Document Title

Summary of the fate and behaviour in the environment for fluopicolide

Part 1

Data Requirement(s)

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Section 7: Fate and behaviour in the environment

According to the Guidance Document SANCO/10181/2013 for applicants on preparing dossiers for the approval of a chemical active substance

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on behalf of

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[1] It is suggested that applicants adopt a similar approach to showing revisions and version history as outlined in SANCO/10180/2013 Chapter 4, 'How to revise an Assessment Report'.

Table of Contents

CA 7 FATE AND BEHAVIOUR IN THE ENVIRONMENT

Fluopicolide (AE C638206) was included in Annex I to Council Directive 91/414/EEC in 2010 (Commission Directive 2010/15/EU, Entry into Force on June 1, 2010). The expiration of approval of fluopicolide is May 31, 2023 (Commission Implementing Regulation (EU) 2017/1527). The Supplementary Dossier contains only data which were not submitted at the time of the Annex I inclusion of fluopicolide under Council Directive 91/414/EEC and which were therefore not evaluated during the first EU review. All data which were already submitted by Bayer AG (former Bayer CropScience) for the Annex I inclusion under Council Directive 91/414/EEC are contained in the Draft Assessment Report (DAR) and its Addenda, and are included in the Baseline Dossier provided by Bayer AG.

Fluopicolide is a fungicidal active substance developed by Bayer. It is the only active substance in Europe representing a class of chemistry (pyridinylmethyl-benzamides) with a unique mode of action via delocalization of a spectrin-like protein in the Oomycetes fungi.

Fluopicolide has a long track record of safe use in a large number of targeted crops within horticulture, e.g. cucumbers, lettuce and on arable crops (e.g. potato).

Fluopicolide is active against a wide range of Oomycete fungi, the causal agents of devastating plant diseases of economic importance in EU-27 such as potato late blight (*Phytophthora infestans*) or downy mildew diseases in a broad range of crops.

It provides effective, long lasting protection at low application rates against Oomycetes diseases at different stage of development of the fungi, giving flexibility of use to the farmer.

Fluopicolide can be formulated with other active ingredients in different types of formulations to optimise and complete its activity.

The development of resistances of Oomycetes against existing, well-established fungicide groups represent a threat for European farmers by increasing the complexity of their plant protection programs leading to severe economic impacts. With Fluopicolide, farmers in EU-27 have access to a modern tool for their integrated crop protection programs, contributing to effective and sustainable management of resistance development and preserving high level of protection against Oomycete diseases.

By reducing the Oomycete damages, applications of Fluopicolide on target crops contribute to the achievement of optimum yield and quality, thus securing sufficient supply of high-quality potatoes and horticultural produces for European consumer destinations and markets abroad, being it fresh or for the processing industry.

Fate and behaviour of fluopicolide in the environment

The fate and behaviour of fluopicolide in the environment have been investigated in a series of laboratory and outdoor studies using [^{14}C]-labelled compound. Experiments have also been conducted to investigate the behaviour of fluopicolide under field conditions. Additionally, where required, studies have been conducted on metabolites of fluopicolide.

Detailed summaries of studies on fate and behaviour in soil are provided in Document MCA 7, Part 1. An overview of soil DT_{50} values derived for fluopicolide and its metabolites in laboratory and field studies are provided below.

Summary of laboratory aerobic soil DT_{50} values for fluopicolide and its metabolites

Compound	Trigger endpoints DT_{50} range (un-normalised) (d)	No. datasets	No. soils	Modelling endpoints Geometric mean $DegT_{50}$ normalised to 20°C & pF2 (d)
Fluopicolide	47.7 - 1290	22	16	181.6
M-01 (AE C653711, BAM)	135.9 - 3461	26	18	569.5
M-02 (AE C657188, PCA)	0.7 – 4.4	7	7	1.6
M-03 (AE 0608000)	0.1 – 62.6	9	7	17.9 (pH <6) / 0.19 (pH ≥ 6)
M-05 (AE 1344122)	5.6 – 172.1	13	7	25.2
M-10 (AE 1344123)	3.6 – 1000	13	7	35.4
M-11/M-12	31.7 – 242.5	2	2	87.6
M-13	13.3 – 48.4	3	3	20.7
M-14 (AE 1388273)	4.9 – 21.7	5	3	9.4
M-15 (AE 1413903)	102.7 – 113.2	4	4	144.8
M-20 (BCS-BX16566)	2.0 - 144.7	6	6	6.1

Fluopicolide was oxidized in soil to form the metabolite M-03, which is cleaved to form the metabolites M-01 containing the phenyl ring and M-02 containing the pyridyl ring. The other metabolites listed above; M-05, M-10, M-11, M-12, M-13, M-14, M-15 and M-20 are minor soil metabolites, detected in aerobic soil metabolism studies conducted with M-02 or in leachate from a lysimeter study conducted with fluopicolide. An assessment which establishes the non-relevance of the fluopicolide metabolites in groundwater is provided in Document N4.

While fluopicolide and M-01 degraded slowly in laboratory studies other metabolites showed moderate to rapid rates of degradation, except for M-15 which was a very minor soil metabolite not detected in parent soil degradation studies. Experiments have been conducted to investigate the behaviour of fluopicolide and its metabolite M-01 under field conditions.

Summary of field DT_{50} values for fluopicolide and its metabolites

Compound	Trigger endpoints DT_{50} range (un-normalised) (d)	SFO DT_{50} range (un-normalised) (d)	No. sites	Modelling endpoints Geometric mean $DegT_{50}$ (normalised to 20°C & pF2) (d)
Fluopicolide	28 - 403	177.4 – 457.6	12	183
M-01 (AE C653711)	133 - 344	155 - 344	5	146

The degradation of rate of fluopicolide was similar under laboratory and field conditions, so an overall geometric mean $DegT_{50}$ value of 182 days was used in FOCUS modelling calculations. Lower-tier degradation study data for fluopicolide from laboratory and field studies have been evaluated to derive $DegT_{50eq}$ values, which when combined with the higher-tier aged-sorption values yield an overall geometric mean $DegT_{50eq}$ of 121 days for use in exposure modelling (in combination with the mean aged-sorption parameters: F_{ne} 0.508; k_{des} 0.0356).

Overall $DegT_{50eq}$ evaluation results

Compound	$DegT_{50eq}$ range (d)	No. datasets	Geometric mean $DegT_{50eq}$ (d)
Fluopicolide	45.4-532.5	33	121

Fluopicolide is of medium mobility in soil with a geometric mean K_{oc} value of 267.7 mL/g. The metabolites of fluopicolide were more mobile than their parent (geometric mean Koc values range from 0 to 106.9 mL/g). Detailed summaries of studies on adsorption to soil and mobility in soil are provided in Document MCA 7, Part 2, along with summaries of studies on fate and behaviour in water and sediment and in air.

In aquatic water sediment systems fluopicolide dissipated from the water by a combination of degradation and partitioning to the sediment. The compound was shown to be stable to hydrolysis, aqueous photolysis and aerobic mineralization in other studies. An overview of DT_{50} values in aquatic sediment systems derived for fluopicolide is provided below.

Summary of DT_{50} values for fluopicolide in aquatic / sediment systems

Phase	Trigger endpoints DT_{50} range at 20°C (d)	SFO DT_{50} range at 20°C (d)	No. datasets (n)	Geometric mean $DegT_{50}$ at 20°C (d)
Total system	856.3-1340.0	856.3 – 1340.0	2	1071.2
Water phase	5.6 - 228.9	57.0 [A] – 594.5 [B]	2	184.1

After removal of data points prior to the maximum in sediment (76.2% AR at DAT 85), only three data points remained for each system, which was insufficient to provide a robust, meaningful fit.
[A] Pseudo-SFO DT_{50} value derived from the FOMC DT_{90}/3.32
[B] Pseudo-SFO DT_{50} values derived from the slow phase of the DFOP fit

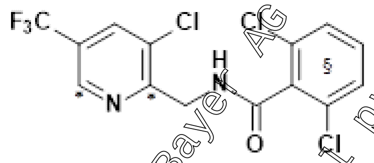
M-01 and M-02 were observed at significant levels in the water phase of one of the water sediment systems. M-03 was not observed in aquatic systems due to its instability in water with hydrolysis DT_{50} values ranging from 0.1 hours to 1.9 days at environmentally relevant pH values (pH 5 to 8). No metabolites requiring risk assessment were formed in sediment.

Comprehensive surveys of public monitoring databases for environmental data and summaries of relevant peer reviewed publications on fluopicolide and its metabolite M-01 are provide in Document MCA 7, Part 3.

CA 7.1 Fate and behaviour in soil

The following summary provides an overview of the behaviour of fluopicolide in soil.

The fate and behaviour of fluopicolide (AE C638206) in soil has been investigated in a comprehensive series of laboratory studies and, when required, extended by data from field experiments. The laboratory studies were all conducted with ^{14}C-labelled active substance. When required to fully define the fate of the molecule, studies have been separately performed with labelling in each of the two rings; uniformly labelled phenyl ring or labelled in the 2 and 6 positions of the pyridyl ring.



* = [2,6-Pyridyl-^{14}C]-ring label § = [Phenyl-U-^{14}C]-ring label

Additionally, where required, studies have been conducted on metabolites of fluopicolide.

The primary metabolic pathway of fluopicolide in soil is oxidation to form the hydroxylated metabolite M-03 (AE 0608000). M-03 in turn is cleaved to form the metabolites M-01 (AE C653711, BAM) containing the phenyl ring and M-02 (AE C657188, PCA) containing the pyridyl ring. While M-02 was rapidly metabolised to a number of further minor metabolites, no metabolites other than M-01 have been detected arising from the phenyl ring. Microbial breakdown of this ring slowly leads to the formation of carbon dioxide and soil bound residues with no intermediate products observed.

Unextracted soil bound residues account for between 5 and 23% of the applied fluopicolide at the end of the studies. Mineralisation to carbon dioxide was slow, with less than 10% being produced in studies conducted with parent radiolabelled in either the pyridyl or phenyl ring.

Supplemental studies have also been conducted to investigate the metabolism of fluopicolide in soil under anaerobic and sterile conditions and to determine if photolysis contributed to the degradation of fluopicolide on soil surfaces.

Under sterile conditions the cleavage of fluopicolide to M-01 and M-02 proceeded at a rate no slower than in viable soil but the subsequent breakdown of the metabolites was not evident. Fluopicolide was slowly degraded under flooded anaerobic conditions. The metabolic pathway was the same as that observed under aerobic conditions, with no unique metabolites formed under anaerobic conditions. Whilst the presence of light accelerated the rate of degradation on soil, no unique metabolites were formed exceeding 0.3% of applied radioactivity.

During the course of these studies, only three metabolites have been observed in significant amounts (i.e. >10% of applied or > 5% at two or more consecutive timepoints or > 5% at the final timepoint).

M-01 and M-03 can be defined as major metabolites exceeding 10% of applied radioactivity. M-01 has been identified in all soils tested with fluopicolide labelled in the phenyl ring, with maximum percentage ranging from 5 to 55%. The occurrence of M-03 has been shown to have a strong pH dependence and the metabolite was only observed as a major metabolite in acidic soils (<pH 6) at a maximum of 11%, whilst in neutral to alkali soils it was either not detected or detected occasionally at a maximum of 3%. M-02 was detected as a minor metabolite in soil which exceeds 5% AR at two or more consecutive timepoints in aerobic soil incubated at 10° C and was observed at a maximum of 7%, exceeding 5% AR at one timepoint only under aerobic conditions at 20 °C before declining to less than 2%.

In studies conducted according to EU requirements for 120 days no other metabolites were detected at levels above 5%. Three further metabolites of M-02 were observed in soil laboratory studies conducted with [2,6-pyridyl-^{14}C]-labelled fluopicolide according to EPA requirements at levels ≤ 5% but were not identified.

Metabolite B was observed at maximum of 5.3% AR, exceeding 5% at one timepoint only. Metabolite C was reported to reach a maximum of 5.5% AR on DAT 273 and declined very slightly to 5.2% AR by DAT 369. This metabolite has been further investigated in a statement (KCA 7.1.2.1.1/06, M-685745-01-1) and it was concluded the region quantified as Metabolite C did not contain a single metabolite at > 5% AR. Metabolite D did not exceed 5% AR in either soil.

Soil metabolites formed from fluopicolide

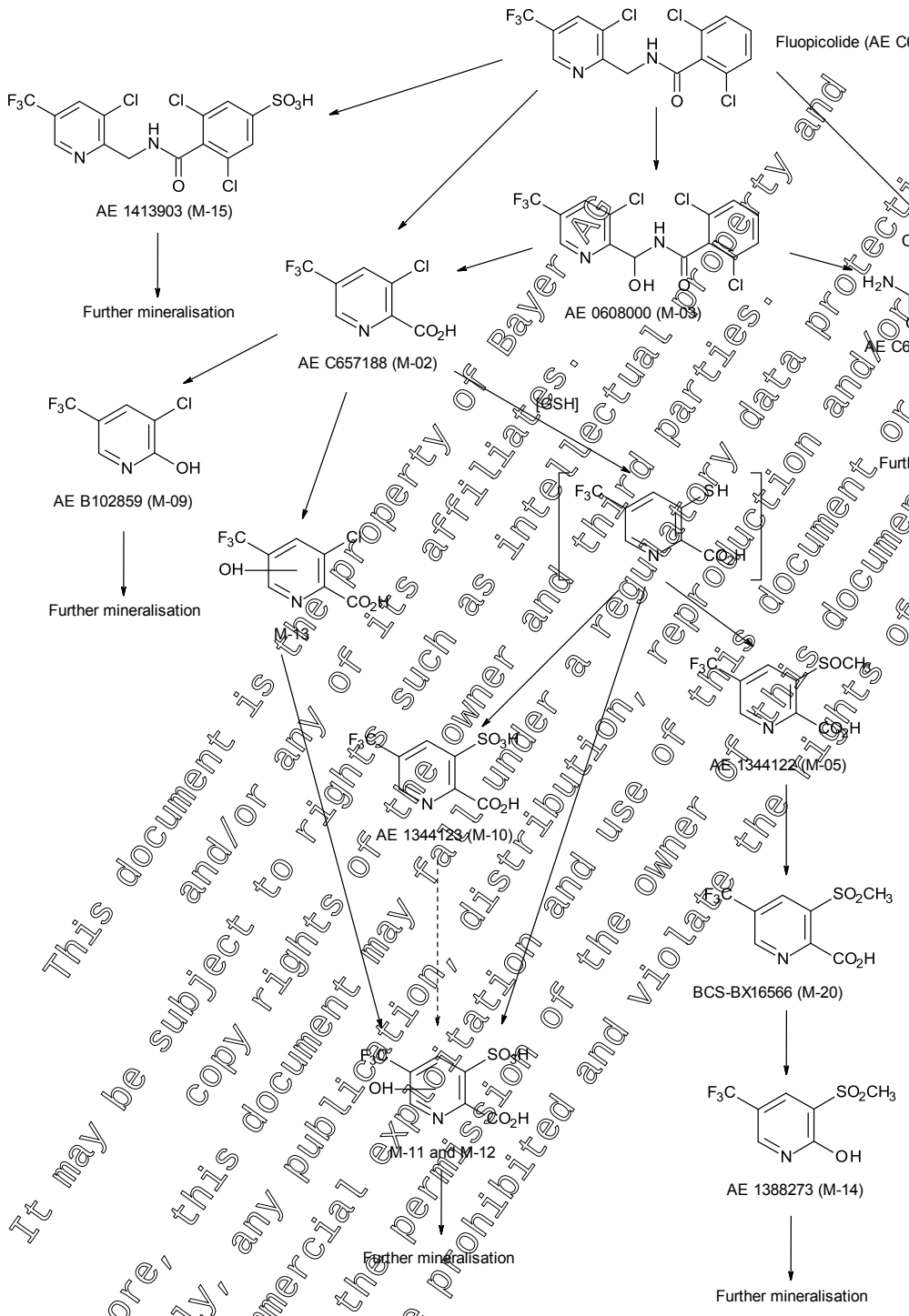
	Metabolite	Aerobic	Anaerobic	Soil Photolysis
Maximum observed in laboratory studies (as % of applied fluopicolide)	M-01 (AE 653711)	55.0	2.1	8.6
	M-02 (AE C657188)	7.3	8.9	2.6
	M-03 (AE 0608000)	10.6	not detected	not detected

The primary metabolic pathway in soil involved steady degradation of fluopicolide initially by hydroxylation of the aliphatic bridge between the two aromatic ring moieties to form the hydroxylated metabolite M-03. This resulted in cleavage of the intact fluopicolide skeleton with formation of the metabolites M-01 containing the phenyl ring and M-02 containing the pyridyl ring. The initial hydroxylation of the parent was relatively slow while the subsequent cleavage of M-03 was generally extremely rapid, although under very acidic conditions it has been observed to be slightly slower. Neither step was enhanced by microbial activity. Microbial breakdown of M-01 slowly leads to the formation of carbon dioxide and soil bound residues with no significant intermediate products observed. M-02 was very rapidly metabolised by microbial activity in soil initially to further minor metabolites and ultimately to carbon dioxide and unextracted soil residues. Metabolism of M-02 proceeded via a number of pathways with the initial steps hydroxylation of the pyridyl ring to form the metabolites M-09 and M-13 and a postulated reaction with glutathione to form a transient intermediate that can be oxidised to M-10, oxidised and hydroxylated to M-11 and M-12, or methylated and oxidised to M-05. M-10 may then be hydroxylated to M-11 and M-12, and M-05 oxidised to M-20 and then hydroxylated to M-14.

The metabolic route of degradation of fluopicolide in soil is shown below. Further metabolism of the minor metabolite M-02 led to the formation of very low levels of metabolites either detected in aerobic soil metabolism studies conducted with [2,6-pyridyl-^{14}C]-M-02 or in leachate from a lysimeter study conducted with fluopicolide. In the lysimeter study conducted with [2,6-pyridyl-^{14}C]-labelled fluopicolide, the pyridyl ring metabolites M-05, M-10, M-11, M-12, M-13 and M-14 were detected at annual average concentrations > 0.1 µg/L in leachate. Additionally M-15, a sulfate metabolite of fluopicolide, was detected in leachate from the lysimeter study at annual average concentrations close to but < 0.1 µg/L.

In laboratory soil studies, DegT$_{50}$ values for fluopicolide at 20ºC ranged from 93.5 to 1037.9 days (mean value 181.6 days, normalised to 20ºC and pF2).

Metabolic pathway for fluopicolide and its metabolites in soil



Summary of $DegT_{50}$ values derived for fluopicolide under laboratory conditions

Applied compound	Study	Soil	Model selected	$DegT_{50}$ un-normalised (d)	$DegT_{50}$ normalised to 20°C and pF2 (d)
Fluopicolide	[redacted], 2003	Münster	SFO	212.0	212.0
		Sarotti	SFO	191.2	191.2
	[redacted] 2003a	Abington (non-sterile)	SFO	348.0	340.2
	[redacted] 2003b	Lamberton	SFO	1290.0	1037.9
	[redacted] 2003c	Lamberton	SFO	358.0	395.8
		Pikeville	DFOP	612.9[a] / 30.1[b]	616.0[a] / 30.3[b]
	[redacted] 016a	Albaro/Marcomcini	DFOP	146.2[a] / 2.8[b]	146.2[a] / 2.8[b]
		Great Chishill	DFOP	312.4[a] / 2.7[b]	312.4[a] / 2.7[b]
		H[redacted]	DFOP	155.5[a] / 7.2[b]	155.5[a] / 7.2[b]
		Mas du Coq	DFOP	216.7[a] / 10.5[b]	193.7[a] / 9.4[b]
		Parcey Meslay	DFOP	202.5[a] / 8.1[b]	202.5[a] / 8.1[b]
		Vilobí d'Onyar	DFOP	93.5[a] / 7.8[b]	93.5[a] / 7.8[b]
	[redacted] 2016b	Dollendorf II	DFOP	111.4[a] / 0.6[b]	111.4[a] / 0.6[b]
		H[redacted]	DFOP	137.7[a] / 4.2[b]	137.7[a] / 4.2[b]
		[redacted]	DFOP	141.3[a] / 6.3[b]	141.3[a] / 6.3[b]
		[redacted]	DFOP	133.5[a] / 9.4[b]	133.5[a] / 9.4[b]
	[redacted] 2019	Abington 2	DFOP	142.1[a] / 1.9[b]	142.1[a] / 1.9[b]
		Lamberton	DFOP	176.1[a] / 2.8[b]	145.1[a] / 2.3[b]
		Lignieres	DFOP	141.4[a] / 1.4[b]	141.4[a] / 1.4[b]
		Münster	DFOP	170.1[a] / 5.3[b]	124.5[a] / 3.9[b]
		Pikeville	DFOP	155.2[a] / 4.1[b]	129.4[a] / 3.5[b]
		Sarotti 2	DFOP	161.2[a] / 1.6[b]	143.6[a] / 1.4[b]
Geometric mean (SFO and DFOP slow phase)					**181.6[c]**

a – Pseudo-SFO value based on slow phase of decline (calculated as $\ln(2)/k_2$ and normalised if applicable)
b – Pseudo-SFO value based on fast phase of decline (calculated $\ln(2)/k_1$ and normalised if applicable)
c – Geometric mean calculated of $DegT_{50}$ values from Lamberton soils prior to calculation of overall geometric mean.

Experiments have been conducted to investigate the behaviour of fluopicolide under field conditions. Fluopicolide was found to have a similar rate of degradation in the field, with $DegT_{50}$ values similar to those observed under laboratory conditions (range 111.9 to 317.4 days, mean 183 days, normalised to 20ºC and pF2).

Summary of $DegT_{50}$ values (normalised to 20°C and pF2) derived for fluopicolide from terrestrial field dissipation studies

	Aerobic field conditions					
Soil type	**Location (country)**	**pH ($CaCl_2$)**	**Depth (cm)**	**St. (χ^2err) (%)**	**Method of calculation**	**$DegT_{50}$ (d) norm**
Silt loam	Burscheid (Germany)	5.9	0-30	9.80	SFO	111.9
Clay	Great Chishill (UK)	7.8	0-50	11.64	SFO	216.9
Sandy loam	Lignieres de Touraine (France)	6.9	0-20	4.82	SFO	158.6
Clay loam	St.Etienne du Grès (France)	8.1	0-40	4.90	SFO	303.2
Clay loam	Albaro di Ronco all'Adige (Italy)	7.7	0-30	9.99	SFO	237.3
Sandy clay loam	Vilobi d'Onyar (Spain)	6.9	0-20	6.20	SFO	166.8
Loamy sand	Philippsburg (Germany)	6.4	0-50	9.477	SFO	199.6
Sandy clay loam	Rödelsee (Germany)	7.4	0-30	21.59	SFO	146.4
Sand	Huntlosen (Germany)	4.9	0-50	15.46	SFO	168.4
Loamy sand	Valencia (Spain)	7.3	0-30	13.95	SFO	317.4
Sandy silt	Appilly (France)	7.1	0-30	11.16	SFO	144.2
Sandy silt loam	Senas (France)	7.6	0-45	9.864	SFO	136.5
					Geometric mean	**183**

An overall geometric mean $DegT_{50}$ value of **182 days** in soil was derived for fluopicolide for use in FOCUS modelling calculations, including both laboratory and field data.

Laboratory studies have also been conducted with the soil metabolites of fluopicolide. In addition, M-01 was observed to form from fluopicolide in parent studies and from M-03 in one study. M-03 was observed to form from fluopicolide in three soils. The number of soils and datasets evaluated to derived $DegT_{50}$ values for each metabolite is summarised below. The degradation rate of M-03 is extremely rapid except in soils which are highly acidic. In very acidic soils (pH < 6) the degradation is bi-phasic with a rapid initial degradation phase followed by a second slower degradation phase.

Summary of $DegT_{50}$ values (normalised to 20°C and pF2) derived for the fluopicolide metabolites under laboratory conditions

Compound	Number of datasets	Number of soils	Geometric Mean $DegT_{50}$ normalised to 20°C & pF2 (d)	Arithmetic Mean Molar Fraction
M-01 (AE C653711)	26	18	569.5	0.80 (from parent)
M-02 (AE C657188)	7	7	1.6	NA [C]
M-03 (AE 0608000)	9	7	17.9 [A] / 0.19 [B]	0.53 [A] / - [B] (from parent)
M-05 (AE 1344122)	13	7	25.2	0.153 (from M-02)
M-10 (AE 1344123)	13	7	35.4	0.129 (from M-02)
M-11/M-12	2	2	87.6	0.044 (from M-02)
M-13	3	3	20.7	0.049 (from M-02)
M-14 (AE 1388273)	5	3	9.4	1 (from M-20)
M-15 (AE 1413903)	4	4	144.8	NA [C]
M-20 (BCS-BX16566)	6	6	6.1	0.021 (from M-02) 0.559 (from M-05)

[A] Geometric mean for soils with pH < 6
[B] Geometric mean for soils with pH ≥ 6
[C] Not applicable as degradation rates for M-02 and M-15 were derived from metabolite dosed studies. For M-02 the overall formation fraction from fluopicolide was set to 1.0 as a conservative assumption. For M-15 a molar formation fraction of 0.0016 from fluopicolide was estimated by inverse modelling of a lysimeter study (KCA 7.1.3.2/08, M-687165-01-1)

While M-01 was found to degrade slowly in these laboratory studies other metabolites showed moderate to rapid rates of degradation. Thus, terrestrial field dissipation studies have also been conducted to investigate the behaviour of M-01 under field conditions. Great care has been taken to exclude any potential enhancement of the degradation rate by surface or leaching processes. Degradation half-lives for M-01 derived from laboratory and field dissipation studies were compared using the EFSA $DegT_{50}$ Endpoint Selector (EFSA, 2014). This comparison indicated that the field $DegT_{50}$ values for M-01 were significantly shorter than the laboratory studies, therefore the geometric mean field $DegT_{50}$ value of **146 days** was used for FOCUS modelling calculations for M-01.

Summary of $DegT_{50}$ values (normalised to 20°C and pF2) derived for M-01 from terrestrial field dissipation studies

	Aerobic field conditions					
Soil type	**Location (country)**	**pH ($CaCl_2$)**	**Depth (cm)**	**St. (χ^2 cor) (%)**	**Method of calculation**	**$DegT_{50}$ (d) norm**
Silt loam	Burscheid (Germany)	5.2	0-110	14.68	SFO	94.0
Sandy loam	Lignieres de Touraine (France)	6.9	0-100	7.82	SFO	191.1
Clay loam	St.Etienne du Grès (France)	8.1	0-50	5.87	SFO	179.9
Clay loam	Albaro di Ronco all'Adige (Italy)	7.7	0-60	13.93	SFO	151.8
Sandy clay loam	Vilobi d'Onyar (Spain)	6.9	0-110	10.94	SFO	136.3
					Geometric mean	**146**

The degradation rate of fluopicolide and M-01 indicate some persistence leading to residual residue levels remaining one year after application. Accumulation studies were conducted to determine fluopicolide and M-01 levels in soil following annual applications over a four year period. Levels in the soil at the end of the studies did not point to significant accumulation. Definitive assessment of the accumulation of fluopicolide and its metabolites in soil is addressed in Document MCP-9 by calculation.

The mobility in soil of fluopicolide and its metabolites was studied in batch equilibrium tests on a variety of different soils. A summary of the calculated adsorption constants and Freundlich exponents are presented below. Fluopicolide is of medium mobility (McCall classification) or low mobility (Briggs classification) in soil. The geometric mean K_{oc} was 267.7 mL/g. The metabolites of fluopicolide were found to be more mobile than their parent.

Compound	**Number of soils**	**Geometric Mean K_{oc} (mL/g)**	**Arithmetic Mean Freundlich Exponent 1/n**
Fluopicolide	24	267.7	0.888
M-01 (AE C653711)	10	24.1	0.914
M-02 (AE C657188)	8	5.7	0.889
M-03 (AE 0608000)	3	106.9	0.971
M-05 (AE 1344122)	7	25.8	0.960
M-10 (AE 1344123)	5	1.8 [A]	- [C]
M-11/M-12	-	- [B]	- [C]
M-13	-	0.41 [B]	- [C]
M-14 (AE 1388273)	9	9.9	0.942
M-15 (AE 1413903)	4	18.8	0.937
M-20 (BCS-BX16566)	4	2.1 [A]	- [C]

[A] Single point K_{oc} measurements as adsorption to soil was too low to conduct Freundlich adsorption isotherms.
[B] No reliable sorption parameters could be derived. A worst-case Koc of 0 is assumed
[C] Default 1/n values of 1.0 assumed

The adsorption of fluopicolide to soil has been shown to increase significantly with time under laboratory conditions. A comprehensive set of time-dependent sorption studies in sixteen soils have been evaluated according to current guidance on aged sorption, resulting in mean aged-sorption parameters of F_{ne} 0.508 and k_{des} 0.0356 for fluopicolide. Comparison of the non-equilibrium and equilibrium K_{d_app} fits showed that strong aged-sorption effects occurred for all soils, as shown by equilibrium fits not being able to adequately describe the increased observed in K_d with time. Lower-tier degradation study data for fluopicolide from laboratory studies and field studies have been evaluated to derive $DegT_{50eq}$ values, which when combined with the higher-tier aged-sorption values yield an overall geometric mean $DegT_{50eq}$ of 121 days for use in exposure modelling (in combination with the mean aged-sorption parameters: F_{ne} 0.508; k_{des} 0.0356).

Overall $DegT_{50eq}$ evaluation results

Soil	$DegT_{50eq}$ (days)	Derivation
L[redacted]	80.5	TDS - PEARLneq
Dollendorf	98.6	TDS - PEARLneq
H[redacted]	69.8	TDS - PEARLneq
W[redacted]	45.4	TDS - PEARLneq
H[redacted]	76.2	TDS - PEARLneq
Great Chishill	170.9	TDS - PEARLneq
Parcey Meslay	111.0	TDS - PEARLneq
Mas du Coq	108.4	TDS - PEARLneq
Albaro	112.2	TDS - PEARLneq
Vilobi d'Ònyar	52.2	TDS - PEARLneq
Abington	97.5	TDS - PEARLneq
Lamberton	91.6	TDS - PEARLneq
Munster	75.4	TDS - PEARLneq
Pikeville	66.8	TDS - PEARLneq
Sarrotti	99.3	TDS - PEARLneq
Lignieres	96.8	TDS - PEARLneq
Munster	178.1	Lab Tier-1 Refit
Sarrotti	198.6	Lab Tier-1 Refit
Abington	256.4	Lab Tier-1 Refit
Lamberton	532.5	Lab Tier-1 Refit
Pikeville	295.2	Lab Tier-1 Refit
Burscheid (Germany)	84.3	Field Scaling factor 1
Great Chishill (UK)	155.8	Field Scaling factor 1
Lignieres de Touraine (France)	109.8	Field Scaling factor 1
St.Etienne du Grès (France)	234.2	Field Scaling factor 1
Albaro di Ronco all'Adige (Italy)	205.3	Field Scaling factor 1
Vilobi d'Onyar (Spain)	92.5	Field Scaling factor 1
Philippsburg (Germany)	158.9	Field Scaling factor 1
Rödelsee (Germany)	109.0	Field Scaling factor 1
Huntlosen (Germany)	124.7	Field Scaling factor 1
Valencia (Spain)	234.4	Field Scaling factor 1
Appilly (France)	107.6	Field Scaling factor 1
Senas (France)	101.3	Field Scaling factor 1
Geometric mean	**121**	

The fate and mobility of fluopicolide and its metabolites in soil were additionally investigated in outdoor experimental exposure assessments. A lysimeter study in acidic silty sand soil (pH 5.2) with low organic carbon content was conducted in Germany and in accordance with BBA guidelines. This study was complimented by a field leaching study conducted in a similar sandy soil. The results of these studies show fluopicolide, M-02 and M-03 will not reach groundwater. A number of metabolites showed potential to reach groundwater in concentration in excess of 0.1 µg/L. The metabolites detected in leachate; M-05, M-10, M-11, M-12, M-13, M-14 and M-15 have been fully identified or characterised to an extent which enabled full risk assessments to be conducted on them. The metabolite M-01, from the phenyl ring, is also mobile in soil and the concentration in groundwater at 1 m depth following use of fluopicolide was established using soils and climate conditions which represented worst-case conditions for leaching.

Definitive assessment of leaching potential of fluopicolide and its metabolites is addressed by FOCUS groundwater modelling provided in Document MCP-9.

CA 7.1.1 Route of degradation in soil

CA 7.1.1.1 Aerobic degradation

Two studies were evaluated during the previous EU review and are still considered as reliable to assess the route of fluopicolide degradation in soil (KCA 7.1.1.1/01 and KCA 7.1.1.1/02).

Report reference	Author, Year	Phenyl Label	Pyridyl Label	Comment
KCA 7.1.1.1/01 M-241049-01-1	[redacted] 2003	✓	✓	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable. Additional summary provided under KCA 7.1.3.2/02
KCA 7.1.1.1/02 M-201230-02-1	[redacted] 2003	✓	✓	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.1.1/03 M-687284-01-1	[redacted] 2020	-	-	New data not yet reviewed.

The aerobic degradation of fluopicolide in soil has been investigated in a total of six studies at 20°C, one study at 25°C and one study at 40°C. The remaining aerobic soil degradation studies are summarised under Point KCA 7.1.2.1.1. No additional metabolites of fluopicolide are formed in aerobic soil studies summarised under KCA 7.1.2.1.1 to those observed in Studies KCA 7.1.1.1/01 and KCA 7.1.1.1/02.

The fungicide fluopicolide does not contain any stereogenic centres, but its metabolite M-03 (AE 0608000) contains a chiral carbon atom and M-05 (AE 1344122) contains a chiral sulfur atom. Consequently, either metabolite could exist as a pair of enantiomers. The EFSA guidance document (EFSA, 2019) on how to perform risks assessments for plant protection products that contain stereoisomers has been used to assess if any further information is required for M-03 or M-05. A statement on the potential for formation of stereoisomers of M-03 is provided under KCA 7.1.1.1/03 (M-687284-01-1). No additional environmental fate information was required for M-05 as it does not exceed the trigger of 5% AR in the environment given in EFSA (2019)*: Guidance of EFSA on risk assessments for active substances of plant protection products that have stereoisomers as components or impurities and for transformation products of active substances that may have stereoisomers.*

Data Point:	KCA 7.1.1.1/01
Report Author:	[redacted]
Report Year:	2003
Report Title:	Route and rate of degradation of [2,6-14C pyridinyl] and [U-14C-benzoyl]-AE C638206 in a European sandy loam under laboratory aerobic conditions at 20 deg. C and determination of aged in situ Kd values at 25 degrees C
Report No:	B004071
Document No:	M-241049-01-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EC of July 1995
Deviations from current test guideline:	Yes. The study design does not conform to current aged sorption guidelines as the aged sorption phase of the study had insufficient timepoints.
Previous evaluation:	yes, evaluated and accepted Tests on aerobic degradation evaluated and accepted in the DAR (2005). Tests on aged sorption evaluated in the DAR (2005) and Addendum 1 to the DAR (2007).
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of fluopicolide was investigated in a European soil under laboratory aerobic conditions for up to 120 days. [Phenyl-U-^{14}C]-labelled fluopicolide or [2,6-pyridyl-^{14}C]-labelled fluopicolide was applied to soil samples at an application rate equivalent to 400 g /ha. Abington soil was classified as a sandy loam soil according to USDA classification. Soil samples were incubated in the dark, at a moisture content equivalent to pF 2 under aerobic conditions at 20 °C. The radiochemical purity was > 99 % for both radiolabelled test items. The specific activities were 5.33 and 5.88 MBq/mg for [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]-labelled fluopicolide, respectively.

Samples were taken for extraction and analysis immediately after treatment (day 0) and after 14, 28, 42, 56, 77, 98 and 120 days of incubation. Sterile samples were taken for analysis at 14, 56, 77 and 120 days. Soil samples were exhaustively extracted with up to four successive extractions with acetonitrile / water (4 / 1, v/v) at ambient temperature followed by one Soxhlet extraction using acetonitrile. Concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC) and normal phase thin layer chromatography (TLC).

The material balances ranged from 93.1 to 103.5% of applied radioactivity (% AR). For samples incubated with [phenyl-U-^{14}C]-fluopicolide, extractable [^{14}C]-residues decreased slightly from a maximum of 98.4% AR at DAT 0 to 92.5% AR by DAT 120. For samples incubated with [2,6-pyridyl-^{14}C]-fluopicolide, extractable radioactivity decreased from 95.1% AR at DAT 0 to a minimum of 82.3% AR by DAT 120.

Non-extractable [^{14}C]-residue increased proportionately with the decrease in extractable radioactivity over the 120 day study. The maximum amount of non-extractable residues was 5.2% (DAT 120) in the phenyl label and 11.9% (DAT 120) in the pyridyl label.

Mineralization to carbon dioxide was a minor pathway, demonstrated by the low amount of radioactivity recovered in the ethanolamine volatile traps for both labels (maximum of 2% of applied). No significant levels of organic volatiles were observed.

After 120 days incubation at 20 °C, fluopicolide degraded to 80.2% of the radioactivity applied in the phenyl label and 77.2% in the pyridyl label. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of 340.0 days and DT_{90} values of 1156.0 days in Abington soil.

Fluopicolide degraded to oxidative cleavage products, M-01 (AE C653711) and M-02 (AE C657188). The quantity of M-01 ranged from 4.5% (DAT 14) to a maximum of 14.4% (DAT 56) and declined thereafter to 12.1% at the end of the 120 day incubation period. M-02 was generally observed at ca. 1% except on DAT 42 when it reached 7.3% of the applied radioactivity. Two minor metabolites (each < 3% of the applied radioactivity) were detected in the pyridyl treated soil extracts only.

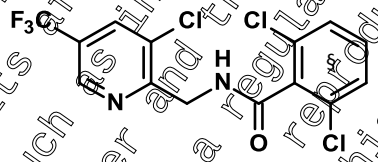
Microbial activity did not enhance fluopicolide degradation. This observation is supported by the presence of 77.2% of fluopicolide observed in the 120 day non-sterile samples compared to 61.9% of fluopicolide observed in the sterile samples during the same incubation period. However, microbial activity did facilitate degradation of the key metabolites, M-01 and M-02. Both M-01 and M-02 accumulated in sterile samples with a maximum of 36.2% and 25.7%, respectively (DAT 120), which compares to M-01 and M-02 maximum in the non-sterile samples of 14.4 and 7.3%, respectively.

I. Materials and Methods

A. Materials

1. Test Items

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

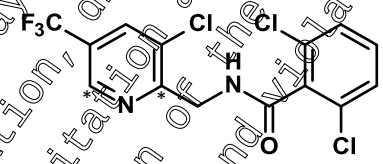


§Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.33 MBq/mg

Radiochemical Purity: 100% (mean of HPLC and TLC analyses)

[2,6-Pyridyl-^{14}C]-fluopicolide



*Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.88 MBq/mg

Radiochemical Purity: 99.97% (mean of HPLC and TLC analyses)

2. Test Soil

The study was performed using one test soil as characterized in Table 7.1.1.1- 1.

Table 7.1.1.1- 1: Physico-chemical properties of test soil

Parameter		Soil
Soil Designation		Abington
Geographic Location		
City		Abington
Country		England, UK
Textural Classification (USDA)		sandy loam
Sand [50 - 2000 µm]	(%)	67
Silt [2 – 50 µm]	(%)	22
Clay [< 2 µm]	(%)	11
pH		
in H_2O (1:1)		7.4
in $CaCl_2$ (1:1)		7.2
Organic Matter (%)		3.8
Organic Carbon (%)*		2.2
Cation Exchange Capacity (meq/100 g)		18.0
Water Holding Capacity (%)		
maximum		56.1
at 1/10 bar		18.8
at 1/3 bar		14.6
Soil Microbial Biomass (µg microbial C/g soil)		
Initial (DAT 1)		376.8
Final (DAT 120)		212.1

* Calculated by dividing organic matter content by 1.72
Biomass samples were untreated

B. Study Design

1. Experimental Conditions

Tests were performed in flow through systems consisting of glass flasks each containing 50 g soil and attached to an ethylene glycol trap to collect organic volatiles followed by an ethanolamine (or 3M sodium hydroxide on selected flasks) trap to collect carbon dioxide. Soil moisture was maintained during incubation with periodic additions of water throughout the study.

The tests were performed at a concentration of approximately 0.41 mg/kg dry weight of soil. The test concentration was based on a field rate of 400 g a.s./ha. The test items [phenyl-U-^{14}C]- or [2,6-pyridyl-^{14}C]-fluopicolide, dissolved in acetonitrile (406 and 460 µL, respectively), were applied drop wise onto the soil surface. Soil samples were adjusted to a moisture content of 18.2%, equivalent to pF 2, five to seven days prior to application. The samples were incubated at 20 ± 1 °C under aerobic conditions in the dark for 120 days.

Selected flasks were sterilized (gamma irradiation) prior to treatment with the test substance, while additional untreated sterile and untreated non-sterile flasks were used as controls and to monitor the viability of the test system by determination of biomass.

In addition, the effect of ageing on potential mobility of fluopicolide and its metabolites was also studied by determining partitioning ratios (*in situ* K_d values) between aqueous and soil phases in additional flasks incubated at 25 °C. The study design is no longer considered valid to assess aged sorption, in particular only four timepoints were taken. Details of this part of the study are provided in KCA 7.1.3.2/02. The results are consistent with later fully complaint guideline studies.

2. Sampling

Single samples were removed for analysis after 0, 14, 28, 42, 56, 77, 98 and 120 days of incubation. For tests using sterile soil, single samples were removed after 14, 56, 77 and 120 days of incubation. Untreated samples were analysed for biomass at the beginning and end of the experiment.

3. Analytical Procedures

Soil samples, including sterile samples, were extracted up to four times successively with acetonitrile/water (4/1, *v/v*) at ambient temperature followed by one Soxhlet extraction using acetonitrile. Radioactivity in extracts was determined by liquid scintillation counting (LSC). Soil extracts were concentrated and analysed by HPLC with radiodetection. Degradation products were identified by comparison of the retention times of reference standards and confirmed by TLC co-chromatography with reference items. A peak of 300 dpm, corresponding to 0.9 ng fluopicolide, was readily determined by TLC and HPLC quantitation methods used.

Volatile radioactivity in volatile traps was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide and M-01 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). For fluopicolide, as the degradation was investigated using two radiolabel positions, and similar behaviour was observed for each, these radiolabels have been considered as true replicates, and included together in a single optimisation. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed between the SFO and FOMC fits for fluopicolide. For the non-sterile Abington soil the FOMC fit resulted in a lower χ^2 err% value, and the DFOP model was also fitted, however the KinGUI run for the DFOP model did not complete successfully. The FOMC fit was not accepted, as extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using this model (EFSA, 2009). The SFO model therefore provided the most appropriate description of fluopicolide degradation in the non-sterile Abington soil.

Metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soil incubated at 20 °C following application of [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]-fluopicolide are summarized in Table 7.1.1.1- 2 to Table 7.1.1.1- 3. The corresponding data for sterile samples are summarized in Table 7.1.1.1- 4 to Table 7.1.1.1- 5.

Table 7.1.1.1- 2: Degradation of [phenyl-U-^{14}C]-fluopicolide at 20 °C in Abington soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)							
	0	14	28	42	56	77	98	120
Fluopicolide	98.4	93.3	90.9	82.7	79.6	82.8	81.5	80.2
M-01 (AE C653711)	n.d.	4.5	6.7	8.0	14.4	11.6	13.2	12.1
Ambient extract	98.4	92.8	95.1	85.3	85.9	90.8	86.9	85.4
Soxhlet extract	n.a.	4.6	2.5	5.8	8.2	4.5	7.8	7.0
Total extractable radioactivity [A]	*98.4*	*97.4*	*97.6*	*91.1*	*94.1*	*95.3*	*94.7*	*92.5*
Non-extractable radioactivity	2.1	0.9	1.9	1.9	3.0	3.6	4.0	5.2
^{14}C-Carbon dioxide including other volatiles [B]	n.a.	0.1	0.5	0.8	0.1	0.9	0.2	0.7
Total radioactivity	100.5	98.4	100.0	93.8	97.2	99.8	98.9	98.4

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[B] Other volatile radioactivity was < 0.05 % AR at any time point

Table 7.1.1.1- 3: Degradation of [2,6-pyridyl-^{14}C]-fluopicolide at 20 °C in Abington soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)							
	0	14	28	42	56	77	98	120
Fluopicolide	95.1	97.0	92.2	80.7	85.5	83.2	77.4	77.2
M-02 (AE C657188)	n.d.	n.d.	1.4	7.3	0.9	1.0	1.3	1.2
Unknown A (R_t 16 min)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.6	0.9
Unknown B (R_t 21 min)	n.d.	n.d.	n.d.	n.d.	n.d.	2.4	3.0	2.8
Ambient extract	95.1	92.6	92.7	83.1	83.2	84.5	79.8	77.6
Soxhlet extract	n.a.	4.4	2.4	4.9	4.8	2.8	5.4	4.7
Total extractable radioactivity [A]	*95.1*	*97.0*	*95.1*	*88.0*	*88.0*	*87.3*	*85.2*	*82.3*
Non-extractable radioactivity	2.4	2.2	3.8	4.6	7.4	8.5	10.0	11.9
^{14}C-Carbon dioxide including other volatiles [B]	n.a.	0.2	0.4	0.5	1.2	1.9	0.1	0.9
Total radioactivity	97.5	99.4	99.3	93.1	96.6	97.7	95.3	95.1

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of applied radioactivity (% AR)
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[B] Other volatile radioactivity was < 0.05 % AR at any time point

Table 7.1.1.1- 4: Degradation of [phenyl-U-^{14}C]-fluopicolide at 20 °C in sterile Abington soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)			
	14	56	77	120
Fluopicolide	86.9	75.3	71.3	61.9
M-01 (AE C653711)	7.3	24.0	27.7	36.2
Ambient extract	93.7	95.1	97.7	93.0
Soxhlet extract	n.a.	4.3	1.3	5.3
Total extractable radioactivity [A]	*93.7*	*99.4*	*99.0*	*98.3*
Non-extractable radioactivity	3.7	2.0	2.1	3.1
^{14}C-Carbon dioxide including other volatiles [B]	n.a.	n.d.	n.d.	n.d.
Total radioactivity	97.4	101.4	101.1	101.4

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of applied radioactivity (% AR)
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[B] Other volatile radioactivity was < 0.05 % AR at any time point

Table 7.1.1.1- 5: Degradation of [2,6-pyridyl-^{14}C]-fluopicolide at 20 °C in sterile Abington soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)			
	14	56	77	120
Fluopicolide	95.2	72.2	75.5	63.1
M-02 (AE C657188)	1.8	16.7	19.8	25.7
Ambient extract	98.6	87.3	94.3	85.2
Soxhlet extract	n.a.	4.8	1.0	3.6
Total extractable radioactivity [A]	*98.6*	*92.1*	*95.3*	*88.8*
Non-extractable radioactivity	4.9	7.0	4.7	8.0
^{14}C-Carbon dioxide including other volatiles [B]	n.a.	n.d.	n.d.	n.d.
Total radioactivity	103.5	99.1	100.0	96.8

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of applied radioactivity (% AR)
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[B] Other volatile radioactivity was < 0.05 % AR at any time point

B. Material Balance

For aerobic degradation samples of Abington soil incubated at 20 °C, material balances ranged from 93.8 to 100.5% AR for [phenyl-U-^{14}C]-fluopicolide and from 93.1 to 99.4% AR for [2,6-pyridyl-^{14}C]-fluopicolide.

For sterile samples of Abington soil incubated at 20 °C, material balances ranged from 97.4 to 101.4% AR for [phenyl-U-^{14}C]-fluopicolide and from 96.8 to 103.5% AR for [2,6-pyridyl-^{14}C]-fluopicolide.

There were no signs for losses of radioactivity during work-up and processing.

C. Extractable and Non-Extractable Residues

For samples incubated with [phenyl-U-^{14}C]-fluopicolide, total extractable radioactivity decreased from 98.4% AR at DAT 0 to 92.5% AR by DAT 120. The total of non-extractable residues (NER) increased from 2.1% AR at DAT 0 to 5.2% AR by the end of the study (DAT 120).

For samples incubated with [2,6-pyridyl-^{14}C]-fluopicolide, total extractable radioactivity decreased from 95.1% AR on DAT 0 to 82.3% AR after 120 days of incubation. NER was 2.4% AR on DAT 0 and increased to 11.9% AR by the end of the study (DAT 120).

For sterile samples incubated with [phenyl-U-^{14}C]-fluopicolide, total extractable radioactivity amounted to 93.7% AR by DAT 14, peaked on DAT 56 (99.4% AR) and then decreased to 98.3% AR by the end of the study (DAT 120). NER amounted to 3.7% AR by DAT 14 and decreased to 3.1% AR after 120 days of incubation.

For sterile samples incubated with [2,6-pyridyl-^{14}C]-fluopicolide, total extractable radioactivity decreased from 98.6% AR by DAT 14 to 88.8% AR after 120 days of incubation. NER amounted to 4.9% AR by DAT 14 and increased to 8.0% AR at the end of the study (DAT 120).

D. Volatile Radioactivity

For samples incubated with [phenyl-U-^{14}C]-fluopicolide, levels of ^{14}C-carbon dioxide formed ranged from 0.1% AR (DAT 14) to 0.9% AR (DAT 77) during incubation. Other volatile radioactivity was < 0.05% AR at all timepoints.

For samples incubated with [2,6-pyridyl-^{14}C]-fluopicolide, values of ^{14}C-carbon dioxide formed were in the range of 0.2% AR (DAT 14) and 1.9% AR (DAT 77) during incubation. Other volatile radioactivity was < 0.05% AR at all timepoints.

For sterile samples, total volatile radioactivity was < 0.05% AR at all timepoints.

E. Degradation of Parent Compound

Following application of [phenyl-U-^{14}C]-fluopicolide, the amount of parent in the total soil extracts (i.e. in ambient and Soxhlet soil extracts) decreased from 98.4% at DAT 0 to 80.2% AR by the end of the study at DAT 120. In corresponding sterile samples, the amount of the active substance decreased from 86.9% AR at DAT 14 to 61.9% AR at DAT 120.

Degradation of [phenyl-U-^{14}C]-fluopicolide in Abington soil was accompanied by the formation of the degradation product M-01 (AE C653711). M-01 was detected at a maximum of 14.4% AR at DAT 56 in non-sterile samples, declining slightly to 12.1% AR by DAT 120. Levels of M-01 increased to a maximum of 36.2% AR by DAT 120 in sterile samples.

Following application of [2,6-pyridyl-^{14}C]-fluopicolide, the amount of parent in the total soil extracts (i.e. in ambient and Soxhlet soil extracts) decreased from 95.1% at DAT-0 to 77.2% AR by the end of the study at DAT 120. In corresponding sterile samples, the amount of the active substance decreased from 95.2% AR at DAT 14 to 63.1% AR at DAT 120.

Degradation of [2,6-pyridyl-^{14}C]-fluopicolide in Abington soil was accompanied by the formation of the degradation product M-02 (AE C657188) and two minor unidentified metabolites. M-02 was detected at a maximum of 7.3% AR at DAT 42, exceeding 5% AR at one timepoint only before rapidly declining *cc.* 1% AR (1.2% by DAT 120). The total unidentified residues amounted to a maximum of 5.6% AR and no single component exceeded 3.0% AR at any sampling interval. In sterile soil only M-02 was observed, which increased to a maximum of 25.7% AR by DAT 120.

F. Degradation Kinetics

Fluopicolide degraded slowly in the Abington sandy loam soil under non-sterile conditions. The reported DT_{50} values were 291 and 274 days (mean = 283 days) for the phenyl- and pyridyl- labels, respectively. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Further details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting DT_{50} values for trigger endpoints are summarised below in Table 7.1.1.1- 6. Best fit kinetics are highlighted in bold.

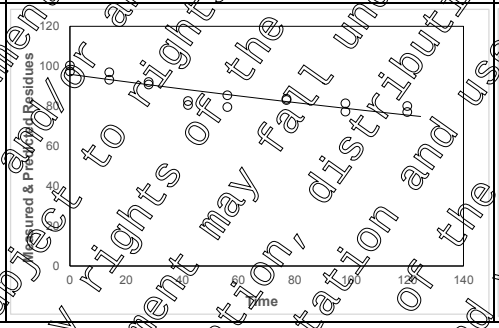
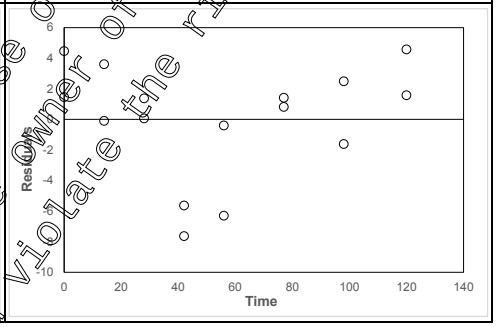
Table 7.1.1.1- 6: Degradation rate of fluopicolide under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ², %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington (non-sterile), Allan (2003a)	**SFO**	**96.04**	**k 0.001992**	**2.89**	**4.54E-07**	**0.001422**	**0.003**	**348**	**1156**
	FOMC	99.34	α 0.1403 β 25.08	2.17	n.r. n.r.	< 0.02499 > 17.32	0.256 67.48	3482.7	>10000

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.1.1- 7: Degradation of fluopicolide under aerobic conditions at 20 °C (best-fit DT_{50} value for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Abington (non-sterile) SFO Allan (2003a)		

II. Conclusion

Fluopicolide slowly degraded in the non-sterile sandy loam soil under aerobic conditions with 80% and 77% of the applied radioactivity remaining as parent compound in the phenyl and pyridyl label treated soils at the end of 120 days. Less than 2% of the radioactivity was detected as $^{14}CO_2$ with both labels, indicating slow mineralization of fluopicolide to CO_2. Organic volatiles were not detected in either label. The primary metabolic pathway involved the oxidative cleavage of fluopicolide to form M-01 (maximum of 14.4% in the phenyl label treated soil) and M-02 (maximum of 7.3% in the pyridyl label treated soil). Further degradation resulted in the formation of minor metabolites only in the pyridyl label treated soil which did not exceed 3% of applied radioactivity.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of 348 days for Abington soil.

Comparison of results from the sterile versus non-sterile soil indicates that the microbial activity did not appear to enhance fluopicolide degradation in the sandy loam soil. However, microbial activity did facilitate the degradation of M-01 and M-02.

Assessment and conclusion by applicant:

The study was conducted in accordance with SETAC 1.1 (1995) and USEPA (= EPA) N, 162-1 (1982). The study is considered valid to assess the aerobic degradation of [phenyl-U-^{14}C]-and [2,6-pyridyl-^{14}C]-fluopicolide in soil.

Data Point:	KCA 7.1.1.1/02
Report Author:	[redacted]
Report Year:	2003
Report Title:	Rate of degradation in two soils (amendment) (14C) AE C638206
Report No:	C037459
Document No:	M-201230-02-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EEC
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of fluopicolide was investigated in two European soils under laboratory aerobic conditions for up to 200 days. [Phenyl-U-^{14}C]-labelled fluopicolide was applied to soil samples at an application rate equivalent to 400 g /ha. The soils used were classified (ADAS classification) as a silty clay loam (Sarotti soil) and a loamy sand (Münster). Soil samples were incubated in the dark, at a moisture content equivalent to pF 2 under aerobic conditions at 20 ºC. The radiochemical purity was 100 % and the specific activity was 5.33 MBq/mg.

Samples were taken for extraction and analysis immediately after treatment (DAT 0) and after 7, 14, 22, 34, 49, 64, 78, 98, 120 days of incubation, and additionally in Münster soil only after 160 and 200 days. Soil samples were exhaustively extracted with acetonitrile / water (4 / 1, v/v) at ambient temperature followed by Soxhlet extraction using acetonitrile (DAT 0 and DAT 7) or with acetonitrile, followed by acetonitrile / water (4/1, *v/v*) at ambient temperatures and finally by Soxhlet extraction with acetonitrile (after DAT 7). Concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC). Selected samples were analysed by thin layer chromatography (TLC) to confirm the results obtained by HPLC. A selected sample was analysed by LC-MS to confirm the structural identity of the degradation products.

Material balances ranged from 98.9 to 86.7% AR in Sarotti soil (mean 91.7% AR) and 103.3 to 93.7% AR (mean 96.6% AR) for Münster soil. Extractable [^{14}C]-residues decreased from a maximum of 98.2% AR at DAT 0 to 77.8% AR by DAT 120 in Sarotti soil and from 102.1% AR on DAT 0 to 90.5% AR by DAT 200 in Münster soil. The amounts of unextractable radioactivity were low, reaching a maximum of 12.0% of applied radioactivity in Sarotti soil and 5.8% in Münster soil.

Radiolabelled carbon dioxide evolved accounted for a maximum of 3% of the applied radioactivity in the Sarotti soil by the end of the study and <1% in the Münster soil. No significant levels of organic volatiles were observed.

Fluopicolide was the principal radiolabelled component detected. Levels of parent accounted for 100% of extracted radioactivity at DAT 0 and declined to 53 and 55% of applied radioactivity at termination of the study in Sarotti and Münster soils respectively. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit un-normalised DT_{50} and DT_{90} values of 191.2 and 635 days in Sarotti soil, and 212.0 and 704.1 days in Münster soil.

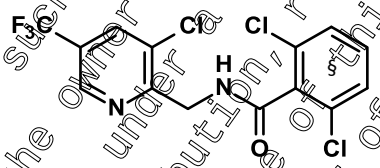
In addition to parent, two metabolites M-01 (AE C653711) and M-03 (AE 0608000, referred to as RPA 427967 in the report) were detected. Levels of M-01 increased over the course of the incubation period and accounted for 25.4% AR at DAT 120 in Sarotti and DAT 200 in Münster soils. M-03 was detected in Münster soil only, and accounted for a maximum of 10.6% AR.

I. Materials and Methods

A. Materials

1. Test Item

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



§ Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.33 MBq/mg

Radiochemical Purity: >100% (mean of HPLC and TLC analyses)

2. Test Soil

The study was performed using two test soils as characterized in Table 7.1.1.1- 8.

Table 7.1.1.1- 8: Physico-chemical properties of test soil

Parameter		Soil	
Soil Designation		Sarotti	Münster
Geographic Location		Hattersheim, D-65795	
City		Frankfurt	Münster-Handorf
Country		Germany	Germany
Textural Classification		Silty clay loam	Loamy sand
Sand [63 - 2000 µm]	(%)	9.3	79.8
Silt [2 – 63 µm]	(%)	69.7	17.2
Clay [< 2 µm]	(%)	21.1	2.9
pH			
in 0.01M KCl		7.4	4.9
Organic Matter (%)		1.6	1.2
Organic Carbon (%)		0.9	0.7
Cation Exchange Capacity (meq/100 g)		13.3	6.3
Water Holding Capacity (%)			
maximum		50.2	33.8
at pF 2 (1/10 bar)		20.2	6.1
at pF 0		62.2	35.9
at pF 2.5		28.3	9.2
Soil Microbial Biomass (mg microbial C /100g soil)			
Initial		50	12
DAT 120		30	20
DAT 200		2	41

Initial biomass samples were untreated, later biomass samples were treated with non-labelled fluopicolide (41 µg) dissolved in acetonitrile (100 µL).

B. Study Design

1. Experimental Conditions

Tests were performed in flow through systems consisting of glass flasks each containing 100 g soil and attached to an ethanediol trap to collect organic volatiles followed by an ethanolamine trap to collect carbon dioxide.

The tests were performed at a concentration of approximately 0.41 mg/kg dry weight of soil. The test concentration was based on a field rate of 400 g a.s./ha. The test item [phenyl-U-^{14}C]-fluopicolide dissolved in acetonitrile (100 µL) was applied to the soil surface. Soil samples were adjusted to a moisture content equivalent to pF 2, seven days prior to application. The samples were incubated at 20 ± 1 °C under aerobic conditions in the dark for up to 200 days.

2. Sampling

Single samples each were removed for analysis after 0, 7, 14, 22, 34, 49, 64, 78, 98 and 120 days incubation in Sarotti soil and 0, 7, 14, 22, 34, 49, 64, 78, 98, 120, 160 and 200 days incubation in Münster soil.

3. Analytical Procedures

DAT 0 and DAT 7 soil samples were extracted at ambient temperature initially with acetonitrile / water (4/1, *v/v*) followed by soxhlet extraction with acetonitrile. At timepoints after DAT 7 soil samples were extracted at ambient temperature initially with acetonitrile, followed by acetonitrile / water (4/1, *v/v*) and finally by soxhlet extraction with acetonitrile. After removal of the soil samples from the incubation flasks, the flasks were soaked in acetone to remove any residual activity.

Radioactivity in extracts was determined by liquid scintillation counting (LSC). Soil extracts were concentrated and analysed by HPLC with radiodetection. Degradation products were identified by comparison of the retention times of reference standards. Selected samples were analysed by thin layer chromatography (TLC) to confirm the results obtained by HPLC. A selected sample was analysed by LC-MS to confirm the structural identity of the degradation products.

Volatile radioactivity in volatile traps was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion. As a result of low recovery of radioactivity in the Sarotti soil the radioactivity remaining associated with soxhlet thimbles was quantified by combustion and / or extraction at ambient temperature with methanol.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide, M-01 and M-03 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each soil between the SFO and FOMC fits for fluopicolide. For the Münster soil, the FOMC fit resulted in a lower χ^2 err% value than the SFO fit, and the DFOP model was therefore also fitted. The resulting DFOP fit was similar visually to the SFO fit, with low confidence in rate constant k_1 (p>0.2), and was not accepted. The FOMC fit was also not accepted, as extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using this model (EFSA, 2009). The SFO model therefore provided the most appropriate description of fluopicolide degradation in the Münster soil. For the Sarotti soil, the SFO model provided a better fit than the FOMC model to the fluopicolide residues, with a lower χ^2 err% value.

Metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soils incubated at 20 °C following application of [phenyl-U-^{14}C]-fluopicolide are summarized in Table 7.1.1.1- 9 to Table 7.1.1.1- 10.

