Behaviour of fluopicolide in plants

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1 Introduction

Fluopicolide (AE C638206) is an acyl picolide class fungicide which is effective at low dose rates against a wide range of oomycete (Phycomycete) diseases including downy mildews (*Plasmopara, Pseudoperonospara, Peronospora, Bremia*), late blight (*Phytophthora*) and some *Pythium* species. Fluopicolide is redistributed via the xylem (acropetal systemic activity) and effective disease control can be achieved from foliar, seed and soil applications. Fluopicolide is targeted for use in root and tuber vegetables, brassica leafy vegetables, bulb vegetables, fruiting vegetables, cucurbits and hops.

Knowledge of the nature of the residue associated with the use of pesticides on target crops and in rotational crops is essential for the development of analytical methods in these crops for enforcement and risk assessment. Therefore metabolism studies in three contrasting target crops and in rotational crops were conducted to determine the metabolic profile of fluopicolide resulting from its use according to good agricultural practices.

2 Behaviour in plants

The behaviour and metabolism of fluopicolide was investigated in potatoes, lettuce and grapes under simulated field conditions. The formulation utilized in the metabolism studies was consistent with the type and composition likely to be used in the commercial products. In all studies crops were treated with a 20 SC (w/w) formulation with a 0.05 % concentration of the adjuvant Crodamol PC.

The fluopicolide structure consists of a substituted phenyl ring and a substituted pyridine ring joined by an amide linkage. Due to the potential lability of the amide, all metabolism investigations were conducted with test material labelled separately as either [U-^{14}C-phenyl]-fluopicolide or [2,6-^{14}C-pyridyl]-fluopicolide, so that metabolites arising from either part of the molecule could be detected (see Mackenzie et al., this issue, page 209).

The behaviour and metabolism of fluopicolide in confined rotational crops of wheat, radish and lettuce was also determined. Either [U-^{14}C-pheny]-fluopicolide or [2,6-^{14}C-pyridyl]-fluopicolide was applied to bare soil at the maximum seasonal application rate after which the soil was aged prior to the planting of the rotational crops.

2.1 Metabolism in potatoes

2.1.1 Method

Potatoes (var. Red Pontiac) were grown under simulated field conditions in multiple steel crop tanks. Potatoes were treated two times at nominal rates of 200 g a.i./ha with [U-^{14}C-phenyl]-fluopi-

colide and [2,6-^{14}C-pyridyl]-fluopicolide, formulated as 20 % SC formulation. The cumulative treatment rate was equal to the proposed maximum seasonal application of 400 g a.i./ha. The first treatment was between BBCH growth stages 31 and 33 (beginning of crop cover to when 30 % of plants meet between rows), and the second treatment was 20 days prior to normal harvest. The raw agricultural commodity, potatoes, was analyzed in duplicate at maturity. Aerial plant part samples were collected at harvest as a potential aid to metabolite identification and are not a raw agricultural commodity of potatoes. Additional trials were carried out at exaggerated application rates (total application 4000 g/ha) to provide additional material for metabolite identification.

The residues in the potato foliage and tuber samples were recovered by an acetonitrile wash and an acetonitrile extraction. Metabolites in the extractable residue were identified and quantified by TLC or HPLC against a standard mix of putative metabolites. Residues remaining in the matrix were determined by combustion analysis. Non-extractable residues were subjected to acid hydrolysis.

2.1.2 Findings

Good agreement was obtained throughout from the duplicate samples of potato tubers. No appreciable differences in total residues were observed between the phenyl-label treated potatoes and the pyridyl-label treated potatoes. The total residue in both treatment labels at harvest was very similar. The vast majority of residue was solvent extractable. Mean results of residue levels and the extraction profiles at maturity are presented in Table 1.

Analysis of the extractable residues showed a qualitatively similar metabolic profile in all potato foliage and tuber tissues. For the phenyl-label and the pyridyl-label treated potatoes, total radioactive residues (TRR) in potato tubers at final harvest were 0.081 mg/kg and 0.053 mg/kg, respectively and in subsequent field residue trials no residues in tubers above the limit of quantification of 0.01 mg/kg were detected. The acetonitrile surface wash removed a small amount of the total residue (11.0-12.6 % TRR). Subsequent acetonitrile extraction removed an additional 71.9-78.4 % of the TRR, leaving only 10.7-15.6 % non-extractable. The extractable residue was concentrated and subjected to a cleanup with a SPE C-18 cartridge before being analyzed. Acid hydrolysis of the extracted fiber removed an additional 5.9-8.7 % of the TRR from tubers, leaving 4.9-6.9 % in the fiber.

Metabolites in the acetonitrile washes and acetonitrile extracts of the potato samples were identified and quantified by comparison with putative metabolites by reverse phase thin layer chromatography. Qualitative chromatography was performed on one-dimensional normal phase TLC. For the phenyl-label treated potatoes, the parent compound, fluopicolide was found in potatoes at 51.1 % of TRR. Lesser amounts of AE C653711 (25.4 % of TRR) and minor amounts of AE C643890 (2.4 % of TRR) were also identified. For the pyridyl-label treated potatoes, fluopicolide was found in potatoes at 70.2 % of TRR. Lesser amounts of AE C657188 (12.0 % of TRR) and minor amounts of AE C643890 (1.7 % of TRR) were also identified.

The known soil metabolites AE C653711 and AE C657188 were not thought to be

Table 1: Distribution of fluopicolide and metabolites in potatoes and grapes.

	Potatoes				**Grapes**			
Radiolabel	[U-^{14}C-phenyl]		[2,6-^{14}C-pyridyl]		[U-^{14}C-phenyl]		[2,6-^{14}C-pyridyl]	
Application type	Foliar				Foliar			
Application rate (g/ha)	2 x 200				167+116+116			
Pre-harvest interval (d)	20				21			
Commodity	Potatoes				Grapes			
TRR (mg/kg)	0.081		0.053		1.265		1.040	
	mg/kg[a]	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Fluopicolide	0.041	51.1	0.037	70.2	1.152	91.2	0.910	87.4
AE C653711	0.021	25.4	n.a.	n.a.	0.026	2.0	n.a.	n.a.
AE C657188	n.a.	n.a.	0.007	12.0	n.a.	n.a.	0.024	2.3
AE C643890	0.003	2.4	0.001	1.7	0.002	0.2	n.d.	n.d.
Total identified	**0.064**	**78.8**	**0.045**	**83.9**	**1.180**	**93.4**	**0.934**	**89.7**
Total unidentified	0.004	5.6[b]	0.003	5.4[b]	0.029	2.3[c]	0.045	4.3[c]
Non-extractable residue (NER)	0.013	15.6	0.006	10.7	0.055	4.3	0.063	6.0
NER acid hydrolysate	0.007	8.7	0.004	5.9	-	-	-	-
Acid extracted matrix	0.006	6.9	0.003	4.9	-	-	-	-
Total	**0.081**	**100**	**0.053**	**100**	**1.265**	**100**	**1.040**	**100**

n.a.: not applicable, n.d.: not detected
[a] Results expressed as mg equivalent fluopicolide per kilogram fresh weight
[b] No single metabolite comprised > 2 % of TRR
[c] No single metabolite comprised > 1 % of TRR

formed in plants directly but formed part of the crop residue as a result of plant uptake from soil.
The proposed metabolic pathway of fluopicolide in potatoes is shown in Fig. 1. Fluopicolide is metabolized in potatoes to AE C653711, AE C657188, and minor amounts of AE C643890.

