)	2	46	0.21, 0.23 (0.22)	
	EC	0.15 0.20	200 200	51-55 69-71 (14 days)	2	36 42 46 51 56	0.091 0.14 0.11, 0.14 (0.13) 0.16 0.14	
Canada, 2013 Carberry, MB (AC Morgan) Outdoor	SC	0.15 0.20	200 200	52-53 71-73 (14 days)	2	36	0.14, 0.14 (0.14)	
	EC	0.15 0.20	200 200	52-53 71-73 (14 days)	2	36	0.14, 0.16 (0.15)	
Canada, 2013 Alvena, SK (Leggett) Outdoor	SC	0.15 0.21	45 50	49-52 71-72 (13 days)	2	56	0.27, 0.28 (0.28)	
	EC	0.15 0.21	45 50	49-52 71-72 (13 days)	2	56	0.30, 0.33 (0.32)	
Canada, 2013 Josephburg, AB (Morgan) Outdoor	SC	0.15 0.21	200 200	47-51 70-71 (15 days)	2	61	0.18, 0.19 (0.19)	
	EC	0.16 0.21	200 200	47-51 70-71 (15 days)	2	61	0.18, 0.18 (0.18)	
Canada, 2013 Minto, MB (Summit) Outdoor	SC	0.15 0.20	200 200	45-52 71-73 (11 days)	2	42	0.10, 0.11 (0.11)	
	EC	0.15 0.20	200 200	45-52 71-73 (11 days)	2	33 38 42 47 53	0.063 0.084 0.092, 0.096 (0.094) 0.054 0.089	

Portion analysed: grain

[a] Re-treatment interval is given in parenthesis.

[b] Mean of replicate field samples is given in parenthesis.

[c] ground application,

[d] aerial application

Maize Cereals

Maize

The Meeting received 20 trials (17 at harvest trials, three decline trials) on field corn and 3 trials (at harvest trials) on popcorn which were conducted in the USA (Oakes, 2015: TK0163563). In each of these trials, pydiflumetofen was applied to maize as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. Two applications at a nominal rate of 0.13 kg ai/ha were made. The first application was made 7 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, grain samples were taken 30 ± 2 DALA. In the decline trials additional grain samples were collected at 20, 25, 35 and 40 ± 2 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from maize grain was 92% at 0.01 mg/kg fortified, 103% at 1.0 mg/kg fortified and 99% at 2.0 mg/kg fortified. Maize grain samples were stored at ca -20 °C for a maximum of 10 months between sampling and analysis.

Table 67 Residues of pydiflumetofen on maize grain from supervised trials in the USA

Maize grains country, year (variety)	Application					DALA Days	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.20 0.25 / year	93[c] 45[d]		2	30		
Field corn								TK0163563
USA, 2014 Alton, NY (Mycogen 232180) Outdoor	SC	0.13 0.13	281 281	83 85-87	2	28	<0.010, <0.010 (<u><0.010</u>)	Mean recovery for pydiflumetofen: 92% (n=10) at 0.01 mg/kg 103% (n=10) at 1.0 mg/kg 99% (n=1) at 2.0 mg/kg Sampling to analysis: 157-315 days
	EC	0.13 0.13	281 281	83 85-87	2	28	<0.010, <0.010 (<0.010)	
USA, 2014 Seven Springs, NC (DeKalb 6805) Outdoor	SC	0.13 0.13	365 327	85 85-86	2	28	<0.010, <0.010 (<u><0.010</u>)	
	EC	0.13 0.13	383 365	85 85-86	2	28	<0.010, <0.010 (<0.010)	
USA, 2014 Fitchburg, WI[e] (G96A69-3111) Outdoor	SC	0.13 0.13	290 187	85-86 86-87	2	31	<0.010, <0.010 (<u><0.010</u>)	
	EC	0.13 0.13	281 196	85-86 86-87	2	31	<0.010, <0.010 (<0.010)	
USA, 2014 Grandview, IA (P1360CHR) Outdoor	SC	0.13 0.12	327 355	85-8787-89	2	31	<0.010, <0.010 (<u><0.010</u>)	
	EC	0.13 0.13	327 365	85-8787-89	2	31	<0.010, <0.010 (<0.010)	
USA, 2014 Dunbar, NE (Producers 7268TXRIB) Outdoor	SC	0.13 0.13	140 131	85-85 85-87	2	28	<0.010, <0.010 (<u><0.010</u>)	
	EC	0.13 0.13	140 131	85-85 85-87	2	28	<0.010, <0.010 (<0.010)	
USA, 2014 Cresco, IA (Pioneer K11) Outdoor	SC	0.13 0.13	234 234	85-85 85-85	2	31	<0.010, <0.010 (<u><0.010</u>)	
	EC	0.13 0.13	234 234	85-85 85-85	2	31	<0.010, <0.010 (<0.010)	
USA, 2014 Cherry Grove, MN (Stine R9424SS Blend) Outdoor	SC	0.13 0.13	234 234	85-85 85-85	2	31	<0.010, <0.010 (<u><0.010</u>)	
	EC	0.13 0.13	234 234	85-85 85-85	2	31	<0.010, <0.010 (<0.010)	
USA, 2014 Hedrick, IA (P1360HR) Outdoor	SC	0.13 0.13	327 355	83-85 85-87	2	29	<0.010, <0.010 (<u><0.010</u>)	
	EC	0.13 0.13	327 365	83-85 85-87	2	29	<0.010, <0.010 (<0.010)	
USA, 2014 Coon Rapids, IA (1142) Outdoor	SC	0.13 0.13	150 168	85-85 85-85	2	28	<0.010, <0.010 (<u><0.010</u>)	
	EC	0.13 0.13	159 168	85-85 85-85	2	28	<0.010, <0.010 (<0.010)	
USA, 2014 Gardner, KS	SC	0.13 0.12	140 140	85-87 87-87	2	28	<0.010, <0.010 (<u><0.010</u>)	

Maize grains country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[ab]	Ref
(5N-410) Outdoor	EC	0.13 0.13	150 150	85-87 87-87	2	28	<0.010, <0.010 (<0.010)	
USA, 2014 Tolna, ND (NuTech 781) Outdoor	SC	0.13 0.12	37 37	85-87 85-87	2	30	<0.010, <0.010 (<0.010)	
	EC	0.13 0.13	37 37	85-87 85-87	2	30	<0.010, <0.010 (<0.010)	
USA, 2014 Kirksville, MO (Pioneer P1498AM) Outdoor	SC	0.13 0.13	47 47	85-85 85-87	2	31	<0.010, <0.010 (<0.010)	
	EC	0.13 0.13	47 47	85-85 85-87	2	31	<0.010, <0.010 (<0.010)	
USA, 2014 Jefferson, IA (J1248) Outdoor	SC	0.13 0.13	19 19	85-85 85-85	2	30	<0.010, <0.010 (<0.010)	
	EC	0.13 0.13	19 19	85-85 85-85	2	30	<0.010, <0.010 (<0.010)	
USA, 2014 Carlyle, IL (FS 66JV1 RIB) Outdoor	SC	0.13 0.13	112 122	85-87 85-87	2	30	<0.010, <0.010 (<0.010)	
	EC	0.13 0.13	112 122	85-87 85-87	2	30	<0.010, <0.010 (<0.010)	
USA, 2014 Fisk, MO (Agventure RL8899YHB) Outdoor	SC	0.13 0.12	187 187	85-85 85-85	2	29	<0.010, <0.010 (<0.010)	
	EC	0.13 0.13	187 187	85-85 85-85	2	29	<0.010, <0.010 (<0.010)	
USA, 2014 Fairfield, IA (P0993HR) Outdoor	SC	0.13 0.13	224 224	87-89 87-89	2	32	<0.010, <0.010 (<0.010)	
	EC	0.13 0.13	206 206	87-89 87-89	2	32	<0.010, <0.010 (<0.010)	
USA, 2014 Northwood, ND (DKC35-54RIB) Outdoor	SC	0.13 0.13	187 187	85-87 87-87	2	30	<0.010, 0.014 (0.012)	
	EC	0.13 0.13	187 187	85-87 87-87	2	30	<0.010, <0.010 (<0.010)	
USA, 2014 Springfield, NE (Pioneer 0987) Outdoor	SC	0.13 0.12	131 131	85-85 87-87	2	19 25 29 33 38	<0.010 <0.010 <0.010, <0.010 (<0.010) <0.010 <0.010	
	EC	0.12 0.13	131 131	85-85 87-87	2	29	<0.010, <0.010 (<0.010)	
USA, 2014 Perry, IA (P1248) Outdoor	SC	0.12 0.13	168 168	85-85 85-85	2	20 26 30 34 40	<0.010 <0.010 <0.010, <0.010 (<0.010) <0.010 <0.010	
	EC	0.12 0.12	178 168	85-85 85-85	2	30	<0.010, <0.010 (<0.010)	
USA, 2014 Raymondville, TX (Dyna-Gro H6284162 Brand 58V24) Outdoor	SC	0.13 0.13	196 196	85-85 85-85	2	29	<0.010, <0.010 (<0.010)	
	EC	0.13 0.13	196 196	85-85 85-85	2	20 24 29 35 42	<0.010 <0.010 <0.010, <0.010 (<0.010) <0.010 <0.010	
Popcorn								
USA, 2014 Fitchburg, WI[f] (Vogel M2101) Outdoor	SC	0.13 0.13	178 187	85-86 86-87	2	31	<0.010, <0.010 (<0.010)	
	EC	0.13 0.13	178 187	85-86 86-87	2	31	<0.010, <0.010 (<0.010)	
USA, 2014 York, NE	SC	0.13 0.13	187 187	79-79 87-87	2	30	<0.010, <0.010 (<0.010)	

Maize grains country, year (variety)	Application					DALA Days	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
(Snowpuff) Outdoor	EC	0.13 0.13	187 187	79-79 87-87	2	30	<0.010, <0.010 (<0.010)	
USA, 2014 San Angelo, TX (Robust R997) Outdoor	SC	0.13 0.13	178 178	85-85 87-87	2	28	<0.010, <0.010 (<0.010) c 0.010	
	EC	0.13 0.13	178 178	85-85 87-87	2	28	<0.010, <0.010 (<0.010) c 0.010	

Portion analysed: grain

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

[e] Address: HOBO unit/NOAA Station474961 at Madison, WI ~1.8 miles, Application dates (1st): 25 Sep 2014

[f] Address: HOBO unit/NOAA Station474961 at Madison, WI ~1.8 miles, Application dates (1st): 07 Oct 2014

Sweet Corns

Sweet corn (Corn-on-the-cob) (kernels plus cob with husk removed)

The Meeting received 12 trials (10 at harvest trials, two decline trials) on sweet corn which were conducted in the USA (Oakes, 2015: TK0223529). In each of these trials, a SC formulations (200 g ai/L) was applied twice to sweet corn as a foliar treatment at a nominal rate of 0.13 kg ai/ha. The first application was made 6 to 9 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, whole ear samples (kernel plus cob with husk) were taken 7 ± 1 DALA. In the decline trials additional samples were collected at 1, 3, 10 and 14 ± 1 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from sweet corn ears was 110% at 0.01 mg/kg fortified and 116% at 1.0 mg/kg fortified. Sweet corn ear samples were stored at ca -18 °C for a maximum of 7.1 months between sampling and analysis.

Table 68 Residues of pydiflumetofen on sweet corn from supervised trials in the USA

Sweet corn country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	*SC*	*0.13* *0.25 / year*	*93*[b] *345*[c]		*2*	*7*		
USA, 2014 Germansville, PA (Mirai 421) Outdoor	SC	0.12 0.12	187 187	65-69 71-73	2	7	<0.010, <0.010 (<u>≤0.010</u>)	TK0223529 Mean recovery for pydiflumetofen: 110% (n=3) at 0.01 mg/kg 116% (n=3) at 1.0 mg/kg Sampling to analysis: 143-217 days
USA, 2014 North Rose, NY (BC-0805) Outdoor	SC	0.12 0.12	215 215	71-73 73-75	2	1 3 7 10 14	<0.010 <0.010 <0.010, <0.010 (<u>≤0.010</u>) <0.010 <0.010	
USA, 2014 Suffolk, VA (Bilicious) Outdoor	SC	0.12 0.13	112 150	67-69 69-71	2	7	<0.010, <0.010 (<u>≤0.010</u>)	
USA, 2014 High Springs, FL (Passion II) Outdoor	SC	0.12 0.12	150 150	64-66 66-67	2	6	<0.010, <0.010 (<u>≤0.010</u>)	
USA, 2014 Gardner, ND (Honey Select TSW) Outdoor	SC	0.13 0.13	47 47	67-69 73-75	2	7	<0.010, <0.010 (<u>≤0.010</u>)	
USA, 2014 Richland, IA	SC	0.12 0.13	224 187	71-71 73-73	2	1 3	<0.010 <0.010	

Sweet corn country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[a]	Ref
(XTRA-Tender 2573 F1) Outdoor						7 10 15	<0.010, <0.010 (<0.010) <0.010 <0.010	
USA, 2014 Lime Springs, IA (Ambrosia) Outdoor	SC	0.12 0.12	234 234	70-70 73-73	2	8	<0.010, <0.010 (<0.010)	
USA, 2014 York, NE (Obsession II) Outdoor	SC	0.12 0.12	178 187	63-65 71-71	2	7	<0.010, <0.010 (<0.010)	
USA, 2014 Geneva, MN (Revelation) Outdoor	SC	0.12 0.12	168 168	71-72 72-73	2	7	<0.010, <0.010 (<0.010)	
USA, 2014 Madera, CA (Silver Queen) Outdoor	SC	0.13 0.12	290 281	71-73 73-75	2	7	<0.010, <0.010 (<0.010)	
USA, 2014 Rupert, IA (I.O Chief Hybrid) Outdoor	SC	0.13 0.13	37 47	60-61 73-75	2	7	<0.010, <0.010 (<0.010)	
USA, 2014 Hillsboro, OR (Honey and Pearl) Outdoor	SC	0.12 0.12	178 178	70-73 73-75	2	7	<0.010, <0.010 (<0.010)	

Portion analysed: ears (kernel + cob with husk removed)

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

Oilseeds and oilfruits

Small seed oilseeds

Rape seed

The Meeting received 21 trials (17 at harvest trials, four decline trials) on rape seed which were conducted in Canada (Sagan, 2015: TK0103848) and the USA (Smith, 2015: TK0163606). In each of these trials, pydiflumetofen was applied to rape seed as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. One application at 0.13 kg ai/ha was followed by a second application at 0.20 kg ai/ha. The first application was made 13–22 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS, COC or MSO. At each trial, rape seed samples were taken 30 ± 2 DALA. In the decline trials additional samples were collected at 20, 25, 35 and 40 ± 1 DALA. Samples were allowed to dry for 0–25 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from rape seed was 102% at 0.01–1.0 mg/kg fortified (Canada) and 104% at 0.01–1.0 mg/kg fortified (USA). Rape seed samples were stored at -10 °C or below for a maximum of 15 months between sampling and analysis.

Table 69 Residues of pydiflumetofen on rape seed supervised trials in Canada and the USA

Rape seed country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
GAP, USA	SC	0.20 0.33 / year	93[d] 45[e]	(14 days)	1-2	30		
USA, 2013 Northwood, ND (5440) Outdoor	SC	0.12 0.20	140 140	62 67 (14 days)	2	20 25 30 35 40	0.26 0.19 0.068, 0.072 (0.070) 0.069 0.026	TK0163606 Mean recovery for pydiflumetofen: 104% (n=4) at 0.01 mg/kg 104% (n=4) at 1.0 mg/kg Sampling to analysis: 211-303 days
	EC	0.13 0.20	140 140	62 67 (14 days)	2	30	0.046, 0.048 (0.047)	
USA, 2013 Gardner, ND (DKL51-45) Outdoor	SC	0.13 0.20	140 187	64-66 75-77 (19 days)	2	25	0.028, 0.030 (0.029) c 0.012	
	EC	0.12 0.22	187 187	64-66 75-77 (18days)	2	25	0.048, 0.051 (0.050) c 0.012	
USA, 2013 Verona, WI (Hyclass 947 RR) Outdoor	SC	0.12 0.20	252 252	78-79 80-81 (14 days)	2	30	0.16, 0.21 (0.18)	
	EC	0.12 0.20	262 252	78-79 80-81 (14 days)	2	30	0.18, 0.19 (0.18)	
USA, 2013 Rice, MN (DKL72-55) Outdoor	SC	0.12 0.20	47 47	64 67 (14 days)	2	31	0.083, 0.092 (0.087)	
	EC	0.12 0.20	47 47	64 67 (14 days)	2	31	0.095, 0.096 (0.095)	
USA, 2013 Carrington, ND (LL 5440) Outdoor	SC	0.12 0.20	140 140	64-65 66-67 (14 days)	2	31	0.030, 0.034 (0.032) c 0.012	
	EC	0.12 0.20	140 140	64-65 66-67 (14 days)	2	31	0.019, 0.020 (0.020) c 0.012	
USA, 2013 Cleveland, ND (DKL51-45) Outdoor	SC	0.13 0.21	196 187	60-65 73-75 (14 days)	2	29	0.15, 0.20 (0.17)	
	EC	0.12 0.20	187 187	60-65 73-75 (14 days)	2	29	0.14, 0.19 (0.17)	
USA, 2013 Jerome, ID (A7191) Outdoor	SC	0.12 0.20	168 159	59-60 65-67 (14 days)	2	32	0.016, 0.016 (0.016)	
	EC	0.12 0.21	168 168	59-60 65-67 (14 days)	2	32	0.020	
USA, 2013 Ephrata, WA (1R072CZZZZ) Outdoor	SC	0.13 0.20	187 187	65-66 79-80 (14 days)	2	20 25 30 35 40	0.020 0.025 0.037, 0.045 (0.041) 0.014 0.023	
	EC	0.12 0.20	187 187	65-66 79-80 (14 days)	2	30	0.018, 0.019 (0.018)	
Canada, 2014 Elm Creek, MB (VT500)	SC	0.12 0.21	200 200	63-65 67 (13 days)	2	29	0.041, 0.054 (0.048)	TK0103848

Rape seed country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH[a]	no.	DALA Days	Residues, mg/kg[bc]	Ref
Outdoor	EC	0.12 0.21	200 200	63-65 67 (13 days)	2	29	0.050, 0.061 (0.056)	Mean recovery for pydiflumetofen: 102% (n=6) at 0.01 mg/kg 109% (n=3) at 0.1 mg/kg 99% (n=6) at 1.0 mg/kg Sampling to analysis: 88-453 days
Canada, 2013 Pike Lake, SK (73-74 RR) Outdoor	SC	0.13 0.20	200 200	63-65 65-69 (14 days)	2	29	0.18, 0.19 (0.19) c 0.028	
	EC	0.13 0.20	200 200	63-65 65-69 (14 days)	2	29	0.049, 0.050 (0.050) c 0.028	
Canada, 2013 Taber, AB (72-55 RR) Outdoor	SC	0.13 0.21	45 45	59-61 67-69 (13 days)	2	31	0.63, 0.74 (0.69)	
	EC	0.12 0.21	45 45	59-61 67-69 (13 days)	2	31	0.055, 0.060 (0.058)	
Canada, 2013 Hanley, SK (V-12-1) Outdoor	SC	0.13 0.20	200 200	67-69 74-75 (15 days)	2	31	0.14, 0.14 (0.14)	
	EC	0.13 0.20	200 200	67-69 74-75 (15 days)	2	31	0.11, 0.12 (0.12)	
Canada, 2013 Elgin, MB[f] (Liberty Link Invigor L120) Outdoor	SC	0.13 0.21	200 200	62-63 66-67 (15 days)	2	30	0.013, 0.028 (0.021)	
	EC	0.13 0.21	200 200	62-63 66-67 (15 days)	2	21 25 30 35 41	0.060 0.011 0.012, 0.014 (0.013) 0.031 0.010	
Canada, 2013 Rosthern, SK (73-45 RR) Outdoor	SC	0.12 0.19	45 45	62-63 64-65 (14 days)	2	29	0.45, 0.46 (0.46) c 0.032	
	EC	0.12 0.19	45 45	62-63 64-65 (14 days)	2	29	0.32, 0.33 (0.33) c 0.032	
Canada, 2013 Carberry, MB (73-75 Dekalb) Outdoor	SC	0.13 0.20	45 45	62-63 67-69 (13 days)	2	31	0.027, 0.068 (0.048)	
	EC	0.13 0.20	45 45	62-63 67-69 (13 days)	2	31	0.025, 0.031 (0.028)	
Canada, 2013 Shilo, MB (73-75 Dekalb) Outdoor	SC	0.13 0.20	200 200	60-62 69-71 (13 days)	2	30	0.036, 0.041 (0.039)	
	EC	0.13 0.20	200 200	60-62 69-71 (13 days)	2	30	0.036, 0.056 (0.046)	
Canada, 2013 Alvena, SK (45H29) Outdoor	SC	0.13 0.22	200 200	64-65 69-74 (13 days)	2	31	0.068, 0.12 (0.094)	
	EC	0.13 0.21	200 200	64-65 69-74 (13 days)	2	31	0.061, 0.068 (0.065)	
Canada, 2013 Wakaw, AK (Roundup Ready) Outdoor	SC	0.14 0.21	200 200	74-75 73-74 (15 days)	2	30	0.31, 0.39 (0.35)	
	EC	0.14 0.20	200 200	74-75 73-74 (15 days)	2	30	0.22, 0.26 (0.24)	
Canada, 2013 Lamont, AB	SC	0.13 0.21	200 200	63-65 67-69	2	29	0.13, 0.16 (0.15)	

Rape seed country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
(RR Cantera 1918) Outdoor				(14 days)				
	EC	0.13 0.21	200 200	63-65 67-69 (14 days)	2	29	0.049, 0.061 (0.055)	
Canada, 2013 Josephburg, AB (RR Cantera 1918) Outdoor	SC	0.13 0.21	200 200	65-67 67-71 (14 days)	2	30	0.057, 0.14 (0.099)	
	EC	0.13 0.21	200 200	65-67 67-71 (14 days)	2	20 24 30 35 41	0.068 0.060 0.044, 0.053 (0.049) 0.099 0.11	
Canada, 2013 Elgin, MB[g] (Liberty Link Invigor L120) Outdoor	SC	0.13 0.21	200 200	60-61 68-69 (22 days)	2	30	0.016, 0.038 (0.027)	
	EC	0.13 0.21	200 200	60-61 68-69 (22 days)	2	30	0.012, 0.016 (0.014)	

Portion analysed: seed

[a] Re-treatment interval is given in parenthesis.

[b] Mean of replicate field samples is given in parenthesis.

[c] c: sample from control plot

[d] ground application,

[e] aerial application

[f] Address: 210 South Railway St, Minto, MB R0K 1M0 (From Elgin, MB on Hwy 23, travel 0.8 km west; turn south for 2.4 km on the gravel road; turn west onto field; travel along edge of field ~ 200 m), Application dates (1st): 15 Jul 2013

[g] Address: 210 South Railway St, Minto, MB R0K 1M0 (From Elgin, MB on Hwy 23; travel 0.8 km west; turn south for 2.4 km on gravel road; turn west onto field and travel along edge of field ~ 200 m), Application dates (1st): 08 Jul 2013

Other oilseeds

Peanut

The Meeting received 12 trials (10 at harvest trials, two decline trials) on peanut which were conducted in the USA (Mäyer, 2015: TK0163595). In each of these trials, pydiflumetofen was applied to peanut as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. Four applications at a nominal rate of 0.050 kg ai/ha were made. The first application was made 56 ± 2 days prior to harvest and subsequent applications were made at 14 day intervals. All applications were made in tank-mix with an adjuvant, NIS, COC or MSO. At each trial, peanut nutmeat samples were taken 14 DALA. In the decline trials additional nutmeat samples were collected at 7, 10, 17 and 21 DALA. Samples were allowed to dry for 4–14 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from peanut nutmeat was 91% at 0.01 mg/kg fortified, 105% at 0.1 mg/kg fortified and 103% at 1 mg/kg fortified. Nutmeat samples were stored at -10 °C or below for a maximum of 11 months between sampling and analysis.

Table 70 Residues of pydiflumetofen on peanut from supervised trials in the USA

Peanut country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.05 0.20 / year	93[b] 45[c]		4	14		
USA, 2013 Chula, GA[d] (Georgia 06G) Outdoor	SC	0.050 0.051 0.051 0.053	178 187 187 178	73-74 75-76 78-80 82-83	4	14	<0.01, <0.01 (<u><0.01</u>)	TK0163545 Mean recovery for pydiflumetofen: 91% (n=5) at
	EC	0.050 0.050	178 178	73-74 75-76	4	7 10	<0.01 <0.01	

Peanut country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[a]	Ref
		0.050 0.051	187 178	78-80 82-83		14 17 21	<0.01, <0.01 (<0.01) <0.01 <0.01	0.01 mg/kg 105% (n=1) at 0.1 mg/kg 103% (n=4) at 1 mg/kg
USA, 2013 Colbert, GA (Georgia 06G) Outdoor	SC	0.051 0.050 0.051 0.051	234 224 243 243	71-73 73-75 75-77 77-79	4	14	<0.01, <0.01 (<0.01)	Sampling to analysis: 231-349 days
	EC	0.051 0.050 0.050 0.050	234 224 243 243	71-73 73-75 75-77 77-79	4	14	<0.01, <0.01 (<0.01)	
USA, 2013 Athens, GA (Georgia 06G) Outdoor	SC	0.050 0.050 0.050 0.050	224 224 234 234	73-75 75-77 75-78 77-79	4	7 10 14 17 21	<0.01 <0.01 <0.01, <0.01 (<0.01) <0.01 <0.01	
	EC	0.050 0.051 0.051 0.051	234 224 234 234	73-75 75-77 75-78 77-79	4	14	0.017, 0.019 (0.018)	
USA, 2013 Blackville, SC (Gregory) Outdoor	SC	0.051 0.051 0.051 0.051	131 131 131 131	69-71 61-63 77-81 82-84	4	14	<0.01, <0.01 (<0.01)	
	EC	0.051 0.050 0.051 0.051	131 131 131 131	69-71 61-63 77-81 82-84	4	14	<0.01, <0.01 (<0.01)	
USA, 2013 Lenox, GA (Georgia 06G) Outdoor	SC	0.048 0.051 0.050 0.050	47 47 47 37	70-71 72-73 75-77 83-84	4	14	<0.01, <0.01 (<0.01)	
	EC	0.053 0.052 0.052 0.052	47 47 47 37	70-71 72-73 75-77 83-84	4	14	<0.01, <0.01 (<0.01)	
USA, 2013 Mt. Olive, NC (Bailey) Outdoor	SC	0.052 0.053 0.051 0.051	47 28 47 47	73 75 81 83-85	4	15	<0.01, <0.01 (<0.01)	
	EC	0.050 0.052 0.050 0.051	37 28 37 47	73 75 81 83-85	4	15	<0.01, <0.01 (<0.01)	
USA, 2013 Chula, GA[e] (Georgia 06G) Outdoor	SC	0.050 0.050 0.050 0.050	168 215 215 215	81-82 83-84 85-87 88-89	4	14	<0.01, <0.01 (<0.01)	
	EC	0.050 0.050 0.050 0.050	168 215 215 206	81-82 83-84 85-87 88-89	4	14	<0.01, <0.01 (<0.01)	
USA, 2013 Seven Springs, NC (Bailey) Outdoor	SC	0.050 0.049 0.049 0.051	150 252 271 262	71 75 79-81 85-88	4	12	<0.01, <0.01 (<0.01)	
	EC	0.051 0.051 0.051 0.051	150 262 281 271	71 75 79-81 85-88	4	12	<0.01, <0.01 (<0.01)	

Peanut country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[a]	Ref
USA, 2013 Oviedo, FL (Georgia 06G) Outdoor	SC	0.051 0.052 0.050 0.050	281 290 281 281	81 83 85 87	4	14	<0.01, <0.01 (<u>0.01</u>)	
	EC	0.051 0.052 0.051 0.050	281 290 281 281	81 83 85 87	4	14	<0.01, <0.01 (<0.01)	
USA, 2013 Charlotte, TX (Florida 07) Outdoor	SC	0.051 0.051 0.051 0.050	178 206 196 206	77-79 77-79 77-79 81-85	4	14	0.016, 0.021 (0.018)	
	EC	0.051 0.051 0.050 0.050	178 206 196 206	77-79 77-79 77-79 81-85	4	14	<0.01, 0.015 (0.012)	
USA, 2013 Hinton, OK (Tamnut OL06) Outdoor	SC	0.051 0.050 0.051 0.050	112 187 140 206	75-79 76-79 79-81 79-81	4	15	<0.01, <0.01 (<u>0.01</u>)	
	EC	0.050 0.051 0.051 0.050	112 187 140 206	75-79 76-79 79-81 79-81	4	15	<0.01, <0.01 (<0.01)	
USA, 2013 Dill City, OK (Tamnut OL06) Outdoor	SC	0.050 0.051 0.050 0.052	252 178 178 215	73-75 74-76 77-79 81-82	4	15	<0.01, <0.01 (<0.01)	
	EC	0.050 0.050 0.051 0.051	252 178 178 206	73-75 74-76 77-79 81-82	4	15	<0.01, 0.013 (0.012)	

Portion analysed: nutmeat

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

[d] Address: SEAgR Chula, GA Weather Station ~0.25 miles, Application dates (1st): 27 Aug 2013

[e] Address: Georgia Automated Monitoring Network ~4 miles, Application dates (1st): 20 Sep 2013

Animal feeds

Legume animal feeds

Pea vines and hay

The Meeting received five trials (four at harvest trials, one decline trial) on pea vines and hay which were conducted in the USA (Oakes, 2015: TK0103860). In each of these trials, pydiflumetofen was applied to peas as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. Two applications at a nominal rate of 0.20 kg ai/ha were made. The first application was made 14 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, vines and hay samples were taken 14 DALA. In the decline trials additional samples were collected at 7, 10, 17 and 21 DALA. Hay samples were allowed to dry for 3–18 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from pea vines was 107% at 0.01–25 mg/kg fortified and from pea hay was 100% at 0.01–25 mg/kg fortified. Pea vines and hay samples were stored at -18 °C or below for a maximum of 18 and 23 months between sampling and analysis.

Table 71 Residues of pydiflumetofen on pea vines and hay from supervised trials in USA

Pea vines & hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
GAP, USA	SC	0.20 0.40/ yr	93[b] 45[c]		2	0			
USA, 2013 Mount Hood, OR (Columbia Green Pea) Outdoor	SC	0.21 0.20	168 150	16-17 59-60	2	14	Vines Hay	0.62, 1.1 (0.88) 2.0, 4.8 (3.4)	TK0163595 Mean recovery for pydiflumetofen: 109% (n=3) at 0.01 mg/kg 110% (n=2) at 0.1 mg/kg 93% (n=1) at 25 mg/kg for vines, 99% (n=4) at 0.01 mg/kg 103% (n=2) at 0.1 mg/kg 94% (n=1) at 20 mg/kg 103% (n=3) at 25 mg/kg for hay Sampling to analysis: 377-553 days for vines, 374-710 days for hay
	EC	0.19 0.20	159 159	16-17 59-60	2	14	Vines Hay	0.40, 0.54 (0.47) 1.8, 1.9 (1.8)	
USA, 2013 Jerome, ID (Latah Yellow) Outdoor	SC	0.21 0.20	187 187	35-37 59-63	2	14	Vines Hay	0.21, 0.28 (0.25) 1.3, 1.8 (1.5)	
	EC	0.20 0.21	187 187	35-37 59-63	2	14	Vines Hay	0.35, 0.36 (0.36) 1.6, 2.1 (1.8)	
USA, 2013 Payette, ID (Austrian Winter Pea) Outdoor	SC	0.20 0.20	234 234	36-39 51-55	2	14	Vines Hay	2.7, 2.9 (2.8) 16, 18 (17)	
	EC	0.20 0.20	234 234	36-39 51-55	2	14	Vines Hay	1.5, 1.7 (1.6) 8.9, 11 (10)	
USA, 2013 Acequia, ID (Cascadia) Outdoor	SC	0.20 0.20	122 122	13-15 37-39	2	7 10 14 17 21	Vines	0.84 0.51 0.23, 0.23 (0.23) 0.24 0.12	
						7 10 14 17 21	Hay	6.3 3.4 1.5, 1.6 (1.6) 1.5 0.85	
	EC	0.20 0.20	122 122	13-15 37-39	2	14	Vines Hay	0.40, 0.45 (0.42) 3.0, 3.1 (3.0)	
USA, 2013 American Falls, ID (Progress #9) Outdoor	SC	0.20 0.20	37 37	33-35 38-60	2	14	Vines Hay	0.78, 1.0 (0.90) 4.8, 7.1 (5.9)	
	EC	0.20 0.20	37 37	33-35 38-60	2	14	Vines Hay	0.68, 0.76 (0.72) 4.0, 4.0 (4.0)	

Vines: received basis, Hay: dry weight basis

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

Peanut hay

The Meeting received 12 trials (10 at harvest trials, two decline trials) on peanut which were conducted in the USA (Smith, 2015: TK0163545). In each of these trials, pydiflumetofen was applied to peanut as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. Four applications at a nominal rate of 0.050 kg ai/ha were made. The first application was made 56 ± 2 days prior to harvest and subsequent applications were made at 14 day intervals. All applications were made in tank-mix with an adjuvant, NIS, COC or MSO. At each trial, peanut hay samples were taken 14 DALA. In the decline trials additional nutmeat samples were collected at 7, 10, 17 and 21 DALA. Samples were allowed to dry for 4–14 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from peanut hay was 102% at 0.01 mg/kg fortified, 118% at 1 mg/kg fortified and 82% at 50 mg/kg fortified. Peanut hay samples were stored at -10 °C or below for a maximum of 12 months between sampling and analysis.