Table 7.1.1.1- 9: Degradation of [phenyl-U-^{14}C]-fluopicolide at 20 °C in Sarotti soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)					
	0	7	14	22	34	49
Fluopicolide	98.2	81.5	76.2	79.4	75.6	72.3
M-01 (AE C653711)	nd	4.4	6.4	7.7	10.0	12.2
M-03 (AE 0608000)	nd	nd	nd	nd	nd	nd
Total extractable radioactivity[A]	98.2	85.9	82.6	87.2	85.8	84.5
Apparatus Wash	na	0.02	0.02	0.02	0.02	0.01
Extracted thimble combusted	na	0.0	3.6	na	na	0.2
Non-extractable radioactivity	0.7	0.7	3.1	2.4	4.8	6.0
^{14}C-Carbon dioxide [B]	na	0.1	0.2	0.2	0.4	0.8
Total radioactivity	98.9	86.7	89.5	89.8	91.0	91.5

Compound	Incubation time (DAT)			
	64	78	98	120
Fluopicolide	75.7	70.2	64.5	52.8
M-01 (AE C653711)	10.2	17.3	18.8	25.0
M-03 (AE 0608000)	nd	nd	nd	nd
Total extractable radioactivity[A]	85.9	87.4	85.3	77.8
Apparatus Wash	0.02	0.01	0.01	0.01
Extracted thimble combusted	na	na	0.1	na
Non-extractable radioactivity	5.2	4.4	6.5	12.0
^{14}C-Carbon dioxide [B]	0.8	2.0	2.0	2.5
Total radioactivity	91.9	93.8	91.9	92.4

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of applied radioactivity (% AR)
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts, including extracts of Soxhlet thimbles.
[B] Other volatile radioactivity was negligible at all timepoints

Table 7.1.1.1- 10: Degradation of [phenyl-U-^{14}C]-fluopicolide at 20 °C in Münster soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)					
	0	7	14	22	34	49
Fluopicolide	102.1	90.3	90.6	86.5	82.5	79.4
M-01 (AE C653711)	nd	1.6	2.4	3.2	5.0	6.8
M-03 (AE 0608000)	nd	1.9	3.0	4.2	5.2	6.7
Total extractable radioactivity[A]	102.1	93.8	96.0	93.8	92.6	92.8
Apparatus Wash	na	nd	0.02	0.02	0.02	0.03
Non-extractable radioactivity	0.2	0.2	0.4	0.5	1.1	1.2
^{14}C-Carbon dioxide [B]	na	0.0	0.0	0.0	0.0	0.1
Total radioactivity	102.3	94.0	96.4	94.4	93.7	94.1

Compound	Incubation time (DAT)					
	64	78	98	120	160	200
Fluopicolide	76.6	82.2	72.4	68.0	64.1	54.8
M-01 (AE C653711)	9.3	11.2	14.5	14.5	16.5	25.4
M-03 (AE 0608000)	7.8	8.3	9.7	10.6	10.4	10.3
Total extractable radioactivity[A]	93.7	101.7	96.6	93.0	91.1	90.5
Apparatus Wash	0.02	0.01	0.01	0.09	nd	0.01
Non-extractable radioactivity	0.9	1.9	1.5	2.8	3.8	5.8
^{14}C-Carbon dioxide [B]	0.1	0.1	0.2	0.3	0.2	0.7
Total radioactivity	94.7	103.3	98.3	96.2	95.1	97.0

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of applied radioactivity (% AR)
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts
[B] Other volatile radioactivity was negligible at all timepoints

B. Material Balance

Material balances ranged from 98.9 to 86.7% AR in Sarotti soil and 103.3 to 93.7% AR for Münster soil.

C. Extractable and Non-Extractable Residues

For samples of Sarotti soil incubated with [phenyl-U-^{14}C]-fluopicolide, total extractable radioactivity decreased from 98.2% AR on DAT 0 to 77.8% AR by DAT 120. The total of non-extractable residues (NER) increased from 0.7% AR at DAT 0 to 12.0% AR byt the end of the study (DAT 120).

For samples of Münster soil incubated with [phenyl-U-^{14}C]-fluopicolide, total extractable radioactivity decreased from 102.1% AR on DAT 0 to 90.5% AR after 200 days of incubation. NER was 0.2% AR at DAT 0 and increased to 5.8% AR by the end of the study (DAT 200).

D. Volatile Radioactivity

Radiolabelled carbon dioxide evolved accounted for a maximum of 2.5% of the applied radioactivity in Sarotti soil by the end of the study and <1% in the Münster soil. Other volatile radioactivity was negligible at all timepoints.

E. Degradation of Parent Compound

Fluopicolide was the principal radiolabelled component detected. Levels of parent accounted for 100% of extracted radioactivity at DAT 0 and declined to 53 and 55% of applied radioactivity at termination of the study in Sarotti and Münster soils respectively. In addition to parent material, M-01 (AE C653711, 2,6-dichlorobenzamide, BAM) was detected. Levels increased over the course of the incubation period and accounted for 25% of applied radioactivity at DAT 120 in Sarotti and DAT 200 in Münster soils. A second metabolite M-03 (AE 0608000, called RPA 427967 in the report) was detected in Münster soil only, and accounted for 10% of applied radioactivity at study termination.

F. Degradation Kinetics

Fluopicolide degraded slowly in Sarotti silty clay loam and Münster loamy sand soils under non-sterile conditions. The reported DT_{50} values were 194 and 266 days for Sarotti and Münster soils, respectively. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.1.1- 11. Best fit kinetics are highlighted in bold.

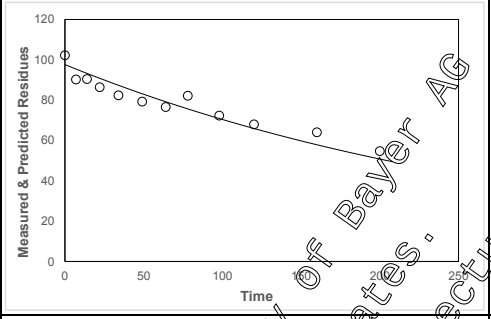
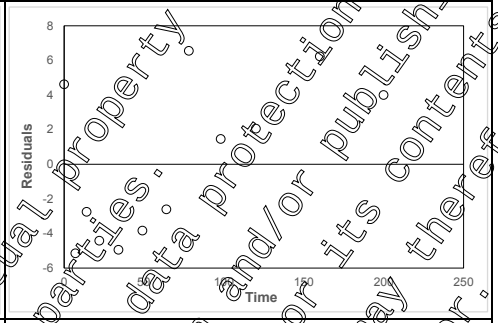
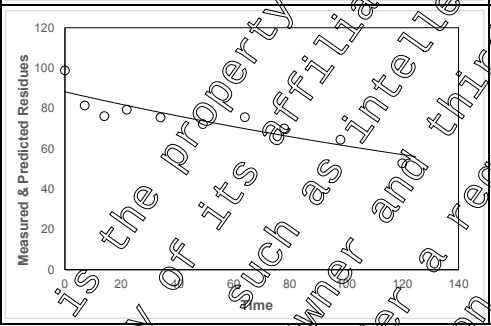
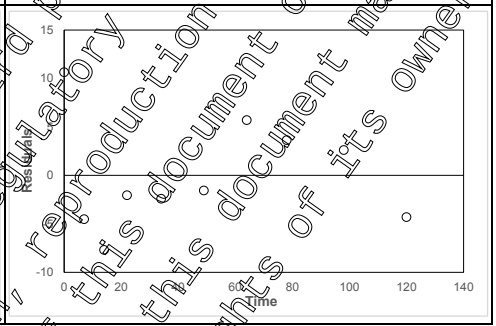
Table 7.1.1.1- 11: Degradation rate of fluopicolide under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2 - % error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Münster, Keirs (2003)	**SFO**	**97.68**	**k 0.00327**	**4.43**	**7.63E-11**	**0.002595**	**0.004**	**212**	**704.1**
	FOMC	96.95	α 0.455 β 100.7	3.56	n.r. n.r.	0.1406 -3.064	0.769 204.4	361.2	>10000
	DFOP	96.59	k1 0.255 k2 0.002841 g 0.01594	3.74	0.2082 1.57E-11 n.r.	-0.3506 0.002321 0.003067	0.861 0.003 0.029	238.3	804.9
Sarotti, Keirs (2003)	**SFO**	**88.25**	**k 0.003626**	**5.65**	**3.04E-05**	**0.002281**	**0.005**	**191.2**	**635**
	FOMC	89.46	α 0.5823 β 115.4	5.9	n.r. n.r.	-1.488 -440.8	2.652 671.5	264	5901

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.1.1- 12: Degradation of fluopicolide under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Münster SFO Keirs (2003)		
Sarotti SFO Keirs (2003)		

III. Conclusion

[Phenyl-U-^{14}C]-labelled fluopicolide was slowly degraded in Sarotti and Münster soils under aerobic conditions, with 53% and 55% of the applied radioactivity remaining as parent compound, respectively, at the end of the incubation period. Less than 3% of the radioactivity was detected as $^{14}CO_2$, indicating very slow mineralization of fluopicolide to CO_2. Organic volatiles were not detected in either soil. The primary metabolic pathway involved the oxidation of fluopicolide to form the hydroxylated metabolite M-03 (AE 0608000), which was then cleaved to form M-02 (AE C653711) containing the phenyl ring. M-02 was detected as a major degradation product reaching a maximum of 25.4% at the end of the incubation period. M-03 was observed in the acidic Münster soil only, where it reached a maximum of 10.6% of applied at DAT 120.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} values of 191.2 days and 212.0 days were determined for the degradation fluopicolide in non-sterile Sarotti and Münster soils, respectively.

Assessment and conclusion by applicant

The study was conducted in accordance with SETAC 1.1 (1995). The study is considered valid to assess the aerobic degradation of [phenyl-U-^{14}C]-fluopicolide in soil.

Data Point:	KCA 7.1.1.1/03
Report Author:	[redacted]
Report Year:	2020
Report Title:	Fluopicolide - Statement on potential for formation of stereogenic elements: M-03 (AE 0608000)
Report No:	VC/19/039C
Document No:	M-687284-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	None
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	not applicable
Acceptability/Reliability:	Yes

Executive Summary

This document addresses whether any further data or risk assessments are required for potential stereogenic elements associated with fluopicolide. Fluopicolide does not contain any stereogenic centres but forms a metabolite M-03 (AE 0608000) which contains one chiral carbon atom and consequently could exist as a pair of enantiomers. The EFSA guidance document (EFSA, 2019) on how to perform risks assessments for plant protection products that contain stereoisomers has been used to assess if any further information is required for M-03.

M-03 has been observed only in soil matrices where it can exceed 5% AR in acidic soils dosed with parent. The metabolite is readily degraded in acidic soils and very rapidly degraded in soils at neutral or slightly alkaline soil pH. No information on the composition of possible enantiomers of M 03 formed in soil or their individual transformation or interconversion is available. M-03 has not been observed as a metabolite in plant, animal or water matrices.

Ecotoxicological studies for M-03 are available with the soil organisms *Eisenia fetida* and *Folsomia candida*, and on microbial nitrogen transformation. No effects on survival and reproduction were seen for *E. fetida* and *F. candida* up to 100 mg/kg, the highest concentration tested. For *Hypoaspis aculeifer* no ecotoxicological study with M-03 is available. However, an endpoint is extrapolated from the study with the parent active substance assuming 10-fold higher toxicity compared to the parent active substance. Endpoints are corrected by a factor of 2 as the Log P for M-03 is > 2. The process of microbial nitrogen transformation was not adversely impacted up to 2.78 mg/kg (effects on nitrate formation rate < 25%), the highest concentration tested.

Information is not available on whether a specific stereoisomer of M-03 is enriched in ecotoxicological studies and/or whether the ecotoxicity properties of M-03 stereoisomers are comparable. An additional safety factor of 2 is advised by EFSA to account for the remaining uncertainty with regard to potential isomerization of M-03.

Ecotoxicological endpoint [b]		PEC_{soil}	TER	Critical trigger
E. fetida	$NOEC_{corr}$ ≥ 25 mg/kg [a, b] (with uncertainty factor)	0.030 mg/kg	≥833.3	5
F. candida	$NOEC_{corr}$ ≥ 25 mg/kg [a, b] (with uncertainty factor)	0.030 mg/kg	≥833.3	5
H. aculeifer	$NOEC_{corr}$ = 25 mg/kg * [a, b] (with uncertainty factor)	0.030 mg/kg	≥833.3	5
N-transformation	Effects < 25% at 1.39 mg/kg [b] (with uncertainty factor)	0.030 mg/kg	≥46.3	1

* NOEC extrapolated from Hypoaspis aculeifer reproduction study with the active substance (NOEC ≥ 1000 mg as/kg), assuming 10x higher toxicity of M-03 compared to the parent active substance
[a] As LogP for M-03 is >2 the ecotoxicological endpoints for *E. fetida* and *F. candida* are divided by a correction factor of 2 ($NOEC_{corr.}$ = NOEC corrected)
[b] Endpoint corrected by an additional safety factor of 2 to account for uncertainty with regard to potential isomerization of M-03 (EFSA, 2019)

No unacceptable risk to soil organisms is anticipated from enantiomers of M-03 forming from fluopicolide.

I. Materials and Methods

Formation and decline of M-03 in soil

In soil the primary degradation pathway of fluopicolide initially involves oxidation of parent to form the hydroxylated metabolite M-03 (AE 0608000) which contains one chiral carbon atom and which consequently could exist as a pair of enantiomers.

M-03 exceeds 5% of applied radioactivity in three soils (Münster, Lamberton and Pikeville soils) and so further assessment of the impact of the enantiomers of M-03 in the environment is needed.

Table 7.1.1.1- 13: Maximum formation of primary metabolites in aerobic soil studies treated with [^{14}C]-fluopicolide

Report	Soil	Soil pH ($CaCl_2$)	Maximum M-03 detected (% AR)
[redacted] 2003; M-201230-02-1	Münster	4.9 [A]	10.6
[redacted] 2003; M-241052-01-1	Lamberton (25°C)	5.9	6.3 / 7.8 [B]
[redacted] 2019; M-655056-01-1	Pikeville	4.5	6.6

nd = Not detected
[A] pH in KCl
[B] Maximum formation of M-03 in soil treated with [U-^{14}C-phenyl]-fluopicolide / [2,6-^{14}C-pyridyl]-fluopicolide

The extent of M-03 levels in soil has strong pH dependence and the metabolite was only observed as a major metabolite in acidic soils (<pH 6) at a maximum of 10.6% AR, whilst in neutral to alkali soils it was either not detected or detected only occasionally at low levels. The degradation rate of M-03 is extremely rapid except in soils which are highly acidic. In very acidic soils the degradation is bi-phasic with a rapid initial degradation phase followed by a second slower degradation phase.

Table 7.1.1.1- 14: Best-fit soil DT_{50} values for M-03 in laboratory soil studies determined at 20 °C

Report	Soil	pH ($CaCl_2$)	Best-fit kinetic	χ2 err %	t-test	Best-fit DT_{50} (days)	Best-fit DT_{90} (days)
[redacted] 2003; M-241188-01-1	Abington	7.2	SFO	1.467	p=0.01147	0.1	0.3
	Sarotti	7.1	SFO	3.113	p=0.02458	0.1	0.3
	Münster	4.9	HS	12.22	k1: p=1.45e-06 k2: p= -	4.4	644.9
	Pikeville	5.4	DFOP	8.478	k1: p=4.98e 09 k2: p=0.149	2.1	9.1
[redacted] 2016; M-565219-01-1	Brierlow	5.3	SFO	9.455	p=6.21E-09	2.5	8.4
	H[redacted]	6.0	SFO	7.977	p=4.53E-10	0.9	2.9

Kinetic evaluation of trigger endpoints is taken from M-685680-01-1

M-03 was included as an analytical target in a number of field dissipation and accumulation studies. The metabolite was detected in the 0-10 cm soil depth at two out of the six trial sites. In Huntlosen soil (pH 5.3) M-03 was found at concentrations in individual replicates ranging from < LOQ to 0.019 mg/kg throughout the trial. In Philippsburg soil (pH 6.4), M-03 was detected only in two soil samples throughout the trial at a maximum of 0.006 mg/kg. In all other soils M-03 was analysed for but not detected (LOQ 0.005 mg/kg). It was never detected in the accumulation phase of field trials.

A lysimeter study ([redacted] 2004; M-218506-01-1) was conducted in Münster soil with [2,6-^{14}C-pyridyl]-fluopicolide. Leachate samples were collected regularly for 3 years. M-03 was included as reference standard but the metabolite was never detected in leachate throughout the duration of the study. M-03 was also included as an analytical target in a field leaching study ([redacted], 2003; M-223180-01-2) conducted with fluopicolide at the Philippsburg site. Suction samplers (45) were employed to collect soil water at 5 different depths (at 30, 50, 85, 120 and 150 cm soil depth) throughout the soil profile at *ca.* 70 timepoints over three years. M-03 was detected only once throughout the three years of the study at a concentration of 0.067 µg/L at 30 cm depth.

Formation and decline of M-03 in aquatic environments

M-03 (AE 0608000) was not detected in aquatic environments.

As discussed earlier the metabolite was not detected in lysimeter leachate and was detected only once in soil water in a field leaching study. Both studies were conducted with acidic soils in which there is greatest potential for detecting M-03 which is less rapidly degraded in such soils.

The hydrolysis of M-03 ([redacted] 2004; M-236241-01-2) was investigated to provide further information on its fate in the aquatic environment. The metabolite was shown to be hydrolytically labile under acidic, neutral and alkaline conditions at 20°C. The rate of hydrolysis was strongly dependent on pH with best-fit DT50 values of 7.3 minutes, 39.1 minutes, 4.4 hours and 45.5 hours at pH 8, 7, 6 and 5, respectively.

Table 7.1.1.1- 15: Best-fit aquatic DT_{50} values for M-03 in a sterile hydrolysis study determined at 20 °C

Buffer pH	Best-fit kinetic	χ2 err %	t-test	Best-fit DT_{50} (hours)	Best-fit DT_{90} (hours)
5.1	SFO	0.423	p < 0.01	45.5	151
6.1	DFOP	0.491	k1 < 0.01, k2 < 0.01	4.43	16
7.1	DFOP	1.24	k1 < 0.01, k2 < 0.01	0.651	2.76
8.1	DFOP	1.12	k1 < 0.01, k2 < 0.01	0.121	0.5

II. Results and Discussion

The fluopicolide metabolite M-03 has not been observed as a metabolite in plant, animal or water matrices, but exceeds 5% AR in soil and so further assessment of the impact of the enantiomers of M-03 in the environment is needed. No information on the composition of possible enantiomers formed in soil or their individual transformation or interconversion is available.

Ecotoxicological risk assessment for M-03 in soil

Ecotoxicological studies for M-03 are available with the soil organisms *Eisenia fetida* and *Folsomia candida*, and on microbial nitrogen transformation. No effects on survival and reproduction were seen for *E. fetida* and *F. candida* up to 100 mg/kg, the highest concentration tested. For *Hypoaspis aculeifer* no ecotoxicological study with M-03 is available. However, an endpoint is extrapolated from the study with the parent active substance assuming 10-fold higher toxicity compared to the parent active substance. Endpoints are corrected by a factor of 2 as the Log P for M-03 is > 2. The process of microbial nitrogen transformation was not adversely impacted up to 2.78 mg/kg (effects on nitrate formation rate < 25%), the highest concentration tested.

Test system	Study duration	Relevant ecotoxicological endpoint	Reference
Eisenia fetida, reproduction (OECD 222)	56 days	$NOEC_{corr}$ ≥ 50 mg/kg [a]	[redacted] 2016; M-557757-01-1
Folsomia candida, reproduction (OECD 232)	28 days	$NOEC_{corr}$ ≥ 50 mg/kg [a]	[redacted] 2016; M-558337-01-1
Hypoaspis aculeifer, reproduction (extrapolated from a.s. study assuming 10x higher toxicity than the parent active substance)		$NOEC_{corr}$ 50 mg/kg*[a]	[redacted] 2015; M-548042-01-1
N-transformation (OECD 216)	28 days	Effects < 25% at 2.78 mg/kg	[redacted] 2016; M-555852-01-1

* NOEC extrapolated from *Hypoaspis aculeifer* reproduction study with the active substance (NOEC ≥ 1000 mg a.s./kg), assuming 10x higher toxicity of M-03 compared to the parent active substance

[a] As LogP for M-03 is >2 the ecotoxicological endpoints for *E. fetida*, *F. candida,* and *H. aculeifer* are divided by a correction factor of 2 (NOEC$_{corr.}$ = NOEC corrected)

The soil risk assessment for M-03 for *E. fetida*, *F. candida*, *H. aculeifer*, and N transformation has a high margin of safety. The critical trigger values in the soil risk assessment for M-03 are exceeded by factor > 333 for *E. fetida*, *F. candida, H. aculeifer* and by factor > 92 for N-transformation.

Ecotoxicological endpoint		PEC_{soil}	TER	Critical trigger
E. fetida	$NOEC_{corr}$ ≥ 50 mg/kg [a]	0.030 mg/kg	≥1666.7	5
F. candida	$NOEC_{corr}$ ≥ 50 mg/kg [a]	0.030 mg/kg	≥1666.7	5
H. aculeifer	$NOEC_{corr}$ ≥ 50 mg/kg *[a]	0.030 mg/kg	≥1666.7	5
N-transformation	Effects < 25% at 2.78 mg/kg	0.030 mg/kg	≥92.7	1

* NOEC extrapolated from Hypoaspis aculeifer reproduction study with the active substance (NOEC ≥ 1000 mg a.s./kg), assuming 10x higher toxicity of M-03 compared to the parent active substance
[a] As LogP for M-03 is >2 the ecotoxicological endpoints for *E. fetida* and *F. candida* are divided by a correction factor of 2 ($NOEC_{corr.}$ = NOEC corrected)

Information is not available on whether a specific stereoisomer of M-03 is enriched in the ecotoxicological studies listed above and/or whether the ecotoxicity properties of M-03 stereoisomers are comparable. For this case EFSA (2019) proposes an uncertainty factor is used in the ecotoxicological risk assessment. For two isomers the EFSA guidance document (EFSA, 2019) advises the No Observed Effects Concentration (NOEC) can be divided by 2 provided the TER is exceeded. Considering an additional safety factor of 2 the risk assessment would still indicate no unacceptable risk for soil organisms. The risk assessment shows a high margin of safety.

Applying an additional safety factor = 2 on ecotoxicological endpoints to account for remaining uncertainty with regard to potential isomerization of M-03				
Ecotoxicological endpoint [b]		**PEC_{soil}**	**TER**	**Critical trigger**
E. fetida	$NOEC_{corr}$ ≥ 25 mg/kg [a, b] (with uncertainty factor)	0.030 mg/kg	≥833.3	5
F. candida	$NOEC_{corr}$ ≥ 25 mg/kg [a, b] (with uncertainty factor)	0.030 mg/kg	≥833.3	5
H. aculeifer	$NOEC_{corr}$ ≥ 25 mg/kg * [a, b] (with uncertainty factor)	0.030 mg/kg	≥833.3	5
N-transformation	Effects < 25% at 1.39 mg/kg [b] (with uncertainty factor)	0.030 mg/kg	≥46.3	1

* NOEC extrapolated from Hypoaspis aculeifer reproduction study with the active substance (NOEC ≥ 1000 mg a.s./kg), assuming 10x higher toxicity of M-03 compared to the parent active substance
[a] As LogP for M-03 is >2 the ecotoxicological endpoints for *E. fetida* and *F. candida* are divided by a correction factor of 2 ($NOEC_{corr.}$ = NOEC corrected)
[b] Endpoint corrected by an additional safety factor of 2 to account for uncertainty with regard to potential isomerization of M-03 (EFSA, 2019)

No unacceptable risk to soil organisms is concluded from enantiomers of M-03 forming from fluopicolide.

III. Conclusion

The fungicide fluopicolide does not contain any stereogenic centres. The primary degradation pathway initially involves oxidation of parent to form the hydroxylated metabolite M-03 (AE 0608000) which contains one chiral atom and which consequently could exist as a pair of enantiomers. However this metabolite is transient, being rapidly cleaved to form further metabolites which also do not contain any stereogenic centres.

It has been demonstrated that there is no unacceptable risk from enantiomers of M-03 formed in soil from fluopicolide.

Assessment and conclusion by applicant:

The position paper is considered valid to aid assessment of the route of degradation of fluopicolide in soil.

CA 7.1.1.2 Anaerobic degradation

The route of anaerobic degradation of fluopicolide in soil has been investigated in KCA 7.1.1.2/01, which was evaluated during the previous EU review and is still considered acceptable to assess the anaerobic degradation of fluopicolide. No new anaerobic data is submitted.

Report reference	Author, Year	Phenyl Label	Pyridyl Label	Comment
KCA 7.1.1.2/01 M-241050-01-1	[redacted] 2003	✓	✓	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.

Data Point:	KCA 7.1.1.2/01
Report Author:	[redacted]
Report Year:	2003
Report Title:	Route and rate of degradation of [2,6-14C-pyridinyl] and [U-14C-benzoyl]-AE C638206[in a European sandy loam under laboratory anaerobic conditions at 20 degrees C
Report No:	B004072
Document No:	M-241050-01-1
Guideline(s) followed in study:	EU (=EEC) 95/36/EC of July 1995
Deviations from current test guideline:	Yes. The soil was not incubated under aerobic conditions for 30 days prior to flooding. Due to the rate of degradation of fluopicolide in aerobic soil this will not impact significantly on the results of the study.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of fluopicolide was investigated in a European soil flooded with an overlying layer of water under laboratory anaerobic conditions. Abington soil was classified as a sandy loam soil according to USDA classification. Once the systems were anaerobic [phenyl-U-^{14}C]-labelled fluopicolide or [2,6-pyridyl-^{14}C]-labelled fluopicolide was applied to evenly over the surface of the water at an application rate equivalent to 400 g /ha. The radiochemical purity was > 99 % for both radiolabelled test items. The specific activities were 5.33 and 5.88 MBq/mg for [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]-labelled fluopicolide, respectively. Soil samples were then incubated in the dark under flooded anaerobic conditions at 20 °C for up to 120 days.

Samples were taken for extraction and analysis immediately after treatment (DAT 0) and after 16, 28, 56, 84 and 120 days of incubation. For each analysis, the water and soil were separated and analyzed separately. Soil samples were exhaustively extracted with up to four successive extractions with acetonitrile / water (4 / 1, v/v) at ambient temperature followed by one Soxhlet extraction using acetonitrile. Water samples and concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC) and the identity of degradation products confirmed by normal phase thin layer chromatography (TLC).

Recovery of radioactivity was quantitative throughout the study except for one sample (DAT 84 treated with [2,6-pyridyl-^{14}C]-fluopicolide). Overall mean mass balances were 95.9% AR samples treated with [phenyl-U-^{14}C]-fluopicolide and 94.5% AR for samples treated with [2,6-pyridyl-^{14}C]-fluopicolide.

The majority of the applied radioactivity was extractable using acetonitrile/water at ambient temperature. Radioactivity from Soxhlet extractions, which used acetonitrile only, showed only a slight increase over the course of the study. At study termination (DAT 120) in the phenyl labelled treatment 11%, 72%, and 8% AR was recovered in the water phase, ambient extract, and Soxhlet extract, respectively. Similarly in the pyridyl labelled treatment, 14%, 71%, and 7% of applied were recovered in the water phase, ambient extract and Soxhlet extract, respectively at termination.

Non-extractable residue increased proportionately with the decrease in extractable radioactivity over the 120 day study. The maximum amount of non-extractable residues was 4.5% AR (DAT 120) in the phenyl label and 4.4 % AR (DAT 120) in the pyridyl label.

Mineralization to carbon dioxide was a very minor pathway under anaerobic conditions (maximum of 0.1% AR). There was no volatilization of fluopicolide or its metabolites.

After 120 days anaerobic incubation at 20 °C, fluopicolide slowly degraded to 88.6% of the radioactivity applied in the phenyl label and 83.0% in the pyridyl label. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of 585.4 days and DT_{90} value of 1945 days in Abington soil.

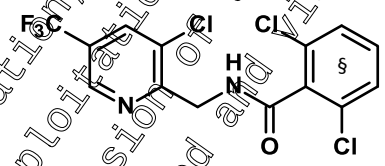
The metabolic pathway under anaerobic conditions was the same as that observed under aerobic conditions, with cleavage of fluopicolide to form M-01 (maximum of 2.1% in the phenyl label treated soil) and M-02 (maximum of 8.9% in the pyridyl label treated soil). No unique metabolites were formed under anaerobic conditions.

I. Materials and Methods

A. Materials

1. Test Items

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

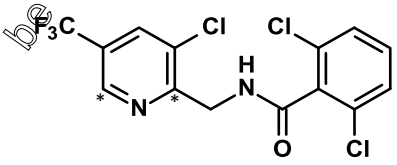


§ Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.33 MBq/mg

Radiochemical Purity: 100% (mean of HPLC and TLC analyses)

[2,6-Pyridyl-^{14}C]-fluopicolide



* Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.88 MBq/mg

Radiochemical Purity: 99.97% (mean of HPLC and TLC analyses)

2. Test Soil

The study was performed using one test soil as characterized in Table 7.1.1.2- 1.

Table 7.1.1.2- 1: Physico-chemical properties of test soil

Parameter		Soil
Soil Designation		Abington
Geographic Location		
City		Abington
Country		England, UK
Textural Classification (USDA)		sandy loam
Sand [50 - 2000 µm]	(%)	67
Silt [2 – 50 µm]	(%)	22
Clay [< 2 µm]	(%)	11
pH		
in H_2O (1:1)		7.4
in $CaCl_2$ (1:1)		7.2
Organic Matter (%)		3.8
Organic Carbon (%)*		2.2
Cation Exchange Capacity (meq/100 g)		18.0
Water Holding Capacity (%)		
maximum		56.1
at 1/10 bar		18.8
at 1/3 bar		14.6
Bulk Density (disturbed, g/cm³)		1.26
Soil Microbial Biomass (µg microbial C/g soil)		
Initial (DAT 0)		90.8
Final (DAT 126)		71.6

* Calculated by dividing organic matter content by 1.72
Biomass samples were untreated.

B. Study Design

1. Experimental Conditions

The test system consisted of flasks containing 50 g soil (dry weight basis) that were flooded with an overlying layer of deionised water. The depth of the surface water was approximately 1 cm above the soil surface and was maintained throughout the course of the study. Humidified nitrogen was passed through each treated flask continuously to maintain anaerobic conditions. Tests were performed in flow through systems consisting of glass flasks each containing 50 g soil and attached to an ethylene glycol trap to collect organic volatiles followed by an ethanolamine trap to collect carbon dioxide. Soil samples were flooded 54 days prior to application in order to allow the system to become anaerobic.

The tests were performed at a concentration of approximately 0.41 mg/kg dry weight of soil. The test concentration was based on a field rate of 400 g a.s./ha. The test items [phenyl-U-^{14}C]- or [2,6-pyridyl-^{14}C]-fluopicolide, dissolved in acetonitrile (406 and 460 µL, respectively), were applied drop wise onto the surface of the water. The samples were incubated at 20 ± 1 °C under anaerobic conditions in the dark for up to 120 days.

2. Sampling

Single samples were removed for analysis after 0, 16, 28, 56, 84 and 120 days of incubation. Untreated samples were analysed for biomass at the beginning and end of the experiment.

3. Analytical Procedures

For each analysis, the water and soil were separated and analyzed separately. The soil was extracted extracted up to four times at ambient temperature with acetonitrile/water (4/1, v/v). After ambient extractions, the soil was further extracted by Soxhlet using only acetonitrile. Radioactivity in the water phase, extracted from soil and in the volatile traps was quantified by liquid scintillation counting (LSC). The remaining soil residue was combusted to quantify non-extractable soil residue.

Water samples were filtered and analysed by HPLC with radiodetection directly. Ambient soil extracts and Soxhlet extracts were concentrated and analysed separately by HPLC with radiodetection. Degradation products were identified by comparison of the retention times of reference standards and confirmed by TLC co-chromatography with reference items. A peak of 300 dpm, corresponding to 0.9 ng fluopicolide, was readily determined by TLC and HPLC quantitation methods used.

Volatile radioactivity in volatile traps was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). As the degradation was investigated using two radiolabel positions, and similar behaviour was observed for each, these radiolabels have been considered as true replicates, and included together in a single optimisation.

The un-normalised data was best fit by the simple first order (SFO) model in Abington soil, with an $\chi 2$ error of 1.82%.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soil incubated at 20 °C following application of [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]-fluopicolide are summarized in Table 7.1.1.2- 2 to Table 7.1.1.2- 3.

Table 7.1.1.2- 2: Degradation of [phenyl-U-^{14}C]-fluopicolide at 20 °C in Abington soil under anaerobic conditions [% AR]

Compound	Incubation time (DAT)					
	0	16	28	56	84	120
Fluopicolide	96.3	97.2	91.6	90.7	85.6	88.6
M-01 (AE C653711)	nd	1.6	0.4	0.4	0.6	2.1
Water Phase	76.2	18.3	12.6	13.8	12.6	11.0
Ambient Extracts	21.1	76	75.2	76.4	67.7	71.9
Soxhlet Extracts	n/a	4.5	4.5	4.4	6.2	7.8
Total Soil Extracted [A]	21.1	80.5	79.7	77.8	73.9	79.7
Non-extractable radioactivity	1.4	1.7	2.8	3.8	3.7	4.5
^{14}C-Carbon dioxide including other volatiles [B]	na	nd	nd	nd	nd	0.1
Total radioactivity	98.7	100.5	95.2	95.4	90.3	95.3

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] The total soil extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[B] Other volatile radioactivity was < 0.05 % AR at any time point

Table 7.1.1.2- 3: Degradation of [2,6-pyridyl-^{14}C]-fluopicolide at 20 °C in Abington soil under anaerobic conditions [% AR]

Compound	Incubation time (DAT)					
	0	16	28	56	84	120
Fluopicolide	95.5	93.9	90	92.9	82.5	83.0
M-02 (AE C657188)	nd	nd	nd	0.5	0.7	8.9
Water Phase	70.9	21.1	17.5	14	12.2	14.3
Ambient Extracts	24.6	69.3	69.2	74.6	65.5	70.6
Soxhlet Extracts	na	3.5	3.9	5.0	5.7	7.0
Total Soil Extracted [A]	24.6	72.8	73.1	79.6	71.2	77.6
Non-extractable radioactivity	1.1	1.9	3.0	3.8	4.3	4.4
^{14}C-Carbon dioxide including other volatiles [B]	na	nd	nd	nd	nd	nd
Total radioactivity	96.6	95.5	93.6	97.4	87.7	96.3

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] The total soil extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[B] Other volatile radioactivity was < 0.05 % AR at any time point

B. Material Balance

For samples of Abington soil incubated at 20 °C, material balances ranged from 90.3 to 100.5% AR for [phenyl-U-^{14}C]-fluopicolide (mean 95.9% AR) and from 87.7 to 97.4% AR for [2,6-pyridyl-^{14}C]-fluopicolide (mean 94.5% AR).

C. Extractable and Non-Extractable Residues

The majority of the applied radioactivity was extractable throughout the study. For samples incubated with [phenyl-U-^{14}C]-fluopicolide, total extractable radioactivity ranged from 98.8% AR at DAT 16 to 86.5% AR at DAT 84. At study termination (DAT 120) 11.0%, 71.9% and 7.8% was recovered in the water phase, ambient extracts and Soxhlet extracts, respectively. The total of non-extractable residues (NER) increased from 1.4% AR at DAT 0 to 4.5% AR by the end of the study (DAT 120).

For samples incubated with [2,6-pyridyl-^{14}C]-fluopicolide, total extractable radioactivity ranged from 95.5% AR at DAT 0 to 83.4% AR at DAT 84. At study termination (DAT 120) 14.3%, 70.6% and 7.0% was recovered in the water phase, ambient extracts and Soxhlet extracts, respectively. The total of non-extractable residues (NER) increased from 1.1% AR at DAT 0 to 4.4% AR by the end of the study (DAT 120).

D. Volatile Radioactivity

Mineralization to carbon dioxide was a very minor pathway under anaerobic conditions. Only trace amounts of radioactivity were recovered in the volatile traps containing ethanolamine (maximum of 0.1% of applied). There was no volatilization of fluopicolide or its metabolites.

E. Degradation of Parent Compound

Fluopicolide slowly degraded in soil under anaerobic conditions with 88.6% and 83.0% of the applied radioactivity remaining as parent compound in the phenyl and pyridyl label treated soil by DAT 120.

Following application of [phenyl-U-^{14}C]-fluopicolide, the active substance was degraded to form M-01 (called AE C653711, BAM in the report), which was observed at peak levels of 2.1% AR by DAT 120 in anaerobic soil.

Following application of [2,6-pyridyl-^{14}C]-fluopicolide, the active substance was degraded to form M-02 (called AE C657188, PCA in the report), which was observed at a maximum of 8.9% AR by DAT 120.

F. Degradation Kinetics

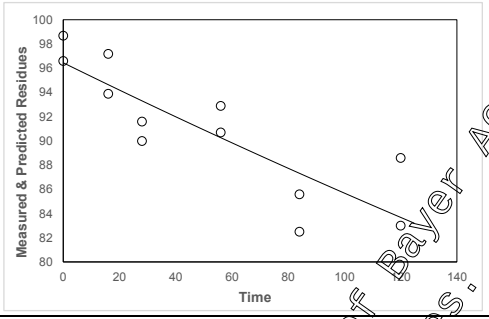
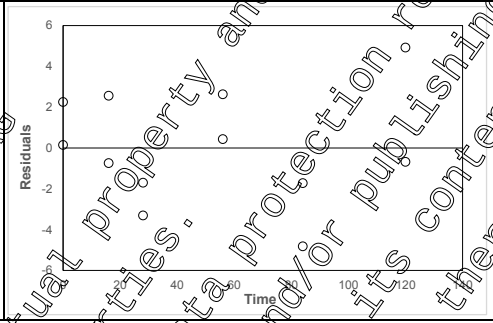
Fluopicolide degraded slowly in the Abington sandy loam soil under anaerobic conditions. The reported DT_{50} values were 471 and 377 days (mean = 424 days) for the phenyl- and pyridyl- labels, respectively. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.1.2- 4.

Table 7.1.1.2- 4: Degradation rate of fluopicolide under anaerobic conditions at 20 ºC (best-fit DT_{50} values for trigger endpoints)

Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ², %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
SFO	**96.45**	**k 0.001184**	**1.82**	**0.000204**	**0.0007368**	**0.002**	**585.4**	**1945**
FOMC	97.86	α 0.1011 β 37.7	1.77	n.r. n.r.	-0.07646 -84.84	0.279 160.2	>10000	>10000
DFOP	97.83	k1 0.01462 k2 2.34E-14 g 0.1602	2	0.427 0.5 n.r.	-0.1359 -0.01288 -1.973	0.165 0.013 2.294	>10000	>10000

Best fit model highlighted in bold

Table 7.1.1.2- 5: Degradation of fluopicolide under anaerobic conditions in Abington soil at 20 °C with time

Soil Model Reference	Modelled vs observed	Residuals
Abington SFO M-241050-01-1		

III. Conclusion

Fluopicolide slowly degraded in sandy loam soil under anaerobic conditions. Initial dissipation of fluopicolide into the soil phase during the first two weeks of the study was rapid but thereafter the rate of dissipation from the water phase was slower. Approximately 89% and 83% of the applied radioactivity remained in the combined aqueous and soil extracts as fluopicolide in the phenyl and pyridyl label treated soils, respectively, at the end of 120 days. Less than 1% of the radioactivity was detected in the ethanolamine traps indicating very slow mineralization of fluopicolide to CO_2 under anaerobic conditions. Organic volatiles were not detected with either radiolabelled treatment. Unextracted soil bound residues accounted for 5% AR at the end of the study.

The metabolic pathway under anaerobic conditions was the same as that observed under aerobic conditions, with cleavage of fluopicolide to form M-01 (maximum of 2.1% in the phenyl label treated soil) and M-02 (maximum of 8.9% in the pyridyl label treated soil). No unique metabolites were formed under anaerobic conditions

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of 585.4 days for Abington soil under anaerobic conditions.

Assessment and conclusion by applicant:

The study was conducted in accordance with SETAC 1.1 (1995) and USEPA (= EPA) N, 162-1 (1982). The study is considered valid to assess the anaerobic degradation of [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]-fluopicolide in soil.

CA 7.1.1.3 Soil photolysis

The soil photolysis of fluopicolide has been investigated in Studies KCA 7.1.1.3/01, KCA 7.1.1.3/02 and KCA 7.1.1.3/03 which were evaluated during the previous EU review and are still considered acceptable. No new soil photolysis data is submitted.

Report reference	Author, Year	Phenyl Label	Pyridyl Label	Comment
KCA 7.1.1.3/01 M-201037-03-1	[redacted] 2009	×	✓	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.1.3/02 M-201033-03-1	[redacted]1999	✓	×	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.1.3/03 M-201038-03-1	[redacted]2005	✓	×	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.1.3/04 M-300764-03-1	[redacted] 2009	✓	-	Submitted and reviewed for first approval of fluopicolide, 2008 and 2009. Superseded.
KCA 7.1.1.3/05 M-286182-01-1	[redacted] 2007	-	✓	Submitted and reviewed for first approval of fluopicolide, 2007 and 2009. Not accepted. Superseded.

In addition, two statements submitted to the PRAPeR Expert Meetings in 2007 to 2009 are included in the supplementary dossier for procedural reasons. However, both statements have been superseded as they were written prior to FOCUS guidance document on degradation kinetics (2014). Neither statement is valid as all field dissipation studies have been re-evaluated according to EFSA guidance document (2014) following the procedures recommended for determining bulk soil $DegT_{50matrix}$ values for use in exposure modelling, which exclude soil surface processes such as soil photolysis. Consequently KCA 7.1.1.3/04 and KCA 7.1.1.3/05 are not summarised in this dossier.

Data Point:	KCA 7.1.1.3/01
Report Author:	[redacted]
Report Year:	2009
Report Title:	(Pyridyl-2, 6-14C) labelled AE C638206: photodegradation on sandy loam soil- report amendment 2
Report No:	18768
Document No:	M-201037-03-1
Guideline(s) followed in study:	EU (=EEC): 91/414
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The original soil photolysis report M-201037-01-1, which was evaluated and accepted in the DAR (2005), is included in the Baseline dossier. This report has subsequently been amended twice. The first report amendment (M-201037-02-1) added the initials 'OECD' to the GLP statement and has not been included in either the Baseline Dossier or this MCA 7 Dossier because it has been superseded by a second report amendment (KCA 7.1.1.3/01, M-201037-03-1) which is summarised below. In the second report amendment, part of Appendix 7 has been removed. In the original report Appendix 7 contained an extrapolation of natural sunlight measured outdoors with a Radiolux sensor to justify the light intensity used in the Heraeus suntest unit. However the light intensity value measured outdoors was unrealistically high as the sensor was not appropriate for measurements of natural sunlight outdoors. It should be noted that this had no effect on the calculation of the light intensity in the suntest experiment, which was measured by the Radiolux sensor under the correct conditions. The only impact of the outdoor measurements on the study is that the soil samples will have been irradiated at higher levels or for a longer period than was strictly necessary. To avoid confusion the report was amended by removing the extrapolation calculation from Appendix 7.

Executive Summary

The photolytic degradation of fluopicolide on soil surfaces was investigated in air-dried Abington sandy loam soil, prepared as a thin-layer on glass incubation vessels with a thickness of about 3 mm. The test item, [2,6-pyridyl-^{14}C]-fluopicolide, dissolved in acetonitrile was applied evenly to the soil surface at an application rate equivalent to 11.2 mg/kg. Treated soil samples were exposed to artificial irradiation from a Xenon lamp (with < 290 nm cut-off filter) with continuous irradiation for a period of 15 days at 20 ± 3 °C. A set of dark control samples treated at the same application rate were incubated in the dark at 20 ± 3 °C.

The mean recoveries of radioactivity were 97.1 % and 96.6 % AR for the irradiated and dark controls, respectively.

Fluopicolide was the principal radiolabelled component detected. In the irradiated samples, fluopicolide slowly degraded to 87% AR after 15 days. Mineralization to carbon dioxide was low at < 1 % AR. The degradation rate of fluopicolide was slightly enhanced in the presence of light leading to the formation of M-02 (called AE C657188 in the report) at 3% AR and two other very minor photodegradation products (each < 0.3% AR). No degradation of fluopicolide occurred during incubation in the dark.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014). A summary of the results is shown below. The best fit DT_{50} value calculated for fluopicolide was 235 days summer sunlight at 30-50 °N.

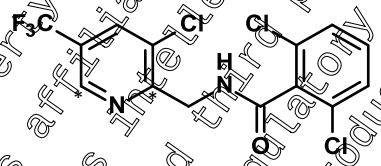
Soil	Best fit model	χ^2 – error [%]	DT_{50} [days summer sunlight @ 30-50 °N]	DT_{90} [days summer sunlight @ 30-50 °N]
Abington – pyridyl label	SFO	1.26	235	780

I. Materials and Methods

A. Materials

1. Test Items

[2,6-Pyridyl-^{14}C]-fluopicolide



* Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.99 MBq/mg (162 µCi/mg)

Radiochemical Purity: 99.8% (mean of HPLC and TLC analyses)

2. Test Soil

The study was performed using one test soil as characterized in Table 7.1.1.3- 1.

Table 7.1.1.3- 1: Physico-chemical properties of test soil

Parameter	Soil
Soil Designation	Abington
Geographic Location	
City	Abington
Country	England, UK
Textural Classification (USDA)	sandy loam
Sand [50 - 2000 µm] (%)	64.72
Silt [2 – 50 µm] (%)	18.64
Clay [< 2 µm] (%)	16.65
pH	
in H_2O (1:1)	7.6
in $CaCl_2$ (1:1)	7.4
Organic Carbon (%)	2.1
Cation Exchange Capacity (meq/100 g)	14.6

* USDA textural classification data taken from Appendix 5

B. Study Design

1. Experimental Conditions

Portions of fresh soil (5 g) were added to borosilicate glass incubation vessels, fitted with quartz glass covers. Water was added to form a slurry and the soil air dried at ambient temperature to give a soil depth of *ca*. 3mm. The soil samples were incubated inside glass units with moistened carbon dioxide free air drawn over the soil surface. A series of three traps for volatile products was connected to each unit. The first trap was empty (to prevent flow back), the second contained ethanediol to trap volatile organic components and the third contained ethanolamine to trap liberated $^{14}CO_2$.

A preliminary investigation was conducted over three days to determine an approximate rate of photodegradation of fluopicolide.

The test item [2,6-pyridyl-^{14}C]-fluopicolide (56 µg), dissolved in acetonitrile (100 µL), was applied evenly to the soil surface. Units were either irradiated continuously with light from an Heraeus Suntest xenon lamp, or incubated in the dark (non-irradiated samples). The application rate was equivalent to 11.2 mg/kg. The temperature of both irradiated and non-irradiated samples was maintained at a temperature of 20 ± 3 ºC throughout the incubation period. The photolytic degradation of fluopicolide was studied with continuous illumination under artificial sunlight for a period of up to 15 days, equivalent to 30 days of natural summer sunlight. The artificial sunlight was provided by a xenon arc lamp with filters to cut off any radiation below 290nm.

2. Sampling

In the preliminary investigation a single replicate was removed at time zero, 1, 2 and 3 days. A non-irradiated sample was maintained for 3 days.

In the definitive study duplicate soil samples were taken from both irradiated and non-irradiated systems after 0, 3, 5, 7, 10 and 15 days. One replicate was taken for analysis and the other was stored without further analysis. The replicate samples were planned for structural analysis of potential photo-degradation products which were not formed.

3. Analytical Procedures

Soil samples were extracted at ambient temperature initially with acetonitrile, followed by acetonitrile / water (4/1, v/v). After removal of the soil samples from the incubation flasks, the flasks were soaked in acetone to remove any residual activity. Radioactivity extracted from soil was quantified by liquid scintillation counting (LSC).

Soil extracts were pooled and concentrated prior to analysis by HPLC. Degradation products were identified by comparison of the retention times of reference standards.

Volatile radioactivity in volatile traps and radioactivity in apparatus washes was determined by LSC.

Non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics

No degradation kinetics were determined in the report. DT_{50} and DT_{90} values for the degradation of fluopicolide under irradiated conditions have been calculated from the reported data.

The total recovery of radioactivity reported for each sample was variable and the recovery in the 0 DAT samples was higher than the other samples (especially for the phenyl label, KCA 7.1.1.3/02). All fluopicolide residues were, therefore, corrected based on the total recoveries reported for each sample prior to entry in kinetic models. This study with [2,6-pyridyl-^{14}C]-fluopicolide and the soil photolysis study with [phenyl-U-14C]-fluopicolide (KCA 7.1.1.3/02) used the same (Abington) soil but were conducted two years apart. They were therefore considered to be separate incubation systems and analysed in separate sets of optimisations.

The radiation intensity in both studies was measured pre incubation and at termination for each sample, these intensity measurements were used to normalise the sampling interval of each sample to days of summer sunlight days at 30-50 °N in accordance with OECD guidance:

$$d = \frac{h \times r}{0.75 \times 12}$$

Where d = days of summer sunlight
h = hours of continuous study irradiation
r = ratio of study radiation intensity (irradiance) to that of summer sunlight
0.75 = correction for diurnal variation of natural sunlight
12 = conversion of hours to days

Draft OECD guidance for soil photolysis studies reports a mean mid-summer solar irradiance intensity of 67 W/m^2 at 30-50 °N in the UVA wavelength (300-400 nm). The radiation intensity in the studies was measured and reported in W/m^2 for the wavelength 300-800 nm. The irradiance reported for each sample was therefore factored for comparison with summer sunlight intensity (calculation of r factor) according to the following breakdown of the sunlight spectra provided by CIE (1989)[1]:

Wavelength **Proportion of total global radiation**
300-400 nm: 6.8%
400-800 nm: 55.4%
800-2450 nm: 37.8%

Kinetic model input data are summarised below.

Table 7.1.1.3- 2: Kinetic input data

Actual DAT (days)	Label	Reported W/m^2 (300-800 nm)	Calculated W/m^2 (300-400 nm)	r	Days summer sunlight @ 30-50°N	Fluopicolide (%AR corrected for total recovery)
0	Pyridyl				0.0	97.9
3	Pyridyl	479.0	32.4	0.78	6.3	92.6
5	Pyridyl	461.5	50.5	0.75	10.0	91.9
7	Pyridyl	454.5	49.7	0.74	13.8	90.9
10	Pyridyl	409.0	44.7	0.67	17.8	89.7
15	Pyridyl	419.5	45.9	0.68	27.4	90.0

[1] CIE (Commission Internationale De L´Eclairage) (1989): Solar Spectral Irradience Publication CIE, No. 85 (Technical Report)

The input data were fitted with single first order (SFO), first order multi compartment (FOMC) and dual first order in parallel (DFOP) kinetics using KinGUI v2.1 in accordance with the FOCUS (2006, 2014) guidance for deriving trigger endpoints. Confidence in the resulting parameters was assessed visually and from probability values for a t-test of the rate parameters for the SFO and DFOP models and assessment of the confidence intervals of the α and β FOMC parameters. The χ^2 error% metric was used to determine statistical goodness of fit.

Fluopicolide residues in samples incubated under dark conditions showed negligible decline and all decline in residues observed under irradiated conditions was assumed to be attributable to photolysis.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity under irradiated conditions and in the dark following application of [2,6-pyridyl-^{14}C]-fluopicolide are summarized in Table 7.1.1.3- 3 and Table 7.1.1.3- 4.

Table 7.1.1.3- 3: Photodegradation of [2,6-pyridyl-^{14}C]-fluopicolide at 20 °C in Abington soil under continuous irradiated conditions [% AR]

Compound	Incubation time (DAT)					
	0	3	5	7	10	15
Fluopicolide	98.44	90.87	87.97	87.33	85.98	86.68
M-02 (AE C657188)	nd	1.84	2.23	2.35	2.64	2.6
AE C648995	nd	nd	nd	nd	nd	0.29
Unidentified 1	nd	nd	nd	nd	nd	0.22
Soil Extracts	98.44	92.71	90.19	89.68	88.62	89.80
Apparatus Wash	na	0.02	0.05	0.01	0.02	0.02
Non-extractable radioactivity	2.12	5.30	5.38	6.17	6.83	6.12
^{14}C-Carbon dioxide	na	0.19	0.12	0.21	0.32	0.35
Organic volatiles	na	0.01	0.02	0.02	0.04	0.03
Total radioactivity	100.56	98.14	95.76	96.09	95.83	96.32

na: not analysed, nd: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel

Table 7.1.1.3- 4: Degradation of [2,6-pyridyl-^{14}C]-fluopicolide at 20 °C in Abington soil under non-irradiated conditions in the dark [% AR]

Compound	Incubation time (DAT)				
	3	5	7	10	15
Fluopicolide	96.65	92.99	91.65	94.35	92.95
M-02 (AE C657188)	nd	nd	nd	nd	nd
AE C648995	nd	nd	nd	nd	nd
Unidentified 1	nd	nd	nd	nd	nd
Soil Extracts	96.65	92.99	91.65	94.35	92.95
Apparatus Wash	0.08	0.14	0.09	0.05	0.06
Non-extractable radioactivity	3.18	2.46	3.22	2.17	2.82
^{14}C-Carbon dioxide	nd	nd	nd	nd	0.02
Organic volatiles	nd	nd	nd	nd	0.01
Total radioactivity	99.90	95.59	94.97	96.58	95.86

nd: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel

B. Material Balance

Material balances ranged from 95.8 to 100.6% AR for irradiated samples treated with [2,6-pyridyl-^{14}C]-fluopicolide (mean 97.1% AR) and from 95.0 to 99.9% AR for samples incubated in the dark (mean 96.6% AR).

C. Extractable and Non-Extractable Residues

The majority of the applied radioactivity was extractable throughout the study. For irradiated samples treated with [2,6-pyridyl-^{14}C]-fluopicolide, total extractable radioactivity ranged from 98.4% AR at DAT 0 to 88.6% AR at DAT 10. At study termination (DAT 15) 89.8% AR was recovered in soil extracts. The total of non-extractable residues (NER) was low throughout the study, increasing from 2.1% AR at DAT 0 to 6.8% AR by DAT 10, and then decreasing slightly to 6.1% AR by the end of the study (DAT 15).

For samples incubated in the dark with [2,6-pyridyl-^{14}C]-fluopicolide, total extractable radioactivity ranged from 96.7% AR at DAT 3 to 91.7% AR at DAT 7. At study termination (DAT 15) 93.0% AR was recovered in the soil extracts. The total of non-extractable residues (NER) was low ranging from 2.2% to 3.2% AR throughout the study.

D. Volatile Radioactivity

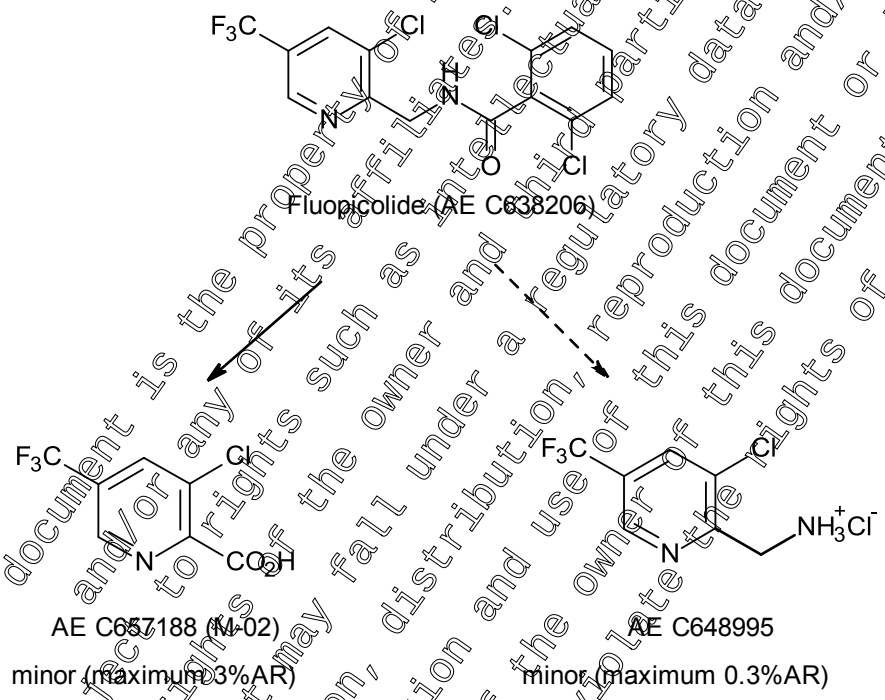
Radiolabelled carbon dioxide evolved accounted for <1% of the applied radioactivity in both the irradiated and non-irradiated samples. Only trace amounts of radioactivity were recovered in the volatile traps containing ethanediol (maximum of 0.04% of applied) confirming there was no volatilization of fluopicolide or its metabolites.

E. Degradation of Parent Compound

Fluopicolide was the principal radiolabelled component detected. Levels of parent accounted for all extracted radioactivity in Day 0 soil extracts and in non-irradiated samples throughout both the preliminary and main studies. Levels of parent declined over the irradiation period of the main study to 87% of applied radioactivity after 15 days. The degradation product M-02 (called AE C657188 in the report) accounted for 3% at the end of the irradiation period. Two other minor metabolites, AE C648995 and an unidentified metabolite were detected at the final sampling point. Each accounted for <0.3% of applied radioactivity.

The proposed photolytic route of degradation of [2,6-pyridyl-^{14}C]-fluopicolide in soil is presented in Figure 7.1.1.3- 1.

Figure 7.1.1.3- 1: Photolytic route of degradation of [2,6-pyridyl-^{14}C]-fluopicolide in soil



F. Degradation Kinetics

The presence of light slightly enhanced the degradation rate of fluopicolide on soil surfaces. DT_{50} values were not reported. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). A summary of the fit statistics in the standard EFSA kinetics template are shown below.

The best fit DT_{50} value calculated for fluopicolide was 235 days summer sunlight at 30-50 °N.

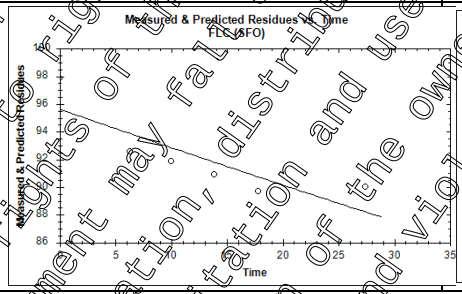
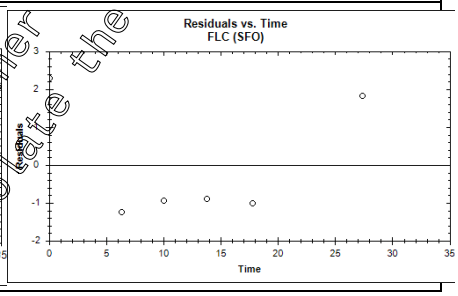
Table 7.1.1.3- 5: Degradation rate of [2,6-pyridyl-^{14}C]-fluopicolide under irradiated conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Kinetic model	M_0	Parameter [k, k1, k2, g, tb, a, β]	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
SFO	**95.6**	**k=0.002952**	**1.26**	**0.0163**	**0.001149**	**0.005**	**235**	**780**
FOMC	97.9	α=0.023527 β=0.631907	0.3545	n.r. n.r.	0.010801 -0.547341	0.036 1.811	>1000	>1000
DFOP	97.9	k1=0.1501 k2=2.418E-14 g=0.0838	0.3413	0.1315 0.5 n.r.	-0.04065 -0.003132 3.213E-03	0.341 0.003 0.164	>1000	>1000
Decision summary (persistence endpoints): FOMC and DFOP both provided a slight decrease in χ^2-error. However, parameter confidence was poor for both biphasic models and there were too few samples to ascertain a definitive biphasic behaviour. The SFO model provided an acceptable fit and was selected for trigger endpoints								

n.r. not relevant
Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.1.3- 6: Graphical representation of final kinetic fit of [2,6-pyridyl-^{14}C]-fluopicolide under irradiated conditions at 20 °C

Soil Model Reference	Modelled vs observed	Residuals
Abington – pyridyl label SFO Keirs D.C., Lowrie C. 2001a		

III. Conclusion

The degradation rate of [2,6-pyridyl-^{14}C]-fluopicolide was slightly enhanced in the presence of light leading to the formation of M-02 (AE C657188) and two other very minor photodegradation products. No degradation of fluopicolide occurred during incubation in the dark.

Assessment and conclusion by applicant:

The study was conducted in accordance with SETAC 1.1 (1995) and USEPA (= EPA) N, 161-3 (1982). The study is considered valid to assess the photolytic degradation of [2,6-pyridyl-^{14}C]-fluopicolide in soil.

Data Point:	KCA 7.1.1.3/02
Report Author:	[redacted]
Report Year:	2009
Report Title:	[14C]-AE C638206: soil photolysis - Report amendment 3
Report No:	17299
Document No:	M-201033-03-1
Guideline(s) followed in study:	EU (=EEC): 91/414; SETAC: March 1995
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The original soil photolysis report M-201033-01-1, which was evaluated and accepted in the DAR (2005), is included in the Baseline dossier. This report has subsequently been amended twice. A report amendment (M-201033-02-1) added the initials 'OECD' to the GLP statement and has not been included in either the Baseline Dossier or this MCA 7 Dossier because it has been superseded by a later report amendment (KCA 7.1.1.3/02, M-201033-03-1) which is summarised below. In the later report amendment, part of Appendix 7 has been removed. In the original report Appendix 7 contained an extrapolation of natural sunlight measured outdoors with a Radiolux sensor to justify the light intensity used in the Heraeus suntest unit. However the light intensity value measured outdoors was unrealistically high as the sensor was not appropriate for measurements of natural sunlight outdoors. It should be noted that this had no effect on the calculation of the light intensity in the suntest experiment, which was measured by the Radiolux sensor under the correct conditions. The only impact of the outdoor measurements on the study is that the soil samples will have been irradiated at higher levels or for a longer period than was strictly necessary. To avoid confusion the report was amended by removing the extrapolation calculation from Appendix 7.

Executive Summary

The photolytic degradation of fluopicolide on soil surfaces was investigated in air-dried Abington sandy loam soil, prepared as a thin-layer on glass incubation vessels with a thickness of about 3 mm. The test item, [phenyl-U-^{14}C]- fluopicolide, dissolved in acetonitrile was applied evenly to the soil surface at an application rate equivalent to 1.23 mg/kg. Treated soil samples were exposed to artificial irradiation from a Xenon lamp (with < 290 nm cut-off filter) with continuous irradiation for a period of 15 days at 20 ± 3 °C. A set of dark control samples treated at the same application rate were incubated in the dark at 20 ± 3 °C.

The mean recoveries of radioactivity were 97.5 % and 99.0 % AR for the irradiated and dark controls, respectively.

Fluopicolide was the principal radiolabelled component detected. In the irradiated samples, fluopicolide slowly degraded to 72% AR after 15 days. Mineralization to carbon dioxide was low at <3 % AR. The degradation rate of fluopicolide was slightly enhanced in the presence of light leading to the formation of M-01 (AE C653711) at 8.6% AR. No degradation of fluopicolide occurred during incubation in the dark.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014). A summary of the results is shown below. The best fit DT_{50} value calculated for fluopicolide was 158 days summer sunlight at 30-50 °N.