2.2 Metabolism in grapes

2.2.1 Method

Grapevines (var. Sunbelt and Niagara) were grown under greenhouse conditions in pots. Grapevines were treated three times at nominal rates of 167, 116.5 and 116.5 g a.i./ha with [U-^{14}C-phenyl]-fluopicolide and [2,6-^{14}C-pyridyl]-fluopicolide, formulated as a 20 % SC formulation. Additionally plants were treated three times at nominal rates of 1.67, 1.16 and 1.16 kg a.i./ha [U-^{14}C-phenyl]-fluopicolide and [2,6-^{14}C-pyridyl]-fluopicolide, formulated as a 20 % SC formulation, to provide material for metabolite identification if needed. The cumulative treatment rate was equal to the proposed labelled maximum seasonal application of 400 g a.i./ha. The applications were made between BBCH stages 55 and 57 (Day Zero), between BBCH stages 71 and 73 (Immature harvest), and 21 days prior to harvest maturity.
Plants were harvested on Day Zero, immediately prior to the second treatment

and at final harvest maturity. Residues in the early grape foliage samples were recovered by surface wash with acetonitrile followed by extraction with acetonitrile. Residue remaining in the extracted fiber was determined by combustion. The remaining samples, grapes and grape foliage, were harvested at maturity. Only grapes are considered to be a raw agricultural commodity. Total residues at final harvest were determined by summing the initial acetonitrile surface wash and combustion of ground plant material prior to extraction. Extraction of the grapes and vines with acetonitrile resulted in the release of the majority of the remaining radioactive residue. The acetonitrile extracts of final harvest grapes were concentrated prior to chromatographic analysis.
Metabolites in the extractable residue were identified and quantified by TLC against a mix of putative metabolites.

2.2.2 Findings

Good agreement was obtained throughout from the duplicate samples of grape and foliage samples. The total residue in both treatment labels at each harvest and in each sample type (i.e. grape foliage and grapes) were very similar. In immature grape foliage, residues declined slightly between the initial treatment and just prior to the second treatment. The vast majority of residue at all timepoints was solvent extractable. Mean results of residue levels and the extraction profiles at each timepoint are presented in Table 1.
Analysis of the extractable residues showed a qualitatively similar metabolic profile in all foliage and grape samples. For the phenyl-label and the pyridyl-label treated grapes, total residues in the grapes at final harvest were 1.265 mg/kg and 1.040 mg/kg, respectively. The acetonitrile surface wash removed a significant amount of the radioactive residue (46.1-62.5 % TRR). Subsequent acetonitrile extraction removed an additional 33.2-48.0 % of the TRR, leaving 4.3-6.0 % non-extractable. The acetonitrile surface wash and the acetonitrile extract were concentrated before being analyzed.
Metabolites in the acetonitrile washes and acetonitrile extracts of the grape samples were identified and quantified by comparison with putative metabolites by reverse phase thin layer chromatography. Qualitative chromatography was performed on one-dimensional normal phase TLC. For the phenyl-label treated vines, fluopicolide was found in grapes at 91.2 % of TRR. Lesser amounts of AE C653711 (2.0 % TRR), and AE C643890 (0.2 % TRR) were also identified. For the pyridyl-label treated vines, fluopicolide was found in grapes at 87.4 % of TRR. Lesser amounts of AE C657188 (2.3 % TRR) were also identified. The small percentages of the TRR which are attributed to metabolites indicate that uptake and metabolism of fluopicolide from foliar surfaces does not greatly contribute to the nature of the residue in plants.
The proposed metabolic pathway of fluopicolide in grapevines is shown in Fig. 1. Fluopicolide is slowly metabolized in grapevines to AE C653711, AE C657188, and minor amounts of AE C643890.

2.3 Metabolism in lettuce

2.3.1 Method

Lettuce (var. Black Seeded Simpson) were grown under simulated field conditions in multiple steel crop tanks. Lettuce were treated two times with a foliar ap-

plication at nominal rates of 200 g a.i./ha per application with [U-^{14}C-phenyl]-fluopicolide and [2,6-^{14}C-pyridyl]-fluopicolide, formulated as a 20 % SC formulation. The cumulative treatment rate was equal to the proposed labelled maximum seasonal application of 400 g a.i./ha. The applications were made approximately six weeks after planting and 14 days prior to harvest maturity. In addition, one application of a soil drench at a nominal rate of 200 g a.i./ha per application with [U-^{14}C-phenyl]-fluopicolide formulated as a 20 % SC formulation was made approximately six weeks after planting.

Plants were harvested on Day Zero, immediately prior to the second treatment (immature harvest), and at final harvest maturity. Residues in the early lettuce leaf samples (Day Zero) from the foliar treated plants were recovered by surface wash with acetonitrile followed by extraction with acetonitrile. The residue remaining in the extracted fiber was determined by combustion. Residues in the immature harvest and final harvest lettuce samples from the foliar treatment areas were determined by summing the initial acetonitrile surface wash and combustion of ground plant material prior to extraction. Residues in the immature harvest and final harvest lettuce samples from the soil drench treatment areas were determined by combustion of ground plant material prior to extraction.

Extraction of the lettuce leaves with acetonitrile resulted in the release of the majority of the remaining radioactive residue. The acetonitrile extracts of lettuce leaves at all sampling events were concentrated prior to chromatographic analysis. All analyses were performed on duplicate samples of lettuce leaves. Metabolites in the extractable residue were identified and quantitated by TLC against a mix of putative metabolites.

2.3.2 Findings

Good agreement was obtained throughout from the duplicate samples of lettuce. No appreciable differences in total residues were observed between the phenyl-label treated lettuce and the pyridyl-label treated lettuce when the treatment was applied as a foliar application. The total residues in both treatment labels at each harvest were very similar. In immature lettuce leaves from the foliar treatment areas, residues declined significantly between the initial application and just prior to the second application, with initial mean residue levels of 10.837-13.359 mg/kg at Day Zero declining to 1.307-1.327 mg/kg at day 21 (pre-second treatment). At final harvest, the mean residue in lettuce leaves from the foliar treatment areas was 13.385-14.503 mg/kg. Significant differences in total residues were observed between the phenyl-label foliar application and the soil drench. In immature lettuce leaves, the TRR for the soil drench applied treated lettuce was only 0.076 mg/kg and the mean residue at final harvest 0.175 mg/kg. The vast majority of residue (> 95 % of TRR) at all timepoints was solvent extractable. Mean results of residue levels and the extraction profiles at each timepoint are presented in the Table 2.

Analysis of the final harvest extractable residues showed a qualitatively similar metabolic profile in lettuce samples. For the phenyl-label and the pyridyl-label foliar treated lettuce, total residues in the lettuce at final harvest were 13.385 mg/kg and 14.503 mg/kg, respectively. The ace-

tonitrile surface wash removed a significant amount of the radioactive residue (84.0-84.6 % TRR). Subsequent acetonitrile extraction removed an additional 14.8-15.1 % of the TRR, leaving 0.7-1.0 % non-extractable. The acetonitrile surface wash and the acetonitrile extract were concentrated before being analyzed.