Table 72 Residues of pydiflumetofen on peanut hay from supervised trials in the USA

Peanut hay country, year (variety)	Application					DALA Days	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.050 0.20 / year	93[c] 45[d]		4	14		
USA, 2013 Chula, GA[e] (Georgia 06G) Outdoor	SC	0.050 0.051 0.051 0.053	178 187 187 178	73-74 75-76 78-80 82-83	4	14	4.7, 5.2 (5.0)	TK0163545 Mean recovery for pydiflumetofen: 102% (n=5) at 0.01 mg/kg 118% (n=5) at 1 mg/kg 82% (n=1) at 50 mg/kg Sampling to analysis: 261-357 days
	EC	0.050 0.050 0.050 0.051	178 178 187 178	73-74 75-76 78-80 82-83	4	7 10 14 17 21	4.0 6.6 5.4, 9.8 (7.6) 12 5.1	
USA, 2013 Colbert, GA (Georgia 06G) Outdoor	SC	0.051 0.050 0.051 0.051	234 224 243 243	71-73 73-75 75-77 77-79	4	14	5.0, 6.8 (5.9)	
	EC	0.051 0.050 0.050 0.050	234 224 243 243	71-73 73-75 75-77 77-79	4	14	12, 12 (12)	
USA, 2013 Athens, GA (Georgia 06G) Outdoor	SC	0.050 0.050 0.050 0.050	224 224 234 234	73-75 75-77 75-78 77-79	4	7 10 14 17 21	4.8 4.0 2.5, 3.4 (3.0) 3.1 2.5	
	EC	0.050 0.051 0.051 0.051	234 224 234 234	73-75 75-77 75-78 77-79	4	14	4.2, 4.4 (4.3)	
USA, 2013 Blackville, SC (Gregory) Outdoor	SC	0.051 0.051 0.051 0.051	131 131 131 131	69-71 61-63 77-81 82-84	4	14	6.9, 7.8 (7.4)	
	EC	0.051 0.050 0.051 0.051	131 131 131 131	69-71 61-63 77-81 82-84	4	14	12, 14 (13)	
USA, 2013 Lenox, GA (Georgia 06G) Outdoor	SC	0.048 0.051 0.050 0.050	47 47 47 37	70-71 72-73 75-77 83-84	4	14	3.7, 5.5 (4.6)	
	EC	0.053 0.052 0.052 0.052	47 47 47 37	70-71 72-73 75-77 83-84	4	14	4.3, 5.1 (4.7)	
USA, 2013 Mt. Olive, NC (Bailey) Outdoor	SC	0.052 0.053 0.051 0.051	47 28 47 47	73 75 81 83-85	4	15	4.1, 4.2 (4.1)	
	EC	0.050 0.052 0.050 0.051	37 28 37 47	73 75 81 83-85	4	15	12, 12 (12)	
USA, 2013 Chula, GA[f] (Georgia 06G) Outdoor	SC	0.050 0.050 0.050 0.050	168 215 215 215	81-82 83-84 85-87 88-89	4	14	0.012, 0.023 (0.018) c 0.027	
	EC	0.050 0.050 0.050 0.050	168 215 215 206	81-82 83-84 85-87 88-89	4	14	0.037, 0.039 (0.038) c 0.027	

Peanut hay country, year (variety)	Application					DALA	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.	Days		
USA, 2013 Seven Springs, NC (Bailey) Outdoor	SC	0.050 0.049 0.049 0.051	150 252 271 262	71 75 79-81 85-88	4	12	15, 16 (15)	
	EC	0.051 0.051 0.051 0.051	150 262 281 271	71 75 79-81 85-88	4	12	15, 15 (15)	
USA, 2013 Oviedo, FL (Georgia 06G) Outdoor	SC	0.051 0.052 0.050 0.050	281 290 281 281	81 83 85 87	4	14	4.2, 4.3 (4.3)	
	EC	0.051 0.052 0.051 0.050	281 290 281 281	81 83 85 87	4	14	7.4, 11 (9.2)	
USA, 2013 Charlotte, TX (Florida 07) Outdoor	SC	0.051 0.051 0.051 0.050	178 206 196 206	77-79 77-79 77-79 81-85	4	14	3.0, 3.2 (3.1)	
	EC	0.051 0.051 0.050 0.050	178 206 196 206	77-79 77-79 77-79 81-85	4	14	2.1, 2.8 (2.4)	
USA, 2013 Hinton, OK (Tamnut OL06) Outdoor	SC	0.051 0.050 0.051 0.050	112 187 140 206	75-79 76-79 79-81 79-81	4	15	2.7, 3.3 (3.0) c 0.051	
	EC	0.050 0.051 0.051 0.050	112 187 140 206	75-79 76-79 79-81 79-81	4	15	4.5, 4.5 (4.5) c 0.051	
USA, 2013 Dill City, OK (Tamnut OL06) Outdoor	SC	0.050 0.051 0.050 0.052	252 178 178 215	73-75 74-76 77-79 81-82	4	15	0.79, 1.2 (1.0)	
	EC	0.050 0.050 0.051 0.051	252 178 178 206	73-75 74-76 77-79 81-82	4	15	1.9, 2.0 (2.0)	

Portion analysed: hay, dry weight basis

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

[e] Address: SEAgR Chula, GA Weather Station ~0.25 miles, Application dates (1st): 27 Aug 2013

[f] Address: Georgia Automated Monitoring Network ~4 miles, Application dates (1st): 20 Sep 2013

Soya bean forage and hay

The Meeting received 21 trials (17 at harvest trials, four decline trials) on soya bean forage and hay which were conducted in the USA (Oakes, 2015: TK0103860). In each of these trials, pydiflumetofen was applied to soya bean as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. Two applications at a nominal rate of 0.15 kg ai/ha were made. The first application was made 7 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, forage and hay samples were taken 0 DALA. In the decline trials additional samples were collected at 3, 7, 14 ± 1 DALA. Hay samples were allowed to dry for 1–12 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from soya bean forage was 89% at 0.01 mg/kg fortified, 90% at 0.1 mg/kg

fortified and 79% at 10–40 mg/kg fortified, and from soya bean hay was 99% at 0.01 mg/kg fortified, 98% at 0.1 mg/kg fortified and 104% at 20–100 mg/kg fortified. Soya bean forage and hay samples were stored at -18 °C or below for a maximum of 20 months between sampling and analysis.

Table 73 Residues of pydiflumetofen on soya bean forage and hay from supervised trials in the USA

Soya bean forage & hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
GAP, Argentina	SC	0.045	100[c] 15[d]		2	30			
GAP, CA & USA	Do not feed soybean forage and hay.								
USA, 2013 Chula, GA (Pioneer 97M50) Outdoor	SC	0.15 0.15	178 187	72-73 73-74	2	0 0	Forage Hay	7.5, 9.3 (8.4) 45, 47 (46)	TK0103860 Mean recovery for pydiflumetofen: 89% (n=8) at 0.01 mg/kg 90% (n=9) at 0.1 mg/kg 79% (n=4) at 10-40 mg/kg for forage, 99% (n=8) at 0.01 mg/kg 98% (n=9) at 0.1 mg/kg 104% (n=4) at 20-100 mg/kg for hay Sampling to analysis: 268-611 days for forage, 316-580 days for hay
	EC	0.15 0.15	178 187	72-73 73-74	2	0 0	Forage Hay	7.6, 7.7 (7.7) 14, 22 (18)	
USA, 2013 Seven Springs, NC (Asgrow 6931) Outdoor	SC	0.15 0.15	140 159	71 73	2	0 0	Forage Hay	14, 15 (15) 33, 40 (37)	
	EC	0.14 0.15	131 159	71 73	2	0 0	Forage Hay	13, 17 (15) 37, 39 (38)	
USA, 2013 Pollard, AR (5N451R2) Outdoor	SC	0.15 0.15	187 187	71-72 72-73	2	0 0	Forage Hay	3.1, 3.3 (3.2) 22, 25 (23) c 0.013	
	EC	0.15 0.15	187 187	71-72 72-73	2	0 0	Forage Hay	4.9, 6.3 (5.6) 20, 25 (22) c 0.013	
USA, 2013 Fisk, MO (5N451R2) Outdoor	SC	0.15 0.15	187 187	71-72 72-73	2	0 0	Forage Hay	6.5, 6.9 (6.7) 21, 27 (24) c 0.011	
	EC	0.15 0.14	187 187	71-72 72-73	2	0 3 8 14	Forage	6.6, 6.8 (6.7) 6.7 2.8 2.4	
						0 3 8 14	Hay	21, 28 (24) 19 6.6 4.1 c 0.011	
USA, 2013 Proctor, AR (HBK LL4950) Outdoor	SC	0.15 0.15	140 140	71-72 74-75	2	0 0	Forage Hay	5.3, 6.7 (6.0) 13, 15 (14)	
	EC	0.15 0.15	140 140	71-72 74-75	2	0 0	Forage Hay	7.0, 7.5 (7.3) 12, 16 (14)	
USA, 2013 Jefferson, IA (93Y13) Outdoor	SC	0.15 0.14	103 234	69-69 70-71	2	0 3 7 14	Forage	24, 25 (24) 13 9.4 7.7	
						0 3 7 14	Hay	86, 96 (91) 61 61 37	
	EC	0.14 0.14	103 243	69-69 70-71	2	0 0	Forage Hay	14, 16 (15) 55, 60 (57)	
USA, 2013 Gardner, KS (Willcross RR2428N) Outdoor	SC	0.15 0.15	37 37	71-72 73-74	2	0 0	Forage Hay	6.8, 8.0 (7.4) 21, 28 (25)	
	EC	0.15 0.15	37 37	71-72 73-74	2	0 0	Forage Hay	8.5, 9.5 (9.0) 24, 33 (28)	
USA, 2013 Geneva, MN	SC	0.15 0.15	168 168	67-69 69-71	2	0 0	Forage Hay	9.9, 10 (10) 27, 30 (29)	

Soya bean forage & hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
(Pioneer 91Y41) Outdoor	EC	0.15 0.15	168 168	67-69 69-71	2	0 0	Forage Hay	12, 12 (12) 42, 49 (45)	
USA, 2013 Lawrence, KS (MG3981-NR2) Outdoor	SC	0.14 0.14	140 140	69-70 71-72	2	0 0	Forage Hay	7.3, 8.3 (7.8) 38, 45 (41)	
	EC	0.15 0.14	140 140	69-70 71-72	2	0 0	Forage Hay	4.6, 8.2 (6.4) 41, 43 (42)	
USA, 2013 Perry, IA (93Y13) Outdoor	SC	0.15 0.14	19 19	65-65 69-69	2	0 0	Forage Hay	15, 17 (16) 41, 43 (42)	
	EC	0.15 0.15	19 19	65-65 69-69	2	0 0	Forage Hay	8.0, 14 (11) 34, 45 (40)	
USA, 2013 York, NE (S28-U7) Outdoor	SC	0.15 0.15	196 196	65-65 70-70	2	0 0	Forage Hay	11, 11 (11) 64, 65 (64)	
	EC	0.15 0.15	196 196	65-65 70-70	2	0 0	Forage Hay	9.6, 9.7 (9.6) 45, 55 (50)	
USA, 2013 Stafford, KS (93Y72) Outdoor	SC	0.15 0.15	206 206	71-72 71-71	2	0 0	Forage Hay	8.8, 9.6 (9.2) 23, 29 (26)	
	EC	0.15 0.15	206 206	71-72 71-71	2	0 0	Forage Hay	8.8, 10 (9.5) 23, 30 (26)	
USA, 2013 Royalton, MN (PB 0954RR) Outdoor	SC	0.14 0.15	187 187	64-65 65-66	2	0 0	Forage Hay	11, 13 (12) 37, 42 (39) c0.012	
	EC	0.14 0.15	187 187	64-65 65-66	2	0 0	Forage Hay	12, 12 (12) 38, 39 (39) c0.012	
USA, 2013 Northwood, ND (11R02 RR2Y) Outdoor	SC	0.15 0.15	140 140	64-65 70-72	2	0 0	Forage Hay	8.8, 8.9 (8.9) 40, 55 (48)	
	EC	0.15 0.15	140 140	64-65 70-72	2	0 0	Forage Hay	9.4, 10 (9.8) 59, 66 (63)	
USA, 2013 Springfield, NE (NK28U7) Outdoor	SC	0.15 0.14	131 131	69-71 71-73	2	0 0	Forage Hay	7.6, 8.4 (8.0) 40, 40 (40)	
	EC	0.15 0.15	131 131	69-71 71-73	2	0 0	Forage Hay	6.6, 7.7 (7.1) 32, 33 (33)	
USA, 2013 Verona, WI (NK S20-Y2) Outdoor	SC	0.14 0.15	196 224	67-69 69-71	2	0 0	Forage Hay	8.8, 8.9 (8.9) 51, 53 (52)	
	EC	0.15 0.15	196 224	67-69 69-71	2	0 0	Forage Hay	7.3, 8.9 (8.1) 51, 60 (55)	
USA, 2013 Hedrick, IA (93Y15-PA35) Outdoor	SC	0.15 0.15	168 168	71 75	2	0 4 6 14	Forage	4.6, 6.6 (5.6) 5.4 7.0 5.6	
						0 4 6 14	Hay	31, 44 (38) 31 20 22 c 0.011	
	EC	0.15 0.15	168 168	71 75	2	0 0	Forage Hay	5.4, 6.5 (6.0) 32, 32 (32) c 0.011	
USA, 2013 Bloomfield, MS (R2 46X29) Outdoor	SC	0.15 0.15	187 196	72-73 73-74	2	0 0	Forage Hay	4.1, 4.3 (4.2) 11, 11 (11)	
	EC	0.15 0.15	187 187	72-73 73-74	2	0 3 8 14	Forage	2.6, 3.2 (2.9) 2.3 1.5 1.2	

Soya bean forage & hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
						0 3 8 14	Hay	13, 14 (14) 11 7.5 4.8	
USA, 2013 Campbell, MN (Asgrow 0832) Outdoor	SC	0.15 0.15	187 187	65-66 69-69	2	0 0	Forage Hay	8.6, 12 (10) 43, 64 (53)	
	EC	0.15 0.15	187 187	65-66 69-69	2	0 0	Forage Hay	12, 12 (12) 56, 60 (58)	
USA, 2013 Richland, IA (93Y60) Outdoor	SC	0.15 0.15	215 215	71 75	2	0 0	Forage Hay	7.6, 9.5 (8.5) 41, 61 (51)	
	EC	0.15 0.15	215 215	71 75	2	0 0	Forage Hay	8.0, 8.7 (8.4) 39, 42 (40)	
USA, 2013 Cooper, IA (93Y13) Outdoor	SC	0.15 0.15	103 103	65-65 69-69	2	0 0	Forage Hay	14, 21 (17) 58, 62 (60)	
	EC	0.15 0.15	112 103	65-65 69-69	2	0 0	Forage Hay	17, 18 (17) 78, 79 (79)	

Forage: received basis, Hay: dry weight basis

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

Straw, fodder and forage of cereal grains

Barley straw

The Meeting received 21 trials (17 at harvest trials, four decline trials) on barley which were conducted in Canada (Sagan, 2015: TK0163517) and the USA (Oakes, 2015: TK0163584). In each of these trials, pydiflumetofen was applied to barley as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. One application at 0.15 kg ai/ha was followed by a second application at 0.20 kg ai/ha. The first application was made 9–15 days prior to the second application, and the second application was made at BBCH 71. All applications were made in tank-mix with an adjuvant, NIS, COC or MSO. At each trial, straw samples were taken at normal grain harvest maturity which occurred at 16–68 days after the second (last) application. In the decline trials additional straw samples were collected at 10 and 5 ± 1 day before normal harvest, and 5 and 10 ± 1 days after normal harvest.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from barley straw was 92% at 0.01–26 mg/kg fortified (Canada) and 93% at 0.01–50 mg/kg fortified (USA). Barley straw samples were stored at -10 °C or below (Canada) or at -18 °C or below (USA) for a maximum of 18 months between sampling and analysis.

Table 74 Residues of pydiflumetofen on barley straw from supervised trials in Canada and the USA

Barley straw country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
GAP, USA	SC	0.20 0.35 / year	93[d] 45[e]	(14 days)	1-2	Do not apply after *BBCH 71*		
USA, 2013 Suffolk, VA (Atlantic) Outdoor	SC	0.15 0.21	122 112	55-56 71-71 (15 days)	2	36	6.2, 7.5 (6.8) c 0.014	TK0163584
	EC	0.15 0.21	122 112	55-56 71-71 (15 days)	2	36	7.1, 7.1 (7.1) c 0.014	Mean recovery for pydiflumetofen: 91% (n=4) at 0.01 mg/kg 91% (n=5) at 0.1 mg/kg 98% (n=5) at 10-50 mg/kg
USA, 2013 Richland, IA (Robust) Outdoor	SC	0.15 0.20	168 262	50 73 (13 days)	2	49 54 59 63 68	2.5 0.58 1.0, 3.3 (2.2) 2.4 1.5	

Barley straw country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
	EC	0.15 0.20	59	50 73 (13 days)	2	59	2.1, 2.6 (2.3)	Sampling to analysis: 103-561 days
USA, 2013 York, NE (Lacey) Outdoor	SC	0.15 0.21	187 150	45-45 73-73 (14 days)	2	21	5.3, 6.5 (5.9)	
	EC	0.14 0.20	187 150	45-45 73-73 (14 days)	2	21	5.6, 7.4 (6.5)	
USA, 2013 Bagley, IA (Robust) Outdoor	SC	0.14 0.21	94 112	61-61 71-71 (13 days)	2	28	6.4, 7.5 (7.0) c 0.012	
	EC	0.16 0.20	103 103	61-61 71-71 (13 days)	2	28	15, 17 (16) c 0.012	
USA, 2013 Cleveland, ND (Tradition) Outdoor	SC	0.16 0.21	47 47	70-71 81-82 (13 days)	2	16	14, 16 (15)	
	EC	0.15 0.21	56 47	70-71 81-82 (13 days)	2	16	14, 15 (14)	
USA, 2013 Grand Island, NE (Lacey) Outdoor	SC	0.15 0.20	150 196	45-45 73-73 (14 days)	2	27	3.6, 6.0 (4.8)	
	EC	0.15 0.20	150 196	45-45 73-73 (14 days)	2	27	5.8, 7.0 (6.4)	
USA, 2013 Carrington, ND (Robust) Outdoor	SC	0.15 0.20	140 140	45-45 71-71 (14 days)	2	52	5.2, 5.6 (5.4)	
	EC	0.15 0.20	140 140	45-45 71-71 (14 days)	2	52	6.3, 7.2 (6.7)	
USA, 2013 Prosser, NE (Lacey) Outdoor	SC	0.15 0.20	168 168	61-65 71-71 (14 days)	2	24 29 34 39 45	3.4 1.9 1.7, 1.8 (1.7) 1.7 1.5	
	EC	0.15 0.20	168 168	61-65 71-71 (14 days)	2	34	1.9, 3.4 (2.7)	
USA, 2013 Jerome, ID (Moravian 69) Outdoor	SC	0.15 0.20	187 159	61-65 71-73 (14 days)	2	45	1.1, 1.1 (1.1)	
	EC	0.15 0.20	187 159	61-65 71-73 (14 days)	2	45	1.1, 1.7 (1.4)	
USA, 2013 Porterville, CA (UC937) Outdoor	SC	0.15 0.20	290 281	69-71 73-77 (13 days)	2	21	4.4, 7.0 (5.7) c 0.029	
	EC	0.15 0.20	290 281	69-71 73-77 (13 days)	2	21	5.1, 5.6 (5.3) c 0.029	
USA, 2013 Parkdale, OR (Haybet) Outdoor	SC	0.15 0.20	37 47	69-69 85-85 (13 days)	2	26	6.9, 8.9 (7.9)	
	EC	0.15 0.19	37 47	69-69 85-85 (13 days)	2	26	8.0, 8.2 (8.1)	
USA, 2013 Payette, ID (Westbred 501) Outdoor	SC	0.15 0.20	234 234	41-45 71-71 (14 days)	2	44	2.4, 2.6 (2.5)	
	EC	0.15 0.21	234 243	41-45 71-71 (14 days)	2	44	1.2, 1.4 (1.3)	

Barley straw country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
Canada, 2013 Taber, AB (CDC Austenson) Outdoor	SC	0.16 0.21	200	58-65 69-73 (13 days)	2	36	5.3, 6.3 (5.8)	TK0163517
	EC	0.16 0.21	200	58-65 69-73 (13 days)	2	36	3.6, 3.8 (3.7)	Mean recovery for pydiflumetofen: 94% (n=6) at 0.01 mg/kg 94% (n=5) at 10 mg/kg 83% (n=3) at 26 mg/kg
Canada, 2013 Boissevain, MB (Legacy) Outdoor	SC	0.16 0.22	45	39-41 71-73 (13 days)	2	47	1.5, 2.2 (1.9)	
	EC	0.16 0.21	45	39-41 71-73 (13 days)	2	47	0.67, 1.3 (0.99)	
Canada, 2013 Elgin, MB (Bentley) Outdoor	SC	0.15 0.21	45	43-49 68-71 (9 days)	2	42	3.8, 4.1 (4.0)	Sampling to analysis: 151-204 days
	EC	0.15 0.22	45	43-49 68-71 (9 days)	2	42	1.8, 2.5 (2.2)	
Canada, 2013 Rosthern, SK (AC Metcalf) Outdoor	SC	0.14 0.20	200	59-61 69-71 (14 days)	2	41	11, 19 (15)	
	EC	0.14 0.20	200	59-61 69-71 (14 days)	2	41	16, 20 (18)	
Canada, 2013 Waldheim, SK (AC Metcalf) Outdoor	SC	0.16 0.20	200	51-61 71-75 (14 days)	2	50	8.6, 11 (9.8)	
	EC	0.16 0.20	200	51-61 71-75 (14 days)	2	50	7.6, 8.1 (7.9)	
Canada, 2013 Carberry, MB (Conlon) Outdoor	SC	0.15 0.20	200	53-54 71 (15 days)	2	40	3.5, 4.2 (3.9)	
	EC	0.15 0.20	200	53-54 71 (15 days)	2	40	3.0, 3.3 (3.2)	
Canada, 2013 Josephburg, AB (Coalition) Outdoor	SC	0.16 0.22	45	47-52 71-73 (15 days)	2	48	4.5, 4.6 (4.6)	
	EC	0.15 0.21	45	47-52 71-73 (15 days)	2	48	1.8, 2.4 (2.1)	
Canada, 2013 Minto, MB[f] (Bentley) Outdoor	SC	0.15 0.20	200	41-45 71,77 (13 days)	2	42	1.5, 1.9 (1.7)	
	EC	0.15 0.20	200	41-45 71-77 (13 days)	2	31 38 42 46 51	1.0 1.2 1.6, 1.9 (1.8) 1.5 1.8	
Canada, 2013 Minto, MB[g] (AC Metcalf) Outdoor	SC	0.16 0.21	200	39-41 69-73 (11 days)	2	48	2.7, 3.9 (3.3)	
	EC	0.16 0.21	200	39-41 69-73 (11 days)	2	38 43 48 52 57	1.5 2.0 1.2, 1.4 (1.3) 2.1 1.8	

Portion analysed: straw, as received basis

[a] Re-treatment interval is given in parenthesis.

[b] Mean of replicate field samples is given in parenthesis.

[c] c: sample from control plot

[d] ground application,

[e] aerial application

[f] Address: 210 South Railway St, Minto, MB R0K 1M0 (From Minto, drive 2.4 km north on Hwy #10, turning east on gravel road 29N at Double Diamond Farm Supply. Travel east for 3.5 km. The trials are located immediately north of this road.), Application dates (1st): 03 Jul 2013

[g] Address: 210 South Railway St, Minto, MB R0K 1M0 (From Minto, travel north on Hwy #10 for 0.8 km, turn east on gravel road at Manitoba Hydro station. Travel 0.8 km to farm entrance, turn south and travel ~ 300 m. Turn west and travel ~ 400 m to the trial site.), Application dates (1st): 05 Jul 2013

Barley hay

The Meeting received 21 trials (17 at harvest trials, four decline trials) on barley which were conducted in Canada (Sagan, 2015: TK0163517) and the USA (Oakes, 2015: TK0163584). In each of these trials, pydiflumetofen was applied to barley as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. A single application at 0.15 kg ai/ha was made at BBCH 31. All applications were made in tank-mix with an adjuvant, NIS, COC or MSO. At each trial, hay samples were taken 7 ± 1 DALA. In the decline trials additional samples were collected at 0, 3, 10 and 14 ± 1 DALA. Hay samples were allowed to dry for 2–28 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from barley hay was 84% at 0.01–82 mg/kg fortified (Canada) and 93% at 0.01–100 mg/kg fortified (USA). Barley hay samples were stored at -10 °C or below (Canada) or at -18 °C or below (USA) for a maximum of 20 months between sampling and analysis.

Table 75 Residues of pydiflumetofen on barley hay from supervised trials in Canada and the USA

Barley hay country, year (variety)	Application					DALA Days	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.15	93[c] 45[d]		1	7		
USA, 2013 Suffolk, VA (Atlantic) Outdoor	SC	0.15	112	30-31	1	6	4.3, 4.5 (4.4)	TK0163584 Mean recovery for pydiflumetofen: 90% (n=5) at 0.01 mg/kg 86% (n=5) at 0.1 mg/kg 102% (n=5) at 10-100 mg/kg Sampling to analysis: 148-595 days
	EC	0.15	112	30-31	1	6	4.0, 4.1 (4.1)	
USA, 2013 Richland, IA (Robust) Outdoor	SC	0.15	168	31	1	0 3 7 10 13	79 25 3.0, 3.1 (3.1) 2.4 1.6	
	EC	0.15	168	31	1	7	8.6, 9.8 (9.2)	
USA, 2013 York, NE (Lacey) Outdoor	SC	0.15	187	30-31	1	7	4.0, 4.4 (4.2)	
	EC	0.15	187	30-31	1	7	4.1, 4.2 (4.2)	
USA, 2013 Bagley, IA (Robust) Outdoor	SC	0.15	187	25-31	1	7	4.8, 6.2 (5.5)	
	EC	0.15	196	25-31	1	7	4.5, 5.4 (4.9)	
USA, 2013 Cleveland, ND (Tradition) Outdoor	SC	0.16	47	30-35	1	8	10, 11 (11) c 0.010	
	EC	0.15	47	30-35	1	8	5.0, 6.6 (5.8) c 0.010	
USA, 2013 Grand Island, NE (Lacey) Outdoor	SC	0.15	187	30-31	1	8	1.2, 1.7 (1.4)	
	EC	0.15	187	30-31	1	8	0.81, 0.83 (0.82)	
USA, 2013 Carrington, ND (Robust) Outdoor	SC	0.14	140	25-25	1	7	4.0, 6.1 (5.1)	
	EC	0.15	140	25-25	1	7	7.9, 8.9 (8.4)	
USA, 2013 Prosser, NE (Lacey) Outdoor	SC	0.15	168	29-30	1	0 3 7 10 15	19 9.1 4.4, 5.2 (4.8) 2.6 0.51	

Barley hay country, year (variety)	Application					DALA	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.	Days		
							c 0.023	
	EC	0.15	168	29-30	1	7	24, 28 (26) c 0.023	
USA, 2013 Jerome, ID (Moravian 69) Outdoor	SC	0.15	187	29-31	1	7	6.5, 8.3 (7.4)	
	EC	0.14	187	29-31	1	7	9.9, 11 (10)	
USA, 2013 Porterville, CA (UC937) Outdoor	SC	0.15	281	30-31	1	6	3.5, 5.1 (4.3)	
	EC	0.15	281	30-31	1	6	6.8, 6.9 (6.8)	
USA, 2013 Parkdale, OR (Haybet) Outdoor	SC	0.15	37	30-31	1	7	8.2, 8.3 (8.3)	
	EC	0.15	37	30-31	1	7	8.8, 9.6 (9.2)	
USA, 2013 Payette, ID (Westbred 501) Outdoor	SC	0.15	243	29-31	1	7	12, 12 (12)	
	EC	0.15	243	29-31	1	7	14, 15 (14)	
Canada, 2013 Taber, AB (CDC Austenson) Outdoor	SC	0.16	200	29-31	1	8	4.1, 4.6 (4.4)	TK0163517
	EC	0.16	200	29-31	1	8	3.1, 3.9 (3.5)	
Canada, 2013 Boissevain, MB (Legacy) Outdoor	SC	0.15	45	30-31	1	7	4.8, 5.3 (5.1)	Mean recovery for pydiflumetofen: 90% (n=6) at 0.01 mg/kg 81% (n=5) at 10 mg/kg 77% (n=3) at 82 mg/kg Sampling to analysis: 209-241 days
	EC	0.14	45	30-31	1	7	3.8, 4.4 (4.1)	
Canada, 2013 Elgin, MB (Bentley) Outdoor	SC	0.16	45	30-31	1	7	5.4, 5.9 (5.7)	
	EC	0.15	45	30-31	1	7	3.0, 3.2 (3.1)	
Canada, 2013 Rosthern, SK (AC Metcalf) Outdoor	SC	0.14	200	29-31	1	8	5.2, 5.8 (5.5)	
	EC	0.14	200	29-31	1	8	3.6, 4.4 (4.0)	
Canada, 2013 Waldheim, SK (AC Metcalf) Outdoor	SC	0.15	200	22-32	1	6	17, 17 (17)	
	EC	0.15	200	22-32	1	6	13, 15 (14)	
Canada, 2013 Carberry, MB (Conlon) Outdoor	SC	0.15	200	31	1	7	10, 15 (13)	
	EC	0.15	200	31	1	7	12, 12 (12)	
Canada, 2013 Josephburg, AB (Coalition) Outdoor	SC	0.16	45	31-32	1	7	5.1, 6.8 (6.0)	
	EC	0.16	45	31-32	1	7	2.9, 3.3 (3.1)	
Canada, 2013 Minto, MB[e] (Bentley) Outdoor	SC	0.15	200	30-31	1	7	4.0, 4.0 (4.0)	
	EC	0.15	200	30-31	1	0 3 7 10 15	72 42 3.5, 3.5 (3.5) 1.5 0.91	
Canada, 2013 Minto, MB[f] (AC Metcalf) Outdoor	SC	0.15	200	14-15/31	1	7	1.5, 1.8 (1.7)	
	EC	0.15	200	14-15/31	1	0 3 7 10 14	57 9.8 2.5, 3.0 (2.8) 1.8 0.36	

Portion analysed: hay, dry weight basis

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

[e] Address: 210 South Railway St, Minto, MB R0K 1M0 (From Minto, drive 2.4 km north on Hwy #10, turning east on gravel road 29N at Double Diamond Farm Supply. Travel east for 3.5 km. The trials are located immediately north of this road.), Application dates (1st): 17 Jun 2013

[f] Address: 210 South Railway St, Minto, MB R0K 1M0 (From Minto, travel north on Hwy #10 for 0.8 km, turn easton gravel road at Manitoba Hydro station. Travel 0.8 km to farm entrance, turn south and travel ~ 300 m. Turn west and travel ~ 400 m to the trial site.), Application dates (1st): 21 Jun 2013

Maize forage

The Meeting received 20 trials (15 at harvest trials, five decline trials) on field corn which were conducted in the USA (Oakes, 2015: TK0163563). In each of these trials, pydiflumetofen was applied to maize as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. A single application at 0.25 kg ai/ha was made. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, forage samples were taken 7 ± 1 DALA. In the decline trials additional samples were collected at 0, 1, 3, 10, 14, 21 and 28 ± 1 DALA (not all dates at all trials).

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from field corn forage was 101% at 0.01 mg/kg fortified, 98% at 1.0 mg/kg fortified and 104% at 6.0–10 mg/kg fortified. Maize forage samples were stored at -18 °C or below for a maximum of 9.9 months between sampling and analysis.

Table 76 Residues of pydiflumetofen on field corn forage from supervised trials in the USA

Maize forage country, year (variety)	Application					DALA Days	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.20 0.25 / year	93[c] 45[d]		1	7		
Field corn								TK0163563
USA, 2014 Alton, NY (Mycogen 232180) Outdoor	SC	0.25	281	75-79	1	7	0.54, 1.3 (0.92)	Mean recovery for pydiflumetofen: 101% (n=8) at 0.01 mg/kg 98% (n=9) at 1.0 mg/kg 104% (n=2) at 6.0-10 mg/kg Sampling to analysis: 189-302 days
	EC	0.25	281	75-79	1	7	1.2, 1.4 (1.3)	
USA, 2014 Seven Springs, NC (DeKalb 6805) Outdoor	SC	0.25	327	79-80	1	7	0.56, 0.58 (0.57)	
	EC	0.25	327	79-80	1	7	0.43, 1.4 (0.79)	
USA, 2014 Fitchburg, WI (G96A69-3111) Outdoor	SC	0.25	178	71-72	1	7	0.35, 0.41 (0.38)	
	EC	0.25	178	71-72	1	7	0.28, 0.41 (0.34)	
USA, 2014 Grandview, IA (P1360CHR) Outdoor	SC	0.25	150	81-83	1	0 1 3 7 10 14 21 28	0.99 1.4 0.89 1.0, 1.1 (1.0) 0.59 0.39 0.33 0.40	
	EC	0.26	159	81-83	1	7	1.2, 0.86 (1.0)	
USA, 2014 Dunbar, NE (Producers 7268TXRIB) Outdoor	SC	0.25	131	75-76	1	0 1 3 7 10 14 21 28	2.5 3.1 2.5 2.3, 2.5 (2.4) 2.1 2.4 1.7 2.1	
	EC	0.25	131	75-76	1	7	1.4, 1.7 (1.6)	
USA, 2014	SC	0.25	234	74-74	1	7	0.39, 0.58 (0.49)	

Maize forage country, year (variety)	Application					DALA	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.	Days		
Cresco, IA (Pioneer K11) Outdoor	EC	0.25	234	74-74	1	7	1.5, 1.8 (1.6)	
USA, 2014 Cherry Grove, MN (Stine R9424SS Blend) Outdoor	SC	0.25	234	72-72	1	7	0.52, 0.77 (0.64)	
	EC	0.25	234	72-72	1	7	0.57, 0.82 (0.69)	
USA, 2014 Hedrick, IA (P1360HR) Outdoor	SC	0.25	122	75-76	1	7	0.33, 0.56 (0.45)	
	EC	0.25	122	75-76	1	7	0.36, 0.51 (0.43)	
USA, 2014 Coon Rapids, IA (1142) Outdoor	SC	0.25	215	73-74	1	7	0.35, 0.99 (0.67) c 0.010	
	EC	0.25	215	73-74	1	7	0.13, 1.0 (0.58) c 0.010	
USA, 2014 Gardner, KS (5N-410) Outdoor	SC	0.24	140	75-75	1	7	0.88, 2.3 (1.6) c 0.014	
	EC	0.26	150	75-75	1	7	2.1, 2.1 (2.1) c 0.014	
USA, 2014 Tolna, ND (NuTech 781) Outdoor	SC	0.25	37	83-83	1	6	1.6, 2.5 (2.0)	
	EC	0.25	37	83-83	1	6	1.7, 1.8 (1.8)	
USA, 2014 Kirksville, MO (Pioneer P1498AM) Outdoor	SC	0.26	47	73-75	1	7	1.1, 1.8 (1.5)	
	EC	0.25	47	73-75	1	7	1.3, 1.5 (1.4)	
USA, 2014 Jefferson, IA (J1248) Outdoor	SC	0.27	19	71-72	1	7	0.56, 0.72 (0.64)	
	EC	0.26	19	71-72	1	7	0.13, 0.21 (0.17)	
USA, 2014 Carlyle, IL (FS 66JV1 RIB) Outdoor	SC	0.26	112	73-75	1	7	0.81, 1.0 (0.91)	
	EC	0.26	112	73-75	1	7	0.53, 1.1 (0.81)	
USA, 2014 Fisk, MO (Agventure RL8899YHB) Outdoor	SC	0.25	187	79-83	1	7	1.7, 1.7 (1.7)	
	EC	0.25	187	79-83	1	7	1.8, 2.6 (2.2)	
USA, 2014 Fairfield, IA (P0993HR) Outdoor	SC	0.25	206	81-83	1	7	1.3, 1.4 (1.3)	
	EC	0.25	206	81-83	1	7	1.2, 1.3 (1.3)	
USA, 2014 Northwood, ND (DKC35-54RIB) Outdoor	SC	0.25	187	83-83	1	7	0.84, 1.2 (1.0)	
	EC	0.26	187	83-83	1	7	0.81, 0.84 (0.82)	
USA, 2014 Springfield, NE (Pioneer 0987) Outdoor	SC	0.25	140	75-77	1	0 2 7 9 14	3.1 2.1 1.1, 1.1 (1.1) 2.8 0.30	
	EC	0.25	140	75-77	1	7	0.84, 1.2 (1.0)	
USA, 2014 Perry, IA (P1248) Outdoor	SC	0.25	206	73-73	1	0 3 7 10 14	2.9 0.66 0.32, 0.34 (0.33) 0.74 0.33	
	EC	0.25	206	73-73	1	7	0.91, 1.1 (1.0)	
USA, 2014	SC	0.27	196	75-79	1	7	4.5, 5.2 (4.9)	

Maize forage country, year (variety)	Application					DALA Days	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
Raymondville, TX (Dyna-Gro H6284162 Brand 58V24) Outdoor	EC	0.27	196	75-79	1	0 3 7 10 14	5.7 5.2 3.6, 5.3 (4.4) 2.6 3.0	

Portion analysed: forage, as received basis

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

The Meeting received 12 trials (10 at harvest trials, two decline trials) on sweet corn which were conducted in the USA (Oakes, 2015: and TK0223529). In each of these trials, an SC formulation (200 g ai/L) was applied twice to sweet corn as a foliar treatment at a nominal rate of 0.13 kg ai/ha. The first application was made 6 to 9 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, forage samples were taken 7 ± 1 DALA. In the decline trials additional samples were collected at 1, 3, 10 and 14 ± 1 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from sweet corn forage was 118% at 0.01 mg/kg fortified, 118% at 1.0 mg/kg fortified, 86% at 10 mg/kg fortified and 90% at 15 mg/kg fortified. Sweet corn forage samples were stored at ca -18 °C for a maximum of 9.6 months between sampling and analysis.