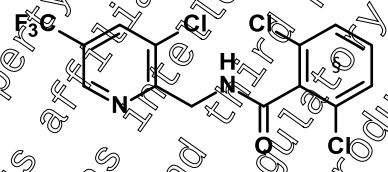
Soil	Best fit model	χ^2 – error [%]	DT_{50} [days summer sunlight @ 30-50 °N]	DT_{90} [days summer sunlight @ 30-50 °N]
Abington – phenyl label	DFOP	0.70	158	552

I. Materials and Methods

A. Materials

1. Test Items

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



* Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.33 MBq/mg (144 µCi/mg)

Radiochemical Purity: >98%

2. Test Soil

The study was performed using one test soil as characterized in Table 7.1.1.3- 7.

Table 7.1.1.3- 7: Physico-chemical properties of test soil

Parameter	Soil
Soil Designation	Abington
Geographic Location	
City	Abington
Country	England, UK
Textural Classification (USDA)	sandy loam
Sand [50 - 2000 µm] (%)	61.64
Silt [2 – 50 µm] (%)	20.81
Clay [< 2 µm] (%)	17.55
pH	
in H_2O (1:1)	8.0
in $CaCl_2$ (1:1)	7.3
Organic Carbon (%)	2.4
Cation Exchange Capacity (meq/100 g)	21.0

*USDA textural classification data taken from Appendix 5 (Soil Batch 99/01)

B. Study Design

1. Experimental Conditions

Portions of fresh soil (5 g) were added to borosilicate glass incubation vessels, fitted with quartz glass covers. Water was added to form a slurry and the soil air dried at ambient temperature to give a soil depth of *ca*. 3mm. The soil samples were incubated inside glass units with moistened carbon dioxide free air drawn over the soil surface. A series of three traps for volatile products was connected to each unit. The first trap was empty (to prevent flow back), the second contained ethanediol to trap volatile organic components and the third contained ethanolamine to trap liberated $^{14}CO_2$.

A preliminary investigation was conducted over three days to determine an approximate rate of photodegradation of fluopicolide.

The test item [phenyl-U-^{14}C]-fluopicolide (56.5 µg), dissolved in acetonitrile (100 µL), was applied evenly to the soil surface. Units were either irradiated continuously with light from an Heraeus Suntest xenon lamp or incubated in the dark (non-irradiated samples). The application rate was equivalent to 11.3 mg/kg. The temperature of both irradiated and non-irradiated samples was maintained at a temperature of 20 ± 3 ºC throughout the incubation period. The photolytic degradation of fluopicolide was studied with continuous illumination under artificial sunlight for a period of up to 15 days, equivalent to 30 days of natural summer sunlight. The artificial sunlight was provided by a xenon arc lamp with filters to cut off any radiation below 290nm.

2. Sampling

In the preliminary investigation a single replicate was removed at time zero, 1, 2 and 3 days. A non-irradiated sample was maintained for 3 days.

In the definitive study duplicate soil samples were taken from both irradiated and non-irradiated systems after 0, 3, 5, 7, 10 and 15 days. One replicate was taken for analysis and the other was stored without further analysis. The replicate samples were planned for structural analysis of potential photo-degradation products which were not formed.

3. Analytical Procedures

Soil samples were extracted at ambient temperature initially with acetonitrile, followed by acetonitrile / water (4/1, v/v). After removal of the soil samples from the incubation flasks, the flasks were soaked in acetone to remove any residual activity. Radioactivity extracted from soil was quantified by liquid scintillation counting (LSC).

Soil extracts were pooled and concentrated prior to analysis by HPLC. Degradation products were identified by comparison of the retention times of reference standards.

Volatile radioactivity in volatile traps and radioactivity in apparatus washes was determined by LSC.

Non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics

No degradation kinetics were determined in the report. DT_{50} and DT_{90} values for the degradation of fluopicolide under irradiated conditions have been calculated from the reported data.

The total recovery of radioactivity reported for each sample was variable and the recovery in the 0 DAT samples was higher than the other samples. All fluopicolide residues were, therefore, corrected based on the total recoveries reported for each sample prior to entry in kinetic models. This study with [phenyl-U-14C]-fluopicolide and the soil photolysis study with [2,6-pyridyl-^{14}C]-fluopicolide (KCA 7.1.1.3/01) used the same (Abington) soil but were conducted two years apart. They were therefore considered to be separate incubation systems and analysed in separate sets of optimisations.

The radiation intensity in both studies was measured pre incubation and at termination for each sample, these intensity measurements were used to normalise the sampling interval of each sample to days of summer sunlight days at 30-50 °N in accordance with OECD guidance.

$$d = \frac{h \times r}{0.75 \times 12}$$

Where d = days of summer sunlight
h = hours of continuous study irradiation
r = ratio of study radiation intensity (irradiance) to that of summer sunlight
0.75 = correction for diurnal variation of natural sunlight
12 = conversion of hours to days

Draft OECD guidance for soil photolysis studies reports a mean mid-summer solar irradiance intensity of 67 W/m^2 at 30-50 °N in the UVA wavelength (300-400 nm). The radiation intensity in the studies was measured and reported in W/m^2 for the wavelength 300-800 nm. The irradiance reported for each sample was therefore factored for comparison with summer sunlight intensity (calculation of r factor) according to the following breakdown of the sunlight spectra provided by CIE (1989)[2].

Wavelength	**Proportion of total global radiation**
300-400 nm:	6.8%
400-800 nm:	55.4%
800-2450 nm:	37.8%

Kinetic model input data are summarised below

Table 7.1.1.3- 8: Kinetic input data

Actual DAT (days)	Label	Reported W/m^2 (300-800 nm)	Calculated W/m^2 (300-400 nm)	r	Days summer sunlight @ 30-50°N	Fluopicolide (%AR corrected for total recovery)
0	Phenyl				0.0	93.5
3	Phenyl	418.0	45.9	0.68	5.5	86.4
5	Phenyl	423.0	46.2	0.69	9.2	86.7
7	Phenyl	470.0	51.4	0.77	14.3	83.7
10	Phenyl	480.0	52.5	0.78	20.9	80.9
15	Phenyl	470.0	50.4	0.77	30.7	78.8

The input data were fitted with single first order (SFO), first order multi compartment (FOMC) and dual first order in parallel (DFOP) kinetics using KinGUI v2.1 in accordance with the FOCUS (2006, 2014) guidance for deriving trigger endpoints. Confidence in the resulting parameters was assessed visually and from probability values for a t-test of the rate parameters for the SFO and DFOP models and assessment of the confidence intervals of the α and β FOMC parameters. The χ^2 error% metric was used to determine statistical goodness of fit.

Fluopicolide residues in samples incubated under dark conditions showed negligible decline and all decline in residues observed under irradiated conditions was assumed to be attributable to photolysis.

[2] CIE (Commission Internationale De L´Eclairage) (1989): Solar Spectral Irradience Publication CIE, No. 85 (Technical Report)

II. Results and Discussion

The distribution and characterisation of radioactivity under irradiated conditions and in the dark following application of [phenyl-U-^{14}C]- fluopicolide are summarized in Table 7.1.1.3- 9 and Table 7.1.1.3- 10.

Table 7.1.1.3- 9: Photodegradation of [phenyl-U-^{14}C]- fluopicolide at 20 °C in Abington soil under continuous irradiated conditions [% AR]

Compound	Incubation time (DAT)					
	0	3	5	7	10	15
Fluopicolide	102.10	81.35	79.51	82.84	80.92	71.60
M-01 (AE C653711) [A]	nd	4.20	4.55	5.95	6.94	8.58
Soil Extracts	102.10	85.55	84.06	88.79	87.86	80.18
Apparatus Wash	nd	0.85	0.76	0.19	0.24	0.44
Non-extractable radioactivity	7.13	7.58	6.36 [B]	7.09	8.01	7.24
^{14}C-Carbon dioxide	na	0.16	0.55	2.90	3.72	2.93
Organic volatiles	na	nd	nd	0.02	0.18	0.02
Total radioactivity	109.20	94.14	91.73	98.99	100.00	90.81

na: not analysed, nd: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] This metabolite was incorrectly identified as AE C643805 in the report but was subsequently correctly identified as M-01 (AE C653711) in Document KCA 7.1.3/03, M-201038-03-1
[B] Residue was further extracted using acetonitrile - water (1: 1, v/v) and released 2.94 % AR

Table 7.1.1.3- 10: Degradation of [phenyl-U-^{14}C]- fluopicolide at 20 °C in Abington soil under non-irradiated conditions in the dark [% AR]

Compound	Incubation time (DAT)				
	3	5	7	10	15
Fluopicolide	91.61	89.61	94.61	96.96	95.13
M-01 (AE C653711) [A]	nd	nd	nd	nd	nd
Soil Extracts	91.61	89.61	94.61	96.96	95.13
Apparatus Wash	0.20	2.70	1.59	0.93	0.65
Non-extractable radioactivity	4.26	4.23	3.87	4.17	4.38
^{14}C-Carbon dioxide	nd	nd	nd	nd	nd
Organic volatiles	nd	nd	nd	nd	nd
Total radioactivity	96.07	96.54	100.10	102.10	100.20

nd: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] This metabolite was incorrectly identified as AE C643805 in the report but was subsequently correctly identified as M-01 (AE C653711) in Document KCA 7.1.3/03, M-201038-03-1.

B. Material Balance

Material balances ranged from 90.8 to 109.2% AR for irradiated samples treated with [phenyl-U-^{14}C]-fluopicolide (mean 97.5% AR) and from 96.1 to 102.1% AR for samples incubated in the dark (mean 99.0% AR).

C. Extractable and Non-Extractable Residues

The majority of the applied radioactivity was extractable throughout the study. For irradiated samples treated with [phenyl-U-^{14}C]- fluopicolide, total extractable radioactivity ranged from 102.1% AR at DAT 0 to 80.2% AR at DAT 15 at study termination. Non-extractable residues (NER) were low reaching a maximum of 8.0% AR (DAT 10) in the irradiated samples.

For samples incubated in the dark with [phenyl-U-^{14}C]-fluopicolide, total extractable radioactivity ranged from 89.6% AR to 97.0% AR throughout the incubation period. At study termination (DAT 15) 95.1% AR was recovered in the soil extracts. The total of non-extractable residues (NER) was low ranging from 3.9% to 4.4% AR throughout the study.

D. Volatile Radioactivity

Radiolabelled carbon dioxide evolved accounted for < 4% of the applied radioactivity in the irradiated samples. Only trace amounts of radioactivity were recovered in the volatile traps containing ethanediol (maximum of 0.2% AR) confirming there was no volatilization of fluopicolide or its metabolites. Neither carbon dioxide nor volatile organic compounds were detected in the non-irradiated experiment.

E. Degradation of Parent Compound

Fluopicolide was the principal radiolabelled component detected. Levels of parent accounted for all extracted radioactivity in Day 0 soil extracts and in non-irradiated samples throughout both the preliminary and main studies. Levels of parent declined over the irradiation period of the main study to 72% of applied radioactivity after 15 days. A degradation product accounted for 9% AR at the end of the irradiation period. This metabolite was incorrectly identified as AE C643805 in the report but was subsequently correctly identified as M-01 (AE C633711).

F. Degradation Kinetics

The presence of light slightly enhanced the degradation rate of fluopicolide on soil surfaces. DT_{50} values were not reported. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). A summary of the fit statistics in the standard EFSA kinetics template are shown below.

The best fit DT_{50} value calculated for fluopicolide was 158 days summer sunlight at 30-50 °N.

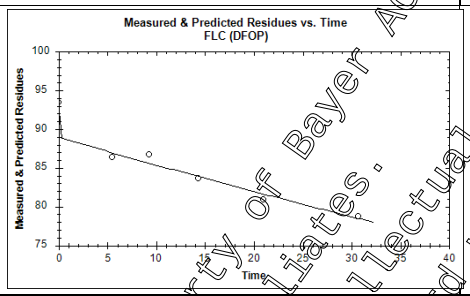
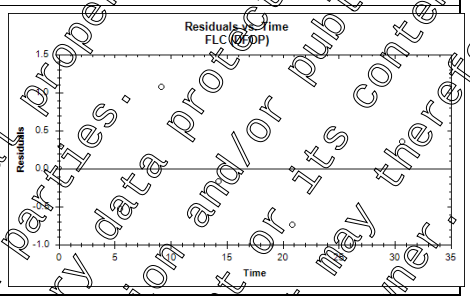
Table 7.1.1.3- 11: Degradation rate of [phenyl-U-^{14}C]- fluopicolide under irradiated conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Kinetic model	M_0	Parameter [k, k1, k2, g, tb, a, β]	χ^2 %-error	Prob > t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
SFO	91.	k=0.005308	1.376	0.00186	0.003594	0.007	131	434
FOMC	93.3	α=0.08727 β=5.31867	0.7893	n.r. n.r.	0.02787 -2.35883	0.147 12.996	>1000	>1000
DFOP	**93.5**	**k1=582.4 k2=0.004077 g=0.0492**	**0.7018**	**<2e-16 0.0116 n.r.**	**582.4 0.002839 1.984E-02**	**582.381 0.005 0.079**	**158**	**552**
Decision summary (persistence endpoints): FOMC provided an improved fit over SFO. DFOP provided the best fit and both k1 and k2 parameter estimates were statistically robust. DFOP selected as best fit for trigger endpoints.								

n.r. not relevant
Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.1.3- 12: Graphical representation of final kinetic fit of [phenyl-U-^{14}C]- fluopicolide fluopicolide under irradiated conditions at 20 °C

Soil Model Reference	Modelled vs observed	Residuals
Abington – phenyl label DFOP Mackie J.A. 1999a		

III. Conclusion

The degradation rate of [phenyl-U-^{14}C]-fluopicolide was slightly enhanced in the presence of light leading to the formation of M-01 (AE C653711). No unique photodegradation products were detected. No degradation of fluopicolide occurred during incubation in the dark.

Assessment and conclusion by applicant:

The study was conducted in accordance with SETAC 1.1 (1995) and USEPA (= EPA) N, 161-3 (1982). The study is considered valid to assess the photolytic degradation of [phenyl-U-^{14}C]-fluopicolide in soil.

Data Point:	KCA 7.1.1.3/03
Report Author:	[redacted]
Report Year:	2005
Report Title:	(Benzene Ring-U-14C)-AE C638206: Re-analysis of soil photolysis extracts generated from Inveresk project no. 394309 (Agredoc reference number C011545)
Report No:	18816
Document No:	M-201038-03-1
Guideline(s) followed in study:	EU (=EEC): 91/414; SETAC: March 1995; EPA Pesticide Assessment Guidelines, Subdivision N, Paragraph 161-3 (Oktober 1982)
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The original report M-201038-01-1, which was evaluated and accepted in the DAR (2005), is included in the Baseline dossier. This report has subsequently been amended twice. The first report amendment (M-201038-02-1) corrected some typographical errors and has not been included in either the Baseline Dossier or this MCA 7 Dossier because it has been superseded by a second report amendment (KCA 7.1.1.3/03, M-201038-03-1) which is summarised below. The second report amendment (M-201038-03-1) corrected some typographical errors and added the initials 'OECD' to the GLP statement.

Executive Summary

A degradation product of [phenyl-U-^{14}C]-fluopicolide was incorrectly identified as AE C643805 in the report KCA 7.1.1.3/02, [redacted] 2005; M-201038-03-1. Soil extracts from the photolysis study were re-examined by HPLC and LC/MS and the degradation product was shown to be M-01 (AE C653711).

I. Materials and Methods

This study was conducted as a supplementary study to that summarised previously [KCA 7.1.1.3/02, [redacted] 2005; M-201038-03-1]. The objective was to re-analyse selected soil photolysis extracts generated in Report M-201038-03-1 to confirm the identity of the degradation product detected.

1. Experimental Conditions

Not applicable.

2. Sampling

Soil extracts from DAT 10 and DAT 15 irradiated samples were re-analysed.

3. Analytical Procedures

Analytical standards of fluopicolide, AE C643805 and M-01 (AE C653711) were used.

HPLC analysis of the soil extracts in the original report characterised the degradation product as AE C643805 based on its retention time in the HPLC system used in that study. The retention times of certified standards of AE C643805 and M-01 (AE C653711) were very close in this HPLC system. A new HPLC system was developed to separate these compounds. The soil extract from the irradiated DAT 10 sample was re analysed using the new HPLC method. The soil extract from the irradiated DAT 15 sample was analysed by LC-MS.

4. Determination of degradation kinetics

Not applicable.

II. Results and Discussion

Analysis of DAT 10 irradiated soil extract in an HPLC method which separated the compounds fluopicolide, AE C643805 and M-01 (AE C653711) confirmed the presence of radiolabelled peaks which co-chromatographed with fluopicolide and M-01. Fluopicolide and M-01 accounted for 80% and 7% of the applied radioactivity respectively. These values are similar to those found in the original study and provide confirmation of the stability of the residue during storage. No radiolabelled peak with a retention time corresponding to AE C643805 was observed.

Analysis of DAT 15 irradiated soil extract by LC-MS confirmed the presence of fluopicolide as the main component and M-01 (AE C653711). AE C643805 was not present at detectable levels.

III. Conclusion

The degradation product detected during the soil photolysis study conducted with [phenyl-U-^{14}C]-fluopicolide [KCA 7.1.1.3/02, M-201038-03-1] was identified as M-01 (AE C653711).

Assessment and conclusion by applicant:

The study was conducted in accordance with SETAC 1.1 (1995) and USEPA (= EPA) N, 161-3 (1982). The study is considered valid to assess the photolytic degradation of [phenyl-U-^{14}C]-fluopicolide in soil.

Data Point:	KCA 7.1.1.3/04
Report Author:	[redacted]
Report Year:	2009
Report Title:	Statement (Version 3) - The light intensity measured during the studies on the phototransformation of fluopicolide on soil surfaces and the transfer of experimental results to environmental phototransformation half-lives
Report No:	MEF-08/185
Document No:	M-300764-03-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted Addendum 2 to the DAR (2008)
GLP/Officially recognised testing facilities:	not applicable
Acceptability/Reliability:	Yes

The original statement M-300764-02-1 which was evaluated and accepted in the Addendum 2 to the DAR (2008), is included in the Baseline dossier. This statement was subsequently amended in response to a data requirement from the PRAPeR Expert Meeting 62 (January 2009). In the final version of the statement (KCA 7.1.1.3/04, M-300764-03-1), soil photolysis DT_{50} values derived using data from KCA 7.1.1.3/01, M-201037-03-1 and KCA 7.1.1.3/02, M-201038-03-1 in samples irradiated continuously with light from an Heraeus Suntest xenon lamp were used to provide environmental photo-transformation DT_{50} values at different locations in Europe as requested by the PRAPeR Expert Meetings. However, M-300764-03-1 has been superseded as the experimental DT_{50} values (reported in the original dosser and DAR, 2005) were derived prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. Consequently M-300764-03-1 is not summarised in this dossier. However, for procedural reasons the position paper is included in the dossier.

Data Point:	KCA 7.1.1.3/05
Report Author:	[redacted]
Report Year:	2007
Report Title:	Fluopicolide - Relevance of photolysis in soil degradation studies
Report No:	MEF-06/495
Document No:	M-286182-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted Addendum 1 to the DAR (2007)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The purpose of statement KCA 7.1.1.3/05, M-286182-01-1 was to assess the impact of photodegradation on the soil degradation rate of fluopicolide in field dissipation studies. The statement is summarised in Addendum 1 to the DAR (2007) and was discussed, but not accepted, at the PRAPeR report of meeting 37 (December 2007) on fluopicolide. The document was superseded by the later statement addressing the photolytic half-life calculations for latitudes 40° N and 45° N prepared in response to the requests from the PRAPeR report of meeting 62 (January 2009) on fluopicolide (KCA 7.1.1.3/04, M-300764-03-1). Consequently M-286182-01-1 is not summarised in this dossier. However, for procedural reasons the position paper is included in the dossier.

CA 7.1.2 Rate of degradation in soil

On overall summary of the trigger endpoint DT_{50} values and modelling endpoint $DegT_{50}$ values derived for fluopicolide and its metabolite in laboratory studies is summarised below.

Summary of laboratory aerobic soil DT_{50} values for fluopicolide and its metabolites

Compound	Trigger endpoints DT_{50} range (un-normalised) (d)	No. datasets	No. soils	Modelling endpoints Geometric Mean $DegT_{50}$ normalised to 20°C & pF2 (d)	Arithmetic Mean Molar Fraction
Fluopicolide	47.7 - 1290	22	16	181.6	-
M-01 (AE C653711)	135.9 - 3461	26	18	569.5	0.80 (from parent)
M-02 (AE C657188)	0.7 – 4.4	7	7	1.6	NA [C]
M-03 (AE 0608000)	0.1 – 62.6	9	7	17.9 [A] / 0.19 [B]	0.53 [A] / - [B] (from parent)
M-05 (AE 1344122)	5.6 – 172.1	13	7	25.2	0.153 (from M-02)
M-10 (AE 1344123)	3.6 – 1000	13	7	35.4	0.129 (from M-02)
M-11/M-12	31.7 – 242.5	2	2	87.6	0.044 (from M-02)
M-13	13.3 – 48.4	3	3	20.7	0.049 (from M-02)
M-14 (AE 1388273)	4.9 – 21.7	5	3	9.4	1 (from M-20)
M-15 (AE 1413903)	102.7 – 113.2	4	4	144.8	NA [C]
M-20 (BCS-BX16566)	2.0 - 144.7	6	6	6.1	0.021 (from M-02) 0.559 (from M-05)

[A] Geometric mean for soils with pH < 6
[B] Geometric mean for soils with pH ≥ 6
[C] Not applicable as degradation rates for M-02 and M-15 were derived from metabolite dosed studies. For M-02 the overall formation fraction from fluopicolide was set to 1.0 as a conservative assumption. For M-15 a molar formation fraction of 0.0016 from fluopicolide was estimated by inverse modelling of a lysimeter study (KCA 7.1.3.2/08, M-687165-01-1)

It should be noted M-05, M-10, M-11, M-12, M-13, M-14, M-15 and M-20 are minor soil metabolites, detected in aerobic soil metabolism studies conducted with [2,6-pyridyl-^{14}C]-M-02 or in leachate from a lysimeter study conducted with fluopicolide. While fluopicolide and M-01 degraded slowly in laboratory studies other metabolites showed moderate to rapid rates of degradation, except for M-15 which was a very minor soil metabolite not detected in parent soil degradation studies. Experiments have been conducted to investigate the behaviour of fluopicolide and its metabolite M-01 under field conditions.

Summary of field DT_{50} values for fluopicolide and its metabolites

Compound	Trigger endpoints DT_{50} range (un-normalised) (d)	SFO DT_{50} range (un-normalised) (d) [B]	No. sites	Modelling endpoints Geometric Mean $DegT_{50}$ (normalised to 20°C & pF2) (d)
Fluopicolide	28 [A] - 403	177.4 – 457.6	12	183
M-01 (AE C653711)	133 - 344	155 - 344	5	146 [C]

[A] Lowest value from cropped soil site, not considered for SFO DT_{50} range (un-normalised)
[B] Worst-case SFO DT_{50} (un-normalised) used for PEC_{soil} calculation
[C] Geometric mean field $DegT_{50}$ value (normalised to 20°C & pF2) used for FOCUS modelling

Fluopicolide was found to have a similar rate of degradation in the field, with $DegT_{50}$ values similar to those observed under laboratory conditions. An overall geometric mean $DegT_{50}$ value of **182 days** in soil was derived for fluopicolide for use in FOCUS modelling calculations, including both laboratory and field data.

CA 7.1.2.1 Laboratory studies

CA 7.1.2.1.1 Aerobic degradation of the active substance

The aerobic degradation of fluopicolide in soil has been investigated in a total of six studies at 20°C, one study at 25°C and one study at 10°C.

Five studies were evaluated during the previous EU review and are still considered as reliable to assess the behaviour of fluopicolide in soil (KCA 7.1.2.1.1/01, KCA 7.1.2.1.1/02, KCA 7.1.2.1.1/03, KCA 7.1.2.1.1/04 and KCA 7.1.2.1.1/05). For procedural reasons two of the previously submitted studies also have to be included under Point KCA 7.1.2.1.1 in the current dossier (KCA 7.1.2.1.1/04 and KCA 7.1.2.1.1/05) but the summaries are provided in full only in Point KCA 7.1.1.1. A statement about the levels of an unidentified minor metabolite (called Metabolite C in the study) is provided in document KCA 7.1.2.1.1/06.

In addition, studies KCA 7.1.2.1.1/07, KCA 7.1.2.1.1/08 and KCA 7.1.2.1.1/09, time dependent sorption studies conducted with [phenyl-U-^{14}C]-labelled fluopicolide, are provided as new data not yet reviewed.

Finally, a report describing the kinetic evaluation of the degradation behaviour of fluopicolide and its metabolites in soil under aerobic laboratory conditions is provided (KCA 7.1.2.1.1/10).

Report reference	Author, Year	Phenyl Label	Pyridyl Label	Comment
KCA 7.1.2.1.1/01 M-241052-01-1	[redacted] 2003	✓	✓	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.2.1.1/02 M-241051-01-1	[redacted] 2003	✓	✓	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.2.1.1/03 M-241053-01-1	[redacted] 2003	✓	✓	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.2.1.1/04 M-241049-01-1	[redacted] 2003	✓	✓	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable. Summary provided under KCA 7.1.1.1/01. Additional summary provided under KCA 7.1.3.2/02
KCA 7.1.2.1.1/05 M-201230-02-1	[redacted] 2003	✓	✗	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable. Summary provided under KCA 7.1.1.1/02.
KCA 7.1.2.1.1/06 M-685745-01-1	[redacted] 2020	-	-	New data not yet reviewed.
KCA 7.1.2.1.1/07 M-555570-01-1	[redacted] 016	✓	✗	New data not yet reviewed. Additional summary provided under KCA 7.1.3.2/03
KCA 7.1.2.1.1/08 M-550687-01-1	[redacted] 2016	✓	✗	New data not yet reviewed. Additional summary provided under KCA 7.1.3.2/04
KCA 7.1.2.1.1/09 M-655056-01-1	[redacted] 2019	✓	✗	New data not yet reviewed. Additional summary provided under KCA 7.1.3.2/05
KCA 7.1.2.1.1/10 M-685680-01-1	[redacted] 2020	-	-	New data not yet reviewed.

Data Point:	KCA 7.1.2.1.1/01
Report Author:	[redacted]
Report Year:	2003
Report Title:	The route and rate of degradation of [2,6-14C-pyridinyl] and [U-14C-benzoyl]-AE C638206 in two soils under laboratory aerobic conditions at 25 degrees C
Report No:	B004074
Document No:	M-241052-01-1
Guideline(s) followed in study:	USEPA (=EPA): Sec. N 162-1
Deviations from current test guideline:	Yes. According to OECD 307 soil laboratory studies should not normally exceed 120 days. The soils were incubated under aerobic conditions for 369 days as required by US EPA guidelines at the time the study was conducted. Final biomass samples are low compared to initial measurements.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of fluopicolide was investigated in two soils from the USA under laboratory aerobic conditions for up to 369 days. [Phenyl-U-^{14}C]-labelled fluopicolide or [2,6-pyridyl-^{14}C] labelled fluopicolide was applied to soil samples at an application rate equivalent to 400 g /ha. Lamberton soil was classified as a sandy clay loam and Pikeville soil as a loamy sand according to USDA classification. Soil samples were incubated in the dark, at a moisture content equivalent to 75% of ⅓ bar under aerobic conditions at 25 °C. The radiochemical purity was > 99 % for both radiolabelled test items. The specific activities were 5.33 and 5.88 MBq/mg for [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C] labelled fluopicolide, respectively.

Samples were taken for extraction and analysis immediately after treatment (day 0) and after 14, 31, 60, 94, 116, 188, 273, and 369 days of incubation. Soil samples were exhaustively extracted with up to four successive extractions with acetonitrile / water (4 / 1, v/v) at ambient temperature followed by a Soxhlet extraction using acetonitrile. Concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC). Results from HPLC characterization were confirmed by mass spectrometry.

Mass balances in the Lamberton soil ranged from 86.3% to 97.6% of the applied radioactivity (mean 92.5% AR) for samples treated with [phenyl-U-^{14}C]-fluopicolide and 84.8% to 96.1% AR (mean 89.8% AR) for samples treated with [2,6-pyridyl-^{14}C]-fluopicolide. Mass balances in the Pikeville soil ranged from 83.3% to 98.3% AR (mean 94.5% AR) for samples treated with [phenyl-U-^{14}C]-fluopicolide and 78.5% to 98.0% AR (mean 91.7% AR) for samples treated with [2,6-pyridyl-^{14}C]-fluopicolide. The low recoveries in mass balance could not be attributed to a single factor such as dosing errors, extraction losses, volatile losses or adsorption to laboratory equipment.

The majority of the radioactivity was extractable in both soils. Radioactivity in soil extracts from Lamberton soil decreased from 94.4 and 84.8% AR at DAT 0 to 84.1 and 62.1% AR at DAT 369 in the phenyl and pyridyl labelled samples, respectively. Radioactivity in soil extracts from Pikeville soil decreased from 98.1 and 97.8% AR at DAT 0 to 70.3 and 64.3% AR at DAT 369 in the phenyl and pyridyl labelled samples, respectively.

Non-extractable soil residues increased concurrently with the decrease in extractable radioactivity in both soils. In Lamberton soil at DAT 369, non-extractable residues reached a maximum of 23% in the pyridyl treatment, compared to 5% AR in the phenyl treatment at the same timepoint suggesting the formation of soil residues from the pyridine ring. Similar differences were observed in Pikeville soil where non-extractable residues reached a maximum of 19% AR in the pyridyl treatment (DAT 188) compared to 11% AR in the phenyl treatment at the same timepoint.

Mineralization to carbon dioxide was a minor pathway, demonstrated by the low amount of radioactivity recovered in the ethanolamine volatile traps for both labels (maximum of 1.6% of applied). No significant levels of organic volatiles were observed.

After 369 days incubation at 25 °C in Lamberton soil, fluopicolide degraded to 40.4% of the radioactivity applied in the phenyl label and 45.3% AR in the pyridyl label. In Pikeville soil over the same time period, fluopicolide degraded to 49.3% of the radioactivity applied in the phenyl label and 53.5% AR in the pyridyl label. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit un-normalised DT_{50} values of 358 days in Lamberton soil and 424.9 days in Pikeville soil.

The primary metabolic pathway for fluopicolide in soil was proposed to be the formation of an oxidative addition product M-03 (AE 0608000), followed by cleavage to form M-01 (AE C653711) and M-02 (AE C657188).

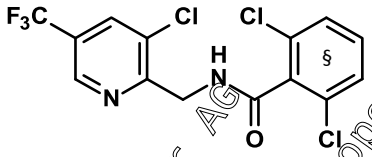
M-03 was observed in both radiolabelled treatments in both soils, reaching a maximum of 7.8% in Lamberton soil in the pyridyl labelled samples (DAT 116) before declining to 6.0% by the next timepoint and to 3.3% AR by the final timepoint. M-01 was also observed in both soils in the phenyl samples, increasing steadily to a maximum of 40.2% AR in Lamberton soil and 19.3% AR in Pikeville soil by the end of the study (DAT 369). M-02 was observed at a maximum of 4.7% AR (DAT 94) in Lamberton soil and 3.2% AR (DAT 369) in Pikeville soil. In addition, three minor unidentified metabolites were detected in the pyridyl treated soil extracts only. Metabolite B was observed at maximum of 5.3% AR, exceeding 5% at one timepoint only in Lamberton soil on DAT 273. Metabolite C was reported to reach a maximum of 5.5% AR on DAT 273 and declined very slightly to 5.2% AR by DAT 369 in Lamberton soil. This metabolite has been further investigated in a statement (See KCA 7.1.2.1.1/06, M-685745-01-1) and it was concluded the region quantified as Metabolite C did not contain a single metabolite at > 5% AR. Metabolite D did not exceed 5% AR in either soil.

I. Materials and Methods

A. Materials

1. Test Items

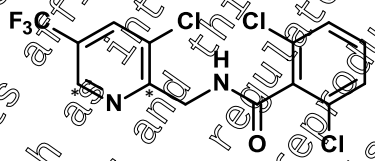
[Phenyl-U-^{14}C]-fluopicolide



§ Denotes position of [^{14}C]-radiolabel

Specific Activity:	5.33 MBq/mg
Radiochemical Purity:	100% (mean of HPLC and TLC analyses)

[2,6-Pyridyl-^{14}C]-fluopicolide



* Denotes position of [^{14}C]-radiolabel

Specific Activity:	5.88 MBq/mg
Radiochemical Purity:	99.97% (mean of HPLC and TLC analyses)

2. Test Soil

The study was performed using one test soil as characterized in Table 7.1.2.1.1- 1.

Table 7.1.2.1.1- 1: Physico-chemical properties of test soil

Parameter		Soil	
Soil Designation		Lamberton	Pikeville
Geographic Location			
City		Lamberton	Pikeville
Country		Minnesota, USA	North Carolina, USA
Batch Number		EFS-92	EFS-96
Textural Classification (USDA)		Sandy clay loam	Loamy sand
Sand [50 - 2000 µm]	(%)	48	86
Silt [2 – 50 µm]	(%)	24	8
Clay [< 2 µm]	(%)	28	6
pH			
in H_2O (1:1)		5.9	6.3
in $CaCl_2$ (1:1)		5.9	5.7
Organic Matter (%)		6.1	2.8
Organic Carbon (%) *		3.5	1.6
Cation Exchange Capacity (meq/100 g)		24.6	5.7
Water Holding Capacity (%)			
maximum		77.1	35.5
at 1/10 bar		39.4	16.8
at 1/3 bar		30.9	11.5
at 15 bar		19.3	4.2
Moisture Content During Incubation (%)		23.1	8.6
Bulk Density (disturbed, g/cm³)		1.09	1.39
Soil Microbial Biomass (µg microbial C /g soil)			
Initial (DAT 0)		508.3	85.4
Final (DAT 369)		164.9	30.5

* Calculated by dividing organic matter content by 1.72

B. Study Design

1. Experimental Conditions

Tests were performed in flow through systems consisting of glass flasks each containing 50 g soil and attached to an ethylene glycol trap to collect organic volatiles followed by an ethanolamine (or sodium hydroxide) trap to collect carbon dioxide. Humidified air was passed through the samples. Soil was adjusted to 75% of the 1/3 bar water holding capacity which was maintained throughout the course of the study.

The tests were performed at a concentration of approximately 0.41 mg/kg dry weight of soil. The test concentration was based on a field rate of 400 g a.s./ha. The test items [phenyl-U-^{14}C]- or [2,6-pyridyl-^{14}C]-fluopicolide, dissolved in acetonitrile (406 and 460 µL, respectively), were applied drop wise onto the soil surface. Soil samples were adjusted to a moisture content equivalent to 75% of ⅓ bar, at least two days prior to application. The samples were incubated at 25 ± 1 °C under aerobic conditions in the dark for 369 days.

2. Sampling

Single samples each were removed for analysis after 0, 14, 31, 60, 94, 116, 188, 273, and 369 days of incubation. Untreated samples were analysed for biomass at the beginning and end of the experiment.

3. Analytical Procedures

Soil samples were extracted up to four times successively with acetonitrile/water (4/1, v/v) at ambient temperature followed by a Soxhlet extraction with acetonitrile. Radioactivity in extracts was determined by liquid scintillation counting (LSC). Soil extracts were concentrated and analysed by HPLC with radiodetection. Degradation products were identified by comparison of the retention times of reference standards. Selected extracts were analysed by LC/MS/MS for confirmation of the major peaks identified by HPLC. A peak of 300 dpm, corresponding to 0.9 ng fluopicolide, was readily determined by the TLC and HPLC quantitation methods used.

Volatile radioactivity in volatile traps was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide, M-01 and M-03 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). For fluopicolide, as the degradation was investigated using two radiolabel positions, and similar behaviour was observed for each, these radiolabels have been considered as true replicates, and included together in a single optimisation. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each soil between the SFO and FOMC fits for fluopicolide. For the Lamberton soil, the SFO model provided the best fit to the fluopicolide residues, with the lowest χ^2 err% value. For the Pikeville soil, the FOMC model provided a better fit than the SFO model to the fluopicolide residues, and the DFOP model was therefore also fitted. DFOP was selected as the most appropriate model for fluopicolide, with the best visual fit and lowest χ^2 err% value.

Metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soils incubated at 25 °C following application of [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]-fluopicolide are summarized in Table 7.1.2.1.1- 2 to Table 7.1.2.1.1- 5.

Table 7.1.2.1.1- 2: Degradation of [phenyl-U-^{14}C]-fluopicolide at 25 °C in Lamberton soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)								
	0	14	31	60	94	116	188	273	369
Fluopicolide	94.4	85.0	82.3	85.8	65.3	69.4	64.2	63.0	40.4
M-01 (AE C653711)	nd	0.4	nd	3.5	11.1	18.1	20.6	25.3	40.2
M-03 (AE 0608000)	nd	nd	nd	4.5	6.5	4.3	4.5	6.3	3.1
Ambient extract	89.9	75.7	69.6	74.3	70.1	75.6	71.5	74.5	66.0
Soxhlet extract	4.5	9.7	12.7	19.5	12.8	16.2	18.8	20.6	18.1
Total extractable radioactivity [A]	94.4	85.4	82.3	93.8	82.9	91.8	90.3	95.1	84.1
Non-extractable radioactivity	0.6	1.4	4.0	2.9	6.5	4.9	4.2	2.3	4.8
^{14}C-Carbon dioxide including other volatiles [B]	na	0.0	0.0	0.1	0.0	0.1	0.0	0.2	0.2
Total radioactivity	95.0	86.8	86.3	96.8	89.4	96.8	94.5	97.6	89.1

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[B] Other volatile radioactivity was < 0.05 % AR at all timepoints

Table 7.1.2.1.1- 3: Degradation of [2,6-pyridyl-^{14}C]-fluopicolide at 25 °C in Lamberton soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)								
	0	14	31	60	94	116	188	273	369
Fluopicolide	83.9	82.6	81.0	81.3	69.2	68.0	63.4	54.6	45.3
M-02 (AE C657188)	nd	nd	nd	nd	4.4	4.2	3.4	3.0	2.6
M-03 (AE 0608000)	nd	nd	nd	4.8	3.8	7.8	6.0	3.9	3.3
Metabolite B	nd	nd	nd	nd	nd	nd	nd	5.3	2.3
Metabolite C [A]	nd	nd	nd	nd	nd	nd	nd	5.5	5.2
Metabolite D	nd	nd	nd	nd	nd	nd	nd	4.8	2.3
Ambient extract	83.9	71.8	70.8	66.9	64.1	66.5	54.0	55.4	47.9
Soxhlet extract	10.9	10.8	10.2	19.2	13.6	13.6	19.4	21.7	14.2
Total extractable radioactivity [B]	84.8	82.6	81.0	86.1	77.7	80.1	73.4	77.1	62.1
Non-extractable radioactivity	0.3	3.0	5.2	5.1	10.5	14.8	22.6	18.8	22.6
^{14}C-Carbon dioxide including other volatiles [C]	na	0.0	0.0	0.1	0.0	0.0	0.0	0.2	0.1
Total radioactivity	85.1	85.6	86.2	91.3	88.2	94.9	96.0	96.1	84.8

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] The region quantified as Metabolite C did not contain a single metabolite at > 5% AR. See statement KCA 7.1.2.1.1/06, [redacted] 2020; M-685745-01-1 for further details.
[B] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[C] Other volatile radioactivity was < 0.05 % AR at all timepoints

Table 7.1.2.1.1- 4: Degradation of [phenyl-U-^{14}C]-fluopicolide at 25 °C in Pikeville soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)								
	0	14	31	60	94	116	188	273	369
Fluopicolide	97.6	91.1	81.7	80.3	68.6	68.6	78.5	57.3	49.6
M-01 (AE C653711)	nd	4.7	14.3	12.5	20.5	20.5	4.5	18.6	19.3
M-03 (AE 0608000)	nd	nd	nd	1.4	nd	nd	1.2	1.8	1.6
Ambient extract	97.6	83.5	80.3	72.3	67.7	67.7	57.6	60.9	47.0
Soxhlet extract	0.5	12.3	15.7	21.9	21.4	23.0	29.6	18.9	33.3
Total extractable radioactivity [A]	98.1	95.8	96.0	94.2	89.1	90.7	87.2	79.8	70.3
Non-extractable radioactivity	0.1	0.7	2.5	3.1	6.6	4.9	10.8	6.4	13.6
^{14}C-Carbon dioxide including other volatiles [B]	na	0.8	0.1	0.2	0.1	0.1	0.0	0.4	0.0
Total radioactivity	98.2	97.3	98.3	97.5	95.8	95.7	98.0	86.6	83.8

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[B] Other volatile radioactivity was < 0.05 % AR at all timepoints

Table 7.1.2.1.1- 5: Degradation of [2,6-pyridyl-^{14}C]-fluopicolide at 25 °C in Pikeville soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)								
	0	14	31	60	94	116	188	273	369
Fluopicolide	96.8	89.1	82.4	81.3	75.5	67.2	74.2	58.3	53.5
M-02 (AE C657188)	nd	nd	1.1	nd	1.5	1.9	nd	2.6	3.2
M-03 (AE 0608000)	nd	nd	nd	0.5	1.3	1.5	0.5	2.0	2.8
Metabolite B	nd	nd	nd	nd	nd	2.7	nd	3.3	0.8
Metabolite C	nd	nd	nd	nd	nd	nd	nd	nd	nd
Metabolite D	nd	2.8	3.1	3.5	nd	nd	nd	2.2	4.0
Ambient extract	96.8	82.5	76.1	64.8	59.2	54.4	52.1	51.6	43.5
Soxhlet extract	1.0	9.8	12.6	21.0	19.1	20.6	23.3	17.8	20.8
Total extractable radioactivity [A]	97.8	92.3	88.7	85.8	78.3	75.0	75.4	69.4	64.3
Non-extractable radioactivity	0.2	3.3	7.6	10.0	12.2	16.2	19.3	8.9	16.7
^{14}C-Carbon dioxide including other volatiles [B]	na	0.3	1.0	1.6	0.2	0.4	0.0	0.2	0.2
Total radioactivity	98.0	95.9	97.3	97.4	90.7	91.6	94.7	78.5	81.2

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[B] Other volatile radioactivity was < 0.05 % AR at all timepoints

B. Material Balance

Mass balances in the Lamberton soil ranged from 86.3% to 97.6% of the applied radioactivity (mean 92.5% AR) for samples treated with [phenyl-U-^{14}C]-fluopicolide and 84.8% to 96.1% AR (mean 89.8% AR) for samples treated with [2,6-pyridyl-^{14}C]-fluopicolide.

Mass balances in the Pikeville soil ranged from 83.3% to 98.3% AR (mean 94.5% AR) for samples treated with [phenyl-U-^{14}C]-fluopicolide and 78.5% to 98.0% AR (mean 91.7% AR) for samples treated with [2,6-pyridyl-^{14}C]-fluopicolide.

The low recoveries in mass balance could not be attributed to a single factor such as dosing errors, extraction losses, volatile losses or adsorption to laboratory equipment.

C. Extractable and Non-Extractable Residues

The majority of the radioactivity was extractable in both soils. For samples of Lamberton soil incubated with [phenyl-U-^{14}C]-fluopicolide, total extractable radioactivity decreased from 94.4% AR at DAT 0 to 84.1% AR by DAT 369. For Lamberton soil incubated with [2,6-pyridyl-^{14}C]-fluopicolide, total extractable radioactivity decreased from 84.8% AR on DAT 0 to a minimum of 62.1% AR after 369 days.

Radioactivity in the combined soil extracts of Pikeville soil incubated with [phenyl-U-^{14}C]-fluopicolide decreased from 98.1% at Day 0 to 70.3% at DAT 369 and from 97.8% at DAT 0 to 64.3% at DAT 369 in samples incubated with [2,6-pyridyl-^{14}C]-fluopicolide

Non-extractable soil residues increased concurrently with the decrease in extractable radioactivity in both soils. In Lamberton soil, at DAT 369, non-extractable residues reached a maximum of 22.6% AR in the pyridyl treatment, compared to 4.8% AR in the phenyl treatment at the same timepoint suggesting the formation of soil residues from the pyridine ring. Similar differences were observed in Pikeville soil where non-extractable residues reached a maximum of 19.3% AR in the pyridyl treatment (DAT 188) compared to 10.8% in the phenyl treatment at the same timepoint.

D. Volatile Radioactivity

The presence of CO_2 in the ethanolamine trap reached a maximum of 1.6% AR only in the pyridyl label treatment of Pikeville soil at DAT 60. At all other intervals, the recovery of radioactivity in the ethanolamine traps was minimal, regardless of soil or label. Volatilization of fluopicolide and metabolites was not considered to be a dissipation pathway under aerobic conditions in soil as indicated by the lack of radioactivity detected (< 0.05% AR) in the ethylene glycol traps during the study.

E. Degradation of Parent Compound

In Lamberton soil treated with the phenyl label, fluopicolide declined to 40.4% AR after 369 days of incubation at 25°C under aerobic conditions. In the same soil, treated with the pyridyl label, fluopicolide declined to 45.3% AR over the same time period. Oxidative cleavage of fluopicolide to form M-01 (AE C653711) and M-02 (AE C657188) was considered the major degradation pathway. Prior to cleavage, formation of an addition product M-03 (AE 0608000) reached maximum levels of 6.5% and 7.8% and 6.5% in the phenyl (DAT 94) and pyridyl (DAT 116) labels, respectively before declining to approximately 3% in both labels at study termination. Other metabolites occurred in the pyridyl treatment only, namely Metabolites B to D, however no single metabolite exceeded 6% of applied. Metabolites B and D were observed at maxima of 5.3 and 4.8%, respectively, after 273 days incubation before declining to <3% at the end of the incubation period (369 days). Metabolite C was observed at a maximum of 5.5% after 273 days incubation before declining slightly to 5.2% by DAT 369. This metabolite has been further investigated in a statement (See KCA 7.1.2.1.1/06, M-685745-01-1) and it was concluded the region quantified as Metabolite C did not contain a single metabolite at > 5% AR. It was only detected at these two timepoints throughout the incubation period in Lamberton soil by which point the soil biomass had declined by 70% of the initial level. Levels of M-02 reached a maximum of

4.7% at DAT 94 before declining to 2.6% at termination. Levels of M-01 increased steadily to 40.2% by DAT 369.

In the Pikeville soil treated with the phenyl label fluopicolide declined to 49.3% of the applied radioactivity after 369 days incubation. In the pyridyl treatment fluopicolide declined to 53.5% over the same time period. M-03 reached maximum levels of 1.8% (DAT 273) and 2.8% (DAT 369) in the phenyl and pyridyl labels, respectively. Metabolites B and D were observed in the pyridyl treatment only, at maximum levels of 3.3% (DAT 273) and 4.0% (DAT 369), respectively. Metabolite C was not observed in Pikeville soil. Levels of M-02 reached a maximum of 3.2% by DAT 369. Levels of M-01 increased steadily to 19.3% AR at study termination.

The potential leaching risk of pyridyl ring metabolites has been addressed in a lysimeter study conducted with [2,6-pyridyl-^{14}C]-labelled fluopicolide (see KCA 7.1.4.2/01, [redacted] 2004; M-218906-01-1).

On the basis of the chromatographic and mass spectrometric investigations the presence of fluopicolide, M-01 and M-02 were confirmed and Metabolite A was concluded to be M-03. The primary route of degradation in soil was assumed to be via the addition product, M-03 before oxidative cleavage to form two products, M-01 (2,6-dichlorobenzamide; AE C653711) and M-02 (AE C657188). Association with soil or soil constituents to form non-extractable residues occurred with both radiolabels. Non-extractable soil residues were higher in the respective pyridyl labelled treatments compared to the phenyl treatments suggesting that M-02 degrades to components that are associated with the soil matrix.

F. Degradation Kinetics

Fluopicolide degraded slowly in the Lamberton sandy clay loam and Pikeville loamy sand soils under non-sterile conditions. In Lamberton soil reported DT_{50} values were 282 and 270 days (mean = 276 days) for the phenyl- and pyridyl- labels, respectively. In Pikeville soil reported DT_{50} values were 323 and 336 days (mean = 330 days) for the phenyl- and pyridyl- labels, respectively. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.1.1- 6. Best fit kinetics are highlighted in bold.

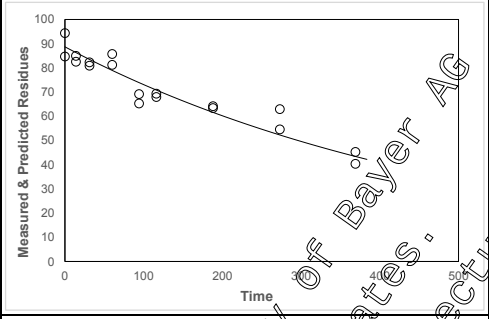
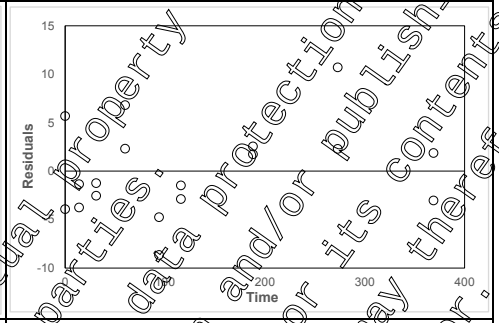
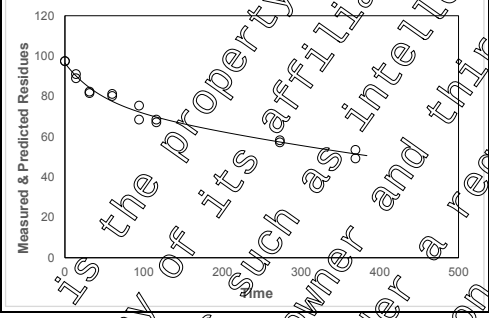
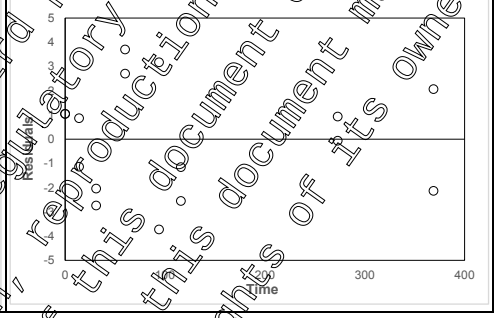
Table 7.1.2.1.1- 6: Degradation rate of fluopicolide under aerobic conditions at 25 ºC (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Lamberton, Allan (2003c)	**SFO**	**88.71**	**k 0.001936**	**4.25**	**<2e-16**	**0.001752**	**0.002**	**358**	**1189.4**
	FOMC	88.19	α 1.753 β 835.6	4.27	n.r. n.r.	-4.007 -2357	7.513 4028	405.2	2271.9
Pikeville, Allan (2003c)	SFO	91.14	k 0.001673	5.64	6.52E-09	0.00129	0.002	414.4	1376.5
	FOMC	96.82	α 0.2417 β 43.71	5.16	n.r. n.r.	0.08526 -21.14	0.398 108.6	725.8	>10000
	DFOP	**96.55**	**k1 0.02302 k2 0.001131 g 0.1914**	**1.97**	**0.00565 6.56E-06 n.r.**	**0.006936 0.0007505 0.11**	**0.039 0.002 0.273**	**424.9**	**1847.5**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.1.1- 7: Degradation of fluopicolide under aerobic conditions at 25 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Lamberton SFO Allan (2003c)		
Pikeville DFOP Allan (2003c)		

III. Conclusion

Fluopicolide slowly degraded in Lamberton and Pikeville soils under aerobic conditions at 25°C. Less than 2% AR was detected in the ethanolamine traps indicating very slow mineralization of fluopicolide to CO_2. Organic volatiles were not detected in either of the treated soils regardless of label position.

The primary metabolic pathway was proposed to be the formation of an oxidative addition product M-03, followed by cleavage to form M-01 and M-02. M-03 was observed at a maximum of 7.8% AR before declining to 3.3% AR by the final timepoint. M-01 was observed in the phenyl samples only reaching a maximum of 40.2% AR by the end of the study. M-02 was observed in the pyridyl samples only at a maximum of 4.7% AR.

Levels of non-extractable residues were higher in general in the pyridyl labelled treatments suggesting that M-02 and other degradates from the pyridyl ring are closely associated with the soil matrix. A number of minor unidentified metabolites were detected from the pyridyl treatments in both soils, none of which individually exceeded 6% AR. It was concluded that these metabolites accumulated to these levels because the microbial viability of the soil had declined significantly on ageing for 273 and 369 days and were unlikely to form at significant levels in the environment (see KCA 7.1.2.1.1/06, [redacted] 2020; M-685745-01-1).

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of 358 and 424.9 days for Lamberton and Pikeville soils, respectively.

Assessment and conclusion by applicant:

The study was conducted in accordance with USEPA (= EPA) N, 162-1 (1982). The study is considered valid to assess the aerobic degradation of [phenyl-U-^{14}C] and [2,6-pyridyl-^{14}C]-fluopicolide in soil.

Data Point:	KCA 7.1.2.1.1/02
Report Author:	[redacted]
Report Year:	2003
Report Title:	The kinetics of degradation of [2,6-14C-pyridinyl] and [U-14C-benzoyl]-AE C638206 in a U.S. loam under laboratory aerobic conditions at 20 degrees C
Report No:	B004073
Document No:	M-241051-01-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EC of July 1995
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The rate of degradation of fluopicolide was investigated in an USA soil under laboratory aerobic conditions for up to 120 days. [Phenyl-U-^{14}C]-labelled fluopicolide or [2,6-pyridyl-^{14}C]-labelled fluopicolide was applied to soil samples at an application rate equivalent to 400 g/ha. Lamberton soil was classified as a loam soil according to USDA classification. Soil samples were incubated in the dark, at a moisture content equivalent to pF 2.5 under aerobic conditions at 20 °C. The radiochemical purity was > 99 % for both radiolabelled test items. The specific activities were 5.33 and 5.88 MBq/mg for [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]-labelled fluopicolide, respectively.

Samples were taken for extraction and analysis immediately after treatment (day 0) and after 14, 28, 42, 56, 77, 98 and 120 days of incubation. Soil samples were exhaustively extracted with up to four successive extractions with acetonitrile / water (4 / 1, v/v) at ambient temperature followed by Soxhlet extraction with acetonitrile. Concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC). Selected samples were analysed by thin layer chromatography (TLC) to confirm the results obtained by HPLC.

Material balances ranged from 88.8 to 99.0% AR for samples incubated with [phenyl-U-^{14}C]-fluopicolide (mean 94.9% AR) and from 87.4 to 99.7% AR for those treated with [2,6-pyridyl-^{14}C]-fluopicolide (mean 94.7% AR). Extractable radioactivity decreased slightly from a maximum of 94.0% AR at DAT 0 to 87.5% AR by DAT 120 for the [^{14}C phenyl]-residues and from 96.2% AR on DAT 0 to 90.9% AR after 120 days of incubation for the [^{14}C pyridyl]-residues. Non-extractable residues increased slightly with the decrease in extractable radioactivity over the 120 day study. The maximum amount of non-extractable residues was observed at DAT 98 at levels of 7.4% in the phenyl label and 9.2 % in the pyridyl label. Very little mineralization to carbon dioxide was observed with ≤ 0.1% of applied radioactivity detected in volatile traps at the end of the study.

The quantity of fluopicolide ranged from 96% and 94 % AR at DAT 0 and declined to 81.6% and 90.9% AR at DAT 120 in the phenyl and pyridyl labels, respectively. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of 1290.0 days and DT_{90} values of 4285.0 days in Lamberton soil. This result is not consistent with the overall behaviour of fluopicolide in all other soils, including a study conducted with the same soil incubated at 25°C, and may be considered as an outlier.

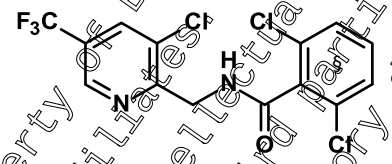
Fluopicolide was degraded initially to a minor metabolite, presumed to be M-03 (AE 0608000), which was degraded to the oxidative cleavage product, M-01 (AE C653711) in the phenyl labelled samples. The corresponding cleavage product M-02 (AE C657188) was not detected in the pyridyl labelled samples. M-03 was observed in both radiolabelled treatments but did not accumulate, reached a maximum of 2.7% in the phenyl labelled samples (DAT 56) and 3.1% in the pyridyl samples (DAT 77). After 42 days, levels of M-01 began to form (2.1% AR) and gradually increased to 4.8% AR by DAT 120.

I. Materials and Methods

A. Materials

1. Test Items

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

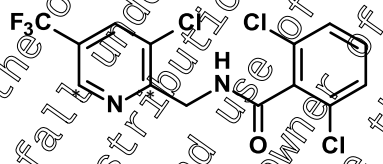


§ Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.33 MBq/mg

Radiochemical Purity: 100% (mean of HPLC and TLC analyses)

[2,6-Pyridyl-^{14}C]-fluopicolide



* Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.88 MBq/mg

Radiochemical Purity: 99.97% (mean of HPLC and TLC analyses)

2. Test Soils

The study was performed using one test soil as characterized in Table 7.1.2.1.1- 8.

Table 7.1.2.1.1- 8: Physico-chemical properties of test soil

Parameter		Soil
Soil Designation		Lamberton
Geographic Location		
City		Lamberton
Country		Minnesota, USA
Textural Classification (USDA)		Loam
Sand [50 - 2000 µm]	(%)	44
Silt [2 – 50 µm]	(%)	30
Clay [< 2 µm]	(%)	26
pH		
in H_2O (1:1)		5.9
in $CaCl_2$ (1:1)		5.6
Organic Matter (%)		5.6
Organic Carbon (%)*		3.3
Cation Exchange Capacity (meq/100 g)		26.4
Water Holding Capacity (%)		
maximum		78.3
at 1/10 bar		41.2
at 1/3 bar		30.2
at 15 bar		20.5
Moisture Content During Incubation (%)		30.2
Bulk Density (disturbed, g/cm³)		1.06
Soil Microbial Biomass (µg microbial C /g soil)		
Initial (DAT 0)		559.5
Final (DAT 121)		344.0

* Calculated by dividing organic matter content by 1.72

B. Study Design

1. Experimental Conditions

Tests were performed in flow through systems consisting of glass flasks each containing 50 g soil and attached to an ethylene glycol trap to collect organic volatiles followed by an ethanolamine (or sodium hydroxide) trap to collect carbon dioxide.

The tests were performed at a concentration of approximately 0.41 mg/kg dry weight of soil. The test concentration was based on a field rate of 400 g a.s./ha. The test items [phenyl-U-^{14}C]- or [2,6-pyridyl-^{14}C]-fluopicolide, dissolved in acetonitrile (406 and 460 µL, respectively), were applied drop wise onto the soil surface. Soil samples were adjusted to a moisture content of 30.2%, equivalent to pF 2.5, two days prior to application. The samples were incubated at 20 ± 1 °C under aerobic conditions in the dark for 120 days.

Additional untreated flasks were used to monitor the viability of the test system by determination of biomass.

2. Sampling

Single samples each were removed for analysis after 0, 14, 28, 42, 56, 77, 98, and 120 days of incubation. Untreated samples were analysed for biomass at the beginning and end of the experiment.

3. Analytical Procedures

Soil samples were extracted up to four times successively with acetonitrile/water (4/1, *v/v*) at ambient temperature followed by one Soxhlet extraction using acetonitrile. Radioactivity in extracts was determined by liquid scintillation counting (LSC). Soil extracts were concentrated and analysed by HPLC with radiodetection. Degradation products were identified by comparison of the retention times of reference standards and confirmed in selected samples by TLC co-chromatography with reference items. A peak of 300 dpm, corresponding to 0.9 ng fluopicolide, was readily determined by TLC and HPLC quantitation methods used.

Volatile radioactivity in volatile traps was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide and M-01 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). For fluopicolide, as the degradation was investigated using two radiolabel positions, and similar behaviour was observed for each, these radiolabels have been considered as true replicates and included together in a single optimisation. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed between the SFO and FOMC fits for fluopicolide in the Lamberton soil. The FOMC fit provided no significant improvement on the SFO fit by visual comparison, and no further bi-phasic fits were performed. Confidence in the degradation rate constant, k, for fluopicolide was slightly low (r^2=0.077), however this is attributed to the relatively slow degradation observed in this study and the parameter estimate was still considered reliable.

Metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soil incubated at 20 °C following application of [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]-fluopicolide are summarized in Table 7.1.2.1.1- 9 to Table 7.1.2.1.1- 10.

Table 7.1.2.1.1- 9: Degradation of [phenyl-U-^{14}C]-fluopicolide at 20 °C in Lamberton soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)							
	0	14	28	42	56	77	98	120
Fluopicolide	94.0	86.4	88.3	90.3	91.7	86.5	84.5	81.6
M-01 (AE C653711)	nd	nd	nd	2.1	2.5	1.2	2.9	4.8
M-03 (AE 0608000) [A]	nd	nd	nd	2.2	2.7	1.9	2.1	1.1
Total extractable radioactivity [B]	94.0	86.4	88.3	94.6	96.9	89.6	89.5	87.5
Non-extractable radioactivity	3.5	2.3	4.0	2.3	2.1	4.9	7.4	5.4
^{14}C-Carbon dioxide including other volatiles [C]	na	0.1	0.1	0.1	nd	nd	nd	nd
Total radioactivity	97.5	88.8	92.4	97.0	99.0	94.5	96.9	92.9

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] Metabolite A proposed to be M-03 (AE 0608000)
[B] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[C] Other volatile radioactivity was < 0.05 % AR at any timepoint

Table 7.1.2.1.1- 10: Degradation of [2,6-pyridyl-^{14}C]-fluopicolide at 20 °C in Lamberton soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)							
	0	14	28	42	56	77	98	120
Fluopicolide	96.2	82.7	80.4	91.1	90.0	89.9	86.8	90.9
M-02 (AE C657188)	nd	nd	nd	nd	nd	nd	nd	nd
M-03 (AE 0608000) [A]	nd	0.5	0.9	1.8	nd	3.1	2.7	nd
Total extractable radioactivity [B]	96.2	83.9	81.3	92.9	90.0	93.4	89.5	90.9
Non-extractable radioactivity	3.5	3.4	4.7	3.6	3.7	5.2	9.2	6.0
^{14}C-Carbon dioxide including other volatiles [C]	na	0.1	0.1	0.1	nd	nd	nd	nd
Total radioactivity	99.7	87.4	86.1	96.6	93.7	98.6	98.7	96.9

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of applied radioactivity (% AR)
[A] Metabolite A proposed to be M-03 (AE 0608000)
[B] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.
[C] Other volatile radioactivity was < 0.05 % AR at all timepoints

B. Material Balance

Material balances ranged from 88.8 to 99.0% AR for samples incubated with [phenyl-U-^{14}C]-fluopicolide (mean 94.9% AR) and from 87.4 to 99.7% AR for those treated with [2,6-pyridyl-^{14}C]-fluopicolide (mean 94.7% AR).