For the phenyl-label soil drench treated lettuce, total residues in the lettuce at final harvest were 0.175 mg/kg. Acetonitrile extraction released 95.9 % of the TRR, leaving 4.1 % non-extractable. The acetonitrile extract were concentrated before being analyzed.

Metabolites in the acetonitrile washes and acetonitrile extracts of the lettuce samples were identified and quantified by comparison with putative metabolites by reverse phase thin layer chromatography. Qualitative chromatography was performed on one-dimensional normal phase TLC. In the foliar phenyl-label treated lettuce, fluopicolide was found at 95.9 % of the TRR while in the soil drench treated lettuce, the parent compound was found at 71.7 % of TRR. The major metabolite found in the soil drench treated lettuce was AE C653711 at 19.8 % of TRR which is presumably the result of uptake from the soil, whereas in the foliar applied treated lettuce, AE C653711 was found at 0.9 % of TRR indicating that uptake and metabolism of fluopicolide from foliar surfaces does not greatly contribute to the nature of the residue in plants. The metabolite AE C643890 was found at 2.8 % of TRR in the soil drench treated lettuce and was not found in the foliar applied treated lettuce. For the pyridyl-label foliar treated lettuce, fluopicolide was found at 96.4 % of TRR. Lesser amounts of AE C657188 (0.6 % TRR) were also identified.

The proposed metabolic pathway of fluopicolide in lettuce is the same as in potatoes or grapes (Fig. 1). Fluopicolide is slowly metabolized in lettuce to AE C653711, AE C657188, and AE C643890. Fluopicolide is metabolized in soil to AE C653711, which is taken up by the lettuce plant.

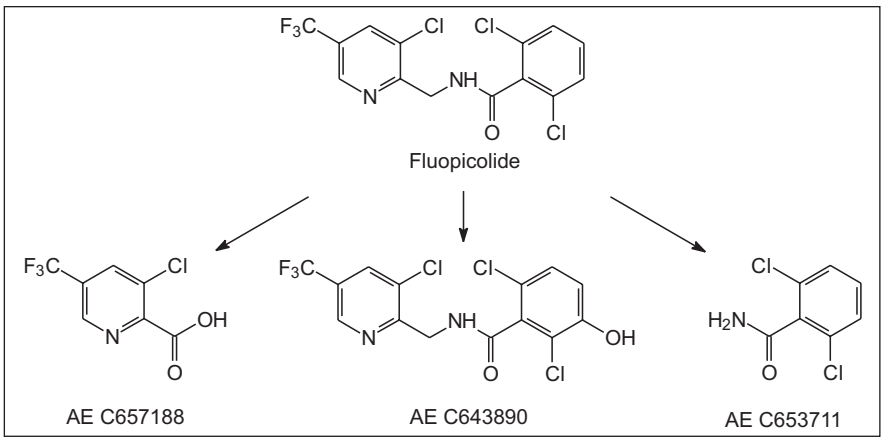


Fig. 1: Proposed metabolic pathway of fluopicolide in potatoes, grapes and lettuce.

Table 2: Distribution of fluopicolide and metabolites in lettuce.

Radiolabel	[U-^{14}C-phenyl]		[2,6-^{14}C-pyridyl]		[U-^{14}C-phenyl]	
Application type	Foliar				Soil drench	
Application rate (g/ha)	2 x 200				1 x 200	
Pre-harvest interval (d)	14				35	
Commodity	Leaves					
TRR (mg/kg)	13.385		14.503		0.175	
	mg/kg[a]	% TRR	mg/kg	% TRR	mg/kg	% TRR
Fluopicolide	12.843	95.9	13.979	96.4	0.128	71.7
AE C653711	0.112	0.9	n.a.	n.a.	0.034	19.8
AE C657188	n.a.	n.a.	0.078	0.6	n.a.	n.a.
AE C643890	n.d.	n.d.	n.d.	n.d.	0.005	2.8
Total identified	**12.955**	**96.8**	**14.056**	**97.0**	**0.167**	**94.3**
Total unidentified	0.335	2.5[b]	0.280	2.0[b]	0.003	1.6[b]
Non-extractable residue (NER)	0.089	0.7	0.140	1.0	0.007	4.1
Total	**13.385**	**100**	**14.503**	**100**	**0.175**	**100**

n.a.: not applicable, n.d.: not detected
[a] Results expressed as mg equivalent fluopicolide per kilogram fresh weight
[b] No single metabolite comprised > 1 % of TRR

2.4 Confined rotational crops

2.4.1 Method

The extent and nature of residue uptake by crops grown in soil previously treated with [^{14}C]-fluopicolide at the maximum expected annual rate and planted after various time intervals has been investigated. Bare loamy sand soil was treated at a rate of 400 g a.i./ha (proposed annual maximum use rate) with [U-^{14}C-phenyl]-fluopicolide or [2,6-^{14}C-pyridyl]-fluopicolide. Rotational crops were planted 29 days, 133 days and 365 days after treatment and grown to maturity. Crops included a leafy vegetable (lettuce), a root crop (radishes) and a small grain (wheat). In addition, soil cores from critical timepoints (treatment, planting and harvest) were taken.

Raw Agricultural Commodities (RACs) were harvested at an immature stage (forage) in wheat, and at maturity in all crops. At maturity, radishes were divided into roots and tops, and wheat was divided into straw (including hulls) and grain. Total residues in RAC were determined by combustion. Residues were characterized as extractable with acetonitrile, acetonitrile/water, acetonitrile/water Soxhlet, or remaining fiber (bound). Wheat straw and forage samples from the [U-^{14}C-phenyl]-fluopicolide treatments containing significant (> 0.05 mg/kg) residues were subjected to sequential hydrolysis with 1 M HCl (approx. 50 °C for 24 hours) and 2 M NaOH (reflux for 2 hours) to release bound residues. Metabolites were identified and quantified by reverse-phase high performance liquid chromatography and confirmed by LC-MS.

2.4.2 Findings

Total radioactive residues in RACs are given in Table 3. Total residues declined with greater soil aging. Mean residues in 29 day RAC ranged from 0.09 mg/kg (radish root) to 13.56 mg/kg (wheat straw), but residues declined greatly in

Table 3: Mean TRR in crops grown in soil treated with fluopicolide at a rate of 400 g a.i./ha and aged for 29, 133 and 365 days prior to planting.

Radiolabel	[U-^{14}C-phenyl]			[2,6-^{14}C-pyridyl]		
RAC	TRR (mg/kg) [a]			TRR (mg/kg)		
	29 DAT [b]	133 DAT	365 DAT	29 DAT	133 DAT	365 DAT
Lettuce	1.01	0.10	0.53	0.27	0.03	0.05
Radish tops	6.40	0.23	1.75	1.96	0.23	0.40
Radish roots	0.13	0.02	0.03	0.09	0.02	0.02
Immature wheat	4.95	0.22	0.86	4.29	0.16	0.24
Wheat grain	0.16	0.02	0.05	2.60	0.10	0.18
Wheat straw	13.56	0.84	2.37	7.05	0.35	1.01

[a] Results expressed as mg equivalent fluopicolide per kilogram fresh weight.
[b] DAT: Days after treatment

Table 4a: Distribution of the TRR in Raw Agricultural Commodities upon extraction; [U-^{14}C-phenyl] label.