Table 77 Residues of pydiflumetofen on sweet corn forage from supervised trials in the USA

Sweet corn forage country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.125 0.25 / year	93[b] 45[c]		2	7		
USA, 2014 Germansville, PA (Mirai 421) Outdoor	SC	0.12 0.12	187 187	65-69 71-73	2	7	0.66, 0.79 (0.73)	TK0223529 Mean recovery for pydiflumetofen: 118% (n=5) at 0.01 mg/kg 118% (n=4) at 1.0 mg/kg 86% (n=3) at 10 mg/kg 90% (n=1) at 15 mg/kg Sampling to analysis: 142-293 days
USA, 2014 North Rose, NY (BC-0805) Outdoor	SC	0.12 0.12	215 215	71-73 73-75	2	1 3 7 10 14	0.65 0.76 0.66, 0.94 (0.80) 0.47 0.65	
USA, 2014 Suffolk, VA (Bilicious) Outdoor	SC	0.12 0.13	112 150	67-69 69-71	2	7	0.57, 0.92 (0.75)	
USA, 2014 High Springs, FL (Passion II) Outdoor	SC	0.12 0.12	150 150	64-66 66-67	2	6	0.85, 1.2 (1.0)	
USA, 2014 Gardner, ND (Honey Select TSW) Outdoor	SC	0.13 0.13	47 47	67-69 73-75	2	7	0.40, 0.48 (0.44)	
USA, 2014 Richland, IA (XTRA-Tender 2573 F1) Outdoor	SC	0.12 0.13	224 187	71-71 73-73	2	1 3 7 10 15	4.0 3.6 1.0, 1.4 (1.2) 1.1 0.77	
USA, 2014 Lime Springs, IA (Ambrosia) Outdoor	SC	0.12 0.12	234 234	70-70 73-73	2	8	0.41, 0.57 (0.49)	
USA, 2014 York, NE (Obsession II)	SC	0.12 0.12	178 187	63-65 71-71	2	7	0.87, 0.94 (0.90)	

Sweet corn forage country, year (variety)	Application					DALA Days	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
Outdoor								
USA, 2014 Geneva, MN (Revelation) Outdoor	SC	0.12 0.12	168 168	71-72 72-73	2	7	1.2, 1.3 (1.2)	
USA, 2014 Madera, CA (Silver Queen) Outdoor	SC	0.13 0.12	290 281	71-73 73-75	2	7	3.4, 4.5 (3.9)	
USA, 2014 Rupert, IA (I.O Chief Hybrid) Outdoor	SC	0.13 0.13	37 47	60-61 73-75	2	7	0.68, 0.69 (0.68)	
USA, 2014 Hillsboro, OR (Honey and Pearl) Outdoor	SC	0.12 0.12	178 178	70-73 73-75	2	7	0.44, 1.0 (0.73)	

Portion analysed: forage, as received basis

[a] Mean of replicate field samples is given in parenthesis.

[b] ground application,

[c] aerial application

Maize fodder

The Meeting received 20 trials (17 at harvest trials, three decline trials) on field corn and 3 trials (at harvest trials) on popcorn which were conducted in the USA (Oakes, 2015: TK0163563). In each of these trials, pydiflumetofen was applied to maize as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. Two applications at a nominal rate of 0.13 kg ai/ha were made. The first application was made 7 ± 1 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, stover samples were taken 30 ± 2 DALA. In the decline trials additional stover samples were collected at 20, 25, 35 and 40 ± 2 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from maize stover as 96% at 0.01 mg/kg fortified, 103% at 1.0 mg/kg fortified and 102% at 3.0–15 mg/kg fortified. Maize stover samples were stored at ca -20 °C for a maximum of 9.1 months between sampling and analysis.

Table 78 Residues of pydiflumetofen on maize stover from supervised trials in the USA

Maize stover country, year (variety)	Application					DALA Days	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
GAP, USA	SC	0.20 0.25 / year	93[c] 45[d]		2	30		
Field corn								TK0163563
USA, 2014 Alton, NY (Mycogen 232180) Outdoor	SC	0.13 0.13	281 281	83 85-87	2	28	0.77, 0.88 (0.82)	Mean recovery for pydiflumetofen: 96% (n=9) at 0.01 mg/kg 103% (n=9) at 1.0 mg/kg 102% (n=2) at 3.0-15 mg/kg Sampling to analysis: 171-277 days
	EC	0.13 0.13	281 281	83 85-87	2	28	0.51, 0.61 (0.56)	
USA, 2014 Seven Springs, NC (DeKalb 6805) Outdoor	SC	0.13 0.13	365 327	85 85-86	2	28	2.9, 3.4 (3.1)	
	EC	0.13 0.13	383 365	85 85-86	2	28	2.1, 3.7 (2.9)	
USA, 2014 Fitchburg, WI[e] (G96A69-3111) Outdoor	SC	0.13 0.13	290 187	85-86 86-87	2	31	3.5, 3.5 (3.5)	
	EC	0.13 0.13	281 196	85-86 86-87	2	31	3.0, 3.7 (3.4)	
USA, 2014 Grandview, IA (P1360CHR) Outdoor	SC	0.13 0.12	327 355	85- 8787- 89	2	31	1.8, 2.5 (2.1)	
	EC	0.13	327	85-	2	31	3.8, 6.2 (5.0)	

Maize stover country, year (variety)	Application					DALA Days	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.			
		0.13	365	8787-89				
USA, 2014 Dunbar, NE (Producers 7268TXRIB) Outdoor	SC	0.13 0.13	140 131	85-85 85-87	2	28	3.1, 6.5 (4.8)	
	EC	0.13 0.13	140 131	85-85 85-87	2	28	3.8, 4.0 (3.9)	
USA, 2014 Cresco, IA (Pioneer K11) Outdoor	SC	0.13 0.13	234 234	85-85 85-85	2	31	1.3, 1.6 (1.5)	
	EC	0.13 0.13	234 234	85-85 85-85	2	31	1.9, 2.0 (1.9)	
USA, 2014 Cherry Grove, MN (Stine R9424SS Blend) Outdoor	SC	0.13 0.13	234 234	85-85 85-85	2	31	3.3, 3.7 (3.5)	
	EC	0.13 0.13	234 234	85-85 85-85	2	31	2.3, 2.4 (2.4)	
USA, 2014 Hedrick, IA (P1360HR) Outdoor	SC	0.13 0.13	327 355	83-85 85-87	2	29	1.4, 1.9 (1.6)	
	EC	0.13 0.13	327 365	83-85 85-87	2	29	2.0, 2.3 (2.1)	
USA, 2014 Coon Rapids, IA (1142) Outdoor	SC	0.13 0.13	150 168	85-85 85-85	2	28	0.94, 1.2 (1.1) c 0.035	
	EC	0.13 0.13	159 168	85-85 85-85	2	28	0.80, 0.92 (0.86) c 0.035	
USA, 2014 Gardner, KS (5N-410) Outdoor	SC	0.13 0.12	140 140	85-87 87-87	2	28	3.0, 3.4 (3.2)	
	EC	0.13 0.13	150 150	85-87 87-87	2	28	3.3, 3.7 (3.5)	
USA, 2014 Tolna, ND (NuTech 781) Outdoor	SC	0.13 0.12	37 37	85-87 85-87	2	30	3.6, 4.1 (3.8)	
	EC	0.13 0.13	37 37	85-87 85-87	2	30	3.9, 4.5 (4.2)	
USA, 2014 Kirksville, MO (Pioneer P1498AM) Outdoor	SC	0.13 0.13	47 47	85-85 85-87	2	31	1.2, 1.7 (1.4)	
	EC	0.13 0.13	47 47	85-85 85-87	2	31	1.2, 1.8 (1.5)	
USA, 2014 Jefferson, IA (J1248) Outdoor	SC	0.13 0.13	19 19	85-85 85-85	2	30	0.43, 0.46 (0.44)	
	EC	0.13 0.13	19 19	85-85 85-85	2	30	0.93, 1.6 (1.3)	
USA, 2014 Carlyle, IL (FS 66JV1 RIB) Outdoor	SC	0.13 0.13	112 122	85-87 85-87	2	30	1.5, 2.0 (1.7)	
	EC	0.13 0.13	112 122	85-87 85-87	2	30	2.5, 2.7 (2.6)	
USA, 2014 Fisk, MO (Agventure RL8899YHB) Outdoor	SC	0.13 0.12	187 187	85-85 85-85	2	29	2.8, 3.1 (3.0) c 0.019	
	EC	0.13 0.13	187 187	85-85 85-85	2	29	3.3, 4.1 (3.7) c 0.019	
USA, 2014 Fairfield, IA (P0993HR) Outdoor	SC	0.13 0.13	224 224	87-89 87-89	2	32	1.4, 2.0 (1.7)	
	EC	0.13 0.13	206 206	87-89 87-89	2	32	2.2, 2.4 (2.3)	
USA, 2014 Northwood, ND (DKC35-54RIB) Outdoor	SC	0.13 0.13	187 187	85-87 87-87	2	30	3.0, 3.3 (3.2)	
	EC	0.13 0.13	187 187	85-87 87-87	2	30	2.0, 3.2 (2.6)	
USA, 2014 Springfield, NE (Pioneer 0987) Outdoor	SC	0.13 0.12	131 131	85-85 87-87	2	19 25 29 33 38	2.1 1.6 0.92, 1.3 (1.1) 1.1 0.93	
	EC	0.12 0.13	131 131	85-85 87-87	2	29	1.3, 2.0 (1.6)	

Maize stover country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[ab]	Ref
USA, 2014 Perry, IA (P1248) Outdoor	SC	0.12 0.13	168 168	85-85 85-85	2	20 26 30 34 40	2.6 2.2 1.6, 2.2 (1.9) 3.0 2.9 c 0.020	
	EC	0.12 0.12	178 168	85-85 85-85	2	30	1.1, 2.3 (1.7) c 0.020	
USA, 2014 Raymondville, TX (Dyna-Gro H6284162 Brand 58V24) Outdoor	SC	0.13 0.13	196 196	85-85 85-85	2	29	12, 14 (13)	
	EC	0.13 0.13	196 196	85-85 85-85	2	20 24 29 35 42	16 14 11, 12 (12) 11 7.3	
Popcorn								
USA, 2014 Fitchburg, WI[f] (Vogel M2101) Outdoor	SC	0.13 0.13	178 187	85-86 86-87	2	31	2.3, 2.4 (2.3)	
	EC	0.13 0.13	178 187	85-86 86-87	2	31	3.4, 3.5 (3.4)	
USA, 2014 York, NE (Snowpuff) Outdoor	SC	0.13 0.13	187 187	79-79 87-87	2	30	1.1, 1.4 (1.2)	
	EC	0.13 0.13	187 187	79-79 87-87	2	30	1.4, 1.8 (1.6)	
USA, 2014 San Angelo, TX (Robust R997) Outdoor	SC	0.13 0.13	178 178	85-85 87-87	2	28	4.5, 4.9 (4.7)	
	EC	0.13 0.13	178 178	85-85 87-87	2	28	4.3, 5.6 (5.0)	

Portion analysed: stover, as received basis

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

[e] Address: HOBO unit/NOAA Station474961 at Madison, WI ~1.8 miles, Application dates (1st): 25 Sep 2014

[f] Address: HOBO unit/NOAA Station474961 at Madison, WI ~1.8 miles, Application dates (1st): 07 Oct 2014

The Meeting received 12 trials (10 at harvest trials, two decline trials) on sweet corn which were conducted in the USA (Oakes, 2015: TK0223529). In each of these trials, a SC formulations (200 g ai/L) was applied twice to sweet corn as a foliar treatment at a nominal rate of 0.13 kg ai/ha. The first application was made 6 to 9 days prior to the second applications. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, stover samples were taken 7 ± 1 DALA. In the decline trials additional samples were collected at 1, 3, 10 and 14 ± 1 DALA. Samples were allowed to dry for 2–14 days before collection

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from sweet corn stover as 97% at 0.01 mg/kg fortified, 106% at 1.0 mg/kg fortified and 83% at 15 mg/kg fortified. Sweet corn stover samples were stored at ca -18 °C for a maximum of 9.6 months between sampling and analysis.

Table 79 Residues of pydiflumetofen on sweet corn stover from supervised trials in the USA

Sweet corn stover country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Residues, mg/kg[ab]	Ref
GAP, USA	SC	0.125 0.25 / year	93[c] 45[d]		2	7		
USA, 2014 Germansville, PA (Mirai 421) Outdoor	SC	0.12 0.12	187 187	65-69 71-73	2	7	1.9, 2.4 (2.2)	TK0223529 Mean recovery for

Sweet corn stover country, year (variety)	Application					DALA	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.	Days		
USA, 2014 North Rose, NY (BC-0805) Outdoor	SC	0.12 0.12	215 215	71-73 73-75	2	1 3 7 10 14	2.9 1.6 1.5, 1.6 (1.5) 1.6 1.3 c 0.017	pydiflumetofen: 97% (n=3) at 0.01 mg/kg 106% (n=3) at 1.0 mg/kg 83% (n=3) at 15 mg/kg
USA, 2014 Suffolk, VA (Bilicious) Outdoor	SC	0.12 0.13	112 150	67-69 69-71	2	7	1.5, 2.7 (2.1)	
USA, 2014 High Springs, FL (Passion II) Outdoor	SC	0.12 0.12	150 150	64-66 66-67	2	6	1.3, 1.9 (1.6)	Sampling to analysis: 148-293 days
USA, 2014 Gardner, ND (Honey Select TSW) Outdoor	SC	0.13 0.13	47 47	67-69 73-75	2	7	1.2, 1.9 (1.6) c 0.024	
USA, 2014 Richland, IA (XTRA-Tender 2573 F1) Outdoor	SC	0.12 0.13	224 187	71-71 73-73	2	1 3 7 10 15	11 12 3.7, 4.5 (4.1) 2.6 3.3	
USA, 2014 Lime Springs, IA (Ambrosia) Outdoor	SC	0.12 0.12	234 234	70-70 73-73	2	8	0.72, 0.86 (0.79)	
USA, 2014 York, NE (Obsession II) Outdoor	SC	0.12 0.12	178 187	63-65 71-71	2	7	1.3, 1.7 (1.5)	
USA, 2014 Geneva, MN (Revelation) Outdoor	SC	0.12 0.12	168 168	71-72 72-73	2	7	2.2, 2.4 (2.3)	
USA, 2014 Madera, CA (Silver Queen) Outdoor	SC	0.13 0.12	290 281	71-73 73-75	2	7	3.9, 9.3 (6.6)	
USA, 2014 Rupert, IA (I.O Chief Hybrid) Outdoor	SC	0.13 0.13	37 47	60-61 73-75	2	7	1.9, 2.4 (2.1)	
USA, 2014 Hillsboro, OR (Honey and Pearl) Outdoor	SC	0.12 0.12	178 178	70-73 73-75	2	7	0.66, 1.1 (0.88)	

Portion analysed: stover, dry weight basis

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

Oats forage and hay

The Meeting received 28 trials (24 at harvest trials, four decline trials) on oats which were conducted in Canada (Sagan, 2015: TK0163519) and the USA (McDonald, 2015: TK0163581). In each of these trials, pydiflumetofen was applied to oats as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. A single application at 0.15 kg ai/ha was made at BBCH 31. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, forage and hay samples were

taken 7 ± 2 DALA. In the decline trials additional samples were collected at 0, 3, 10 and 14 ± 1 DALA. Hay samples were allowed to dry for 2–36 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from oat forage was 100% at 0.01–51 mg/kg fortified (Canada) and 93% at 0.01–10 mg/kg fortified (USA), and from oat hay was 100% at 0.01–81 mg/kg fortified (Canada) and 95% at 0.01–25 mg/kg fortified (USA). Oat forage and hay samples were stored at -10 °C or below (Canada) or at ca -18 °C (USA) for a maximum of 25 months between sampling and analysis.

Table 80 Residues of pydiflumetofen on oat forage and hay from supervised trials in Canada and the USA

Oats forage & hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
GAP, USA	SC	0.20	93[c] 45[d]		1	7			
USA, 2013 North Rose, NY (Rodeo) Outdoor	SC	0.14	253	32-33	1	7	Forage Hay	0.56, 0.61 (0.58) 0.83, 1.1 (0.95)	TK0163581 Mean recovery for pydiflumetofen: 88% (n=5) at 0.01 mg/kg 101% (n=7) at 0.1 mg/kg 85% (n=4) at 1.0-10 mg/kg for forage, 99% (n=5) at 0.01 mg/kg 92% (n=8) at 0.1 mg/kg 97% (n=6) at 1.0-25 mg/kg for hay Sampling to analysis: 153-443 days for forage, 147-768 days for hay
	EC	0.14	243	32-33	1	7	Forage Hay	1.2, 1.3 (1.2) 1.4, 2.4 (1.9)	
USA, 2014 Athens, GA (SS 76-50) Outdoor	SC	0.15	280	30-32	1	7	Forage Hay	1.0, 1.0 (1.0) c 0.012 2.2, 2.3 (2.3)	
	EC	0.15	280	30-32	1	7	Forage Hay	1.5, 2.1 (1.8) c 0.012 4.3, 4.3 (4.3)	
USA, 2013 Richland, IA (NC Jerry Oats) Outdoor	SC	0.15	159	32	1	7	Forage Hay	2.6, 2.8 (2.7) 6.2, 8.9 (7.6)	
	EC	0.15	159	32	1	7	Forage Hay	2.7, 3.1 (2.9) 7.7, 8.8 (8.2)	
USA, 2013 Gardner, ND (Horsepower) Outdoor	SC	0.15	150	30-31	1	7	Forage Hay	2.7, 2.8 (2.7) 4.8, 5.5 (5.2) c 0.028	
	EC	0.16	150	30-31	1	7	Forage Hay	2.3, 2.7 (2.5) 4.8, 6.2 (5.5) c 0.028	
USA, 2013 Bagley, IA (Reeves) Outdoor	SC	0.15	19	31	1	7	Forage Hay	0.55, 0.51 (0.53) 6.1, 2.0 (4.1) c 0.031	
	EC	0.14	19	31	1	7	Forage Hay	1.3, 1.4 (1.3) 2.7, 3.5 (3.1) c 0.031	
USA, 2013 Campbell, MN (Souris Oats) Outdoor	SC	0.15	187	31	1	0 3 7 10 14	Forage	2.3 1.2 0.57, 0.65 (0.61) 0.55 0.33	
						0 3 7 10 14	Hay	20 8.6 5.1, 5.6 (5.3) 5.7 2.1	
	EC	0.15	187	31	1	7	Forage Hay	0.71, 0.78 (0.75) 4.0, 4.7 (4.3)	

Oats forage & hay country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Commodity	Residues, mg/kg[a]	Ref
USA, 2013 Atlantic, IA (Souris) Outdoor	SC	0.14	234	31	1	0 3 7 10 14	Forage	2.7 1.4 0.93, 1.1 (1.0) 0.58 0.34	
						0 3 7 10 14	Hay	16 6.2 0.81, 0.99 (0.90) 0.69 0.64 c 0.094	
	EC	0.15	252	31	1	7	Forage Hay	0.92, 1.0 (0.98) 0.88, 0.94 (0.91) c 0.094	
USA, 2013 Verona, WI (Badger) Outdoor	SC	0.15	196	30-61	1	7	Forage Hay	1.2, 1.3 (1.2) 3.7, 5.2 (4.5)	
	EC	0.15	196	30-61	1	7	Forage Hay	2.1, 2.5 (2.3) 5.8, 6.0 (5.9)	
USA, 2013 Northwood, ND (Jerry) Outdoor	SC	0.15	140	31	1	7	Forage Hay	1.9, 1.9 (1.9) 3.7, 3.8 (3.7) c 0.010	
	EC	0.14	140	31	1	7	Forage Hay	1.4, 2.3 (1.9) 2.5, 2.6 (2.6) c 0.010	
USA, 2013 Kirksville, MO (NC Jerry Oats) Outdoor	SC	0.15	168	31	1	7	Forage Hay	3.4, 3.7 (3.5) 6.1, 8.1 (7.1)	
	EC	0.15	178	31	1	7	Forage Hay	3.6, 3.8 (3.7) 7.1, 7.8 (7.5)	
USA, 2013 St. Cloud, MN (Morton) Outdoor	SC	0.14	47	31	1	7	Forage Hay	0.36, 0.59 (0.47) 0.54, 0.66 (0.60)	
	EC	0.15	47	31	1	7	Forage Hay	0.56, 0.73 (0.65) 0.46, 0.53 (0.49)	
USA, 2014 Raymondville, TX (VNS) Outdoor	SC	0.15	34	31	1	7	Forage Hay	3.1, 3.4 (3.2) 13, 15 (14)	
	EC	0.15	337	31	1	7	Forage Hay	3.0, 3.4 (3.2) 12, 12 (12)	
USA, 2013 Cleveland, ND (Horsepower) Outdoor	SC	0.14	140	30-31	1	8	Forage Hay	3.7, 4.8 (4.2) 4.9, 5.7 (5.3)	
	EC	0.15	140	30-31	1	8	Forage Hay	2.7, 2.8 (2.8) 3.5, 3.6 (3.5)	
USA, 2013 Carrington, ND (Rockford) Outdoor	SC	0.15	140	31	1	8	Forage Hay	1.0, 1.2 (1.1) 3.1, 3.3 (3.2)	
	EC	0.15	140	31	1	8	Forage Hay	1.5, 1.6 (1.5) 3.7, 3.9 (3.8)	
USA, 2013 Prosser, NE (Horsepower Oats) Outdoor	SC	0.15	47	29-30	1	7	Forage Hay	1.6, 3.0 (2.3) 2.6, 3.9 (3.3)	
	EC	0.15	47	29-30	1	7	Forage Hay	2.0, 2.6 (2.3) 2.7, 5.2 (3.9)	
USA, 2014 San Angelo, TX (TAM 606)	SC	0.15	140	31	1	7	Forage Hay	5.0, 5.9 (5.4) c 0.010 16, 19 (18)	

Oats forage & hay country, year (variety)	Application Form	kg ai/ha	L/ha	BBCH	no.	DALA Days	Commodity	Residues, mg/kg[a]	Ref
Outdoor	EC	0.14	140	31	1	7	Forage Hay	6.4, 7.5 (7.0) c 0.010 24, 26 (25)	
Canada, 2013 Branchton, ON (Bradley) Outdoor	SC	0.14	200	30-31	1	7	Forage Hay	1.5, 1.5 (1.5) 6.3, 6.9 (6.6)	TK0163519 Mean recovery for pydiflumetofen: 99% (n=5) at 0.01 mg/kg 110% (n=4) at 0.1 mg/kg 100% (n=3) at 16 mg/kg 96% (n=3) at 51 mg/kg for forage, 110% (n=6) at 0.01 mg/kg 100% (n=6) at 0.1 mg/kg 88% (n=3) at 81 mg/kg for hay Sampling to analysis: 205-579 days for forage, 201-566 days for hay
	EC	0.14	200	30-31	1	7	Forage Hay	1.4, 1.4 (1.4) 5.3, 6.8 (6.1)	
Canada, 2013 St-Marc-sur-Richelieu, QC (RC Amaze) Outdoor	SC	0.16	200	21-31	1	8	Forage Hay	0.54, 0.54 (0.54) 2.4, 2.4 (2.4)	
	EC	0.15	200	21-31	1	8	Forage Hay	0.68, 0.77 (0.73) 3.0, 3.1 (3.1)	
Canada, 2014 Pike Lake, SK (Leggett) Outdoor	SC	0.14	200	22-31	1	7	Forage Hay	2.0, 2.0 (2.0) 9.1, 11 (10)	
	EC	0.15	200	22-31	1	7	Forage Hay	1.8, 1.9 (1.9) 7.9, 9.3 (8.6)	
Canada, 2013 Hanley, SK (Souris) Outdoor	SC	0.16	200	30-31	1	7	Forage Hay	3.2, 3.9 (3.6) 14, 15 (15)	
	EC	0.16	200	30-31	1	7	Forage Hay	2.6, 3.5 (3.1) 13, 14 (14)	
Canada, 2013 Elgin, MB (Summit) Outdoor	SC	0.16	45	30-31	1	7	Forage Hay	2.7, 3.7 (3.3) 19, 27 (23)	
	EC	0.16	45	30-31	1	7	Forage Hay	0.70, 0.88 (0.79) 5.5, 8.4 (7.0)	
Canada, 2013 Rosthern, SK (Leggett) Outdoor	SC	0.15	45	21-30	1	6	Forage Hay	5.1, 5.5 (5.3) 13, 18 (16)	
	EC	0.15	45	21-30	1	6	Forage Hay	3.0, 3.5 (3.3) 9.2, 11 (10)	
Canada, 2013 Waldheim, SK (Leggett) Outdoor	SC	0.15	200	22-30	1	6	Forage Hay	4.5, 4.9 (4.7) 19, 22 (21)	
	EC	0.15	200	22-30	1	6	Forage Hay	4.3, 8.7 (6.5) 22, 23 (23)	
Canada, 2014 Minto, MB (Pinnacle) Outdoor	SC	0.15	200	30-31	1	7	Forage Hay	0.46, 0.48 (0.47) 1.8, 2.1 (2.0)	
	EC	0.15	200	30-31	1	0 4 7 10 15	Forage	11 0.83 0.32, 0.36 (0.34) 0.15 0.026	
						0 4 7 10 15	Hay	61 7.3 1.5, 1.9 (1.7) 1.2 0.16	
Canada, 2013 Carberry, MB (AC Morgan) Outdoor	SC	0.15	200	31	1	9	Forage Hay	1.6, 1.9 (1.8) 10, 11 (11)	
	EC	0.15	200	31	1	9	Forage Hay	1.3, 1.5 (1.4) 6.8, 8.2 (7.5)	
Canada, 2013 Alvena, SK (Leggett) Outdoor	SC	0.16	50	31-32	1	7	Forage Hay	5.6, 7.5 (6.6) 19, 23 (21) c 0.015	
	EC	0.17	50	31-32	1	7	Forage Hay	3.2, 4.0 (3.6) 20, 22 (21) c 0.015	
Canada, 2013 Josephburg, AB	SC	0.15	200	31-32	1	7	Forage Hay	1.6, 1.6 (1.6) 10, 11 (11)	

Oats forage & hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[a]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
(Morgan) Outdoor	EC	0.15	200	31-32	1	7	Forage Hay	1.4, 1.4 (1.4) 5.3, 5.8 (5.6)	
Canada, 2013 Minto, MB (Summit) Outdoor	SC	0.15	200	30-31	1	7	Forage Hay	0.37, 0.42 (0.40) 1.9, 2.1 (2.0)	
	EC	0.15	200	30-31	1	0 2 7 10 14	Forage	12 2.8 0.56, 0.68 (0.62) 0.34 0.16	
						0 2 7 10 14	Hay	70 17 c 0.020 2.9, 3.1 (3.0) 1.8 0.79	

Forage: received basis, Hay: dry weight basis

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

Oats straw

The Meeting received 28 trials (24 at harvest trials, four decline trials) on oats which were conducted in Canada (Sagan, 2015: TK0163519) and the USA (McDonald, 2015: TK0163581). In each of these trials, pydiflumetofen was applied to oats as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. One application at 0.15 kg ai/ha was followed by a second application at 0.20 kg ai/ha. The first application was made 7–15 days prior to the second application, and the second application was made at BBCH 71. All applications were made in tank-mix with an adjuvant, NIS or COC. At each trial, straw samples were taken at normal grain harvest maturity which occurred at 16–61 days after the second application. In the decline trials additional straw samples were collected at 10 and 5 ± 1 day before normal harvest, and 5 and 10 ± 1 days after normal harvest.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from oat straw was 100% at 0.01–31 mg/kg fortified (Canada) and 97% at 0.01–15 mg/kg fortified (USA). Oat straw samples were stored at -10 °C or below (Canada) or at ca -18 °C (USA) for a maximum of 23 months between sampling and analysis.

Table 81 Residues of pydiflumetofen on oat straw from supervised trials in Canada and the USA

Oats straw country, year (variety)	Application					DALA Days	Residues, mg/kg [a b]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
GAP, USA	SC	0.20 0.35 / year	93[c] 45[d]	(14 days)	2	Do not apply after BBCH 71		
USA, 2013 North Rose, NY (Rodeo) Outdoor	SC	0.15 0.20	253 243	59-61 71-73 (14 days)	2	30	0.30, 0.32 (0.31)	TK0163581
	EC	0.15 0.20	253 243	59-61 71-73 (14 days)	2	30	0.49, 0.84 (0.67)	Mean recovery for pydiflumetofen: 85% (n=5) at 0.01 mg/kg 98% (n=8) at 0.1 mg/kg 104% (n=6) at 1.0-15 mg/kg
USA, 2014 Athens, GA (SS 76-50) Outdoor	SC	0.15 0.20	206 224	61-63 71 (13 days)	2	43	5.5, 7.3 (6.4)	
	EC	0.15 0.20	206 215	61-63 71	2	43	6.6, 7.3 (6.9)	

Oats straw country, year (variety)	Application					DALA Days	Residues, mg/kg [a b]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
				(13 days)				
USA, 2013 Richland, IA (NC Jerry Oats) Outdoor	SC	0.15 0.20	196 178	60 71 (14 days)	2	18	2.9, 3.8 (3.4)	Sampling to analysis: 103-706 days
	EC	0.15 0.20	196 178	60 71 (14 days)	2	18	4.8, 5.3 (5.0)	
USA, 2013 Gardner, ND (Horsepower) Outdoor	SC	0.15 0.21	140 196	67-68 81-83 (12 days)	2	28	4.0, 4.3 (4.1) c 0.026	
	EC	0.14 0.21	140 187	68-69 80-81 (12 days)	2	28	5.2, 7.0 (6.1) c 0.026	
USA, 2013 Bagley, IA (Reeves) Outdoor	SC	0.15 0.20	19 19	59-61 71 (12 days)	2	22	3.7, 4.8 (4.2)	
	EC	0.15 0.18	19 19	59-61 71 (12 days)	2	22	1.1, 2.4 (1.8)	
USA, 2013 Campbell, MN (Souris Oats) Outdoor	SC	0.15 0.20	187 187	39 71 (14 days)	2	24 29 34 39 44	0.95 0.77 0.93, 1.0 (0.96) 0.78 1.0	
	EC	0.15 0.20	187 187	39 71 (14 days)	2	34	1.3, 1.9 (1.6)	
USA, 2013 Atlantic, IA (Souris) Outdoor	SC	0.16 0.20	234 271	55-56 71-73 (14 days)	2	7 11 16 21 25	1.3 2.0 0.76, 0.79 (0.78) 0.74 0.79	
	EC	0.15 0.20	224 280	55-56 71-73 (14 days)	2	16	0.58, 0.74 (0.66)	
USA, 2013 Northwood, ND (Jerry) Outdoor	SC	0.15 0.20	140 140	45-47 71 (15 days)	2	46	0.25, 0.49 (0.37) c 0.015	
	EC	0.14 0.20	140 140	45-47 71 (15 days)	2	46	0.10, 0.12 (0.11) c 0.015	
USA, 2013 Kirksville, MO (NC Jerry Oats) Outdoor	SC	0.15 0.20	168 178	60 71 (14 days)	2	23	1.7, 1.7 (1.7)	
	EC	0.15 0.20	168 178	60 71 (14 days)	2	23	1.6, 1.7 (1.6)	
USA, 2013 St. Cloud, MN (Morton) Outdoor	SC	0.15 0.20	47 47	39-41 71-72 (14 days)	2	33	0.45, 1.1 (0.76)	
	EC	0.14 0.20	47 47	39-41 71-72 (14 days)	2	33	6.7, 8.3 (7.5)	
USA, 2014 Raymondville, TX (VNS) Outdoor	SC	0.15 0.21	327 337	65-69 71 (14 days)	2	19	11, 11 (11)	
	EC	0.15 0.21	337 337	65-69 71 (14 days)	2	19	12, 14 (13)	
USA, 2013 Cleveland, ND	SC	0.14 0.19	187 140	70-71 81-82	2	16	3.3, 4.2 (3.7)	

Oats straw country, year (variety)	Application					DALA Days	Residues, mg/kg [a b]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
(Horsepower) Outdoor				(13 days)				
	EC	0.15 0.21	187 140	70-71 81-82 (13 days)	2	16	5.0, 6.8 (5.9)	
USA, 2013 Carrington, ND (Rockford) Outdoor	SC	0.16 0.20	150 140	44-45 71 (14 days)	2	52	1.1, 1.3 (1.2)	
	EC	0.15 0.20	140 140	44-45 71 (14 days)	2	52	1.4, 1.5 (1.5)	
USA, 2013 Prosser, NE (Horsepower Oats) Outdoor	SC	0.15 0.21	47 47	61-65 71 (14 days)	2	29	1.1, 1.5 (1.3)	
	EC	0.15 0.20	47 47	61-65 71 (14 days)	2	29	0.85, 1.3 (1.1)	
USA, 2014 San Angelo, TX (TAM 606) Outdoor	SC	0.15 0.20	131 140	39-49 71 (13 days)	2	27	3.0, 3.1 (3.1)	
	EC	0.15 0.20	131 131	39-49 71 (13 days)	2	27	2.2, 2.8 (2.5)	
USA, 2014 Brooklyn, WI (Badger) Outdoor	SC	0.15 0.20	234 243	59-61 71-73 (14 days)	2	28	1.8, 2.2 (2.0)	
	EC	0.15 0.20	234 243	59-61 71-73 (14 days)	2	28	0.93, 0.96 (0.95)	
Canada, 2013 Branchton, ON (Bradley) Outdoor	SC	0.16 0.20	200 200	41-45 71 (15 days)	2	40	1.4, 3.7 (2.6)	TK0163519
	EC	0.15 0.19	200 200	41-45 71 (15 days)	2	40	0.92, 1.0 (0.96)	Mean recovery for pydiflumetofen: 100% (n=4) at 0.01 mg/kg 100% (n=4) at 0.1 mg/kg 94% (n=3) at 31 mg/kg
Canada, 2013 St-Marc-sur-Richelieu, QC (RC Amaze) Outdoor	SC	0.16 0.21	200 200	56-57 71-73 (10 days)	2	28	4.5, 5.4 (5.0)	
	EC	0.15 0.20	200 200	56-57 71-73 (10 days)	2	28	2.1, 4.5 (3.3)	
Canada, 2014 Pike Lake, SK (Leggett) Outdoor	SC	0.15 0.19	200 200	51-56 69-71 (14 days)	2	51	0.98, 1.1 (1.0)	Sampling to analysis: 117-500 days
	EC	0.15 0.19	200 200	51-56 69-71 (14 days)	2	51	1.2, 1.5 (1.4)	
Canada, 2013 Hanley, SK (Souris) Outdoor	SC	0.15 0.21	200 200	51-52 71-73 (15 days)	2	37	3.7, 5.1 (4.4)	
	EC	0.15 0.21	200 200	51-52 71-73 (15 days)	2	37	3.0, 4.5 (3.8)	
Canada, 2013 Elgin, MB (Summit) Outdoor	SC	0.15 0.20	45 45	55-59 71-73 (7 days)	2	54	0.98, 1.1 (1.0)	
	EC	0.15 0.20	45 45	55-59 71-73 (7 days)	2	54	0.50, 0.79 (0.65)	
Canada, 2013 Rosthern, SK (Leggett)	SC	0.14 0.20	45 45	59-61 71-73 (14 days)	2	39	11, 23 (17)	

Oats straw country, year (variety)	Application					DALA Days	Residues, mg/kg [a b]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
Outdoor	EC	0.15 0.20	45 45	59-61 71-73 (14 days)	2	39	6.6, 7.0 (6.8)	
Canada, 2013 Waldheim, SK (Leggett) Outdoor	SC	0.16 0.20	200 200	53-56 71-73 (14 days)	2	52	5.5, 7.2 (6.4)	
	EC	0.16 0.20	200 200	53-56 71-73 (14 days)	2	52	6.5, 7.5 (7.0)	
Canada, 2014 Minto, MB (Pinnacle) Outdoor	SC	0.15 0.20	200 200	51-55 69-71 (14 days)	2	46	1.4, 2.0 (1.7)	
	EC	0.15 0.20	200 200	51-55 69-71 (14 days)	2	36 42 46 51 56	0.65 0.65 0.54, 0.88 (0.71) 1.0 0.59	
Canada, 2013 Carberry, MB (AC Morgan) Outdoor	SC	0.15 0.20	200 200	52-53 71-73 (14 days)	2	36	3.0, 3.4 (3.2)	
	EC	0.15 0.20	200 200	52-53 71-73 (14 days)	2	36	2.0, 2.5 (2.3)	
Canada, 2013 Alvena, SK (Leggett) Outdoor	SC	0.15 0.21	45 50	49-52 71-72 (13 days)	2	56	8.6, 9.1 (8.9)	
	EC	0.15 0.21	45 50	49-52 71-72 (13 days)	2	56	5.5, 6.9 (6.2)	
Canada, 2013 Josephburg, AB (Morgan) Outdoor	SC	0.15 0.21	200 200	47-51 70-71 (15 days)	2	61	3.9, 4.7 (4.3)	
	EC	0.16 0.21	200 200	47-51 70-71 (15 days)	2	61	1.7, 3.5 (2.6)	
Canada, 2013 Minto, MB (Summit) Outdoor	SC	0.15 0.20	200 200	45-52 71-73 (11 days)	2	42	0.63, 0.67 (0.65)	
	EC	0.15 0.20	200 200	45-52 71-73 (11 days)	2	33 38 42 47 53	0.42 0.72 0.42, 0.50 (0.46) 0.33 0.49	

Portion analysed: straw, received basis

[a] Re-treatment interval is given in parenthesis.