C. Extractable and Non-Extractable Residues

For samples incubated with [phenyl-U-^{14}C]-fluopicolide, total extractable radioactivity decreased from 94.0% AR at DAT 0 to 87.5% AR by DAT 120. For samples incubated with [2,6-pyridyl-^{14}C]-fluopicolide, total extractable radioactivity decreased from 96.2% AR on DAT 0 to a minimum of 89.5% AR after 98 days, and was 90.9% AR after 120 days of incubation.

Non-extractable residues increased slightly with the decrease in extractable radioactivity over the 120 day study. The maximum amount of non-extractable residues was observed at DAT 98 at levels of 7.4% in the phenyl label and 9.2 % in the pyridyl label.

D. Volatile Radioactivity

Very little mineralization to carbon dioxide was observed with ≤ 0.1% of applied radioactivity detected in volatile traps at the end of the study. Only trace amounts of radioactivity were recovered in the volatile traps containing ethanolamine (maximum of 0.05% of applied). There was no volatilization of fluopicolide or its metabolites.

E. Degradation of Parent Compound

Fluopicolide was the principal radiolabelled component detected. Levels of parent accounted for 100% of extracted radioactivity at DAT 0 and declined slightly to 81.6 and 90.9% of applied radioactivity by the end of the study of the study in the soil samples treated with [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C] labelled fluopicolide, respectively. In addition to parent material, M-01 (AE C653711) was detected at a maximum of 4.8% AR in the soil samples treated with [phenyl-U-^{14}C]-fluopicolide. The corresponding cleavage product M-02 (AE C657188) was not detected in the pyridyl labelled samples. A second metabolite M-03 (AE 0608000) was detected in soil samples treated with both [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]- fluopicolide at maxima of 2.7 and 3.1% AR, respectively.

F. Degradation Kinetics

Fluopicolide degraded slowly in the Lamberton loam soil under non-sterile conditions. The reported DT_{50} values were 365 and 463 days (mean = 414 days) for the phenyl- and pyridyl- labels, respectively. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.1.1- 11. Best fit kinetics are highlighted in bold.

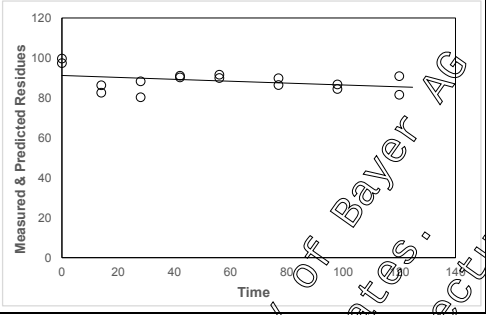
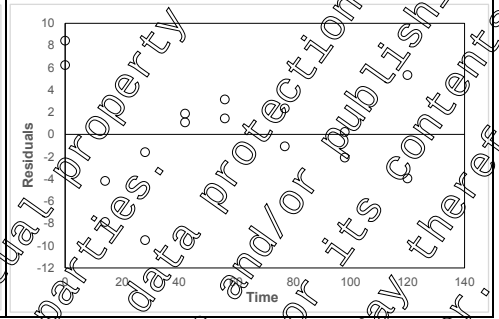
Table 7.1.2.1.1- 11: Degradation rate of fluopicolide under aerobic conditions at 20 ºC (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Lamberton, Allan (2003b)	SFO	91.25	k 0.0005374	3.63	0.077	-0.0001734	0.001	1290	4285
	FOMC	98.46	α 0.00493 β 1.05E-09	2.36	n.r. n.r.	0.00493 9.89E-10	0.005 0	>10000	>10000

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.1.1- 12: Degradation of fluopicolide under aerobic conditions at 20 ºC (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Lamberton SFO Allan (2003b)		

Two studies with Lamberton soil were conducted in the same laboratory by the same study director shortly after each other. However, degradation in this study incubated at 20 °C appeared significantly slower than the study conducted at 25 °C (see KCA 7.1.2.1.101), which cannot be accounted for by temperature or moisture effects. The calculated DT_{50} of 1210 days is therefore not consistent with either the replicate study, or the overall behaviour of fluopicolide in all other soils and may be considered as an outlier.

III. Conclusion

Fluopicolide slowly degraded in the non-sterile loam soil under aerobic conditions. Approximately 82% and 91% of the applied radioactivity remained in the combined soil extracts as fluopicolide in the phenyl and pyridyl label treated soils respectively at the end of 120 days. Less than 0.1% of the radioactivity was detected in the ethanolamine traps indicating very slow mineralization of fluopicolide to CO_2. Organic volatiles were not detected with either radiolabel. The primary metabolic pathway involved formation of M-03 (AE 0608000, maximum of 3.1% AR) followed by cleavage to form M-01 (AE C653711, maximum of 4.8% AR).

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of 1290 days for Lamberton soil. This result is not consistent with the overall behaviour of fluopicolide in all other soils, including a study conducted with the same soil incubated at 25°C, and may be considered as an outlier.

Assessment and conclusion by applicant:

The study was conducted in accordance with SETAC 1.1 (1995) and USEPA (= EPA) N, 162-1 (1982). The study is considered valid to assess the aerobic degradation of [phenyl-U-^{14}C] and [2,6-pyridyl-^{14}C]-fluopicolide in soil.

Data Point:	KCA 7.1.2.1.1/03
Report Author:	[redacted]
Report Year:	2003
Report Title:	Kinetics of Degradation of [2,6-14C-pyridinyl] and [U-14C-benzoyl]-AE C638206 in One Soil at 10 Degrees C under Laboratory Aerobic Conditions
Report No:	B004075
Document No:	M-241053-01-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EC
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The rate of degradation of fluopicolide was investigated in a European soil under laboratory aerobic conditions for up to 131 days. [Phenyl-U-^{14}C]-labelled fluopicolide or [2,6-pyridyl-^{14}C] labelled fluopicolide was applied to soil samples at an application rate equivalent to 400 g /ha. Abington soil was classified as a sandy loam soil according to USDA classification. Soil samples were incubated in the dark, at a moisture content equivalent to 40% maximum water holding capacity under aerobic conditions at 10 ºC. The radiochemical purity was > 98 % for both radiolabelled test items. The specific activities were 5.74 and 5.99 MBq/mg for [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]-labelled fluopicolide, respectively.

Samples were taken for extraction and analysis immediately after treatment (DAT 0) and after 14, 27, 49, 63, 88, 105 and 131 days of incubation. Soil samples were exhaustively extracted with up to four successive extractions with acetonitrile / water (4 + 1, v/v) at ambient temperature followed by Soxhlet extraction with acetonitrile. Concentrated ambient soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC). Soxhlet extracts were not analysed as the levels of radioactivity did not exceed 3% AR at any time throughout the study. The HPLC results were confirmed by LC/MS/MS.

Material balances ranged from 100.7 to 106.4% AR for samples incubated with [phenyl-U-^{14}C]-fluopicolide (mean 103.5% AR) and from 94.4 to 102.5% AR for those treated with [2,6-pyridyl-^{14}C]-fluopicolide (mean 99.4% AR). The majority of the applied radioactivity was extractable at all the timepoints. Percentages of radioactivity were greater than 94% AR in both radiolabelled treatments over the entire length of the study. Non-extractable residues in the pyridyl labelled samples reached 6.1% AR by DAT 131 compared to only 1.3% AR in the phenyl treatment. Very little mineralization to carbon dioxide was observed with < 0.4% of applied radioactivity detected in volatile traps at the end of the study.

The quantity of fluopicolide ranged from 91.9% to 96.3 % AR at Day 0 in the pyridyl and phenyl labels, respectively, and degraded to 84% in both labels by Day 131. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of 671.6 days and DT_{90} values of 2393.0 days in Abington soil.

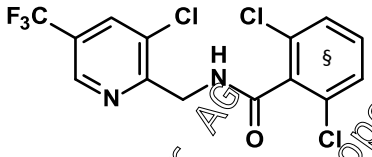
Fluopicolide degraded to form the cleavage products, M-01 (AE C653711) and M-02 (AE C657188). The formation of M-01 steadily increased to a maximum of 16% AR at study termination. M-02 reached a maximum of 6.4% in the pyridyl labelled soil by DAT 63 before degrading slightly to 5% at study termination (DAT 131). This metabolite was degraded to at least four minor unidentified metabolites detected at DAT 131 at < 1% AR each.

I. Materials and Methods

A. Materials

1. Test Items

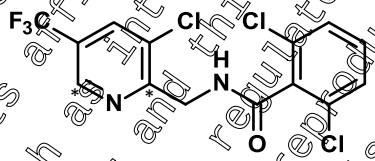
[Phenyl-U-^{14}C]-fluopicolide



§ Denotes position of [^{14}C]-radiolabel

Specific Activity:	5.74 MBq/mg
Radiochemical Purity:	98.3% (by HPLC analysis)

[2,6-Pyridyl-^{14}C]-fluopicolide



* Denotes position of [^{14}C]-radiolabel

Specific Activity:	5.99 MBq/mg
Radiochemical Purity:	98.7% (by HPLC analysis)

2. Test Soils

The study was performed using one test soil as characterized in Table 7.1.2.1.1- 13.

Table 7.1.2.1.1- 13: Physico-chemical properties of test soil

Parameter		Soil
Soil Designation		Abington
Geographic Location		
City		Abington
Country		Cambridgeshire, UK
Textural Classification (USDA)		Sandy loam
Sand [50 - 2000 µm]	(%)	71
Silt [2 – 50 µm]	(%)	22
Clay [< 2 µm]	(%)	7
pH		
in H_2O (1:1)		7.4
in $CaCl_2$ (1:1)		7.2
Organic Matter (%)		4.7
Organic Carbon (%) *		2.7
Cation Exchange Capacity (meq/100 g)		21.1
Water Holding Capacity (%)		
maximum		63.5
at 1/10 bar		18.2
at 1/3 bar		15.8
at 15 bar		11.4
Moisture Content During Incubation (%)		25.0
Bulk Density (disturbed, g/cm^3)		1.11
Soil Microbial Biomass (µg microbial C /g soil)		
Initial (DAT 0)		440.6
Final (DAT 131)		236.9

* Calculated by dividing organic matter content by 1.72

B. Study Design

1. Experimental Conditions

Tests were performed in flow through systems consisting of glass flasks each containing 50 g soil and attached to an ethanolamine trap to collect carbon dioxide.

The tests were performed at a concentration of approximately 0.41 mg/kg dry weight of soil. The test concentration was based on a field rate of 400 g a.s./ha. The test items [phenyl-U-^{14}C]- or [2,6-pyridyl-^{14}C]-fluopicolide, dissolved in acetonitrile (456 and 522 µL, respectively), were applied drop wise onto the soil surface. Soil samples were adjusted to a moisture content of 25%, equivalent to 40% of maximum water holding capacity, two days prior to application. The samples were incubated at 10 ± 1 °C under aerobic conditions in the dark for 131 days.

Additional untreated flasks were used to monitor the viability of the test system by determination of biomass.

2. Sampling

Single samples each were removed for analysis after 0, 14, 27, 49, 63, 88, 105, and 131 days of incubation. Untreated samples were analysed for biomass at the beginning and end of the experiment.

3. Analytical Procedures

Soil samples were extracted up to four times successively with acetonitrile/water (4/1, *v/v*) at ambient temperature followed by a Soxhlet extraction with acetonitrile. Radioactivity in extracts was determined by liquid scintillation counting (LSC). Ambient soil extracts were concentrated and analysed by HPLC with radiodetection. Soxhlet extracts were not analysed as the levels of radioactivity did not exceed 3% at any time throughout the study. Degradation products were identified by comparison of the retention times of reference standards and confirmed in selected samples by LC/MS/MS. A peak of 300 dpm, corresponding to 0.9 ng of fluopicolide in samples treated with [2,6-pyridyl-^{14}C]-fluopicolide or 0.8 ng of fluopicolide in samples treated with [phenyl-U-^{14}C]-fluopicolide, was readily determined by TLC and HPLC quantitation methods used.

Volatile radioactivity in volatile traps was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide and M-01 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). For fluopicolide, as the degradation was investigated using two radiolabel positions, and similar behaviour was observed for each, these radiolabels have been considered as true replicates, and included together in a single optimisation. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

An initial comparison was performed between the SFO and FOMC fits for fluopicolide in the Abington soil. The FOMC model provided a better fit than the SFO model, and the DFOP model was therefore also fitted. The FOMC fit gave the lowest χ^2 err% value but was not accepted, as extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using this model (EFSA, 2009). DFOP was instead selected as the most appropriate model to describe fluopicolide degradation.

Metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soil incubated at 10 °C following application of [phenyl-U-^{14}C]- and [2,6-pyridyl-^{14}C]- fluopicolide are summarized in Table 7.1.2.1.1-14 to Table 7.1.2.1.1- 15.

Table 7.1.2.1.1- 14: Degradation of [phenyl-U-^{14}C]-fluopicolide at 10 °C in Abington soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)							
	0	14	27	49	63	88	105	131
Fluopicolide	96.3	95.4	92.4	86.9	89.3	84.1	87.0	84.3
M-01 (AE C653711)	4.0	6.1	7.8	10.3	11.8	12.4	14.4	16.3
Ambient extracts	100.3	101.5	100.2	97.5	101.5	96.5	102.0	100.6
Soxhlet extract	1.1	1.8	2.1	2.6	2.6	2.7	3.0	3.1
Total extractable radioactivity [A]	101.4	103.3	102.3	100.1	104.1	99.2	105.0	103.7
Non-extractable radioactivity	na	1.2	1.0	1.0	1.2	1.2	1.3	1.3
^{14}C-Carbon dioxide	na	0.2	0.3	0.3	na	0.3	0.1	0.0
Total radioactivity	101.4	104.7	103.6	101.4	105.3	100.7	106.4	105.0

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.

Table 7.1.2.1.1- 15: Degradation of [2,6-pyridyl-^{14}C]-fluopicolide at 10 °C in Abington soil under aerobic conditions [% AR]

Compound	Incubation time (DAT)							
	0	14	27	49	63	88	105	131
Fluopicolide	91.9	89.9	89.5	89.2	87.4	87.8	85.7	83.9
M-02 (AE C657188)	0.7	2.6	6.2	5.8	6.4	3.6	5.2	5.0
Ambient extracts	93.4	92.5	95.7	95.8	94.3	95.5	93.7	93.0
Soxhlet extract	1.0	1.8	2.1	2.0	3.0	2.7	2.7	1.6
Total extractable radioactivity [A]	94.4	94.3	97.8	97.6	97.3	98.2	96.4	94.6
Non-extractable radioactivity	na	0.6	1.3	2.9	3.3	3.9	5.0	6.1
^{14}C-Carbon dioxide	na	0.2	0.1	0.3	na	0.4	0.1	0.2
Total radioactivity	94.4	95.1	99.2	100.8	100.6	102.5	101.5	100.9

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] The total extractable radioactivity was calculated as sum of ambient and Soxhlet extracts.

Four minor unidentified pyridyl ring metabolites were also detected but were not quantified as they accounted for a maximum of ca. 4% in total (each less than 1%) at Day 131.

B. Material Balance

Material balances ranged from 100.7 to 106.4% AR for samples incubated with [phenyl-U-^{14}C]-fluopicolide (mean 103.5% AR) and from 94.4 to 102.5% AR for those treated with [2,6-pyridyl-^{14}C]-fluopicolide (mean 99.4% AR).

C. Extractable and Non-Extractable Residues

The majority of the applied radioactivity was extractable at all the timepoints. Percentages of radioactivity were greater than 94% AR in both radiolabelled treatments over the entire length of the study. Non-extractable residues in the pyridyl labelled samples reached 6.1% AR by DAT 131 compared to only 1.3% AR in the phenyl treatment.

D. Volatile Radioactivity

Very little mineralization to carbon dioxide was observed with <1% AR detected in the volatile traps at the end of the study (maximum of 0.4% of applied in the pyridyl labelled samples).

E. Degradation of Parent Compound

The quantity of fluopicolide ranged from 91.9% to 96.3% AR at DAT 0 in the pyridyl and phenyl labels, respectively, and degraded to 84% in both labels by DAT 131. Fluopicolide degraded to form the cleavage products, M-01 (AE C653711) and M-02 (AE C657188). The formation of M-01 steadily increased to a maximum of 16% AR at study termination. No other metabolites were detected from the phenyl labelled treatment. Levels of M-02 reached a maximum of 6.4% in the pyridyl labelled soil by DAT 63 before degrading slightly to 5% at study termination (DAT 131). This metabolite degraded to at least four minor metabolites which were detected at DAT 131 at *ca*. 4% AR in total, each individually less than 1%.

F. Degradation Kinetics

Fluopicolide degraded slowly in the Abington sandy loam soil under aerobic conditions at 10 °C. The reported DT_{50} values were 581 and 752 days (mean = 667 days) for the phenyl- and pyridyl- labels, respectively. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.1.1- 16. Best fit kinetics are highlighted in bold.

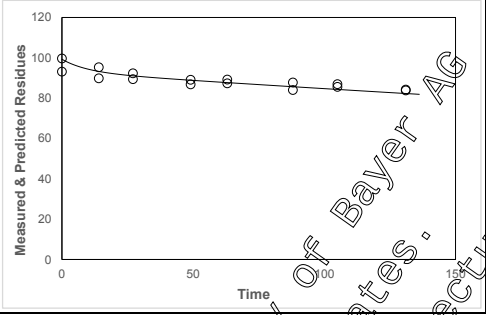
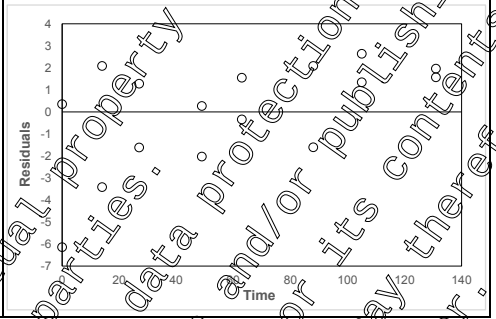
Table 7.1.2.1.1- 16: Degradation rate of fluopicolide under aerobic conditions at 10 ºC (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington Allan (2003d)	SFO	96.46	k 0.001359	1.79	1.52E-05	0.0008619	0.002	510	1694
	FOMC	98.93	α 0.06124 β 9.129	1.13	n.r. n.r.	0.03328 -1.463	0.089 19.72	>10000	>10000
	DFOP	**99.35**	**k1 0.104 k2 0.0009347 g 0.06329**	**1.51**	**0.000806 9.46E-07 n.r.**	**0.04852 0.0006634 0.04512**	**0.159 0.001 0.081**	**671.6**	**2393**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.1.1- 17: Degradation of fluopicolide under aerobic conditions at 10 ºC (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Abington DFOP Allan (2003d)		

III. Conclusion

Fluopicolide slowly degraded in the Abington sandy loam soil under aerobic conditions at 10 ºC. Approximately 84% of the applied radioactivity remained in the soil extracts as parent in both the phenyl and pyridyl label treated soils at the end of 131 days. Less than 0.4% of the radioactivity was detected in the ethanolamine traps indicating very slow mineralization of fluopicolide to CO_2.

The primary metabolic pathway involved the oxidative cleavage of fluopicolide to form M-01 (AE C653711) and M-02 (AE C657188) at maxima of 16.3% AR at DAT 131 and 6.4% AR at DAT 63, respectively.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of 671.6 days for Abbington soil.

Assessment and conclusion by applicant:

The study was conducted in accordance with SETAC 1.1 (1995). The study is considered valid to assess the aerobic degradation of [phenyl-UL-^{14}C] and [2,6-pyridyl-^{14}C]-fluopicolide in soil.

Data Point:	KCA 7.1.2.1.1/04
Report Author:	[redacted]
Report Year:	2003
Report Title:	Route and rate of degradation of [2,6-14C pyridinyl] and [U-14C-benzoyl]-AE C638206 in a European sandy loam under laboratory aerobic conditions at 20 deg. C and determination of aged in situ Kd values at 25 degrees C
Report No:	B004071
Document No:	M-241049-01-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EC of July 1995
Deviations from current test guideline:	Yes. The study design does not conform to current aged sorption guidelines as the aged sorption phase of the study had insufficient timepoints.
Previous evaluation:	yes, evaluated and accepted Tests on aerobic degradation evaluated and accepted in the DAR (2005). Tests on aged sorption evaluated in the DAR (2005) and Addendum 1 to the DAR (2007).
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The details of this study are fully summarised under point KCA 7.1.1.1/01.

Data Point:	KCA 7.1.2.1.1/05
Report Author:	[redacted]
Report Year:	2003
Report Title:	Rate of degradation in two soils (amendment) (14C)-AE C638206
Report No:	C037459
Document No:	M-201230-02-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EEC
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The details of this study are fully summarised under point KCA 7.1.1.1/02.

Data Point:	KCA 7.1.2.1.1/06
Report Author:	[redacted]
Report Year:	2020
Report Title:	Fluopicolide - Statement on the levels of soil metabolite C from M-241052-01-1 (Allan, 2003)
Report No:	VC/19/039B
Document No:	M-685745-01-1
Guideline(s) followed in study:	None
Deviations from current test guideline:	none
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	not applicable
Acceptability/Reliability:	Yes

Executive Summary

This document addresses an unidentified metabolite formed in an aerobic soil study treated with [2,6-pyridyl-^{14}C]-fluopicolide (KCA 7.1.2.1.1/01, [redacted] 2003, M-241052-01-1) which requires further consideration according to current data requirements. The study was conducted in 2003 at which time there was no need to identify metabolites formed at <10% AR and an unidentified metabolite (Metabolite C) was reported at levels above 5% AR at two consecutive timepoints.

Following re-examination of the study raw data it was possible to confirm the reported levels of Metabolite C at the final two timepoints in the Lamberton soil extracts represented a region of radioactivity which contained multiple minor peaks just above the baseline detection level of the HPLC system. It is concluded the region quantified as Metabolite C does not contain a single metabolite at > 5% AR. Consequently, none of the metabolites reported in this study require further consideration in fluopicolide risk assessments.

I. Materials and Methods

In Document KCA 7.1.2.1.1/01, M-241052-01-1 the route and rate of degradation of fluopicolide was investigated in two soils from the USA under laboratory aerobic conditions for up to 369 days. [2,6-Pyridyl-^{14}C] labelled fluopicolide or [phenyl-U-^{14}C]-labelled fluopicolide was applied to soil samples of Lamberton and Pikeville soils. Soil samples were incubated in the dark, at a moisture content equivalent to 75% of ⅓ bar under aerobic conditions at 25 ºC.

In soil samples treated with [2,6-pyridyl-^{14}C]-labelled fluopicolide three minor unidentified degradates were observed in addition to M-02 (AE C657188) and M-03 (AE 0608000); Metabolite B, Metabolite C and Metabolite D.

Metabolite B was only reported to exceed 5% at one timepoint (Maximum 5.3% AR, Day 273). It was observed at a maximum of 5.3% AR after 273 days incubation in Lamberton soil before declining to 2.3% at the end of the incubation period (369 days). In Pikeville soil Metabolite B was observed at a maximum of 3.3% AR after 273 days incubation, declining to 0.8% by 369 days.

Metabolite C was reported to reach a maximum of 5.5% AR on Day 273 and declined very slightly to 5.2% AR by Day 369 in Lamberton soil. It was not observed at earlier timepoints in Lamberton soil and was not observed at all in Pikeville soil.

Metabolite D did not exceed 5% AR (maximum 4.8% AR, Day 273). It was observed at a maximum of 4.8% AR after 273 days incubation in Lamberton soil before declining to 2.3% at the end of the incubation period (369 days). In Pikeville soil Metabolite D was observed throughout the incubation period at levels ranging from 2.2 to 4.0% AR.

According to the guidance applicable at the time the study was conducted there was no need to identify metabolites formed at <10% AR. However Metabolite C requires further consideration according to current data requirements as it exceeded 5% at two consecutive timepoints.

II. Results and Discussion

Biomass levels in soil

Soils in Document KCA 7.1.2.1.1/01, M-241052-01-1 were incubated under aerobic conditions for a total of 369 days as required by US EPA guidelines (EPA 162-1:1982. Untreated samples were analysed for biomass at the beginning (DAT 0) and end of the experiment (DAT 369). Final biomass measurements compared to initial measurements are summarised below.

Table 7.1.2.1.1- 18: Biomass measurements

Parameter	Soil	
Soil Designation	Lamberton	Pikeville
Organic Carbon (%) *	3.5	1.6
Soil Microbial Biomass (µg microbial C /g soil)		
Initial (Day 0)	508.3	85.4
Final (Day 369)	164.9	30.5
Soil Microbial Biomass (% organic carbon)		
Initial (Day 0)	1.5	0.5
Final (Day 369)	0.5	0.2

* Calculated by dividing organic matter content by 1.72

Initial biomass levels were 508.3 µg microbial C /g soil in Lamberton soil and 85.4 µg microbial C /g soil in Pikeville soil, which represented 1.5 and 0.5% of the total organic carbon, respectively. By the end of the incubation period biomass levels had dropped to *ca.* 30% of initial levels in Lamberton soil and 35% of initial levels in Pikeville soil. The final biomass levels at DAT 369 were 164.9 µg microbial C /g soil in Lamberton soil and 30.5 µg microbial C /g soil in Pikeville soil, which represented 0.5 and 0.2% of the total organic carbon, respectively. These values are both well below the OECD 307 recommendation to have at least 1% of total organic carbon soil microbial biomass.

HPLC Radiochromatograms of Lamberton soil extracts

The raw data in Document KCA 7.1.2.1.1/01, M-241052-01-1 was re-examined to confirm whether reported levels of 'Metabolite C' represented a single metabolite. Metabolite C was only detected in soil extracts of Lamberton soil treated with [2,6-^{14}C-pyridyl]-fluopicolide after incubation for 273 and 369 days. It was not detected at earlier timepoints or in soil extracts of Pikeville soil treated with [2,6-^{14}C-pyridyl]-fluopicolide.

Metabolite C was observed largely in the ambient soil extracts. Metabolite C was a wide region of radioactivity eluting between 14 to 20 minutes (with RRT values ranging from 0.3 to 0.5). In chromatograms from both Day 273 and 369 the region quantified as Metabolite C was not a single peak but a region containing multiple minor peaks just above the baseline detection level of the HPLC system. Thus, the region quantified as Metabolite C does not contain a single metabolite at > 5% AR. This region was detected when the microbial viability of the soil had declined significantly on ageing for 273 and 369 days and consequently is unlikely to form at significant levels in the environment under natural conditions.

III. Conclusion

Following re-examination of the HPLC chromatograms from KCA 7.1.2.1.1/01 (M-241052-01-1, [redacted] 2003) it was possible to establish the unidentified Metabolite C was a region of radioactivity containing multiple minor peaks which eluted over a period of 5 minutes rather than a distinct single peak. It was concluded the region quantified as Metabolite C did not contain a single metabolite at > 5% AR. Consequently none of the metabolites reported in this study requires further consideration in fluopicolide risk assessments.

Assessment and conclusion by applicant:

The position paper is considered valid to aid assessment of the route and rate of degradation of [2,6-pyridyl-^{14}C]-fluopicolide in soil.

Data Point:	KCA 7.1.2.1.1/07
Report Author:	[redacted]
Report Year:	2016
Report Title:	[Phenyl-UL-^{14}C]Fluopicolide: Degradation and time - Dependent sorption in soils
Report No:	EnSa-15-0475
Document No:	M-555570-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307 US EPA OCSPP Test Guideline No. 835.4100 / 835.4200 OECD Test Guideline No. 106 (only in parts) Guidance on how aged sorption studies for pesticides should be conducted, analysed and used in the regulatory process; Food and Environmental Agency, York, UK; 2012
Deviations from current test guideline:	none
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation and time-dependence of sorption of fluopicolide was studied in four soils under aerobic conditions in the laboratory in the dark at 20 ± 1 °C and 54.3% of the maximum water holding capacity for 126 days. In addition, the rate of degradation of fluopicolide was determined in the study.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
L[redacted]	sandy loam	6.5	1.5
Dollendorf II	clay loam	7.3	4.8
L[redacted]	loam	5.0	1.8
H[redacted]	silt loam	6.1	1.9

[Phenyl-U-^{14}C]-labelled fluopicolide was applied to soil samples at an application rate of 0.44 mg/kg dry weight. The radiochemical purity and specific activity were > 98% and 5.50 MBq/mg, respectively.

Samples were taken for extraction and analysis immediately after treatment (day 0) and after 2, 7, 10, 14, 28, 57, 85 and 126 days of incubation. Soil samples were first desorbed with 0.01M calcium chloride solution for 24 hours at 20 °C at a soil : solution ratio of 1:3 (w/w) to determine the desorbable portion of the test item from aged soil. The soil residue was then exhaustively extracted with three further successive extractions with acetonitrile/water 4/1 (v/v) at ambient temperature, followed by a microwave extraction with acetonitrile/water (4/1, *v/v*) at 70 °C. Desorption supernatants and concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC).

Recovery of radioactivity was quantitative throughout the study. Overall mean mass balances were 98.9% AR for L[redacted] soil, 99.0% AR for Dollendorf soil, 100.1% AR for L[redacted] soil and 100.0% AR for H[redacted] soil.

Desorbable residues in aqueous 0.01 M calcium chloride solution reached 32.6, 20.0, 32.6 and 28.9% AR in L[redacted], Dollendorf, L[redacted] and H[redacted] soils, respectively by the end of the study (DAT-126). Total extractable residues (i.e. residues desorbed by aqueous 0.01 M calcium chloride solution and residues in organic soil extracts) decreased from 97.8% AR at DAT-0 to 84.1% AR by DAT-126 in I[redacted] soil, from 87.8 to 77.7% AR in Dollendorf soil, from 96.8 to 81.3% AR in L[redacted] soil and from 95.7 to 86.7% AR in H[redacted] soil.

Non-extractable soil residues (NER) increased concurrently with the decrease in extractable radioactivity in all soils, reaching maxima of 11.7, 13.7 and 11.1% AR in L[redacted], L[redacted] and H[redacted] soils at DAT-126. In Dollendorf soil NER levels reached a maximum of 17.6% AR at DAT 57 before declining slightly to 14.5% AR by DAT-126.

The maximum amount of carbon dioxide formed was 4.9, 8.9, 5.5 and 4.5% AR in the four soils by the end of the study (DAT-126). No significant levels of organic volatiles were observed (≤ 0.1% AR).

After 126 days incubation at 20 °C, fluopicolide degraded to 46.4, 45.7, 31.3 and 43.3% of the applied radioactivity in the four soils. M-01 (AE C653711) was also observed in all soils, increasing steadily to maxima of 37.7, 32.0, 47.4 and 43.5% AR by the end of the study. It was identified by LC/MS/MS after isolation from concentrated desorption solutions. In addition, a minor unidentified metabolite was detected in L[redacted] soil at a maximum of 4.6% AR on DAT-10 which declined to 2.6% AR by DAT-126.

The effect of aged sorption to soil was determined for fluopicolide and showed a significant increase with time. Apparent sorption coefficients ($K_{d, app}$) increased with time in all soils by a factor of 1.96 to 2.98 (mean 2.60). Further details specific to the aged sorption of fluopicolide are provided in Section 7.1.3.2.

Degradation kinetics for fluopicolide provided in the report were conducted in accordance with FOCUS guidance document on degradation kinetics (2014). The best-fit DT_{50} values were 107, 112, 48.0 and 96.4 days in L[redacted], Dollendorf, L[redacted] and H[redacted] soils, respectively. A re-evaluation of the degradation kinetics resulted in similar best-fit un-normalised DT_{50} values of 47.7 to 110.2 days.

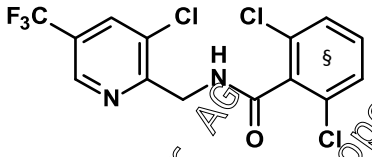
Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)	Visual Assessment
L[redacted] (sandy loam)	DFOP	107	431	1.4	Good
Dollendorf II (clay loam)	DFOP	112	982	2.3	Good
L[redacted] (loam)	DFOP	48.0	308	1.2	Good
H[redacted] (silt loam)	DFOP	96.4	418	1.0	Good

I. Materials and Methods

A. Materials

1. Test Items

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



§ Denotes position of [^{14}C]-radiolabel

Specific Activity:	5.50 MBq/mg
Radiochemical Purity:	>98% (TLC) & >99% (HPLC)

2. Test Soil

The study was performed using four German soils as characterized in Table 7.1.2.1.c-19. The same batches of soils were also used in OECD 106 adsorption desorption studies (see KCA 7.1.3.1/03).

Table 7.1.2.1.1- 19: Physico-chemical properties of test soils

Parameter	Soil							
Soil Designation	L[redacted]		Dollendorf II		[redacted]		H[redacted]	
Soil ID	[redacted]		DD		[redacted]		[redacted]	
Geographic Location City Country	Monheim, Germany		Blankenheim, Germany		Monheim, Germany		Burscheid, Germany	
Batch Number	20140828		20140827		20140828		20140828	
Soil Taxonomic Classification (USDA)	Sandy, mixed, mesic Typic Cambudoll		Fine-loamy, mixed, active, frigid Typic Eutrudept		Loamy, mixed, mesic Typic Argudalf		Loamy, mixed, mesic Typic Argudalf	
Textural Classification (USDA)	Sandy loam		Clay loam		Loam		Silt loam	
Sand [50 - 2000 µm] (%)	75		25		49		15	
Silt [2 – 50 µm] (%)	16		44		32		68	
Clay [< 2 µm] (%)	9		31		19		17	
pH								
in $CaCl_2$ (1:1)	6.5		7.3		5.0		6.1	
in H_2O (1:1)	6.8		7.6		5.3		6.4	
Saturated paste	6.8		7.5		5.3		6.3	
in KCl (1:1)	6.3		7.1		4.7		5.9	
Organic Matter (%) *	2.6		8.3		3.1		3.3	
Organic Carbon (%)	1.5		4.8		1.8		1.9	
Cation Exchange Capacity (meq/100 g)	8.2		18.8		9.7		10.5	
Water Holding Capacity								
Maximum (g H_2O per 100 g DW)	50.5		95.3		68.9		71.4	
at 1/10 bar (%)	13.2		37.9		20.4		34.7	
Moisture Content During Incubation (%)	54.3% MWHC		54.3% MWHC		54.3% MWHC		54.3% MWHC	
Bulk Density (disturbed, g/cm³)	1.23		0.87		1.04		1.04	
Soil Microbial Biomass (µg microbial C /g soil)	BIO⁻	BIO⁺	BIO⁻	BIO⁺	BIO⁻	BIO⁺	BIO⁻	BIO⁺
Initial (DAT 0)	623		2064		798		854	
Mid (DAT 57)	372	358	1800	1764	471	460	661	514
Final (DAT 126)	226	211	1449	1408	305	265	406	397

* Calculated by multiplying organic carbon content by 1.724
MWHC = Maximum Water Holding Capacity
BIO⁻ samples were untreated
BIO⁺ samples were treated with 400 µL of methanol/water (1:1 v/v)

B. Study Design

1. Experimental Conditions

Tests were performed in static systems consisting of an Erlenmeyer flask containing 100 g soil (dry weight equivalents) fitted with a trap attachment (permeable for oxygen) containing soda lime for absorption of carbon dioxide and a polyurethane foam plug for adsorption of volatile organic compounds.

The tests were performed at a concentration of approximately 0.44 mg/kg dry weight of soil. The test item [phenyl-U-^{14}C]-fluopicolide dissolved in methanol/water (1:1, v/v) (400 µL) was applied drop wise onto the soil surface. Soil samples were adjusted to a moisture content equivalent to 54.3% of maximum water holding capacity, four days prior to application. The samples were incubated at 20 ± 1 °C under aerobic conditions in the dark for 126 days. Soil moisture was maintained during incubation by addition of de-ionized water after 29, 70 and 98 days of incubation. No significant losses of moisture were observed throughout the study.

2. Sampling

Duplicate samples were removed for analysis after 0, 2, 7, 10, 14, 28, 57, 85 and 126 days of incubation. Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment (DAT 0, 57 and 126).

3. Analytical Procedures

Soil samples were first shaken with 0.01M calcium chloride solution for 24 hours at 20 °C on an overhead shaker at 16 rpm to determine the desorbable portion of the test item from aged soil. A soil-to-solution ratio of 1:3 was used for all soils. Soil samples were then extracted three times with acetonitrile/water (4/1, *v/v*) at ambient temperature followed by a microwave extraction with acetonitrile/water (4/1, v/v) at 70 °C. After each extraction step, extract and soil were separated by centrifugation.

Radioactivity in extracts was determined by liquid scintillation counting (LSC). Desorption supernatants were analysed directly by HPLC with radiodetection. Ambient and microwave soil extracts were pooled and concentrated prior to analysis by HPLC. The maximum HPLC LOD was determined as 0.5%AR. The concentration procedure for soil extracts was established as quantitative (recovery 96.4%). HPLC column recovery was also quantitative (recovery 101.9 to 103.3%). The maximum HPLC LOD was determined as 1.3%AR. The primary chromatographic method for analysis of soil extracts was a reverse phase C18 HPLC method. Selected extracts were analysed by a second confirmatory phenyl-hexyl phase HPLC method. Selected desorption supernatants (DAT-57) were concentrated and the radiopeak corresponding to the major degradation product isolated, prior to analysis by LC/MS/MS for identification of M-01.

With the exception of the time zero samples, trap attachments were removed for analysis at each sampling time. Soda lime from the trap attachment was transferred into an Erlenmeyer flask, aqueous hydrochloric acid (18%) added dropwise and any liberated carbon dioxide collected in trapping vessels containing scintillation cocktail. The polyurethane foam plug was extracted with ethyl acetate for approximately 5 minutes in an ultrasonic bath to desorb any volatile organic compounds. The radioactivity content of these samples was determined by LSC.

The polyurethane foam plug was extracted with ethyl acetate for approximately 5 minutes in an ultrasonic bath to desorb any volatile organic compounds. The radioactivity content was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

A test of the extraction efficiency using a simplified extraction method, suitable for terrestrial field dissipation samples, was performed with spare samples of all soils. Duplicate samples of each soil were processed at DAT-126 and single samples at DAT-130. At DAT-126 soil samples were extracted for 15 minutes by microwave extraction with acetonitrile/water (4/1, *v/v*) at 70 °C. At DAT-130 soil samples were extracted for 3, 15 and 30 minutes by microwave extraction with acetonitrile/water (4/1, *v/v*) at 70 °C. After the extraction step, extract and soil were separated by centrifugation. Radioactivity in extracts was determined by LSC and the microwave soil extracts were concentrated prior to analysis by HPLC. The extraction efficiency of the simplified and standard (exhaustive) extraction methods for total extractable residues, fluopicolide and M-01 were shown to be comparable.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The degradation of fluopicolide was best described by the double first order in parallel (DFOP) model in all soils based on lowest chi^2 error values and visual assessments of fits.

Additionally, modelling endpoints for the degradation of fluopicolide and M-01 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each soil between the SFO and FOMC fits for fluopicolide. For all four soils, the FOMC model provided a better fit than the SFO model to the fluopicolide residues, and the DFOP model was therefore also fitted. For the Dollendorf II soil, the FOMC fit resulted in the lowest χ^2 err% value; this fit was not accepted, however, as extrapolation beyond the experimental period is not recommended for deriving robust DT$_{90}$ values using the FOMC model (EFSA, 2009). DFOP was instead selected as the most appropriate model to describe fluopicolide degradation in the Dollendorf II soil. The DFOP model provided the best visual fit to the fluopicolide residues in the other three soils.

Metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soils incubated at 20 °C following application of [phenyl-U-^{14}C]- fluopicolide are summarized in Table 7.1.2.1.1- 20 to Table 7.1.2.1.1- 23.

Table 7.1.2.1.1- 20: Degradation of [phenyl-U-^{14}C]-fluopicolide in L[redacted] soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	14	28	57	85	126
Fluopicolide	Mean	93.4	92.3	86.2	83.4	79.8	74.3	61.4	53.3	46.4
	SD	± 0.7	± 0.6	± 0.4	± 1.0	± 0.8	± 0.1	± 0.6	± 0.0	± 1.1
M-01 (AE C653711)	Mean	2.6	5.0	8.4	9.8	12.5	18.0	27.3	32.7	37.7
	SD	± 0.0	± 0.1	± 0.6	± 0.6	± 0.6	± 0.0	± 0.1	± 0.7	± 0.6
u3	Mean	1.5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD	± 0.1								
u4	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u5	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	4.2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD	± 0.1								
$CaCl_2$ solution	Mean	33.7	29.8	29.1	27.9	28.2	28.3	30.2	30.8	32.6
	SD	± 0.4	± 0.5	± 0.4	± 0.6	± 0.6	± 0.3	± 0.3	± 1.0	± 0.2
Ambient Extract	Mean	61.2	64.7	62.5	62.5	60.8	60.0	53.8	49.7	45.7
	SD	± 0.5	± 0.3	± 0.7	± 0.9	± 0.6	± 0.2	± 0.6	± 0.2	± 0.1
Microwave Extract	Mean	2.3	2.7	3.0	3.8	3.3	4.1	4.6	5.5	5.8
	SD	± 0.1	± 0.1	± 0.0	± 0.0	± 0.2	± 0.0	± 0.2	± 0.1	± 0.2
Total Extractable Residues	Mean	97.8	97.2	94.7	93.4	92.3	92.3	88.6	86.0	84.1
	SD	± 0.8	± 0.7	± 1.0	± 0.4	± 0.2	± 0.0	± 0.5	± 0.7	± 0.5
Carbon Dioxide	Mean	n.a.	0.8	1.1	1.2	1.2	1.7	2.7	1.8	4.9
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 1.8	± 0.0
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	1.6	2.6	2.9	4.0	3.7	5.3	7.0	9.6	11.7
	SD	± 0.1	± 0.0	± 0.1	± 0.4	± 0.0	± 0.0	± 0.1	± 0.7	± 0.1
Total Recovery	Mean	99.3	100.6	98.6	98.5	97.2	99.4	98.4	97.4	100.7
	SD	± 0.8	± 0.7	± 1.2	± 0.0	± 0.1	± 0.0	± 0.6	± 1.9	± 0.4

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 21: Degradation of [phenyl-U-^{14}C]-fluopicolide in Dollendorf II soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	14	28	57	85	126
Fluopicolide	Mean	84.3	90.6	83.8	81.5	78.9	71.9	64.4	51.6	45.7
	SD	± 0.7	± 0.0	± 0.2	± 0.9	± 0.2	± 0.3	± 2.2	± 1.3	± 0.4
M-01 (AE C653711)	Mean	2.3	5.8	8.1	9.5	10.8	16.3	22.4	28.5	32.0
	SD	± 0.2	± 0.3	± 0.2	± 0.3	± 0.2	± 0.4	± 0.5	± 0.3	± 0.3
u3	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u4	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u5	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	3.1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD	± 0.6								
$CaCl_2$ solution	Mean	13.7	14.0	14.0	14.5	15.0	16.0	18.1	20.2	20.0
	SD	± 0.0	± 0.0	± 0.3	± 0.1	± 0.4	± 0.2	± 0.4	± 0.3	± 0.2
Ambient Extract	Mean	68.4	78.0	72.2	72.4	70.4	64.2	52.3	50.8	51.6
	SD	± 1.6	± 0.4	± 0.4	± 0.4	± 0.4	± 0.8	± 4.4	± 4.5	± 0.9
Microwave Extract	Mean	5.7	4.4	5.7	4.1	4.3	8.0	6.3	9.1	6.2
	SD	± 0.5	± 0.2	± 0.6	± 0.7	± 0.2	± 0.0	± 1.3	± 2.7	± 0.1
Total Extractable Residues	Mean	87.8	96.4	91.9	91.0	89.7	88.2	76.8	80.1	77.7
	SD	± 1.3	± 0.4	± 0.4	± 1.2	± 0.0	± 0.6	± 2.7	± 1.6	± 1.1
Carbon Dioxide	Mean	n.a.	0.6	1.0	1.3	1.2	2.1	4.0	5.7	8.9
	SD		± 0.0	± 0.0	± 0.0	± 0.1	± 0.1	± 0.0	± 0.2	± 0.3
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	9.2	4.2	5.6	6.3	5.9	9.0	17.6	14.0	14.5
	SD	± 1.0	± 0.2	± 0.3	± 0.4	± 0.1	± 0.1	± 2.9	± 1.5	± 0.2
Total Recovery	Mean	97.0	101.2	98.5	98.6	96.8	99.3	98.4	99.8	101.2
	SD	± 0.3	± 0.1	± 0.7	± 0.8	± 0.3	± 0.6	± 0.2	± 0.1	± 0.6

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues

Table 7.1.2.1.1- 22: Degradation of [phenyl-U-^{14}C]-fluopicolide in L[redacted] soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	14	28	57	85	126
Fluopicolide	Mean	93.8	90.3	76.2	72.6	67.9	57.3	45.4	38.3	31.3
	SD	± 0.4	± 0.2	± 0.4	± 1.1	± 0.9	± 0.7	± 0.0	± 0.3	± 0.1
M-01 (AE C653711)	Mean	2.3	5.1	12.9	15.5	20.4	31.8	39.1	45.7	47.4
	SD	± 0.1	± 0.2	± 0.6	± 0.7	± 0.0	± 0.2	± 0.5	± 0.3	± 0.6
u3	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u4	Mean	n.d.	2.9	4.4	4.6	3.5	1.7	2.6	2.6	2.6
	SD		± 0.1	± 0.1	± 0.0	± 0.7	± 0.1	± 0.1	± 0.1	± 0.3
u5	Mean	< LOD	< LOD	< LOD	n.d.	< LOD	n.d.	< LOD	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	< LOD	2.9	4.4	4.6	3.5	1.7	2.6	2.6	2.6
	SD		± 0.1	± 0.1	± 0.0	± 0.7	± 0.1	± 0.1	± 0.1	± 0.3
$CaCl_2$ solution	Mean	26.4	25.7	25.8	27.0	27.3	29.1	31.1	32.9	32.6
	SD	± 0.1	± 0.3	± 0.0	± 0.1	± 0.0	± 0.1	± 0.5	± 0.4	± 0.1
Ambient Extract	Mean	67.5	69.4	63.8	61.5	60.0	55.8	49.5	45.2	40.7
	SD	± 0.3	± 0.1	± 0.1	± 0.5	± 0.1	± 0.7	± 0.1	± 0.1	± 0.1
Microwave Extract	Mean	2.8	3.4	4.2	4.2	4.9	6.0	6.7	8.5	8.0
	SD	± 0.1	± 0.1	± 0.3	± 0.1	± 0.2	± 0.1	± 0.1	± 0.1	± 0.1
Total Extractable Residues	Mean	96.8	98.5	93.9	92.2	92.1	90.9	87.3	86.6	81.3
	SD	± 0.6	± 0.4	± 0.1	± 0.4	± 0.2	± 0.9	± 0.6	± 0.5	± 0.2
Carbon Dioxide	Mean	n.a.	0.8	1.1	1.0	1.3	1.9	3.0	4.1	5.5
	SD		± 0.0	± 0.0	± 0.1	± 0.0	± 0.0	± 0.1	± 0.0	± 0.1
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	2.2	3.1	4.3	4.9	5.0	7.4	9.7	12.2	13.7
	SD	± 0.0	± 0.3	± 0.1	± 0.0	± 0.1	± 0.1	± 0.2	± 0.3	± 0.4
Total Recovery	Mean	98.9	102.4	99.3	98.5	98.5	100.2	100.0	102.8	100.6
	SD	± 0.6	± 0.2	± 0.1	± 0.5	± 0.1	± 0.8	± 0.5	± 0.2	± 0.3

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 23: Degradation of [phenyl-U-^{14}C]-fluopicolide in H[redacted] soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	14	28	57	85	126
Fluopicolide	Mean	92.0	90.2	81.0	79.1	75.2	69.2	58.3	50.9	43.3
	SD	± 0.3	± 0.8	± 0.3	± 0.1	± 0.1	± 0.1	± 0.9	± 0.6	± 0.1
M-01 (AE C653711)	Mean	2.6	6.6	12.5	14.4	16.6	23.0	32.2	38.0	43.5
	SD	± 0.2	± 0.1	± 0.1	± 0.2	± 0.2	± 0.1	± 0.5	± 0.4	± 0.0
u3	Mean	< LOD	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u4	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u5	Mean	< LOD	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	3.4	3.2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD	± 0.5	± 0.0							
$CaCl_2$ solution	Mean	23.1	21.4	22.5	23.2	22.5	23.3	26.0	27.5	28.9
	SD	± 0.5	± 0.0	± 0.2	± 0.1	± 0.3	± 0.3	± 0.4	± 0.3	± 0.0
Ambient Extract	Mean	69.4	71.9	67	65.5	64.5	62.9	58.1	53.3	49.8
	SD	± 0.1	± 0.6	± 0.5	± 0.3	± 0.3	± 0.4	± 1.0	± 0.4	± 0.1
Microwave Extract	Mean	3.2	3.8	4.0	4.7	4.7	6.0	6.4	8.1	8.0
	SD	± 0.1	± 0.1	± 0.0	± 0.0	± 0.0	± 0.0	± 0.1	± 0.0	± 0.0
Total Extractable Residues	Mean	95.7	97.0	93.5	93.5	91.7	92.2	90.5	88.9	86.7
	SD	± 0.8	± 0.9	± 0.5	± 0.3	± 0.1	± 0.0	± 1.3	± 0.2	± 0.1
Carbon Dioxide	Mean	n.a.	0.7	1.0	1.3	1.2	1.6	2.5	3.2	4.5
	SD		± 0.0	± 0.0	± 0.2	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	2.4	3.2	3.9	4.2	4.7	6.2	8.3	9.8	11.1
	SD	± 0.0	± 0.0	± 0.2	± 0.1	± 0.1	± 0.2	± 0.2	± 0.2	± 0.0
Total Recovery	Mean	98.1	101.0	98.5	99.0	97.6	100.2	101.3	102.0	102.3
	SD	± 0.8	± 0.9	± 0.4	± 0.4	± 0.1	± 0.3	± 1.5	± 0.0	± 0.2

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues

B. Material Balance

Mean mass balances were 98.9% AR for L[redacted] soil (range from 97.2 to 100.7% AR), 99.0% AR for Dollendorf soil (range from 96.8 to 101.2% AR), 100.1% AR for L[redacted] soil (range from 98.5 to 102.8% AR) and 100.0% AR for H[redacted] soil (range from 97.6 to 102.3% AR).

The results confirm there were no significant losses of radioactivity during sample processing.

C. Extractable and Non-Extractable Residues

Desorbable residues in aqueous 0.01 M $CaCl_2$ solution ranged from 33.7 to 27.9% AR L[redacted] soil over the course of the study (mean 30.1 % AR), initially declining from 33.7 % AR at DAT-0 to 27.9% AR at DAT-10 and then increased to 32.6% AR at DAT-126. In Dollendorf and L[redacted] soils desorbable residues increased from DAT-0 to DAT-85 from 13.7 to 20.2% AR and from 26.4 to 32.9% AR, respectively, and remained constant until the end of the study (DAT-126). Desorbable residues in H[redacted] soil ranged between 21.4 and 23.3% AR from DAT-0 to DAT-28 and increased to 28.9% AR at DAT-126.

Total extractable residues (i.e. residues desorbed by aqueous 0.01 M $CaCl_2$ solution and residues in organic soil extracts) decreased from DAT-0 to DAT-126 from 97.8 to 84.1% AR in L[redacted] soil, from 87.8 to 77.7% AR in Dollendorf soil, from 96.8 to 81.3% AR in L[redacted] soil and from 95.7 to 86.7% AR in H[redacted] soil.

Non-extractable soil residues increased concurrently with the decrease in extractable radioactivity in all soils. Non-extractable residues (NER) increased from DAT-0 to DAT-126 from 1.6 to 11.7% AR in L[redacted] soil, from 9.2 to 14.5% AR in Dollendorf soil, from 2.2 to 13.7% AR in L[redacted] soil and from 2.4 to 11.1% AR in H[redacted] soil.

D. Volatile Radioactivity

The maximum amount of carbon dioxide formed was 4.9, 8.9, 5.5 and 4.5% AR in L[redacted], Dollendorf, L[redacted] and H[redacted] soils, respectively by the end of the study (DAT-126). Formation of volatile organic compounds (VOC) was insignificant with values of ≤ 0.1% AR at all timepoints in all soils.

E. Degradation of Parent Compound

The amount of fluopicolide in the total soil extracts (i.e. in aqueous desorption solution and organic soil extracts) decreased from 93.4 at DAT-0 to 46.4% AR at DAT-126 in L[redacted] soil, from 84.3 to 45.7% AR in Dollendorf soil, from 93.8 to 51.3% AR in L[redacted] soil and from 92.0 to 43.3% AR in H[redacted] soil.

Degradation of fluopicolide was accompanied by the formation of one degradation product, M-01 (AE C653711) which was observed at a maximum of 47.4% AR at DAT-126 in L[redacted] soil. M-01 was identified by co-chromatography with an analytical standard and by LC/MS/MS after isolation of the radiopeak from desorption solutions. The total unidentified residues amounted to a maximum of 4.6% AR and no single component exceeded 4.6% AR at any sampling interval in any soil.

F. Degradation Kinetics

Reported DT_{50} values of fluopicolide under aerobic conditions were 107, 112, 48.0 and 96.4 days in [redacted] Dollendorf, L[redacted] and H[redacted] soils, respectively. The experimental data was best described by a double first order in parallel (DFOP) kinetic model. Details are provided below in Table 7.1.2.1.1- 24.

Table 7.1.2.1.1- 24: Reported degradation rate of fluopicolide under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} (days)	DT_{90} (days)
L[redacted]	DFOP	98.42	k1 1.477e-01 k2 4.960e-03 g 1.511e-01	1.4	0.00114 5.44e-11 - -	- - 1.146e-01	- - 0.188	107	431
Dollendorf II	DFOP	94.52	k1 0.034062 k2 0.001829 g 0.397855	2.3	0.00638 0.15099	- - 0.150830	- - 0.645	112	982
L[redacted]	DFOP	98.36	k1 1.165e-01 k2 6.188e-03 g 3.287e-01	1.2	3.65e-08 2.65e-11	- - 2.948e-01	- - 0.363	48.0	308
H[redacted]	DFOP	97.65	k1 1.901e-01 k2 5.010e-03 g 1.897e-01	1.0	4.78e-06 1.41e-13	- - 1.680e-01	- - 0.211	96.4	418

In addition, the experimental data for the degradation of fluopicolide and M-01 has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting DT_{50} values for trigger endpoints for fluopicolide are summarised below in Table 7.1.2.1.1- 25. Best fit kinetics are highlighted in bold. The results are very similar to reported best fit DT_{50} values.

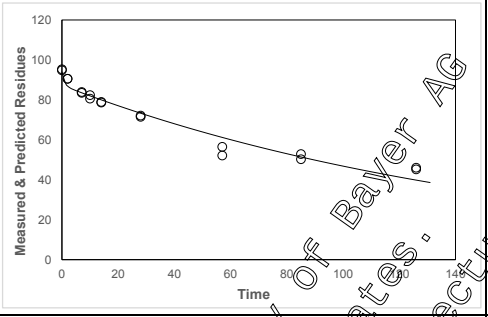
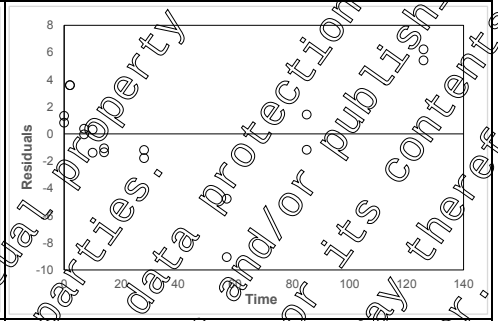
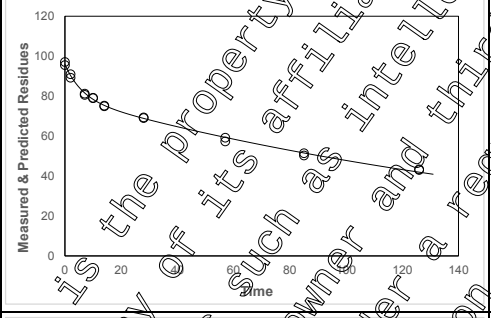
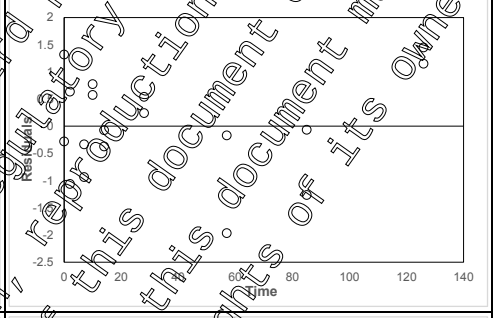
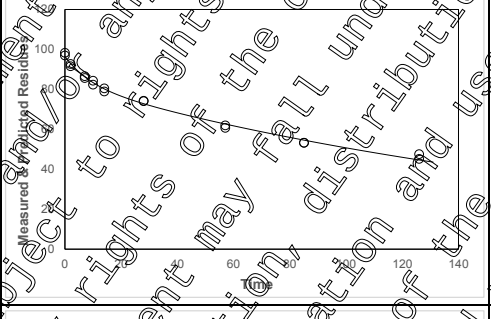
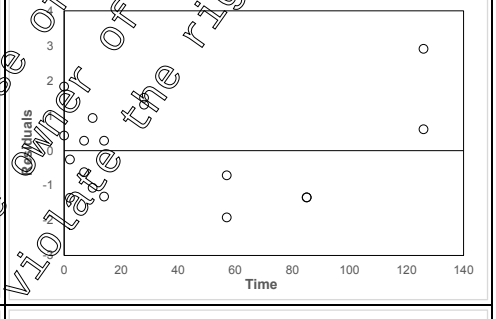
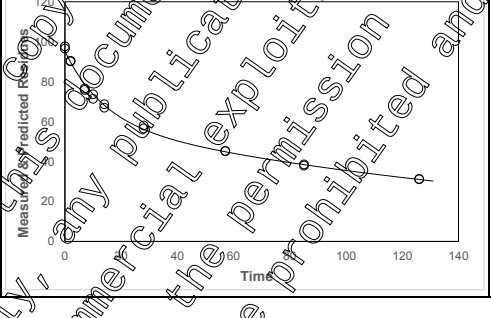
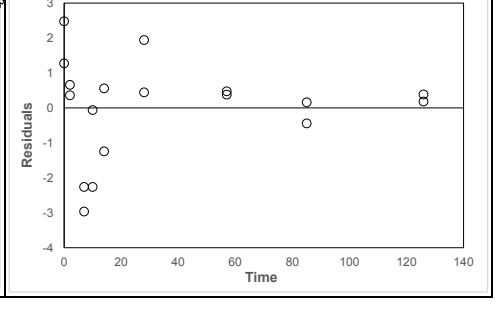
Table 7.1.2.1.1- 25: Re-evaluated degradation rate of fluopicolide under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Dollendorf, [redacted] (2016b)	SFO	90.88	k 0.007374	4.95	1.94E-13	0.006143	0.009	94	312.2
	FOMC	95.01	α 0.3622 β 19	1.95	n.r. n.r.	0.2842 10.62	0.44 27.38	109.8	>10000
	DFOP	**94.06**	**k1 1.097 k2 0.006222 g 0.07153**	**4.13**	**0.00456 <2e-16 n.r.**	**0.3256 0.005461 0.05601**	**1.868 0.007 0.087**	**99.5**	**358.1**
[redacted] (2016b)	SFO	94.64	k 0.01008	8.95	5.96E-09	0.007478	0.013	68.8	228.4
	FOMC	95.38	α 0.2929 β 11.39	1.97	n.r. n.r.	0.2494 7.52	0.336 15.26	110	>10000
	DFOP	**95.88**	**k1 0.1652 k2 0.005033 g 0.1751**	**0.906**	**2.17E-10 <2e-16 n.r.**	**0.1295 0.004696 0.1545**	**0.201 0.005 0.196**	**99.5**	**419.3**
L[redacted] (2016b)	SFO	94.88	k 0.00787	5.27	5.15E-13	0.006506	0.009	88.1	292.6
	FOMC	96.43	α 0.3706 β 22.33	1.54	n.r. n.r.	0.3039 14.43	0.437 30.24	122.6	>10000
	DFOP	**96.57**	**k1 0.1103 k2 0.004904 g 0.1415**	**1.32**	**3.59E-06 <2e-16 n.r.**	**0.0704 0.004362 0.1041**	**0.15 0.005 0.179**	**110.2**	**438.4**
[redacted] (2016b)	SFO	89.2	k 0.01235	8.69	1.76E-09	0.009337	0.015	56.1	186.5
	FOMC	95.96	α 0.4526 β 12.5	1.49	n.r. n.r.	0.4098 10.02	0.495 14.97	45.3	2013
	DFOP	**95.32**	**k1 0.07342 k2 0.005194 g 0.374**	**1.74**	**9.14E-13 7.08E-13 n.r.**	**0.06062 0.004297 0.3268**	**0.086 0.006 0.421**	**47.7**	**353.2**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.1.1- 26: Degradation of fluopicolide under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Dollendorf DFOP [redacted] (2016b)		
[redacted] DFOP [redacted] (2016b)		
L[redacted] DFOP [redacted] (2016b)		
[redacted] DFOP [redacted] (2016b)		

G: Time-Dependent Sorption

The sorption of fluopicolide in soil increased steadily with time. Apparent sorption coefficients ($K_{d, app}$) increased from DAT-0 to DAT-126 in all four soils. The values are summarised in Table 7.1.2.1.1-27.

Table 7.1.2.1.1- 27: Apparent sorption coefficients ($K_{d, app}$) (expressed as mL/g)

DAT	Mean SD	L [redacted]	Dollendorf	L [redacted]	[redacted]
0	Mean	5.69	16.19	7.02	8.7
	SD	± 0.00	± 0.00	± 0.01	± 0.01
2	Mean	6.52	20.66	7.9	10.21
	SD	± 0.00	± 0.00	± 0.00	± 0.02
7	Mean	7.69	20.67	9.33	11.61
	SD	± 0.02	± 0.00	± 0.01	± 0.03
10	Mean	8.13	21.47	10.08	12.44
	SD	± 0.02	± 0.01	± 0.01	± 0.01
14	Mean	8.63	21.12	10.49	13.65
	SD	± 0.01	± 0.06	± 0.03	± 0.01
28	Mean	10.21	23.18	12.1	14.64
	SD	± 0.02	± 0.02	± 0.04	± 0.01
57	Mean	11.74	22.98	14.47	18.32
	SD	± 0.03	± 0.10	± 0.04	± 0.02
85	Mean	13.32	26.41	16.78	20.89
	SD	± 0.07	± 0.07	± 0.02	± 0.02
126	Mean	14.62	31.76	20.36	25.97
	SD	± 0.05	± 0.07	± 0.03	± 0.03
Factor [A]		2.57	1.96	2.90	2.98
Mean Factor		2.60			

Apparent Sorption Coefficients ($K_{d, app}$) are called Time-Dependent Sorption Ratios (R_{TDS}) in the report.
[A] Calculated as $K_{d, app}$ DAT-126 divided by $K_{d, app}$ DAT-0

The results are more fully discussed under Section 7.1.3.2 (see KCA 7.1.3.2/03).