Plot (day)	Crop part	Initial	CH_3CN		CH_3CN/water		CH_3CN/water Soxhlet		NER (fiber)	
		mg/kg [a]	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
29	Lettuce	1.013	0.939	92.7	n.d.	n.d.	0.055	5.4	0.019	1.9
	Radish tops	6.705	6.346	94.6	0.253	3.8	0.060	0.9	0.046	0.7
	Radish roots	0.143	0.127	88.8	n.d.	n.d.	0.012	8.6	0.004	2.6
	Wheat forage	4.949	3.803	76.8	n.d.	n.d.	0.920	18.6	0.226	4.6
	Wheat grain	0.158	0.052	32.7	0.080	50.8	0.005	3.0	0.021	13.5
	Wheat straw	13.560	4.823	35.6	4.202	31.0	0.655	4.8	3.881	28.6
133	Lettuce	0.115	0.109	95.0	0.002	1.5	n.d.	n.d.	0.004	3.5
	Radish tops	0.240	0.225	93.4	0.012	4.9	n.d.	n.d.	0.004	1.7
	Radish roots	0.023	0.021	89.9	0.002	6.9	n.d.	n.d.	0.001	3.2
	Wheat forage	0.225	0.178	79.3	0.025	10.9	0.007	3.1	0.015	6.7
	Wheat grain	0.020	0.010	49.4	0.003	14.8	n.d.	n.d.	0.007	35.8
	Wheat straw	0.843	0.537	63.7	0.159	18.9	0.017	2.0	0.130	15.4
365	Lettuce	0.619	0.590	95.3	0.010	1.7	n.d.	n.d.	0.019	3.0
	Radish tops	2.006	1.928	96.1	0.053	2.7	n.d.	n.d.	0.024	1.2
	Radish roots	0.036	0.034	95.0	< 0.001	1.2	n.d.	n.d.	0.001	3.9
	Wheat forage	0.865	0.677	78.2	0.103	11.9	0.023	2.6	0.063	7.3
	Wheat grain	0.054	0.004	7.2	0.027	50.4	0.004	8.1	0.019	34.3
	Wheat straw	2.373	0.741	31.2	0.636	26.8	0.161	6.8	0.835	35.2

[a] Results expressed as mg equivalent fluopicolide per kilogram fresh weight.
n.d.: not determined

the 133 day and 365 day aging periods. The 133 Day crop residues ranged from 0.02 mg/kg (radish root) to 0.84 (wheat straw). The 365 Day crop residues were observed to increase slightly, ranging from 0.02 mg/kg (radish root) to 2.37 mg/kg (wheat straw). This is considered to be a result of seasonal variation. The 133 Day plots were planted in October and developed through the winter. In contrast, the 365 Day plots were planted in March and developed through the summer when the plants would be more metabolically active. The soil residues showed a general decline after treatment, although there was some variability in the results.

The distribution of radioactivity following extraction of crop samples is given in Table 4. Residues in all RACs were characterized as extractable with acetonitrile, extractable by acetonitrile/water (1:1), with acetonitrile/water Soxhlet, or non-extractable. The percent of extractable residues was generally consistent in each RAC across the aging periods. In most RACs, the large majority of radioactivity was recovered in the acetonitrile extract. The exceptions were wheat grain and straw, where a significant percentage was found in the more polar extracts, and which had a higher percentage of non-extractable residues. There were four RACs with non-extractable residue greater than 10 % and greater than 0.05 mg/kg:

Table 4b: Distribution of the TRR in Raw Agricultural Commodities upon extraction; [2,6-^{14}C-pyridyl] label.

Plot (day)	Crop part	Initial	CH_3CN		CH_3CN/water		CH_3CN/water Soxhlet		NER (fiber)	
		mg/kg [a]	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
29	Lettuce	0.302	0.263	87.1	n.d.	n.d.	0.025	8.1	0.015	4.8
	Radish tops	2.097	1.812	86.4	n.d.	n.d.	0.260	12.4	0.025	1.2
	Radish roots	0.116	0.106	91.2	n.d.	n.d.	0.007	6.3	0.003	2.5
	Wheat forage	4.288	3.394	79.1	n.d.	n.d.	0.789	18.4	0.105	2.5
	Wheat grain	2.600	0.842	32.4	1.533	58.9	0.051	2.0	0.175	6.7
	Wheat straw	7.054	3.851	54.6	2.451	34.7	0.314	4.4	0.438	6.2
133	Lettuce	0.034	0.031	91.3	0.002	5.5	n.d.	n.d.	0.001	3.2
	Radish tops	0.237	0.215	90.7	0.020	8.3	n.d.	n.d.	0.002	1.0
	Radish roots	0.025	0.022	87.2	0.002	7.9	n.d.	n.d.	0.001	4.9
	Wheat forage	0.156	0.122	78.2	0.026	17.0	0.003	2.0	0.004	2.8
	Wheat grain	0.096	0.039	40.8	0.051	53.2	n.d.	n.d.	0.006	6.0
	Wheat straw	0.348	0.229	66.0	0.083	24.0	0.012	3.6	0.023	6.5
365	Lettuce	0.058	0.046	80.5	0.005	9.1	n.d.	n.d.	0.006	10.4
	Radish tops	0.419	0.356	84.9	0.049	11.6	n.d.	n.d.	0.015	3.5
	Radish roots	0.032	0.027	85.1	0.003	9.9	n.d.	n.d.	0.002	5.0
	Wheat forage	0.243	0.197	81.1	0.031	12.6	n.d.	n.d.	0.015	6.3
	Wheat grain	0.178	0.013	7.3	0.147	82.4	0.008	4.6	0.010	5.8
	Wheat straw	1.009	0.348	34.6	0.448	44.4	0.084	8.4	0.128	12.7

[a] Results expressed as mg equivalent fluopicolide per kilogram fresh weight.
n.d.: not determined

29 Day wheat straw (phenyl-label), 133 Day straw (phenyl-label), and 365 Day straw (both labels). These fibers were subjected to extraction with 1 N HCl followed by 2 N NaOH. Radioactive residue which remained non-extractable after sequential treatment with acid and base was less than 0.05 mg/kg, except in 29 Day wheat straw (phenyl-label) where 0.319 mg/kg represented 2.4 % of TRR.

Initial characterization of plant extracts was performed by thin layer chromatography (TLC) and included analysis to establish storage stability of samples. However, the chromatographic properties of the crude extracts were poor in some cases. Although resolution of the reference compounds on TLC was good, components of the crop extracts were not sufficiently resolved for identification and quantitiation. Therefore, extracts of all the RACs were subjected to concentration and filtration to prepare them sufficiently for analysis by RP-HPLC. LC-MS was utilized to confirm these identifications in representative extracts.

The distribution of metabolites identified by HPLC is shown in Table 5a and 5b.

Table 5a: Summary of metabolite identification and characterization of residues in lettuce, radish and wheat following 29, 133 and 365 Day plantback intervals; [U-^{14}C-phenyl] label.