[b] Mean of replicate field samples is given in parenthesis.

[c] ground application,

[d] aerial application

Wheat forage and hay

The Meeting received 33 trials (29 at harvest trials, four decline trials) on wheat which were conducted in Canada (Sagan, 2015: TK0103847) and the USA (Oakes, 2015: TK0163574). In each of these trials, pydiflumetofen was applied to oats as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. A single application at 0.15 kg ai/ha was made at BBCH 31. All applications were made in tank-mix with an adjuvant, NIS, COC or MSO. At each trial, forage and hay samples were taken 7 ± 1 DALA. In the decline trials additional samples were collected at 0, 3, 10 and 14 ± 1 DALA. Hay samples were allowed to dry for 1–22 days before collection.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from wheat forage was 107% at 0.01–10 mg/kg fortified (Canada) and 100% at 0.01–100 mg/kg fortified (USA), and from wheat hay was 97% at 0.01–81 mg/kg fortified (Canada) and 105% at 0.01–75 mg/kg fortified (USA). Wheat forage and hay samples were stored at -10 °C or below (Canada) or at ca -18 °C (USA) for a maximum of 19 months between sampling and analysis.

Table 82 Residues of pydiflumetofen on wheat forage and hay from supervised trials in Canada and the USA

Wheat forage/ hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
GAP, USA	SC	0.20	93[c] a45[d]		1	7			
USA, 2014 Seven Springs, NC (Pioneer 26R20) Outdoor	SC	0.15	140	30-31	1	7	Forage Hay	1.1, 1.2 (1.2) 2.8, 2.8 (2.8)	TK0163574 Mean recovery for pydiflumetofen: 95% (n=7) at 0.01 mg/kg 111% (n=6) at 0.1 mg/kg 95% (n=5) at 1.0-100 mg/kg for forage, 91% (n=5) at 0.01 mg/kg 113% (n=4) at 0.1 mg/kg 109% (n=9) at 1.0-75 mg/kg for hay Sampling to analysis: 148-196 days for forage, 148-589 days for hay
	EC	0.15	140	30-31	1	7	Forage Hay	2.3, 2.7 (2.5) 5.8, 6.7 (6.2)	
USA, 2014 Proctor, AR (Progeny 357) Outdoor	SC	0.15	112	28-29	1	7	Forage Hay	1.4, 1.6 (1.5) 3.4, 4.5 (3.9) c 0.027	
	EC	0.15	112	28	1	7	Forage Hay	2.2, 2.4 (2.3) 6.5, 7.1 (6.8) c 0.027	
USA, 2014 St. Cloud, MN (Arapahoe) Outdoor	SC	0.15	187	31-31	1	7	Forage Hay	0.67, 1.2 (0.95) 0.89, 1.1 (0.98)	
	EC	0.15	187	31-31	1	7	Forage Hay	1.5, 1.7 (1.6) 1.3, 2.4 (1.8)	
USA, 2013 Richland, IA (748) Outdoor	SC	148	159	31	1	0 3 7 10 14	Forage	5.0 1.7 0.92, 1.4 (1.2) 0.82 0.77	
						0 3 7 10 14	Hay	9.1 10 4.1, 4.9 (4.5) 4.2 2.6	
	EC	0.15	159	31	1	7	Forage Hay	1.1, 1.3 (1.2) 4.1, 4.8 (4.4)	
USA, 2014 Stafford, KS (Armour) Outdoor	SC	0.14	206	23-24	1	6	Forage Hay	7.6, 7.9 (7.7) 37, 42 (40)	
	EC	0.15	224	23-24	1	6	Forage Hay	7.1, 8.4 (7.7) 32, 37 (35)	
USA, 2014 Bagley, IA (BW 442) Outdoor	SC	0.14	224	30-31	1	7	Forage Hay	1.0, 1.3 (1.2) 2.5, 3.6 (3.0) c 0.012	
	EC	0.15	224	30-31	1	7	Forage Hay	1.9, 2.5 (2.2) 6.8, 7.6 (7.2) c 0.012	
USA, 2014 Bloomfield, MO (McAlister) Outdoor	SC	0.15	187	29-31	1	7	Forage Hay	1.2, 1.4 (1.3) 6.6, 6.6 (6.6) c 0.012	
	EC	0.14	187	29-31	1	7	Forage Hay	1.9, 1.9 (1.9) 8.9, 10 (9.5) c 0.012	
USA, 2014 Uvalde, TX (Greer) Outdoor	SC	0.15	168	29-29	1	7	Forage Hay	2.3, 2.5 (2.4) 5.8, 5.9 (5.8)	
	EC	0.15	168	29-29	1	7	Forage Hay	2.5, 2.9 (2.7) 6.1, 6.8 (6.5)	
USA, 2013 Cleveland, ND[a)]	SC	0.14	131	30-35	1	7	Forage Hay	1.6, 1.6 (1.6) 7.1, 7.9 (7.5)	

Wheat forage/ hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
(Divide) Outdoor								c 0.020	
	EC	0.15	140	30-35	1	7	Forage Hay	1.6, 1.6 (1.6) 5.7, 6.8 (6.2) c 0.020	
USA, 2013 Carrington, ND (Faller) Outdoor	SC	0.15	140	25-25	1	8	Forage Hay	0.92, 1.1 (1.0) 7.7, 8.0 (7.8)	
	EC	0.15	140	25-25	1	8	Forage Hay	1.8, 1.9 (1.9) 7.8, 8.3 (8.0)	
USA, 2013 Grand Island, NE (Taverse) Outdoor	SC	0.15	187	30-31	1	8	Forage Hay	0.23, 0.25 (0.24) 1.7, 1.8 (1.8)	
	EC	0.15	187	30-31	1	8	Forage Hay	0.12, 0.16 (0.14) 0.35, 0.84 (0.59)	
USA, 2013 Prosser, NE (Barlow) Outdoor	SC	0.15	47	29-30	1	7	Forage Hay	1.6, 1.7 (1.7) 9.9, 9.9 (9.9)	
	EC	0.15	47	29-30	1	7	Forage Hay	1.1, 1.7 (1.4) 9.0, 9.7 (9.4)	
USA, 2013 Cleveland, ND[b] (Glenn) Outdoor	SC	0.14	140	30-35	1	8	Forage Hay	1.7, 2.4 (2.1) 9.2, 15 (12) c 0.037	
	EC	0.15	140	30-35	1	8	Forage Hay	1.7, 2.6 (2.2) 6.6, 6.7 (6.6) c 0.037	
USA, 2014 Jetmore, KS (TAM 112) Outdoor	SC	0.15	47	27-30	1	8	Forage Hay	10, 11 (11) 19, 20 (20) c 0.020	
	EC	0.14	47	27-30	1	8	Forage Hay	6.3, 8.1 (7.2) 7.7, 9.3 (8.5) c 0.020	
USA, 2014 San Angelo, TX (WB-Grainfield) Outdoor	SC	0.15	140	31-31	1	0 2 8 10 14	Forage	21 15 8.8, 12 (10) 6.9 4.3	
						0 2 8 10 14	Hay	62 51 31, 34 (33) 24 11	
	EC	0.15	140	31-31	1	8	Forage Hay	11, 11 (11) 19, 36 (27)	
USA, 2014 Levelland, TX (Weathermaster) Outdoor	SC	0.15	140	25-31	1	7	Forage Hay	5.0, 5.1 (5.0) 15, 17 (16)	
	EC	0.15	140	25-31	1	7	Forage Hay	4.9, 5.8 (5.4) 19, 20 (19)	
USA, 2014 Larned, KS (T-154) Outdoor	SC	0.15	215	23-24	1	7	Forage Hay	2.0, 4.1 (3.1) 16, 18 (17)	
	EC	0.15	215	23-24	1	7	Forage Hay	5.4, 5.4 (5.4) 20, 21 (20)	
USA, 2014 Dill City, OK (Duster) Outdoor	SC	0.15	178	29-31	1	7	Forage Hay	5.7, 6.6 (6.2) 16, 17 (16)	
	EC	0.15	178	29-31	1	7	Forage Hay	4.8, 5.8 (5.3) 15, 16 (15)	
USA, 2014 Hinton, OK (Duster) Outdoor	SC	0.15	112	28-30	1	8	Forage Hay	4.6, 8.0 (6.3) 15, 19 (17)	
	EC	0.15	112	28-30	1	7	Forage Hay	6.2, 6.4 (6.3) 12, 12 (12)	

Wheat forage/ hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
USA, 2014 Jerome, ID (Legacy) Outdoor	SC	0.15	187	30-32	1	7	Forage Hay	3.6, 3.8 (3.7) 11, 12 (12)	
	EC	0.15	187	30-32	1	7	Forage Hay	4.0, 4.3 (4.2) 11, 11 (11)	
Canada, 2013 Pike Lake, SK (Shaw) Outdoor	SC	0.12	45	30-31	1	7	Forage Hay	1.6, 1.6 (1.6) 9.1, 11 (10)	TK0103847 Mean recovery for pydiflumetofen: 105% (n=4) at 0.01 mg/kg 111% (n=4) at 0.1 mg/kg 105% (n=3) at 10 mg/kg for forage, 103% (n=4) at 0.01 mg/kg 98% (n=4) at 0.1 mg/kg 89% (n=3) at 81 mg/kg for hay Sampling to analysis: 196-573 days for forage, 200-577 days for hay
	EC	0.15	45	30-31	1	1 3 7 10 13 1 3 7 13	Forage Hay	2.2 2.4 0.76, 0.89 (0.83) 0.53 1.1 15 6.0 3.2, 3.3 (3.3) 4.3	
Canada, 2014 Vanscoy, SK (CDC Utmost) Outdoor	SC	0.14	200	21-31	1	7	Forage Hay	2.9, 3.8 (3.4) 12, 13 (13)	
	EC	0.14	200	21-31	1	7	Forage Hay	2.4, 2.5 (2.5) 11, 12 (12)	
Canada, 2013 Taber, AB (Bentley) Outdoor	SC	0.15	200	29-31	1	8	Forage Hay	0.85, 1.1 (0.98) 3.9, 4.1 (4.0)	
	EC	0.16	200	29-31	1	8	Forage Hay	0.77, 0.80 (0.79) 3.2, 3.4 (3.3)	
Canada, 2013 Boissevain, MB (Harvest) Outdoor	SC	0.15	45	31	1	7	Forage Hay	0.94, 1.0 (0.97) 5.4, 6.0 (5.7)	
	EC	0.16	45	31	1	7	Forage Hay	0.78, 0.85 (0.82) 3.8, 3.8 (3.8)	
Canada, 2013 Elgin A, MB[c)] (Vesper) Outdoor	SC	0.16	45	30-31	1	7	Forage Hay	3.0, 4.0 (3.5) 17, 19 (18)	
	EC	0.16	45	30-31	1	7	Forage Hay	3.3, 3.9 (3.6) 16, 19 (18)	
Canada, 2013 Elgin B, MB[d)] (Unity) Outdoor	SC	0.15	200	30-31	1	7	Forage Hay	4.1, 5.6 (4.9) 28, 29 (29)	
	EC	0.15	200	30-31	1	7	Forage Hay	3.3, 3.5 (3.4) 18, 19 (19)	
Canada, 2013 Rosthern, SK (Shaw) Outdoor	SC	0.14	200	29-31	1	6	Forage Hay	3.5, 3.6 (3.6) 12, 14 (13)	
	EC	0.14	200	29-31	1	6	Forage Hay	2.7, 2.9 (2.8) 13, 16 (15)	
Canada, 2013 Coalhurst, AB (AC Carberry) Outdoor	SC	0.15	200	29-32	1	8	Forage Hay	2.9, 3.1 (3.0) 16, 17 (17)	
	EC	0.15	200	30-32	1	7	Forage Hay	3.2, 3.4 (3.3) 15, 18 (17)	
Canada, 2013 Carberry, MB (Harvest) Outdoor	SC	0.15	200	31	1	6	Forage Hay	4.3, 4.5 (4.4) 20, 20 (20)	
	EC	0.15	200	31	1	6	Forage Hay	3.7, 3.8 (3.8) 11, 11 (11)	
Canada, 2013 Alvena, SK (Unity) Outdoor	SC	0.14	200	31-32	1	6	Forage Hay	4.0, 5.6 (4.8) 22, 26 (24) c 0.010	
	EC	0.14	200	31-32	1	6	Forage Hay	3.4, 3.7 (3.6) 22, 26 (24) c 0.010	
Canada, 2013 Lamont, AB (Harvest)	SC	0.16	200	31-32	1	7	Forage Hay	3.5, 4.4 (4.0) 18, 22 (20)	
	EC	0.16	200	31-32	1	7	Forage	3.5, 3.6 (3.6)	

Wheat forage/ hay country, year (variety)	Application					DALA Days	Commodity	Residues, mg/kg[ab]	Ref
	Form	kg ai/ha	L/ha	BBCH	no.				
Outdoor							Hay	14, 16 (15)	
Canada, 2014 Josephburg, AB (Harvest) Outdoor	SC	0.15	200	30-32	1	7	Forage Hay	1.2, 1.2 (1.2) 4.5, 6.5 (5.5)	
	EC	0.15	200	30-32	1	7	Forage Hay	1.3, 1.4 (1.4) 4.8, 5.0 (4.9)	
Canada, 2013 Minto, MB (Unity) Outdoor	SC	0.15	200	30-31	1	7	Forage Hay	0.50, 0.54 (0.52) 1.8, 2.0 (1.9)	
	EC	0.15	200	30-31	1	0 2 7 10 14	Forage	8.9 2.8 0.43, 0.48 (0.46) 0.32 0.19	
						0 2 7 10 14	Hay	68 18 2.4, 2.5 (2.5) 1.1 0.68	

Forage: received basis, Hay: dry weight basis

[a] Mean of replicate field samples is given in parenthesis.

[b] c: sample from control plot

[c] ground application,

[d] aerial application

[e] and [f]Address: Jamestown Municipal Airport ~27 miles, Application dates (1st): 02 Jul 2013

[g] and [h] Address: 210 South Railway St, Minto, MB R0K 1M0, Application dates (1st): 26 Jun 2013

Wheat straw

The Meeting received 33 trials (29 at harvest trials, four decline trials) on wheat which were conducted in Canada (Sagan, 2015: TK0103847) and the USA (Oakes, 2015: TK0163574). In each of these trials, pydiflumetofen was applied to wheat as a foliar treatment as SC formulations (200 g ai/L) and EC formulations (100 g ai/L) side-by-side. One application at 0.15 kg ai/ha was followed by a second application at 0.20 kg ai/ha. The first application was made 12–21 days prior to the second application, and the second application was made at BBCH 71. All applications were made in tank-mix with an adjuvant, NIS, COC or MSO. At each trial, straw samples were taken at normal grain harvest maturity which occurred at 16–74 days after the second application. In the decline trials additional straw samples were collected at 10 and 5 ± 1 day before normal harvest, and 5 and 10 ± 1 days after normal harvest.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from wheat straw was 100% at 0.01–41 mg/kg fortified (Canada) and 92% at 0.01–50 mg/kg fortified (USA). Wheat straw samples were stored at -10 °C or below (Canada) or at -18 °C or below (USA) for a maximum of 19 months between sampling and analysis.

Table 83 Residues of pydiflumetofen on wheat straw from supervised trials in Canada and the USA

Wheat straw country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
GAP, USA	SC	0.20 0.35 / year	93[d] 45[e]	(14 days)	2	Do not apply after BBCH 71		
USA, 2014 Seven Springs, NC (Pioneer 26R20) Outdoor	SC	0.15 0.20	243 234	66-67 71-73 (14 days)	2	20	18, 18 (18)	TK0163574
	EC	0.15 0.20	234 234	66-67 71-73 (14 days)	2	20	30, 30 (30)	Mean recovery for pydiflumetofen: 88% (n=1) at

Wheat straw country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
USA, 2014 Proctor, AR (Progeny 357) Outdoor	SC	0.15 0.20	112 150	68-69 71-71 (14 days)	2	23	2.2, 2.3 (2.3)	0.01 mg/kg 96% (n=4) at 0.1 mg/kg 91% (n=7) at 1.0-50 mg/kg
	EC	0.15 0.20	112 150	68-69 71-71 (14 days)	2	23	2.6, 2.9 (2.8)	
USA, 2014 St. Cloud, MN (Arapahoe) Outdoor	SC	0.15 0.20	187 187	48-51 69-71 (14 days)	2	45	2.0, 2.1 (2.1)	Sampling to analysis: 94-579 days
	EC	0.15 0.20	187 187	49-51 69-71 (14 days)	2	45	2.7, 3.7 (3.2)	
USA, 2013 Richland, IA (748) Outdoor	SC	0.15 0.20	215 168	40 71 (14 days)	2	44 48 53 58 62	1.7 1.1 1.3, 1.3 (1.3) 0.54 1.3	
	EC	0.15 0.20	215 168	40 71 (14 days)	2	53	2.1, 2.7 (2.4)	
USA, 2014 Stafford, KS (Armour) Outdoor	SC	0.14 0.20	206 206	50-53 71-75 (14 days)	2	29	3.2, 4.6 (3.9)	
	EC	0.14 0.20	206 206	50-53 71-75 (14 days)	2	29	6.6, 7.1 (6.8)	
USA, 2014 Bagley, IA (BW 442) Outdoor	SC	0.16 0.21	243 196	69-69 71-71 (15 days)	2	33	3.9, 4.7 (4.3)	
	EC	0.16 0.21	243 196	69-69 71-71 (15 days)	2	33	1.8, 2.5 (2.1)	
USA, 2014 Bloomfield, MO (McAlister) Outdoor	SC	0.15 0.20	187 187	43-45 71-71 (13 days)	2	32	4.0, 4.6 (4.3)	
	EC	0.14 0.20	187 187	43-45 71-71 (13 days)	2	32	3.3, 4.3 (3.8)	
USA, 2014 Uvalde, TX (Greer) Outdoor	SC	0.15 0.20	224 234	49-51 71-71 (16 days)	2	33	3.1, 3.6 (3.4)	
	EC	0.15 0.20	224 234	49-51 71-71 (16 days)	2	33	2.2, 2.4 (2.3)	
USA, 2013 Cleveland, ND [f] (Divide) Outdoor	SC	0.15 0.20	187 140	70-71 81-82 (13 days)	2	16	12, 12 (12)	
	EC	0.15 0.20	187 140	70-71 81-82 (13 days)	2	16	15, 16 (16)	
USA, 2013 Carrington, ND (Faller) Outdoor	SC	0.16 0.20	150 140	44-45 71-71 (14 days)	2	52	5.3, 5.7 (5.5) c 0.11	
	EC	0.15 0.20	140 140	44-45 71-71 (14 days)	2	52	3.8, 4.8 (4.3) c 0.11	
USA, 2013 Grand Island, NE (Taverse) Outdoor	SC	0.15 0.20	150 187	45-45 73-73 (12 days)	2	22	5.7, 5.7 (5.7)	
	EC	0.15 0.20	150 187	45-45 73-73 (12 days)	2	22	6.3, 6.3 (6.3)	

Wheat straw country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
USA, 2013 Prosser, NE (Barlow) Outdoor	SC	0.15 0.21	47 47	61-65 71-71 (14 days)	2	29	3.0, 3.6 (3.3)	
	EC	0.15 0.20	47 47	61-65 71-71 (14 days)	2	29	3.0, 3.4 (3.2)	
USA, 2013 Cleveland, ND [g] (Glenn) Outdoor	SC	0.15 0.21	187 150	70-71 81-82 (13 days)	2	16	9.9, 11 (10)	
	EC	0.15 0.20	187 140	70-71 81-82 (13 days)	2	16	12, 17 (15)	
USA, 2014 Jetmore, KS (TAM 112) Outdoor	SC	0.15 0.22	37 47	50-53 71-71 (14 days)	2	47	0.87, 1.3 (1.1)	
	EC	0.15 0.21	37 47	50-53 71-71 (14 days)	2	47	2.4, 3.0 (2.7)	
USA, 2014 San Angelo, TX (WB-Grainfield) Outdoor	SC	0.14 0.20	140 140	49-56 71-73 (16 days)	2	21 27 32 36 42	9.8 5.3 4.9, 5.4 (5.2) 5.5 5.4	
	EC	0.15 0.20	140 140	49-56 71-73 (16 days)	2	32	7.4, 7.7 (7.5)	
USA, 2014 Levelland, TX (Weathermaster) Outdoor	SC	0.15 0.20	140 140	59-59 71-83 (14 days)	2	19	5.0, 6.0 (5.5)	
	EC	0.15 0.20	140 140	59-59 71-83 (14 days)	2	19	5.6, 6.4 (6.0)	
USA, 2014 Larned, KS (T-154) Outdoor	SC	0.15 0.20	215 206	50-53 71-71 (12 days)	2	40	1.3, 2.8 (2.1)	
	EC	0.15 0.20	206 206	50-53 71-71 (12 days)	2	40	3.6, 4.2 (3.9)	
USA, 2014 Dill City, OK (Duster) Outdoor	SC	0.15 0.21	122 131	59-60 71-73 (14 days)	2	28	6.5, 8.3 (7.4)	
	EC	0.15 0.20	112 131	59-60 71-73 (14 days)	2	28	8.8, 10 (9.5)	
USA, 2014 Hinton, OK (Duster) Outdoor	SC	0.14 0.20	122 122	61-69 81-83 (13 days)	2	33	5.7, 6.0 (5.9) c 0.028	
	EC	0.15 0.20	131 122	61-69 81-83 (13 days)	2	33	9.4, 10 (9.9) c 0.028	
USA, 2014 Jerome, ID (Legacy) Outdoor	SC	0.14 0.20	187 196	45-49 65-69 (15 days)	2	74	1.9, 2.0 (2.0)	
	EC	0.15 0.21	196 196	45-49 65-69 (15 days)	2	74	2.0, 2.2 (2.2)	
Canada, 2013 Pike Lake, SK (Shaw) Outdoor	SC	0.15 0.20	45 45	59-65 71-75 (14 days)	2	41	3.8, 5.3 (4.6)	TK0103847
	EC	0.15 0.20	45 45	59-65 71-75 (14 days)	2	29 36 41	2.6 3.2 1.8, 2.2 (2.0)	Mean recovery for pydiflumetofen: 110% (n=5) at

Wheat straw country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
						47 51	4.7 4.0	0.01 mg/kg 100% (n=4) at 0.1 mg/kg 98% (n=3) at 16 mg/kg 92% (n=3) at 41 mg/kg
Canada, 2014 Vanscoy, SK (CDC Utmost) Outdoor	SC	0.15 0.20	200 200	58-61 69-71 (14 days)	2	39	5.7, 6.0 (5.9)	
	EC	0.15 0.20	200 200	58-61 69-71 (14 days)	2	39	3.9, 4.0 (4.0)	
Canada, 2013 Taber, AB (Bentley) Outdoor	SC	0.15 0.20	200 200	61-65 69-73 (13 days)	2	38	2.8, 3.6 (3.2)	Sampling to analysis: 140-504 days
	EC	0.15 0.20	200 200	61-65 69-73 (13 days)	2	38	2.2, 2.6 (2.4)	
Canada, 2013 Boissevain, MB (Harvest) Outdoor	SC	0.15 0.21	45 45	51-59 71-73 (12 days)	2	48	2.5, 2.6 (2.6)	
	EC	0.15 0.20	45 45	51-59 71-73 (12 days)	2	48	1.6, 1.7 (1.7)	
Canada, 2013 Elgin A, MB [h] (Vesper) Outdoor	SC	0.16 0.20	45 45	55-59 71-73 (13 days)	2	44	3.2, 3.6 (3.4)	
	EC	0.16 0.20	45 45	55-59 71-73 (13 days)	2	44	2.4, 2.9 (2.7)	
Canada, 2013 Elgin B, MB [i] (Unity) Outdoor	SC	0.15 0.21	200 200	55-59 71-73 (13 days)	2	44	3.5, 3.7 (3.6)	
	EC	0.16 0.21	200 200	55-59 71-73 (13 days)	2	44	0.77, 0.77 (0.77)	
Canada, 2013 Rosthern, SK (Shaw) Outdoor	SC	0.14 0.21	200 200	59-61 69-71 (14 days)	2	41	15, 17 (16)	
	EC	0.14 0.21	200 200	59-61 69-71 (14 days)	2	41	9.0, 12 (11)	
Canada, 2013 Coalhurst, AB (AC Carberry) Outdoor	SC	0.15 0.20	200 200	45-61 71-73 (14 days)	2	47	2.2, 2.8 (2.5)	
	EC	0.15 0.20	200 200	45-61 71-73 (14 days)	2	47	0.85, 0.91 (0.88)	
Canada, 2013 Carberry, MB (Harvest) Outdoor	SC	0.15 0.20	200 200	55-56 71 (15 days)	2	40	4.2, 4.4 (4.3)	
	EC	0.15 0.20	200 200	55-56 71 (15 days)	2	40	2.6, 3.6 (3.1)	
Canada, 2013 Alvena, SK (Unity) Outdoor	EC	0.16 0.20	200 200	51-53 71-72 (13 days)	2	56	8.1, 8.2 (8.2) c 0.015	
Canada, 2013 Lamont, AB (Harvest) Outdoor	SC	0.16 0.21	200 200	54-59 69-71 (15 days)	2	46	4.7, 5.2 (5.0)	
	EC	0.15 0.21	200 200	54-59 69-71 (15 days)	2	46	3.6, 4.3 (4.0)	
Canada, 2014	SC	0.16	200	51-57	2	48	8.7, 10 (9.4)	

Wheat straw country, year (variety)	Application					DALA Days	Residues, mg/kg[bc]	Ref
	Form	kg ai/ha	L/ha	BBCH[a]	no.			
Josephburg, AB (Harvest) Outdoor		0.21	200	69-71 (21 days)				
	EC	0.15 0.21	200 200	51-57 69-71 (21 days)	2	48	3.9, 4.4 (4.2)	
Canada, 2013 Minto, MB (Unity) Outdoor	SC	0.15 0.20	200 200	51-58 71-73 (14 days)	2	45	1.3, 1.5 (1.4)	
	EC	0.15 0.20	200 200	51-58 71-73 (14 days)	2	35 40 45 49 54	1.1 0.80 1.3, 1.4 (1.4) 1.4 1.6	

Portion analysed: straw, received basis

[a] Re-treatment interval is given in parenthesis.

[b] Mean of replicate field samples is given in parenthesis.

[c] c: sample from control plot

[d] ground application,

[e] aerial application

[f] and [g] Address: Jamestown Municipal Airport ~27 miles, Application dates (1st): 15 Aug 2013

[h] and [i] Address: 210 South Railway St, Minto, MB R0K 1M0, Application dates (1st): 17 Jul 2013

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting received information on high temperature hydrolysis of pydiflumetofen and the fate of pydiflumetofen residues during the processing of grapes, tomato, soya bean, potato, wheat, barley, oats, maize, sweet corn, rape seed and peanut.

The crops that the Meeting received information on supervised field trials may be processed prior to consumption. Processing factors have been calculated for pydiflumetofen residues in grapes and rape seeds.

High temperature hydrolysis

The hydrolytic stability of [Pyrazole-5-^{14}C]-pydiflumetofen was investigated under conditions representative of food processing (Dixon, 2014: 35072). The conditions used are summarised below.

Temperature, °C	Time, min	pH	Process Represented
90	20	4	Pasteurisation
100	60	5	Baking, Brewing, Boiling
120	20	6	Sterilisation

Test solutions were prepared by adding an aliquot of the radiochemical stock solution to each buffer (citrate buffers were prepared at pH 4, 5 and 6) to give a final test concentration of 1.0 mg/L. Test solutions were dispensed into headspace vials (which were capable of withstanding the high temperature and pressures expected during incubation) and samples were incubated as per the conditions detailed above. The radioactivity content of incubates sampled prior to and following incubation was determined by LSC analysis. Radioactivity content in incubated samples was compared to control samples.

Table 84 Recovery of total radioactivity from hydrolysed samples

Sample	Radioactive Recovery (% of applied radioactivity)		
	pH4, 90 °C, 20 min	pH5, 100 °C, 60 min	pH6, 120 °C, 20 min
Buffer	105.5, 106.0	104.5, 104.5	100.0, 97.9
Buffer + acetonitrile rinse	107.4, 107.7	106.4, 106.3	103.9, 100.4

The total recovery of applied radioactivity ranged from 100.0–107.7% for all hydrolysis experiments. Characterisation and identification of the radiolabelled components in treated solutions in this study was carried out by co-chromatography against

reference standards using TLC. Quantification of the degradates was conducted using the TLC data. In addition the presence of parent [Pyrazole-5-^{14}C]-pydiflumetofen in incubated and non-incubated (control) samples, was confirmed using HPLC.

Table 85 Identification of radioactivity under the conditions for processing simulation

Identified components	Recovery of applied [Pyrazole-5-^{14}C]-pydiflumetofen [1.0 mg/L]					
	pH 4, 90 °C, 20min		pH 5, 100 °C, 60 min		pH 6, 120 °C, 20 min	
	%AR	mg/L	%AR	mg/L	%AR	mg/L
Pydiflumetofen	97.5, 96.2	0.998, 0.989	96.5, 96.0	0.982, 0.978	95.5, 95.7	0.940, 0.923
SYN545547	0.26, 0.63	0.003, 0.006	0.35, 0.62	0.004, 0.006	0.64, 0.78	0.006, 0.008
SYN547891	0.20, 0.51	0.002, 0.005	0.51, 0.37	0.005, 0.004	0.28, 0.08	0.003, 0.001
Origin	1.29, 1.62	0.013, 0.017	1.69, 1.71	0.017, 0.017	1.88, 1.66	0.018, 0.016
Unidentified	0.78, 1.09	0.008, 0.011	0.97, 1.33	0.010, 0.014	1.66, 1.74	0.016, 0.017
Total	100, 100	1.02, 1.03	100, 100	1.02, 1.02	100, 100	0.984, 0.964

%AR: Results are based on the assumption that the chromatographed radioactivity represents 100% of the applied radioactivity.

[Pyrazole-5-^{14}C]-pydiflumetofen was found to be hydrolytically stable in buffer solutions at pH 4, 5 and 6 at temperatures simulating pasteurisation (90 °C), baking/brewing/boiling (100 °C) and sterilisation (120 °C) respectively.

Grapes

Two processing trials on grapes were conducted in Northern France and Germany during 2013. Two applications of an SC formulation containing 200 g/L pydiflumetofen were applied at 200 g ai/ha separated by a 14–15 day interval. Treated grape samples were collected at 21 day (normal commercial harvest) after the final application (Tessier, 2015: S13-03421). Samples were shipped frozen to the analytical facility for residue analysis and chilled to the processing facility.

Fresh whole grapes were processed into grape juice and white wine. Two balance studies and one follow-up study were carried out on each trial, for each process. Relevant industrial practices and standardised procedures were applied to simulate the common processes used by industry for production of grape juice and white wine.

Grape juice processing

Bunches were stemmed and crushed manually. After addition of pectolytic enzymes, the stemmed and crushed grapes were heated to 50–55 °C, maintained at a temperature ranging between 45 °C and 60 °C for two hours and then pressed in a water press. Wet pomace and raw juice were obtained after pressing. The dry pomaces were obtained by placing wet pomace into ovens regulated at 60 °C. The clarification of the raw juice took place for 5 minutes at a temperature of about 85 °C. The juice was filtered over cellulose filter plates “trimming” quality. The filtered juice was then pasteurised for 1 minute at 85 °C.

White wine processing

Bunches were pressed directly. The must and wet pomace were obtained after pressing. Wet pomaces were placed into ovens regulated at 60 °C to obtained dry pomace. After addition of pectolytic enzymes and potassium metabisulphite, the must was decanted after at least 12 hours of settling. Dry active yeast was added to the must. Sugar was added to the must during alcoholic fermentation, in order to increase the probable alcohol content of the specimen to the content. The alcoholic fermentation was considered completed when the density of the wine was stabilised below the value 1000. Potassium metabisulphite were added to the wine. The natural clarification lasted at least four days. After racking, the AF (after alcoholic fermentation) wine was separated from lees. Dry gelatin and potassium metabisulphite were added to wine, to improve the clarification. The wine was kept in demijohn. The demijohns were stored in a cold room (<10 °C) to stabilise the wine with regard to tartaric deposits and so that clarification could be achieved. To remove impurities (solid material), the wine was filtered over cellulose filter plates of 2.5 µm and 1.5 µm porosity under pressure using nitrogen. After this operation, the wine received potassium metabisulphite, which protects the wine from oxidation. The wine was stored in a cold room (about 5–10 °C) for 6 months.

Samples of pre-processed whole fruit and processed commodities were analysed for residues of pydiflumetofen using analytical method GRM061.03A with an LOQ of 0.01 mg/kg for all commodities. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%.

Table 86 Residues of pydiflumetofen in grapes and grape processed commodities

country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg	Processing factor
Germany, 2013 Baden-Württemberg (Riesling)	0.19 0.20	2	21	Whole white grape	0.09, 0.19 Mean 0.14	-
				Stems	0.48	3.4
				Crushed grapes	0.31	2.2
				Raw juice	0.03	0.21
				Wet pomace	1.2	8.3
				Dry pomace	4.9	35
				Clarified juice	0.03	0.21
				Sediments	0.04	0.29
				Filter plate	0.19	1.4
				Filtered juice	0.02	0.14
				Grape juice	0.01	0.07
				Whole white grape	0.17, 0.24 Mean 0.21	-
				Grape juice	0.01	0.05
				Whole white grape	0.15, 0.25 Mean 0.20	-
				Must	0.32	1.6
				Wet pomace	0.39	2.0
				Dry pomace	2.6	13
				Must deposit	2.0	9.8
				AF wine	0.12	0.60
				Lees	0.59	3.0
				Deposit	0.13	0.65
				White young wine	0.11	0.55
				White aged wine	0.12	0.60
				Whole white grape	0.18, 0.24 Mean 0.21	-
				Must	0.13	0.62
				Wet pomace	0.32	1.6
				Dry pomace	1.7	7.9
				White aged wine	0.11	0.52
Northern France, 2013 Centre (Chardonnay)	0.20 0.21	2	21	Whole white grape	0.79, 1.0 Mean 0.91	-
				Stems	1.5	1.6
				Crushed grapes	0.53	0.58
				Raw juice	0.03	0.03
				Wet pomace	1.7	1.9
				Dry pomace	9.4	10
				Clarified juice	0.04	0.04
				Sediments	0.09	0.10
				Filter plate	0.23	0.25
				Filtered juice	0.02	0.02
				Grape juice	0.02	0.02
				Grape juice	0.02	0.02
				Whole white grape	0.79, 1.0 Mean 0.91	-
				Must	0.40	0.44
				Wet pomace	2.1	2.3
				Dry pomace	8.4	9.2
				Must deposit	3.0	3.3
				AF wine	0.12	0.13
				Lees	0.80	0.88
				Deposit	0.10	0.11
				White young wine	0.09	0.10
				White aged wine	0.10	0.11
				Must	0.12	0.13
				Wet pomace	2.7	3.0
				Dry pomace	9.6	11
				White aged wine	0.07	0.08

AF wine: After alcoholic fermentation, MLF wine: After malolactic fermentation, Young wine: At bottling

Aged wine: 6 months after bottling

Two processing trials were conducted on grape in Southern France and Italy during 2013. Two applications of an SC formulation of pydiflumetofen containing 200 g/L were applied at a rate of 200 g ai/ha separated by an interval of 14 days. Treated samples were collected at 21 day (normal commercial harvest) after the final application (Tessier, 2015: S13-03422). Field samples for processing were shipped chilled to the processing facility.