III. Conclusion

Fluopicolide was moderately degraded and mineralized in four Germans soils; L [redacted], Dollendorf, L [redacted] and L [redacted], under aerobic conditions at 20°C in the dark. Reported best fit DT_{50} values ranged from 48 to 112 days in the tested soils. Re-evaluated best fit DT_{50} values were very similar, ranging from 47.7 to 110.2 days.

The primary objective of the study was to investigate the sorption of fluopicolide, determined under equilibrium conditions, following its aging in soil under aerobic conditions in the dark under laboratory conditions. The time-dependent sorption ratio increased throughout the incubation period (126 days) by a factor of 1.96 to 2.98 in the four soils tested.

Formation of carbon dioxide was significant (up to 8.9% AR) by the end of the study indicating the potential for complete mineralization of fluopicolide and its degradation products. One major degradation product, M-01 (AE C653711), was identified with a maximum of 47.4% AR.

Formation of non-extractable residues (NER) was up to 14.5% AR at study end, which is an indication for biotic degradation of fluopicolide.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002), US EPA 835.4100 / 835.4200 (2008) and in parts, where relevant, to OECD 106 (2000). The study is considered valid to assess the aerobic degradation of [phenyl-U-^{14}C]-fluopicolide in soil.

The study is valid to assess the changes in sorption of fluopicolide with time in accordance with guidance provided by Food and Environment Research Agency (2019) on conducting aged sorption studies. A kinetic assessment of the time dependent sorption (TDS) parameters is provided in KCA 7.1.3.2.

Data Point:	KCA 7.1.2.1.1/08
Report Author:	[redacted]
Report Year:	2016
Report Title:	[Phenyl-UL-14C]Fluopicolide: Degradation and time- dependent sorption in 6 soils from field dissipation trials
Report No:	EnSa-15-0510
Document No:	M-550687-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307 US EPA OPPTS Test Guideline No. 835.4100, 835.4200 OECD Test Guideline No. 106 (only in parts) Guidance on how aged sorption studies for pesticides should be conducted, analysed and used in the regulatory process; Food and Environmental Agency, York, UK, 2012
Deviations from current test guideline:	Study: none; Analytical methods part: none
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation and time-dependence of sorption of fluopicolide was studied in six soils under aerobic conditions in the laboratory in the dark at 20 ± 1 °C and 53.9% of the maximum water holding capacity for 120 days. In addition, the rate of degradation of fluopicolide was determined in the study.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
H[redacted]	Silt loam	6.1	0.7
Great Chishill	Clay	7.3	2.1
Parcey Meslay	Loam	6.7	1.3
Mas du Coq	Clay loam	7.6	0.9
Albaro/Marcomcini	Silty clay	7.2	2.1
Vilobi d'Onyar	Sandy loam	6.3	0.8

[Phenyl-U-^{14}C]-labelled fluopicolide was applied to soil samples at an application rate of 0.44 mg/kg dry weight. The radiochemical purity and specific activity were > 99% and 5.73 MBq/mg, respectively.

Samples were taken for extraction and analysis immediately after treatment (day 0) and after 7, 10, 30, 44, 59, 91 and 120 days of incubation. Soil samples were first desorbed with 0.01M calcium chloride solution for 24 hours at 20 °C at a soil : solution ratio of 1:3 (w/w) to determine the desorbable portion of the test item from aged soil. The soil residue was then exhaustively extracted with three further successive extractions with acetonitrile/water 4/1 (v/v) at ambient temperature, followed by a microwave extraction with acetonitrile/water (4/1, *v/v*) at 70 °C. Desorption supernatants and concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC).

Recovery of radioactivity was quantitative throughout the study. Overall mean mass balances were 99.9% AR for H[redacted] soil, 99.4% AR for Great Chishill soil, 99.3% AR for Parcey Meslay soil, 98.9% AR for Mas du Coq soil, 99.6% AR for Albaro soil and 99.4% AR for Vilobi soil.

Desorbable residues in aqueous 0.01 M calcium chloride solution were 41.7, 20.6, 32.2, 33.5, 35.4 and 48.0% AR in H[redacted] Chishill, Parcey Meslay, Mas du Coq, Albaro and Vilobi soils, respectively by the end of the study (DAT-120). Total extractable residues (i.e. residues desorbed by aqueous 0.01 M $CaCl_2$ solution and residues in organic soil extracts) decreased from 98.7% at DAT-0 to 89.2% AR by DAT-120 in H[redacted] soil, from 96.7 to 86.5% AR in Chishill soil, from 97.3 to 89.5% AR in soil FR09B, from 97.2 to 86.5% AR in Parcey Meslay soil, from 96.1 to 85.8% AR in Albaro soil and from 96.7 to 85.2% AR in Vilobi soil.

Non-extractable soil residues (NER) increased concurrently with the decrease in extractable radioactivity in all soils, reaching maxima of 8.9, 8.3, 9.6, 10.0% AR in H[redacted], Parcey Meslay, Mas du Coq and Vilobi soils at DAT-120. In Great Chishill and Albaro soils NER levels reached maxima of 12.6% and 8.8% AR at DAT 91, before declining slightly to 10.6% and 8.6% AR by DAT-120.

The maximum amount of carbon dioxide formed was 2.0, 1.7, 1.9, 3.0, 4.6 and 3.8% AR in the six soils by the end of the study (DAT-120). No significant levels of organic volatiles were observed (≤ 0.1% AR).

After 120 days incubation at 20 °C, fluopicolide degraded to 46.2, 66.5, 57.9, 59.8, 52.6 and 30.2% of the applied radioactivity in the six soils. M-01 (AE C653711) was also observed in all soils, increasing steadily to maxima of 43.0, 20.0, 31.5, 26.7, 33.2 and 55.0% AR by the end of the study. It was identified by LC/MS/MS after isolation from a concentrated desorption solution. No other degradation products were detected (LOD 1.3% AR).

The effect of aged sorption to soil was determined for fluopicolide and showed a significant increase with time. Apparent sorption coefficients ($K_{d, app}$) increased with time in all soils by a factor of 1.93 to 3.12 (mean 2.49). Further details specific to the aged sorption of fluopicolide are provided in Section 7.1.3.2.

Degradation kinetics for fluopicolide provided in the report were conducted in accordance with FOCUS guidance document on degradation kinetics (2014). The best-fit DT_{50} values were 101, 251, 167, 180, 131 and 54.7days in H[redacted], Chishill, Parcey Meslay, Mas du Coq, Albaro and Vilobi soils, respectively. A re-evaluation of the degradation kinetics resulted in similar best-fit un-normalised DT_{50} values of 54.9 to 250.7 days.

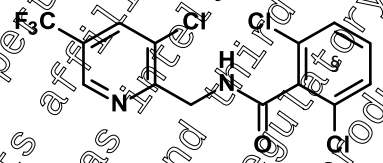
Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)	Visual Assessment
H[redacted] (silt loam)	DFOP	101	471	1.20	Good
Great Chishill (clay)	DFOP	251	991	0.65	Good
Parcey Meslay (loam)	DFOP	167	643	0.70	Good
Mas du Coq (clay loam)	DFOP	180	673	0.53	Good
Albaro (silty clay)	DFOP	131	481	0.78	Good
Vilobi d'Onyar (sandy loam)	DFOP	54.7	277	0.72	Good

I. Materials and Methods

A. Materials

1. Test Items

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



§ Denotes position of [^{14}C]-radiolabel

Specific Activity: 5.73 MBq/mg

Radiochemical Purity: >99% (TLC & HPLC)

2. Test Soil

The study was performed using six European soils from terrestrial field dissipation sites used for fluopicolide (five sites) and its metabolite M-01 (all six sites) as characterized in Table 7.1.2.1.1-28. The same batches of all six soils were also used in OECD 106 adsorption desorption studies (see KCA 7.1.3.1.1/04).

Table 7.1.2.1.1- 28: Physico-chemical properties of test soils

Parameter		Soil					
Soil Designation		[redacted]		Great Ghishill		Parcey Meslay	
Soil ID		VG08		ENG2		FR09B	
Geographic Location							
City		Burscheid,		Cambridgeshire,		Centre-Val de Loire	
Country		Germany		England, UK		France	
Batch Number		20141121		20141125		20141124	
Textural Classification (USDA)		Silt loam		Clay		Loam	
Sand [50 - 2000 µm]	(%)	19		35		31	
Silt [2 – 50 µm]	(%)	57		23		49	
Clay [< 2 µm]	(%)	24		42		20	
pH							
in $CaCl_2$ (1:1)		6.1		7.5		6.7	
in H_2O (1:1)		6.2		7.5		7.0	
Saturated paste		6.4		7.3		7.7	
in KCl (1:1)		5.6		6.9		6.5	
Organic Matter (%) *		1.2		3.6		2.2	
Organic Carbon (%)		0.7		2.1		1.3	
Cation Exchange Capacity (meq/100 g)		11		27.2		10.7	
Water Holding Capacity							
Maximum (g H_2O per 100 g DW)		54.1		65.3		58.0	
at 1/10 bar (%)		26.7		34.7		23.9	
Moisture Content During Incubation (%)		53.9% MWHC		53.9% MWHC		53.9% MWHC	
Bulk Density (disturbed, g/cm³)		1.08		1.12		1.09	
Soil Microbial Biomass (µg microbial C /g soil)		BIO⁻	BIO⁺	BIO⁻	BIO⁺	BIO⁻	BIO⁺
Initial (Day 1)		222		907		433	
Mid (Day 67)		154	184	697	675	258	233
Final (Day 121/123)		161	154	657	623	250	233

BIO⁻ samples were untreated
BIO⁺ samples were treated with 400 µL of methanol/water (1:1 v/v)

Parameter		Soil		
Soil Designation		Mas de Coq	Albaro/Marcomcini	Vilobi d'Onyar
Soil ID		FR08	IT09	SPA1
Geographic Location City Country		St Etienne du Gres, France	Albaro, Ronco Alladige Italy	Vilobi d'Onyar Catalonia Spain
Batch Number		20141121	20141124	20141125
Textural Classification (USDA)		Clay loam	Silty clay	Sandy loam
Sand [50 - 2000 µm]	(%)	25	17	57
Silt [2 – 50 µm]	(%)	43	41	31
Clay [< 2 µm]	(%)	32	42	12
pH				
in $CaCl_2$ (1:1)		7.6	7.2	6.3
in H_2O (1:1)		7.8	7.4	6.5
Saturated paste		7.7	7.4	6.6
in KCl (1:1)		7.4	6.9	5.9
Organic Matter (%) *		1.6	3.6	1.4
Organic Carbon (%)		0.9	2.1	0.8
Cation Exchange Capacity (meq/100 g)		11.2	20.5	8.7
Water Holding Capacity				
Maximum (g H_2O per 100 g DW)		46.5	68.1	43.8
at 1/10 bar (%)		29.3	36.3	18.3
Moisture Content During Incubation (%)		53.9% MWHC	53.9% MWHC	53.9% MWHC
Bulk Density (disturbed, g/cm³)		1.15	1.00	1.16
Soil Microbial Biomass (µg microbial C /g soil)		BIO^- BIO^+	BIO^- BIO^+	BIO^- BIO^+
Initial (Day 1)		486	578	339
Final (Day 121/123)		359 336	491 483	204 186

BIO^- samples were untreated
BIO^+ samples were treated with 400 µL of methanol/water (1:1 v/v)

B. Study Design

1. Experimental Conditions

Tests were performed in static systems consisting of an Erlenmeyer flask containing 100 g soil (dry weight equivalents) fitted with a trap attachment (permeable for oxygen) containing soda lime for absorption of carbon dioxide and a polyurethane foam plug for adsorption of volatile organic compounds.

The tests were performed at a concentration of approximately 0.44 mg/kg dry weight of soil. The test item [phenyl-U-^{14}C]-fluopicolide dissolved in methanol/water (1:1, v/v) (400 µL) was applied drop wise onto the soil surface. Soil samples were adjusted to a moisture content equivalent to 53.9% of maximum water holding capacity, three days prior to application. The samples were incubated at 20 ± 1 °C under aerobic conditions in the dark for 120 days. Soil moisture was maintained during incubation by addition of de-ionized water after 30, 63 and 91 days of incubation. No significant losses of moisture were observed throughout the study.

2. Sampling

Duplicate samples were removed for analysis after 0, 2, 7, 10, 30, 44, 59, 91 and 120 days of incubation. Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment (Day 1, Day 67 and Day 121/123) for H[redacted], Great Chishill and Parcey Meslay soils and at start and end of the study (Day 1 and Day 121/123) for Mas de Coq, Albaro and Vilobi d'Onyar soils.

3. Analytical Procedures

Soil samples were first shaken with 0.01M calcium chloride solution for 24 hours at 20 °C on an overhead shaker at 16 rpm to determine the desorbable portion of the test item from aged soil. A soil-to-solution ratio of 1:3 was used for all soils. Soil samples were then extracted three times with acetonitrile/water (4/1, *v/v*) at ambient temperature followed by a microwave extraction with acetonitrile/water (4/1, *v/v*) at 70 °C. After each extraction step, extract and soil were separated by centrifugation.

Radioactivity in extracts was determined by liquid scintillation counting (LSC). Desorption supernatants were analysed directly by HPLC with radiodetection. Ambient and microwave soil extracts were pooled and concentrated prior to analysis by HPLC. The concentration procedure for soil extracts was established as quantitative (recovery 96.3%). HPLC column recovery was also quantitative (recovery 101.1%). The maximum HPLC LOD was determined as 1.3%AR. The primary chromatographic method for analysis of soil extracts was a reverse phase C18 HPLC method. Selected extracts were analysed by a second confirmatory phenyl-hexyl phase HPLC method. A selected desorption supernatant (DAT-59, H[redacted] soil) was concentrated and the radiopeak corresponding to the major degradation product isolated, prior to analysis by LC/MS/MS for identification of M-01.

With the exception of the time zero samples, trap attachments were removed for analysis at each sampling time. Soda lime from the trap attachment was transferred into an Erlenmeyer flask, aqueous hydrochloric acid (18%) added dropwise and any liberated carbon dioxide collected in trapping vessels containing scintillation cocktail. The polyurethane foam plug was extracted with ethyl acetate for approximately 5 minutes in an ultrasonic bath to desorb any volatile organic compounds. The radioactivity content of these samples was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

M-15 (AE 1413903) was included as an analytical standard to determine its retention time in the HPLC method (35 minutes). Using this retention time, it was possible to establish that M-15 did not occur at any sampling interval in any of the soils.

A test of the extraction efficiency using a simplified extraction method, suitable for terrestrial field dissipation samples, was performed with spare samples of all soils. Duplicate samples of each soil were processed at DAT-140. Soil samples were extracted for 15 minutes by microwave extraction with acetonitrile/water (4/1, *v/v*) at 70 °C. After the extraction step, extract and soil were separated by centrifugation. Radioactivity in extracts was determined by LSC and the microwave soil extracts were concentrated prior to analysis by HPLC. The extraction efficiency of the simplified and standard (exhaustive) extraction methods for total extractable residues, fluopicolide and M-01 were shown to be comparable.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The degradation of fluopicolide was best described by the double first order in parallel (DFOP) model in all soils based on lowest chi error values and visual assessments of fits.

Additionally, modelling endpoints for the degradation of fluopicolide and M-01 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each soil between the SFO and FOMC fits for fluopicolide. For all six soils, the FOMC model provided a better fit than the SFO model to the fluopicolide residues, and the DFOP model was therefore also fitted. The DFOP model provided the best visual fit to the fluopicolide residues for all soils, with the lowest χ^2 err% value.

Metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soils incubated at 20 °C following application of [phenyl-U-^{14}C]- fluopicolide are summarized in Table 7.1.2.1.1- 29 to Table 7.1.2.1.1- 34.

Table 7.1.2.1.1- 29: Degradation of [phenyl-U-^{14}C]-fluopicolide in H[redacted] soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	30	44	59	91	120
Fluopicolide	Mean	97	92.4	87.1	81.8	68.3	63.6	59.6	50.4	46.2
	SD	± 0.4	± 0.2	± 0.0	± 0.9	± 0.3	± 0.9	± 0.2	± 0.3	± 0.4
M-01 (AE C653711)	Mean	1.6	4.9	10.2	15.9	25.6	30.0	33.7	39.6	43.0
	SD	± 0.1	± 0.2	± 0.2	± 0.2	± 0.1	± 0.2	± 0.0	± 0.0	± 0.2
u3	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u4	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u5	Mean	nd	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
$CaCl_2$ solution	Mean	47.3	44.2	42.6	44.2	42.1	41.3	41.1	41.7	41.7
	SD	± 1.0	± 0.1	± 0.3	± 0.3	± 0.1	± 0.1	± 0.7	± 0.0	± 0.4
Ambient Extract	Mean	49.4	50.5	51.5	49.3	47.7	47.2	46.4	42.4	41.6
	SD	± 0.4	± 0.2	± 0.1	± 1.0	± 0.6	± 0.4	± 1.2	± 0.4	± 0.1
Microwave Extract	Mean	2.1	2.7	3.2	4.2	4.6	5.2	5.8	6.0	6.0
	SD	± 0.2	± 0.3	± 0.2	± 0.1	± 0.1	± 0.3	± 0.3	± 0.0	± 0.1
Total Extractable Residues	Mean	97.6	97.3	97.2	97.7	94.3	93.6	93.4	90	89.2
	SD	± 0.8	± 0.0	± 0.4	± 1.1	± 0.7	± 0.7	± 0.2	± 0.3	± 0.6
Carbon Dioxide	Mean	n.a.	0.1	0.2	0.2	0.4	0.6	0.9	1.5	2.0
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.1	± 0.0	± 0.0
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	1.1	2.1	2.3	2.8	5.5	5.2	6.2	7.6	8.9
	SD	± 0.1	± 0.3	± 0.1	± 0.0	± 0.5	± 0.2	± 0.5	± 0.2	± 0.2
Total Recovery	Mean	99.7	99.5	99.7	100.7	100.3	99.5	100.4	99.1	100.2
	SD	± 0.2	± 0.3	± 0.4	± 1.1	± 0.1	± 0.4	± 0.4	± 0.1	± 0.3

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 30: Degradation of [phenyl-U-^{14}C]-fluopicolide in Great Chishill soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	30	44	59	91	120
Fluopicolide	Mean	96.2	92.7	87.3	84.1	80.5	77.6	76.0	69.4	66.5
	SD	± 0.2	± 0.0	± 0.7	± 0.0	± 0.5	± 0.1	± 0.3	± 0.4	± 0.5
M-01 (AE C653711)	Mean	< LOD	2.2	7.3	4.5	8.7	11.1	13.6	18.6	20.0
	SD		± 0.2	± 0.1	± 0.1	± 0.1	± 0.1	± 0.1	± 1.3	± 0.3
u3	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u4	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u5	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
$CaCl_2$ solution	Mean	28.6	26.2	24.6	24.4	22.1	21.1	20.9	20.3	20.6
	SD	± 0.0	± 0.1	± 0.2	± 0.0	± 0.1	± 0.1	± 0.0	± 0.3	± 0.6
Ambient Extract	Mean	62.6	64.0	64.1	54.9	57.2	57.8	59.0	56.8	54.7
	SD	± 0.4	± 0.0	± 0.8	± 0.5	± 0.1	± 0.8	± 0.6	± 0.1	± 0.3
Microwave Extract	Mean	5.5	4.8	6.0	9.4	10.0	9.8	9.8	11.0	11.3
	SD	± 0.6	± 0.2	± 0.4	± 0.5	± 0.7	± 0.8	± 0.3	± 1.9	± 0.1
Total Extractable Residues	Mean	96.2	95.0	94.6	88.6	89.2	88.6	89.7	88.0	86.5
	SD	± 0.2	± 0.1	± 0.6	± 0.1	± 0.5	± 0.0	± 0.2	± 1.7	± 0.8
Carbon Dioxide	Mean	n.a.	0.1	0.2	0.1	0.4	0.5	0.8	1.2	1.7
	SD		± 0.0	± 0.0	± 0.1	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	2.8	4.4	4.8	9.4	9.9	9.3	8.6	12.6	10.6
	SD	± 0.0	± 0.1	± 0.6	± 0.8	± 1.1	± 0.4	± 0.1	± 2.2	± 0.3
Total Recovery	Mean	99.0	99.4	99.5	98.1	99.5	98.5	99.0	101.8	98.8
	SD	± 0.2	± 0.2	± 0.0	± 0.7	± 0.6	± 0.4	± 0.3	± 3.9	± 1.1

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 31: Degradation of [phenyl-U-^{14}C]-fluopicolide in Parcey Meslay soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	30	44	59	91	120
Fluopicolide	Mean	97.3	95.9	90.2	88.3	80.3	73.8	70.6	63.0	57.9
	SD	± 0.2	± 0.8	± 0.8	± 0.2	± 0.3	± 0.7	± 1.4	± 0.2	± 0.5
M-01 (AE C653711)	Mean	n.d.	3.1	6.0	7.5	15.1	19.0	22.2	27.8	31.5
	SD		± 0.0	± 0.5	± 0.6	± 0.1	± 0.7	± 0.2	± 0.6	± 1.0
u3	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u4	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u5	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
$CaCl_2$ solution	Mean	35.6	32.3	30.2	30.9	29.5	29.5	29.1	30.5	32.2
	SD	± 0.1	± 0.9	± 0.0	± 0.0	± 0.1	± 0.7	± 0.5	± 0.1	± 0.8
Ambient Extract	Mean	59.5	63.9	63.0	60.1	61.1	58.8	58.2	54.3	50.6
	SD	± 0 .3	± 0.2	± 0.3	± 0.6	± 0.3	± 0.6	± 0.7	± 0.8	± 0.3
Microwave Extract	Mean	2.3	2.9	3.2	4.9	4.8	4.5	5.5	6.1	6.7
	SD	± 0.2	± 0.3	± 0.1	± 0.4	± 0.2	± 0.0	± 0.4	± 0.0	± 0.1
Total Extractable Residues	Mean	97.3	99.0	96.3	95.8	95.4	92.7	92.7	90.8	89.5
	SD	± 0.2	± 0.8	± 0.3	± 0.2	± 0.3	± 1.4	± 1.6	± 0.9	± 0.5
Carbon Dioxide	Mean	n.a.	0.1	0.2	0.2	0.5	0.6	0.9	1.4	1.9
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.1	± 0.0	± 0.0
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	0.9	1.4	2.1	2.8	4.1	5.0	6.0	7.5	8.3
	SD	± 0.1	± 0.1	± 0.1	± 0.3	± 0.3	± 0.1	± 0.1	± 0.1	± 0.0
Total Recovery	Mean	98.2	100.5	98.6	98.7	99.9	98.4	99.7	99.7	99.6
	SD	± 0.3	± 0.8	± 0.4	± 0.0	± 0.0	± 1.2	± 1.4	± 0.8	± 0.5

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 32: Degradation of [phenyl-U-^{14}C]-fluopicolide in Mas du Coq soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	30	44	59	91	120
Fluopicolide	Mean	95.8	95.1	91.6	88.7	81.4	75.8	72.6	65.2	59.8
	SD	± 0.3	± 0.3	± 0.6	± 1.0	± 0.9	± 0.8	± 0.3	± 0.1	± 0.2
M-01 (AE C653711)	Mean	<LOD	2.6	4.5	5.6	11.6	16.1	18.7	23.3	26.7
	SD		± 0.1	± 0.3	± 0.1	± 0.0	± 0.2	± 0.4	± 0.5	± 0.4
u3	Mean	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u4	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u5	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	<LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
$CaCl_2$ solution	Mean	49.5	44.0	40.3	40.9	34.9	35.0	34.2	33.3	33.6
	SD	± 0.7	± 0.1	± 0.3	± 0.4	± 0.1	± 0.1	± 0.1	± 0.1	± 0.2
Ambient Extract	Mean	46.2	51.3	52.6	48.6	53.0	50.9	51.1	48.6	45.5
	SD	± 0.1	± 0.1	± 0.1	± 1.5	± 0.5	± 0.1	± 0.0	± 0.2	± 0.4
Microwave Extract	Mean	1.6	2.4	3.2	4.8	5.2	6.0	6.1	6.6	7.5
	SD	± 0.0	± 0.1	± 0.0	± 0.7	± 0.4	± 0.5	± 0.0	± 0.1	± 0.4
Total Extractable Residues	Mean	97.0	97.7	96.1	94.3	93	91.9	91.3	88.5	86.5
	SD	± 0.3	± 0.2	± 0.2	± 1.1	± 0.8	± 0.6	± 0.2	± 0.4	± 0.7
Carbon Dioxide	Mean	n.a.	0.1	0.2	0.2	0.6	0.9	1.4	2.2	3.0
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	0.7	1.6	2.5	4.3	4.9	6.5	6.5	8.2	9.6
	SD	± 0.0	± 0.1	± 0.1	± 0.8	± 0.1	± 0.1	± 0.1	± 0.0	± 0.1
Total Recovery	Mean	97.9	99.4	98.7	98.8	98.5	99.3	99.2	98.9	99.2
	SD	± 0.8	± 0.1	± 0.4	± 0.3	± 0.7	± 0.5	± 0.3	± 0.3	± 0.5

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues

Table 7.1.2.1.1- 33: Degradation of [phenyl-U-^{14}C]-fluopicolide in Albaro soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	30	44	59	91	120
Fluopicolide	Mean	95.2	94.8	87.6	87.5	78.4	73.7	67.9	58.4	52.6
	SD	± 1.3	± 1.0	± 0.8	± 0.6	± 0.7	± 0.4	± 0.7	± 0.3	± 0.4
M-01 (AE C653711)	Mean	< LOD	3.0	7.6	6.8	13.7	19.2	23.1	29.1	33.2
	SD		± 0.0	± 0.0	± 0.3	± 0.2	± 0.3	± 0.8	± 0.3	± 0.2
u3	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u4	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u5	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
$CaCl_2$ solution	Mean	35.4	33.9	32.3	33.1	32.3	33.5	34.0	35.1	35.4
	SD	± 0.2	± 0.1	± 0.0	± 0.3	± 0.3	± 0.1	± 0.4	± 0.3	± 0.2
Ambient Extract	Mean	56.6	60.1	59.7	54.1	53.0	55.0	52.2	47.9	45.2
	SD	± 0.7	± 0.4	± 0.4	± 0.8	± 1.2	± 0.3	± 0.4	± 0.1	± 0.4
Microwave Extract	Mean	4.1	4.0	3.3	7.1	6.8	4.5	4.9	4.6	5.2
	SD	± 0.0	± 0.4	± 0.4	± 0.3	± 0.5	± 0.2	± 0.6	± 0.5	± 0.3
Total Extractable Residues	Mean	95.2	97.9	95.2	94.3	92.1	92.9	91	87.5	85.8
	SD	± 1.3	± 1.0	± 0.8	± 0.3	± 0.9	± 0.2	± 0.2	± 0.0	± 0.3
Carbon Dioxide	Mean	n.a.	0.1	0.2	0.2	0.7	1.2	2.0	3.3	4.6
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.2
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.1	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	2.2	2.6	3.8	5.3	6.4	6.4	6.9	8.8	8.6
	SD	± 0.0	± 0.1	± 0.8	± 0.1	± 0.4	± 0.3	± 0.2	± 0.4	± 0.1
Total Recovery	Mean	97.4	100.6	99.2	99.9	99.2	100.6	99.8	99.6	99.1
	SD	± 1.3	± 0.9	± 0.0	± 0.2	± 0.5	± 0.2	± 0.0	± 0.4	± 0.0

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues

Table 7.1.2.1.1- 34: Degradation of [phenyl-U-^{14}C]-fluopicolide in Vilobi d'Ònyar soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	7	10	30	44	59	91	120
Fluopicolide	Mean	94.4	91.3	82.2	78.6	59.4	53.4	46.6	36.8	30.2
	SD	± 0.1	± 0.4	± 0.8	± 1.2	± 0.4	± 0.1	± 0.3	± 0.3	± 0.2
M-01 (AE C653711)	Mean	2.3	6.5	13.5	20.2	34.8	40.0	45.0	52.1	55.0
	SD	± 0.0	± 0.0	± 0.3	± 0.6	± 0.6	± 0.7	± 1.0	± 0.4	± 1.2
u3	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u4	Mean	n.d.	LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
u5	Mean	n.d.	LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	n.d.	LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
$CaCl_2$ solution	Mean	44.1	44.6	42.3	44.7	45.2	46.0	46.5	48.3	48.0
	SD	± 0.1	± 0.2	± 0.1	± 0.3	± 0.3	± 0.3	± 0.9	± 0.3	± 1.5
Ambient Extract	Mean	50.8	51.5	50.8	50.8	45.3	43.4	41.0	36.2	32.9
	SD	± 0.3	± 0.2	± 0.5	± 0.8	± 0.1	± 0.3	± 0.1	± 0.1	± 0.4
Microwave Extract	Mean	2.0	2.3	2.7	3.5	3.7	4.0	4.1	4.5	4.4
	SD	± 0.2	± 0.0	± 0.2	± 0.2	± 0.1	± 0.1	± 0.1	± 0.3	± 0.1
Total Extractable Residues	Mean	96.7	97.8	95.7	98.9	94.2	93.4	91.6	88.9	85.2
	SD	± 0.1	± 0.4	± 0.5	± 1.2	± 0.2	± 0.5	± 0.7	± 0.1	± 1.1
Carbon Dioxide	Mean	n.a.	0.1	0.2	0.2	0.7	1.1	1.8	2.8	3.8
	SD		± 0.1	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.1
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	0.7	1.3	2.0	2.6	4.1	5.5	6.2	8.5	10.0
	SD	± 0.0	± 0.0	± 0.0	± 0.1	± 0.2	± 0.3	± 0.2	± 0.3	± 0.2
Total Recovery	Mean	97.4	99.1	97.9	101.6	98.9	100.0	99.6	100.2	99.0
	SD	± 0.1	± 0.5	± 0.5	± 1.4	± 0.4	± 0.3	± 0.9	± 0.4	± 1.0

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues

B. Material Balance

Mean mass balances were 99.9% AR for H[redacted] (range from 99.1 to 100.7% AR), 99.4% AR for Great Chishill soil (range from 98.1 to 101.8% AR), 99.3% AR for Parcey Meslay soil (range from 98.2 to 100.5% AR), 98.9% AR for Mas de Coq soil (range from 97.9 to 99.4% AR), 99.6% AR for Albaro soil (range from 98.3 to 100.6% AR) and 99.4% AR for Vilobi d'Ònyar soil (range from 97.4 to 101.6% AR).

The results confirm there were no significant losses of radioactivity during sample processing.

C. Extractable and Non-Extractable Residues

Desorbable residues in aqueous 0.01 M CaCl2 solution decreased from DAT-0 to DAT-120 from 47.3 to 41.7% AR in H[redacted] soil, from 28.6 to 20.6% AR in Chishill soil, from 35.9 to 32.2% AR in Parcey Meslay soil and from 49.5 to 33.5% AR in Mas du Coq soil. In Vilobi soil, desorbable residues in aqueous 0.01 M CaCl2 solution increased from DAT-0 to DAT-120 from 44.0 to 48.0% AR. In Albaro soil desorbable residues in aqueous 0.01 M CaCl2 solution ranged from 32.3 % to 35.4% AR over the course of the study (mean 33.9 % AR), initially declining from 35.4 % at DAT-0 to 32.2% AR at DAT-30 and then increased to 35.4% AR at DAT-120.

Total extractable residues (i.e. residues desorbed by aqueous 0.01 M CaCl2 solution and residues in organic soil extracts) decreased from DAT-0 to DAT-120 from 98.7 to 89.2% AR in H[redacted] soil, from 96.7 to 86.5% AR in Chishill soil, from 97.3 to 89.5% AR in soil FR09B, from 97.2 to 86.5% AR in Parcey Meslay soil, from 96.1 to 85.8% AR in Albaro soil and from 96.7 to 85.2% AR in Vilobi soil.

Non-extractable soil residues increased concurrently with the decrease in extractable radioactivity in all soils. Non-extractable residues (NER) increased from DAT-0 to DAT-120 from 1.1 to 8.8% AR in H[redacted] soil, from 0.9 to 8.3% AR in Parcey Meslay soil, from 0.7 to 9.6% AR in Mas du Coq soil, and from 0.7 to 10.0% AR in Vilobi soil. In Chishill soil, NER increased from DAT-0 to DAT-91 from 2.8 to 12.6% AR and decreased then slightly to 10.6% AR at DAT-120. In Albaro soil, NER increased from DAT-0 to DAT-91 from 2.2 to 8.8% AR and decreased then slightly to 8.6% AR at DAT-120.

D. Volatile Radioactivity

The maximum amount of carbon dioxide formed was 2.0, 1.7, 1.9, 3.0, 4.6 and 3.8 % AR in H[redacted], Chishill, Parcey Meslay, Mas du Coq, Albaro and Vilobi soils, respectively by the end of the study (DAT-120). Formation of volatile organic compounds (VOC) was insignificant with values of ≤ 0.1% AR at all timepoints in all soils.

E. Degradation of Parent Compound

The amount of fluopicolide in the total soil extracts (i.e. in aqueous desorption solution and organic soil extracts) decreased from 97.0% at DAT-0 to 46.2% at DAT-120 in H[redacted] soil, from 96.2 to 66.5% AR in Chishill soil, from 97.3 to 57.9% AR in Parcey Meslay soil, from 95.8 to 59.8% AR in Mas du Coq soil, from 95.2 to 52.6% AR in Albaro soil and from 94.4 to 30.2% AR in Vilobi soil.

Degradation of fluopicolide was accompanied by the formation of one degradation product, M-01 (AE C653711), which was observed at a maximum of 55.0% AR at DAT-120 in Vilobi soil. M-01 was identified by co-chromatography with an analytical standard and by LC/MS/MS after isolation of the radiopeak from a desorption solution. The total unidentified residues were < LOD at any sampling interval in any soil.

F. Degradation Kinetics

Reported DT_{50} values of fluopicolide under aerobic conditions were 101, 251, 167, 180, 131 and 54.7 days in H[redacted], Chishill, Parcey Meslay, Mas du Coq, Albaro and Vilobi soils, respectively. The experimental data was best described by a double first order in parallel (DFOP) kinetic model. Details are provided below in Table 7.1.2.1.1- 35.

Table 7.1.2.1.1- 35: Reported degradation rate of fluopicolide under aerobic conditions at 20 °C (best-fit DT_{50} values for persistence endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} (days)	DT_{90} (days)
[redacted]	DFOP	98.68	k1 9.178e-02 k2 4.354e-03 g 2.237e-01	1.2	4.29e-06 2.39e-10	- - 1.898e-01	- - 0.258	101	471
Great Chishill	DFOP	99.38	k1 2.77e-01 k2 2.175e-03 g 1.374e-01	0.6	2.45e-06 8.62e-13	- - 1.241e-01	- - 0.151	251	991
Parcey Meslay	DFOP	98.13	k1 8.558e-02 k2 3.380e-03 g 1.208e-01	0.7	0.000453 1.02e-10	- - 9.169e-02	- - 0.150	167	643
Mas du Coq	DFOP	97.67	k1 8.987e-02 k2 3.270e-03 g 9.820e-02	0.5	0.000539 8.19e-12	- - 7.429e-02	- - 0.122	180	673
Albaro	DFOP	98.46	k1 2.327e-01 k2 4.592e-03 g 8.785e-02	0.8	0.00287 3.56e-14	- - 6.684e-02	- - 0.109	131	481
Vilobi d'Òny ar	DFOP	96.87	k1 7.978e-02 k2 7.223e-03 g 2.624e-01	0.7	7.73e-08 5.85e-12	- - 2.288e-01	- - 0.296	54.7	277

In addition, the experimental data for the degradation of fluopicolide and M-01 has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting DT_{50} values for trigger endpoints for fluopicolide are summarised below in Table 7.1.2.1.1- 36. Best fit kinetics are highlighted in bold. The results are very similar to reported best fit DT_{50} values.

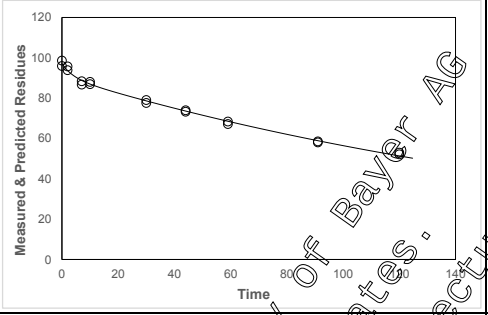
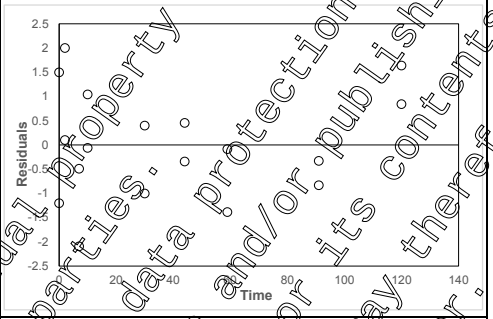
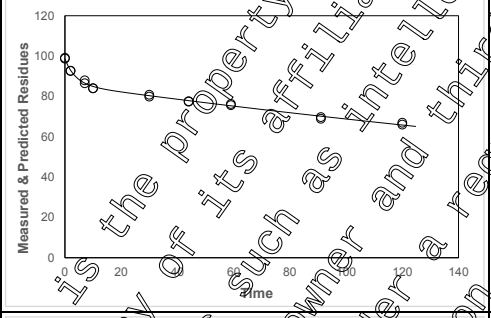
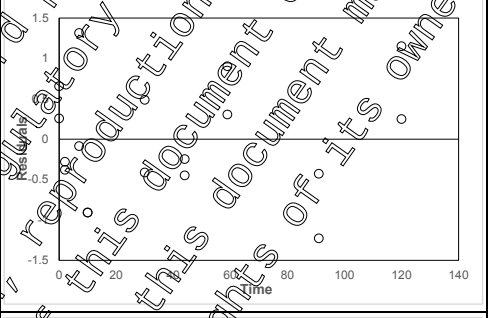
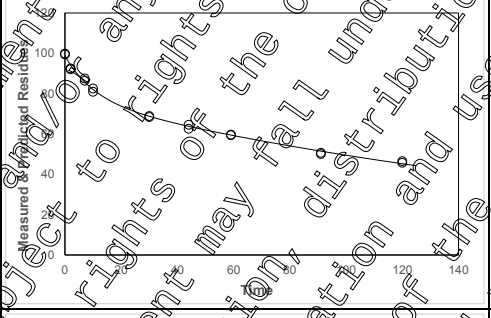
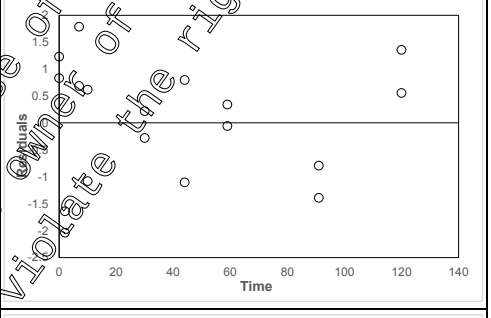
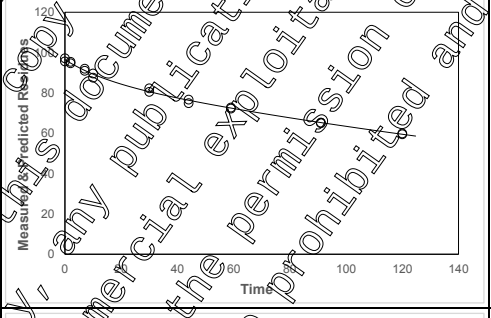
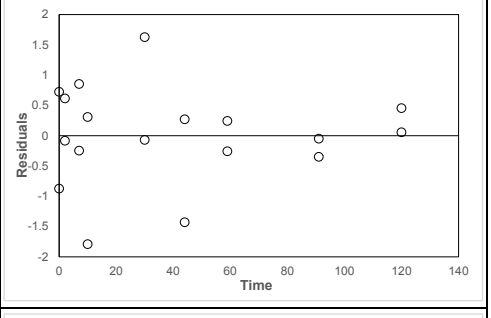
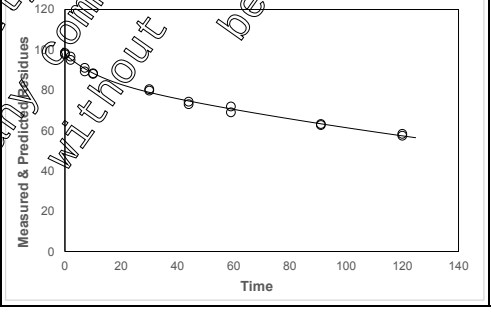
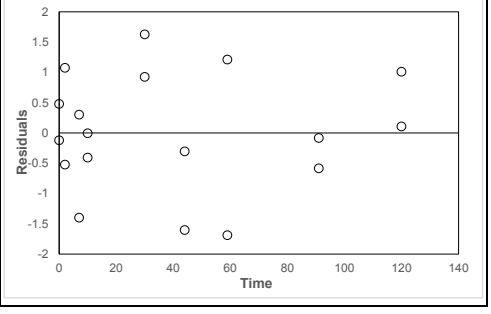
Table 7.1.2.1.1- 36: Re-evaluated degradation rate of fluopicolide under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

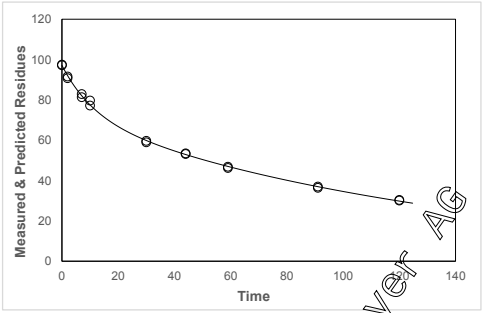
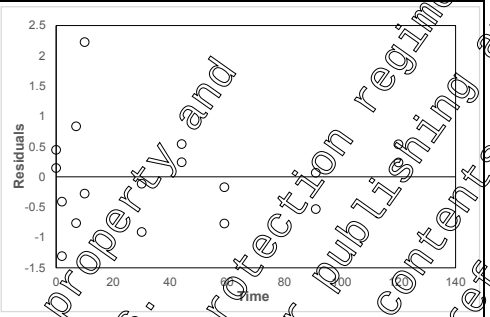
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Albaro/Marcomcini, [redacted] (2016a)	SFO	94.24	k 0.005383	2.18	<2e-16	0.004892	0.006	128.8	427.8
	FOMC	95.86	α 0.5563 β 65.09	1.55	n.r. n.r.	0.3198 23.01	0.793 106.6	161.2	4019
	DFOP	**97.2**	**k1 0.2499 k2 0.004742 g 0.06658**	**0.913**	**0.00172 <2e-16 n.r.**	**0.09565 0.004432 0.04564**	**0.404 0.005 0.088**	**131.6**	**471**
Great Chishill, [redacted] (2016a)	SFO	92.7	k 0.003447	3.66	1.50E-11	0.002764	0.004	201.1	668
	FOMC	96.37	α 0.1235 β 7.602	1.9	n.r. n.r.	0.08991 1.826	0.157 13.38	2096	10000
	DFOP	**98.54**	**k1 0.2582 k2 0.002219 g 0.1279**	**0.658**	**2.83E-08 <2e-16 n.r.**	**0.1869 0.002038 0.115**	**0.33 0.002 0.141**	**250.9**	**975.9**
H[redacted] (2016a)	SFO	99.34	k 0.01003	8.61	1.05E-10	0.007872	0.012	69.1	229.6
	FOMC	98.98	α 0.3038 β 12.15	1.6	n.r. n.r.	0.2679 8.935	0.34 15.36	106.8	>10000
	DFOP	**98.67**	**k1 0.09601 k2 0.004458 g 0.2171**	**1.19**	**7.06E-14 <2e-16 n.r.**	**0.08116 0.00399 0.1882**	**0.111 0.005 0.246**	**100.6**	**461.6**
Mas du Coq, [redacted] (2016a)	SFO	94.56	k 0.004277	1.84	<2e-16	0.003862	0.005	162.1	538.4
	FOMC	96.5	α 0.3824 β 50.86	0.666	n.r. n.r.	0.29 31.11	0.475 70.62	260.7	>10000
	DFOP	**96.57**	**k1 0.06602 k2 0.003199 g 0.09496**	**0.495**	**6.42E-06 <2e-16 n.r.**	**0.04119 0.002847 0.06684**	**0.091 0.004 0.123**	**185.5**	**688.6**
Parcey Meslay, [redacted] (2016a)	SFO	95.27	k 0.004859	2.63	<2e-16	0.004258	0.005	142.7	473.9
	FOMC	97.68	α 0.3101 β 29.62	0.981	n.r. n.r.	0.2456 18.24	0.375 41.01	247.3	>10000
	DFOP	**98.02**	**k1 0.08553 k2 0.003422 g 0.1172**	**0.702**	**4.50E-08 <2e-16 n.r.**	**0.06156 0.003065 0.09078**	**0.109 0.004 0.144**	**166.1**	**636.4**
Vilobi d'Onyar, [redacted] (2016a)	SFO	94.27	k 0.01261	6.13	4.99E-15	0.01078	0.014	55	182.5
	FOMC	97.02	α 0.5975 β 23.07	1.61	n.r. n.r.	0.5141 17.12	0.681 29.03	50.5	1065
	DFOP	**97.15**	**k1 0.08873 k2 0.007416 g 0.2516**	**0.76**	**4.20E-14 <2e-16 n.r.**	**0.07528 0.006893 0.2231**	**0.102 0.008 0.28**	**54.9**	**271.4**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.1.1- 37: Degradation of fluopicolide under aerobic conditions at 20 ºC (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Albaro/Marcomcini DFOP [redacted] (2016a)		
Great Chishill DFOP [redacted] (2016a)		
[redacted] (2016a)		
Mas du Coq DFOP [redacted] (2016a)		
Parcey Meslay DFOP [redacted] (2016a)		

Soil Model Reference	Modelled vs observed	Residuals
Vilobi d'Ònyar DFOP [redacted] (2016a)		

G: Time-Dependent Sorption

The sorption of fluopicolide in soil increased steadily with time. Apparent sorption coefficients ($K_{d,app}$) increased from DAT-0 to DAT-120 in all six soils. The values are summarised in Table 7.1.2.1.1-38.

Table 7.1.2.1.1- 38: Apparent sorption coefficients ($K_{d, app}$) (expressed as mL/g)

DAT	Mean SD	H [redacted]	Great Chishill	Parcey Meslay	Mas du Coq	Albaro Marcomcini	Vilobi d'Ònyar
0	Mean	2.87	6.57	4.89	2.62	4.66	3.46
	SD	± 0.01	± 0.00	± 0.00	± 0.01	± 0.01	± 0.00
2	Mean	3.34	7.55	6.13	3.32	5.23	3.90
	SD	± 0.00	± 0.01	± 0.03	± 0.00	± 0.01	± 0.02
7	Mean	4.20	8.17	6.95	3.92	5.49	4.70
	SD	± 0.01	± 0.00	± 0.01	± 0.02	± 0.02	± 0.00
10	Mean	4.12	7.96	6.91	3.86	5.52	4.78
	SD	± 0.01	± 0.01	± 0.00	± 0.00	± 0.00	± 0.01
30	Mean	5.21	10.38	8.83	5.52	6.55	5.89
	SD	± 0.03	± 0.01	± 0.01	± 0.01	± 0.01	± 0.01
44	Mean	5.92	11.38	9.74	6.05	7.19	6.47
	SD	± 0.01	± 0.01	± 0.00	± 0.00	± 0.01	± 0.01
69	Mean	6.26	12.37	9.90	6.36	7.48	6.77
	SD	± 0.03	± 0.00	± 0.00	± 0.00	± 0.02	± 0.02
91	Mean	7.41	13.90	11.21	7.61	8.00	8.12
	SD	± 0.02	± 0.04	± 0.03	± 0.03	± 0.02	± 0.01
120	Mean	8.26	15.00	11.14	8.16	9.00	8.55
	SD	± 0.01	± 0.02	± 0.02	± 0.03	± 0.05	± 0.01
Factor [A]		2.88	2.28	2.28	3.12	1.93	2.47
Mean Factor		2.49					

Apparent Sorption Coefficients ($K_{d, app}$) are called Time-Dependent Sorption Ratios (R_{TDS}) in the report.
[A] Calculated as $K_{d, app}$ DAT-120 divided by $K_{d, app}$ DAT-0

The results are more fully discussed under Section 7.1.3.2 (see KCA 7.1.3.2/04).

III. Conclusion

Fluopicolide was well to moderately degraded and mineralized in six European field soils; H[redacted], Great Chishill, Parcey Meslay, Mas du Coq, Albaro and Vilobi d'Onyar, under aerobic conditions at 20°C in the dark. Best fit DT_{50} values ranged from 54.7 to 251 days in the tested soils. Re-evaluated best fit DT_{50} values were similar, ranging from 54.9 to 250.7 days.

The primary objective of the study was to investigate the sorption of fluopicolide, determined under equilibrium conditions, following its aging in soil under aerobic conditions in the dark under laboratory conditions. The time-dependent sorption ratio increased throughout the incubation period (120 days) by a factor of 1.93 to 3.12 in the six soils tested.

Formation of carbon dioxide was significant (up to 4.6% AR) by the end of the study indicating the potential for complete mineralization of fluopicolide and its degradation products. One major degradation product, M-01 (AE C653711), was identified with a maximum of 35.0% AR.

Formation of non-extractable residues (NER) was up to 10.6% AR at study end, which is an indication for biotic degradation of fluopicolide.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002), US EPA 835.4100 / 835.4200 (2008) and in parts, where relevant, to OECD 106 (2000). The study is considered valid to assess the aerobic degradation of [phenyl-U-^{14}C]fluopicolide in soil.

The study has been designed so that it is valid to assess the changes in sorption of fluopicolide with time in accordance with guidance provided by Food and Environment Research Agency (2019) on conducting aged sorption studies. A kinetic assessment of the time dependent sorption (TDS) parameters is provided in KCA 7.1.3.2.

Data Point:	KCA 7.1.2.1.1/09
Report Author:	[redacted]
Report Year:	2019
Report Title:	[phenyl-UL-14C]fluopicolide: Degradation and time - Dependent sorption in 6 soils
Report No:	EnSa-16-0983
Document No:	M-655056-01-1
Guideline(s) followed in study:	- OECD Test Guideline 307 (April, 2002) - US EPA OCSPP Test Guideline No. 835.4100 / 835.4200 - OECD Test Guideline No. 106 (January, 2000; only in parts) - Guidance on how aged sorption studies for pesticides should be conducted, analysed and used in the regulatory process; Food and Environmental Agency, York, UK, 2012
Deviations from current test guideline:	none
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation and time-dependence of sorption of fluopicolide was studied in six soils under aerobic conditions in the laboratory in the dark at 20 ± 1 °C and 53.9% of the maximum water holding capacity for 120 days, with the exception of Lignieres soil which was unintentionally incubated at a soil moisture content equivalent to 72.9% of the maximum water holding capacity. In addition, the rate of degradation of fluopicolide was determined in the study.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
Lamberton	loam	5.6	2.6
Sarotti	silty clay loam	6.9	1.4
Münster	loamy sand	5.6	1.2
Pikeville	loamy sand	4.5	1.8
Abington	sandy loam	7.3	2.6
Lignieres	sandy loam	5.7	0.8

[Phenyl-UL-^{14}C]-labelled fluopicolide was applied to soil samples at an application rate of ca. 0.41 mg/kg dry weight. The radiochemical purity and specific activity were > 98% and 5.50 MBq/mg, respectively.

Samples of Lamberton and Münster soils were taken for extraction and analysis immediately after treatment (day 0) and 2, 8, 13, 30, 44, 56, 90 and 119 days of incubation and samples of Sarotti, Pikeville, Abington and Lignieres soils after 0, 2, 8, 10, 28, 45, 59, 85 (86, Lignieres soil) and 120 days of incubation. Soil samples were first desorbed with 0.01M calcium chloride solution for 24 hours at 20 °C at a soil : solution ratio of 1:5 (w/w) to determine the desorbable portion of the test item from aged soil. The soil residue was then exhaustively extracted with three further successive extractions with acetonitrile/water 4:1 (v/v) at ambient temperature, followed by a microwave extraction with acetonitrile/water (4/1, v/v) at 70 °C. Desorption supernatants and concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC).

Recovery of radioactivity was quantitative throughout the study. Overall mean mass balances were 95.4% AR for Lamberton soil, 98.6% AR for Sarotti soil, 96.9% AR for Münster soil, 98.5% AR for Pikeville soil, 97.5% AR for Abington soil and 98.8% AR for Lignieres soil.

Desorbable residues in aqueous 0.01 M calcium chloride solution were 16.8, 26.3, 35.9, 17.3, 26.5 and 35.7% AR in Lamberton, Sarotti, Münster, Pikeville, Abington and Lignieres soils, respectively by the end of the study (DAT-119/120). Total extractable residues (i.e. residues desorbed by aqueous 0.01 M CaCl2 solution and residues in organic soil extracts) decreased from DAT-0 to DAT-119/120 from 90.2 to 80.2% AR in Lamberton soil, from 96.4 to 79.6% AR in Sarotti soil, from 95.4 to 86.8% AR in Münster soil, from 96.1 to 80.6% AR in Pikeville soil, from 94.3 to 78.3% AR in Abington soil and from 101.0 to 84.9% AR in Lignieres soil.

Non-extractable residues (NER) increased from DAT-0 to DAT-119/120 from 3.8 to 13.3% AR in Lamberton soil, from 3.6 to 13.4% AR in Sarotti soil, from 0.8 to 6.5% AR in Münster soil, from 1.7 to 13.4% AR in Pikeville soil, from 2.6 to 10.2% AR in Abington soil and from 1.4 to 8.9% AR in Lignieres soil.

The maximum amount of carbon dioxide formed was 3.2, 5.3, 3.8, 3.7, 8.1 and 4.5% AR in the six soils by the end of the study (DAT-120). No significant levels of organic volatiles were observed (≤ 0.1% AR).

After 119/120 days incubation at 20 °C, fluopicolide degraded to 32.2, 54.1, 50.4, 47.9, 45.9 and 50.1% of the applied radioactivity in the six soils. M-01 (AE C653711) was also observed in all soils, increasing steadily to maxima of 24.8, 31.7, 22.8, 28.5 and 34.8% AR by the end of the study (DAT-119/120) in Lamberton, Münster, Pikeville, Abington and Lignieres soils. In Sarotti soil, M-01 reached a maximum of 23.4% AR at DAT-85 before declining slightly to 21.1% AR by the end of the study. M-03 was observed in Pikeville soil only at a maximum of 6.6% AR at DAT-85 before declining slightly to 5.2% AR by DAT-120. M-01 and M-03 were identified by LC/MS/MS after isolation from concentrated extracts. No other unidentified degradation products exceeded 3.3% AR.

The effect of aged sorption to soil was determined for fluopicolide and showed a significant increase with time. Apparent sorption coefficients ($K_{d\ app}$) increased with time in all soils by a factor of 1.62 to 2.53 (mean 2.22). Further details specific to the aged sorption of fluopicolide are provided in Section 7.1.3.2.

Degradation kinetics for fluopicolide provided in the report were conducted in accordance with FOCUS guidance document on degradation kinetics (2014). The best-fit DT_{50} values were 149, 131, 135, 114, 116 and 117 days in Lamberton, Sarotti, Münster, Pikeville, Abington and Lignieres soils, respectively. A re-evaluation of the degradation kinetics resulted in similar best-fit un-normalised DT_{50} values of 119.2 to 158.3 days.

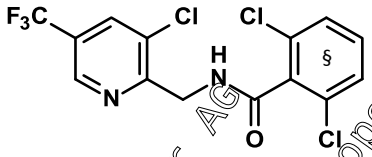
Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)	Visual Assessment
Lamberton (loam)	DFOP	149	597	1.6	Good
Sarotti (silty clay loam)	DFOP	131	515	1.4	Good
Münster (loamy sand)	DFOP	135	524	0.7	Good
Pikeville (loamy sand)	DFOP	114	480	1.4	Good
Abington (sandy loam)	DFOP	116	451	1.3	Good
Lignieres (sandy loam)	DFOP	117	453	1.5	Good

I. Materials and Methods

A. Materials

1. Test Items

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



§ Denotes position of [^{14}C]-radiolabel

Specific Activity:	5.50 MBq/mg
Radiochemical Purity:	>98% (HPLC)

2. Test Soils

The study was performed using six soils from agricultural areas as characterized in Table 7.1.2.1.1- 39. Five of these soils were collected from the same sites as earlier laboratory aerobic soil studies (Lamberton, Sarotti, Münster, Pikeville and Abington soils) and one soil was from a terrestrial field dissipation site used for fluopicolide and its metabolite M-01 (Lignieres soil). The same batches of all six soils were also used in OECD 106 adsorption desorption studies (see CA 7.1.3.1.1/05).

Table 7.1.2.1.1- 39: Physico-chemical properties of test soils

Parameter		Soil					
Soil Designation		Lamberton		Sarotti		Münster	
Soil ID		LB		SR2		MS	
Geographic Location							
City		Lamberton, Minnesota		Hattersheim, Hesse		Münster, Northrine-Westfalia	
Country		USA		Germany		Germany	
Batch Number		100416-S		20161109		20161014	
Textural Classification (USDA)		Loam		Silty clay loam		Loamy sand	
Sand [50 - 2000 µm]	(%)	51		14		79	
Silt [2 – 50 µm]	(%)	28		54		20	
Clay [< 2 µm]	(%)	21		32		1	
pH							
in $CaCl_2$ (1:1)		5.6		6.9		5.6	
in H_2O (1:1)		5.8		7.0		6.0	
Saturated paste		5.7		6.9		6.0	
in KCl (1:1)		5.2		6.4		5.4	
Organic Matter (%) *		4.5		2.4		2.1	
Organic Carbon (%)		2.6		1.4		1.2	
Cation Exchange Capacity (meq/100 g)		20.7		16.2		6.6	
Water Holding Capacity							
Maximum (g H_2O per 100 g DW)		59.4		54.8		32.9	
at 1/10 bar (%)		42.3		35.1		27.4	
Moisture Content During Incubation (%)		53.9% MWHC		53.9% MWHC		53.9% MWHC	
Bulk Density (disturbed, g/cm³)		1.09		1.08		1.37	
Soil Microbial Biomass (µg microbial C /g soil)		BIO⁻	BIO⁺	BIO⁻	BIO⁺	BIO⁻	BIO⁺
Initial (Day 0)		468		447		204	
Mid (Day 60)		377	424	458	461	114	136
Final (Day 120)		342	324	370	371	116	124

BIO⁻ samples were untreated
BIO⁺ samples were treated with 400 µL of methanol/water (1:1 v/v)

Parameter		Soil					
Soil Designation		Pikeville		Abington		Lignieres	
Soil ID		PV		AB2		LN	
Geographic Location							
City		Pikeville, North Carolina		Abington, Cambridgeshire		Lignieres de Touraine, Indre-et Loire,	
Country		USA		UK		France	
Batch Number		100516-S		16/069		20160912	
Textural Classification (USDA)		Loamy sand		Sandy loam		Sandy loam	
Sand [50 - 2000 µm]	(%)	73		66		72	
Silt [2 – 50 µm]	(%)	26		20		16	
Clay [< 2 µm]	(%)	1		14		12	
pH							
in $CaCl_2$ (1:1)		4.5		7.3		5.7	
in H_2O (1:1)		4.9		7.4		6.0	
Saturated paste		4.8		7.3		6.0	
in KCl (1:1)		4.3		7.0		5.2	
Organic Matter (%) *		3.1		4.5		1.4	
Organic Carbon (%)		1.8		2.6		0.8	
Cation Exchange Capacity (meq/100 g)		6.0		19.2		11.8	
Water Holding Capacity							
Maximum (g H_2O per 100 g DW)		44.5		59.7		41.6	
at 1/10 bar (%)		31.0		20.3		20.3	
Moisture Content During Incubation (%)		53.9% MWHC		53.9% MWHC		72.9% MWHC	
Bulk Density (disturbed, g/cm³)		1.20		1.11		1.25	
Soil Microbial Biomass (µg microbial C /g soil)		BIO^-	BIO^+	BIO^-	BIO^+	BIO^-	BIO^+
Initial (Day 0)		182		1146		284	
Mid (Day 60)		194	186	1059	1035	188	180
Final (Day 120)		132	135	840	849	116	129

BIO^- samples were untreated
BIO^+ samples were treated with 400 µL of methanol/water (1:1 v/v)

B. Study Design

1. Experimental Conditions

Tests were performed in static systems consisting of an Erlenmeyer flask containing 100 g soil (dry weight equivalents) fitted with a trap attachment (permeable for oxygen) containing soda lime for absorption of carbon dioxide and a polyurethane foam plug for adsorption of volatile organic compounds.

The tests were performed at a concentration of 0.399 mg/kg dry weight of soil to Lignieres soil, 0.41 mg/kg DW to Lamberton and Münster soils, and 0.42 mg/kg DW to Abington, Pikeville and Sarotti soils. The test item [phenyl-U-^{14}C]-fluopicolide dissolved in methanol/water (1:1, v/v) (400 µL) was applied drop wise onto the soil surface. Samples from five soils (Lamberton, Sarotti, Münster, Pikeville, Abington) were adjusted to a moisture content equivalent to 53.9% of maximum water holding capacity, three to four days prior to application. Samples Lignieres soil were unintentionally incubated at a mean soil moisture content equivalent to 72.9% of the maximum water holding capacity. This does not appear

to have had an impact of the degradation of fluopicolide. All samples were incubated at 20 ± 1 °C under aerobic conditions in the dark for 120 days. Soil moisture was maintained during incubation by addition of de-ionized water after 30, 60 and 90 days of incubation. No significant losses of moisture were observed throughout the study.

2. Sampling

Duplicate samples of Lamberton and Münster soils were removed for analysis after 0, 2, 8, 13, 30, 44, 56, 90 and 119 days of incubation and duplicate samples of Sarotti, Pikeville, Abington and Lignieres soils after 0, 2, 8, 10, 28, 45, 59, 85 (86, Lignieres soil) and 120 days of incubation. Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment (Day 0, Day 60 and Day 120).

3. Analytical Procedures

Soil samples were first shaken with 0.01M calcium chloride solution for 24 hours at 20 °C on an overhead shaker at 30 rpm to determine the desorbable portion of the test item from aged soil. A soil-to-solution ratio of 1:3 was used for all soils. Soil samples were then extracted three times with acetonitrile/water (4/1, *v/v*) at ambient temperature followed by a microwave extraction with acetonitrile/water (4/1, *v/v*) at 70 °C. After each extraction step, extract and soil were separated by centrifugation.