Plot (day)	Crop part	Ext. residue	AE C657378		AE C653711		AE C643890	
		% TRR	% TRR	mg/kg [a]	% TRR	mg/kg	% TRR	mg/kg
29	Lettuce	98.1	n.d.	n.d.	81.2	0.822	n.d.	n.d.
	Radish tops	98.4	n.d.	n.d.	65.3	4.381	n.d.	n.d.
	Radish roots	97.4	n.d.	n.d.	43.2	0.062	n.d.	n.d.
	Wheat forage	95.4	32.7	1.619	6.3	0.312	< 1.0	< 0.049
	Wheat grain	79.9	n.d.	n.d.	3.6	0.006	13.1	0.021
	Wheat straw	71.4	13.6	1.844	3.4	0.461	n.d.	n.d.
133	Lettuce	96.5	n.d.	n.d.	60.9	0.070	n.d.	n.d.
	Radish tops	98.3	n.d.	n.d.	77.3	0.186	n.d.	n.d.
	Radish roots	96.8	n.d.	n.d.	54.9	0.013	n.d.	n.d.
	Wheat forage	92.3	28.9	0.065	5.1	0.011	n.d.	n.d.
	Wheat grain	65.3	23.3	0.004	19.0	0.003	n.d.	n.d.
	Wheat straw	84.6	14.6	0.123	25.5	0.215	n.d.	n.d.
365	Lettuce	97.0	n.d.	n.d.	87.0	0.539	n.d.	n.d.
	Radish tops	98.8	n.d.	n.d.	87.5	1.755	n.d.	n.d.
	Radish roots	96.2	n.d.	n.d.	60.9	0.022	n.d.	n.d.
	Wheat forage	92.7	59.3	0.513	14.8	0.128	n.d.	n.d.
	Wheat grain	65.7	24.5	0.013	17.9	0.010	n.d.	n.d.
	Wheat straw	64.8	28.0	0.663	5.1	0.121	n.d.	n.d.

n.d.: not detected

[a] Results expressed as mg equivalent fluopicolide per kg fresh weight

[b] Total Identified: sum of identified metabolites and total conjugates

The principal metabolites identified in phenyl-labelled experiments were fluopicolide, AE C653711, and in the wheat only AE C657378 (3-hydroxy-AE C653711). AE C643890, which is the 3-hydroxy derivative of the parent, was detected at a quantifiable level only in 29 Day wheat grain and forage (13.1 %, 0.021 mg/kg and 1.0 %, 0.049 mg/kg parent equivalents, respectively).

The principal metabolites identified in pyridyl-labelled experiments included fluopicolide, AE C657188 and AE 1344122. AE C653598, the amide congener of AE C657188, was detected in some 365 Day RACs and in 133 Day straw, but not exceeding 9.5 % or 0.028 mg/kg (0.048 mg/kg parent equivalents). AE C643890 was detected in 29 Day wheat forage at 1.4 %, 0.060 mg/kg. Finally, AE B102859 was detected in some RACs at a maximum concentration of 0.052 mg/kg (0.1 mg/kg parent equivalents).

Subsequent field residue trials in rotational crops showed significantly lower residues than those seen in the confined rotational crop study. As an example, no residues of fluopicolide in wheat grain were observed above the limit of quantification of 0.01 mg/kg.

In addition to the unconjugated metabolites, several conjugated species were fully characterized (in wheat in which they were most abundant) by LC-MS. The proposed structures are hydroxylated or sulfhydrylated versions of the parent, conjugated to glucose, malonic acid, glyceric acid or amino acids. In several of the remaining RACs, two regions of radioactivity were observed and labelled region A and B in the corresponding HPLC chromatographs. Direct comparison was not possible, but based on relative retention times, the components in region A and B were found to correspond to conjugates characterized in 29 Day wheat samples.

The proposed major metabolic pathway for fluopicolide in rotational crops is given in Fig. 2. Fluopicolide and its metabolites AE C653711 (from the phenyl ring) and AE C657188 (from the pyridyl ring) were detected in all crops. AE C653711 and AE C657188 were not thought to be formed in plants di-

	Fluopicolide		Total conjugates		Largest single unknown		Total IDed [b]
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
	11.1	0.112	n.d.	n.d.	1.2	0.012	92.3
	24.5	1.644	n.d.	n.d.	1.7	0.116	89.8
	47.9	0.069	n.d.	n.d.	1.1	0.002	91.1
	36.6	1.812	19.6	0.97	n.d.	n.d.	95.4
	27.3	0.043	n.d.	n.d.	13.4	0.021	44.0
	23.1	3.132	25.5	3.458	3.6	0.488	65.7
	26.6	0.031	n.d.	n.d.	1.9	0.002	87.5
	15.1	0.036	n.d.	n.d.	1.7	0.004	92.4
	28.2	0.006	n.d.	n.d.	6.4	0.001	83.1
	23.3	0.052	21.0	0.047	4.8	0.011	78.3
	7.0	0.001	n.d.	n.d.	4.5	0.001	49.3
	15.5	0.131	17.9	0.150	4.7	0.040	73.5
	2.1	0.013	n.d.	n.d.	2.4	0.015	89.1
	3.8	0.076	n.d.	n.d.	1.5	0.030	91.3
	24.2	0.009	n.d.	n.d.	2.4	0.001	85.1
	4.8	0.042	3.0	0.026	4.3	0.037	81.9
	7.3	0.004	n.d.	n.d.	3.1	0.002	49.7
	7.2	0.172	4.9	0.116	3.5	0.083	45.2

rectly but formed part of the crop residue by plant uptake from soil. AE C657188 is known to be rapidly metabolized to AE 1344122 in soil. No metabolites other than AE C653711 have been detected in soil arising from the phenyl ring.

Fluopicolide taken up by rotational crops was further metabolized to form hydroxylated or thiolated (addition of -SH) versions, and then formed a variety of conjugates with glucose, malonic acid, glyceric acid or amino acids. Occasionally very low concentrations of the unconjugated hydroxylated fluopicolide (AE C643890) were observed in wheat. These plant metabolic processes were most significant in wheat forage and straw. All of the derivatization observed was associated with the phenyl ring of the fluopicolide structure. In the [2,6-^{14}C-pyridyl]-labelled experiment some derivatization and conjugation was observed in lettuce and radish (tops only), while the equivalent concentration in the [U-^{14}C-phenyl]-labelled experiment represented such low percentages of the TRR that they were not resolved from background radioactivity.

The pyridyl ring metabolites AE C657188, AE 1344122, AE C653598

Table 5b: Summary of metabolite identification and characterization of residues in lettuce, radish and wheat following 29, 133 and 365 Day plantback intervals; [2,6-^{14}C-pyridyl] label.