Fresh grapes were processed into raisins, red wine and grape seed oil. Two balance studies and one follow-up study were carried out on each trial, for each process. Relevant industrial practices and standardised procedures were applied to simulate the common processes used by industry for production of raisins, red wine and grape seed oil.

Raisin (soaking) processing

Grapes were stemmed by hand and soaked in equal amount of soaking solution (50 g of potassium bicarbonate per litre of water) for one minute. The berries were drained and put in an oven, on drawers covered with baking paper regulated at 60 °C. The drying was stopped when the weight of the dried berries were decreased to at least 65%.

Red wine processing

Bunches of red grapes were stemmed and crushed. The must was sulphited with addition of potassium metabisulphite and dry activated yeasts were added to begin alcoholic fermentation. Each working day, the solid and liquid phases were mixed for homogenisation. Sugar was added if the probable degree of alcohol of the harvest was insufficient. The progress of the alcoholic fermentation was followed by measuring the density and the temperature of the must on each working day. When the alcoholic fermentation (AF) finished (the density stabilizing < 1000), the AF wine was run off to the tank (free-run wine) and the solid part was pressed with water press to recover the maximum quantity of wine. The pressed wine was added to the free-run wine. Wet pomace specimens were taken and the remaining pomace was dried in an oven regulated at approximately 60 °C to produce dry pomace. A seeding of lactic bacteria was made to the wine after pressing to allow the malolactic fermentation to begin. Paper chromatography was performed each week in order to follow the progress of the malolactic fermentation. Once the malolactic fermentation finished, the wine was sulphited. After the completion of malolactic fermentation, the wine was racked for at least four days. The wine was separated from lees and clarified with gelatin in a cold room (temperature <10 °C) for at least 14 days to prevent possible tartaric precipitation. After racking, the wine was filtered and potassium metabisulphite was added. Wine was bottled and stored in a cold room at a temperature between 5 °C and 10 °C. After six months of storage, the aged wine was bottled again.

Grape seed oil processing

The kept wet pomace was dried in an oven regulated at approximately 60 °C until the drying was deemed visually complete. The dried pomace was sieved with a help of a grading unit to pull off the grape seeds from pomace. The grape seeds were introduced into the press. The cake was collected in a plastic container placed at the press head exit and the oil in a plastic bucket placed under the press head. Obtained oil was separated in two samples to produce raw oil and refined oil. After a settling of 30 minutes, the oil was centrifuged and then filtered to produce raw oil. Soda (115 g/L) was added and the mixing was maintained approximately during 30 minutes in an oven at 90 °C to produce refined oil. After a settling of 30 minutes, the oil was separated to waste.

Whole fruit and grape processed fractions were analysed for residues of pydiflumetofen using analytical method GRM061.03A, with a limit of quantification (LOQ) of 0.01 mg/kg. GRM061.03A method was validated for the analysis of pydiflumetofen in dry raisins, must, red wine (at bottling), refined oil, cake (after pressing) and dry pomace. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%.

Table 87 Residues of pydiflumetofen in grapes and grape processed commodities

country, year (variety)	Application		DALA Days	Commodity	Residues, mg/kg	Processing factor
	kg ai/ha	no.				
Southern France, 2013 Languedoc-Roussillon (Mourvèdre)	0.20 0.20	2	21	Whole grape Stems Berries Soaked liquid Soaking berries Raisin	0.15, 0.23 Mean 0.19 0.15 0.15 0.03 0.10 0.45	- 0.79 0.79 0.16 0.53 2.4
				Whole grape Raisin	0.22, 0.24 Mean 0.23 0.57	- 2.5

country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg	Processing factor
				Whole grape	0.16, 0.17 Mean 0.17	-
				Stems	0.30	1.8
				Must	0.29	1.7
				AF wine	0.05	0.29
				Wet pomace	0.73	4.3
				Dry pomace	4.0	24
				MLF wine	0.04	0.24
				Lees	0.73	4.3
				Sediments	0.07	0.41
				Red wine	0.04	0.24
				Aged wine	0.04	0.24
				Whole grape	0.17, 0.19 Mean 0.18	-
				Must	0.37	2.1
				Wet pomace	1.1	6.3
				Dry pomace	2.5	14
				Aged wine	0.03	0.17
				Grape seeds	0.86	-
				Waste	3.1	3.7
				Cake	0.60	0.70
				Raw oil	0.65	0.76
				Waste soap	1.0	1.2
				Refined oil	0.88	1.0
				Raw oil	1.1	1.3
				Refined oil	0.96	1.1
Italy, 2013 Emilia Romagna (Barbera)	0.20 0.20	2	21	Whole grape	0.07, 0.07 Mean 0.07	-
				Stems	0.45	6.4
				Berries	0.03	0.43
				Soaked berries	0.02	0.29
				Soaking liquid	0.02	0.29
				Raisin	0.12	1.7
				Whole grape	0.03, 0.05 Mean 0.04	-
				Raisin	0.19	4.8
				Whole grape	0.06, 0.14 Mean 0.10	-
				Stems	0.19	1.9
				Must	0.15	1.5
				AF wine	0.02	0.20
				Wet pomace	0.44	4.4
				Dry pomace	1.8	18
				MLF wine	0.01	0.10
				Lees	0.47	4.7
				Sediments	0.07	0.70
				Red wine	0.01	0.10
				Aged wine	0.01	0.10
				Whole grape	0.04, 0.05 Mean 0.05	-
				Must	0.15	3.0
				Wet pomace	0.40	8.0
				Dry pomace	1.7	35
				Aged wine	<0.01	<0.20
				Grape seeds	0.52	-
				Waste	3.0	5.8
				Cake	0.42	0.81
				Raw oil	0.61	1.2
				Waste soap	0.17	0.33
				Refined oil	0.56	1.1
				Raw oil	0.42	0.71
				Refined oil	0.37	0.71

AF wine: After alcoholic fermentation, MLF wine: After malolactic fermentation, Young wine: At bottling

Aged wine: 6 months after bottling

Two grape field trials were conducted in the USA to generate grape samples for processing, received two foliar broadcast spray applications at the nominal rate of 1.0 kg ai/ha/application, at 28 and 14 days before harvest (Salzman, 2015: TK0103853). All spray mixtures included an adjuvant (crop oil concentrate or non-ionic surfactant). Bulk grape samples were collected at 14 days after the last foliar spray application (DALA) to generate processed fraction samples. The bulk grape bunch samples were processed to produce pre-processing grapes, wet pomace, juice, and raisin samples.

After the whole fruit samples were collected, whole grapes from random bunch were carefully destemmed by hand and packed one layer deep on dehydrator trays. The trays were staked onto one dehydrator and operated at 54–60 °C for about 26 hours to produce raisins. Any wastes from the hand destemming operation were returned to the bulk grape samples.

The remaining grapes were passed through a Crusher/Destemmer to remove stems and produce grape mash. The mash was layered into cloth stacks on a hydraulic press and pressed to separate juice from wet pomace. While the mash was pressed, juice samples were collected. The juice samples were a minimum of four pounds. After pressing, the wet pomace stacks were mixed and a minimum of 0.91 kg samples collected. Within 15 minutes of collection, samples were placed in frozen storage at temperatures of <-15 °C.

Method GRM061.03A was used to determine the levels of pydiflumetofen. The LOQ, defined as the lowest fortification level, was 0.01 mg/kg for all commodities. Concurrent procedural recoveries for pydiflumetofen from processed grape commodity samples analysed were between 92% and 108%.

Table 88 Residues of pydiflumetofen in grapes and grape processed commodities

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg	Processing factor
	kg ai/ha	water, L/ha	BBCH	no.				
USA, 2013	1.01	758	88-89	2	14	Grapes	1.4, 2.1, 2.9 Mean 2.1	-
Madera/CA	1.01	758	88-89			Wet pomace	2.1, 2.3 Mean 2.2	1.0
(Thompson						Juice	1.1, 1.3 Mean 1.2	0.57
Seedless)						Raisins	4.1, 4.3 Mean 4.2	2.0
USA, 2013	1.00	543	83-85	2	14	Grapes	0.26, 0.30, 0.34 Mean 0.30	-
Kingsburg/CA	1.03	552	85-89			Wet pomace	0.64, 0.76 Mean 0.70	2.4
(Thompsons)						Juice	0.14, 0.15 Mean 0.15	0.49
						Raisins	0.83, 0.85 Mean 0.84	2.8

Tomato

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw tomato fruits and processed fractions of tomato fruits. Two tests were carried out in the USA following exaggerated treatment with pydiflumetofen 200 g ai/L SC formulation during the 2013 growing season (Salzman, 2015: TK0163552). In all tests, two foliar applications were made to the crop each at a rate of 625 g ai/ha, 5 × the critical USA GAP, with a 7 day interval. Samples of tomato were taken 0 DALA.

Samples were analysed for residues of pydiflumetofen analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%. Processed tomato samples were stored at *ca* -10 °C for a maximum of 17 months between sampling and analysis.

Table 89 Residues of pydiflumetofen in processed commodities of tomato

Tomato country, year (variety)	Application		DALA Days	Commodity	Residues, mg/kg	Processing Factor
	kg ai/ha	no.				
USA, 2013	0.61	2	0	Fruit	0.10, 0.12, 0.17 mean 0.13	
Kettleman City, CA	0.63			Paste	0.10, 0.11 mean 0.11	0.82
(Galilea)				Puree	0.051, 0.055 mean 0.053	0.41
Outdoor				Washed & Peeled	<0.01, <0.01 mean <0.01	≤0.077
				Canned	<0.01, <0.01 mean <0.01	≤0.077
				Sun Dried	1.3, 1.3 mean 1.3	9.9
				Juice (pasteurised)	<0.01, <0.01 mean <0.01	≤0.077
				Wet Pomace	0.36, 0.49 mean 0.43	3.3
				Dried Pomace	5.8, 5.8 mean 5.8	45
USA, 2013	0.62	2	0	Fruit	0.18, 0.23, 0.25 mean 0.22	
Porterville, CA	0.62			Paste	0.12, 0.12 mean 0.12	0.55
(AB2, Roma type)				Puree	0.046, 0.068 mean 0.057	0.26

Tomato country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg	Processing Factor
Outdoor				Washed & Peeled	<0.01, <0.01 mean <0.01	≤0.046
				Canned	<0.01, <0.01 mean <0.01	≤0.046
				Sun Dried	2.3, 2.4 mean 2.3	11
				Juice (pasteurised)	<0.01, <0.01 mean <0.01	≤0.046
				Wet Pomace	0.89, 1.1 mean 0.99	4.5
				Dried Pomace	7.6, 8.9 mean 8.2	38

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in tomato fruits (prior to processing)

For calculations, 0.01 mg/kg was used for fractions with <LOQ residues.

Soya bean

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw soya bean seeds and processed fractions of soya bean seeds. Two tests were carried out in the USA following exaggerated treatment with pydiflumetofen 200 g ai/L SC formulation during the 2013 growing season (Oakes, 2015: TK0103860). In all tests, two foliar applications were made to the crop each at a rate of 600 g ai/ha, 3 × the critical USA GAP, with a 7 day interval. Samples of soya bean seeds were taken 14 DALA.

Samples were analysed for residues of pydiflumetofen using analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%. Processed soya bean samples were stored at -18 °C or below for a maximum of 14 months between sampling and analysis.

Table 90 Residues of pydiflumetofen in processed commodities of soya beans

Soya bean country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg	Processing Factor
USA, 2013	0.60	2	14	Seed	0.16, 0.18, 0.20 mean 0.18	
Richland, IA	0.60			Meal	0.011, 0.020 mean 0.016	0.090
(93Y60)				Hulls	0.65, 0.68 mean 0.67	3.7
Outdoor				Refined Oil	0.047, 0.055 mean 0.051	0.29
				Flour	<0.01, 0.010 mean 0.010	≤0.056
				Milk	<0.01, <0.01 mean <0.01	≤0.056
				Tofu	0.016, 0.036 mean 0.026	0.15
				Soy Sauce	ND, <0.01 mean <0.01	≤0.056
				Miso	0.027, 0.035 mean 0.031	0.17
				Pollard	0.063, 0.065 mean 0.064	0.36
				Crude Oil	0.18, 0.19 mean 0.19	1.1
				AGF	20, 23 mean 21	121
USA, 2013	0.60		14	Seed	0.14, 0.15, 0.17 mean 0.15	
Cooper, IA	0.61	2		Meal	0.010, 0.010 mean 0.010	0.065
(93Y13)				Hulls	0.45, 0.45 mean 0.45	2.9
Outdoor				Refined Oil	0.010, 0.014 mean 0.012	0.078
				Flour	<0.01, <0.01 mean <0.01	≤0.064
				Milk	ND, <0.01 mean <0.01	≤0.064
				Tofu	0.021, 0.021 mean 0.021	0.14
				Soy Sauce	ND, ND mean <0.01	≤0.064
				Miso	0.012, 0.015 mean 0.014	0.091
				Pollard	0.038, 0.041 mean 0.040	0.26
				Crude Oil	0.013, 0.075 mean 0.044	0.29
				AGF	21, 27 mean 24	156

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in soya bean seed (prior to processing)

For calculations, 0.01 mg/kg was used for fractions with <LOQ residues.

Potato

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw potato tubers and processed fractions of potato tubers. Two tests were carried out in the USA following exaggerated treatment with pydiflumetofen 200 g ai/L

SC formulation during the 2013 growing season (Smith, 2015: TK0163562). In all tests, three foliar applications were made to the crop each at a rate of 625 g ai/ha, 5 × the critical USA GAP, with a 7 day interval. Samples of potato were taken 7 DALA.

Samples were analysed for residues of pydiflumetofen using analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%. Processed potato samples were stored at *ca* -10 °C for a maximum of 18 months between sampling and analysis.

Table 91 Residues of pydiflumetofen in processed commodities of potato

Potato country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg	Processing Factor
USA, 2013 Jerome, ID (Ranger Russet) Outdoor	0.62 0.62 0.63	3	6	Tuber Flakes Chips Wet Peel Peeled Peeled and Boiled Unpeeled and Boiled Unpeeled and Baked Crisps Cooking Liquid Starch Dried Pulp Protein	<0.01, <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 0.029, 0.033 mean 0.031	 ≥3.1
USA, 2013 American Falls, ID (Russet Burbank) Outdoor	0.62 0.62 0.62	3	6	Tuber Flakes Chips Wet Peel Peeled Peeled and Boiled Unpeeled and Boiled Unpeeled and Baked Crisps Cooking Liquid Starch Dried Pulp Protein	<0.01, 0.014, 0.042 mean 0.022 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 0.044, 0.049 mean 0.047 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 <0.01, <0.01 mean <0.01 0.093, 0.098 mean 0.096 0.041, 0.050 mean 0.045	 ≤0.45 ≤0.45 2.1 ≤0.45 ≤0.45 ≤0.45 ≤0.45 ≤0.45 ≤0.45 ≤0.45 4.3 2.1

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in potato tubers (prior to processing)

For calculations, 0.01 mg/kg was used for fractions with <LOQ residues.

Wheat

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw wheat grain and processed fractions of wheat grain. Two tests were carried out in the USA following exaggerated treatment with pydiflumetofen 200 g ai/L SC formulation during the 2014 growing season (Oakes, 2015: TK0163574). In all tests, two foliar applications were made to the crop each at a rate of 750 g ai/ha followed by 1,000 g ai/ha, 5 × the critical USA GAP, with a 13 day interval. Samples of wheat grain were taken at normal crop harvest maturity.

Samples were analysed for residues of pydiflumetofen using analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%. Processed samples were stored at -18 °C or below for a maximum of 10 months between sampling and analysis.

Table 92 Residues of pydiflumetofen in processed commodities of wheat

wheat country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg[a]	Processing Factor
USA, 2014 Bloomfield, MO (McAlister) Outdoor	0.73 1.0	2	32	Grain AGF Bran Flour Middlings Shorts Germ	0.052, 0.25, 0.39 mean 0.23 135, 141 mean 138 0.57, 0.62 mean 0.59 0.076, 0.080 mean 0.078 0.13, 0.13 mean 0.13 0.21, 0.21 mean 0.21 0.23, 0.24 mean 0.24	 598 2.6 0.34 0.56 0.90 1.0

wheat country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg[a]	Processing Factor
				Gluten	0.41, 0.66 mean 0.53	2.3
				Starch	<0.010, 0.013 mean 0.012	0.050
				Gluten Feed Meal	0.49, 0.62 mean 0.56	2.4
				Milled Byproducts	0.43, 0.72 mean 0.58	2.5
				Whole Meal Flour	0.17, 0.18 mean 0.18	0.77
				Whole Meal Bread	0.098, 0.11 mean 0.10	0.45
USA, 2014 Hinton, OK (Duster) Outdoor	0.74 1.0	2	35	Grain	0.089, 0.85, 0.92 mean 0.62	
				AGF	76, 81 mean 79 c 0.023	127
				Bran	1.1, 1.2 mean 1.2	1.9
				Flour	0.17, 0.19 mean 0.18	0.30
				Middlings	0.28, 0.29 mean 0.28	0.46
				Shorts	0.34, 0.35 mean 0.34	0.55
				Germ	1.1, 1.2 mean 1.2	1.9
				Gluten	0.60, 0.79 mean 0.69	1.1
				Starch	<0.010, 0.014 mean 0.012	0.019
				Gluten Feed Meal	0.76, 0.80 mean 0.78	1.3
				Milled Byproducts	5.7, 6.3 mean 6.0	9.7
				Whole Meal Flour	0.44, 0.47 mean 0.46	0.73
				Whole Meal Bread	0.24, 0.27 mean 0.25	0.41

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in wheat grain (prior to processing)

[a] c: sample from control plot

Barley

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw barley grain and processed fractions of barley grain. Two tests were carried out in the USA following exaggerated treatment with pydiflumetofen 200 g ai/L SC formulation during the 2013 growing season (Oakes, 2015: TK0163584). In all tests, two foliar applications were made to the crop each at a rate of 750 g ai/ha followed by 1,000 g ai/ha, 5 × the critical USA GAP, with a 14 day interval. Samples of barley grain were taken at normal crop harvest maturity.

Samples were analysed for residues of pydiflumetofen using analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%. Processed barley samples were stored at -18 °C or below for a maximum of 16 months between sampling and analysis.

Table 93 Residues of pydiflumetofen in processed commodities of barley

Barley country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg[a]	Processing Factor
USA, 2013 Bagley, IA (Robust) Outdoor	0.72 0.99	2	28	Grain	12, 12, 13 mean 12 c 0.034	
				Pearled Barley	0.26, 0.34 mean 0.30	0.024
				Bran	2.9, 3.0 mean 3.0	0.24
				Flour	2.8, 3.0 mean 2.9	0.23
USA, 2013 Carrington, ND (Robust) Outdoor	0.69 0.99	2	52	Grain	0.71, 0.75, 0.85 mean 0.77 c 0.035	
				Pearled Barley	0.047, 0.049 mean 0.048	0.062
				Bran	0.34, 0.40 mean 0.37	0.48
				Flour	0.17, 0.18 mean 0.17	0.23

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in barley grain (prior to processing)

[a] c: sample from control plot

Oats

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw oat grain and processed fractions of oat grain. Two tests were carried out in the USA following exaggerated treatment with pydiflumetofen 200 g ai/L SC formulation during the 2013 growing season (McDonald, 2015: TK0163581). In all tests, two foliar applications were made to the crop each at a rate of 750 g ai/ha followed by 1 000 g ai/ha, 5 × the critical USA GAP, with a 14 day interval. Samples of oat grain were taken at normal crop harvest maturity.

Samples were analysed for residues of pydiflumetofen using analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%. Processed oat samples were stored at -18 °C or below for a maximum of 20 months between sampling and analysis.

Table 94 Residues of pydiflumetofen in processed commodities of oats

Oats country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg[a]	Processing Factor
USA, 2013	0.62	2	18	Grain	3.5, 3.7, 3.8 mean 3.7	
Richland, IA	1.0			Rolled Oats	0.046, 0.049 mean 0.048	0.013
(NC Jerry Oats)				Bran	0.060, 0.064 mean 0.062	0.017
Outdoor				Flour	0.20, 0.30 mean 0.25	0.068
				Husks	10, 17 mean 14 c 0.014	3.8
USA, 2013	0.60	2	28	Grain	3.6, 4.2, 4.6 mean 4.1 c 0.035	
Gardner, ND	0.98			Rolled Oats	0.046, 0.053 mean 0.050	0.012
(Horsepower)				Bran	0.049, 0.057 mean 0.053	0.013
Outdoor				Flour	0.095, 0.13 mean 0.11	0.027
				Husks	12, 13 mean 12 c 0.012	3.0

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in oats grain (prior to processing)

[a] c: sample from control plot

Maize

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw maize grain and processed fractions of maize grain. Two tests were carried out in the USA following exaggerated treatment with pydiflumetofen 200 g ai/L SC formulation during the 2014 growing season (Oakes, 2015: TK0163563). In all tests, two foliar applications were made to the crop each at a rate of 630 g ai/ha, 5 × the critical USA GAP, with a 7 day interval. Samples of maize grain were taken 30 DALA.

Samples were analysed for residues of pydiflumetofen using analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%. Processed maize samples were stored at *ca* -20 °C for a maximum of 8 months between sampling and analysis.

Table 95 Residues of pydiflumetofen in processed commodities of maize

Maize country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg[a]	Processing Factor
USA, 2014	0.62	2	32	Grain	0.008, 0.009, 0.009	
Fairfield, IA	0.62				mean 0.0086	
(P0993HR)				AGF	0.60, 0.62 mean 0.61	71
Outdoor				Milled Byproducts	0.023, 0.023 mean 0.023	2.7
				Wet-Milled Germ	0.018, 0.020 mean 0.019	2.2
				Wet-Milled Starch	<0.010, <0.010 mean <0.010	≤1.2
				Wet-Milled Gluten	0.013, 0.020 mean 0.017	1.9
				Wet-Milled Gluten Meal	0.027, 0.029 mean 0.028	3.3
				Wet-Milled Refined Oil	0.012, 0.014 mean 0.013	1.5
				Dry-Milled Grits	<0.010, <0.010 mean <0.010	≤1.2
				Dry-Milled Meal	<0.010, <0.010 mean <0.010	≤1.2
				Dry-Milled Flour	<0.010, <0.010 mean <0.010	≤1.2
				Dry-Milled Hulls	0.047, 0.053 mean 0.050	5.8
				Dry-Milled Germ	<0.010, <0.010 mean <0.010	≤1.2
				Dry-Milled Refined Oil	<0.010, <0.010 mean <0.010	≤1.2
				Wet-Milled Flour	<0.010, <0.010 mean <0.010	≤1.2
USA, 2014	0.61	2	30	Grain	0.021, 0.024, 0.026 mean 0.024	
Northwood, ND	0.63			AGF	1.4, 1.9 mean 1.6 c 0.11	69
(DKC35- 54RIB)				Milled Byproducts	0.032, 0.049 mean 0.041	1.7
Outdoor				Wet-Milled Germ	0.047, 0.047 mean 0.047	2.0
				Wet-Milled Starch	<0.010, <0.010 mean <0.010	≤0.42
				Wet-Milled Gluten	0.013, 0.020 mean 0.017	0.70
				Wet-Milled Gluten Meal	0.062, 0.083 mean 0.073	3.1
				Wet-Milled Refined Oil	0.052, 0.057 mean 0.055	2.3
				Dry-Milled Grits	<0.010, <0.010 mean <0.010	≤0.42
				Dry-Milled Meal	0.022, 0.022 mean 0.022	0.93

Maize country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg[a]	Processing Factor
				Dry-Milled Flour	0.037, 0.041 mean 0.039	1.6
				Dry-Milled Hulls	0.085, 0.090 mean 0.088	3.7
				Dry-Milled Germ	0.021, 0.022 mean 0.022	0.91
				Dry-Milled Refined Oil	<0.010, <0.010 mean <0.010	≤0.42
				Wet-Milled Flour	<0.010, <0.010 mean <0.010	≤0.42

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in maize grain (prior to processing)

For calculations, 0.01 mg/kg was used for fractions with <LOQ residues.

[a] c: sample from control plot

Sweet corn

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw sweet corn ears and processed fractions of sweet corn ears. Two tests were carried out in the USA following exaggerated treatment with pydiflumetofen 200 g ai/L SC formulation during the 2014 growing season (Oakes, 2015: TK0223529). In all tests, two foliar applications were made to the crop each at a rate of 625 g ai/ha, 5 × the critical USA GAP, with a 7-day interval. Samples of sweet corn were taken 7 DALA.

Samples were analysed for residues of pydiflumetofen using analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%. Processed sweet corn samples were stored at *ca* -18 °C for a maximum of 7 months between sampling and analysis.

Table 96 Residues of pydiflumetofen in processed commodities of sweet corn

Sweet corn country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg	Processing Factor
USA, 2014	0.63	2	6	Ears	<0.010, <0.010, <0.010 mean <0.010	
High Springs, FL	0.62			Canned Corn	<0.010, <0.010 mean <0.010	nc
(Passion II)				Cannery Waste	1.6, 1.6 mean 1.6	≥159
Outdoor				Cream Corn	<0.010, <0.010 mean <0.010	nc
				Frozen Corn	<0.010, <0.010 mean <0.010	nc
USA, 2014	0.62	2	7	Ears	<0.010, <0.010, <0.010 mean <0.010	
York, NE	0.61			Canned Corn	<0.010, <0.010 mean <0.010	nc
(Obsession II)				Cannery Waste	1.8, 1.9 mean 1.9	≥186
Outdoor				Cream Corn	<0.010, <0.010 mean <0.010	nc
				Frozen Corn	<0.010, <0.010 mean <0.010	nc

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in sweet corn ears (prior to processing)

For calculations, 0.01 mg/kg was used for fractions with <LOQ residues.

nc: not able to calculate processing factor (all values <LOQ).

Rape seed

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw rape seed and processed fractions of rape seed. Two tests were carried out in the USA following exaggerated treatment with pydiflumetofen 200 g ai/L SC formulation or 100 g ai/L EC formulation during the 2013 growing season (Smith, 2015: TK0163606). In all tests, two foliar applications were made to the crop each at a rate of 625 g ai/ha followed by 1 000 g ai/ha, 5 × the critical USA GAP, with a 14-day interval. Samples of rape seed were taken 30 DALA.

Samples were analysed for residues of pydiflumetofen analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The mean recovery for pydiflumetofen from rape seed as 93%, from meal as 91% and from refined oil as 90% at 0.01–1.0 mg/kg fortified. Processed rape seed samples were stored at -10 °C or below for a maximum of 10 months between sampling and analysis.

Table 97 Residues of pydiflumetofen in processed commodities of rape seed

Rape seed country, year (variety)	Application Form	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg[a]	Processing Factor
USA, 2013	EC	0.62	2	30	Seed	0.31, 0.31, 0.32 mean 0.31 c 0.011	
Gardner, ND		0.99			Meal	0.029, 0.029 mean 0.029	0.094

Rape seed country, year (variety)	Application			DALA Days	Commodity	Residues, mg/kg[a]	Processing Factor
	Form	kg ai/ha	no.				
(DKL51-45) Outdoor					Refined Oil	0.11, 0.11 mean 0.11	0.36
USA, 2013 Carrington, ND (LL 5440) Outdoor	SC	0.59 1.0	2	31	Seed Meal Refined Oil	0.69, 0.77, 0.78 mean 0.74 0.062, 0.067 mean 0.065 0.28, 0.29 mean 0.28	0.087 0.38

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in rape seed (prior to processing)

[a] c: sample from control plot

Peanut

The study was conducted to determine the magnitude of residues of pydiflumetofen in/on raw peanut nutmeat and processed fractions of peanut. Two tests were carried out in the USA following exaggerated treatment during the 2013 growing season (Smith, 2015: TK0163545). In one trial four foliar broadcast applications of pydiflumetofen 200 g ai/L SC formulation were applied to the crop at a total nominal rate of 1 000 g ai/ha (5× the critical USA GAP) separated by 14 days. In the second trial four foliar broadcast applications of pydiflumetofen 100 g ai/L EC formulation were applied at a rate of 1 000 g ai/ha separated by an interval of 14 days. Samples of peanut nutmeat were taken 14 DALA.

Samples were analysed for residues of pydiflumetofen following analytical method GRM061.03A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for pydiflumetofen in each matrix were within 70–120%. Processed peanut samples were stored at -10 °C or below for a maximum of 18 months between sampling and analysis.

Table 98 Residues of pydiflumetofen in processed commodities of peanut

Peanut country, year (variety)	Application			DALA Days	Commodity	Residues, mg/kg	Processing Factor
	Form	kg ai/ha	no.				
USA, 2013 Chula, GA (Georgia 06G) Outdoor	EC	0.25 ×4	4	14	Nutmeat Meal Refined Oil	<0.01, <0.01, <0.01 mean <0.01 0.013, 0.013 mean 0.013 0.022, 0.022 mean 0.022	≥1.3 ≥2.2
USA, 2013 Seven Springs, NC (Bailey) Outdoor	SC	0.25 ×4	4	12	Nutmeat Meal Refined Oil	0.024, 0.025, 0.028 mean 0.025 <0.01, <0.01 mean <0.01 0.059, 0.060 mean 0.060	≤0.40 2.4

Processing Factor = Pydiflumetofen residues in processed commodity/ Pydiflumetofen residues in peanut nutmeat (prior to processing)

For calculations, 0.01 mg/kg was used for fractions with <LOQ residues.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The Meeting received lactating dairy cow feeding studies.

Lactating dairy cow

Study 1

A residue feeding study in lactating cows was conducted in the UK to measure the residue levels, confirm plateau of residues and concentrations of pydiflumetofen and metabolites in lactating ruminant tissues and milk following daily dosing with pydiflumetofen over a 28 day exposure period (Harris, 2015: 35775).

Eleven lactating Holstein/Friesian cows were split into 4 groups for use in the study. Two control cows (Group 1) received gelatin dosing capsules containing no active substance. Pydiflumetofen was administered orally in gelatin capsules once daily to the remaining three groups of three cows at rates based on average daily dietary intakes. The nominal feeding rates were 15 ppm dry matter (DM) (Group 2, 1 × rate), 45 ppm DM (Group 3, 3 × rate) and 150 ppm DM (Group 4, 10 × rate). The capsules were administered daily for 28 consecutive days (Study Days 1-28). The actual mean daily dose rates achieved ranged between 15.67–

15.70 ppm DM for Group 2 animals (1×), 47.09–47.28 ppm DM for Group 3 animals (3×) and 152.3–152.56 ppm DM for Group 4 animals (10×). The test substance pydiflumetofen was demonstrated to be stable in the dosing medium.

Milk was collected twice daily with the afternoon sampling combined with the next morning's sample. Milk samples were collected and analysed from individual cows on Days -1 (pre-dosing), 1, 3, 5, 7, 10, 14, 17, 21, 24 and 28. Skimmed milk and cream samples were produced from milk collected from all animals on Study Days 14 and 28.

Animal 1 (control animal, Group 1) was prematurely euthanised on welfare grounds on Day 11 due to an underlying health condition (lameness), unrelated to on-study procedures. A replacement control animal was assigned (Animal 11). All animals, with exception of Animal 1 were killed within 22–24 hours following the final dose (28th dose) by captive bolt, pithing and exsanguination. Samples of liver, kidney, muscle (composite of loin and hind leg) and fat (perirenal, mesenterial and subcutaneous) were collected from all animals. Samples were stored frozen until analysed.

Samples of milk and tissues were analysed for residues of pydiflumetofen and the 2,4,6-trichlorophenol (TCP) metabolite. In addition dairy samples (milk, skimmed milk and cream) were analysed for the metabolites SYN548264 and SYN508272 and samples of liver and kidney were analysed for the metabolites SYN547897 and SYN548263.

All matrices were analysed for pydiflumetofen using Method GRM061.06A. All matrices were analysed for metabolite 2,4,6-trichlorophenol (sum of free and conjugated) following enzymic hydrolysis of sample extracts using Method GRM061.07A. SYN548264 and SYN508272 (metabolites) were measured in milk, skimmed milk and cream as the free metabolites using Method GRM061.08A. SYN548263 and SYN547897 were measured in liver and kidney, as the sum of free and conjugated metabolite, following enzymic hydrolysis of sample extracts using Method GRM061.09A. The LOQ for pydiflumetofen and metabolites was 0.01 mg/kg for each analyte. The overall mean recoveries from concurrent fortifications of pydiflumetofen, 2,4,6-TCP, SYN548264, SYN508272, SYN548263 and SYN547897 in each lactating cow matrices were within 70–120%.

The maximum frozen storage intervals (*ca* -20 °C) prior to analysis were 306 days for milk, 317 days for skimmed milk, 308 days for cream, 323 days for liver, 326 days for kidney, 258 days for muscle and 316 days for fat.