Radioactivity in extracts was determined by liquid scintillation counting (LSC). Desorption supernatants were analysed directly by HPLC with radiodetection. Ambient and microwave soil extracts were pooled and concentrated prior to analysis by HPLC. The concentration procedure for soil extracts was established as quantitative (recovery 97.1 to 103.6%). HPLC column recovery was also quantitative (recovery 104.1%). The maximum HPLC LOD was determined as 1.6% AR. The primary chromatographic method for analysis of soil extracts was a reverse phase C18 HPLC method. Selected extracts were analysed by a second confirmatory phenyl-hexyl phase HPLC method. Selected desorption supernatant and ambient soil extracts were concentrated and radiopeaks corresponding to the two degradation products isolated, prior to analysis by LC/MS/MS for identification of M-01 and M-03.

With the exception of the time zero samples, trap attachments were removed for analysis at each sampling time. Soda lime from the trap attachment was transferred into an Erlenmeyer flask, aqueous hydrochloric acid (18%) added dropwise and any liberated carbon dioxide collected in trapping vessels containing scintillation cocktail. The polyurethane foam plug was extracted with ethyl acetate to desorb any volatile organic compounds. The radioactivity content of these samples was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The degradation of fluopicolide was best described by the double first order in parallel (DFOP) model in all soils based on lowest chi^2 error values and visual assessments of fits.

Additionally, modelling endpoints for the degradation of fluopicolide, M-01 and M-03 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each soil between the SFO and FOMC fits for fluopicolide. For all six soils, this comparison indicated a bi-phasic pattern in the fluopicolide residues, and the DFOP model was therefore also fitted. The DFOP model provided the best visual fit to the fluopicolide residues for all of the soils, with the lowest χ^2 err% value.

Metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soils incubated at 20 °C following application of [phenyl-U-^{14}C]- fluopicolide are summarized in Table 7.1.2.1.1- 40 to Table 7.1.2.1.1-45.

Table 7.1.2.1.1- 40: Degradation of [phenyl-U-^{14}C]-fluopicolide in Lamberton soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	8	13	30	44	56	90	119
Fluopicolide	Mean	88.0	84.2	79.1	77.5	69.2	66.9	66.7	58.7	52.2
	SD	± 0.3	± 0.4	± 1.1	± 0.1	± 1.1	± 0.9	± 0.2	± 0.3	± 0.3
M-01 (AE C653711)	Mean	< LOD	2.3	5.0	6.3	10.3	11.8	16.0	22.9	24.8
	SD		± 0.3	± 0.4	± 0.5	± 1.3	± 0.4	± 0.4	± 1.0	± 1.2
M-03 (AE 0608000)	Mean	n.d.	n.d.	n.d.	n.d.	< LOD	< LOD	< LOD	< LOD	< LOD
	SD									
ROI 2	Mean	< LOD	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	< LOD	n.d.	n.d.	2.1
	SD									
$CaCl_2$ solution	Mean	15.3	13.0	12.4	11.9	11.3	11.6	13.1	14.0	16.8
	SD	0.3	0.2	0.2	0.3	0.2	0.3	0.6	0.3	0.1
Ambient Extract	Mean	70.0	66.7	64.4	63.1	57.8	56.0	59.1	58.5	48.4
	SD	0.2	0.1	1.3	1.7	0.2	0.6	0.4	0.8	0.4
Microwave Extract	Mean	4.8	7.6	7.4	8.9	11.1	11.8	11.6	10.6	15.0
	SD	0.2	0.6	0.1	0.9	0.3	0.1	0.4	0.4	0.3
Total Extractable Residues	Mean	89.1	86.5	84.1	83.8	79.7	78.7	82.7	81.6	79.1
	SD	± 1.5	± 0.2	± 1.5	± 0.6	± 0.2	± 0.6	± 0.6	± 0.7	± 0.6
Carbon Dioxide	Mean	n.a.	0.7	1.6	1.8	2.2	2.5	2.5	3.0	3.2
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.1	± 0.0	± 0.0
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	3.8	7.9	8.4	9.9	11.4	12.4	10.4	11.6	13.3
	SD	± 0.2	± 0.8	± 1.2	± 0.3	± 0.6	± 0.7	± 0.5	± 0.1	± 0.6
Total Recovery	Mean	92.9	95.1	94.2	95.5	93.4	93.6	95.6	96.1	95.6
	SD	± 1.6	± 0.7	± 0.4	± 0.3	± 0.4	± 1.2	± 0.0	± 0.6	± 0.1

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 41: Degradation of [phenyl-U-^{14}C]-fluopicolide in Sarotti soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	8	10	28	45	59	85	120
Fluopicolide	Mean	93.4	90.5	83.7	83.2	77.0	73.1	66.7	58.1	54.1
	SD	± 1.4	± 0.5	± 1.3	± 1.5	± 1.4	± 0.7	± 0.3	± 1.7	± 0.4
M-01 (AE C653711)	Mean	2.1	4.2	5.9	7.9	11.4	15.5	16.9	23.4	21.1
	SD	± 0.1	± 0.5	± 0.7	± 0.7	± 0.1	± 0.1	± 0.2	± 0.4	± 1.9
M-03 (AE 0608000)	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
ROI 2	Mean	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.5	4.3
	SD								± 0.9	± 0.5
$CaCl_2$ solution	Mean	39.7	35.7	33.2	32.4	30.0	29.3	26.6	31.4	26.4
	SD	0.4	0.2	0.6	0.8	0.1	0.3	0.1	0.5	0.7
Ambient Extract	Mean	54.2	56.6	52.1	55.7	53.8	54.6	50.9	46.1	46.4
	SD	0.4	0.3	0.2	0.0	1.4	0.5	0.5	0.2	0.2
Microwave Extract	Mean	2.6	2.5	4.3	3.1	4.7	4.8	6.2	6.6	6.9
	SD	0.1	0.1	0.1	0.0	0.1	0.0	0.4	0.4	0.1
Total Extractable Residues	Mean	96.4	94.7	89.6	91.1	88.4	88.6	83.6	84.0	79.6
	SD	± 0.7	± 0.0	± 0.6	± 0.7	± 1.5	± 0.8	± 0.0	± 0.4	± 0.9
Carbon Dioxide	Mean	n.a.	1.0	1.7	1.7	2.4	2.8	2.0	4.0	5.3
	SD		± 0.1	± 0.1	± 0.0	± 0.0	± 0.0	± 1.3	± 0.0	± 0.1
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	3.6	4.7	5.7	5.5	7.4	8.7	10.2	11.2	13.4
	SD	± 0.0	± 0.1	± 0.0	± 0.0	± 0.4	± 0.2	± 0.0	± 0.1	± 0.1
Total Recovery	Mean	100.0	100.4	97.0	98.4	98.2	100.1	95.8	99.1	98.3
	SD	± 0.7	± 0.2	± 0.5	± 0.7	± 1.1	± 0.6	± 1.3	± 0.3	± 0.9

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 42: Degradation of [phenyl-U-^{14}C]-fluopicolide in Münster soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	8	13	30	44	56	90	119
Fluopicolide	Mean	90.9	91.4	83.3	80.3	74.1	69.5	66.6	59.2	50.4
	SD	± 0.2	± 0.3	± 1.4	± 1.3	± 0.3	± 0.6	± 0.5	± 0.6	± 0.3
M-01 (AE C653711)	Mean	< LOD	3.3	6.7	12.1	16.4	20.0	22.7	28.9	31.7
	SD		± 0.4	± 0.2	± 0.4	± 0.2	± 0.3	± 0.6	± 0.5	± 0.2
M-03 (AE 0608000)	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	< LOD	< LOD	< LOD
	SD									
ROI 2	Mean	2.9	< LOD	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD	± 0.1								
Sum of Unid./Diff. Residues [A]	Mean	n.d.	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	4.4
	SD									± 0.1
$CaCl_2$ solution	Mean	34.6	31.9	28.6	28.9	30.7	30.9	31.1	31.3	36.0
	SD	± 0.3	± 0.7	± 0.4	± 0.5	± 0.4	± 0.3	± 0.3	± 0.4	± 0.6
Ambient Extract	Mean	59.4	62.5	60.2	61.4	56.9	55.3	55.3	53.8	46.4
	SD	± 0.5	± 0.1	± 0.9	± 1.4	± 0.1	± 0.1	± 0.1	± 0.6	± 0.7
Microwave Extract	Mean	1.5	2.0	1.8	2.2	3.0	3.4	3.6	4.0	4.4
	SD	± 0.1	± 0.1	± 0.2	± 0.2	± 0.0	± 0.2	± 0.0	± 0.1	± 0.1
Total Extractable Residues	Mean	93.8	95.5	90.6	92.4	90.5	89.4	89.2	88.1	86.4
	SD	± 0.3	± 0.1	± 1.2	± 1.7	± 0.4	± 0.3	± 0.1	± 0.2	± 0.1
Carbon Dioxide	Mean	n.a.	0.7	1.3	1.7	2.3	2.5	2.5	3.2	3.8
	SD		± 0.0	± 0.3	± 0.1	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	0.8	2.0	3.2	3.1	3.6	4.3	4.6	5.4	6.5
	SD	± 0.0	± 0.1	± 0.2	± 0.1	± 0.0	± 0.1	± 0.0	± 0.2	± 0.2
Total Recovery	Mean	94.6	98.1	94.6	97.2	96.4	96.3	96.3	97.6	96.7
	SD	± 0.3	± 0.2	± 1.3	± 1.7	± 0.4	± 0.3	± 0.1	± 0.4	± 0.1

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 43: Degradation of [phenyl-U-^{14}C]-fluopicolide in Pikeville soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	8	10	28	45	59	85	120
Fluopicolide	Mean	93.3	93.4	80.0	79.6	72.7	68.1	61.1	56.4	47.8
	SD	± 0.2	± 0.4	± 0.7	± 0.6	± 0.6	± 1.3	± 0.1	± 0.1	± 1.4
M-01 (AE C653711)	Mean	< LOD	< LOD	4.1	6.6	11.1	14.9	19.8	20.5	22.8
	SD			± 0.3	± 1.6	± 0.1	± 0.5	± 0.9	± 0.5	± 0.5
M-03 (AE 0608000)	Mean	< LOD	2.1	5.1	4.4	5.5	6.3	3.6	6.6	5.2
	SD		± 0.2	± 0.1	± 0.8	± 0.2	± 0.5	± 1.0	± 0.6	± 0.3
ROI 2	Mean	1.0	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD	± 0.0								
Sum of Unid./Diff. Residues [A]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	4.6
	SD									± 0.8
$CaCl_2$ solution	Mean	23.3	21.3	19.3	19.0	18.0	17.4	16.7	20.2	17.3
	SD	± 0.1	± 0.1	± 0.6	± 0.5	± 0.0	± 0.0	± 0.5	± 0.4	± 0.7
Ambient Extract	Mean	70.3	72.1	64.2	66.4	61.6	61.4	56.9	51.6	49.7
	SD	± 0.7	± 0.1	± 0.6	± 0.4	± 0.4	± 1.2	± 0.2	± 0.1	± 0.4
Microwave Extract	Mean	2.6	3.7	5.9	5.3	9.8	9.8	10.9	11.8	13.6
	SD	± 0.1	± 0.0	± 0.1	± 0.0	± 0.1	± 0.2	± 0.7	± 0.4	± 0.0
Total Extractable Residues	Mean	95.1	96.6	89.2	90.7	89.3	88.4	84.5	83.6	80.6
	SD	± 0.5	± 0.2	± 1.1	± 0.8	± 0.3	± 1.3	± 0.0	± 0.1	± 0.3
Carbon Dioxide	Mean	n.a.	0.4	1.7	1.8	2.3	2.6	2.8	3.2	3.7
	SD		± 0.0	± 0.1	± 0.0	± 0.0	± 0.1	± 0.0	± 0.0	± 0.0
Volatile Organic Compounds	Mean	n.a.	0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	1.7	3.2	5.8	6.0	6.8	9.3	10.8	11.6	13.4
	SD	± 0.2	± 0.0	± 0.1	± 0.1	± 0.1	± 0.1	± 0.3	± 0.1	± 0.2
Total Recovery	Mean	96.8	100.3	96.7	98.4	98.4	100.4	98.1	98.4	97.6
	SD	± 0.6	± 0.2	± 0.9	± 0.9	± 0.1	± 1.5	± 0.3	± 0.0	± 0.5

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 44: Degradation of [phenyl-U-^{14}C]-fluopicolide in Abington soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	8	10	28	45	59	85	120
Fluopicolide	Mean	91.9	89.8	80.1	82.0	73.9	68.0	64.7	57.9	45.9
	SD	± 0.3	± 0.9	± 0.1	± 0.8	± 1.1	± 0.6	± 0.8	± 0.5	± 0.0
M-01 (AE C653711)	Mean	2.5	5.2	9.5	9.5	14.8	20.1	22.9	22.9	28.5
	SD	± 0.7	± 0.2	± 0.2	± 0.9	± 0.3	± 0.1	± 0.0	± 0.8	± 0.4
M-03 (AE 0608000)	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
ROI 2	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
Sum of Unid./Diff. Residues [A]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.7	3.9
	SD								± 0.2	± 0.9
$CaCl_2$ solution	Mean	24.7	24.5	24.2	24.2	24.9	26.3	26.4	30.6	26.5
	SD	± 0.4	± 0.1	± 0.2	± 0.1	± 0.3	± 0.1	± 0.4	± 0.3	± 0.3
Ambient Extract	Mean	67.6	68.6	63.1	64.9	60.8	58.7	57.4	49.4	47.6
	SD	± 0.7	± 1.3	± 0.1	± 0.1	± 0.5	± 0.4	± 0.1	± 0.3	± 0.3
Microwave Extract	Mean	2.1	2.0	2.4	2.4	3.0	3.3	3.9	3.5	4.3
	SD	± 0.1	± 0.0	± 0.1	± 0.1	± 0.0	± 0.0	± 0.2	± 0.1	± 0.0
Total Extractable Residues	Mean	94.3	95.1	89.6	91.4	88.7	88.2	87.6	83.5	78.3
	SD	± 1.0	± 1.1	± 0.1	± 0.2	± 0.8	± 0.5	± 0.7	± 0.5	± 0.6
Carbon Dioxide	Mean	n.a.	0.9	1.5	2.0	2.8	3.6	4.2	5.7	8.1
	SD		± 0.2	± 0.4	± 0.0	± 0.0	± 0.1	± 0.3	± 0.0	± 0.1
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	2.6	3.5	4.2	4.2	5.2	6.2	7.2	8.4	10.2
	SD	± 0.2	± 0.0	± 0.0	± 0.0	± 0.1	± 0.1	± 0.2	± 0.0	± 0.0
Total Recovery	Mean	96.9	99.5	95.3	97.6	96.7	98.0	99.0	97.6	96.6
	SD	± 0.9	± 1.4	± 0.3	± 0.2	± 0.9	± 0.7	± 0.2	± 0.4	± 0.5

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.1- 45: Degradation of [phenyl-U-^{14}C]-fluopicolide in Lignieres soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)								
		0	2	8	10	30	45	59	86	120
Fluopicolide	Mean	96.3	92.3	85.0	86.5	76.0	74.4	69.2	58.1	50.1
	SD	± 0.5	± 1.9	± 0.4	± 1.0	± 0.9	± 0.5	± 2.1	± 0.7	± 0.9
M-01 (AE C653711)	Mean	1.4	3.2	7.9	7.0	12.8	10.7	18.3	23.3	34.8
	SD	± 0.4	± 0.9	± 0.4	± 0.5	± 1.0	± 0.1	± 3.0	± 0.4	± 0.2
M-03 (AE 0608000)	Mean	n.d.	< LOD	< LOD	n.d.	< LOD	n.d.	n.d.	n.d.	n.d.
	SD									
ROI 2	Mean	3.3	2.5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD	± 0.1	± 0.0							
Sum of Unid./Diff. Residues [A]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD									
$CaCl_2$ solution	Mean	42.0	39.9	35.3	33.0	30.4	27.7	31.0	27.7	33.8
	SD	± 0.6	± 1.5	± 0.7	± 0.2	± 0.3	± 0.5	± 1.8	± 0.3	± 0.4
Ambient Extract	Mean	57.0	57.4	55.2	57.5	54.7	51.5	51.4	46.3	43.9
	SD	± 0.4	± 2.1	± 0.8	± 0.7	± 0.0	± 0.4	± 0.1	± 0.1	± 0.6
Microwave Extract	Mean	2.1	2.4	3.0	3.2	4.1	5.9	5.2	7.5	5.3
	SD	± 0.1	± 0.1	± 0.1	± 0.1	± 0.0	± 0.4	± 0.6	± 0.2	± 0.4
Total Extractable Residues	Mean	101.0	99.6	92.8	93.5	88.8	85.1	87.5	81.4	84.9
	SD	± 0.9	± 3.6	± 0.0	± 0.4	± 0.0	± 0.4	± 0.9	± 0.6	± 0.7
Carbon Dioxide	Mean	n.a.	0.7	1.7	1.6	2.3	2.4	3.0	3.7	4.5
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.1	± 0.1	± 0.1
Volatile Organic Compounds	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues	Mean	1.4	2.5	4.4	4.1	7.5	8.8	7.6	8.5	8.9
	SD	± 0.0	± 0.1	± 0.2	± 0.1	± 0.1	± 0.2	± 0.1	± 0.1	± 0.3
Total Recovery	Mean	102.4	102.8	98.6	99.2	98.6	96.3	98.1	93.6	98.3
	SD	± 0.9	± 3.7	± 0.2	± 0.3	± 0.1	± 0.6	± 0.8	± 0.4	± 0.4

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

B. Material Balance

Mean mass balances were 95.4% AR for Lamberton soil (range from 93.8 to 97.5% AR), 98.6% AR for Sarotti soil (range from 95.8 to 100.4% AR), 96.9% AR for Münster soil (range from 95.0 to 98.9% AR), 98.5% AR for Pikeville soil (range from 96.7 to 100.7% AR), 97.5% AR for Abington soil (range from 95.3 to 99.5% AR) and 98.8% AR for Lignieres soil (range from 93.6 to 102.8% AR).

The results confirm there were no significant losses of radioactivity during sample processing.

C. Extractable and Non-Extractable Residues

Desorbable residues in aqueous 0.01 M $CaCl_2$ solution decreased from DAT-0 to DAT-119/120 from 39.7 to 26.3% AR in Sarotti soil, from 23.3 to 17.3% AR in Pikeville soil and from 42.0 to 35.7% AR in Lignieres soil. In the other soils, desorbable residues remained almost constant, amounting to 15.5 and 16.8% AR (Lamberton soil), 34.6 and 35.9% AR (Münster soil) and 24.7 and 26.5% AR (Abington soil) at DAT-0 and DAT-119/120, respectively.

Total extractable residues (i.e. residues desorbed by aqueous 0.01 M CaCl2 solution and residues in organic soil extracts) decreased from DAT-0 to DAT-119/120 from 90.2 to 80.2% AR in Lamberton soil, from 96.4 to 79.6% AR in Sarotti soil, from 95.4 to 86.8% AR in Münster soil, from 96.1 to 80.6% AR in Pikeville soil, from 94.3 to 78.3% AR in Abington soil and from 101.0 to 84.9% AR in Lignieres soil.

Non-extractable residues (NER) increased from DAT-0 to DAT-119/120 from 3.8 to 13.3% AR in Lamberton soil, from 3.6 to 13.4% AR in Sarotti soil, from 0.8 to 6.5% AR in Münster soil, from 1.7 to 13.4% AR in Pikeville soil, from 2.6 to 10.2% AR in Abington soil and from 1.4 to 8.9% AR in Lignieres soil.

D. Volatile Radioactivity

The maximum amount of carbon dioxide formed was 3.2, 5.3, 3.8, 3.7, 8.1 and 4.5% AR in Lamberton, Sarotti, Münster, Pikeville, Abington and Lignieres soils, respectively by the end of the study (DAT-119/120). Formation of volatile organic compounds (VOC) was insignificant with values of ≤ 0.1% AR at all timepoints in all soils.

E. Degradation of Parent Compound

The amount of fluopicolide in the total soil extracts (i.e. in aqueous desorption solution and organic soil extracts) decreased from 88.0% at DAT-0 to 52.2% by the end of the study in Lamberton soil, 93.4 to 54.1% AR in Sarotti soil, from 90.9 to 50.4% AR in Münster soil, from 93.3 to 47.9% AR in Pikeville soil, from 91.9 to 45.9% AR in Abington soil and from 96.3 to 50.1% AR in Lignieres soil.

Degradation of fluopicolide was accompanied by the formation of two degradation products, M-01 (AE C653711) and M-03 (AE 0608000). M-01 was observed in all six soils and detected at a maximum of 34.8% AR in Lignieres soil at DAT-120. M-03 was observed in Pikeville soil only, at a maximum of 6.6% AR at DAT-85, exceeding 5% AR at two consecutive timepoints. M-01 and M-03 were identified by co-chromatography with analytical standards and by LC/MS/MS including accurate mass determination after isolation of the radiopeaks from soil desorption supernatants and extracts. The total unidentified residues amounted to a maximum of 4.6% AR and no single component exceeded 3.3% AR at any sampling interval in any soil.

F. Degradation Kinetics

Reported DT_{50} values of fluopicolide under aerobic conditions were 149, 131, 135, 114, 116 and 117 days in Lamberton, Sarotti, Münster, Pikeville, Abington and Lignieres soils, respectively. The experimental data was best described by a double first order in parallel (DFOP) kinetic model. Details are provided below in Table 7.1.2.1.1- 46.

Table 7.1.2.1.1- 46: Reported degradation rate of fluopicolide under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} (days)	DT_{90} (days)
Lamberton	DFOP	93.88	k1 0.513035 k2 0.003587 g 0.148002	1.6	0.0011 1.55e-11 -	- - 0.175	- - 0.013634	149	597
Sarotti	DFOP	100.0	k1 5.259e-01 k2 4.189e-03 g 1.351e-01	1.4	0.00327 5.43e-12 -	- - 1.065e-01	- - 0.164	131	515
Münster	DFOP	96.20	k1 2.085e-01 k2 4.134e-03 g 1.282e-01	0.7	0.000194 1.59e-13 -	- - 1.084e-01	- - 0.148	135	524
Pikeville	DFOP	98.61	k1 2.197e-01 k2 4.400e-03 g 1.750e-01	1.4	0.000323 4.69e-11 -	- - 1.455e-01	- - 0.205	114	480
Abington	DFOP	97.09	k1 4.018e-01 k2 4.803e-13 g 1.282e-01	1.2	0.00197 2.44e-13 -	- - 1.037e-01	- - 0.153	116	451
Lignieres	DFOP	102.4	k1 5.610e-01 k2 4.787e-03 g 1.242e-01	1.5	0.00767 2.68e-12 -	- - 9.340e-02	- - 0.155	117	453

In addition, the experimental data for the degradation of fluopicolide, M-01 and M-03 has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting DT_{50} values for trigger endpoints for fluopicolide are summarised below in Table 7.1.2.1.1- 47. Best fit kinetics are highlighted in bold. The results are very similar to reported best fit DT_{50} values.

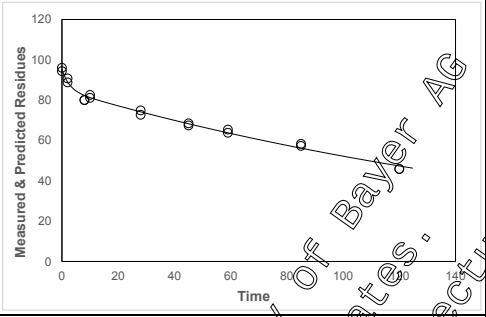
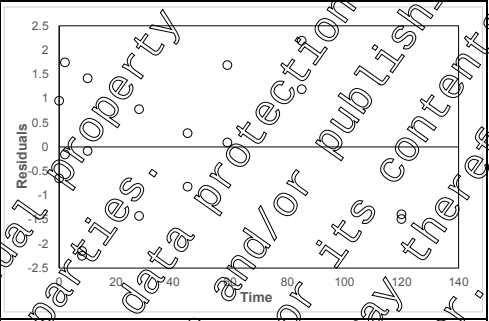
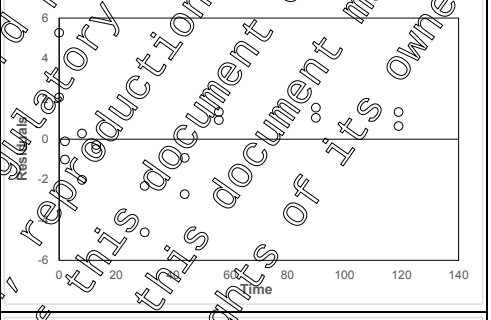
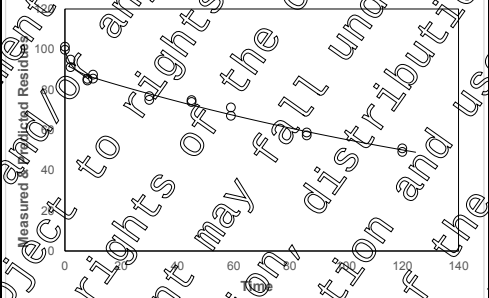
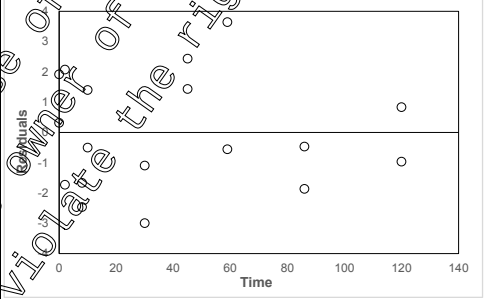
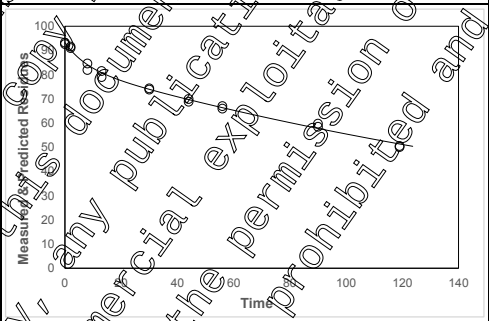
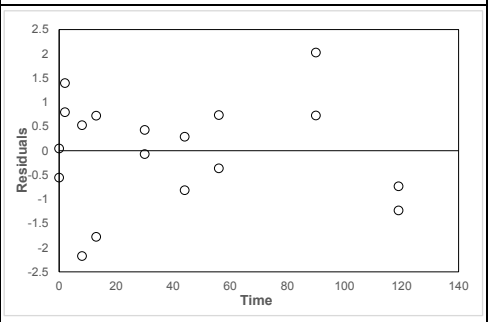
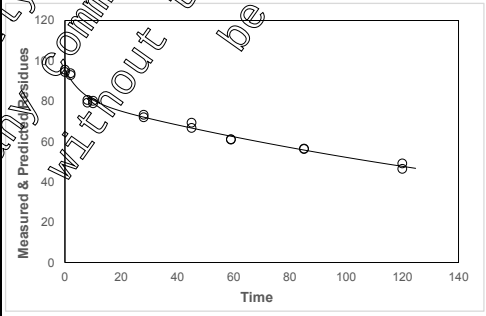
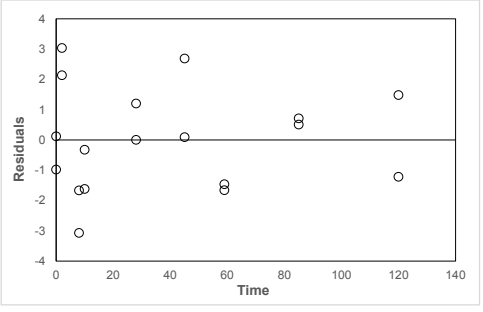
Table 7.1.2.1.1- 47: Re-evaluation of degradation rate of fluopicolide under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

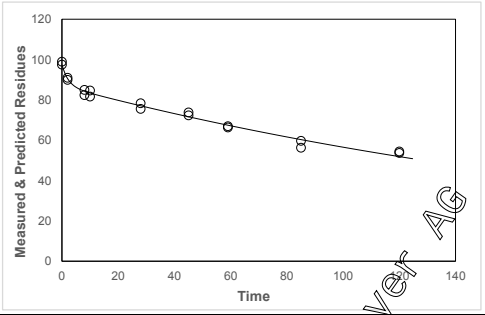
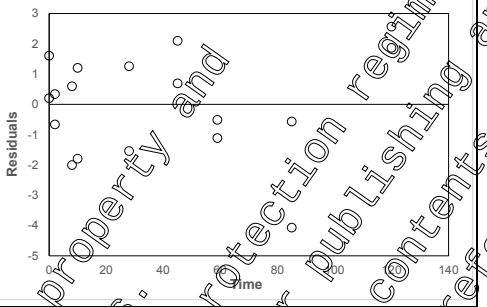
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington 2, [redacted] (2019)	SFO	90.9	k 0.006245	3.81	1.46E-15	0.005376	0.007	111	368.7
	FOMC	95.39	α 0.2186 β 9.099	3.94	n.r. n.r.	0.1563 2.889	0.281 15.31	207.6	>10000
	DFOP	**95.15**	**k1 0.3704 k2 0.004877 g 0.1058**	**1.41**	**9.000341 <2e-16 n.r.**	**0.1787 0.004513 0.08236**	**0.562 0.005 0.129**	**119.2**	**449.2**
Lamberton, [redacted] (2019)	SFO	84.39	k 0.004403	3.66	1.22E-13	0.003681	0.005	157.4	522.9
	FOMC	86.65	α 0.3865 β 47.11	2.69	n.r. n.r.	0.1507 0.3272	0.622 93.9	236.1	10000
	DFOP	**87.63**	**k1 0.2445 k2 0.003936 g 0.06748**	**2.45**	**0.0411 5.19E-16 n.r.**	**-0.02224 0.00342 0.04309**	**0.511 0.004 0.092**	**158.3**	**567.2**
Lignieres, [redacted] (2019)	SFO	93.62	k 0.005431	3.33	<2e-16	0.004742	0.006	127.6	424
	FOMC	94.36	α 1.654 β 258.7	3.31	n.r. n.r.	-2.002 -406.4	5.311 923.9	134.6	781.9
	DFOP	**99.48**	**k1 0.4856 k2 0.004903 g 0.09175**	**1.63**	**0.021 <2e-16 n.r.**	**0.03702 0.004464 0.06356**	**0.934 0.005 0.12**	**121.7**	**450**
Münster, [redacted] (2019)	SFO	90.46	k 0.005501	3.04	<2e-16	0.00479	0.006	126	418.6
	FOMC	93.39	α 0.2806 β 20.36	2.18	n.r. n.r.	0.2075 9.798	0.354 30.91	220.3	>10000
	DFOP	**93.25**	**k1 0.1305 k2 0.004075 g 0.106**	**0.925**	**1.35E-06 <2e-16 n.r.**	**0.08613 0.003707 0.08059**	**0.175 0.004 0.131**	**142.6**	**537.6**
Pikeville, [redacted] (2019)	SFO	89.87	k 0.006034	4.41	1.02E-15	0.005016	0.007	114.9	381.6
	FOMC	94.83	α 0.2662 β 13.52	2.96	n.r. n.r.	0.1778 3.257	0.355 23.79	169.2	>10000
	DFOP	**95.58**	**k1 0.1673 k2 0.004466 g 0.1468**	**1.78**	**3.80E-05 <2e-16 n.r.**	**0.09185 0.003877 0.1097**	**0.243 0.005 0.184**	**119.7**	**480**
Sarotti 2, [redacted] (2019)	SFO	91.3	k 0.006094	3.6	2.05E-14	0.004313	0.006	136.1	452
	FOMC	95.32	α 0.2553 β 17.34	2.91	n.r. n.r.	0.1582 3.332	0.352 31.34	244.6	>10000
	DFOP	**97.39**	**k1 0.4303 k2 0.004301 g 0.1066**	**1.5**	**0.00375 <2e-16 n.r.**	**0.1361 0.003886 0.07785**	**0.724 0.005 0.135**	**135**	**509.2**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.1.1- 48: Degradation of fluopicolide under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Abington 2 DFOP [redacted] (2019)		
Lamberton DFOP [redacted] (2019)		
Lignieres DFOP [redacted] (2019)		
Münster DFOP [redacted] (2019)		
Pikeville DFOP [redacted] (2019)		

Soil Model Reference	Modelled vs observed	Residuals
Sarotti 2 DFOP [redacted] (2019)		

G: Time-Dependent Sorption

The sorption of fluopicolide in soil increased steadily with time. Apparent sorption coefficients ($K_{d,app}$) increased from DAT-0 to DAT-120 in all six soils. The values are summarised in Table 7.1.2.1.1-49.

Table 7.1.2.1.1- 49: Apparent sorption coefficients ($K_{d, app}$) (expressed as mL/g)

DAT	Mean SD	Lamberton	Sarotti	Münster	Pikeville	Abington	Lignieres
0	Mean	15.21	4.05	5.47	9.25	8.46	4.29
	SD	± 0.01	± 0.00	± 0.01	± 0.01	± 0.01	± 0.00
2	Mean	17.95	4.91	6.4	10.92	9.21	4.7
	SD	± 0.02	± 0.02	± 0.01	± 0.02	± 0.04	± 0.00
8	Mean	18.16	5.16	7.35	11.91	9.84	5.49
	SD	± 0.00	± 0.03	± 0.03	± 0.02	± 0.07	± 0.05
10/13	Mean	17.93	5.67	7.95	12.87	10.06	5.97
	SD	± 0.06	± 0.04	± 0.00	± 0.01	± 0.01	± 0.02
28/30	Mean	24.24	7.03	8.53	15.62	11.28	7.43
	SD	± 0.01	± 0.02	± 0.02	± 0.03	± 0.01	± 0.01
44/45	Mean	27.66	7.46	8.9	19.27	11.91	6.82
	SD	± 0.08	± 0.00	± 0.07	± 0.01	± 0.02	± 0.03
56/59	Mean	29.85	8.32	10.11	20.2	13.19	8.32
	SD	± 0.01	± 0.01	± 0.05	± 0.03	± 0.07	± 0.03
85/90	Mean	39.86	43	12.44	24.52	12.36	12.2
	SD	± 0.07	± 0.01	± 0.04	± 0.02	± 0.01	± 0.02
119/120	Mean	38.54	8.72	12.57	21.94	13.7	10.05
	SD	± 0.05	± 0.04	± 0.01	± 0.02	± 0.02	± 0.03
Factor [A]		2.53	2.15	2.30	2.37	1.62	2.34
Mean Factor		2.22					

Apparent Sorption Coefficients ($K_{d, app}$) are called Time-Dependent Sorption Ratios (R_{TDS}) in the report.
[A] Calculated as $K_{d, app}$ DAT-119/120 divided by $K_{d, app}$ DAT-0

The results are more fully discussed under Section 7.1.3.2 (see KCA 7.1.3.2/05).

III. Conclusion

Fluopicolide was moderately degraded and mineralized in six tested soils; Lamberton, Sarotti, Münster, Pikeville, Abington and Lignieres, under aerobic conditions at 20°C in the dark. Reported best fit DT_{50} values ranged from 114 to 149 days in the tested soils. Re-evaluated best fit DT_{50} values were very similar, ranging from 119.2 to 158.3 days.

The primary objective of the study was to investigate the sorption of fluopicolide, determined under equilibrium conditions, following its aging in soil under aerobic conditions in the dark under laboratory conditions. The time-dependent sorption ratio increased throughout the incubation period (119/120 days) by a factor of 1.62 to 2.53 in the six soils tested.

Formation of carbon dioxide was significant (up to 8.1% AR) by the end of the study indicating the potential for complete mineralization of fluopicolide and its degradation products. One major degradation product, M-01 (AE C653711) was identified at a maximum of 34.8% AR. In addition a second degradation product M-03 (AE 0608000) was identified at a maximum of 6.6% AR, exceeding 5% AR at two consecutive timepoints.

Formation of non-extractable residues (NER) was up to 13.4% AR at study end, which is an indication for biotic degradation of fluopicolide.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002), US EPA 835.4100 / 835.4200 (2008) and in parts, where relevant, to OECD 106 (2000). The study is considered valid to assess the aerobic degradation of [phenyl-U-^{14}C]-fluopicolide in soil.

The study has been designed so that it is valid to assess the changes in sorption of fluopicolide with time in accordance with guidance provided by Food and Environment Research Agency (2019) on conducting aged sorption studies. A kinetic assessment of the time dependent sorption (TDS) parameters is provided in KCA 7.1.3.2.

Data Point:	KCA 7.1.2.1.1/10
Report Author:	[redacted]
Report Year:	2020
Report Title:	Fluopicolide (FLC) and metabolites - Kinetic evaluation of degradation behaviour in soil under aerobic laboratory conditions
Report No:	VC/19/041E
Document No:	M-685680-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	None
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of fluopicolide and its metabolites under laboratory aerobic conditions has been investigated in a total of nineteen studies (KCA 7.1.1.1/01, M-241049-01-1, [redacted] 2003a; KCA 7.1.1.1/02, M-201230-02-1, [redacted] 2003; KCA 7.1.2.1.1/01, M-241052-01-1, [redacted] 2003c; KCA 7.1.2.1.1/02, M-241051-01-1, [redacted] 2003b; KCA 7.1.2.1.1/03, M-241053-01-1, [redacted] 2003d; KCA 7.1.2.1.1/07, M-555570-01-1, [redacted] 2016; KCA 7.1.2.1.1/08, M-550687-01-1, [redacted] 2016; KCA 7.1.2.1.1/09, M-655056-01-1, [redacted] 2019; KCA 7.1.2.2/01, M-234320-01-1, [redacted] 2002; KCA 7.1.2.2/02, M-241188-01-1, [redacted] 2003; KCA 7.1.2.2/03, M-219824-01-1, [redacted] 2003; KCA 7.1.2.2/04, M-241410-01-2, [redacted] 2003a; KCA 7.1.2.2/05, M-241411-01-2, [redacted] 2003b; KCA 7.1.2.2/06, M-234349-01-2, [redacted], 2003; KCA 7.1.2.2/08, M-585202-01-1, [redacted] 2017; KCA 7.1.2.2/09, M-581364-01-1, [redacted] 2017; KCA 7.1.2.2/10, M-565219-01-1, [redacted] 2016a; KCA 7.1.2.2/11, M-565223-01-1, [redacted] 2016b and KCA 7.1.2.2/12, M-565224-01-1, [redacted] 2016c).

Kinetic analysis was conducted for these studies to derive parameters suitable for use as trigger endpoints and modelling endpoints. The metabolites included in the analysis were: M-01 (AE C653711, BAM), M-02 (AE C657188, PCA), M-03 (AE 0608000), M-05 (AE 1344122), M-10 (AE 1344123), M-11/12, M-13, M-14 (AE 1388273), M-15 (AE 1413903) and M-20 (BCS-BX16566).

The model fits and statistical evaluation of the results were carried out with the KinGUI software tool, 2.1. The selection of the most appropriate kinetic model was based on a detailed analysis including visual assessment, χ^2err statistics, randomness of residuals, and t-test significance, following current FOCUS guidance (2006, 2014a).

The mean $DegT_{50}$ values (20 °C and pF2) and molar fractions selected for use as modelling endpoints are summarised below.

Compound	Number of datasets [A]	Number of soils [B]	Geometric Mean $DegT_{50}$ normalised to 20°C and pF2 (d)	Arithmetic Mean ffm
Fluopicolide	22 [C]	16	181.6 [D]	-
M-01	26 [C]	18	569.5 [D]	0.850 (from M-03) 0.757 [E] (from parent)
M-02	7	7	1.6	Not applicable [K]
M-03	9	7	17.9 [F,G] / 0.19 [H]	0.53 [I] / - [J] (from parent)
M-05	13	7	25.2	0.153 (from M-02)
M-10	13	7	35.4	0.129 (from M-02)
M-11/M-12	2	2	87.6	0.044 (from M-02)
M-13	3	3	20.7	0.049 (from M-02)
M-14	5	3	9.4	1 (from M-20)
M-15	4	4	144.8	Not applicable [K]
M-20	6	6	6.1	0.021 (from M-02) 0.559 (from M-05)

[A] Where appropriate results from different radiolabels in the same soils treated as replicates for kinetic fits
[B] Some soils were used more than once
[C] Study performed at 10°C not included in modelling endpoint.
[D] Geometric mean calculated of $DegT_{50}$ values from Lamberton soils prior to calculation of overall geometric mean.
[E] Arithmetic mean calculated of formation fractions from Lamberton soil prior to calculation of overall arithmetic mean.
[F] Geometric mean calculated for Munster soils prior to calculation of overall value
[G] Geometric mean soils with pH < 6
[H] Geometric mean soils with pH > 6
[I] Arithmetic mean soils with pH < 6
[J] Arithmetic mean soils with pH > 6
[K] Degradation rates for M-02 and M-15 were derived from metabolite dosed studies.

Metabolite M-01 can be formed either directly from fluopicolide, or from metabolite M-03, which itself is formed from fluopicolide. However, formation *via* M-03 was only observed in a few soils. The overall molar fraction of fluopicolide that degrades to M-01, either directly or *via* M-03 has been calculated for each soil ($ffm_{FLC \rightarrow M\text{-}01} = [ffm_{FLC \rightarrow M\text{-}03} \times ffm_{M\text{-}03 \rightarrow M\text{-}01}]$), to simplify modelling of M-01 formation if required. The arithmetic mean overall formation fraction of M-01 from fluopicolide is 0.80.

I. MATERIALS AND METHODS

The objective of this study was a kinetic evaluation of the degradation behaviour of fluopicolide and eleven of its metabolites (M-01, M-02, M-03, M-05, M-10, M-11/12, M-13, M-14, M-15 and M-20) in soils under laboratory conditions in the dark. This evaluation was performed to derive kinetic parameters suitable for use as trigger endpoints and modelling endpoints.

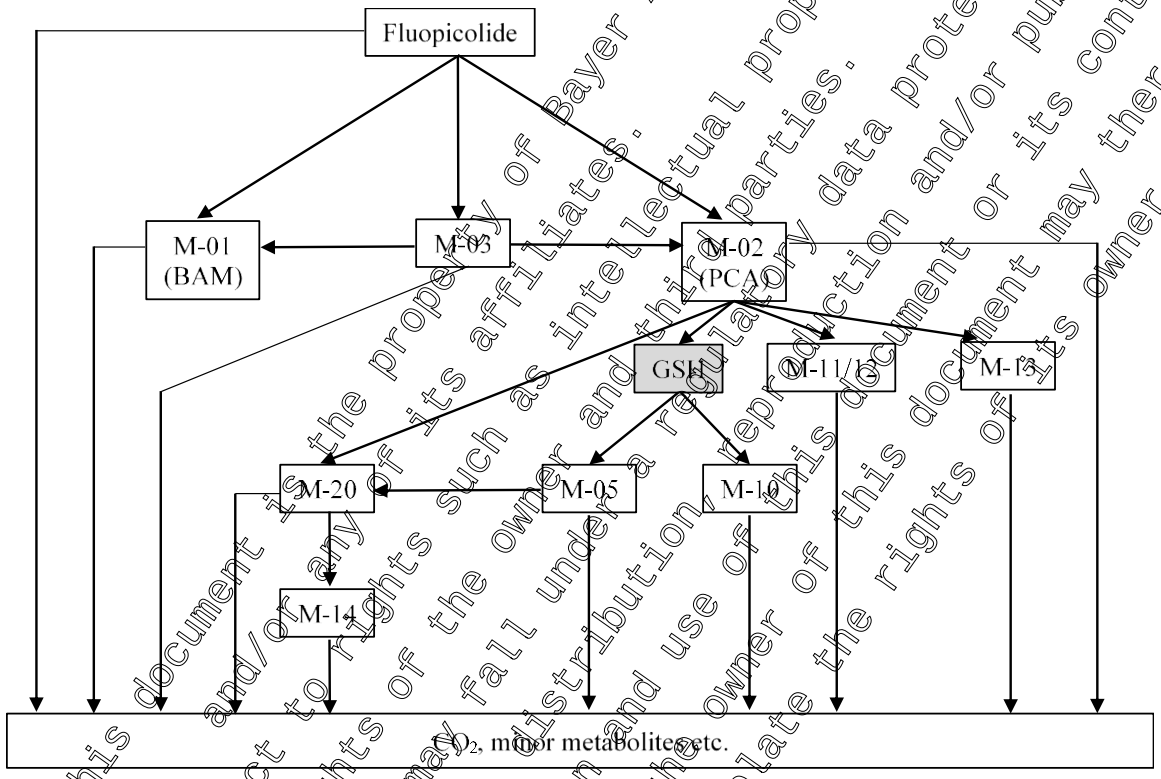
The modelling analysis was based on residue data from nineteen studies covering a range of soil types. The kinetic parameters that provided the most appropriate fit to the measured data were identified, based on a mathematical optimisation algorithm and a visual/statistical assessment. The model fit and statistical evaluation of the results were carried out with the KinGUI software tool (version 2.1).

A. Degradation scheme

The degradation scheme for fluopicolide and its metabolites modelled in the kinetic evaluation is given in Figure 7.1.2.1.1- 1 (for metabolite M-15, the only data available were from an M-15-applied study, therefore this metabolite is not included in the scheme).

The compartment "GSH" denotes a ghost compartment, representing a glutathione conjugate. Initial fits were conducted without this compartment, and it was subsequently added only where its inclusion improved the final fits.

Figure 7.1.2.1.1- 1 Simulated degradation scheme for fluopicolide and its metabolites



B. Experimental data

1. Fluopicolide data

The aerobic degradation of fluopicolide has been measured in eight reports (KCA 7.1.1.1/01, M-241049-01-1, [redacted] 2003a; KCA 7.1.1.1/02, M-201230-02-1, [redacted] 2003; KCA 7.1.2.1.1/01, M-241052-01-1, [redacted] 2003c; KCA 7.1.2.1.1/02, M-241051-01-1, [redacted] 2003b; KCA 7.1.2.1.1/03, M-241053-01-1, [redacted] 2003d; KCA 7.1.2.1.1/07, M-555570-01-1, [redacted] 2016; KCA 7.1.2.1.1/08, M-550687-01-1, [redacted] 2016 and KCA 7.1.2.1.1/09, M-655056-01-1, [redacted] 2019) in a total of 23 datasets in 16 different soils (2 datasets at 25 °C, 20 datasets at 20°C and 1 dataset at 10 °C). Table 7.1.2.1.1- 50 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 50: Test soils used for fluopicolide

#	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	KCA 7.1.1.1/02 M-201230-02-1 [redacted] 2003	Munster	Germany	Loamy sand	20	4.9	0.7
2		Sarrotti	Germany	Silty clay loam	20	7.4	0.9
3	KCA 7.1.1.1/01 M-241049-01-1, [redacted] 2003a	Abington (non-sterile)	UK	Sandy loam	20	7.5	2.2
4	KCA 7.1.2.1.1/02 M-241051-01-1 [redacted] 2003b	Lamberton	USA	Loam	20	5.6	3.3
5	KCA 7.1.2.1.1/01 M-241052-01-1 [redacted] 2003c	Lamberton	USA	Sandy clay loam	25	5.9	3.8
6		Pikeville	USA	Loamy sand	25	5.7	1.6
7	KCA 7.1.2.1.1/03 M-241053-01-1 [redacted] 2003d	Abington [A]	UK	Sandy loam	10	7.2	2.7
8	KCA 7.1.2.1.1/08 M-550687-01-1 [redacted] 2016a	Albaro/Marconcini	Italy	Silty clay	20	7.2	2.1
9		Great Chishill	UK	Clay	20	7.3	2.1
10		H[redacted]	Germany	Silt loam	20	6.1	0.7
11		Mas du Coq	France	Clay loam	20	7.6	0.9
12		Parcey Meslay	France	Loam	20	6.7	1.3
13		Vilobi d'Onyar	Spain	Sandy loam	20	6.3	0.8
14	KCA 7.1.2.1.1/07 M-555570-01-1 [redacted] 2016b	Dollendorf II	Germany	Sandy loam	20	6.5	1.5
15		H[redacted]	Germany	Clay loam	20	7.3	4.8
16		L[redacted]	Germany	Loam	20	5.0	1.8
17		L[redacted]	Germany	Silt loam	20	6.1	1.9
18	KCA 7.1.2.1.1/09 M-655056-01-1 [redacted] 2019	Abington 2	UK	Sandy loam	20	7.3	2.6
19		Lamberton	USA	Loam	20	5.6	2.6
20		Lignieres	France	Sandy loam	20	5.7	0.8
21		Munster	Germany	Loamy sand	20	5.6	1.2
22		Pikeville	USA	Loamy sand	20	4.5	1.8
23		Sarotti 2	Germany	Silty clay loam	20	6.9	1.4

[A] Study performed at 10°C. Data used for trigger endpoint only. Not used for modelling endpoint.

2. M-01 data

The aerobic degradation of M-01 has been evaluated in all studies in which fluopicolide was applied (2 datasets at 25 °C, 20 datasets at 20°C and 1 dataset at 10 °C). In addition, the aerobic degradation of the metabolite has been studied in two soils in which M-01 was applied (KCA 7.1.2.1.2/01, M-234320-01-1, [redacted] 2002) and in two soils in which M-03 was applied (KCA 7.1.2.1.2/02, M-241188-01-1, [redacted] 2003). A total of 27 datasets in 18 different soils (4 datasets at 25 °C, 22 datasets at 20°C and 1 dataset at 10 °C) were evaluated. Table 7.1.2.1.1- 51 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 51: Test soils used for M-01

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC. [%]
1	Fluopicolide	KCA 7.1.1.1/02 M-201230-02-1 [redacted] 2003	Munster	Germany	Loamy sand	20	4.9	0.7
2			Sarrotti	Germany	Silty clay loam	20	7.4	0.9
3		KCA 7.1.1.1/01 M-241049-01-1, [redacted] 2003a	Abington (non-sterile)	UK	Sandy loam	20	7.5	2.2
4		KCA 7.1.2.1.1/02 M-241051-01-1 [redacted] 2003b	Lamberton	USA	Loam	20	5.6	3.3
5		KCA 7.1.2.1.1/01 M-241052-01-1 [redacted] 2003c	Lamberton	USA	Sandy clay loam	25	5.9	2.5
6			Pikeville	USA	Loamy sand	25	5.7	1.6
7		KCA 7.1.2.1.1/03 M-241053-01-1 [redacted] 2003d	Abington 2	UK	Sandy loam	10	7.2	2.7
8		KCA 7.1.2.1.1/08 M-550687-01-1 [redacted] 2016a	Albaro Marcomcini	Italy	Silty clay	20	7.2	2.1
9			Great Chishill	UK	Clay	20	7.3	2.1
10			H[redacted]	Germany	Silt loam	20	6.1	0.7
11			Mas du Coq	France	Clay loam	20	7.6	0.9
12			Parcey Meslay	France	Loam	20	6.7	1.3
13			Vilobi d'Onyar	Spain	Sandy loam	20	6.3	0.8
14		KCA 7.1.2.1.1/07 M-555570-01-1 [redacted] 2016b	Dollendorf II	Germany	Sandy loam	20	6.5	1.5
15			H[redacted]	Germany	Clay loam	20	7.3	4.8
16			L[redacted]	Germany	Loam	20	5.0	1.8
17			L[redacted]	Germany	Silt loam	20	6.1	1.9
18		KCA 7.1.2.1.1/09 M-653056-01-1 [redacted] 2019	Abington 2	UK	Sandy loam	20	7.3	2.6
19			Lamberton	USA	Loam	20	5.6	2.6
20			Lignieres	France	Sandy loam	20	5.7	0.8
21			Münster	Germany	Loamy sand	20	5.6	1.2
22			Pikeville	USA	Loamy sand	20	4.5	1.8
23			Sarotti 2	Germany	Silty clay loam	20	6.9	1.4
24	M-01	KCA 7.1.2.1.2/01 M-234320-01-1 [redacted] 2002	Bethany	USA	Sandy loam	25	4.8	1.6 [B]
25			North Dakota	USA	Sandy loam	25	7.7	9.6 [B]
26	M-03	KCA 7.1.2.1.2/02 M-241188-01-1 [redacted] 2003	Münster	Germany	Loamy sand	20	4.9	1.8
27			Pikeville	USA	Sandy loam	20	5.4	1.1

[A] Study performed at 10°C. Data used for trigger endpoint only. Not used for modelling endpoint.
[B] Organic matter

3. M-02 data

The aerobic degradation of M-02 has been evaluated in two studies in which the metabolite was applied (7 soils at 20°C). Table 7.1.2.1.1- 52 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 52: Test soils used for M-02

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	M-02	KCA 7.1.2.1.2/03 M-219824-01-1, [redacted] 2003	Abington	UK	Sandy loam	20	7.2	2.6
2			Münster	Germany	Loamy sand	20	5.4	1.1
3			Sarotti	Germany	Silt loam	20	7.5	1.3
4		KCA 7.1.2.1.2/09, M-581364-01-1, [redacted] 2017	Dollendorf	Germany	Loam	20	6.9	5.6
5			H[redacted]	Germany	Silt loam	20	5.9	1.8
6			L[redacted]	Germany	Loamy sand	20	5.2	1.8
7			[redacted]	Germany	Sandy loam	20	4.9	2.1

4. M-03 data

The aerobic degradation of M-03 has been evaluated in three acidic soils (KCA 7.1.1.1/02, M-201230-02-1, [redacted] 2003; KCA 7.1.2.1.1/01, M-241052-01-1, [redacted] 2003c and KCA 7.1.2.1.1/09, M-655056-01-1, [redacted] 2019) in which fluopicolide was applied (1 dataset at 25 °C, 2 datasets at 20°C). In addition, the aerobic degradation of the metabolite has been studied in a two reports in which M-03 was applied (KCA 7.1.2.1.2/02, M-241188-01-1, [redacted] 2003 and KCA 7.1.2.1.2/10, M-565219-01-1, [redacted] 2016a). A total of nine datasets in seven different soils (1 dataset at 25 °C, 8 datasets at 20°C) were evaluated. Table 7.1.2.1.1- 53 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 53: Test soils used for M-03

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	Fluopicolide	KCA 7.1.1.1/02 M-201230-02-1 [redacted] 2003	Munster	Germany	Loamy sand	20	4.9	0.7
2		KCA 7.1.2.1.1/01 M-241052-01-1 [redacted] 2003c	Lamberton	USA	Sandy clay loam	25	5.9	3.5
3		KCA 7.1.2.1.1/09 M-655056-01-1 [redacted] 2019	Pikeville	USA	Loamy sand	20	4.5	1.8
4	M-03	KCA 7.1.2.1.2/02 M-241188-01-1 [redacted] 2003	Abington	UK	Sandy loam	20	7.2	3.2
5			Münster	Germany	Loamy sand	20	4.9	1.8
6			Pikeville	USA	Sandy loam	20	5.4	1.1
7			Sarotti	Germany	Silt loam	20	7.1	2.0
8		KCA 7.1.2.1.2/10 M-565219-01-1 [redacted] 2016a	Brierlow	UK	Silt loam	20	5.3	4.5
9			H[redacted]	Germany	Silt loam	20	6.0	2.0

5. M-05 data

The aerobic degradation of M-05 has been evaluated in both studies in which M-02 was applied (7 soils at 20°C). In addition, the aerobic degradation of M-05 has been evaluated in two studies in which the metabolite was applied (6 soils at 20°C). A total of 13 datasets in seven different soils (all at 20°C) were evaluated. Table 7.1.2.1.1- 54 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 54: Test soils used for M-05

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	M-02	KCA 7.1.2.1.2/03 M-219824-01-1, [redacted] 2003	Abington	UK	Sandy loam	20	7.2	2.6
2			Münster	Germany	Loamy sand	20	5.4	1.1
3			Sarotti	Germany	Silt loam	20	7.5	1.3
4		KCA 7.1.2.1.2/09, M-581364-01-1, [redacted] 2017	Dollendorf	Germany	Loam	20	6.9	5.6
5			H[redacted]	Germany	Silt loam	20	5.9	1.8
6			L[redacted]	Germany	Loamy sand	20	5.2	1.8
7			[redacted]	Germany	Sandy loam	20	4.9	2.1
8	M-05	KCA 7.1.2.1.2/04 M-241410-01-2 [redacted] 2003a	Abington	UK	Sandy loam	20	7.2	2.6
9			Münster	Germany	Loamy sand	20	5.4	1.1
10			Sarotti	Germany	Silt loam	20	7.5	1.3
11		KCA 7.1.2.1.2/11 M-565223-01-1 [redacted] 2016b	H[redacted]	Germany	Silt loam	20	5.8	1.9
12			L[redacted]	Germany	Loamy sand	20	5.3	1.5
13			L[redacted]	Germany	Sandy loam	20	5.1	1.9

6. M-10 data

The aerobic degradation of M-10 has been evaluated in both studies in which M-02 was applied (7 soils at 20°C). In addition, the aerobic degradation of M-10 has been evaluated in two studies in which the metabolite was applied (6 soils at 20°C). A total of 13 datasets in seven different soils (all at 20°C) were evaluated. Table 7.1.2.1.1- 55 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 55: Test soils used for M-10

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	M-02	KCA 7.1.2.1.2/03 M-219824-01-1 [redacted] 2003	Abington	UK	Sandy loam	20	7.2	2.6
2			Münster	Germany	Loamy sand	20	5.4	1.1
3			Sarotti	Germany	Silt loam	20	7.5	1.3
4		KCA 7.1.2.1.2/09, M-581364-01-1: [redacted] 2017	Dollendorf	Germany	Loam	20	6.9	5.6
5			H[redacted]	Germany	Silt loam	20	5.9	1.8
6			L[redacted]	Germany	Loamy sand	20	5.2	1.8
7			W[redacted]	Germany	Sandy loam	20	4.9	2.1
8	M-10	KCA 7.1.2.1.2/05, M-241411-01-2 [redacted] 2003b	Abington	UK	Sandy loam	20	7.2	2.6
9			Münster	Germany	Loamy sand	20	5.4	1.1
10			Sarotti	Germany	Silt loam	20	7.5	1.3
11		KCA 7.1.2.1.2/12 M-565224-01-1 [redacted] 2016c	H[redacted]	Germany	Silt loam	20	5.8	1.9
12			L[redacted]	Germany	Loamy sand	20	5.3	1.5
13			L[redacted]	Germany	Sandy loam	20	5.1	1.9

7. M-11/M-12 data

The aerobic degradation of M-11/M-12 has been evaluated in two soils in one of the studies in which M-02 was applied. Table 7.1.2.1.1- 56 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 56: Test soils used for M-11/M-12

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	M-02	KCA 7.1.2.1.2/03 M-219824-01-1, [redacted] 2003	Abington	UK	Sandy loam	20	7.2	2.6
2			Münster	Germany	Loamy sand	20	5.4	1.1

8. M-13 data

The aerobic degradation of M-13 has been evaluated in three soils in one of the studies in which M-02 was applied. Table 7.1.2.1.1- 57 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 57: Test soils used for M-13

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	M-02	KCA 7.1.2.1.2/03 M-219824-01-1, [redacted] 2003	Abington	UK	Sandy loam	20	7.2	2.6
2			Münster	Germany	Loamy sand	20	5.4	1.1
3			Sarotti	Germany	Silt loam	20	7.5	1.3

9. M-14 data

The aerobic degradation of M-14 has been evaluated in two soils form one of the studies in which M-05 was applied. In addition, the aerobic degradation of M-14 has been evaluated in a study in which the metabolite was applied (3 soils at 20°C). A total of five datasets in three different soils (all at 20°C) were evaluated. Table 7.1.2.1.1- 58 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 58: Test soils used for M-14

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	M-05	KCA 7.1.2.1.2/04 M-241410-01-2 [redacted] 2003a	Abington	UK	Sandy loam	20	7.2	2.6
2			Sarotti	Germany	Silt loam	20	7.5	1.3
3	M-14	KCA 7.1.2.1.2/06 M-244149-01-2 [redacted] 2003	Abington	UK	Sandy loam	20	7.2	2.6
4			Münster	Germany	Loamy sand	20	5.4	1.1
5			Sarotti	Germany	Silt loam	20	7.5	1.3

10. M-15 data

The aerobic degradation of M-15 has been evaluated in a study in which the metabolite was applied (four soils at 20°C). Table 7.1.2.1.1- 59 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 59: Test soils used for M-15

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	M-15	KCA 7.1.2.1.2/08, M-585202-01-1, [redacted] 2017	Dollendorf	Germany	Clay loam	20	7.3	4.9
2			H[redacted]	Germany	Silt loam	20	6.0	1.8
3			L[redacted]	Germany	Sandy loam	20	6.0	1.5
4			[redacted]	Germany	Sandy loam	20	5.3	1.9

11. M-20 data

The aerobic degradation of M-20 has been evaluated in one study in which M-02 was applied and one in which M-05 was applied. A total of 6 soils (all at 20°C) were evaluated. Table 7.1.2.1.1- 60 summarises the physico-chemical properties of the test soils used.

Table 7.1.2.1.1- 60: Test soils used for M-20

#	Applied compound	Report	Soil	Source	Texture (USDA)	Temp (°C)	pH	OC [%]
1	M-02	KCA 7.1.2.1.2/09, M-581364-01-1, [redacted] 2017	H[redacted]	Germany	Silt loam	20	5.9	1.8
2			L[redacted]	Germany	Loamy sand	20	5.2	1.8
3			[redacted]	Germany	Sandy loam	20	4.9	2.1
4	M-05	KCA 7.1.2.1.2/04 M-240410-01-2 [redacted] 2003a	Abington	UK	Sandy loam	20	7.2	2.6
5			Münster	Germany	Loamy sand	20	5.4	1.1
6			Sarotti	Germany	Silt loam	20	7.5	1.3

C. Data pre-processing

Residue data were checked for consistency and obvious outliers, and where outliers were removed this has been indicated clearly in this report. Experimental data sets and data points were weighted equally in the kinetic analysis. True replicates were used individually in the optimisations, while analytical replicates were averaged prior to curve fitting. For studies where degradation was investigated using multiple radiolabel positions, and similar behaviour was observed for each, these radiolabels have been considered as true replicates, and included together in a single optimisation.

Following the FOCUS Kinetics guidance (FOCUS 2006, 2014a), reported residues below the Limit of Quantification (LOQ) or Limit of Detection (LOD) were adjusted as follows:

- Residues between LOD and LOQ were set to the measured values, if given in the study report, or 0.5 (LOQ + LOD) otherwise.
- Samples <LOD just after a detectable amount were set to ½ LOD, with all subsequent samples <LOD omitted unless later samples >LOQ were reported.
- For metabolites, samples <LOD before the formation phase were also adjusted. The last point before the first detectable amount of metabolite was set to ½ LOD, with prior non-detects omitted.
- All mass at t=0 was assumed to be the applied compound, therefore initial residues for the applied compound were set to the total recovery at t=0 multiplied, where applicable, by the radiochemical purity of the test solution. Initial metabolite residues were set to 0.

II. RESULTS AND DISCUSSION

The endpoints derived for fluopicolide and each of its metabolites are summarised in Table 7.1.2.1.1-61 to Table 7.1.2.1.1- 71 for modelling endpoints and in Table 7.1.2.1.1- 72 to Table 7.1.2.1.1- 82 for trigger endpoints.