Plot (day)	Crop part	Ext. residue	AE C653598		AE 1344122		AE C657188	
		% TRR	% TRR	mg/kg [a]	% TRR	mg/kg	% TRR	mg/kg
29	Lettuce	95.2	n.d.	n.d.	13.0	0.039	17.4	0.053
	Radish tops	98.8	n.d.	n.d.	3.3	0.069	10.4	0.217
	Radish roots	97.5	n.d.	n.d.	9.6	0.011	33.5	0.039
	Wheat forage	97.5	n.d.	n.d.	3.8	0.163	43.0	1.844
	Wheat grain	93.3	n.d.	n.d.	13.1	0.341	69.6	1.809
	Wheat straw	93.8	n.d.	n.d.	7.7	0.544	7.0	0.494
133	Lettuce	96.8	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	Radish tops	99.0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	Radish roots	95.1	n.d.	n.d.	2.9	0.001	9.6	0.002
	Wheat forage	97.2	n.d.	n.d.	41.0	0.064	5.4	0.008
	Wheat grain	94.0	n.d.	n.d.	66.6	0.064	10.9	0.010
	Wheat straw	93.5	9.4	0.033	1.2	0.004	2.1	0.007
365	Lettuce	89.6	9.0	0.005	7.8	0.005	11.8	0.007
	Radish tops	96.5	n.d.	n.d.	5.1	0.022	27.1	0.114
	Radish roots	95.0	9.5	0.003	5.3	0.002	10.0	0.003
	Wheat forage	93.7	6.3	0.015	18.3	0.045	8.2	0.020
	Wheat grain	94.2	n.d.	n.d.	64.9	0.116	14.2	0.025
	Wheat straw	87.3	4.8	0.048	14.2	0.143	4.1	0.042

n.d.: not detected

[a] Results expressed as mg equivalent fluopicolide per kg fresh weight

[b] Total Identified: sum of identified metabolites and total conjugates

and AE B102859 were observed in rotational crops. AE C657188 and AE 1344122 are known to be formed in small quantities in soil and may have been taken up from the soil directly. In lettuce and radish AE C657188 was usually the most abundant pyridyl ring metabolite. In wheat forage and grain the percentage of AEC657188 declined after the 29 day planting with the percentage of AE 1344122 detected increasing at both the 133 and 365 Day plantings, although more importantly the concentrations were lower with time.

AE C653711 taken up by wheat grown as a rotational crop was further metabolized to form AE C657378 (3-hydroxy-AE C653711). Further derivatives of the phenyl ring were observed in wheat forage including glucose-malonoic acid conjugates of AE C657378. AE C653711 was detected in all rotational crops but further metabolism of this metabolite to AE C657378 or conjugates of AE C657378 was only observed in wheat. The concentrations (in mg/kg) of fluopicolide, of derivatives and conjugates of hydroxylated or thiolated versions and the small amounts of AE C643890 (hydroxylated fluopicolide) in individual

	AE B102859		AE C643890		Fluopicolide		Total conjugates		Largest single unknown		Total IDed [b]
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
	5.3	0.016	n.d.	n.d.	35.8	0.108	4.6	0.014	10.5	0.032	76.1
	4.8	0.100	n.d.	n.d.	51.1	1.072	13.7	0.287	11.3	0.238	83.3
	n.d.	n.d.	n.d.	n.d.	41.1	0.048	n.d.	n.d.	4.0	0.005	84.2
	n.d.	n.d.	1.4	0.060	33.7	1.445	13.4	0.566	1.7	0.073	95.3
	n.d.	n.d.	n.d.	n.d.	1.8	0.046	n.d.	n.d.	1.9	0.048	84.5
	n.d.	n.d.	n.d.	n.d.	34.9	2.462	40.9	2.884	2.2	0.155	90.5
	n.d.	n.d.	n.d.	n.d.	79.9	0.027	n.d.	n.d.	4.8	0.002	79.9
	n.d.	n.d.	n.d.	n.d.	72.2	0.171	n.d.	n.d.	8.3	0.020	72.2
	19.1	0.005	n.d.	n.d.	54.9	0.014	n.d.	n.d.	1.6	<0.001	86.5
	10.5	0.016	n.d.	n.d.	26.2	0.041	n.d.	n.d.	2.9	0.005	83.1
	n.d.	n.d.	n.d.	n.d.	3.2	0.003	n.d.	n.d.	5.0	0.005	80.7
	21.5	0.075	n.d.	n.d.	25.7	0.089	25.7	0.089	2.7	0.009	85.6
	3.7	0.002	n.d.	n.d.	41.5	0.024	6.7	0.004	1.3	0.001	80.5
	6.0	0.025	n.d.	n.d.	25.2	0.106	21.3	0.089	4.7	0.020	84.7
	n.d.	n.d.	n.d.	n.d.	55.8	0.018	n.d.	n.d.	3.5	0.001	80.6
	9.9	0.024	n.d.	n.d.	27.8	0.068	4.1	0.010	4.4	0.011	74.6
	n.d.	n.d.	n.d.	n.d.	2.9	0.005	n.d.	n.d.	2.2	0.004	82.0
	n.d.	n.d.	n.d.	n.d.	27.5	0.277	19.3	0.195	4.1	0.041	69.9