Table 99 Pydiflumetofen residues in bovine milk (mg/kg)

Dose rate	Animal	Study Day										
		-1	1	3	5	7	10	14	17	21	24	28
Control	1 or 11	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
15 ppm (1×)	2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	3	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	4	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	Mean	-	-	-	-	-	-	-	-	-	-	-
45 ppm (3×)	5	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	6	ND	ND	ND	<0.01	<0.01	<0.01	ND	ND	<0.01	ND	<0.01
	7	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Mean	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
150 ppm (10×)	8	ND	<0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.01	0.02
	9	ND	<0.01	0.01	0.01	0.01	<0.01	<0.01	<0.01	<0.01	0.01	0.01
	10	ND	<0.01	0.01	0.01	0.01	0.01	0.01	<0.01	0.01	0.02	0.01
	Mean	ND	<0.01	0.01	0.01	0.01	<0.01	<0.01	<0.01	<0.01	0.01	0.01

NA = not analysed (<LOQ for the previous dose group)

ND = not detected (<0.0025 mg/kg)

Table 100 2,4,6-Trichlorophenol (TCP) residues in bovine milk (mg/kg)

Dose rate	Animal	Study Day										
		-1	1	3	5	7	10	14	17	21	24	28
Control	1 or 11	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
15 ppm (1×)	2	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	3	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	4	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Mean	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
45 ppm (3×)	5	ND	<0.01	0.01	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
	6	ND	<0.01	<0.01	0.01	0.02	0.01	0.02	0.01	0.01	0.02	0.01
	7	ND	<0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
	Mean	ND	<0.01	<0.01	0.01	0.02	0.01	0.02	0.01	0.01	0.02	0.01
150 ppm	8	ND	0.06	0.09	0.10	0.10	0.09	0.09	0.08	0.08	0.08	0.08

Dose rate	Animal	Study Day										
		-1	1	3	5	7	10	14	17	21	24	28
(10×)	9	ND	0.06	0.09	0.06	0.10	0.08	0.08	0.09	0.10	0.10	0.08
	10	ND	0.06	0.09	0.08	0.07	0.09	0.08	0.07	0.08	0.08	0.06
	Mean	ND	0.06	0.09	0.08	0.09	0.09	0.08	0.08	0.09	0.09	0.07

NA = not analysed (<LOQ for the previous dose group)

ND = not detected (<0.0025 mg/kg)

Table 101 SYN548264 residues in bovine milk (mg/kg)

Dose rate	Animal	Study Day										
		-1	1	3	5	7	10	14	17	21	24	28
Control	1 or 11	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
15 ppm (1×)	2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	3	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	4	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	Mean	-	-	-	-	-	-	-	-	-	-	-
45 ppm (3×)	5	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	6	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	7	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	Mean	-	-	-	-	-	-	-	-	-	-	-
150 ppm (10×)	8	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	9	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	10	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01
	Mean	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

NA = not analysed (<LOQ for the previous dose group)

ND = not detected (<0.0025 mg/kg)

Table 102 SYN508272 residues in bovine milk (mg/kg)

Dose rate	Animal	Study Day										
		-1	1	3	5	7	10	14	17	21	24	28
Control	1 or 11	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
15 ppm (1×)	2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	3	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	4	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	Mean	-	-	-	-	-	-	-	-	-	-	-
45 ppm (3×)	5	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	6	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	7	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	Mean	-	-	-	-	-	-	-	-	-	-	-
150 ppm (10×)	8	ND	ND	ND	ND	ND	ND	<0.01	ND	ND	ND	ND
	9	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	<0.01	ND	<0.01
	10	ND	ND	ND	<0.01	ND	<0.01	<0.01	ND	<0.01	<0.01	0.01
	Mean	ND	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

NA = not analysed (<LOQ for the previous dose group)

ND = not detected (<0.0025 mg/kg)

Table 103 Pydiflumetofen and metabolite residues in bovine skimmed milk and cream (mg/kg)

Dose rate	Study Day	Animal	Pydiflumetofen		2,4,6-TCP		SYN548264		SYN508272	
			Individual	Mean	Individual	Mean	Individual	Mean	Individual	Mean
Skimmed milk										
Control	14	1	NA	-	NA	-	NA	-	NA	-
	28	11	NA	-	NA	-	NA	-	NA	-
15 ppm (1×)	14	2	NA	-	<0.01	<0.01	ND	ND	NA	-
		3	NA		<0.01		ND		NA	
		4	NA		<0.01		ND		NA	

Dose rate	Study Day	Animal	Pydiflumetofen		2,4,6-TCP		SYN548264		SYN508272	
			Individual	Mean	Individual	Mean	Individual	Mean	Individual	Mean
	28	2	NA	-	<0.01	<0.01	ND	ND	NA	-
		3	NA		<0.01		ND		NA	
		4	NA		<0.01		ND		NA	
45 ppm (3×)	14	5	NA	-	0.01	0.01	<0.01	<0.01	NA	-
		6	NA		0.01		<0.01		NA	
		7	NA		0.01		<0.01		NA	
	28	5	NA	-	0.01	0.01	<0.01	<0.01	NA	-
		6	NA		0.01		<0.01		NA	
		7	NA		0.01		ND		NA	
150 ppm (10×)	14	8	ND	<0.01	0.08	0.08	<0.01	<0.01	<0.01	<0.01
		9	ND		0.08		<0.01		<0.01	
		10	<0.01		0.07		ND		<0.01	
	28	8	ND	ND	0.06	0.08	0.01	<0.01	<0.01	<0.01
		9	ND		0.09		<0.01		<0.01	
		10	ND		0.08		0.01		<0.01	
Cream										
Control	14	1	NA	-	NA	-	NA	-	NA	-
	28	11	NA	-	NA	-	NA	-	NA	-
15 ppm (1×)	14	2	0.01	0.01	<0.01	<0.01	NA	-	NA	-
		3	0.01		<0.01		NA		NA	
		4	0.01		0.02		NA		NA	
	28	2	0.01	0.01	<0.01	<0.01	NA	-	NA	-
		3	0.01		<0.01		NA		NA	
		4	0.01		0.01		NA		NA	
45 ppm (3×)	14	5	0.04	0.03	0.02	0.01	NA	-	NA	-
		6	0.02		0.02		NA		NA	
		7	0.03		0.02		NA		NA	
	28	5	0.04	0.04	0.02	0.01	NA	-	NA	-
		6	0.03		0.02		NA		NA	
		7	0.04		0.02		NA		NA	
150 ppm (10×)	14	8	0.14	0.13	0.06	0.06	<0.01	<0.01	ND	ND
		9	0.12		0.06		<0.01		ND	
		10	0.14		0.06		<0.01		ND	
	28	8	0.20	0.16	0.05	0.05	<0.01	<0.01	ND	ND
		9	0.14		0.05		<0.01		ND	
		10	0.13		0.05		<0.01		ND	

NA = not analysed (<LOQ for the previous dose group)

ND = not detected (<0.0025 mg/kg)

Table 104 Pydiflumetofen and 2,4,6-TCP residues in bovine muscle and fat (mg/kg)

Dose rate	Pydiflumetofen		2,4,6-TCP	
	Individual	Mean	Individual	Mean
Muscle				
Control	NA	-	NA	-
15 ppm (1×)	NA	-	NA	-
45 ppm (3×)	<0.01, <0.01, <0.01	<0.01	NA	-
150 ppm (10×)	<0.01, <0.01, <0.01	<0.01	<0.01, <0.01, <0.01	<0.01
Perirenal fat				
Control	NA	-	NA	-
15 ppm (1×)	0.01, 0.01, 0.01	0.01	ND, ND, ND	ND
45 ppm (3×)	0.06, 0.04, 0.04	0.05	<0.01, ND, 0.01	<0.01
150 ppm (10×)	0.11, 0.07, 0.05	0.08	<0.01, 0.01, <0.01	<0.01
Mesenterial fat				
Control	NA	-	NA	-
15 ppm (1×)	0.01, 0.01, 0.02	0.01	NA	-
45 ppm (3×)	0.06, 0.04, 0.05	0.05	NA	-
150 ppm (10×)	0.17, 0.07, 0.05	0.10	ND, ND, <0.01	<0.01

Dose rate	Pydiflumetofen		2,4,6-TCP	
	Individual	Mean	Individual	Mean
Subcutaneous fat				
Control	NA	-	NA	-
15 ppm (1×)	<0.01, <0.01, 0.02	<0.01	NA	-
45 ppm (3×)	<0.01, 0.04, 0.03	0.02	NA	-
150 ppm (10×)	0.11, 0.03, <0.01	0.05	<0.01, <0.01, <0.01	<0.01

NA = not analysed (<LOQ for the previous dose group)

ND = not detected (<0.0025 mg/kg)

Table 105 Pydiflumetofen and metabolite residues in bovine liver and kidney (mg/kg)

Dose rate	Pydiflumetofen		2,4,6-TCP		SYN547897		SYN548263	
	Individual	Mean	Individual	Mean	Individual	Mean	Individual	Mean
Liver								
Control	NA	-	NA	-	NA	-	NA	-
15 ppm (1×)	0.01, 0.02, 0.01	0.01	<0.01, <0.01, <0.01	<0.01	0.02, 0.04, 0.06	0.04	NA	-
45 ppm (3×)	0.05, 0.04, 0.03	0.04	0.03, 0.02, 0.03	0.03	0.36, 0.19, 0.10	0.22	ND, ND, ND	ND
150 ppm (10×)	0.09, 0.12, 0.07	0.09	0.06, 0.08, 0.08	0.07	0.56, 0.59, 0.52	0.56	<0.01, <0.01, <0.01	<0.01
Kidney								
Control	NA	-	NA	-	NA	-	NA	-
15 ppm (1×)	NA	-	0.01, 0.01, 0.01	0.01	0.03, 0.06, 0.06	0.05	<0.01, <0.01, <0.01	<0.01
45 ppm (3×)	<0.01, <0.01, <0.01	<0.01	0.05, 0.05, 0.04	0.05	0.24, 0.12, 0.15	0.17	0.02, 0.02, 0.02	0.02
150 ppm (10×)	0.03, 0.02, 0.01	0.02	0.14, 0.21, 0.17	0.17	0.31, 0.58, 0.36	0.41	0.07, 0.10, 0.07	0.08

NA = not analysed (<LOQ for the previous dose group)

ND = not detected (<0.0025 mg/kg)

Residues of pydiflumetofen were found in whole milk, skimmed milk, cream, muscle, liver, kidney and fat samples at the highest dose level (10×), with the highest mean residues found in cream (0.16 mg/kg) and mesenterial fat (0.10 mg/kg). Residues of pydiflumetofen were also found at low levels (<0.01 mg/kg) in fatty matrices (cream and fat samples) and liver at the 1× dose level. Residues of pydiflumetofen reached a plateau concentration in whole milk at approximately 3 days after dosing began.

Residues of 2,4,6-trichlorophenol were found in whole milk, skimmed milk, cream, muscle, liver, kidney and fat at the highest dose level (10×), with the highest mean residues found in kidneys (0.17 mg/kg) followed by dairy commodities and liver (0.06–0.08 mg/kg). Residues of 2,4,6-trichlorophenol were also found in samples from the 1× dose group at low levels (<0.01 mg/kg) in dairy commodities, liver and kidney. Residues of 2,4,6-trichlorophenol reached a plateau concentration in whole milk at approximately 3-5 days after dosing began.

Residues of SYN548264 and SYN508272 were detected in dairy samples from the high dose group (10×) at low levels only (up to 0.01 mg/kg), and were not found in samples from the lower dose groups (1× and 3×), with exception of skimmed milk, where low levels of SYN548264 were detected in the 3× dose group samples (<0.01 mg/kg).

Residues of SYN548263 above 0.01 mg/kg were not seen in liver samples from the high dose group. SYN548263 residues were higher in the kidney samples with mean levels of 0.08 mg/kg detected in the high dose group samples (10×) and <0.01 mg/kg detected in the low dose group samples (1×).

Residues of SYN547897 were the highest found in the liver and kidney samples analysed. In kidney samples, mean residues of SYN547897 were 0.41 mg/kg in the highest dose group (10×) and 0.05 mg/kg in the lowest dose group (1×). In liver samples, mean SYN547897 levels were 0.56 mg/kg in the highest dose group (10×) and 0.04 mg/kg in the lowest dose group (1×).

Study 2

A second residue feeding study in lactating cows was conducted in the UK to measure the residue levels and concentrations of pydiflumetofen metabolite SYN547897 in lactating ruminant tissues (liver and kidney) following daily dosing with pydiflumetofen over a 28 day dosing period (Mills, 2016: 37460).

Eleven lactating Holstein Friesian/Ayrshire cows were split into 4 groups for use in the study. Two control cows (Group 1) received gelatin dosing capsules containing no active substance. Pydiflumetofen was administered orally in gelatin capsules once daily to the remaining three groups of three cows at rates based on average daily dietary intakes. The nominal feeding rates were 15 ppm dry matter (DM) (Group 2, 1× rate), 45 ppm DM (Group 3, 3× rate) and 150 ppm DM (Group 4, 10× rate). The capsules were administered daily for 28 consecutive days (Study Day 1–28). The actual mean daily dose rates achieved ranged between 15.2–15.2 ppm DM for Group 2 animals (1×), 44.1– 44.2 ppm DM for Group 3 animals (3×) and 149.3–150.3 ppm DM for Group 4 animals (10×).

Milk was collected twice daily with the afternoon sampling combined with the next morning's sample. Milk samples were collected from individual cows on Days -1 (pre-dosing), 1, 3, 5, 7, 10, 14, 17, 21, 24 and 28. Skimmed milk and cream samples were produced from milk collected from Animal 9 on Study Days 14 and 28.

All animals, with the exception of Animal 2, were killed within 22–24 hours following the final dose (28th dose) by captive bolt, pithing and exsanguination. Samples of liver, kidney, muscle (composite of loin and hind leg) and fat (perirenal, mesenterial and subcutaneous) were collected from all animals. All liver and kidney samples were stored frozen for analysis. The remaining tissue and milk samples were stored frozen but not required for analysis.

Samples of liver and kidney were analysed for SYN547897 using analytical method GRM061.09A The LOQ for SYN547897 was 0.01 mg/kg for both tissues. The maximum frozen storage intervals (*ca* -20 °C) prior to analysis were 7 days for liver and kidney.

The mean procedural recoveries for SYN547897 from the overall recovery results were 84% ± 15 (n=4) for liver and 89% ± 13 (n=4) for kidney.

Table 106 SYN547897 residues in bovine liver and kidney (mg/kg)

Dose rate	Liver		Kidney	
	Individual	Mean	Individual	Mean
Control	<0.01	-	<0.01	-
15 ppm (1×)	0.04, 0.07, 0.07	0.06	0.09, 0.08, 0.06	0.08
45 ppm (3×)	0.19, 0.23, 0.33	0.25	0.19, 0.14, 0.17	0.17
150 ppm (10×)	0.30, 0.51, 1.0	0.60	0.41, 0.49, 0.41	0.44

In liver samples, mean SYN547897 levels were 0.60 mg/kg in the highest dose group (10×) and 0.06 mg/kg in the lowest dose group (1×). In kidney samples, mean residues of SYN547897 were 0.44 mg/kg in the highest dose group (10×) and 0.08 mg/kg in the lowest dose group (1×). All control samples analysed were found to be <LOQ (0.01 mg/kg).

Laying hens

A feeding study in laying hens was conducted to obtain the magnitude of residue data in egg and tissue samples as a result of feeding pydiflumetofen treated feed to poultry for 28 days (McDonald, 2015: TK0103796).

Fifty four laying hens were split into 5 groups for use in the study. Fourteen control hens (Group 1), which were split into four replicate sub-groups and received untreated feed. Each of the four pydiflumetofen dosing groups were split into 3 replicate sub-groups of 3–4 hens each. The nominal feeding rates were 3 ppm dry matter (DM) (Group 2, 1× rate), 9 ppm DM (Group 3, 3× rate), and 30 ppm DM (Group 4 and Group 5, 10× rate). The treated feed was offered to Groups 2, 3, 4 and 5 *ad libitum* for 28 consecutive days (Study Days 1–28). Following the 28-day feeding phase, hens in one control sub-group and in the three replicate sub-groups in Group 5, began a 14-day depuration phase where they were only fed non-treated feed.

During the dosing phase, whole eggs were collected on Study Days 0, 1, 3, 7, 10, 14, 17 and 24. On days 21, and 28, eggs were collected and the egg yolk and white were separated, and the whites and yolks from each sub-group were composited. In Group 5, eggs were collected during the depuration phase and separated into whites and yolks on days 31, 34, 38 and 42. Samples were stored frozen until analysed. All animals, with exception of those in Group 5 were euthanised on study day 28, within 6 hours following the removal of treated feed. Necropsy for the 10× depuration hens occurred on days 31 (3 days post feed removal), 35 (7 days post feed removal) and 42 (4 days post feed removal). Necropsy for the control depuration hens occurred on day 42. Hens were euthanised by cervical dislocation and decapitation followed by exsanguination. Samples of liver, kidney, muscle (composite of breast and thigh), skin with attached fat, and abdominal plus peritoneal fat were collected from all animals.

Egg and tissue samples were analysed for pydiflumetofen using Method GRM061.06A and analysed for 2,4,6-trichlorophenol (2,4,6-TCP) using Method GRM061.07A by LC-MS/MS. The LOQ for pydiflumetofen and 2,4,6-TCP was 0.01 ppm in all animal matrices. The mean procedural recoveries from the overall recoveries results for the laying hen sample analysis sets were within 70–120% for pydiflumetofen and 2,4,6-TCP. The storage period for treated samples, prior to extraction for analysis, were 182–202 days.

Table 107 Pydiflumetofen and 2,4,6-TCP residues in whole eggs (mg/kg)

Dose rate	Animal	Study Day							
		0	1	3	7	10	14	17	24
Pydiflumetofen									
Control	1	NA	NA	NA	NA	NA	NA	NA	NA
3 ppm (1×)	2A	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	2B	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	2C	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
9 ppm (3×)	3A	ND	<0.01	<0.01	<0.01	<0.01	0.011	0.011	<0.01
	3B	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	3C	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	0.01	<0.01
30 ppm (10×)	4A	ND	<0.01	0.027	0.024	0.021	0.019	0.021	<0.01
	4B	ND	<0.01	0.027	0.024	0.026	0.025	0.019	0.026
	4C	ND	<0.01	0.026	0.023	0.017	0.017	0.027	0.021
30 ppm (Depuration)	5A	ND	<0.01	0.026	0.022	0.023	0.017	0.015	0.016
	5B	ND	ND	0.023	0.024	0.016	0.015	0.017	0.019
	5C	ND	<0.01	0.023	0.025	0.020	0.025	0.019	0.020
	Mean	<0.01	<0.01	0.025	0.024	0.020	0.020	0.020	0.019
2,4,6-TCP									
Control	1	NA	NA	NA	NA	NA	NA	NA	NA
3 ppm (1×)	2A	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	2B	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	2C	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
9 ppm (3×)	3A	ND	ND	<0.01	0.011	0.012	<0.01	<0.01	0.010
	3B	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	3C	ND	<0.01	<0.01	0.011	0.011	0.011	0.013	0.011
	Mean	<0.01	<0.01	0.011	0.011	0.011	0.010	0.011	0.010
30 ppm (10×)	4A	ND	<0.01	0.011	0.027	0.026	0.039	0.036	0.029
	4B	ND	<0.01	<0.01	0.020	0.018	0.021	0.026	0.020
	4C	ND	<0.01	0.013	0.024	0.024	0.028	0.025	0.030
30 ppm (Depuration)	5A	ND	<0.01	0.015	0.037	0.036	0.042	0.038	0.043
	5B	ND	ND	<0.01	0.030	0.034	0.033	0.039	0.039
	5C	ND	<0.01	0.012	0.024	0.028	0.029	0.031	0.029
	Mean	<0.01	<0.01	0.012	0.027	0.028	0.033	0.033	0.032

NA = Not analysed

ND = Not detected

Table 108 Pydiflumetofen and 2,4,6-TCP residues in egg whites (mg/kg)

Dose rate	Animal	Study Day					
		21	28	31	35	38	42
Pydiflumetofen							
Control	1	NA	NA	NA	NA	NA	NA
3 ppm (1×)	2A	<0.01	<0.01	-	-	-	-
	2B	<0.01	<0.01	-	-	-	-
	2C	<0.01	<0.01	-	-	-	-
	Mean	<0.01	<0.01	-	-	-	-
9 ppm (3×)	3A	0.015	<0.01	-	-	-	-
	3B	<0.01	<0.01	-	-	-	-
	3C	<0.01	<0.01	-	-	-	-
	Mean	0.01	<0.01	-	-	-	-

Dose rate	Animal	Study Day					
		21	28	31	35	38	42
30 ppm (10×)	4A	0.038	0.016	-	-	-	-
	4B	0.027	0.028	-	-	-	-
	4C	0.022	0.029	-	-	-	-
30 ppm (Depuration)	5A	0.019	0.034	<0.01	-	-	-
	5B	0.023	0.021	<0.01	ND	-	-
	5C	0.031	0.030	<0.01	ND	ND	ND
	Mean	0.03	0.03	<0.01	<0.01	<0.01	<0.01
2,4,6-TCP							
Control	1	NA	NA	NA	NA	NA	NA
3 ppm (1×)	2A	<0.01	<0.01	-	-	-	-
	2B	<0.01	<0.01	-	-	-	-
	2C	<0.01	<0.01	-	-	-	-
	Mean	<0.01	<0.01	-	-	-	-
9 ppm (3×)	3A	<0.01	<0.01	-	-	-	-
	3B	<0.01	<0.01	-	-	-	-
	3C	<0.01	<0.01	-	-	-	-
	Mean	<0.01	<0.01	-	-	-	-
30 ppm (10×)	4A	<0.01	<0.01	-	-	-	-
	4B	<0.01	<0.01	-	-	-	-
	4C	<0.01	<0.01	-	-	-	-
30 ppm (Depuration)	5A	<0.01	<0.01	ND	-	-	-
	5B	<0.01	<0.01	<0.01	ND	-	-
	5C	<0.01	<0.01	ND	ND	ND	ND
	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

NA = Not analysed

ND = Not detected

Table 109 Pydiflumetofen and 2,4,6-TCP residues in egg yolks (mg/kg)

Dose rate	Animal	Study Day					
		21	28	31	35	38	42
Pydiflumetofen							
Control	1	NA	NA	NA	NA	NA	NA
3 ppm (1×)	2A	<0.01	<0.01	-	-	-	-
	2B	<0.01	<0.01	-	-	-	-
	2C	<0.01	<0.01	-	-	-	-
	Mean	<0.01	<0.01	-	-	-	-
9 ppm (3×)	3A	<0.01	<0.01	-	-	-	-
	3B	<0.01	<0.01	-	-	-	-
	3C	<0.01	<0.01	-	-	-	-
	Mean	<0.01	<0.01	-	-	-	-
30 ppm (10×)	4A	<0.01	<0.01	-	-	-	-
	4B	<0.01	<0.01	-	-	-	-
	4C	<0.01	0.010	-	-	-	-
30 ppm (Depuration)	5A	<0.01	0.012	<0.01	-	-	-
	5B	<0.01	<0.01	<0.01	<0.01	-	-
	5C	0.012	0.011	<0.01	ND	ND	ND
	Mean	0.01	0.01	<0.01	<0.01	<0.01	<0.01
2,4,6-TCP							
Control	1	NA	NA	NA	NA	NA	NA
3 ppm (1×)	2A	<0.01	0.013	-	-	-	-
	2B	<0.01	<0.01	-	-	-	-
	2C	<0.01	<0.01	-	-	-	-
	Mean	<0.01	0.01	-	-	-	-
9 ppm (3×)	3A	0.024	0.026	-	-	-	-
	3B	0.016	0.014	-	-	-	-
	3C	0.013	0.026	-	-	-	-
	Mean	0.02	0.02	-	-	-	-

Dose rate	Animal	Study Day					
		21	28	31	35	38	42
30 ppm (10×)	4A	0.043	0.052	-	-	-	-
	4B	0.049	0.038	-	-	-	-
	4C	0.062	0.062	-	-	-	-
30 ppm (Depuration)	5A	0.058	0.068	0.048	-	-	-
	5B	0.054	0.062	0.052	<0.01	-	-
	5C	0.055	0.041	0.034	<0.01	ND	ND
	Mean	0.05	0.05	0.04	<0.01	<0.01	<0.01

NA = Not analysed

ND = Not detected

Pydiflumetofen and 2,4,6-TCP residues achieved a plateau level in whole eggs early in the course of the study. Residues of 2,4,6-TCP occurred a few days later than the plateau for pydiflumetofen. Eggs collected on days 21 and 28 were separated into whites and yolks. Pydiflumetofen and 2,4,6-TCP residues were eliminated quickly after removal from the diet and return to levels below LOQ within a few days. Pydiflumetofen were slightly higher in egg whites than in egg yolks. 2,4,6-TCP was higher in egg yolks than in egg whites.

Table 110 Pydiflumetofen and 2,4,6-TCP residues in chicken tissues collected following 28 days of dosing (mg/kg)

Dose rate	Pydiflumetofen		2,4,6-TCP	
	Individual	Mean	Individual	Mean
Muscle (Breast + Thigh)				
Control	NA	-	NA	-
3 ppm (1×)	NA	-	NA	-
9 ppm (3×)	NA	-	NA	-
30 ppm (10×)	<0.01, <0.01, <0.01	<0.01	<0.01, <0.01, <0.01	<0.01
Liver				
Control	NA	-	NA	-
3 ppm (1×)	NA	-		
9 ppm (3×)	NA	-		
30 ppm (10×)	<0.01, <0.01, <0.01	<0.01	<0.01, 0.01, <0.01	<0.01
Kidney				
Control	NA	-	NA	-
3 ppm (1×)	ND, ND, ND	<0.01	<0.01, <0.01, <0.01	<0.01
9 ppm (3×)	ND, <0.01, <0.01	<0.01	0.018, 0.013, 0.019	0.02
30 ppm (10×)	<0.01, <0.01, <0.01	<0.01	0.050, 0.037, 0.049	0.05
Skin + Fat				
Control	NA	-	NA	-
3 ppm (1×)	NA	-	NA	-
9 ppm (3×)	NA	-	NA	-
30 ppm (10×)	<0.01, <0.01, <0.01	<0.01	<0.01, <0.01, <0.01	<0.01
Fat (abdominal + peritoneal)				
Control	NA	-	NA	-
3 ppm (1×)	NA	-	NA	-
9 ppm (3×)	NA	-	NA	-
30 ppm (10×)	<0.01, <0.01, <0.01	<0.01	<0.01, <0.01, <0.01	<0.01

NA = not analysed

ND = not detected

Table 111 Pydiflumetofen and 2,4,6-TCP residues in chicken tissues of 30 ppm depuration (mg/kg)

Sample	Pydiflumetofen			2,4,6-TCP		
	Day 31	Day 35	Day 42	Day 31	Day 35	Day 42
Muscle (Breast + Thigh)	ND	ND	ND	ND	ND	ND
Liver	ND	ND	ND	ND	ND	ND
Kidney	ND	ND	ND	ND	ND	ND
Skin + Fat	ND	ND	ND	ND	ND	ND
Fat (abdominal + peritoneal)	ND	ND	ND	ND	ND	ND

ND = not detected

Pydiflumetofen residues did not occur at levels at or above LOQ in any poultry tissue at feeding levels up to 30 ppm DM. Residues of 2,4,6-TCP did not occur at levels at or above LOQ in any poultry tissue at feeding levels up to 30 ppm DM except for kidney. The residues were very low levels and may be found at feeding levels of 9 ppm DM or higher.

APPRAISAL

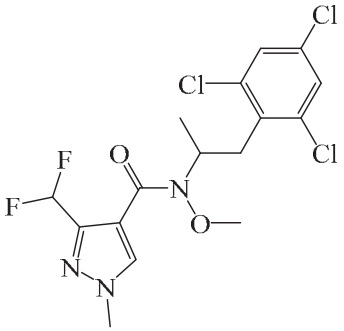
Pydiflumetofen was evaluated for the first time by the Meeting. At the Forty-ninth Session of the CCPR (2017), the compound was scheduled for evaluation as a new compound by the 2018 JMPR.

Pydiflumetofen is a broad-spectrum fungicide of the chemical group of N-methoxy-(phenyl-ethyl)-pyrazole-carboxamide and belongs to the SDHI (Succinate Dehydrogenase Inhibitors) fungicide group. It inhibits succinate dehydrogenase in complex II of fungal mitochondrial respiration.

The Meeting received information on physical and chemical properties, animal and plant metabolism, environment fate, rotational crop residues, analytical methods, storage stability, use pattern, supervised trials, and fate of residues in processing.

N-methoxy-*N*-[(*RS*)-1-methyl-2-(2,4,6-trichlorophenyl)-ethyl]-3-(difluoromethyl)-

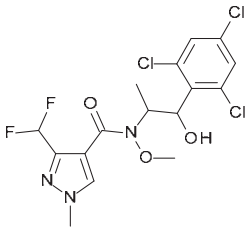
1-methylpyrazole-4-carboxamide



Pydiflumetofen is a 1:1 mixture of the enantiomers.

In this appraisal, the following abbreviated names were used for metabolites.

SYN545547	SYN547891	SYN547897
		MW = 442,7
3-(difluoromethyl)-1-ethyl-N-[1-methyl-2- (2,4,6-trichlorophenyl) ethyl]pyrazole-4- carboxamide	3-(difluoromethyl)-N-methoxy-N-[1-methyl-2-(2,4,6-trichlorophenyl) ethyl]-1H-pyrazole-4-carboxamide	3-(difluoromethyl)-N-methoxy-1-methyl-N-[1-methyl-2-(2,4,6- trichloro-3-hydroxy-phenyl)ethyl] pyrazole-4-carboxamide
SYN547948	SYN548263	SYN548264

		
3-(difluoromethyl)-N-[2-hydroxy-1-methyl-2-(2,4,6-trichlorophenyl) ethyl]-N-methoxy-1- methyl-pyrazole-4-carboxamide	2-[[3-(difluoromethyl)-1-methyl- pyrazole-4-carbonyl]-methoxy-amino] propanoic acid	2-[[3-(difluoromethyl)-1-methyl-pyrazole-4-carbonyl]amino] propanoic acid
SYN508272	NOA449410	2,4,6-TCP
		MW=197.45
3-(difluoromethyl)-1-methyl-pyrazole-4- carboxamide	3-(difluoromethyl)-1-methyl- pyrazole-4-carboxylic acid	2,4,6-trichlorophenol
Hydroxylated pydiflumetofen		
Hydroxylated N-methoxy-N-[1-methyl-2-(2,4,6-trichlorophenyl)-ethyl]-3-(difluoromethyl)-1-methylpyrazole-4- carboxamide		

Physical and chemical properties

Pydiflumetofen has a higher solubility in organic solvents in comparison to water and is not volatile. Pydiflumetofen was shown to be hydrolytically and photolytically stable.

Plant metabolism

The Meeting received plant metabolism studies on tomato, wheat and oilseed rape with pydiflumetofen labelled with ^{14}C at two different rings ([phenyl-U-^{14}C] and [pyrazole-5-^{14}C]).

In a tomato metabolism study, [^{14}C]-pydiflumetofen was applied to tomato plants grown in a glasshouse with two foliar spray applications at total rate of 0.40 kg ai/ha. As a separate study, [^{14}C]-pydiflumetofen was applied as a single application direct to soil containing tomato plants grown in a glasshouse at a rate of 20 mg ai/plant. Residue levels (0.64 md eq/kg) were highest in the foliar treated fruit harvested 14 DALA. Surface wash with acetonitrile extracted ≥ 89% TRR in the foliar experiment. Total extractability was ≥ 98% TRR in all commodities extracted with acetonitrile/water.

In the foliar spray experiment, the highest residues of pydiflumetofen were detected in 14 DALA fruit (0.59–0.61 mg/kg, 92–97% TRR). Metabolism was limited and metabolites identified were SYN545547 and SYN547891. Residues of SYN545547 and SYN547891 accounted for a maximum of 3.6% TRR and 1.6% TRR respectively.

In the soil treated experiment, total radioactive residues in the fruit were low (≤ 0.013 md eq/kg). The principal component detected in fruit harvested 103 days after treatment (DAT) was pydiflumetofen accounting for 4.1% TRR (0.001 mg/kg). Unidentified components in fruit accounted for 89% TRR (0.008 md eq/kg) with no individual component > 12% TRR (0.002 md eq/kg).

In a wheat metabolism study, [^{14}C]-pydiflumetofen was applied twice to spring wheat as spray applications at a total rate of 0.25 kg ai/ha. Residues increased from forage to straw to a maximum of 1.5 md eq/kg; residues in grain were the lowest of all commodities with residues ≤ 0.057 md eq/kg. Extractability was ≥85% TRR in all commodities extracted with acetonitrile/water (80:20).

The highest residues of pydiflumetofen were detected in straw (1.1–1.2 mg/kg, ≥76% TRR), and the lowest in grain (0.030–0.046 mg/kg, ≥82% TRR) at 50 days after last application (DALA). Metabolism was limited and metabolites identified were the de-alkylated molecules, SYN545547 and SYN547891. Residues of SYN545547 accounted for ≤3.9% TRR with the largest residue detected in straw (0.059 md eq/kg, 3.9% TRR). Residues of SYN547891 accounted for ≤8.3% TRR with the highest residue in straw (0.065 md eq/kg, 4.3% TRR).

In an oilseed rape metabolism study, [^{14}C]-pydiflumetofen was applied to oilseed rape as a spray application at a rate of 0.15 kg ai/ha. Residues in trash were 0.061–0.062 md eq/kg and residues in seed were 0.019–0.020 md eq/kg at 62 DAT. Extractability was 72–75% TRR in seed extracted with acetonitrile/water (80:20) plus hexane and 76–81% TRR in trash extracted with acetonitrile /water (80:20).

The highest residues of pydiflumetofen were detected in trash (0.018–0.032 mg/kg, ≥ 30%TRR); lower residues were detected in seed (0.007–0.012 mg/kg, ≥ 39% TRR). Metabolism was limited. Low residues of SYN545547 (≤3.7% TRR, ≤0.002 md eq/kg) and SYN547891 (≤ 5.1% TRR, ≤0.003 md eq/kg) were detected in trash. Low residues of SYN545547 (≤6.1% TRR, ≤ 0.001 md eq/kg) and SYN547891 (≤2.7% TRR, ≤0.001 md eq/kg) were detected in seed. Unknown residues of the [pyrazole-^{14}C] label (35% TRR, 0.022 md eq/kg) remained in trash, but they were comprised of 10 individual components and none individually exceeding 8.4% TRR (0.005 md eq/kg).

In summary, pydiflumetofen was the major component of the residues found in wheat, tomato and oilseed rape. SYN545547 was formed by reduction of the parent molecule and SYN547891 was formed by demethylation of the pyrazole ring but they were not present as significant residues in plants.

Animal metabolism

The Meeting received farm animal metabolism studies with pydiflumetofen on lactating goat and laying hens. The metabolism and distribution of pydiflumetofen in farm animals were investigated using the [phenyl-U-^{14}C] and [pyrazole -5-^{14}C]-pydiflumetofen.

Metabolism in rats was summarised and evaluated by the WHO panel of the JMPR in 2018.

Lactating goats were orally dosed with radiolabelled pydiflumetofen daily for 7 consecutive days at a nominal rate of 100 ppm dietary dry matter intake. The radioactive balance for goats was greater than 94% with the majority of the radioactivity excreted in the urine (30–32% AR) and faeces (46–53% AR).

Following the administration of [^{14}C]-pydiflumetofen, TRRs were 7.0–8.8 md eq/kg in liver, 1.7–2.3 md eq/kg in kidney, 0.10–0.14 md eq/kg in muscle and 0.22–0.28 md eq/kg in fat. Residues in milk achieved a plateau concentration of approximately 0.091–0.13 md eq/kg after 2–6 days of dosing. Following solvent extraction with aqueous acetonitrile and hexane for milk and fat, and with aqueous acetonitrile for liver, kidney and muscle, residue extractabilities were generally high (≥ 83% TRR) for milk, kidney, muscle and fat. Residue extractability was lower in liver (47–50% TRR). Where appropriate fractions were also subject to organic solvent/water partition and β-glucuronidase enzyme hydrolysis procedures prior to chromatographic analysis.

Pydiflumetofen %TRRs were greatest in fat (67–74% TRR, 0.15–0.21 mg/kg), muscle (13–24% TRR, 0.018–0.025 mg/kg), milk (8.7–16% TRR, 0.011–0.019 mg/kg) and liver (2.0–8.2% TRR, 0.18–0.57 mg/kg) with much smaller percentage TRRs being found in kidney (0.5–0.8% TRR, 0.011–0.014 mg/kg).

SYN548264 (N-desmethoxy carboxylic acid) occurred at its highest %TRR in milk (29% TRR, 0.038 md eq/kg, found in its free form). Much lower %TRRs were detected in kidney (0.8% TRR, 0.019 md eq/kg, conjugated form only) and in muscle (0.6% TRR, 0.001 md eq/kg, found in its free form only). It was not detected in liver or fat.

SYN508272 (amide) occurred at its highest %TRRs in muscle (18% TRR, 0.024 md eq/kg) and milk (11% TRR, 0.014 md eq/kg) both were found in the free form only. Much lower %TRRs were detected in kidney (1.5% TRR, 0.036 md eq/kg; found in both its free and conjugated forms) and in fat (1.0% TRR, 0.003 md eq/kg, in its free form only). It was not detected in liver.