A. Modelling endpoints

Table 7.1.2.1.1- 61: Summary of modelling endpoints derived for fluopicolide

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)
Fluopicolide	[redacted] 2003	Münster	SFO	212.0	212.0
		Sarotti	SFO	191.2	191.2
	[redacted] 2003a	Abington (non-sterile)	SFO	348.0	340.2
	[redacted] 2003b	Lamberton	SFO	1290.0	1037.9
	[redacted] 2003c	Lamberton	SFO	358.0	395.8
		Pikeville	DFOP	612.9[a] / 30.1[b]	616.0[a] / 30.3[b]
	[redacted] 2016a	Albaro Marcomcini	DFOP	146.2[a] / 2.8[b]	146.2[a] / 2.8[b]
		Great Chishill	DFOP	312.4[a] / 2.7[b]	312.4[a] / 2.7[b]
		H[redacted]	DFOP	155.5[a] / 7.2[b]	155.5[a] / 7.2[b]
		Mas du Coq	DFOP	216.7[a] / 10.5[b]	193.7[a] / 9.4[b]
		Parcey Meslay	DFOP	202.5[a] / 8.1[b]	202.5[a] / 8.1[b]
		Vilobi d'Onyar	DFOP	93.5[a] / 7.8[b]	93.5[a] / 7.8[b]
	[redacted] 2016b	Dollendorf II	DFOP	111.4[a] / 0.6[b]	111.4[a] / 0.6[b]
		H[redacted]	DFOP	137.7[a] / 4.2[b]	137.7[a] / 4.2[b]
		L[redacted]	DFOP	141.3[a] / 6.3[b]	141.3[a] / 6.3[b]
		L[redacted]	DFOP	133.5[a] / 9.4[b]	133.5[a] / 9.4[b]
	[redacted] 2019	Abington 2	DFOP	142.1[a] / 1.9[b]	142.1[a] / 1.9[b]
		Lamberton 2	DFOP	176.1[a] / 2.8[b]	145.1[a] / 2.3[b]
		Lignieres	DFOP	141.4[a] / 1.4[b]	141.4[a] / 1.4[b]
		Münster	DFOP	170.1[a] / 5.3[b]	124.5[a] / 3.9[b]
		Pikeville	DFOP	155.2[a] / 4.1[b]	129.4[a] / 3.5[b]
		Sarotti 2	DFOP	161.2[a] / 1.6[b]	143.6[a] / 1.4[b]
Geometric mean (SFO and DFOP slow phase)					**181.6[c]**

a – Pseudo-SFO value based on slow phase of decline, calculated as $\ln(2)/k_2$ and normalised if applicable)
b – Pseudo-SFO value based on fast phase of decline, calculated $\ln(2)/k_1$ and normalised if applicable)
c – Geometric mean calculated of DT_{50} values from Lamberton soils prior to calculation of overall geometric mean.

Table 7.1.2.1.1- 62: Summary of modelling endpoints derived for M-01 (AE C653711)

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)	ffm from FLC (direct)	ffm from M-03
Fluopicolide	[redacted] 2003	Münster	SFO	1000[a]	1000[a]	0.3914	0.3892
		Sarotti	SFO	1000[a]	1000[a]	0.798	-
	[redacted] 2003a	Abington (non-sterile)	SFO	1000[a]	1000[a]	0.8406	-
	[redacted] 2003b	Lamberton	SFO	1000[a]	1000[a]	0.7156	-
	[redacted] 2003c	Lamberton	SFO	1000[a]	1000[a]	0.4067	1
		Pikeville	SFO	173.1	174.0	1	-
	[redacted] 2016a	Albaro/Marcomcini	SFO	417.3	417.3	0.8262	-
		Great Chishill	SFO	1000[a]	1000[a]	0.6013	-
		H[redacted]	SFO	571.7	571.7	0.8953	-
		Mas du Coq	SFO	472.2	422.2	0.8075	-
		Parcey-Meslay	SFO	908.4	908.4	0.8286	-
		Vilobi d'Onyar	SFO	323.9	323.9	0.9776	-
	[redacted] 2016b	Dollendorf II	SFO	159.7	159.7	0.819	-
		H[redacted]	SFO	869.3	869.3	0.8773	-
		L[redacted]	SFO	556.2	556.2	0.8156	-
		L[redacted]	SFO	1000[a]	1000[a]	0.8022	-
	[redacted] 2019	Abington 2	SFO	175.6	175.6	0.7879	-
		Lamberton	SFO	1000[a]	1000[a]	0.7252	-
		Lignieres	SFO	1000[a]	1000[a]	0.6264	-
		Münster	SFO	294.7	215.6	0.9101	-
		Pikeville	SFO	135.9	113.3	0.39	1
		Sarotti 2	SFO	267.1	237.9	0.6227	-
M-01	[redacted] 2002	Bethany	SFO	1858.0	2077.6	-	-
		North Dakota	SFO	568.8	913.6	-	-
M-03	[redacted] 2003	Münster	SFO	1000[a]	1000[a]	-	0.9302
		Pikeville	SFO	1000[a]	1000[a]	-	0.9302
				Geometric mean		**569.5[d]**	-	-
				Arithmetic mean		-	**0.757[e]**	**0.850**

a – Conservative default value
b – Pseudo-SFO value based on slow phase of decline (calculated as $\ln(2)/k_2$ and normalised if applicable)
c – Pseudo-SFO value based on fast phase of decline (calculated $\ln(2)/k_1$ and normalised if applicable)
d – Geometric mean calculated of DT_{50} values from Lamberton soil prior to calculation of overall geometric mean.
e – Arithmetic mean calculated of formation fractions from Lamberton soil prior to calculation of overall arithmetic mean.

Table 7.1.2.1.1- 63: Summary of modelling endpoints derived for M-02 (AE C657188)

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)
M-02	[redacted] 2003	Abington	SFO	4.4	4.4
		Münster	SFO	3.5	3.5
		Sarotti	SFO	4.4	4.4
	[redacted] 017	Dollendorf	SFO	1.1	1.1
		H[redacted]	SFO	1.1	0.9
		L[redacted]	SFO	0.7	0.7
		[redacted]	SFO	0.7	0.7
				Geometric mean	**1.6**

Table 7.1.2.1.1- 64: Summary of modelling endpoints derived for M-03 (AE 0608000)

Applied compound	Study	Soil	Soil pH	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)	ffm from FLC
Fluopicolide	[redacted] 2003	Münster	4.9	SFO	62.6	62.6	0.6086
	[redacted] 2003c	Lamberton	5.9	SFO	49.3	54.5	0.5933
	[redacted] 2019	Pikeville	4.5	SFO	29.1	24.4	0.4009
M-03	[redacted] 2003	Abington	7.2	SFO	0.1	0.1	-
		Münster	4.9	DFOP	1000[a]	1000[a]	-
		Pikeville	5.4	DFOP	2.7[b]	2.2[b]	-
		Sarotti	7.1	SFO	0.1	0.08	-
		Bierlow (BL)	5.3	SFO	2.5	2.5	-
	[redacted] 2016a	H[redacted]	6.0	SFO	0.9	0.9	-
				Geometric mean (pH <6)		**17.9[c]**	**-**
				Arithmetic mean (pH <6)		**-**	**0.53**
				Geometric mean (soil pH ≥6)		**0.19**	**-**
				Arithmetic mean (pH ≥6)		**-**	**-**

a – DFOP k_2 parameter fixed to conservative default value
b – Pseudo-SFO DT_{50} value derived as $DT_{90}/3.32$ (and normalized if applicable)
c – Geometric mean calculated for Münster soils prior to calculation of overall value

Table 7.1.2.1.1- 65: Summary of modelling endpoints derived for M-05 (AE 1344122)

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)	ffm from M-02 (PCA)
M-02	[redacted] 2003	Abington	SFO	29.4	29.4	0.2581[a]
		Münster	SFO	172.1	172.1	0.1557[a]
		Sarotti	SFO	45.5	42.3	0.1811[a]
	[redacted] 2017	Dollendorf	SFO	9.3	9.3	0.1528
		H[redacted]	SFO	11.2	9.3	0.0859
		L[redacted]	SFO	5.6	5.6	0.1438
		[redacted]	SFO	8.1	8.1	0.0918
M-05	[redacted] 2003a	Abington	SFO	62.2	62.2	-
		Münster	SFO	136.1	136.1	-
		Sarotti	SFO	34.9	32.5	-
	[redacted] 2016b	H[redacted]	SFO	32.5	32.5	-
		L[redacted]	SFO	16.8	16.8	-
		L[redacted]	SFO	19.0	19.0	-
				Geometric mean	**25.2**	
				Arithmetic mean	**-**	**0.153**

a – Factored formation fraction: ff M-02-ghost × ff ghost-M-05

Table 7.1.2.1.1- 66: Summary of modelling endpoints derived for M-10 (AE 1344123)

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)	ffm from M-02 (PCA)
M-02	[redacted] 2003	Abington	SFO	5.4	5.4	0.1436[a]
		Münster	SFO	1000[b]	1000[b]	0.0335
		Sarotti	SFO	14.6	13.6	0.0796[a]
	[redacted] 2017	Dollendorf	SFO	3.5	3.5	0.1997
		H[redacted]	SFO	20.2	16.8	0.1265
		L[redacted]	SFO	88.2	88.2	0.1502
		[redacted]	SFO	5.8	5.8	0.1686
M-10	[redacted] 2003b	Abington	SFO	31.6	31.6	-
		Münster	SFO	241.9	241.9	-
		Sarotti	SFO	21.3	19.8	-
	[redacted] 2016c	H[redacted]	SFO	21.6	21.6	-
		L[redacted]	SFO	83.9	83.9	-
		L[redacted]	HS	228.8[c]	228.8[c]	-
					Geometric mean	**35.4**	**-**
					Arithmetic mean	**-**	**0.129**

a – Factored formation fraction: ff M-02-ghost × ff ghost-M-05
b – Conservative default value
c - Conservative modelling endpoint DT_{50} value derived from the HS rate constant for the slow phase of degradation

Table 7.1.2.1.1- 67: Summary of modelling endpoints derived for M-11/12

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)	ffm from M-02 (PCA)
M-02	[redacted] 2003	Abington	SFO	31.7	31.7	0.0117
		Münster	SFO	242.5	242.5	0.0711
Geometric mean					**87.6**	-
Arithmetic mean						**0.044**

Table 7.1.2.1.1- 68: Summary of modelling endpoints derived for M-13

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)	ffm from M-02 (PCA)
M-02	[redacted] 2003	Abington	SFO	13.3	13.3	0.0667
		Münster	SFO	48.4	48.4	0.0286
		Sarotti	SFO	14.8	13.8	0.0507
Geometric mean					**20.7**	-
Arithmetic mean					-	**0.049**

Table 7.1.2.1.1- 69: Summary of modelling endpoints derived for M-14 (AE 1388273)

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)	ffm from M-20
M-05	[redacted] 2003a	Abington	SFO	16.4	16.4	1
		Sarotti	SFO	21.7	20.1	1
M-14	[redacted] 2003	Abington	SFO	4.9	4.9	-
		Münster	SFO	8.2	8.2	-
		Sarotti (SLS)	SFO	5.8	5.4	-
Geometric mean					**9.4**	-
Arithmetic mean					-	**1**

Table 7.1.2.1.1- 70: Summary of modelling endpoints derived for M-15 (AE 1413903)

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)
M-15	[redacted] 2016d	Dollendorf II	DFOP	172.5[a]	172.5[a]
		H[redacted]	DFOP	137.9[a]	137.9[a]
		L[redacted]	DFOP	139.6[a]	139.6[a]
		L[redacted]	DFOP	132.4[a]	132.4[a]
Geometric mean					**144.8**

a – Pseudo-SFO value based on slow phase of decline (calculated as $\ln(2)/k_2$)

Table 7.1.2.1.1- 71: Summary of modelling endpoints derived for M-20 (BCS-BX16566)

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{50} normalised (d)	ffm from M-02 (PCA)	ffm from M-05
M-02	[redacted] 2017	H[redacted]	SFO	4.0	3.3	0.0638	0
		L[redacted]	SFO	6.9	6.9	0	0.6664
		[redacted]	SFO	2.7	2.7	0	1
M-05	[redacted] 2003a	Abington	SFO	3.1	3.1		0.5445
		Münster	SFO	144.7	144.7	-	0.7786
		Sarotti	SFO	2.0	1.9	-	0.3644
			Geometric mean		6.1	-	-
			Arithmetic mean		-	0.021	0.559

B. Trigger endpoints

Table 7.1.2.1.1- 72: Summary of trigger endpoints derived for fluopicolide

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
Fluopicolide	[redacted] 2003	Münster	SFO	212.0	704.1
		Sarotti	SFO	191.2	635.0
	[redacted] 2003a	Abington (non-sterile)	SFO	348.0	1156.0
	[redacted] 2003b	Lamberton	SFO	1290.0	4285.0
	[redacted] 2003c	Lamberton	SFO	358.0	1189.4
		Pikeville	DFOP	424.9	1847.5
	[redacted] 2003d	Abington[a]	DFOP	671.6	2393.0
	[redacted] 2016a	Albaro Marcomcini	DFOP	131.6	471.0
		Great Chishill	DFOP	250.7	975.9
		H[redacted]	DFOP	100.6	461.6
		Mas du Coq	DFOP	185.5	688.6
		Parcey Meslay	DFOP	166.1	636.4
		Vilobi d'Onyar	DFOP	54.9	271.4
	[redacted] 2016b	Dollendorf II	DFOP	99.5	358.1
		H[redacted]	DFOP	99.5	419.3
		L[redacted]	DFOP	110.2	438.4
		La[redacted]	DFOP	47.7	353.2
	[redacted] 2019	Abington 2	DFOP	119.2	449.2
		Lamberton	DFOP	158.3	567.2
		Lignieres	DFOP	121.7	450.0
		Münster	DFOP	142.6	537.6
		Pikeville	DFOP	119.7	480.0
		Sarotti 2	DFOP	135.0	509.2

a – Study performed at 10°C

Table 7.1.2.1.1- 73: Summary of trigger endpoints derived for M-01 (BAM)

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
Fluopicolide	[redacted] 2003	Münster	SFO	1000[a]	3322[a]
		Sarotti	SFO	1000[a]	3322[a]
	[redacted] 2003a	Abington (non-sterile)	SFO	1000[a]	3322[a]
	[redacted] 2003b	Lamberton	SFO	1000[a]	3322[a]
	[redacted], 2003c	Lamberton	SFO	1000[a]	3322[a]
		Pikeville	SFO	173.1	574.9
	[redacted] 2003d	Abington[b]	SFO	1000[a]	3322[a]
	[redacted] 2016a	Albaro/Marcomcini	SFO	417.3	1386.0
		Great Chishill	SFO	1000[a]	3322[a]
		H[redacted]	SFO	571.7	1899.2
		Mas du Coq	SFO	472.2	1568.7
		Parcey/Mesley	SFO	908.4	3017.6
		Vilobi d'Onyar	SFO	323.9	1076.1
	[redacted] 2016b	Dollendorf II	SFO	159.7	530.6
		H[redacted]	SFO	869.3	2887.7
		L[redacted]	SFO	556.2	1847.6
		L[redacted]	SFO	1000[a]	3322[a]
	[redacted] 2019	Abington 2	SFO	175.6	583.5
		Lamberton	SFO	1000[a]	3322[a]
		Lignieres	SFO	1000[a]	3322[a]
		Münster	SFO	294.7	979.1
		Pikeville	SFO	135.9	451.6
		Sarotti 2	SFO	267.1	887.3
M-01	[redacted] 2002	Bethany	DFOP	3461.0	>10000
		North Dakota	SFO	568.8	1889.0
M-03	[redacted] 2003	Münster	SFO	1000[a]	3322[a]
		Pikeville	SFO	1000[a]	3322[a]

a – Fixed to conservative default value
b – Study performed at 10°C

Table 7.1.2.1.1- 74: Summary of trigger endpoints derived for M-02 (PCA)

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
M-02 (PCA)	[redacted] 2003	Abington	SFO	4.4	14.5
		Münster	SFO	3.5	11.6
		Sarotti	SFO	4.4	14.5
	[redacted] 2017	Dollendorf	DFOP	1.0	4.0
		H[redacted]	SFO	1.1	3.6
		L[redacted]	SFO	0.7	2.2
		[redacted]	SFO	0.7	2.4

Table 7.1.2.1.1- 75: Summary of trigger endpoints derived for M-03

Applied compound	Study	Soil	Soil pH	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
Fluopicolide	[redacted] 2003	Münster	4.9	SFO	62.6	208.0
	[redacted] 2003c	Lamberton	5.9	SFO	49.3	163.7
	[redacted] 2019	Pikeville	4.5	SFO	29.3	97.4
M-03	[redacted] 2003	Abington	7.2	SFO	0.1	0.3
		Münster	4.9	DFOP	4.4	1225.0
		Pikeville	5.4	DFOP	2.1	9.1
		Sarotti	7.1	SFO	0.1	0.3
	[redacted] 2016a	Brierlow (BL)	5.3	SFO	2.5	8.4
		H[redacted]	6.0	SFO	0.9	2.9

Table 7.1.2.1.1- 76: Summary of trigger endpoints derived for M-05

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
M-02 (PCA)	[redacted] 2003	Abington	SFO	29.4	97.5
		Münster	SFO	172.1	571.6
		Sarotti	SFO	45.5	151.1
	[redacted] 2017	Dollendorf	SFO	9.3	34.0
		H[redacted]	SFO	11.2	37.1
		L[redacted]	SFO	5.6	18.5
		[redacted]	SFO	8.1	26.9
M-05	[redacted] 2003a	Abington	SFO	62.2	206.5
		Münster	SFO	136.1	452.2
		Sarotti	SFO	34.9	116.1
	[redacted] 2016b	H[redacted]	SFO	22.5	74.6
		L[redacted]	SFO	16.8	55.8
		L[redacted]	SFO	19.0	63.2

Table 7.1.2.1.1- 77: Summary of trigger endpoints derived for M-10

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
M-02 (PCA)	[redacted] 2003	Abington	SFO	5.4	18.1
		Münster	SFO	1000[a]	3322[a]
		Sarotti	SFO	14.6	48.4
	[redacted] 2017	Dollendorf	SFO	3.6	11.9
		H[redacted]	SFO	20.2	67.2
		L[redacted]	SFO	88.2	293.0
		[redacted]	SFO	5.8	19.2
M-10	[redacted] 2003b	Abington	SFO	31.6	104.9
		Münster	SFO	241.9	803.6
		Sarotti	SFO	21.3	70.9
	[redacted] 2016c	H[redacted]	SFO	21.6	71.6
		L[redacted]	DFOP	77.3	>10000
		L[redacted]	DFOP	20.2	>10000

a – Fixed to conservative default value

Table 7.1.2.1.1- 78: Summary of trigger endpoints derived for M-11/12

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
M-02 (PCA)	[redacted] 2003	Abington	SFO	31.7	105.1
		Münster	SFO	242.5	805.5

Table 7.1.2.1.1- 79: Summary of trigger endpoints derived for M-13

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
M-02 (PCA)	[redacted] 2003	Abington	SFO	13.3	44.3
		Münster	SFO	48.4	160.6
		Sarotti	SFO	14.8	49.2

Table 7.1.2.1.1- 80: Summary of trigger endpoints derived for M-14

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
M-05	[redacted] 2003a	Abington	SFO	16.4	54.4
		Sarotti	SFO	21.7	72.0
M-14	[redacted] 2003	Abington	SFO	4.9	16.4
		Münster	SFO	8.2	27.3
		Sarotti (SLS)	SFO	5.8	19.3

Table 7.1.2.1.1- 81: Summary of trigger endpoints derived for M-15

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
M-15	[redacted] 2016d	Dollendorf II	DFOP	105.7	506.1
		H[redacted]	DFOP	113.2	433.3
		L[redacted]	DFOP	103.2	427.4
		L[redacted]	DFOP	102.7	410.0

Table 7.1.2.1.1- 82: Summary of trigger endpoints derived for M-20

Applied compound	Study	Soil	Model selected	DT_{50} un-normalised (d)	DT_{90} un-normalised (d)
M-02 (PCA)	[redacted] 2017	H[redacted]	SFO	4.0	13.3
		L[redacted]	SFO	6.9	22.9
		[redacted]	SFO	2.7	8.9
M-05	[redacted] 2003a	Abington	SFO	3.1	10.3
		Münster	SFO	144.7	480.8
		Sarotti	SFO	2.0	6.7

C. Overall formation fraction of M-01 from fluopicolide

Metabolite M-01 can be formed either directly from fluopicolide or from metabolite M-03, which itself is formed from fluopicolide. However, formation *via* M-03 was only observed in a few soils. The overall molar fraction of fluopicolide that degrades to M-01, either directly or *via* M-03, is summarised for each soil in Table 7.1.2.1.1- 83, calculated as $ffm_{FLC \rightarrow M\text{-}01} + [ffm_{FLC \rightarrow M\text{-}03} \times ffm_{M\text{-}03 \rightarrow M\text{-}01}]$, to simplify modelling of M-01 formation if required.

The arithmetic mean overall formation fraction of M-01 from fluopicolide is 0.80.

Table 7.1.2.1.1- 83: Overall formation fraction of M-01 from fluopicolide

Applied compound	Study	Soil	ffm FLC→M-01	ffm FLC→M-03	ffm M-03→M-01	Overall ffm from FLC
Fluopicolide	[redacted] 2003	Münster	0.3914	0.6086	0.3892	0.6283
		Sarotti	0.798	-	-	0.798
	[redacted] 2003a	Abington (non-sterile)	0.8406	-	-	0.8406
	[redacted] 2003b	Lamberton	0.7156	-	-	0.7156
	[redacted] 2003c	Lamberton	0.4067	0.5933	1	1
		Pikeville	1	-	-	1
	[redacted] 2016a	Albaro/Marcomcini	0.8262	-	-	0.8262
		Great Chishill	0.6013	-	-	0.6013
		H[redacted]	0.8953	-	-	0.8953
		Mas du Coq	0.8075	-	-	0.8075
		Parcey Meslay	0.8286	-	-	0.8286
		Vilobi d'Onyar	0.9776	-	-	0.9776
	[redacted] 016b	Dollendorf II	0.819	-	-	0.819
		P[redacted]	0.8773	-	-	0.8773
		L[redacted]	0.8156	-	-	0.8156
		[redacted]	0.8022	-	-	0.8022
	[redacted] 2019	Abington 2	0.7879	-	-	0.7879
		Lamberton	0.7252	-	-	0.7252
		Lignières	0.6264	-	-	0.6264
		Münster	0.9101	-	-	0.9101
		Pikeville	0.39	0.4009	1	0.7909
		Sarotti 2	0.6227	-	-	0.6227
					Arithmetic mean	**0.80[a]**

a – Arithmetic mean calculated of overall formation fractions from Lamberton soil prior to calculation of overall arithmetic mean.

D. EFSA template and graphical representations for kinetic fittings

The results of each kinetic evaluations are provided in the standard EFSA template for fluopicolide and its metabolites in Table 7.1.2.1.1- 84 to Table 7.1.2.1.1- 94. Graphical representations are provided in Table 7.1.2.1.1- 95 to Table 7.1.2.1.1- 105 for the best fit modelling endpoints. Similar graphical representations for the best fit trigger endpoints are provided in the summaries of the aerobic soil degradation studies.

Table 7.1.2.1.1- 84: Standard EFSA template for kinetic fitting for fluopicolide

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Münster, [redacted] (2003)	SFO	97.68	k 0.00327	4.43	7.63E-11	0.002595	0.004	212	704.1
	FOMC	96.95	α 0.455 β 100.7	3.56	n.r. n.r.	0.1406 -3.064	0.769 204.4	361.2	>10000
	DFOP	96.59	k1 0.255 k2 0.002841 g 0.01594	3.74	0.2082 1.37E-11 n.r.	-0.3506 0.002321 0.003067	0.861 0.003 0.029	238.3	8049
Sarotti, [redacted] (2003)	SFO	88.25	k 0.003626	5.65	3.04E-05	0.002281	0.005	191.2	635
	FOMC	89.46	α 0.5823 β 115.4	5.9	n.r. n.r.	-1.488 -440.8	2.652 671.5	264	5901
Abington (non-sterile), [redacted] (2003a)	SFO	96.04	k 0.001992	2.89	4.54E-07	0.001422	0.003	348	1156
	FOMC	99.34	α 0.1403 β 25.08	2.17	n.r. n.r.	0.02499 -17.32	0.256 67.48	3482.7	>10000
Lamberton, [redacted] (2003b)	SFO	91.25	k 0.0005334	3.63	0.077	-0.0001734	0.001	1290	4285
	FOMC	98.46	α 0.00493 β 1.05E-09	2.36	n.r. n.r.	0.00493 9.89E-10	0.005 0	>10000	>10000
Lamberton, [redacted] (2003c)	SFO	88.71	k 0.001936	4.23	<2e-16	0.001752	0.002	358	1189.4
	FOMC	88.19	α 1.753 β 835.6	4.27	n.r. n.r.	-4.007 -2357	7.513 4028	405.2	2271.9
Pikeville, [redacted] (2003c)	SFO	91.14	k 0.001673	5.64	6.52E-09	0.00129	0.002	414.4	1376.5
	FOMC	96.82	α 0.2417 β 43.71	5.16	n.r. n.r.	0.08526 -21.14	0.398 108.6	725.8	>10000
	DFOP	96.55	k1 0.02302 k2 0.001131 g 0.1914	1.97	0.00565 6.56E-06 n.r.	0.006936 0.0007505 0.11	0.039 0.002 0.273	424.9	1847.5
Abington, [redacted] (2003d)	SFO	96.46	k 0.001359	1.76	1.52E-05	0.0008619	0.002	510	1694
	FOMC	98.93	α 0.06124 β 9.129	1.13	n.r. n.r.	0.03328 -1.463	0.089 19.72	>10000	>10000
	DFOP	99.35	k1 0.104 k2 0.0009347 g 0.06329	1.51	0.000806 9.46E-07 n.r.	0.04852 0.0006634 0.04512	0.159 0.001 0.081	671.6	2393
Albaro/ Marcomcint, [redacted] (2016a)	SFO	94.24	k 0.005383	2.18	<2e-16	0.004892	0.006	128.8	427.8
	FOMC	95.86	α 0.5563 β 65.09	1.55	n.r. n.r.	0.3198 23.61	0.793 106.6	161.2	4019
	DFOP	97.2	k1 0.2499 k2 0.004742 g 0.06658	0.913	0.00172 <2e-16 n.r.	0.09565 0.004432 0.04564	0.404 0.005 0.088	131.6	471

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Great Chishill, [redacted] (2016a)	SFO	92.7	k 0.003447	3.66	1.50E-11	0.002764	0.004	201.1	668
	FOMC	96.37	α 0.1233 β 7.602	1.9	n.r. n.r.	0.08991 1.826	0.157 15.38	2096	>10000
	DFOP	98.54	k1 0.2582 k2 0.002219 g 0.1279	0.658	2.83E-08 <2e-16 n.r.	0.1869 0.002038 0.115	0.33 0.002 0.141	250.7	975.9
H[redacted] (2016a)	SFO	99.34	k 0.01003	8.61	1.05E-10	0.007872	0.012	69.1	229.6
	FOMC	98.98	α 0.3038 β 12.15	1.6	n.r. n.r.	0.2679 8.935	0.34 15.36	106.8	>10000
	DFOP	98.67	k1 0.09601 k2 0.004458 g 0.2171	1.19	7.06E-14 <2e-16 n.r.	0.08116 0.00399 0.1882	0.111 0.005 0.246	100.6	461.6
Mas du Coq, [redacted] (2016a)	SFO	94.56	k 0.004277	1.84	<2e-16	0.003862	0.005	162.1	538.4
	FOMC	96.3	α 0.3824 β 50.86	0.666	n.r. n.r.	0.29 31.11	0.475 70.62	260.7	>10000
	DFOP	96.57	k1 0.06602 k2 0.003199 g 0.09496	0.495	6.42E-06 <2e-16 n.r.	0.04119 0.002847 0.00684	0.091 0.004 0.123	185.5	688.6
Parcey Meslay, [redacted] (2016a)	SFO	95.47	k 0.004859	2.63	<2e-16	0.004258	0.005	142.7	473.9
	FOMC	97.68	α 0.3101 β 29.62	0.981	n.r. n.r.	0.2456 18.24	0.375 41.01	247.3	>10000
	DFOP	98.02	k1 0.08553 k2 0.003422 g 0.1272	0.702	4.50E-08 <2e-16 n.r.	0.06156 0.003065 0.09078	0.109 0.004 0.144	166.1	636.4
Vilobi d'Onyar, [redacted] (2016a)	SFO	94.27	k 0.01261	6.13	4.99E-15	0.01078	0.014	55	182.5
	FOMC	97.02	α 0.5975 β 28.07	1.61	n.r. n.r.	0.5141 17.12	0.681 29.03	50.5	1065
	DFOP	97.15	k1 0.08873 k2 0.007416 g 0.2516	0.76	4.20E-14 <2e-16 n.r.	0.07528 0.006893 0.2231	0.102 0.008 0.28	54.9	271.4
Dollendorf, [redacted] (2016b)	SFO	90.88	k 0.007374	4.95	1.94E-13	0.006143	0.009	94	312.2
	FOMC	95.01	α 0.3622 β 19	1.95	n.r. n.r.	0.2842 10.62	0.44 27.38	109.8	>10000
	DFOP	94.06	k1 1.097 k2 0.006222 g 0.07153	4.167	0.00456 <2e-16 n.r.	0.3256 0.005461 0.05601	1.868 0.007 0.087	99.5	358.1
K[redacted] (2016b)	SFO	94.64	k 0.01008	8.95	5.96E-09	0.007478	0.013	68.8	228.4
	FOMC	95.38	α 0.2929 β 11.39	1.97	n.r. n.r.	0.2494 7.52	0.336 15.26	110	>10000
	DFOP	95.88	k1 0.1652 k2 0.005033 g 0.1751	0.906	2.17E-10 <2e-16 n.r.	0.1295 0.004696 0.1545	0.201 0.005 0.196	99.5	419.3
L[redacted] (2016b)	SFO	94.88	k 0.00787	5.27	5.15E-13	0.006506	0.009	88.1	292.6
	FOMC	96.43	α 0.3706 β 22.33	1.54	n.r. n.r.	0.3039 14.43	0.437 30.24	122.6	>10000
	DFOP	96.57	k1 0.1103 k2 0.004904 g 0.1415	1.32	3.59E-06 <2e-16 n.r.	0.0704 0.004362 0.1041	0.15 0.005 0.179	110.2	438.4

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] (2016b)	SFO	89.2	k 0.01235	8.69	1.76E-09	0.009337	0.015	56.1	186.5
	FOMC	95.96	α 0.4526 β 12.5	1.49	n.r. n.r.	0.4098 10.02	0.495 14.97	45.3	2013
	DFOP	95.32	k1 0.07342 k2 0.005194 g 0.374	1.74	9.14E-13 7.08E-13 n.r.	0.06062 0.004297 0.3268	0.086 0.006 0.421	47.4	353.2
Abington 2, [redacted] (2019)	SFO	90.9	k 0.006245	3.81	1.46E-15	0.005576	0.007	111	368.9
	FOMC	95.39	α 0.2186 β 9.099	3.94	n.r. n.r.	0.1563 2.889	0.281 15.31	207.6	>10000
	DFOP	95.15	k1 0.3704 k2 0.004877 g 0.1058	1.41	0.000341 <2e-16 n.r.	0.1787 0.004513 0.08236	0.362 0.005 0.129	119.2	449.2
Lamberton, [redacted] (2019)	SFO	84.39	k 0.004403	3.66	1.22E-13	0.003681	0.005	157.4	522.9
	FOMC	86.65	α 0.3865 β 47.11	2.69	n.r. n.r.	0.1507 0.3272	0.622 93.9	236.1	>10000
	DFOP	87.63	k1 0.2445 k2 0.003936 g 0.06748	2.45	0.0411 5.19E-16 n.r.	-0.02224 0.00342 0.04309	0.511 0.004 0.092	158.3	567.2
Lignieres, [redacted] (2019)	SFO	93.62	k 0.005431	3.63	<2e-16	0.004742	0.006	127.6	424
	FOMC	94.36	α 1.654 β 258.7	3.31	n.r. n.r.	-2.002 -406.4	5.311 923.9	134.6	781.9
	DFOP	99.48	k1 0.4856 k2 0.004903 g 0.09175	1.63	0.921 <2e-16 n.r.	0.03702 0.004464 0.06356	0.934 0.005 0.12	121.7	450
Münster, [redacted] (2019)	SFO	90.46	k 0.005501	3.04	<2e-16	0.004794	0.006	126	418.6
	FOMC	93.39	α 0.2806 β 20.36	2.18	n.r. n.r.	0.2075 9.798	0.354 30.91	220.3	>10000
	DFOP	93.25	k1 0.1305 k2 0.004075 g 0.106	0.925	1.35E-06 <2e-16 n.r.	0.08613 0.003707 0.08059	0.175 0.004 0.131	142.6	537.6
Pikeville, [redacted] (2019)	SFO	89.87	k 0.006034	4.41	1.02E-15	0.005016	0.007	114.9	381.6
	FOMC	94.83	α 0.2662 β 13.52	2.96	n.r. n.r.	0.1778 3.257	0.355 23.79	169.2	>10000
	DFOP	95.58	k1 0.1673 k2 0.004466 g 0.1468	1.78	3.80E-05 <2e-16 n.r.	0.09185 0.003877 0.1097	0.243 0.005 0.184	119.7	480
Sarotti 2, [redacted] (2019)	SFO	91.3	k 0.005094	3.6	2.05E-14	0.004313	0.006	136.1	452
	FOMC	95.32	α 0.2553 β 17.34	2.91	n.r. n.r.	0.1582 3.332	0.352 31.34	244.6	>10000
	DFOP	97.39	k1 0.4303 k2 0.004301 g 0.1066	1.5	0.00375 <2e-16 n.r.	0.1361 0.003886 0.07785	0.724 0.005 0.135	135	509.2

Table 7.1.2.1.1- 85: Standard EFSA template for kinetic fitting for M-01

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Münster, [redacted] (2003) P_{SFO}-$M2_{SFO}$ + $M1_{SFO}$-$M2_{SFO}$; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.3914 (FLC) ff 0.3892 (M03)	11.1	n.r. n.r. n.r.	n.r. 0.0863 -0.0593	n.r. 0.7666 0.8317	1000	3322
Sarotti, [redacted] (2003) P_{SFO}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.798 (FLC)	13.8	n.r. n.r.	n.r. 0.4868	n.r. 1.101	1000	3322
Abington (non-sterile), [redacted] (2003a) P_{SFO}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.8406 (FLC)	23.3	n.r. n.r.	n.r. 0.5376	n.r. 1.141	1000	3322
Lamberton, [redacted] (2003b) P_{SFO}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.7156 (FLC)	28.4	n.r. n.r.	n.r. -0.2543	n.r. 1.705	1000	3322
Lamberton, [redacted] (2003c) P_{SFO}-$M2_{SFO}$ + $M1_{SFO}$-$M2_{SFO}$; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.4067 (FLC) ff 1 (M03)	14.9	n.r. n.r. n.r.	n.r. 0.1027 1	n.r. 0.7076 1	1000	3322
Pikeville, [redacted] (2003c) P_{DFOP}-M_{SFO}	SFO	-	k 0.004005 ff 1 (FLC)	9.91	1.95E-07 n.r.	0.002966 1	0.005 1	173.1	574.9
Abington, [redacted] (2003d) P_{DFOP}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 1 (FLC)	2.48	n.r. n.r.	n.r. 0.743	n.r. 1.254	1000	3322
Albaro Marcomcini, [redacted] (2016a) P_{DFOP}-M_{SFO}	SFO	-	k 0.001661 ff 0.8262 (FLC)	3.88	0.04012 n.r.	-0.0001371 0.7273	0.003 0.925	417.3	1386
Great Chishall, [redacted] (2016a) P_{DFOP}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.6013 (FLC)	12.7	n.r. n.r.	n.r. 0.5468	n.r. 0.6554	1000	3322
H[redacted] (2016a) P_{DFOP}-M_{SFO}	SFO	-	k 0.001212 ff 0.8953 (FLC)	2.34	0.0127 n.r.	0.000202 0.8278	0.002 0.9625	571.7	1899.2
Mas du Coq, [redacted] (2016a) P_{DFOP}-M_{SFO}	SFO	-	k 0.001468 ff 0.8075 (FLC)	3.33	0.0404 n.r.	-0.0001241 0.7254	0.003 0.8917	472.2	1568.7
Parcey Meslay, [redacted] (2016a) P_{DFOP}-M_{SFO}	SFO	-	k 0.000763 ff 0.8286 (FLC)	2.32	0.156 n.r.	-0.0006904 0.7446	0.002 0.9126	908.4	3017.6
Vilobi d'Onyar, [redacted] (2016a) P_{DFOP}-M_{SFO}	SFO	-	k 0.00214 ff 0.9776 (FLC)	2.09	5.59E-06 n.r.	0.001342 0.9256	0.003 1.03	323.9	1076.1
Dollendorf, [redacted] (2016b) P_{DFOP}-M_{SFO}	SFO	-	k 0.004339 ff 0.819 (FLC)	1.88	6.67E-07 n.r.	0.002926 0.6823	0.006 0.9507	159.7	530.6

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
H [redacted] (2016b) P_{DFOP}-M_{SFO}	SFO	-	k 0.0007974 ff 0.8773 (FLC)	3.47	0.0672 n.r.	-0.0002184 0.8091	0.002 0.9456	869.3	2887.7
L [redacted] (2016b) P_{DFOP}-M_{SFO}	SFO	-	k 0.001246 ff 0.8156 (FLC)	4.23	0.0682 n.r.	-0.0003499 0.7176	0.003 0.9132	556.2	1847.6
L [redacted] (2016b) P_{DFOP}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.8022 (FLC)	3.22	n.r. n.r.	n.r. 0.7699	n.r. 0.8344	1000	3322
Abington 2, [redacted] (2019) P_{DFOP}-M_{SFO}	SFO	-	k 0.003946 ff 0.7879 (FLC)	5.47	0.001314 n.r.	0.001589 0.6683	0.006 0.9065	175.6	583.5
Lamberton, [redacted] (2019) P_{DFOP}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.7252 (FLC)	5.68	n.r. n.r.	n.r. 0.6431	n.r. 0.8042	1000	3322
Lignieres, [redacted] (2019) P_{DFOP}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.6264 (FLC)	5.15	n.r. n.r.	n.r. 0.5509	n.r. 0.7014	1000	3322
Münster, [redacted] (2019) P_{DFOP}-M_{SFO}	SFO	-	k 0.002352 ff 0.9101 (FLC)	3.31	0.00555 n.r.	0.0006487 0.8083	0.004 1.013	294.7	979.1
Pikeville, [redacted] (2019) P_{DFOP}-$M2_{SFO}$ + $M1_{SFO}$-$M2_{SFO}$	SFO	-	k 0.005099 ff 0.39 (FLC) ff 1 (M03)	7.52	0.00104 n.r. n.r.	0.002038 0.2771 1	0.008 0.5121 1	135.9	451.6
Sarotti 2, [redacted] (2019) P_{DFOP}-M_{SFO}	SFO	-	k 0.002595 ff 0.6227 (FLC)	9.87	0.07672 n.r.	-0.0008778 0.4856	0.006 0.7612	267.1	887.3
Münster, [redacted] (2003) P_{DFOP}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.9328 (M03)	11	n.r. n.r.	n.r. 0.7517	n.r. 1.116	1000	3322
Münster, [redacted] (2003) P_{DFOP}-M_{SFO}; DT_{50} fixed to 1000 d; k_2 for M-03 fixed to 1000d	SFO	-	k 0.0006931 ff 0.9302 (M03)	11	n.r. n.r.	n.r. 0.7544	n.r. 1.104	1000	3322
Pikeville, [redacted] (2003) P_{DFOP}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.9302 (M03)	5.81	n.r. n.r.	n.r. 0.8475	n.r. 1.016	1000	3322
Bethany, [redacted] (2002)	SFO	1.136	k 0.000373	1.66	1.42E-08	0.0002874	0	1858	6173
	FOMC	1.164	α 0.03657 β 9.731	0.684	n.r. n.r.	0.02637 0.1196	0.047 19.34	>10000	>10000
	DFOP	1.164	k1 0.03968 k2 0.0001796 g 0.06902	0.636	0.00481 0.000215 n.r.	0.01266 9.68E-05 0.05032	0.067 0 0.088	3461	>10000

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
North Dakota, [redacted] (2002)	SFO	1.189	k 0.001219	1.97	4.09E-07	0.0009774	0.001	568.8	1889
	FOMC	1.22	α 0.1042 β 23.91	0.818	n.r. n.r.	0.06679 3.417	0.142 44.41	>10000	>10000
	DFOP	1.219	k1 0.01957 k2 0.0001919 g 0.1711	0.768	0.0559 0.3696 n.r.	-0.002188 -0.0009037 -0.004748	0.041 0.001 0.347	2634	>10000

Table 7.1.2.1.1- 86: Standard EFSA template for kinetic fitting for M-02

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003)	SFO	103.2	k 0.1591	8.51	3.07E-10	0.129	0.189	4.4	14.5
	FOMC	102.4	α 2.07E+07 β 1.35E+08	9.46	n.r. n.r.	2.07E+07 1.35E+08	2.07E+07 1.35E+08	4.5	15.1
Münster, [redacted] (2003)	SFO	104.2	k 0.1986	17.2	9.69E-08	0.1462	0.251	3.5	11.6
	FOMC	104.7	α 182.2 β 907.5	18.5	n.r. n.r.	-20550 -1.03E+05	20910 1.05E+05	3.5	11.5
Sarotti, [redacted] (2003)	SFO	103.3	k 0.159	9.83	2.74E-08	0.1239	0.194	4.4	14.5
	FOMC	102.2	α 1.03E+05 β 6.87E+05	11.2	n.r. n.r.	1.03E+05 6.87E+05	1.03E+05 6.87E+05	4.6	15.3
Dollendorf II, [redacted] (2017)	SFO	103.7	k 0.6056	5.1	<2e-16	0.5658	0.645	1.1	3.8
	FOMC	103.8	α 2.494 β 2.678	0.726	n.r. n.r.	2.21 2.223	2.778 3.134	0.9	4.1
	DFOP	103.8	k1 0.8133 k2 0.1837 g 0.8648	0.639	<2e-16 2.32E-08 n.r.	0.7319 0.1351 0.8056	0.895 0.232 0.924	1	4
H[redacted] (2017)	SFO	104.1	k 0.637	1.79	<2e-16	0.6172	0.657	1.1	3.6
	FOMC	104.1	α 10.85 β 15.51	1.71	n.r. n.r.	-2.618 -5.618	24.31 36.63	1	3.7
L[redacted] (2017)	SFO	103.1	k 1.051	0.719	<2e-16	0.9643	1.138	0.7	2.2
	FOMC	103.1	α 4.003 β 2.504	NaN	n.r. n.r.	-4.513 -5.239	12.52 10.25	0.5	1.9
L[redacted] (2017)	SFO	104.2	k 0.9509	0.578	<2e-16	0.9026	0.999	0.7	2.4
	FOMC	104.2	α 5.202 β 4.103	NaN	n.r. n.r.	-2.89 -4.23	13.29 12.44	0.6	2.3

NaN – Not a number (value not calculated by KinGUI)

Table 7.1.2.1.1- 87: Standard EFSA template for kinetic fitting for M-03

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Münster, [redacted] (2003) P_{SFO}-M_{SFO}	SFO	-	k 0.01107 ff 0.6086 (FLC)	3.89	9.02E-13 n.r.	0.009174 0.4484	0.013 0.7727	62.6	208
Lamberton, [redacted] (2003c) P_{SFO}-M_{SFO}	SFO	-	k 0.01406 ff 0.5933 (FLC)	21.1	0.002 n.r.	0.00505 0.2924	0.023 0.8973	49.3	163.7
Pikeville, [redacted] (2019) P_{DFOP}-M_{SFO}	SFO	-	k 0.02364 ff 0.4009 (FLC)	16.8	6.25E-07 n.r.	0.01533 0.3921	0.032 0.4996	29.3	97.4
Abington, [redacted] (2003)	SFO	92.28	k 6.889	1.47	0.01147	6.402	7.376	0.1	0.3
Münster, [redacted] (2003)	SFO	105.1	k 0.1641	20.5	2.00E-05	0.1066	0.222	4.2	14
	FOMC	103.4	α 1.184 β 5.945	18.3	n.r. n.r.	-0.09832 -3.203	2.467 15.09	4.7	35.6
	DFOP	103.8	k1 0.2007 k2 0.0003466 g 0.8471	12.9	7.56E-05 0.476 n.r.	0.1224 -0.01078 0.7095	0.279 0.011 0.985	4.4	1225
	DFOP (k2 fixed)	103.9	k1 0.2027 k2 0.0006931 g 0.8436	12.2	1.45E-06 n.r. n.r.	0.1461 n.r. 0.7674	0.259 n.r. 0.92	4.4	644.9
Pikeville, [redacted] (2003)	SFO	100.6	k 0.3262	11.4	1.52E-10	0.2794	0.373	2.1	7.1
	FOMC	100.9	α 4.361 β 12.08	11.7	n.r. n.r.	-1.989 -7.579	10.71 31.74	2.1	8.4
	DFOP	100.9	k1 0.3746 k2 0.01155 g 0.9232	8.48	4.98E-09 0.149 n.r.	0.3095 -0.009459 0.8622	0.44 0.033 0.984	2.1	9.1
Sarotti, [redacted] (2003)	SFO	93.63	k 6.903	3.11	0.02458	5.857	7.95	0.1	0.3
Brierlow (BL), [redacted] (2016a)	SFO	0.04213	k 0.2757	9.46	6.21E-09	0.2297	0.322	2.5	8.4
	FOMC	0.04213	α 4211 β 15270	10.1	n.r. n.r.	4211 15270	4211 15270	2.5	8.4
H [redacted] (2016a)	SFO	0.04089	k 0.7914	7.98	4.53E-10	0.7003	0.882	0.9	2.9
	FOMC	0.04089	α 9949 β 12570	8.62	n.r. n.r.	9949 12570	9949 12570	0.9	2.9

Table 7.1.2.1.1- 88: Standard EFSA template for kinetic fitting for M-05

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003) P_{SFO}-ghost-M_{SFO}	SFO	-	k 0.02361 ff 0.2581 (PCA)	7.09	2.02E-07 n.r.	0.01709 -15530	0.03 15190	29.4	97.5
Münster, [redacted] (2003) P_{SFO}-ghost-M_{SFO}	SFO	-	k 0.004028 ff 0.1557 (PCA)	5.95	0.003051 n.r.	0.001425 0.447	0.007 0.4895	172.1	571.6
Sarotti, [redacted] (2003) P_{SFO}-ghost-M_{SFO}	SFO	-	k 0.01524 ff 0.1811 (PCA)	5.81	1.64E-06 n.r.	0.01072 0.4022	0.02 0.5172	45.5	151.1
Dollendorf II, [redacted] (2017) P_{SFO}-M_{SFO}	SFO	-	k 0.07468 ff 0.1528 (PCA)	3.02	<2e-16 n.r.	0.06905 0.1455	0.08 0.1601	9.3	30.8
Dollendorf II, [redacted] (2017) P_{DFOP}-M_{SFO}	SFO	-	k 0.07419 ff 0.1548 (PCA)	3.8	2e-16 n.r.	0.06816 0.1481	0.08 0.1616	9.3	31
H[redacted] 2017) P_{SFO}-M_{SFO}	SFO	-	k 0.0621 ff 0.8859 (PCA)	14.5	1.37E-07 n.r.	0.04237 0.0723	0.082 0.0996	11.2	37.1
L[redacted] (2017) P_{SFO}-M_{SFO}	SFO	-	k 0.1245 ff 0.1438 (PCA)	7.2	<2e-16 n.r.	0.1097 0.1325	0.139 0.155	5.6	18.5
L[redacted] (2017) P_{SFO}-M_{SFO}	SFO	-	k 0.08564 ff 0.0918 (PCA)	7.91	2.62E-15 n.r.	0.07183 0.0833	0.099 0.1001	8.1	26.9
Abington, [redacted] (2003a)	SFO	96.98	k 0.01115	2.36	<2e-16	0.01041	0.012	62.2	206.5
	FOMC	96.96	α 91.57 β 8094	2.51	n.r. n.r.	-2587 -2.33E+05	2770 2.49E+05	62.3	208.6
Münster, [redacted] (2003a)	SFO	96.83	k 0.005092	2.85	<2e-16	0.004512	0.006	136.1	452.2
	FOMC	98.69	α 0.7676 β 101.2	1.9	n.r. n.r.	0.2406 4.019	1.295 198.5	148.5	1932
	DFOP	98.68	k1 0.0126 k2 2.28E-14 g 0.5758	1.91	0.139 0.5 n.r.	-0.009647 -0.0139 -0.7609	0.035 0.014 1.912	160.9	>10000
Sarotti, [redacted] (2003a)	SFO	94.42	k 0.01984	4.09	<2e-16	0.01984	0.02	34.9	116.1
	FOMC	94.57	α 1.79E+08 β 8.95E+09	4.37	n.r. n.r.	1.79E+08 8.95E+09	1.79E+08 8.95E+09	34.6	115
H[redacted] (2016b)	SFO	0.02551	k 0.03086	3.6	1.21E-11	0.02765	0.034	22.5	74.6
	FOMC	0.02551	α 1.01E+05 β 3.28E+06	3.84	n.r. n.r.	1.01E+05 3.28E+06	1.01E+05 3.28E+06	22.5	74.6
L[redacted] (2016b)	SFO	0.02603	k 0.04174	4.94	7.06E-11	0.03635	0.046	16.8	55.8
	FOMC	0.0263	α 4.8 β 102.9	4.84	n.r. n.r.	-2.268 -68.98	11.87 274.7	16	63.3
L[redacted] 2016b)	SFO	0.02557	k 0.03642	6.57	1.83E-09	0.03089	0.042	19	63.2
	FOMC	0.02584	α 4.075 β 97.64	6.6	n.r. n.r.	-2.693 -90	10.84 285.3	18.1	74.2

Table 7.1.2.1.1- 89: Standard EFSA template for kinetic fitting for M-10

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003) P_{SFO}-ghost-M_{SFO}	SFO	-	k 0.1274 ff 0.1436 (PCA)	20.8	0.006874 n.r.	0.03415 -2.64E+08	0.221 2.64E+08	5.4	18.1
Münster, [redacted] (2003) P_{SFO}-M_{SFO}; DT_{50} fixed to 1000 d	SFO	-	k 0.0006931 ff 0.0335 (PCA)	20.5	n.r. n.r.	n.r. 0.0282	n.r. 0.0417	1000	3322
Sarotti, [redacted] (2003) P_{SFO}-ghost-M_{SFO}	SFO	-	k 0.04754 ff 0.0796 (PCA)	27.4	0.004972 n.r.	0.032 0.1588	0.08 0.2489	14.6	48.4
Dollendorf II, [redacted] (2017) P_{SFO}-M_{SFO}	SFO	-	k 0.1997 ff 0.1997 (PCA)	3.28	<2e-16 n.r.	0.1786 0.1825	0.221 0.2171	3.5	11.5
Dollendorf II, [redacted] (2017) P_{DFOP}-M_{SFO}	SFO	-	k 0.1934 ff 0.2002 (PCA)	4.02	<2e-16 n.r.	0.1732 0.1842	0.214 0.216	3.6	11.9
H[redacted] (2017) P_{SFO}-M_{SFO}	SFO	-	k 0.03428 ff 0.1265 (PCA)	4.53	2.10E-15 n.r.	0.02879 0.1183	0.04 0.1348	20.2	67.2
L[redacted], (2017) P_{SFO}-M_{SFO}	SFO	-	k 0.007859 ff 0.1502 (PCA)	11.4	6.54E-06 n.r.	0.004739 0.1349	0.011 0.165	88.2	293
[redacted] (2017) P_{SFO}-M_{SFO}	SFO	-	k 0.1199 ff 0.1686 (PCA)	15.5	8.94E-08 n.r.	0.08261 0.1345	0.157 0.2027	5.8	19.2
Abington, [redacted] (2003b)	SFO	110.9	k 0.02195	8.03	6.09E-09	0.01852	0.025	31.6	104.9
	FOMC	110.9	α 10150 β 4.63E+05	8.57	n.r. n.r.	8501 4.63E+05	11800 4.63E+05	31.6	104.9
Münster, [redacted] (2003b)	SFO	100.9	k 0.002865	2.43	3.68E-05	0.00211	0.004	241.9	803.6
	FOMC	102.5	α 0.2448 β 42.87	2.31	n.r. n.r.	-0.1525 -76.51	0.642 162.3	684.9	>10000
	DFOP	105.1	k1 1888 k2 0.002565 g 0.061	2.04	NA 0.000233 n.r.	NA 0.001834 0.02595	NA 0.003 0.096	245.7	873
Sarotti, [redacted] (2003b)	SFO	109.4	k 0.03248	11.2	9.06E-08	0.02576	0.039	21.3	70.9
	FOMC	109.4	α 19160 β 5.90E+05	12	n.r. n.r.	15050 5.90E+05	23270 5.90E+05	21.3	70.9
H[redacted] [redacted] (2016c)	SFO	101.1	k 0.03214	5.34	1.61E-09	0.0273	0.037	21.6	71.6
	FOMC	101.1	α 2.33E+05 β 7.26E+06	5.7	n.r. n.r.	2.33E+05 7.26E+06	2.33E+05 7.26E+06	21.6	71.6

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
L [redacted] (2016c)	SFO	95.96	k 0.008261	4.79	7.25E-09	0.006864	0.01	83.9	278.7
	FOMC	98.66	α 0.762 β 54.72	3.57	n.r. n.r.	0.2004 -5.75	1.324 115.2	81.2	1068
	DFOP	98.68	k1 0.02068 k2 2.34E-14 g 0.6267	3.47	0.171 0.5 n.r.	-0.0202 -0.02341 -0.7739	0.062 0.023 2.027	7.3	>10000
L [redacted] (2016c)	SFO	94.31	k 0.02498	13.3	2.37E-06	0.01772	0.032	27.8	92.4
	FOMC	99.46	α 0.8631 β 17.73	8.12	n.r. n.r.	0.4364 1.976	1.29 33.48	21.8	237.7
	DFOP	98.97	k1 0.04804 k2 2.34E-14 g 0.8041	6.14	0.000137 0.5 n.r.	0.02849 -0.007782 0.6095	0.068 0.008 0.999	20.2	>10000
	HS	98.33	k1 0.03424 k2 0.003029 tb 37.96	5.18	2.30E-10 0.0688 n.r.	0.0298 -0.0007402 27.81	0.039 0.007 48.11	20.2	369.1

Table 7.1.2.1.1- 90: Standard EFSA template for kinetic fitting for M-11/M-12

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003) P_{SFO}-M_{SFO}	SFO	-	k 0.0219 ff 0.0177 (PCA)	23.5	0.004777 n.r.	0.00678 0.0114	0.037 0.0239	31.7	105.1
Münster, [redacted] (2003) P_{SFO}-M_{SFO}	SFO	-	k 0.002858 ff 0.0711 (PCA)	12.8	0.1349 n.r.	0.002091 0.0488	0.008 0.0932	242.5	805.5

Table 7.1.2.1.1- 91: Standard EFSA template for kinetic fitting for M-13

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003) P_{SFO}-M_{SFO}	SFO	-	k 0.05197 ff 0.0667 (PCA)	35.9	0.01813 n.r.	0.006296 0.0338	0.098 0.0993	13.3	44.3
Münster, [redacted] (2003) P_{SFO}-M_{SFO}	SFO	-	k 0.01433 ff 0.0286 (PCA)	32.8	0.04371 n.r.	-0.001371 0.0155	0.03 0.0417	48.4	160.6
Sarotti, [redacted] (2003) P_{SFO}-M_{SFO}	SFO	-	k 0.04684 ff 0.0507 (PCA)	34.2	0.01462 n.r.	0.008081 0.0269	0.086 0.0746	14.8	49.2

Table 7.1.2.1.1- 92: Standard EFSA template for kinetic fitting for M-14

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003a) P_{SFO}-M_{SFO}	SFO	-	k 0.04234 ff 1 (M20)	10.5	6.34E-07 n.r.	0.02908 1	0.056 1	16.4	54.4
Sarotti, [redacted] (2003a) P_{SFO}-M_{SFO}	SFO	-	k 0.032 ff 1 (M20)	7.61	42e-16 n.r.	0.032 1	0.032 1	21.7	72
Abington, [redacted] (2003)	SFO	11.81	k 0.1404	3.75	<2e-16	0.1345	0.146	4.9	16.4
	FOMC	11.88	α 10.59 β 70.34	3.58	n.r. n.r.	-0.6072 -9.509	21.78 150.2	4.8	17.1
Münster, [redacted] (2003)	SFO	11.85	k 0.08429	4.02	3.01E-15	0.0795	0.089	8.2	27.3
	FOMC	11.85	α 606.5 β 7187	4.29	n.r. n.r.	-55090 -6.54E+05	56310 6.68E+05	8.2	27.3
Sarotti (SLS), [redacted] (2003)	SFO	11.89	k 0.1194	4	1.33E-15	0.113	0.126	5.8	19.3
	FOMC	12.05	α 6.376 β 47.38	3.17	n.r. n.r.	1.655 8.062	11.1 86.69	5.4	20.6

Table 7.1.2.1.1- 93: Standard EFSA template for kinetic fitting for M-15

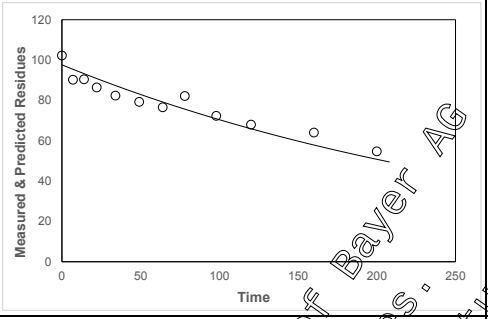
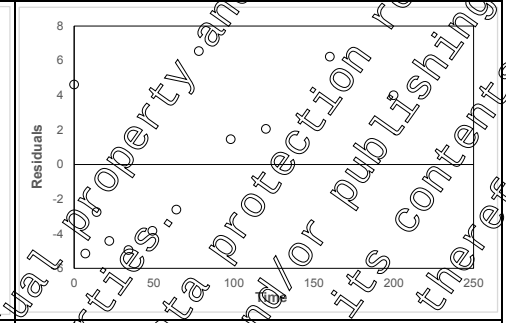
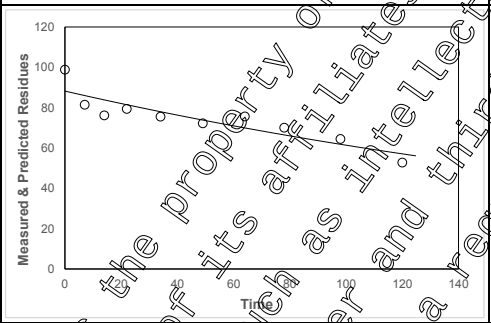
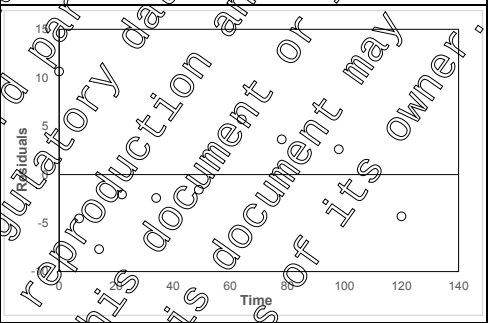
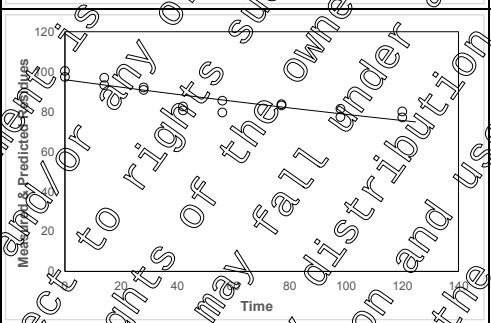
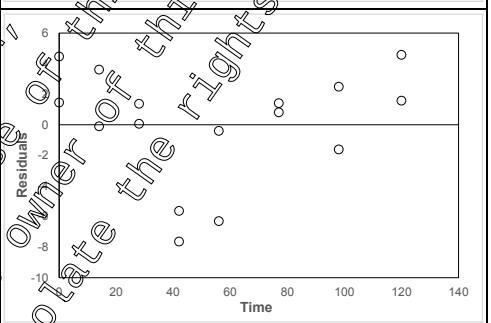
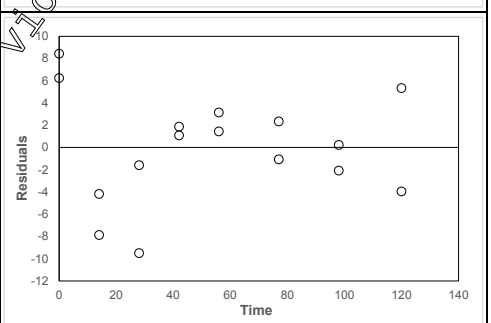
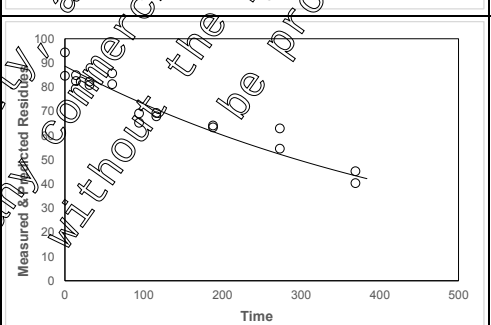
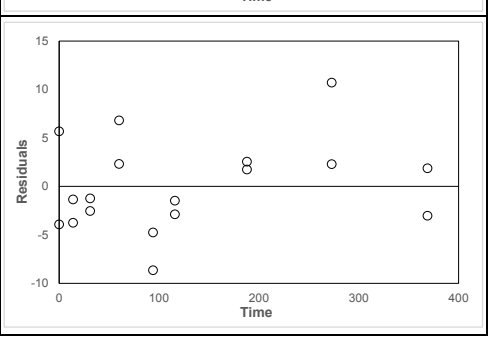
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Dollendorf II, [redacted] (2016d)	SFO	76.03	k 0.00625	7.16	4.77E-06	0.004431	0.008	110.9	368.4
	FOMC	85.5	α 0.1742 β 2.727	2.92	n.r. n.r.	0.1288 0.4348	0.22 5.018	143.1	>10000
	DFOP	86.12	k1 0.2768 k2 0.004019 g 0.2353	1.08	0.000202 2.90E-08 n.r.	0.1648 0.003352 0.2027	0.389 0.005 0.268	105.7	506.1
H[redacted] (2016d)	SFO	83.39	k 0.005497	3.9	6.52E-08	0.00439	0.007	126.1	418.9
	FOMC	84.69	α 0.8885 β 116.4	3.96	n.r. n.r.	-0.8053 -176.7	2.582 409.4	137.5	1437
	DFOP	91.95	k1 22 k2 0.005928 g 0.1168	1.49	<2e-16 1.07E-08 n.r.	22 0.004264 0.06913	22 0.006 0.165	113.2	433.3
[redacted] (2016d)	SFO	89.93	k 0.006216	5.6	2.43E-07	0.004821	0.008	111.5	370.5
	FOMC	94.18	α 0.3379 β 19.77	5.28	n.r. n.r.	0.08529 -9.301	0.59 48.83	134	>10000
	DFOP	99.69	k1 0.4867 k2 0.004965 g 0.1652	2.94	0.0123 4.13E-08 n.r.	0.1157 0.004114 0.12	0.858 0.006 0.21	103.2	427.4
L[redacted] (2016d)	SFO	83.81	k 0.005842	5.32	3.38E-07	0.004494	0.007	118.7	394.2
	FOMC	85.02	α 0.8946 β 108.5	5.54	n.r. n.r.	-0.9788 -194.8	2.768 411.9	127	1315
	DFOP	94.35	k1 22.61 k2 0.005237 g 0.1437	2.95	NA 1.58E-08 n.r.	NA 0.004414 0.09579	NA 0.006 0.192	102.7	410