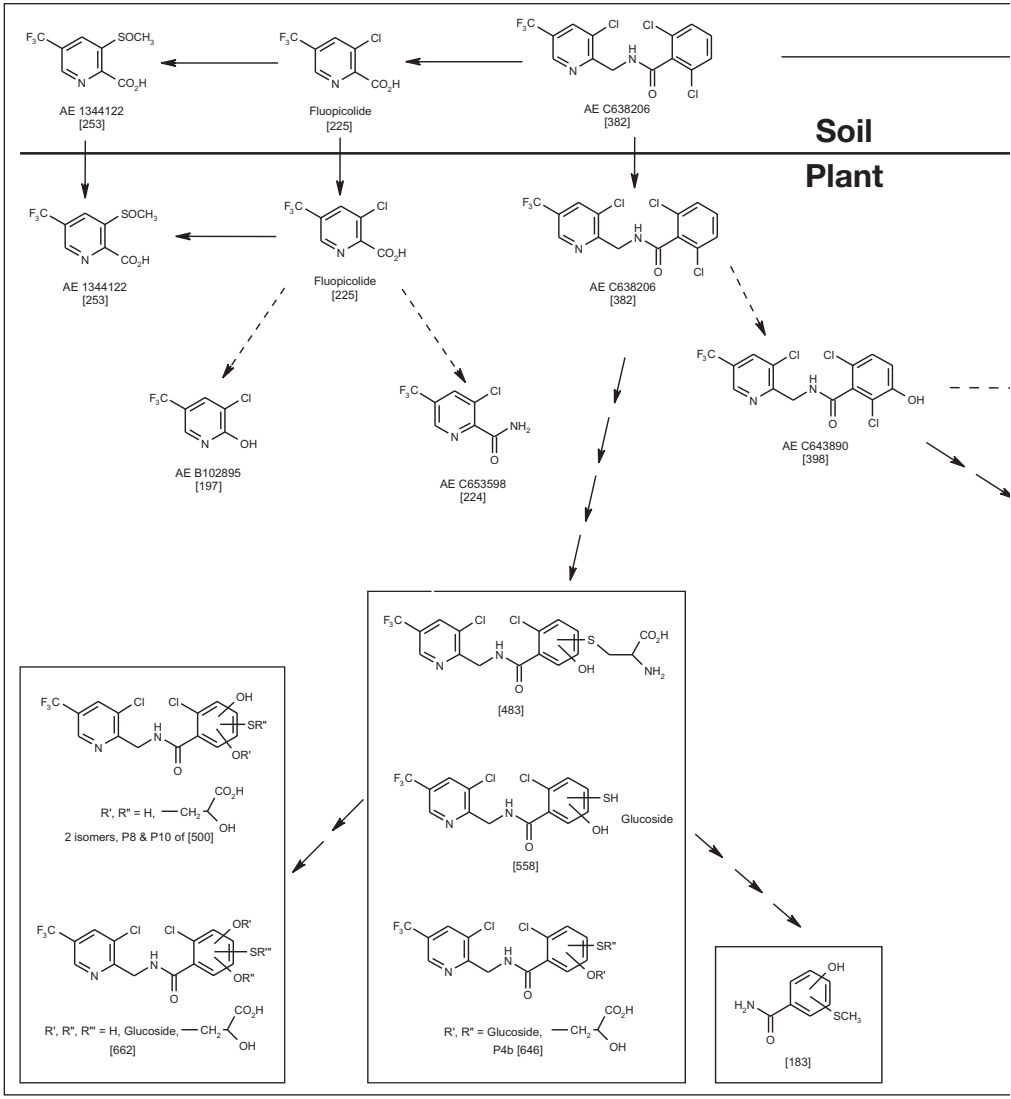
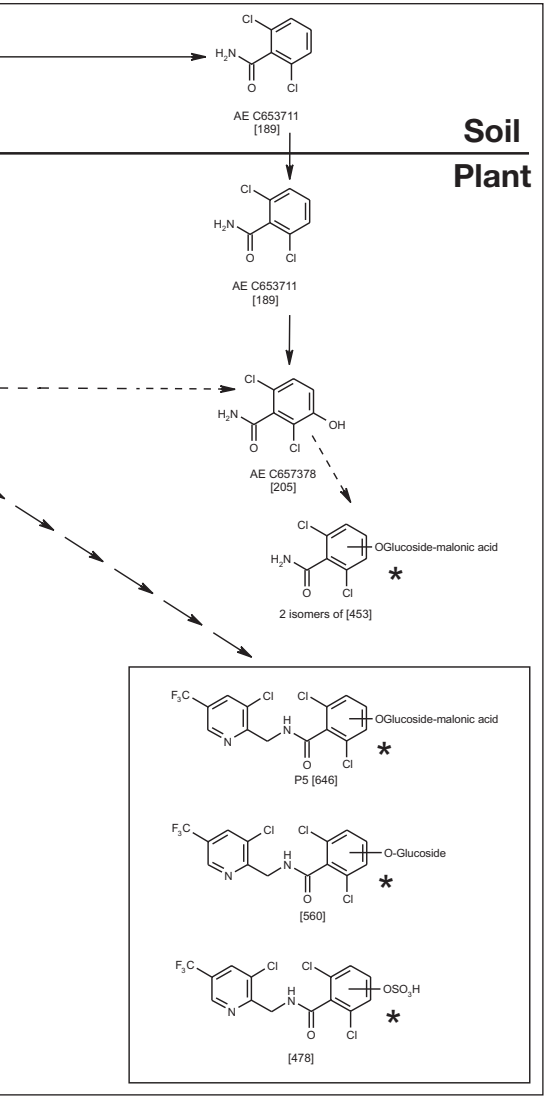


Fig. 2: Proposed pathway of metabolism of fluopicolide in rotational crops. Drawn arrows: major pathway, dotted arrows: minor pathway, molar mass in squared brackets. *: It was not possible to define the position of substituents in the phenyl ring by mass spectroscopy. Co-chromatography with reference standards confirmed the hydroxyl groups in AE C643890 and AE C657378 as being in the 3-position and therefore the most probable position of the conjugates in the structures marked (*) is also in the 3-position.



RACs were similar at each harvest in the [U-^{14}C-phenyl]-labelled and [2,6-^{14}C-pyridyl]-labelled experiments.

This was not true for metabolites formed from the individual pyridyl or phenyl rings and thus the TRR in individual RACs at each harvest in the [U-^{14}C-phenyl]-labelled and [2,6-^{14}C-pyridyl]-labelled experiments were different. The metabolites arising from the phenyl ring, AE C653711 and AE C657378 (3-hydroxy-AE C653711), formed a larger proportion of the TRR with time (although the concentrations were lower with time) in the individual RACs compared to metabolites arising from the pyridyl ring. AE C657188 and AE 1344122 are much more rapidly metabolized in soil than AE C653711 and higher concentrations of the phenyl ring metabolites would be expected in rotational crops based on the different stabilities of the ring structures in soil. An exception to this was observed in wheat grain where the concentrations in the [2,6-^{14}C-pyridyl]-labelled experiment were higher, because AE C657188 and AE 1344122 (both pyridine carboxylic acid moieties) were transported to grain, as expected from the known phloem mobility of aromatic carboxylic acids.

3 Summary

Behaviour of fluopicolide in plants

The metabolic pathway of fluopicolide was similar in three contrasting primary crops (potatoes, grapes and lettuce). Residues were comprised of fluopicolide, AEC653711 (from the phenyl ring), AE C657188 (from the pyridyl ring) and AE C643890 (hydroxylated fluopicolide). The major component of the residue in all plant parts was fluopicolide. Significant amounts of other metabolites were only detected in potato tubers (AE C657188 and AE C653711) or in lettuce grown in soil treated with the test item (AE C653711 only). Both metabolites were not thought to be formed in plants directly but formed part of the crop residue by plant uptake from soil. AE C643890 was detected only as a very minor component.

The metabolic pattern in succeeding crops is significantly more complex. When applied to bare soil, fluopicolide was readily taken up by crops in the xylem system and metabolized to numerous related components in rotational crops. Residue components identified in succeeding crops included unchanged fluopicolide, the known soil degradates AE C653711, AE C657188 and AE 1344122 (resulting from uptake from soil), AE C657378 (3-hydroxy-AE C653711), AE C653598 (the amide congener of AE C657188), AE B102859 (decarboxylated AE C657188), and AE C643890 (hydroxylated fluopicolide). In addition, numerous conjugates were fully characterized utilizing LC-MS. Levels of individual components which were not identified or characterized did not exceed 10 % of total radioactive residues.

The behaviour and nature of the residue arising from the use of fluopicolide in a range of contrasting target crops and rotational crops has been fully elucidated.

Zusammenfassung

Untersuchungen zum Verhalten von Fluopicolide in Pflanzen

Der Abbau von Fluopicolide war in den drei untersuchten Hauptkulturen Kartoffeln, Wein und Salat ähnlich. Folgende Rückstände wurden identifiziert: Fluopicolide, AE C653711 (vom Phenylring), AE C657188 (vom Pyridylring) und AE C643890 (hydroxyliertes Fluopicolide). Hauptbestandteil der gefundenen Rückstände in allen Pflanzenteilen war Fluopicolide. Signifikante Mengen anderer Metaboliten wurden nur in Kartoffelknollen gefunden (AE C657188 und AE C653711) oder in Salat (nur AE C653711), der auf mit der Prüfsubstanz behandelten Böden angebaut wurde. Das Auftreten dieser beiden Metaboliten war in Pflanzen nicht zu vermuten, vielmehr ist der Nachweis im Rahmen der Gesamtrückstände auf die Aufnahme der Substanzen aus dem Boden zurückzuführen. In nur geringfügigen Mengen wurde der Metabolit AE C643890 nachgewiesen.