SYN548263 (carboxylic acid) occurred at its highest %TRRs in kidney (17% TRR, 0.39 md eq/kg, in predominantly its conjugated form) and milk (14% TRR, 0.019 md eq/kg, found in its free form only). Much lower %TRRs were detected in muscle (4.9% TRR, 0.007 md eq/kg) and in fat (4.3% TRR, 0.012 md eq/kg), where it was found in the free form only. It was not detected in liver.

NOA449410 (carboxylic acid) occurred at its highest %TRR in kidney (12% TRR, 0.28 md eq/kg, in predominantly its conjugated form). Lower %TRRs were detected in liver (2.9% TRR, 0.25 md eq/kg, found in both the free and conjugated forms) and in muscle and milk (3.6% TRR and 2.6% TRR respectively, ≤0.005 md eq/kg) in only the free form. It was not detected in fat.

2,4,6-TCP occurred at its highest %TRRs in milk (43% TRR, 0.052 md eq/kg) and muscle (9.0% TRR, 0.009 md eq/kg). In both milk and muscle the residue was found predominantly in its sulphate ester conjugated form of the metabolite. Levels of this metabolite were much lower in kidney (1.2% TRR, 0.021 md eq/kg) and liver (0.5% TRR, 0.037 md eq/kg), where it was only found in the conjugated form in both tissues. It was not detected in fat.

Other identified metabolites, hydroxylated metabolite of pydiflumetofen, SYN545547, SYN547948, SYN547897 and SYN547891, were observed (≤ 11% TRR).

Aliquots of liver and kidney PES were subject to protease enzyme hydrolysis which released significant proportions of the residues (liver: 40–46% TRR; kidney: 9.3–15% TRR) into the resulting hydrolysate.

Laying hens were orally dosed with radiolabelled pydiflumetofen daily for 14 days at a dose level of 30 ppm dietary dry matter intake. The radioactive balance for all hens was greater than 81% with the majority of the radioactivity recovered in excreta.

Samples were extracted with solvents (fat: hexane and aqueous acetonitrile, liver, egg yolk, egg white and muscle: aqueous acetonitrile). Good extractability was achieved for the eggs, muscle and fat (≥ 81% TRR). Extractability of liver was lower (≥ 52% TRR). Where appropriate, these fractions were subject to organic solvent/water partition and β-glucuronidase enzyme hydrolysis procedures prior to chromatographic analysis. Mean residues in egg whites achieved a plateau concentration of approximately 0.062–0.064 md eq/kg after 6–7 days of dosing. Mean residues in egg yolks achieved a plateau concentration of approximately 0.34 md eq/kg (phenyl) and 0.12 md eq/kg (pyrazole) after 10 and 7 days of dosing, respectively.

Pydiflumetofen %TRRs were greatest in egg white (27–47% TRR, 0.014–0.025 mg/kg) and fat (17–31% TRR, 0.010–0.017 mg/kg) with much smaller %TRRs being found in muscle (4.7–8.7% TRR, 0.001–0.002 mg/kg), egg yolk (3.0–11% TRR, 0.011–0.012 mg/kg) and liver (0.5–5.3% TRR, 0.001–0.021 mg/kg).

2,4,6-TCP occurred at its highest %TRRs in egg yolk (68% TRR, 0.24 md eq/kg), egg white (15% TRR, 0.008 md eq/kg), muscle (48% TRR, 0.013 md eq/kg) and fat (29% TRR, 0.030 md eq/kg). In eggs and all tissues the residue of this metabolite was found predominantly in its sulphate ester conjugated form.

SYN508272 (amide) occurred at its highest %TRRs in muscle (46% TRR, 0.010 md eq/kg, the free form), egg white (34% TRR, 0.018 md eq/kg, the free form) and fat (9.6% TRR, 0.003 md eq/kg, the free form). Much lower %TRRs were detected in egg yolk (7.2% TRR, 0.008 md eq/kg, the free and conjugated forms) and liver (2.4% TRR, 0.005 md eq/kg, the free and conjugated forms).

NOA449410 (carboxylic acid) occurred at its highest %TRR in egg white (15% TRR, 0.008 md eq/kg, the free form). Lower %TRRs were detected in egg yolk (6.6% TRR, 0.007 md eq/kg, the free and conjugated forms), in fat (3.1% TRR, 0.001 md eq/kg, the free form) and was not detected in liver and muscle.

Other identified metabolites, SYN545547, SYN547948, SYN547897 and SYN547891, were observed (≤ 6.7% TRR).

Separate aliquots of liver and egg yolk PES were subject to protease enzyme hydrolysis which released significant proportions of the residues (liver: 32–46% TRR; egg yolk: 6.1–11% TRR) into the resulting hydrolysate.

In summary, pydiflumetofen was detected in all goat and hen commodities. The most abundant metabolite detected in milk, egg yolk and hen muscle was 2,4,6-TCP. In the goat, SYN548263 and SYN548264 occurred in milk, kidney and muscle. SYN548264 was further metabolised to form SYN508272 and NOA449410. In the hen, the intermediate metabolites SYN548263 and SYN548264 were not observed but SYN508272 and NOA449410 were identified. Protease hydrolysates of PES comprised a complex mixture of unknown and highly polar metabolites in liver (goat and hen) and kidney (goat).

Environmental fate in water and soil

The Meeting received information on hydrolysis, soil photolysis and soil degradation.

In the hydrolytic degradation study, pydiflumetofen was hydrolytically stable at pH 4, 7 and 9 after incubation at 50 °C for 5 days (> 92% of applied radioactivity was recovered as unchanged pydiflumetofen).

In the aqueous photolysis study, pydiflumetofen degraded slowly in sterile buffer (pH 7) with 73–79% of applied radioactivity recovered after incubation at 25 °C for 30 days.

In the soil photolysis study, the DT_{50} of pydiflumetofen was 77 days in dry soils and 197 days in moist soils.

Hydrolysis and photolysis are not considered significant routes of degradation for pydiflumetofen.

In the soil degradation studies, pydiflumetofen was found to remain mostly in the top soil layer (0–10 cm) during soil dissipation field trials (6 different soils) of duration up to 24 months. The DT_{50} of pydiflumetofen was 398–2380 days and the DT_{90} could not be reached within one year after application to bare soil (> 40 months in all trials). Pydiflumetofen is persistent in soil.

Rotational crop studies

The Meeting received a confined rotational crop study with ^{14}C-labelled pydiflumetofen ([phenyl-U-^{14}C] and [pyrazole-5-^{14}C]) and field rotational crop studies.

In a confined rotational crop study, rotational crops (lettuce, wheat and turnip) were sown at 30, 120 and 270 days after a single spray treatment (DAT) of [^{14}C]-pydiflumetofen at a rate of 0.40 kg ai/ha to a sandy loam soil.

Extractability was 83–97% TRR for all analysed commodities. Pydiflumetofen represented the major portion of the residue taken up into rotated wheat, lettuce and turnip commodities up to a maximum of 78% TRR. Maximum residues of pydiflumetofen were detected in 120 DAT wheat straw (29% TRR, 0.063 mg/kg). Uptake of radioactive residues into grain and turnip tubers was low accounting for ≤ 0.008 md eq/kg.

Metabolites identified were SYN5457891 (≤ 0.012 md eq/kg) and SYN545547 (≤ 0.005 md eq/kg) detected in all commodities.

The results show that the metabolism of pydiflumetofen in rotated crops was similar for all crop types. Comparison with primary crop metabolism studies shows that the pathway in rotational crops is consistent with that in primary crops, though the magnitude of residues in rotational crops is lower.

In field rotational crop studies in Europe and the USA, pydiflumetofen SC formulation was applied to bare ground at 0.40–0.50 kg ai/ha (Europe: 0.50 × 1 kg ai/ha, USA: 0.20 × 2 kg ai/ha). The trials were established for each of three rotational crop types (Europe: spinach, carrot and spring barley, USA: spinach or lettuce, radish, and wheat) at several rotational crop plantback intervals (Europe: 30, 60 and 365 days, USA: 30, 60, 90, and 150 days).

Residues of pydiflumetofen were all below 0.02 mg/kg in spinach, lettuce, carrot, radish, cereal whole plant and grains.

Residues of pydiflumetofen were found in spring barley straw, wheat hay and wheat straw. For spring barley straw in the European studies, residues of pydiflumetofen were 0.02–0.06 mg/kg at a 30 day PBI and 0.02–0.09 mg/kg at a 60 day PBI. For wheat straw and hay in the USA study, a peak in residues is apparent at 90 day PBI (0.011–0.12 mg/kg) with a subsequent decline in residues at 150 day PBI (< 0.01–0.067 mg/kg).

The Meeting noted that residues of pydiflumetofen may be taken up by rotational crops. The active substance is very persistent in soil (up to 2380 days DT_{50}) and accumulation following subsequent years of treatment is expected. The submitted field rotational crop study only approximates the annual application rates of the uses evaluated. The Meeting concluded that the information available does not allow estimation of pydiflumetofen residues in rotational crops, especially in view of expected plateau soil concentrations being significantly higher than the rate applied in the field rotational crop study available.

Methods of analysis

The Meeting received descriptions and validation data for analytical methods for residue of pydiflumetofen in plant commodities and for residues of pydiflumetofen, 2,4,6-TCP, SYN548264, SYN548263, SYN508272 and SYN547897 in animal commodities.

In the method for determination of pydiflumetofen in plant, homogenised samples were extracted with acetonitrile: water (80:20 v/v), with clean up via a solid phase extraction. Residues were determined by LC-MS/MS. The method of analysis was validated at various fortification levels with an LOQ of 0.01 mg/kg for pydiflumetofen. A QuEChERS method was also validated for pydiflumetofen residues in plant matrices with an LOQ of 0.01 mg/kg.

In the methods for determination of pydiflumetofen and 2,4,6-TCP in animal commodities, samples were homogenized with acetonitrile: water (80:20 v/v). The fat samples were dissolved into hexane and partitioned into acetonitrile: water (80:20 v/v). An aliquot for the analysis of pydiflumetofen was directly diluted with 0.1% ultra-pure water. A separate aliquot for the analysis of 2,4,6-TCP was hydrolysed by β-glucuronidase and cleaned up with a solid phase extraction. Residues of both compounds were determined by LC-MS/MS. The method of analysis was validated with LOQs of 0.01 mg/kg for pydiflumetofen and 2,4,6-TCP. QuEChERS method was also validated for pydiflumetofen residue in animal commodities with an LOQ of 0.01 mg/kg.

In the method for determination of SYN548264 and SYN508272 in milk, samples were shaken with acetonitrile and diluted with aqueous acetonitrile. Residues of both compounds were determined by LC-MS/MS. The method of analysis was validated with LOQs of 0.01 mg/kg for SYN548264 and SYN508272.

In the method for determination of SYN547897 and SYN548263 in kidney and liver, homogenised samples were extracted with acetonitrile: water (80:20 v/v) and filtered through a C18 cartridge. An aliquot was hydrolysed by β-glucuronidase and cleaned up with a solid phase extraction. Residues of both compounds were determined by LC-MS/MS. The method of analysis was validated with LOQs of 0.01 mg/kg for SYN547897 and SYN548263.

Stability of residues in stored analytical samples

The Meeting received information on the freezer storage stability of pydiflumetofen in plant (lettuce, orange, oilseed rape seed, potato, beans without pods, wheat grain and straw), processed commodities of plants (corn, soya bean, grape and apple fractions) and animal commodities (bovine muscle, liver, fat, milk and eggs).

Storage stability results indicate that pydiflumetofen residues were stable at approximately -18 °C for at least 23 months in lettuce (high water), orange (high acid), oilseed rape seed (high oil), potato (high starch), beans without pods (high protein), wheat grain (high starch) and straw, at approximately -20 °C for at least 12 months in maize (flour, meal and oil), soya bean (flour, milk, oil), grape (raisin) and apple fractions (dried fruit and juice), and at approximately -20 °C for at least 24 months in bovine muscle, liver, fat, milk and eggs.

The Meeting also received information on the freezer storage stability of SYN508272 and SYN548264 in milk, SYN547897 in liver, SYN547897 and SYN548263 in kidney, and 2,4,6-TCP in bovine muscle, liver, kidney, fat, whole milk and egg. The residues of SYN508272, SYN548264, SYN547897, SYN548263 and 2,4,6-TCP were stable at approximately -18 °C for at least 12 months in animal commodities except SYN547897 in liver (up to 9 months).

The demonstrated periods of storage stability cover the sample storage intervals in the residue trials.

Definition of the residue

In plant metabolism studies, parent pydiflumetofen was a major component (30–97% TRR) for foliar treatment in wheat, tomato and oilseed rape. SYN545547 and SYN547891 were identified at 0.001–0.049 md eq/kg (< 10% TRR) in wheat, tomato and oilseed rape. No other individual metabolite was present in the edible plant parts at a level greater than 10% TRR.

The residue profile was similar in rotational crops, for which the only identified compounds were pydiflumetofen, SYN545547, and SYN547891. SYN547891 occurred at > 10%TRR in some commodities; however, in such cases, the concentration levels were < 0.005 md eq/kg.

The Meeting decided that the suitable analyte for enforcement purposes is parent pydiflumetofen in plant commodities.

Although SYN545547 and SYN547891 share a high degree of structural similarity to parent pydiflumetofen and may be toxicologically relevant, the Meeting concluded that dietary exposure to these compounds is likely to be insignificant in comparison to that of the parent residue. Therefore, the Meeting decided that the suitable analyte for dietary risk assessment is pydiflumetofen in plant commodities.

In animal metabolism studies, pydiflumetofen was the principal component of the residue in fat (goat: 67–74% TRR, 0.15–0.21 mg/kg, hen: 17–31% TRR, 0.010–0.017 mg/kg) and egg white (27–47% TRR, 0.014–0.025 mg/kg), a major residue in goat muscle (13–24% TRR, 0.018–0.025 mg/kg), milk (8.7–16% TRR, 0.011–0.019 mg/kg) and goat liver (2.0–8.2% TRR, 0.18–0.57 mg/kg).

The Meeting decided that pydiflumetofen is a suitable analyte for enforcement purposes. Analytical methods are available to determine residues of pydiflumetofen in animal commodities.

In deciding if additional compounds should be included in the residue definition for risk assessment, the Meeting considered the likely occurrence of the compounds and the toxicological properties of those compounds.

2,4,6-TCP (free + conjugated form) was the principal component of the residue in egg yolk (68% TRR, 0.24 md eq/kg), milk (43% TRR, 0.052 md eq/kg) and fat (29% TRR, 0.030 md eq/kg), a major residue in muscle (goat: 9.0% TRR, 0.009 mg/kg, hen: 48% TRR, 0.013 md eq/kg) and egg white (15% TRR, 0.008 md eq/kg).

SYN508272 (free form) was a major residue in milk (11% TRR, 0.014 md eq/kg), muscle (goat: 18% TRR, 0.024 md eq/kg, hen: 46% TRR, 0.010 md eq/kg) and egg white (34% TRR, 0.018 md eq/kg).

SYN548263 was a major component of the residue in milk (free form: 14% TRR, 0.019 md eq/kg) and goat kidney (free + conjugated form: 17% TRR, 0.39 md eq/kg).

SYN548264 (free form) was a major residue in milk (29% TRR, 0.038 md eq/kg).

The other major residue was NOA449410 (free + conjugated form, goat kidney: 12% TRR, 0.28 md eq/kg, egg white: 15% TRR, 0.008 md eq/kg).

In the lactating dairy cow feeding studies, SYN548264 and SYN508272 residues (free form) in milk were < 0.01 mg/kg except for one cow (0.01 mg/kg) at the 150 ppm dose rate. SYN548263 residue (free + conjugated form) were found in kidney (0.02 mg/kg at 45 ppm and 0.08 mg/kg at 150 ppm) but < 0.01 mg/kg in liver. SYN547897 residue (free + conjugated form) were found in liver (0.06 mg/kg at 15 ppm and 0.60 mg/kg at 150 ppm) and kidney (0.08 mg/kg at 15 ppm and 0.44 mg/kg at 150 ppm).

The Meeting concluded that the metabolites 2,4,6-TCP, SYN547897, SYN548263, SYN548264, SYN508272 and NOA449410 may potentially contribute to the dietary exposure. All compounds are covered by the toxicological reference values for parent pydiflumetofen.

2,4,6-TCP (incl. conjugates) was found in all animal commodities. Therefore, the Meeting decided to include 2,4,6-TCP (including conjugates) in the residue definition for dietary exposure, and noted that this compound is not specific to the use of pydiflumetofen and can be formed in animal commodities from other sources.

SYN547897 was identified only in minor proportions in liver and kidney in the goat metabolism study. However, in the dairy cattle feeding study, it represented the major residue in liver and kidney, found at levels 4 to 10 times higher than parent. The Meeting concluded that SYN547897 (including conjugates) should be included in the residue definition for dietary exposure purposes specifically for mammalian liver and kidney.

SYN548263 was identified as a major metabolite in milk and kidney in the lactating goat metabolism study. In milk, its proportion was major (14% TRR), however absolute levels were low (0.019 md eq/kg), following administration of 100 ppm in the diet. In view of the estimated dietary burden for dairy cattle of 1.2 ppm, the contribution of SYN548263 to the dietary exposure via milk was considered insignificant.

In kidney, the dairy cattle feeding study showed quantifiable residues of SYN548263 (incl. conjugates) at feeding levels of 45 ppm or higher. However, the Meeting noted that the metabolite SYN547897 was present at amounts approximately 10 times higher and concluded, that SYN548263 does not contribute significantly to the total residue and toxicological burden in kidney.

SYN548264 was a major residue in milk (up to 29% TRR). However, its presence in milk was not observed in the dairy cattle feeding study following the administration of parent pydiflumetofen up to 150 ppm. The Meeting concluded that SYN548264 does not contribute significantly to the dietary exposure via milk.

SYN508272 was a major residue in milk but found at < 0.01–0.01 mg/kg in the dairy cattle feeding study. SYN508272 was also found as the major residue in muscle (18% TRR in goat and 46% TRR in hen) and egg white (34% TRR), however the levels were low (0.01–0.02 md eq/kg). The toxicity of SYN508272 is considered to be less than that of parent pydiflumetofen. The Meeting concluded that SYN508272 does not contribute significantly to the total residue and toxicological burden in muscle and egg.

NOA44910 (incl. conjugates) was a major residue in goat kidney (12% TRR) and egg white (15% TRR) but the level in egg white was < 0.01 mg/kg. In view of the dietary burden for dairy cattle as above, the expected contribution of NOA449410 to the dietary exposure was not significant.

The Meeting decided that for animal commodities, except mammalian liver and kidney, the definition for the estimation of the dietary exposure is the sum of pydiflumetofen and 2,4,6-TCP (incl. conjugates), expressed as pydiflumetofen. For mammalian liver and kidney, the definition for the estimation of the dietary exposure is the sum of pydiflumetofen, SYN547897 (incl. conjugates) and 2,4,6-TCP (incl. conjugates), expressed as pydiflumetofen.

The octanol/water coefficient (log Pow) of pydiflumetofen is 3.8. Pydiflumetofen in fat is 10 times higher than in muscle, and, in cream, more than 10 times higher than in skimmed milk. The Meeting considered the residue of pydiflumetofen to be fat-soluble.

The Meeting recommended the following residue definition:

Definition of the residue for compliance with the MRL for both plant and animal commodities: *Pydiflumetofen*

Definition of the residue for animal commodities other than mammalian liver and kidney for dietary risk assessment: *sum of pydiflumetofen and 2,4,6-trichlorophenol (2,4,6-TCP) and its conjugates, expressed as pydiflumetofen*

Definition of the residue for mammalian liver and kidney for dietary risk assessment: *sum of pydiflumetofen, 2,4,6-trichlorophenol (2,4,6-TCP) and its conjugates, and 3-(difluoromethyl)-N-methoxy-1-methyl-N-[1-methyl-2-(2,4,6-trichloro-3-hydroxy-phenyl) ethyl]pyrazole-4-carboxamide (SYN547897) and its conjugates, expressed as pydiflumetofen*

The residue is fat-soluble

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for foliar application of pydiflumetofen on grapes, cucumber, summer squash, cantaloupe, tomato, pepper, head lettuces, leaf lettuce, spinach, dry beans, soya bean, dry peas, potato, celery, wheat, barley, oats, maize, sweet corn, oilseed rape and peanut. Residue trials were conducted in Canada and the USA.

Labels from Argentina, Canada and the USA were available.

The Meeting concluded that accumulation of pydiflumetofen in soil and uptake of residues into rotational crops may significantly contribute to the terminal residue in food and feed commodities. The Meeting decided that residues found in annual crops following treatment as a primary crop (cucumber, summer squash, melon, tomato, pepper, head lettuces, leaf lettuce, spinach, dry beans, soya bean (dry), dry peas, potato, celery, wheat, barley, oats, maize, sweet corn, oilseed rape, peanut and pea vines) may substantially underestimate the potential residues and thus impact the estimation of maximum residue levels, STMRs and HRs.

For these crops, maximum residue levels, STMRs and HRs will be estimated by a future Meeting when additional information addressing the contribution of the additional uptake from soil become available.

Berries and other small fruits

Small fruit vine climbing

Grapes

Data were available from supervised trials on grapes in the USA.

The GAP of the USA for grape and small fruits vine climbing (Crop Subgroup 13–07F), except fuzzy kiwifruit allows foliar applications of up to 0.20 kg ai/ha at a maximum annual rate of 0.40 kg ai/ha with a PHI of 14 days.

Pydiflumetofen residues in grapes from independent trials in the USA matching USA GAP were (n=11): < 0.01, 0.053, 0.16 (2), 0.20, 0.29, 0.37, 0.39, 0.50, 0.61 and 0.77 mg/kg.

The Meeting noted that the GAP of the USA is for grape and small fruits vine climbing except fuzzy kiwifruit and grape is the representative commodity for subgroup of small fruit vine climbing in the Codex classification.

Based on the residues in grapes from trials in the USA, the Meeting estimated a maximum residue level of 1.5 mg/kg, a STMR value of 0.29 mg/kg and a HR value of 0.85 mg/kg (based on the highest residue of replicate samples) for pydiflumetofen in the subgroup of small fruit vine climbing.

Fruiting vegetables, Cucurbits

The GAP for cucurbit vegetables, crop group 9 of the USA is two foliar applications of up to 0.13 kg ai/ha at a maximum annual rate of 0.25 kg ai/ha with a PHI of 0 days.

Fruiting vegetables, Cucurbits – Cucumbers and Summer squashes

Cucumber

Data were available from supervised trials on cucumber in the USA.

Pydiflumetofen residues in cucumber from independent trials in the USA matching USA GAP were (n=10): 0.11 (3), 0.12, 0.14, 0.16 and 0.19 mg/kg (outdoor) and 0.11, 0.23 and 0.26 mg/kg (indoor).

Squash, summer

Data were available from supervised trials on summer squash in the USA.

Pydiflumetofen residues in summer squash from independent outdoor trials in the USA matching GAP were (n=5): 0.056, 0.061, 0.10, 0.16 and 0.18 mg/kg.

Fruiting vegetables, Cucurbits – Melons, Pumpkins and Winter Squashes

Melon

Data were available from supervised trials on cantaloupe in the USA.

Pydiflumetofen residues in cantaloupe from independent outdoor trials in the USA matching GAP were (n=6): 0.067, 0.078, 0.11, 0.15, 0.16 and 0.17 mg/kg.

To consider a maximum residue level for a group, residues in individual crops should be similar (e.g. medians should not differ by more than 5×). The Meeting agreed to estimate a maximum residue level for the group of fruiting vegetables, cucurbits. In considering whether to combine data to estimate a maximum residue level, the Meeting recognized that the residue populations from trials on cucumber, summer squash and cantaloupe were not different according to statistical test (Kruskal-Wallis H-test). Therefore the Meeting decided to combine the data from cucumber, summer squash and cantaloupe to estimate a maximum residue level for fruiting vegetables, cucurbits.

The combined pydiflumetofen residues in cucumber, summer squash and cantaloupe were in rank order (n=21): 0.056, 0.061, 0.067, 0.078, 0.10, 0.11 (5), 0.12, 0.14, 0.15, 0.16 (3), 0.17, 0.18, 0.19, 0.23 and 0.26 mg/kg.

As indicated no recommendation could be made for fruiting vegetables, cucurbits.

Fruiting vegetables, other than Cucurbits

The GAP for fruiting vegetables, crop group 8–10 of the USA is two foliar applications of up to 0.13 kg ai/ha at a maximum annual rate of 0.25 kg ai/ha with a PHI of 0 days.

Tomato

Data were available from supervised trials on tomatoes in the USA.

Pydiflumetofen residues in tomato and cherry tomato from independent outdoor trials in the USA matching USA GAP were (n=10+2): 0.030, 0.043, 0.075, 0.077, 0.082, 0.083, 0.11, 0.13, 0.16 and 0.23 mg/kg for tomato and 0.20 and 0.27 mg/kg for cherry tomato.

Peppers

Data were available from supervised trials on peppers in the USA.

Pydiflumetofen residues in bell pepper and non-bell pepper from independent outdoor trials in the USA matching USA GAP were (n=6+3): 0.062, 0.076, 0.081, 0.17, 0.26 and 0.37 mg/kg for bell pepper and 0.088, 0.14 and 0.26 mg/kg for non-bell pepper.

To consider a maximum residue level for a group, residues in individual crops should be similar (e.g. medians should not differ by more than 5×). The Meeting agreed to estimate a maximum residue level for the group of fruiting vegetables, other than cucurbits. In considering whether to combine data to estimate a maximum residue level, the Meeting recognized that the residue populations from trials on tomatoes and peppers were not different according to statistical test (Mann-Whitney U-test). Therefore the Meeting decided to combine the data from tomatoes and peppers to estimate a maximum residue level for fruiting vegetables, other than cucurbits.

The combined pydiflumetofen residues in tomatoes and peppers were in rank order (n=21): 0.030, 0.043, 0.062, 0.075, 0.076, 0.077, 0.081, 0.082, 0.083, 0.088, 0.11, 0.13, 0.14, 0.16, 0.17, 0.20, 0.23, 0.26 (2), 0.27 and 0.37 mg/kg.

As indicated no recommendation could be made for fruiting vegetables, other than cucurbits.

Leafy vegetables (including Brassica leafy vegetables)

Leafy greens

The GAP for leafy greens, crop subgroup 4-16A of the USA is two foliar applications of up to 0.20 kg ai/ha at a maximum annual rate of 0.40 kg ai/ha with a PHI of 0 days.

Lettuce, Head

Data were available from supervised trials on head lettuce in the USA.

Pydiflumetofen residues in head lettuce with wrapper leaves from independent trials in the USA matching GAP were (n=8): 0.51, 0.78, 1.2, 2.3, 2.4, 2.6, 3.0 and 4.5 mg/kg.

Lettuce, Leaf

Data were available from supervised trials on leaf lettuce in the USA.

Pydiflumetofen residues in leaf lettuce from independent trials in the USA matching GAP were (n=8): 1.7, 3.5, 4.4, 5.5, 7.7, 9.7, 11 and 12 mg/kg.

Spinach

Data were available from supervised trials on spinach in the USA.

Pydiflumetofen residues in spinach from independent trials in the USA matching GAP were (n=8): 7.5, 9.2, 9.7, 12, 13 (2), 14 and 16 mg/kg.

To consider a maximum residue level for a group, residues in individual crops should be similar (e.g. medians should not differ by more than 5×). The Meeting agreed to estimate a maximum residue level for the subgroup of leafy greens. In considering whether to combine data to estimate a maximum residue level, the Meeting recognized that the residue populations from trials on head lettuce, leaf lettuce and spinach were significantly different according to statistical test (Kruskal-Wallis H-test). Therefore the Meeting decided to use the dataset from spinach leading to the highest maximum residue level for leafy greens.

As indicated no recommendation could be made for leafy greens.

Pulses

The GAP for dried shelled peas and beans (except soya bean) of Canada and the USA is two foliar applications of up to 0.20 kg ai/ha with an application interval of 14 days, at a maximum annual rate of 0.40 kg ai/ha with a PHI of 14 days.

The GAP for soya bean of Canada and the USA is two foliar applications of up to 0.20 kg ai/ha with an application interval of 7–14 days, at a maximum annual rate of 0.40 kg ai/ha with a PHI of 14 days.

Dry beans

Beans (dry)

Data were available from supervised trials on dry beans in Canada and the USA.

Pydiflumetofen residues in dry beans from independent trials in Canada and the USA matching GAP were (n=10): < 0.01 (5), 0.014, 0.018, 0.060, 0.10 and 0.24 mg/kg.

Soya bean (dry)

Data were available from supervised trials on soya bean in the USA.

Pydiflumetofen residues in soya bean from independent trials in the USA matching GAP were (n=21): < 0.01 (4), 0.011, 0.012, 0.013, 0.016 (2), 0.027, 0.028, 0.029, 0.031, 0.032, 0.036, 0.039, 0.041, 0.060, 0.088, 0.29 and 0.37 mg/kg.

Dry peas

Peas (dry)

Data were available from supervised trials on dry peas in Canada and the USA.

Pydiflumetofen residues in dry peas from independent trials in Canada and the USA matching GAP were (n=10): 0.023 (2), 0.028, 0.035, 0.053, 0.057, 0.059, 0.063, 0.064 and 0.096 mg/kg.

To consider a maximum residue level for a group, residues in individual crops should be similar (e.g. medians should not differ by more than 5×). The Meeting agreed to estimate a maximum residue level for the subgroup of dry beans and the subgroup of dry peas. In considering whether to combine data to estimate a maximum residue level, the Meeting recognized that the residue populations from trials on dry beans, soya bean and dry peas were not different according to statistical test (Kruskal-Wallis H-test). Therefore the Meeting decided to combine the data from dry beans, soya bean and dry peas to estimate a maximum residue level for the subgroup of dry beans and the subgroup of dry peas.

The combined pydiflumetofen residues in dry beans, soya bean and dry peas were in rank order (n=41): < 0.01 (9), 0.011, 0.012, 0.013, 0.014, 0.016 (2), 0.18, 0.023 (2), 0.027, 0.028 (2), 0.029, 0.031, 0.032, 0.035, 0.036, 0.039, 0.041, 0.053, 0.057, 0.059, 0.060 (2), 0.063, 0.064, 0.088, 0.096, 0.10, 0.24, 0.29 and 0.37 mg/kg.

As indicated no recommendation could be made for the subgroup of dry beans and the subgroup of dry peas.

Root and tuber vegetables

Tuberous and corm vegetables

Potato

Data were available from supervised trials on potato in Canada and the USA.

The GAP for tuberous and corm vegetables crop subgroup 1C of the USA is three foliar applications of up to 0.13 kg ai/ha at a maximum annual rate of 0.38 kg ai/ha with a PHI of 7 days.

Pydiflumetofen residues in potatoes from independent trials in Canada and the USA matching GAP were (n=22): < 0.01 (21) and 0.014 mg/kg.

No recommendation can be made for tuberous and corm vegetables.

Stalk and stem vegetables

Stalk and stem vegetables - Stems and Petioles

Celery

Data were available from supervised trials on celery in the USA.

The GAP for leaf petioles vegetables, crop subgroup 22B of the USA is two foliar applications of 0.073–0.20 kg ai/ha at a maximum annual rate of 0.40 kg ai/ha with a PHI of 0 day.

Pydiflumetofen residues in celery from independent trials in the USA matching GAP were (n=8): 2.6, 2.7, 3.9, 4.3, 4.5, 4.8, 5.4 and 8.1 mg/kg.

As indicated no recommendation could be made for stems and petioles.

Cereal grains

The GAP for cereal grains in the USA is a foliar application of up to 0.20 kg ai/ha at a maximum annual rate of 0.35 kg ai/ha, do not apply after full head emergence (Feekes 10.54=BBCH 71).

Wheat, similar grains, and Pseudocereals without husks

Wheat

Data were available from supervised trials on wheat in Canada and the USA.

Pydiflumetofen residues in wheat grains from independent trials in Canada and the USA matching GAP were (n=29): 0.015, 0.025, 0.035, 0.038, 0.040 (2), 0.048, 0.050, 0.057 (3), 0.062 (2), 0.063 (2), 0.067 (2), 0.10 (2), 0.11, 0.12 (4), 0.13, 0.17, 0.19 and 0.23 (2) mg/kg.

As indicated no recommendation could be made for wheat, similar grains, and pseudocereals without husks.

Barley, similar grains, and pseudocereals with husks

Barley

Data were available from supervised trials on barley in Canada and the USA.

Pydiflumetofen residues in barley grains from independent trials in Canada and the USA matching GAP were (n=14): 0.068, 0.081 (2), 0.11, 0.15, 0.19, 0.20, 0.23, 0.31, 0.55, 0.57, 0.66, 0.82 and 3.0 mg/kg.

Oats

Data were available from supervised trials on oats in Canada and the USA.

Pydiflumetofen residues in oats from independent trials in Canada and the USA matching GAP were (n=24): 0.056, 0.079, 0.11, 0.12, 0.14, 0.15 (2), 0.19, 0.20, 0.21, 0.22, 0.23, 0.24, 0.27, 0.32, 0.36, 0.41, 0.44, 0.48, 0.51, 0.54, 0.66, 0.94 and 2.1 mg/kg.

The combined pydiflumetofen residues in barley and oats were in rank order (n=38): 0.056, 0.068, 0.079, 0.081 (2), 0.11 (2), 0.12, 0.14, 0.15 (3), 0.19 (2), 0.20 (2), 0.21, 0.22, 0.23 (2), 0.24, 0.27, 0.31, 0.32, 0.36, 0.41, 0.44, 0.48, 0.51, 0.54, 0.55, 0.57, 0.66 (2), 0.82, 0.94, 2.1 and 3.0 mg/kg.

As indicated no recommendation could be made for barley, similar grains, pseudocereals with husks.

Maize Cereals

Maize

Data were available from supervised trials on field corn and popcorn in Canada and the USA.

The GAP for corn (field corn and popcorn) of the USA is a foliar application of up to 0.20 kg ai/ha at a maximum annual rate of 0.25 kg ai/ha with a PHI of 30 days.

Pydiflumetofen residues in field corn and popcorn from independent trials in Canada and the USA matching GAP were (n=22): < 0.01 (21) and 0.012 mg/kg.

No recommendation can be made for maize cereals.

Sweet Corns

Sweet corn (Corn-on-the-cob) (kernels plus cob with husk removed)

Data were available from supervised trials on sweet corn in the USA.

The GAP for sweet corn in the USA is two foliar application of up to 0.13 kg ai/ha at a maximum annual rate of 0.25 kg ai/ha with a PHI of 7 days.

Pydiflumetofen residues in sweet corn from independent trials in the USA matching GAP were (n=12): < 0.01 (12) mg/kg.

As indicated no recommendation could be made for sweet corn.

Oilseeds and oilfruits

Small seed oilseeds

Rape seed

Data were available from supervised trials on oilseed rape in Canada and the USA.

The GAP for canola (rapeseed, crop subgroup 20A) of Canada and the USA is a foliar application of up to 0.20 kg ai/ha at a maximum annual rate of 0.33 kg ai/ha with a PHI of 30 days.

Pydiflumetofen residues in rape seed from independent trials in Canada and the USA matching GAP were (n=18): 0.020, 0.031, 0.041, 0.046, 0.048, 0.050, 0.056, 0.070, 0.094, 0.095, 0.11, 0.14, 0.15, 0.17, 0.18, 0.35, 0.46 and 0.69 mg/kg.

No recommendation can be made for small seed oilseeds.

Other oilseeds

Peanut

Data were available from supervised trials on peanut in the USA.

The GAP for peanut in the USA is four foliar application of 0.025–0.050 kg ai/ha at a maximum annual rate of 0.20 kg ai/ha with a PHI of 14 day.

Pydiflumetofen residues in peanut nutmeat from independent trials in the USA matching GAP were (n=12): < 0.01 (9), 0.012 and 0.018 (2) mg/kg.

No recommendation can be made for peanut.

Animal feedstuffs

Legume animal feeds

Pea vines and hay

Data were available from supervised trials on pea in the USA.

The GAP for vine and hay of dried shelled peas and beans (except soya bean) in Canada is two foliar applications of up to 0.20 kg ai/ha at a maximum annual rate of 0.40 kg ai/ha with a PHI of 14 days.

Pydiflumetofen residues in pea vines (as received basis) from independent trials in the USA matching GAP were (n=5): 0.36, 0.42, 0.88, 0.90 and 2.8 mg/kg.

As indicated no recommendation could be made for pea vines.

Pydiflumetofen residues in pea hay (dry weight basis) from independent trials in the USA matching GAP were (n=5): 1.8, 3.0, 3.4, 5.9 and 17 mg/kg.

As indicated no recommendation could be made for pea hay until the contribution of residues in dry peas from direct application as well as uptake through the soil can be assessed.

Peanut hay

Data were available from supervised trials on peanut in the USA.

The GAP for peanut in the USA is four foliar application of up to 0.050 kg ai/ha at a maximum annual rate of 0.20 kg ai/ha with a PHI of 14 days.

Pydiflumetofen residues in peanut hay (dry weight basis) from independent trials in the USA matching GAP were (n=11): 2.0, 3.1, 4.3, 4.5, 4.7, 9.2, 12 (3), 13 and 15 mg/kg.

As indicated no recommendation could be made for peanut fodder until the contribution of residues in peanut from direct application as well as uptake through the soil can be assessed.

Soya bean forage and hay

Data were available from supervised trials on soya bean in the USA.

The GAP for soya bean in Canada and the USA does not allow the feeding of treated forage, hay and silage to livestock. The GAP for soya bean of Argentina is two foliar application of up to 0.045 kg ai/ha with a PHI of 30 day.

The trials conducted in the USA di not match the GAP for soya bean in Argentina.

The Meeting agreed that no recommendation could be made for soya bean forage and fodder.

Straw and fodder (dry) of cereal grains

The GAP for cereal grains in the USA is a foliar application of up to 0.20 kg ai/ha at a maximum annual rate of 0.35 kg ai/ha, do not apply after full head emergence (Feekes 10.54=BBCH 71). The GAP for cereal hay in the USA is a foliar application of up to 0.20 kg ai/ha with a PHI of 7 days.

Barley straw and fodder, dry

Data were available from supervised trials on barley in Canada and the USA.

Pydiflumetofen residues in barley straw (correction for an average 89% dry matter content) and hay (dry weight basis) from independent trials in Canada and the USA matching GAP were (n=21): 1.4, 3.7, 4.0, 4.2, 5.1, 5.7, 6.0, 6.5, 6.8, 8.0, 8.4, 9.2 (2), 10, 11, 13, 14, 17, 18, 20 and 26 mg/kg on a dry weight basis.

Oat straw and fodder, dry

Data were available from supervised trials on oats in Canada and the USA.

Pydiflumetofen residues in oats straw (correction for an average 90% dry matter content) and hay (dry weight basis) from independent trials in Canada and the USA matching GAP were (n=29): 1.4, 1.9, 2.0, 2.2, 3.0, 3.1, 3.7, 3.8, 3.9, 4.7, 5.3, 5.5, 5.7, 5.9, 6.6, 7.5, 7.7, 8.2, 8.3, 10, 11 (2), 14, 15, 19, 21, 23 (2) and 25 mg/kg on a dry weight basis.

Wheat straw and fodder, dry

Data were available from supervised trials on wheat in Canada and the USA.

Pydiflumetofen residues in wheat straw (correction for an average 88% dry matter content) and hay (dry weight basis) from independent trials in Canada and the USA matching GAP were (n=31): 1.8, 2.5, 3.6, 4.0, 4.5, 5.7, 6.5, 6.8, 7.2, 8.0, 9.5, 9.9, 10, 11, 12 (2), 13, 16, 17 (2), 18, 19, 20 (4), 24, 29, 33, 34 and 40 mg/kg on a dry weight basis.

The combined pydiflumetofen residues in straw and hay of barley, oats and wheat were in rank order (n=81): 1.4 (2), 1.8, 1.9, 2.0, 2.2, 2.5, 3.0, 3.1, 3.6, 3.7 (2), 3.8, 3.9, 4.0 (2), 4.2, 4.5, 4.7, 5.1, 5.3, 5.5, 5.7 (3), 5.9, 6.0, 6.5 (2), 6.6, 6.8 (2), 7.2, 7.5, 7.7, 8.0 (2), 8.2, 8.3, 8.4, 9.2 (2), 9.5, 9.9, 10 (3), 11 (4), 12 (2), 13 (2), 14 (2), 15, 16, 17 (3), 18 (2), 19 (2), 20 (5), 21, 23 (2), 24, 25, 26, 29, 33, 34 and 40 mg/kg.

As indicated no recommendation could be made for straw and fodder (dry) of cereal grains until the contribution of residues in cereal grains from direct application as well as uptake through the soil can be assessed.

Oat forage

Data were available from supervised trials on oats in Canada and the USA.

The GAP for cereal forage in the USA is a foliar application of up to 0.20 kg ai/ha with a PHI of 7 days.

Pydiflumetofen residues in oats forage (as received basis) from independent trials in Canada and the USA matching GAP were (n=28): 0.47, 0.62, 0.65, 0.73, 0.75, 1.0, 1.2, 1.3, 1.5 (2), 1.6, 1.8 (2), 1.9, 2.0, 2.3 (2), 2.7, 2.9, 3.2, 3.3, 3.6, 3.7, 4.2, 5.3, 6.5, 6.6 and 7.0 mg/kg.

As indicated no recommendation could be made for oat forage until the contribution of residues in cereal grains from direct application as well as uptake through the soil can be assessed.

Wheat forage

Data were available from supervised trials on wheat in Canada and the USA.

The GAP for cereal forage of the USA is a foliar application of up to 0.20 kg ai/ha with a PHI of 7 days.

Pydiflumetofen residues in wheat forage (as received basis) from independent trials in Canada and the USA matching GAP were (n=31): 0.24, 0.52, 0.97, 0.98, 1.2, 1.4, 1.6 (2), 1.7, 1.9 (2), 2.2 (2), 2.3, 2.5, 2.7, 3.3, 3.4, 3.6, 4.0, 4.2, 4.4, 4.8, 4.9, 5.4 (2), 6.2, 6.3, 7.7 and 11 (2) mg/kg.

As indicated no recommendation could be made for wheat forage until the contribution of residues in cereal grains from direct application as well as uptake through the soil can be assessed.

Maize forage

Data were available from supervised trials on field corn in the USA.

The GAP for corn (field corn and popcorn) forage in the USA is a foliar application of up to 0.20 kg ai/ha with a PHI of 7 days.

Pydiflumetofen residues in corn forage (as received basis) from independent trials in the USA matching GAP were (n=20): 0.38, 0.45, 0.64, 0.67, 0.69, 0.79, 0.91, 1.0 (3), 1.3 (2), 1.5, 1.6, 2.0, 2.1, 2.2, 2.4, 2.8 and 4.9 mg/kg.

As indicated no recommendation could be made for maize forage until the contribution of residues in maize from direct application as well as uptake through the soil can be assessed.

Sweet corn forage

Data were available from supervised trials on sweet corn in the USA.

The GAP for sweet corn in the USA is two foliar application of up to 0.13 kg ai/ha at a maximum annual rate of 0.25 kg ai/ha with a PHI of 7 days.

Pydiflumetofen residues in sweet corn forage (as received basis) from independent trials in the USA matching GAP were (n=12): 0.44, 0.49, 0.68, 0.73 (2), 0.75, 0.80, 0.90, 1.0, 1.2 (2) and 3.9 mg/kg.

As indicated no recommendation could be made for sweet corn forage until the contribution of residues in sweet corn from direct application as well as uptake through the soil can be assessed.

Maize fodder

Data were available from supervised trials on field corn and popcorn in the USA.

The GAP for corn (field corn and popcorn) of Canada is two foliar application of 0.10 kg ai/ha at a maximum annual rate of 0.20 kg ai/ha with a 30-day PHI.

Pydiflumetofen residues in corn stover (as received basis) from independent trials in the USA matching GAP were (n=23): 0.82, 1.1, 1.3, 1.5, 1.6 (2), 1.9, 2.1, 2.3, 2.6, 3.0, 3.1, 3.2, 3.4, 3.5 (3), 3.7, 4.2, 4.8, 5.0 (2) and 13 mg/kg.

As indicated no recommendation could be made for maize fodder until the contribution of residues in maize from direct application as well as uptake through the soil can be assessed.

Fate of residues during processing

High temperature hydrolysis

The hydrolytic stability of [pyrazole-5-^{14}C]-pydiflumetofen was studied under conditions of high temperature in sterile aqueous buffers at pH 4, 5 and 6 for periods of up to 60 minutes so as to simulate common processing practices (pasteurization, baking/brewing/boiling, and sterilization). Pydiflumetofen recoveries ranged from 95.5 to 97.5% of the applied radioactivity at the investigated pH and temperature ranges. Pydiflumetofen is considered stable under hydrolytic conditions at high temperatures.

Residues in processed commodities

The fate of pydiflumetofen residues has been examined in grape processing studies. Estimated processing factors and the derived STMR-Ps are summarised in the Table below.

Processing factors, STMR-P and HR-P for food and feed

Crop	Commodity	Calculated processing factors* [best estimate]	maximum residue level (mg/kg)	STMR-P (mg/kg)	HR-P (mg/kg)
		Pydiflumetofen			
Grape	RAC (fruit)	-	1.5	0.29	0.85
	Must	0.13, 0.32, 0.44, 0.62, 1.5, 1.7, 2.1, 3.0 [1.06]	-	0.31	-
	Juice	0.02, 0.02, 0.05, 0.07, 0.49, 0.57 [0.06]	-	0.017	-
	Red wine	< 0.20, 0.10, 0.17, 0.24 [0.135]	-	0.039	-
	White wine	0.08, 0.11, 0.52, 0.60 [0.315]	-	0.091	-
	Dried grapes	1.7, 2.0, 2.4, 2.5, 2.8, 4.8 [2.45]	4	0.71	2.1
	Refined seed oil	0.71, 1.0, 1.1, 1.1 [1.05]	-	0.30	-
	Wet pomace	0.39, 1.0, 1.6, 2.3, 2.4, 3.0, 4.3, 4.4, 6.3, 8.0, 8.3 [3.0]	-	0.87	-

* Each value represents a separate study. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

Residues in animal commodities

Farm animal feeding studies

The Meeting received a lactating dairy cow and a laying hen feeding studies, which provided information on likely residues resulting in animal commodities, milk and eggs from pydiflumetofen residues in the animal diet.

Lactating dairy cows

Holstein/Friesian dairy cows were dosed with pydiflumetofen for 28 days at the equivalent of 15, 45 and 150 ppm in the diet. Residues of pydiflumetofen were below the LOQ (0.01 mg/kg) in muscle at all feeding levels. Whole milk and kidney contained no residue (< 0.01 mg/kg) of pydiflumetofen at the 15 and 45 ppm feeding level. Liver and fat contained pydiflumetofen residues of < 0.01–0.01 mg/kg at the 15 ppm feeding level, 0.02–0.05 mg/kg at the 45 ppm level and 0.05–0.10 mg/kg at the 150 ppm level.

For estimation of dietary exposure, residues of pydiflumetofen and 2,4,6-TCP (free + conjugated forms; total 2,4,6-TCP), expressed as pydiflumetofen were below the LOQ in muscle at all feeding levels. Whole milk contained no residue (<LOQ) of pydiflumetofen and total 2,4,6-TCP, expressed as pydiflumetofen at the 15 ppm feeding level. Residues of pydiflumetofen and total 2,4,6-TCP, expressed as pydiflumetofen in whole milk achieved a plateau concentration of 0.03–0.05 mg/kg at the 45 feeding ppm level and 0.18–0.20 mg/kg at the 150 ppm level.

Residues of pydiflumetofen, total 2,4,6-TCP and SYN547897 (free + conjugated form), expressed as pydiflumetofen in liver and kidney were 0.07–0.08 mg/kg at the 15 ppm feeding level, 0.28–0.31 mg/kg at the 45 ppm level and 0.78 mg/kg at the 150 ppm level.

Laying hens

Laying hens were dosed with pydiflumetofen for 28 days at the equivalent of 3, 9 and 30 ppm in the diet. Residues of pydiflumetofen were below the LOQ (0.01 mg/kg) in muscle, liver, kidney and fat at all feeding levels, and in eggs at the 3 ppm feeding level. At the 30 ppm level, pydiflumetofen residues in eggs were 0.02–0.03 mg/kg from day 3 to day 24.

For estimation of dietary exposure, residues of pydiflumetofen and total 2,4,6-TCP, expressed as pydiflumetofen were below the LOQ in muscle, liver and fat at all feeding levels. Eggs contained no residue (<LOQ) of pydiflumetofen and total 2,4,6-TCP, expressed pydiflumetofen at the 3 ppm feeding level. Residues of pydiflumetofen and total 2,4,6-TCP, expressed as pydiflumetofen in eggs achieved a plateau concentration of 0.03 mg/kg at the 9 ppm feeding level and 0.09 mg/kg at the 30 ppm level. Kidney contained pydiflumetofen and total 2,4,6-TCP, expressed as pydiflumetofen of 0.05 mg/kg at the 9 ppm feeding level and 0.12 mg/kg at the 30 ppm level.

Farm animal dietary burden

The Meeting concluded that the contribution of the additional uptake from soil to residues in feed commodities following treatment as a primary crop may significantly increase the terminal residue. However, the information available for rotational crops was not considered sufficient to estimate median and highest residues in feed commodities. Therefore, the current Meeting did not estimate the farm animal dietary burden and consequently did not estimate residues in animal commodities.

At a future Meeting, the farm animal dietary burden will be estimated when additional information addressing the contribution of soil uptake into feed commodities becomes available.

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 and IESTI assessment.

The Meeting recommended the following residue definitions for pydiflumetofen.

Definition of the residue for compliance with the MRL for both plant and animal commodities: *Pydiflumetofen.*

Definition of the residue for animal commodities other than mammalian liver and kidney for dietary risk assessment: *sum of pydiflumetofen and 2,4,6-trichlorophenol (2,4,6-TCP) and its conjugates, expressed as pydiflumetofen*

Definition of the residue for mammalian liver and kidney for dietary risk assessment: *sum of pydiflumetofen, 2,4,6-trichlorophenol (2,4,6-TCP) and its conjugates, and 3-(difluoromethyl)-N-methoxy-1-methyl-N-[1-methyl-2-(2,4,6-trichloro-3-hydroxyphenyl) ethyl]pyrazole-4-carboxamide (SYN547897) and its conjugates, expressed as pydiflumetofen*

The residue is fat-soluble.

Recommended maximum residue levels, STMRs and HRs for raw commodities

Commodity		Recommended MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New		
FB 2008	Small fruit vine climbing, Subgroup of (includes all commodities in this subgroup)	1.5	0.29	0.85

* at or about the LOQ.

Recommended MRLs, STMR-Ps and HR-Ps for processed commodities

Commodity		Recommended MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New		
DF 0269	Dried grapes (= Currants, Raisins and Sultanas)	4	0.71	2.1

Dietary exposure and feed burden only

Commodity Name	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
Grape juice (pasteurized)	0.017	
Grape must	0.31	
Grape seed oil, refined	0.30	
Grape wet pomace	0.87	
Red wine	0.039	
White wine	0.091	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for pydiflumetofen is 0–0.1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for pydiflumetofen were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2018 JMPR Report. The IEDIs were 0% of the maximum ADI.

The Meeting concluded that long-term dietary exposure to residues of pydiflumetofen from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for pydiflumetofen is 0.3 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for pydiflumetofen were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2018 JMPR Report. The IESTIs varied from 0–20% of the ARfD for children and 0–9% for the general population.

The Meeting concluded that acute dietary exposure to residues of pydiflumetofen from uses considered by the present Meeting is unlikely to present a public health concern.

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34592	Inns L.	2014	SYN545974 - Metabolism of [^{14}C]-SYN545974 in Tomatoes Charles River, UK, 34592 Syngenta Report No. TK0123336 Syngenta File No. SYN545974_10143
33587	Chapleo, S., Inns, L., Johnson, J.	2015	SYN545974 - Metabolism of [^{14}C]-SYN545974 in Oilseed Rape Charles River Edinburgh Ltd, UK, 33587 Syngenta Report No. TK0123337 Syngenta File No. SYN545974_10225
33963	Przeorska, J.	2015	SYN545974 - Metabolism of [^{14}C]-SYN545974 in the Lactating Goat Charles River, Tranent, Edinburgh, EH33 2NE, 33963 Syngenta Report No. TK0061720 Syngenta File No. SYN545974_10284
33964	Przeorska, J.	2015	SYN545974 - Metabolism of [^{14}C]-SYN545974 in the Laying Hen Charles River, Tranent, Edinburgh, EH33 2NE, 33964 Syngenta Report No. TK0103773 Syngenta File No. SYN545974_10282
34316	Chapleo, S., Johnson, J.	2015	SYN545974 –Uptake and Metabolism of [^{14}C]-SYN545974 in Confined Rotational Crops Charles River Edinburgh Ltd, UK, 34316 Syngenta Report No. TK0061717 Syngenta File No. SYN545974_10227
TK0103846	Mäyer T.J.	2015	SYN545974 SC (A19649B) - Field Accumulation in Rotational Crops (30-, 60-, 90 and 150-day Plant Back Intervals) USA 2013 Syngenta Crop Protection LLC, Greensboro USA and ADPEN Laboratories Inc., Jacksonville, USA Syngenta Report No. TK0103846 Syngenta File No. A19649B_50064
S13-01022	Lakaschus S., Lau A.,	2016	SYN545974 - Residue Study on Rotational Crops in the United Kingdom and Germany during 2013 - 2014 Eurofins Agroscience Services Chem GmbH, Hamburg, Germany Syngenta Report No. S13-01022 Syngenta File No. A19649B_10234
S13-01023	Lakaschus S., Lau A.,	2016	SYN545974 - Residue Study on Rotational Crops in Southern France and Italy during 2013 - 2015

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			Eurofins Agroscience Services Chem GmbH, Hamburg, Germany Syngenta Report No. S13-01023 Syngenta File No. A19649B_10235
3200053	Lewis C, Gilbert J,	2015	SYN545974 - ^{14}C-SYN545974: Hydrolysis in Sterile Buffer at pH 4, 7 and 9 Smithers Viscient (ESG) Ltd / Otley Road, Harrogate, North Yorkshire HG3 1PY, UK, 3200053 Syngenta File No. SYN545974_50052
3200128	Hurst L., Mould R.	2014	SYN545974 - Soil Photolysis of ^{14}C-SYN545974 Smithers Viscient (ESG) Ltd., Otley Road, Harrogate, North Yorkshire, HG3 1PY, UK, 3200128 Syngenta File No. SYN545974_50182
SYN/48/01-KIN01	Ford S.	2015	SYN545974 - Laboratory Degradation Kinetics for Trigger and Modelling Endpoints for Parent JSC International Limited, Harrogate, North Yorkshire, UK, SYN/48/01-KIN01 Syngenta File No. SYN545974_10373
S13-02236-FINAL	Finger N.	2015	SYN545974 - Bare Soil Plot Soil Dissipation Study in UK in 2013-2015 Eurofins Agroscience Services GmbH, Carl-Goerdeler-Weg 5, D21684 Stade, Germany, S13-02236-FINAL Syngenta File No. A19649B_10172
S13-02237-FINAL	Finger N.	2015	SYN545974 - Bare Soil Plot Soil Dissipation Study in Germany in 2013-2015 Eurofins Agroscience Services GmbH, Carl-Goerdeler-Weg 5, D21684 Stade, Germany, S13-02237-FINAL Syngenta File No. A19649B_10166
S13-02238-FINAL	Finger N.	2015	SYN545974 - Bare Soil Plot Soil Dissipation Study in Northern France in 2013-2015 Eurofins Agroscience Services GmbH, Carl-Goerdeler-Weg 5, D21684 Stade, Germany S13-02238-FINAL Syngenta File No. A19649B_10168
S13-02239-FINAL	Finger N.	2015	SYN545974 - Bare Soil Plot Soil Dissipation Study in Southern France in 2013-2015 Eurofins Agroscience Services GmbH, Carl-Goerdeler-Weg 5, D21684 Stade, Germany, S13-02239-FINAL Syngenta File No. A19649B_10170
S13-02240-FINAL	Finger N.	2015	SYN545974 - Bare Soil Plot Soil Dissipation Study in Spain in 2013-2015 Eurofins Agroscience Services GmbH, Carl-Goerdeler-Weg 5, D21684 Stade, Germany, S13-02240-FINAL Syngenta File No. A19649B_10171
S13-02241-FINAL	Finger N.	2015	SYN545974 - Bare Soil Plot Soil Dissipation Study in Italy in 2013-2015 Eurofins Agroscience Services GmbH, Carl-Goerdeler-Weg 5, D21684 Stade, Germany, S13-02241-FINAL Syngenta File No. A19649B_10167
GRM061.03A	Huang S-B.	2015	SYN545974 - Analytical Method for Determination of SYN545974 in Crops by LC-MS/MS with Validation Data Syngenta Crop Protection, LLC 410 Swing Road Greensboro, NC 27409 USA, GRM061.03A Syngenta Task No. TK0103794 Syngenta File No. SYN545974_50054
S14-05352	Schulz D., Trumper C.	2015	SYN545974 - Validation of the Syngenta Method GRM061.03A for the Determination of Residues of SYN545974 in Crop Matrices Eurofins Agroscience Services Chem GmbH, Germany, S14-05352 Syngenta Task No. TK0103789 Syngenta File No. SYN545974_10180
S13-03422	Tessier V.	2015	SYN545974 - Residues study on grapes and processed products in Spain, Southern France and Italy in 2013 Eurofins Agroscience Services Chem SAS, 75 chemin de Sommières, 30310 Vergèze, France, S13-03422 Syngenta File No. A19649B_10180
TK0163552	Salzman, F.P.	2015	SYN545974 SC (A19649B) - Magnitude of the Residues in or on Tomatoes and Peppers (Representative Commodities of Fruiting

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			Vegetables Crop Group 8) USA 2013 Syngenta Crop Protection LLC, Greensboro, USA Syngenta Report No. TK0163552 Syngenta File No. A19649B_50034
S14-05402	Meseguer C.	2015	SYN545974 - Validation of the QuEChERS Method for the Determination of Residues of SYN545974 in Crop Matrices by LC-MS/MS Eurofins Agroscience Services Chem SAS, France, S14-05402 Syngenta Task No. TK0253228 Syngenta File No. SYN545974_10174
S14-05729	Khan A.	2015	SYN545974 - Independent Laboratory Validation of the QuEChERS method for the Determination of Residues of SYN545974 in Crop Matrices by LC-MS/MS Eurofins Agroscience Services Chem Ltd, UK, S14-05729 Syngenta Task No. TK0103788 Syngenta File No. SYN545974_10193
ADPEN-2K14-TK0163712-001	Perez, R.	2014	SYN545974 - Evaluation of SYN545974, FDA Multiresidue Method (MRM) Testing ADPEN Laboratories Inc., 11757 Central Parkway, Jacksonville, FL 32224 USA, ADPEN-2K14-TK0163712-001 Syngenta Task No. TK0163712 Syngenta File No. SYN545974_50047
GRM061.06A	Mayer, L.	2015	SYN545974 - Analytical Method (GRM061.06A) for the Determination of SYN545974 in Bovine Milk, Liver, Kidney, Muscle, Fat, Blood and Hen Eggs by LC-MS/MS Syngenta Crop Protection, LLC, 410 Swing Road, Greensboro, NC 27409 USA, GRM061.06A Syngenta Task No. TK0103792 Syngenta File No. SYN545974_50123
36383	Harris, J.	2015	SYN545974 - Validation of an Analytical Method for the Determination of SYN545974 in Bovine Meat, Liver, Kidney, Fat, Milk, Blood and Chicken Eggs Charles River Tranent, Edinburgh, EH33 2NE, UK, 36383 Syngenta Task No. TK0103783 Syngenta File No. SYN545974_10247
1781.7103	Smith, R.	2015	SYN545974 - Independent Laboratory Validation of Analytical Method (GRM061.06A) for the Determination of SYN545974 in Bovine Liver by LC-MS/MS Smithers Viscient, 790 Main Street, 02571-1037 Wareham, MA USA, 1781.7103 Syngenta Task No. TK0272121 Syngenta File No. SYN545974_50208
PTRL Europe ID P 3592 G	Richter, S.	2015	SYN545974: Validation of the QuEChERS Method for the Determination of Residues of SYN545974 in Animal Matrices by LC-MS/MS PTRL Europe GmbH, Germany, PTRL Europe ID P 3592 G Syngenta Task No. TK0208383 Syngenta File No. SYN545974_10169
CEMR-7055	Bradford W. J.	2015	SYN545974 - Independent Laboratory Validation of the QuEChERS Method for the Determination of Residues of SYN545974 in Liver and Milk by LC-MS/MS CEM Analytical Services Limited, UK, CEMR-7055 Syngenta Task No. TK0259229 Syngenta File No. SYN545974_10195
PASC-REP-1467	Xu, A.	2017	SYN545974 - Independent Laboratory Validation of QuEChERS Method for the Determination of Residues of SYN545974 in Egg and Muscle by LC-MS/MS Primera Analytical Solutions Corp., Princeton, NJ USA, PASC-REP-1467 Syngenta Task No. TK0374048 Syngenta File No. SYN545974_50832
GRM061.07A	Mayer L.	2015	SYN545974 - Analytical Method (GRM061.07A) for the Determination of Free and Conjugated 2,4,6-trichlorophenol in Bovine Milk, Liver, Kidney, Muscle, Fat, Blood and Hen Eggs by LC-MS/MS

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			Syngenta Crop Protection, LLC, 410 Swing Road, Greensboro, NC 27409 USA, GRM061.07A Syngenta Task No. TK0103792 Syngenta File No SYN545974_50114
PTRL Europe ID P 3613 G	Senciuc M., Asekunowo J.	2015	SYN545974 - Validation of the Analytical Method GRM 061.07A for the Determination of Residues of Conjugated 2,4,6-Trichlorophenol in Animal Matrices PTRL Europe GmbH, Germany, PTRL Europe ID P 3613 G Syngenta Task No. TK0257521 Syngenta File No SYN545974_10178
R B5134	Schlewitz, P.	2015	SYN545974 - Independent Lab Validation of the Analytical Method for the Determination of Conjugated 2,4,6-Trichlorophenol in Animal Matrices ANADIAG, 16, rue Ampère, 67500 Haguenau, France, R B5134 Syngenta Task No. TK0259228 Syngenta File No. SYN545974_10239
GRM061.08A	Mayer, L.	2015	SYN545974 - Analytical Method (GRM061.08A) for the Determination of SYN548264 and SYN508272 in Bovine Milk by LC-MS/MS Syngenta Crop Protection, LLC, 410 Swing Road, Greensboro, NC 27409 USA, GRM061.08A Syngenta Task No. TK0103792 Syngenta File No. SYN548264_50000
GRM061.09A	Mayer, L.	2015	SYN545974 - Analytical Method (GRM061.09A) for the Determination of Free and Conjugated SYN547897 and SYN548263 in Kidney and Liver by LC-MS/MS Syngenta Crop Protection, LLC, 410 Swing Road, Greensboro, NC 27409 USA, GRM061.09A Syngenta Task No. TK0103792 Syngenta File No. SYN547897_50000
S13-02224	Yozgatli H.P., Feddersen T.	2015	SYN545974 - Storage Stability in Crops Stored Frozen for up to 23 months - Storage Stability Report Eurofins Agroscience Services Chem GmbH, Hamburg, Germany, S13-02224 Syngenta Report No. TK0103785 Syngenta File No. SYN545974_10278
110G850	Sharp S.	2016	SYN545974 - Stability of SYN545974 in Processed Commodities of Soybean, Corn, Apple and Grapes Under Freezer Storage Conditions EPL Bio Analytical Services Niantic, Illinois, 110G850 Syngenta Report No. TK0103784 Syngenta File No. SYN545974_50491
36552	Harris J.	2016	SYN545974 - Storage Stability of SYN545974 in Bovine Muscle, Liver, Milk, Fat and Chicken Eggs (Ongoing Study) Charles River Laboratories, Edinburgh, UK, 36552 Syngenta Report No. TK0103639 Syngenta File No. SYN545974_10291
CEMR-7064	Langridge G.	2016	Pydiflumetofen - Frozen Storage Stability of Residues of SYN508272, SYN548264, SYN547897 and SYN548263 in Animal Matrices CEM Analytical Services Ltd., Wokingham, UK, Interim Report No. CEMR-7064 Syngenta Report No. TK0257525 Syngenta File No. SYN508272_10915
PTRL Europe ID P 3669 G	Senciuc M. and Asekunowo J.	2016	SYN545974 - Storage Stability of Residues of Conjugated 2,4,6-trichlorophenol in Animal Matrices Stored Frozen for up to Twelve Months PTRL Europe GmbH, Ulm, Germany, Interim Report No. PTRL Europe ID P 3669 G Syngenta Report No. TK0257522 Syngenta File No. SYN545974_10280
TK0103853	Salzman F.P.	2015	SYN545974 SC (A19649B) - Magnitude of the Residues in or on Grapes USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0103853, GLP, Unpublished.
TK0174260	Homa K. and Leonard R.	2015	FTH 545 (SYN545974 SC (200)) - Magnitude of the Residue on Cucumber (Field and Greenhouse), IR-4 Project Headquarters,

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TK0174258	Homa K. and Leonard R.	2015	FTH 545 (SYN545974 SC (200)) - Magnitude of the Residue on Cantaloupe, IR-4 Project Headquarters, Report No. IR-4 PR No. 11158; Syngenta Report No. TK0174258, GLP, Unpublished.
TK0163552	Salzman F.P.	2015	SYN545974 SC (A19649B) - Magnitude of the Residues in or on Tomatoes and Peppers (Representative Commodities of Fruiting Vegetables Crop Group 8) USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0163552, GLP, Unpublished.
TK0163598	Oakes T.L.	2015	SYN545974 SC (A19649B) – Magnitude of the Residues in or on Lettuce (Head and Leaf), Spinach, and Celery (Representative Commodities of Crop Groups 4A & 4B) USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0163598, GLP, Unpublished.
TK0163595	Mäyer T.J.	2015	SYN545974 SC (A19649B) and SYN545974 EC (A17573A) - Magnitude of the Residues in or on Dry Bean and Pea (Representative Commodities for Crop Group 6C) USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0163595, GLP, Unpublished.
TK0103849	Sagan K.	2015	SYN545974 EC (A17573A) and SYN545974 SC (A19649B) - Residue Levels on Dry Bean and Pea from Trials Conducted in Canada During 2013, Syngenta Canada Inc, Syngenta Report No. TK0103849, GLP, Unpublished.
TK0103860	Oakes T.L.	2015	SYN545974 SC (A19649B) and SYN545974 EC (A17573A) - Magnitude of the Residues in or on Soybeans USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0103860, GLP, Unpublished.
TK0163562	Smith N.	2015	SYN545974 SC (A19649B) - Magnitude of the Residues in or on Potato as a Representative Crop of Tuberous and Corm Vegetables, Subgroup 1C USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0163562, GLP, Unpublished.
TK0103851	Sagan K.	2015	SYN545974 (A19649B) - Residue Levels on Potatoes from Trials Conducted in Canada During 2013, Syngenta Canada Inc, Syngenta Report No. TK0103851, GLP, Unpublished.
TK0163574	Oakes T.L.	2015	SYN545974 SC (A19649B) and SYN545974 EC (A17573A) - Magnitude of the Residues in or on Wheat USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0163574, GLP, Unpublished.
TK0103847	Sagan K.	2015	SYN545974 EC (A17573A) and SYN545974 SC (A19649B) - Residue Levels on Wheat (Forage, Hay, Grain and Straw) from Trials Conducted in Canada During 2013, Syngenta Canada Inc, Syngenta Report No. TK0103847, GLP, Unpublished.
TK0163584	Oakes T.L.	2015	SYN545974 SC (A19649B) and SYN545974 EC (A17573A) - Magnitude of the Residues in or on Barley USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0163584, GLP, Unpublished.
TK0163517	Sagan K.	2015	SYN545974 EC (A17573A) and SYN545974 SC (A19649B) - Residue Levels on Barley (Hay, Grain and Straw) from Trials Conducted in Canada During 2013, Syngenta Canada Inc, Syngenta Report No. TK0163517, GLP, Unpublished.
TK0163581	McDonald T.J.	2015	SYN545974 SC (A19649B) and SYN545974 EC (A17573A) - Magnitude of the Residues in or on Oats USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0163581, GLP, Unpublished.
TK0163519	Sagan K.	2015	SYN545974 EC (A17573A) and SYN545974 SC (A19649B) - Residue Levels on Oats (Forage, Hay, Grain and Straw) from Trials Conducted in Canada During 2013 and 2014, Syngenta Canada Inc, Syngenta Report No. TK0163519, GLP, Unpublished.
TK0163563	Oakes T.L.	2015	SYN545974 SC (A19649B) and SYN545974 EC (A17573A) - Magnitude of the Residues in or on Field Corn and Popcorn (Maize) USA 2014, Syngenta Crop Protection LLC, Syngenta Report No. TK0163563, GLP, Unpublished.

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TK0163606	Smith N.	2015	SYN545974 SC (A19649B) and SYN545974 EC (A17573A) - Magnitude of the Residues in or on Canola as Representative Crop of Rapeseed, Subgroup 20A USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0163606, GLP, Unpublished.
TK0103848	Sagan K.	2015	SYN545974 EC (A17573A) and SYN545974 SC (A19649B) - Residue Levels on Canola Seed from Trials Conducted in Canada During 2013, Syngenta Canada Inc, Syngenta Report No. TK0103848, GLP, Unpublished.
TK0163545	Smith N.	2015	SYN545974 SC (A19649B) and SYN545974 EC (A17573A) - Magnitude of the Residues in or on Peanut USA 2013, Syngenta Crop Protection LLC, Syngenta Report No. TK0163545, GLP, Unpublished.
35072	Dixon S.	2014	[14C] SYN545974 - Aqueous Hydrolysis at 90, 100 and 120 °C; Report Number: 35072 Charles River, Tranent, Edinburgh, EH33 2NE, UK Syngenta Report No. TK0103777 Syngenta File No. SYN545974_10110
S13-03421	Tessier V.	2015	SYN545974 - Residues Study on Grapes and Processed Products in Germany, Northern France and Hungary in 2013 Syngenta Report No. S13-03421 Syngenta File No. A19649B_10177
S13-03422	Tessier V.	2015	SYN545974 - Residues study on grapes and processed products in Spain, Southern France and Italy in 2013 Eurofins Agroscience Services Chem SAS, 75 chemin de Sommières, 30310 Vergèze, France, S13-03422 Syngenta File No. A19649B_10180
TK0103853	Salzman F.P.	2015	SYN545974 SC (A19649B) - Magnitude of the Residues in or on Grapes USA 2013 Syngenta Crop Protection LLC, Greensboro, USA Syngenta Report No. TK0103853 Syngenta File No. A19649B_50055
35775	Vance C., Anderson R., Harris J.	2015	SYN545974 - Magnitude of Residues in Milk and Tissues of Dairy Cows following Multiple Oral Administrations of SYN545974 Charles River Laboratories, Edinburgh, UK, 35775 Syngenta Report No. TK0103795 Syngenta File No. SYN545974_10288
37460	Mills B., Brown D.	2016	SYN545974 – Magnitude of Residues in Tissues of Dairy Cows following Multiple Oral Administration of SYN545974. Charles River Laboratories, Edinburgh, UK, 37460 Syngenta Report No. TK0294052 Syngenta File No. SYN545974_10421
TK0103796	McDonald, T.	2015	SYN545974 - Magnitude of Residues in Tissue and Eggs Resulting from the Feeding of Three Dose Levels to Poultry During 2014 Syngenta Crop Protection LLC, Greensboro, USA Syngenta Report No. TK0103796 Syngenta File No. SYN545974_50189