Das metabolische Abbauverhalten in nachfolgenden Kulturen ist deutlich komplexer. Bei Applikation von Fluopicolide auf den blanken Boden wurde der Wirkstoff umgehend von den Pflanzen in das Xylem aufgenommen und zu zahlreichen ähnlichen Substanzen metabolisiert. Unter den in den Nachfolgekulturen nachgewiesenen Metaboliten befanden sich Fluopicolide, die bekannten Bodenabbausubstanzen AE C653711, AE C657188

und AE 1344122 (resultierend aus der Aufnahme aus dem Boden), AE C657378 (3-Hydroxy-AE C653711), AE C653598 (die Amido-Verbindung des AE C657188), AE B102859 (decarboxyliertes AE C657188) und AE C643890 (hydroxyliertes Fluopicolide). Darüber hinaus wurden unter Verwendung von LC-MC zahlreiche Konjugationen charakterisiert. Der Anteil einzelner Substanzen, die nicht identifiziert oder charakterisiert werden konnten, betrug nicht mehr als 10 % der nachgewiesenen radioaktiven Gesamtrückstände.
In den vorliegenden Untersuchungen konnte das Verhalten und die Identität der Rückstände, die nach Anwendung von Fluopicolid in einer Reihe unterschiedlicher Ziel- und Nachbaukulturen auftraten, vollständig aufgeklärt werden.

Résumé

Le comportement du fluopicolide dans les plantes

La voie métabolique du fluopicolide était analogue dans trois cultures primaires différentes (pomme de terre, vigne et laitue). Les résidus étaient constitués de fluopicolide, de l'AEC653711 (provenant de l'anneau phényl), de l'AE C657188 (provenant de l'anneau pyridyl) et de l'AE C643890 (fluopicolide hydroxylé). Dans toutes les plantes, le principal composant des résidus était le fluopicolide. D'importantes quantités d'autres métabolites n'ont été détectées que dans les tubercules de pomme de terre (AE C657188 et AE C653711) ou dans les laitues cultivées dans des sols traités avec le produit testé (AE C653711 uniquement). On ne pense pas que les métabolites aient pu se former directement dans les plantes mais plutôt qu'ils faisaient partie des résidus de récolte assimilés par la plante à partir du sol. L'AE C643890 n'a été détecté qu'en tant que composé très mineur.
Dans les cultures successives, le profil métabolique est beaucoup plus complexe. Lorsqu'il est appliqué sur le sol nu, le fluopicolide était facilement assimilé par les cultures et transporté au niveau du xylème, puis métabolisé en de nombreux composés analogues dans les cultures de la rotation. Les résidus identifiés dans les cultures successives comprenaient du fluopicolide sous forme inchangée, les produits de dégradation dans le sol connus AE C653711, AE C657188 et AE 1344122 (résultant de l'assimilation par les racines), AE C657378 (3-hydroxy-AE C653711), AE C653598 (congénère amidique de AE C657188), AE B102859 (AE C657188 décarboxylé) et AE C643890 (fluopicolide hydroxylé). En outre, de nombreux conjugués ont été entièrement caractérisés en faisant appel à la CL-SM. Les taux de composés individuels qui n'ont été ni identifiés, ni caractérisés n'ont pas dépassé 10 % du total des résidus radioactifs.
Le comportement et la nature des résidus provenant de l'utilisation du fluopicolide sur un éventail de cultures cibles diversifiées et autres cultures de la rotation ont été entièrement élucidés.

Resumen

Comportamiento de fluopicolide en plantas

El proceso metabólico de fluopicolide fué similar en tres cultivos primarios disímiles (papas, vid y lechuga). Los residuos encontrados comprendieron a fluopicolide, AEC653711 (del anillo fenilo), AE C657188 (del anillo piridinilo) y

AE C643890 (fluopicolide hidroxilado). El mayor componente de los residuos en todas las partes de las plantas fué fluopicolide. Cantidades significativas de otros metabolitos se detectaron sólo en tubérculos de papas (AE C657188 y AE C653711) o en lechuga proveniente de suelos tratados con el compuesto experimental (AE C653711 solo). Ambos metabolitos supuestamente no se forman directamente en las plantas pero forman parte del residuo porque las plantas los absorben del suelo. AE C643890 se detectó solamente como un componente muy menor.

El proceso metabólico en cultivos sucesores es significativamente más complejo. Si se aplica al suelo, fluopicolide es rápidamente asimilado al xilema por las raíces de los cultivos rotacionales y metabolizado a numerosos componentes relacionados. Los componentes residuales identificados en cultivos sucesores incluyeron fluopicolide sin modificar, los compuestos conocidos de degradación en suelo AE C653711, AE C657188 y AE 1344122 (resultantes por absorción del suelo), AE C657378 (3-hidroxi-AE C653711), AE C653598 (el congénero amida de AE C657188), AE B102859 (AE C657188 descarboxilado), y AE C643890 (fluopicolide hidroxilado). Adicionalmente, numerosos conjugados fueron plenamente caracterizados utilizando LC-MS. Los niveles de componentes individuales que no fueron identificados o caracterizados no excedieron el 10 % del total de residuos radioactivos.

El comportamiento y naturaleza del residuo resultante del uso de fluopicolide en una serie de cultivos objetivo contrastantes y cultivos rotacionales ha sido plenamente elucidado.

Резюме

Исследование путей разложения флуопиколида в растениях

При исследовании разложения флуопиколида во всех трех основных исследованных культурах – картофель, виноград и салат – выявлена подобная схема. Идентифицированы следующие остатки: флуопиколид, AE C653711 (от фенильного кольца), AE C657188 (от пиридильного кольца) и AE C643890 (гидроксилированный флуопиколид). Основным компонентом найденных остатков во всех частях растений был флуопиколид. Заметные количества других метаболитов найдены только в клубнях картофеля (AE C657188 и AE C65371) и салате (AE C653711), возделанном на почвах, обработанных испытываемым продуктом. Появление этих двух метаболитов в растениях нельзя было предположить, а их обнаружение в рамках определения суммарного остатка связано с поглощением веществ из почвы. Метаболит AE C643890 был обнаружен только в незначительных количествах.

Характеристика метаболизма в последующих культурах заметно сложнее. При аппликации флоупиколида на голой почве действующее вещество сразу же поглощается растениями и переходит в ксилем с последующим разложением на многочисленные похожие друг на друга вещества. Среди обнаруженных в последующих культурах метаболитов были флуопиколид, известные субстанции разложения в почве AE C653711, AE C657188 и AE 1344122 (в результате поглощения из почвы), AE C657378 (3-гидрокси-AE C653711), AE C653598

(аминосоединение вещества AE C657188), AE B02859 (декарбоксилированное AE C657188) и AE C643890 (гидроксилированный флуопиколид). Кроме того, хромато-масс-спектрометрическим методом определены многочисленные конъюгации. Доля индивидуальных веществ, идентифицировать или охарактеризовать которых не удалось, составила не более 10 % от обнаруженных радиоактивных суммарных остатков.

В итоге представленных исследований полностью выявлены характеристика и идентичность остатков, появившихся после применения флуопиколида в ряде различных целевых и последующих культур.

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