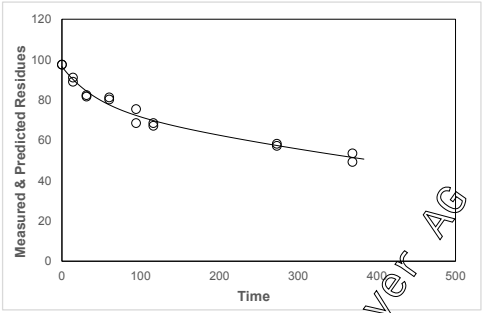
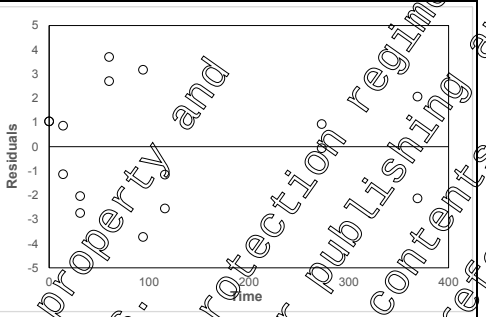
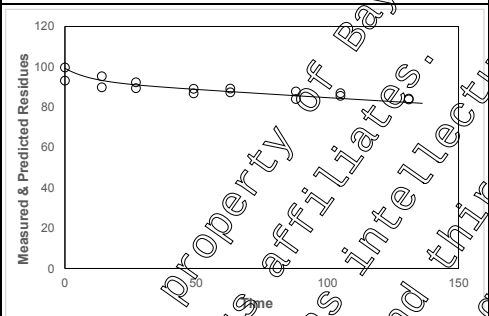
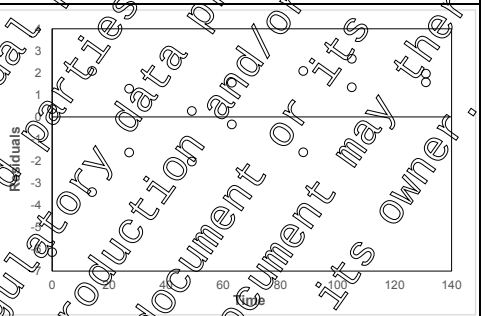
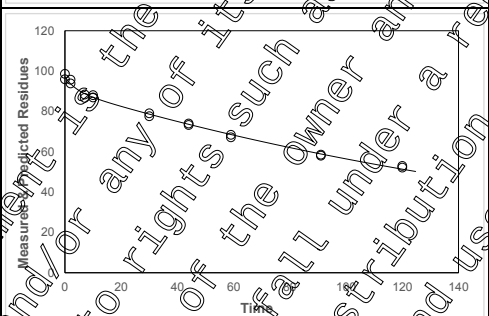
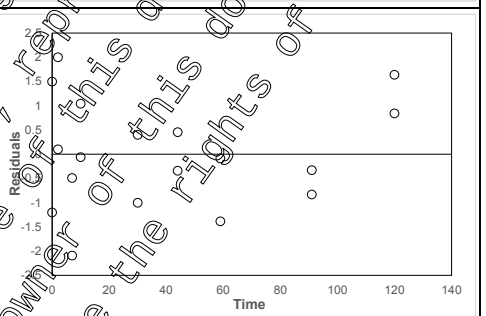
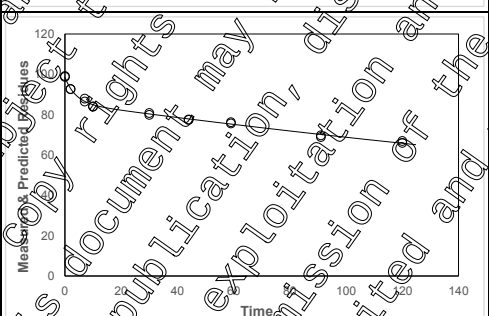
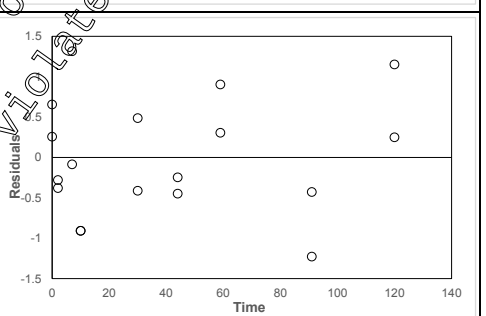
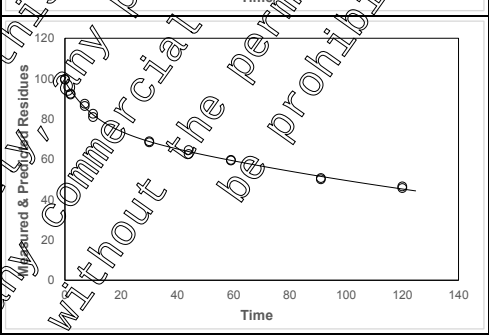
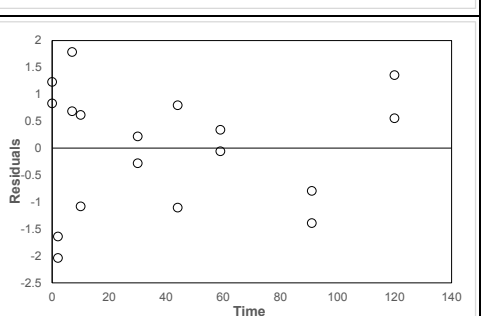
Table 7.1.2.1.1- 94: Standard EFSA template for kinetic fitting for M-20

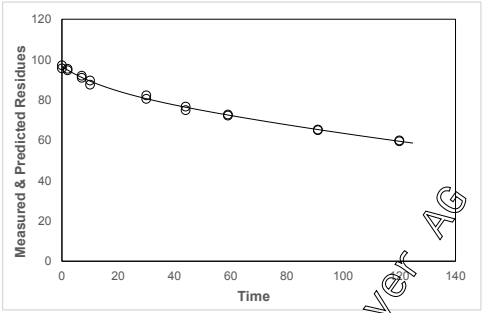
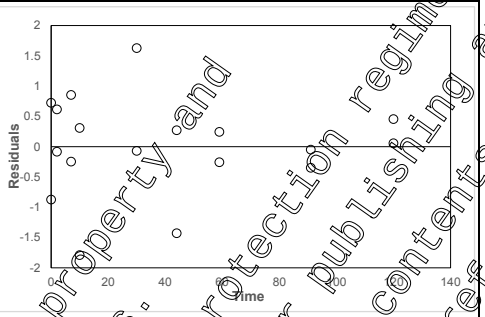
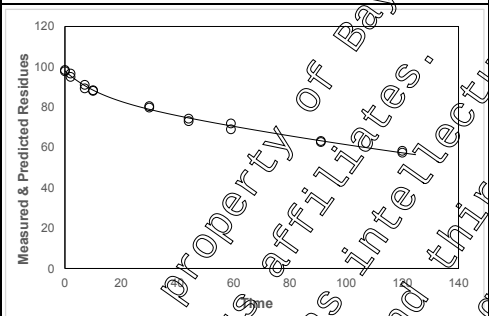
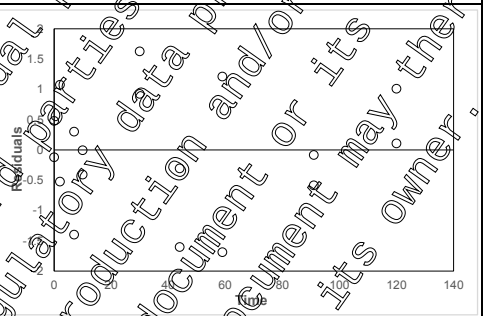
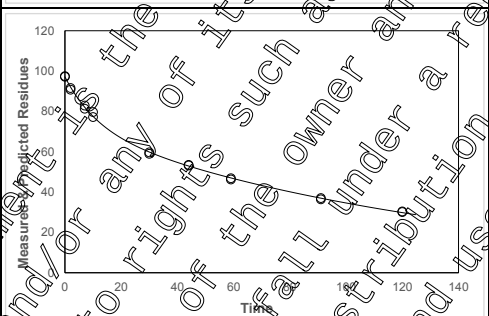
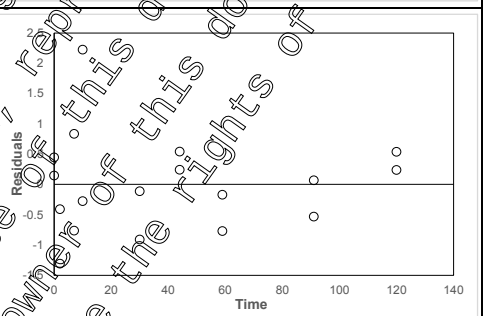
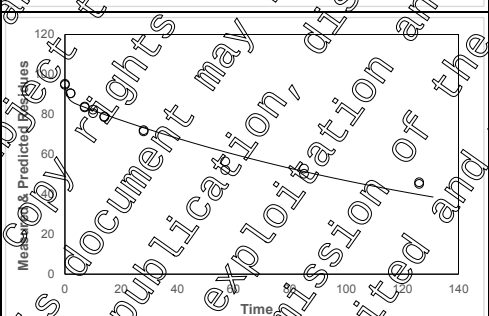
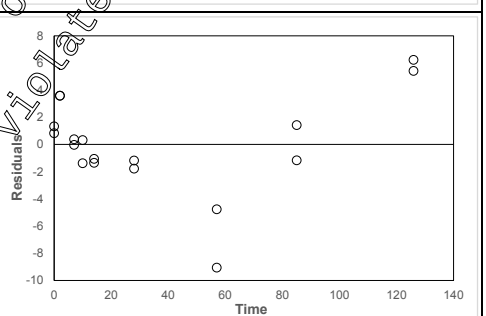
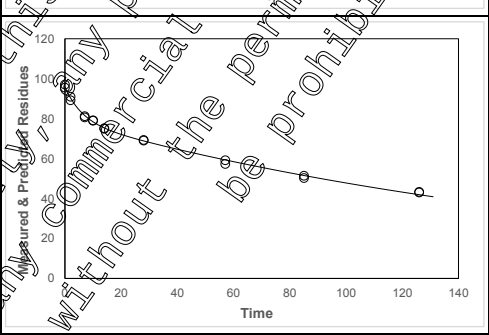
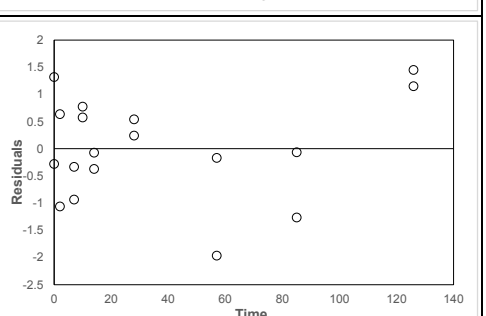
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
H [redacted] (2017) P_{SFO}-M_{SFO}	SFO	-	k 0.1725 ff 0.0638 (PCA)	2	<2e-16 n.r.	0.1543 0.0586	0.191 0.069	4	13.3
I [redacted] (2017) $M1_{SFO}$-$M2_{SFO}$	SFO	-	k 0.1004 ff 0.6664 (M05)	23.8	0.000121 n.r.	0.05137 0.4458	0.149 0.8964	6.9	22.9
I [redacted] (2017) $M1_{SFO}$-$M2_{SFO}$	SFO	-	k 0.2579 ff 1 (M05)	12.6	0.0234 n.r.	0.01143 0.1887	0.504 1.85	2.7	8.9
Abington, [redacted] (2003a) P_{SFO}-$M_{SFO;}$ sink compartment removed and samples <LOD excluded	SFO	-	k 0.2238 ff 0.5445 (M05)	NaN	1.27E-05 n.r.	0.1279 0.3931	0.31 0.6954	3.1	10.3
Münster, [redacted] (2003a) P_{SFO}-M_{SFO}	SFO	-	k 0.004789 ff 0.7786 (M05)	10.9	0.00212 n.r.	0.001775 0.6204	0.008 0.9346	144.7	480.8
Sarotti, [redacted] (2003a) P_{SFO}-$M_{SFO;}$ sink compartment removed and samples <LOD excluded	SFO	-	k 0.3447 ff 0.3644 (M05)	NaN	<2e-16 n.r.	0.3447 0.3644	0.345 0.3644	2	6.7

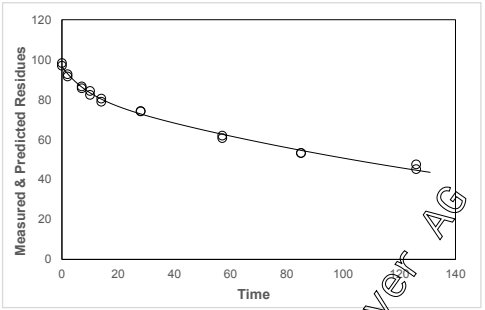
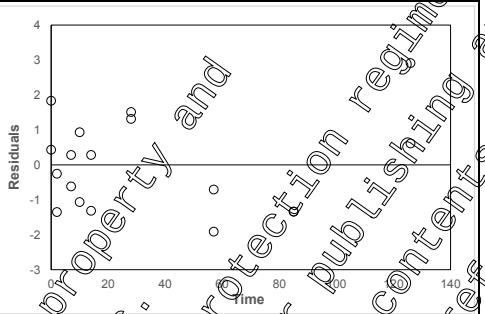
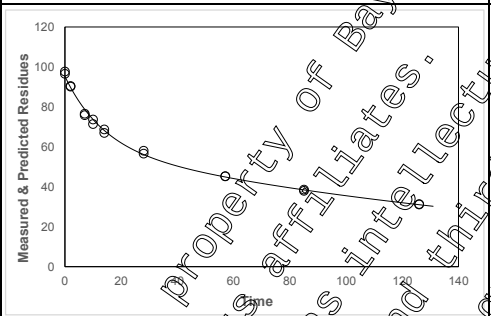
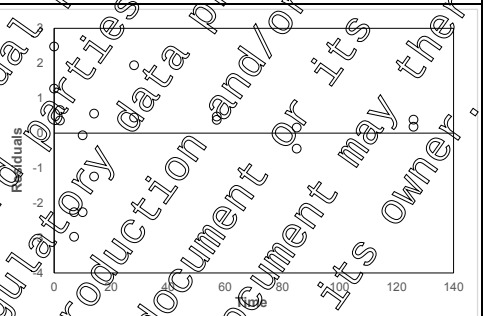
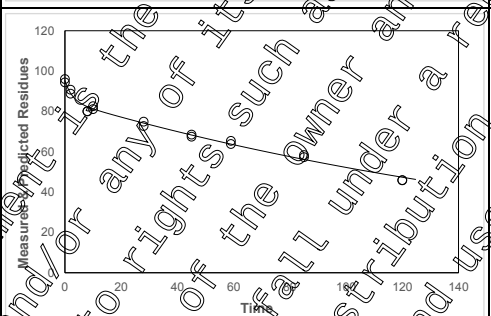
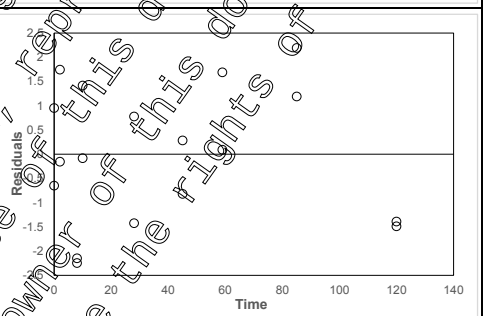
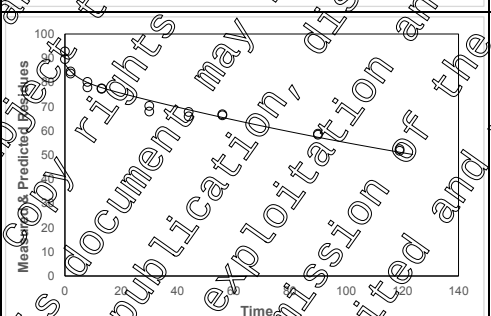
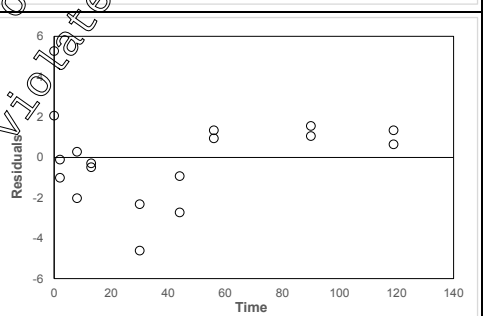
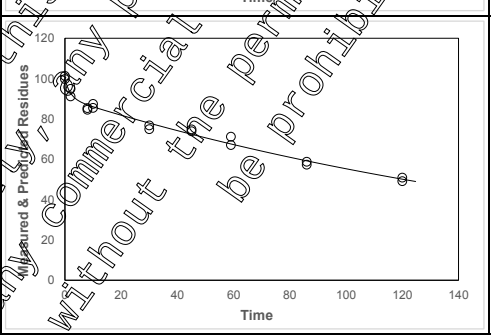
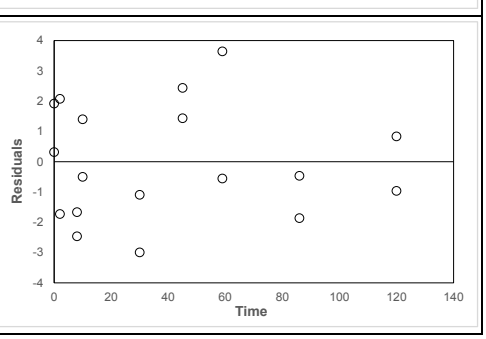
NaN – Not a number (value not calculated by KinGUI)

Table 7.1.2.1.1- 95: Graphical representations of best fit models for modelling endpoints for fluopicolide

Soil Model Reference	Modelled vs observed	Residuals
Münster SFO [redacted] (2003)		
Sarotti SFO [redacted] (2003)		
Abington (non-sterile) SFO [redacted] (2003a)		
Lamberton SFO [redacted] (2003b)		
Lamberton SFO [redacted] (2003c)		

Soil Model Reference	Modelled vs observed	Residuals
Pikeville DFOP [redacted] (2003c)		
Abington DFOP [redacted] (2003d)		
Albaro/Marcomcini DFOP [redacted] (2016a)		
Great Chishill DFOP [redacted] (2016a)		
H[redacted] DFOP [redacted] (2016a)		

Soil Model Reference	Modelled vs observed	Residuals
Mas du Coq DFOP [redacted] (2016a)		
Parcey Meslay DFOP [redacted] (2016a)		
Vilobi d'Ònyar DFOP [redacted] (2016a)		
Dollendorf DFOP [redacted] (2016b)		
H[redacted] DFOP [redacted] (2016b)		

Soil Model Reference	Modelled vs observed	Residuals
L[redacted] DFOP [redacted] (2016b)		
L[redacted] DFOP [redacted] (2016b)		
Abington 2 DFOP [redacted] (2019)		
Lamberton DFOP [redacted] (2019)		
Lignieres DFOP [redacted] (2019)		

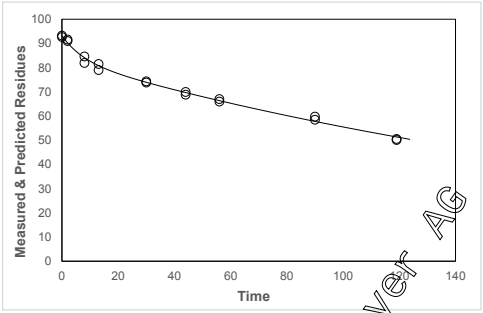
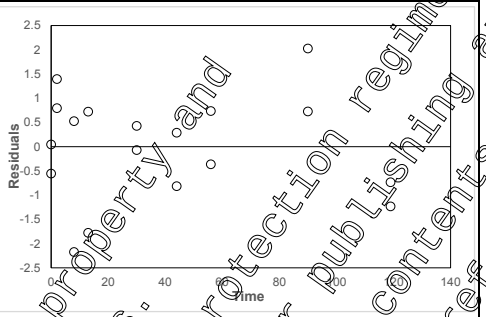
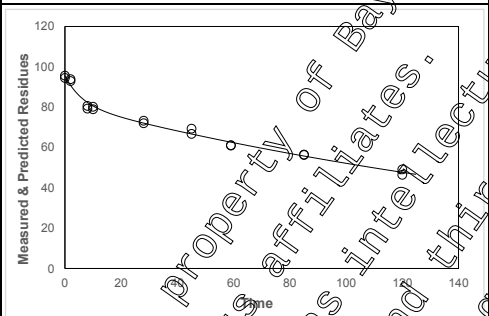
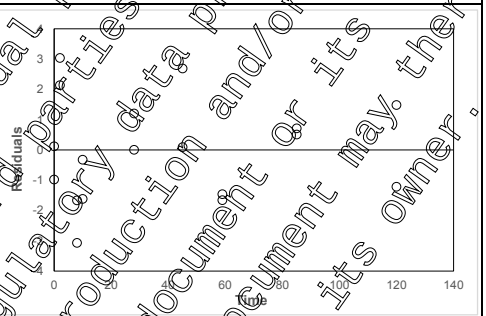
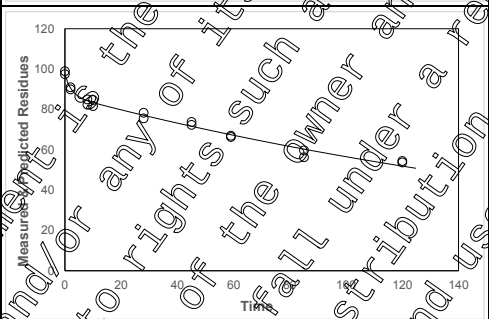
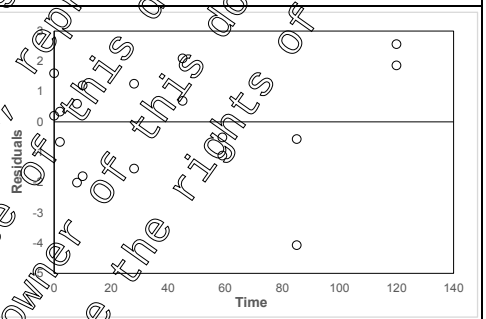
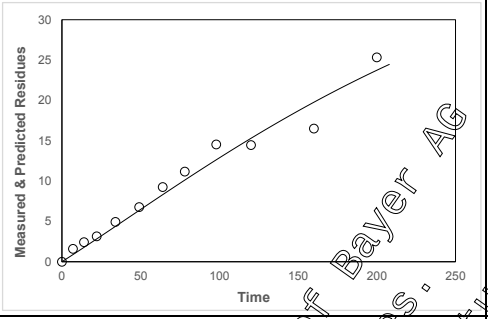
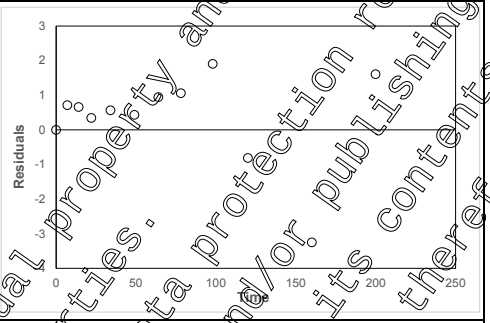
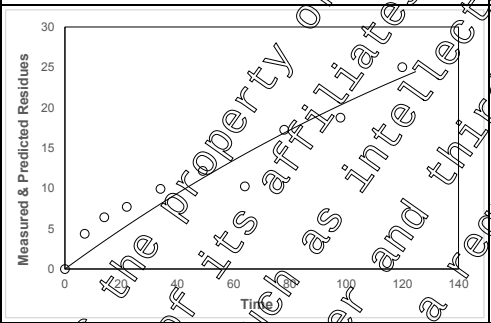
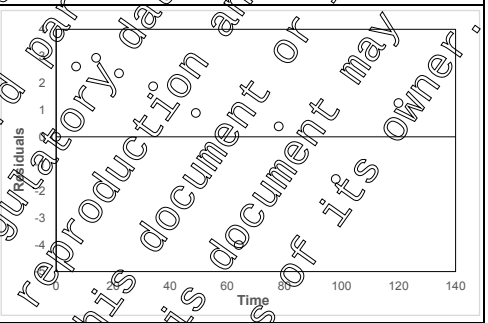
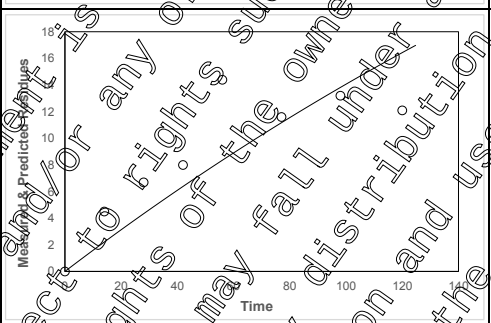
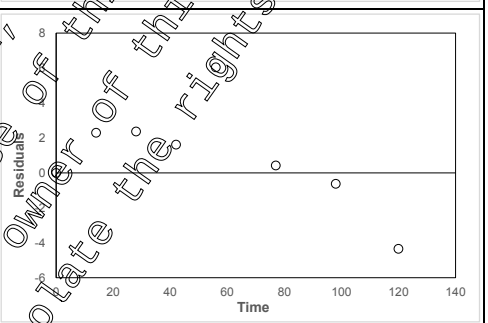
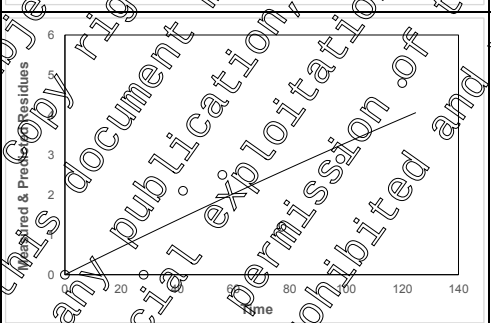
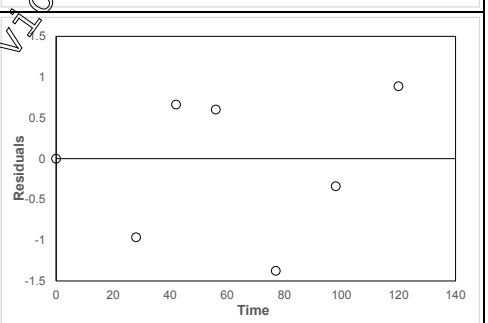
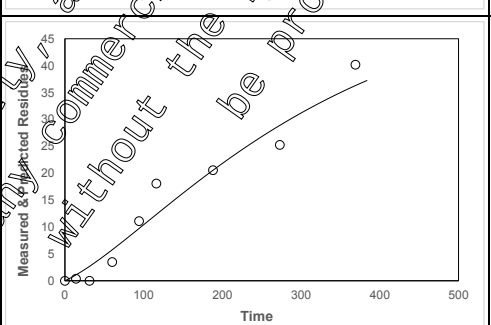
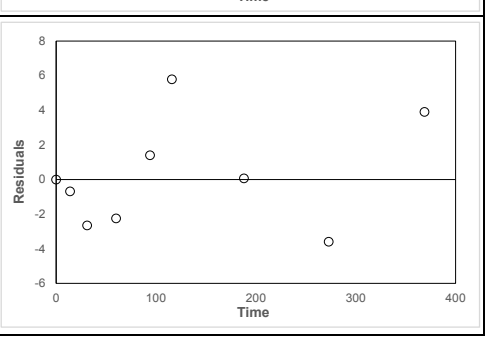
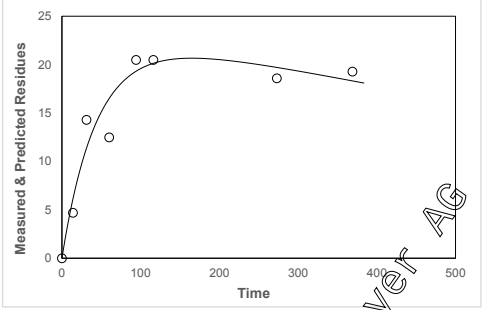
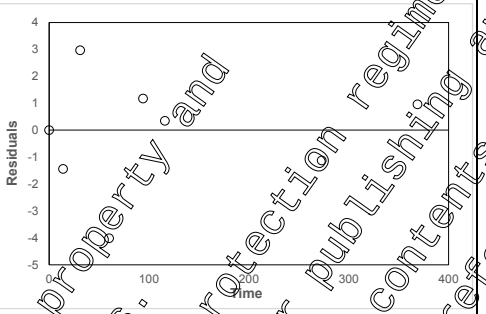
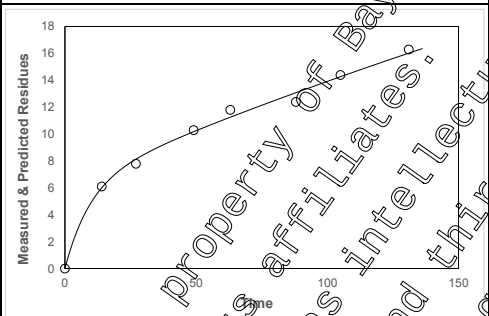
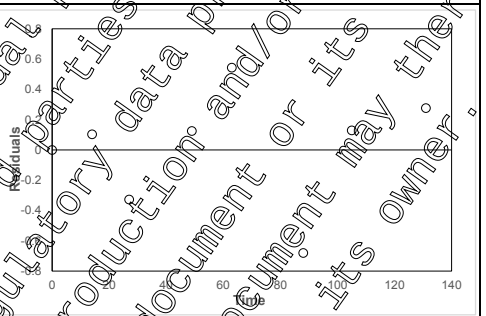
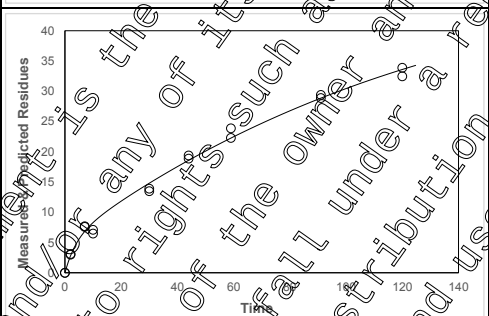
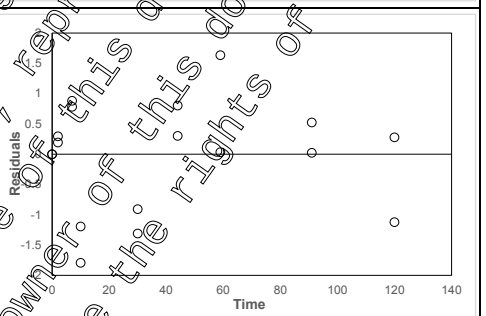
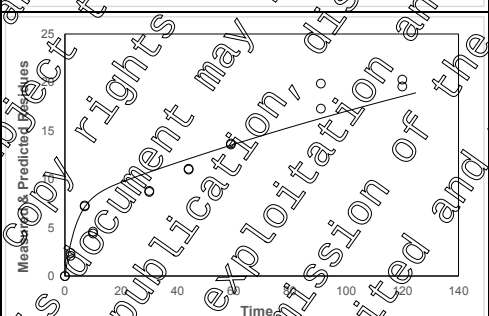
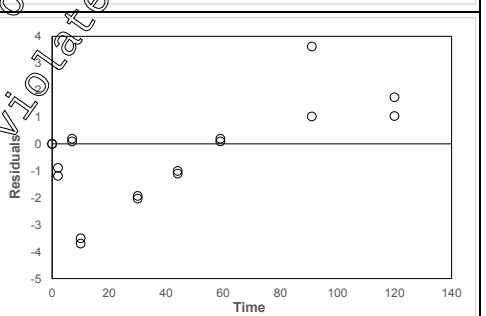
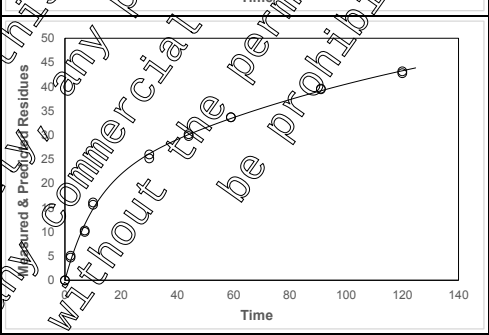
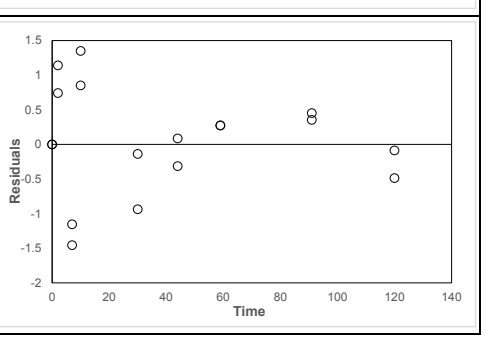
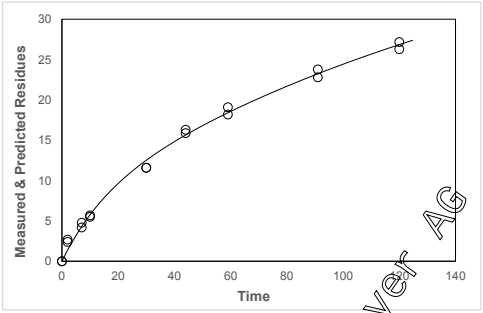
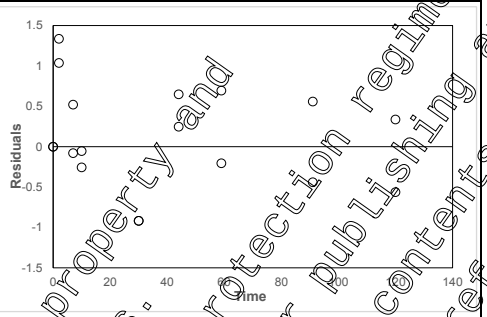
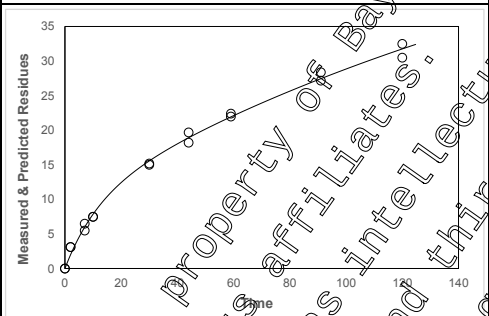
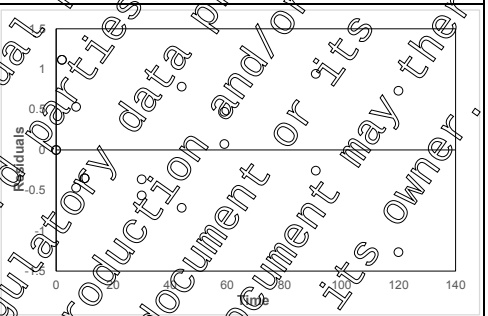
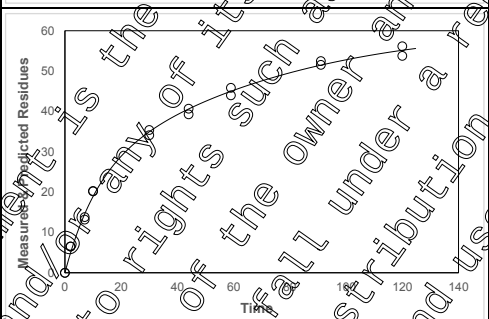
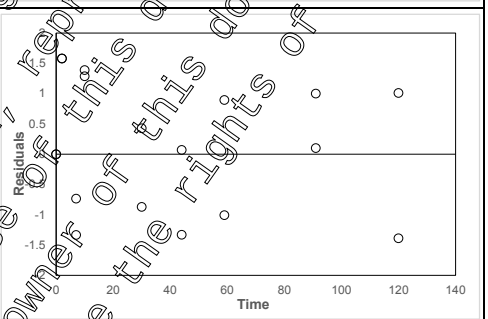
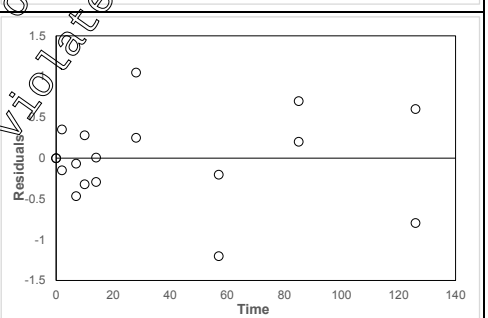
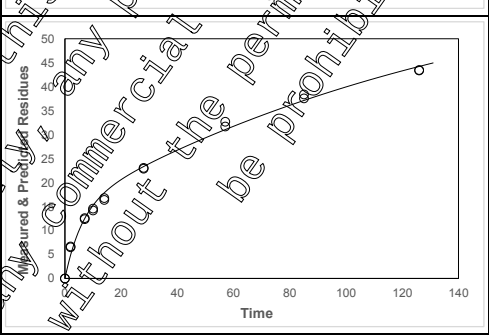
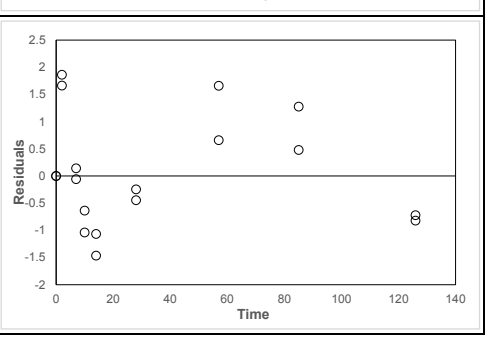
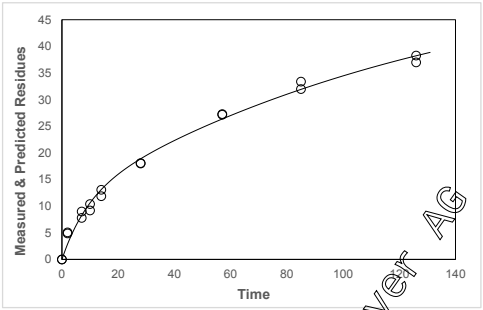
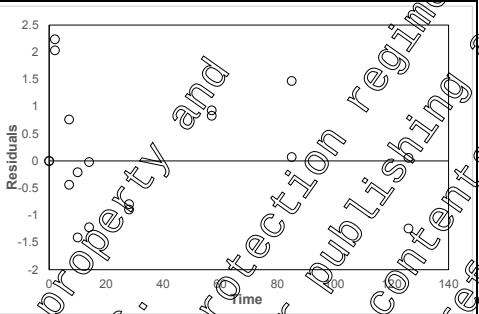
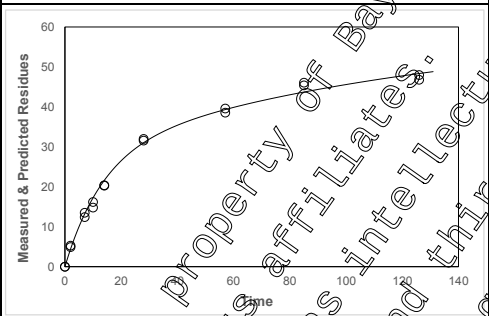
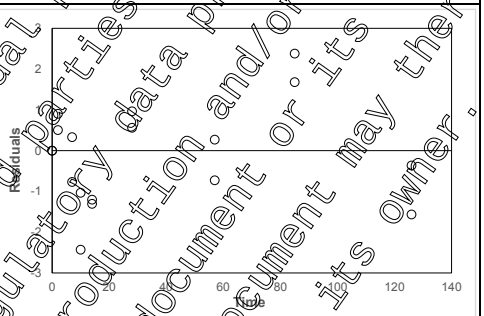
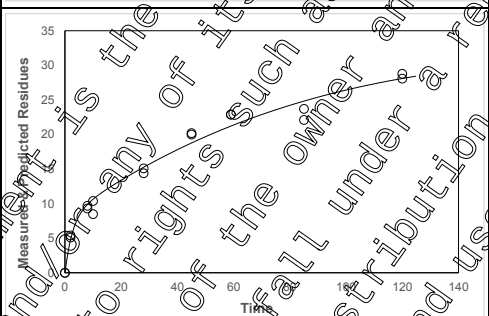
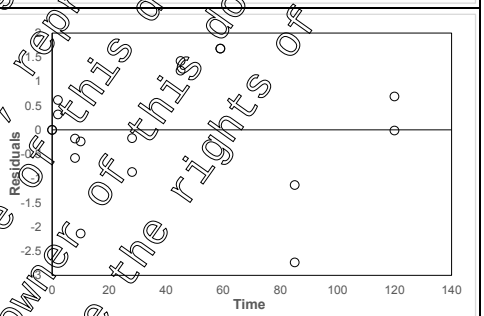
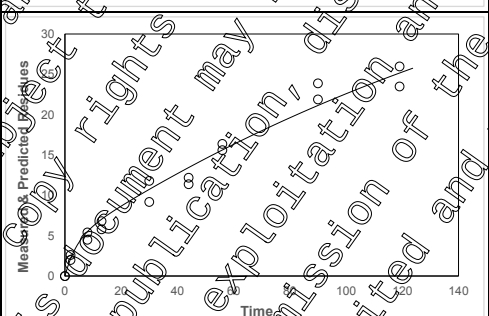
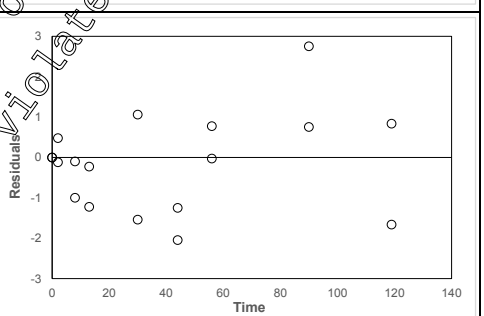
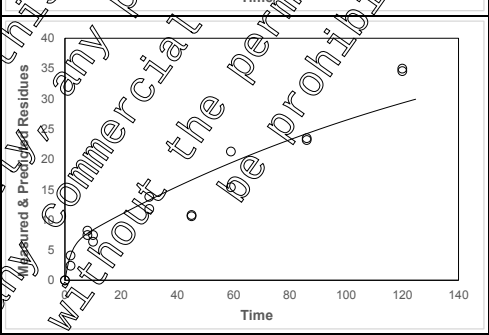
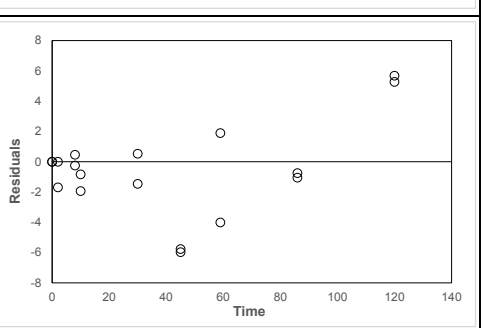
Soil Model Reference	Modelled vs observed	Residuals
Münster DFOP [redacted] (2019)		
Pikeville DFOP [redacted] (2019)		
Sarotti 2 DFOP [redacted] (2019)		

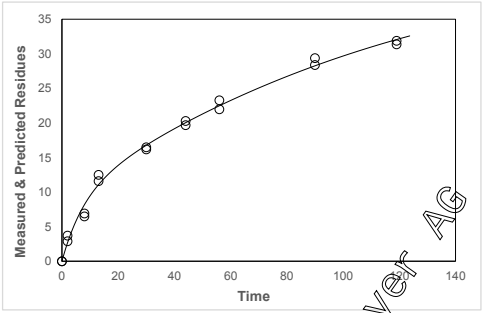
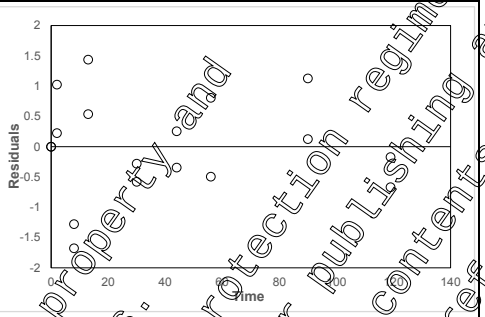
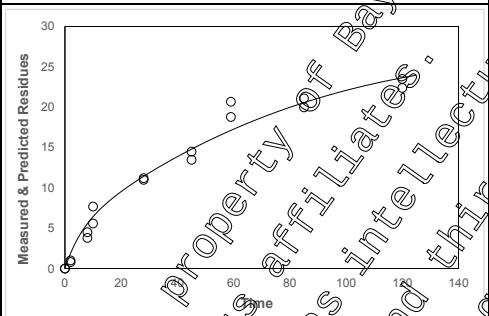
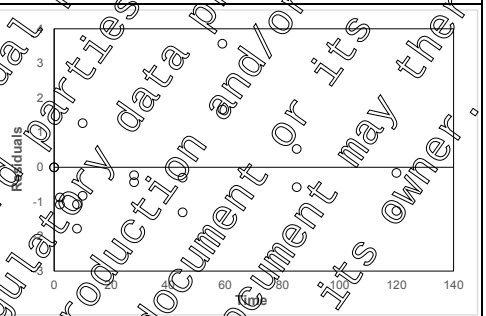
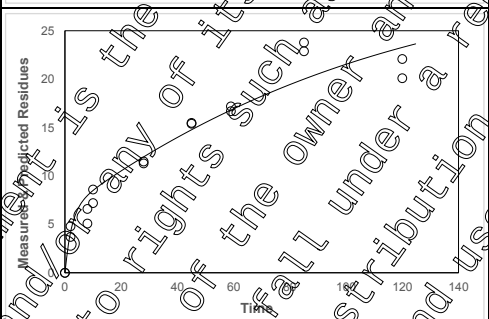
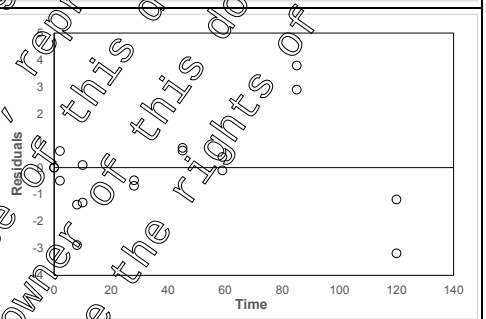
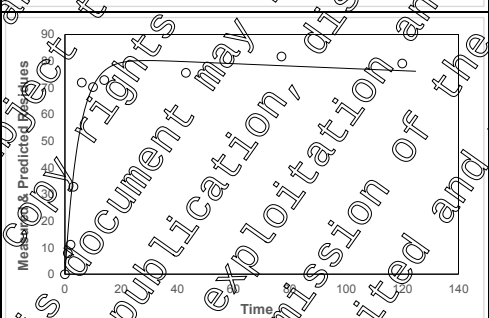
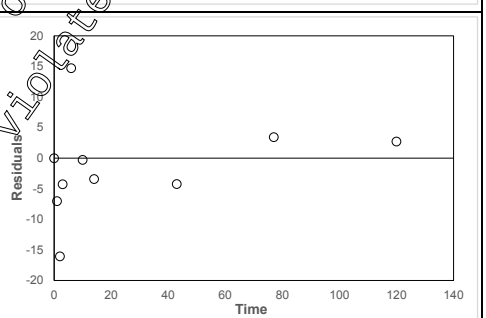
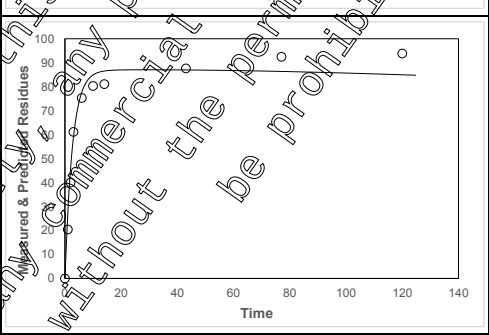
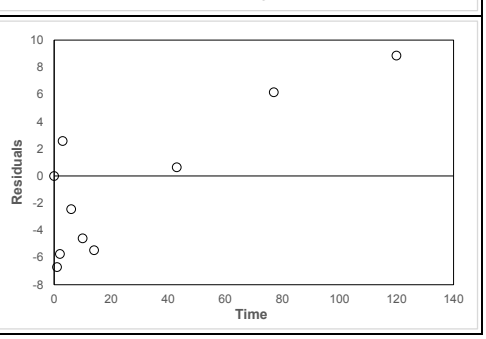
Table 7.1.2.1.1- 96: Graphical representations of best fit models for modelling endpoints for M-01 (AE C653711)

Soil Model Reference	Modelled vs observed	Residuals
Münster SFO [redacted] (2003)		
Sarotti SFO [redacted] (2003)		
Abington (non-sterile) SFO [redacted] (2003a)		
Lamberton SFO [redacted] (2003b)		
Lamberton SFO [redacted] (2003c)		

Soil Model Reference	Modelled vs observed	Residuals
Pikeville SFO [redacted] (2003c)		
Abington SFO [redacted] (2003d)		
Albaro/Marcomcini SFO [redacted] (2016a)		
Great Chishill SFO [redacted] (2016a)		
H[redacted] SFO [redacted] (2016a)		

Soil Model Reference	Modelled vs observed	Residuals
Mas du Coq SFO [redacted] (2016a)		
Parcey Meslay SFO [redacted] (2016a)		
Vilobi d'Ònyar SFO [redacted] (2016a)		
Dollendorf SFO [redacted] (2016b)		
H[redacted] SFO [redacted] (2016b)		

Soil Model Reference	Modelled vs observed	Residuals
L[redacted] SFO [redacted] (2016b)		
[redacted] SFO [redacted] (2016b)		
Abington 2 SFO [redacted](2019)		
Lamberton SFO [redacted](2019)		
Lignieres SFO [redacted](2019)		

Soil Model Reference	Modelled vs observed	Residuals
Münster SFO [redacted] (2019)		
Pikeville SFO [redacted] (2019)		
Sarotti 2 SFO [redacted] (2019)		
Münster SFO [redacted] (2003)		
Pikeville SFO [redacted] (2003)		

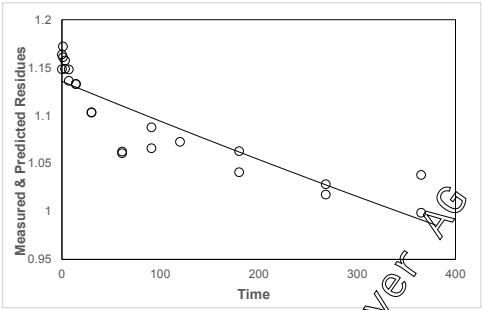
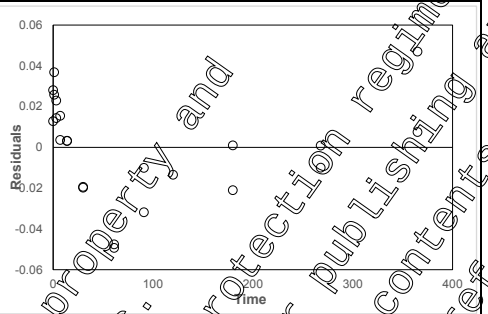
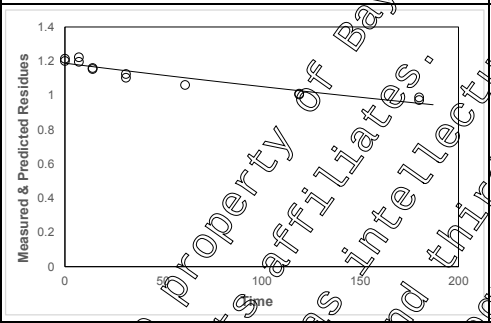
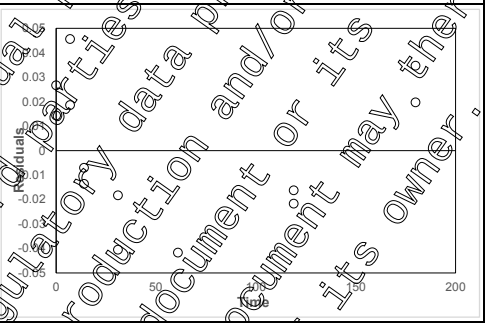
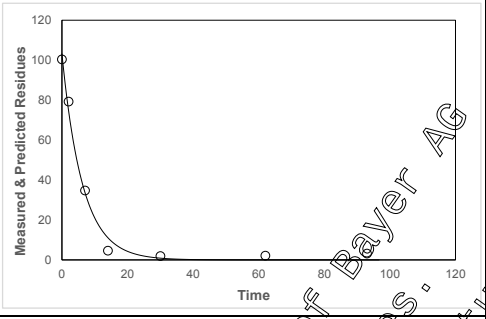
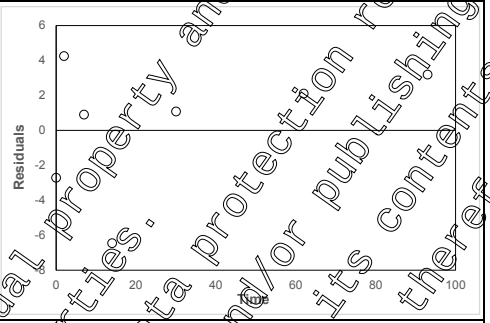
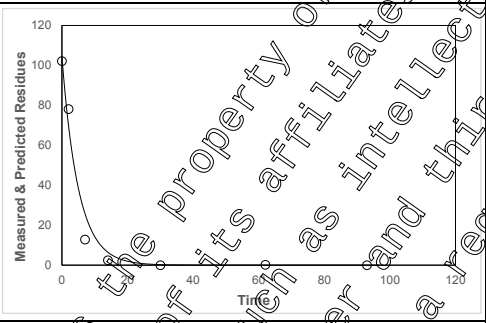
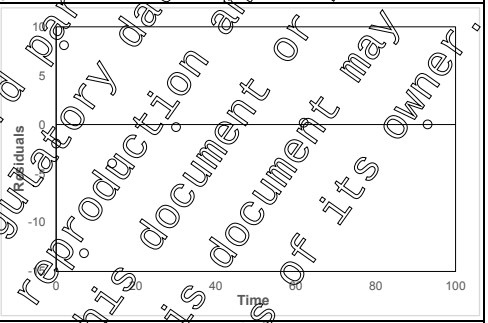
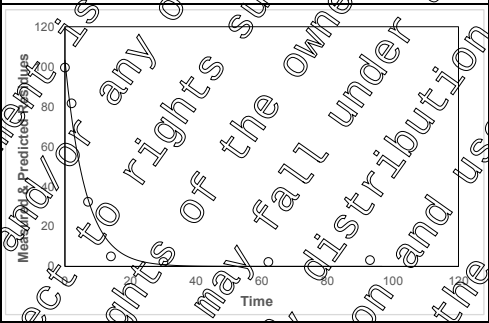
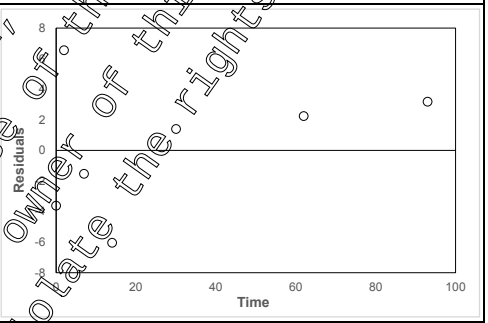
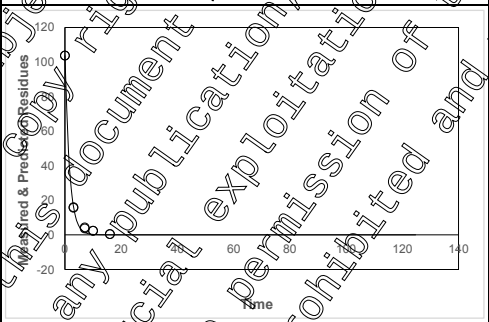
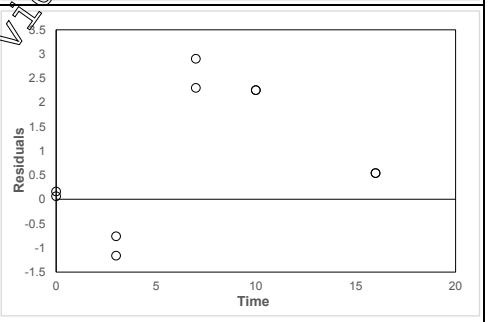
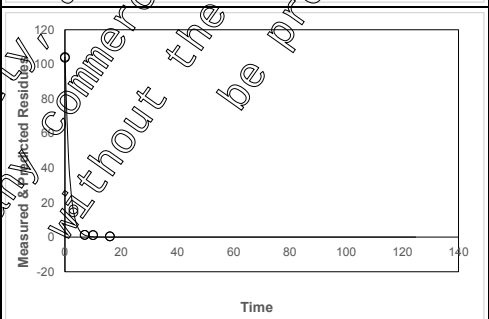
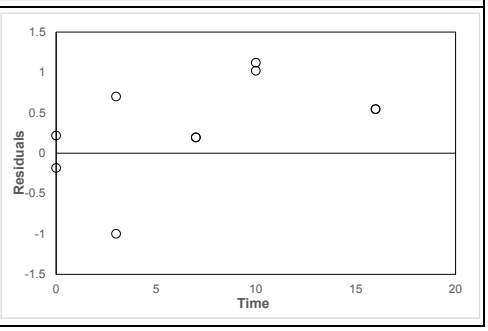
Soil Model Reference	Modelled vs observed	Residuals
Bethany SFO [redacted] (2002)		
North Dakota SFO [redacted] (2002)		

Table 7.1.2.1.1- 97: Graphical representations of best fit models for modelling endpoints for M-02 (AE C657188)

Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003)		
Münster SFO [redacted] (2003)		
Sarotti SFO [redacted] (2003)		
Dollendorf II SFO [redacted] (2017)		
H[redacted] SFO [redacted] (2017)		

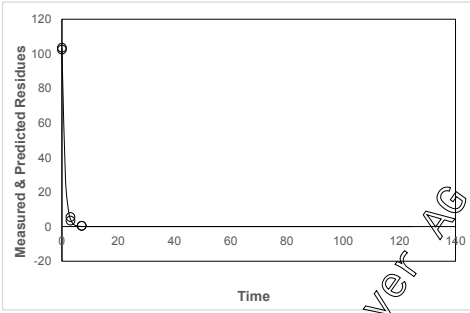
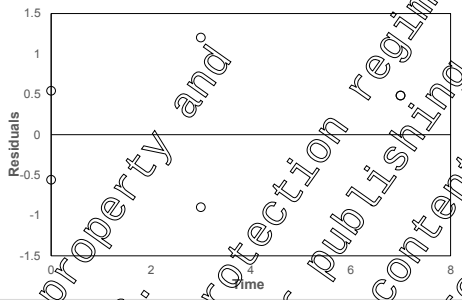
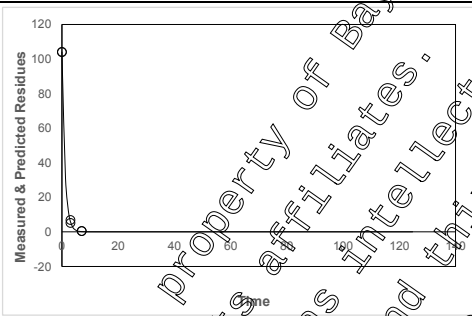
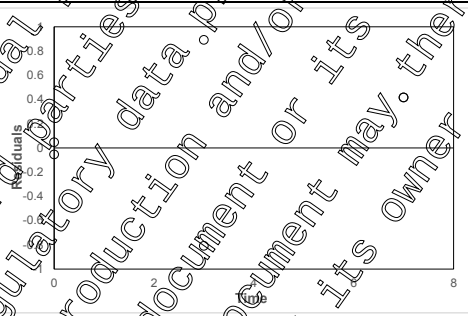
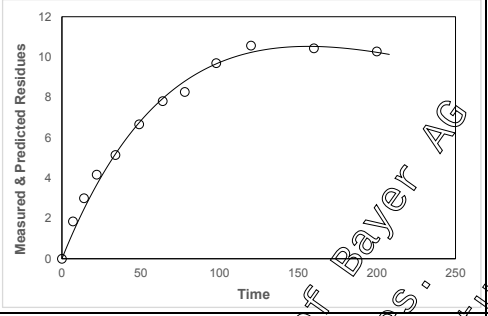
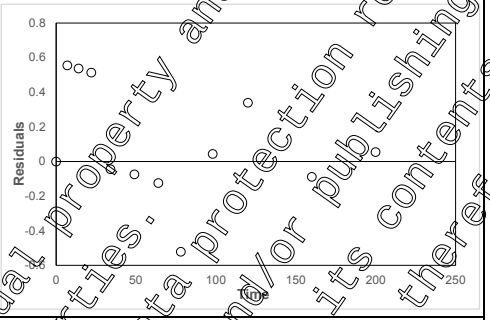
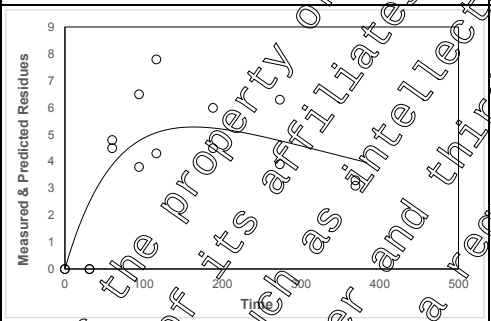
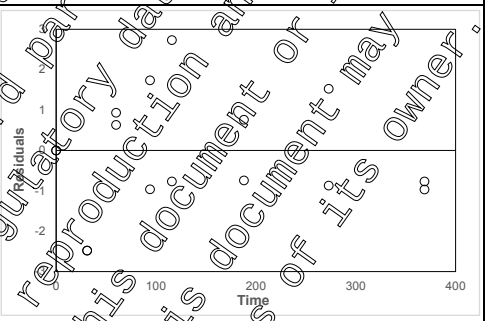
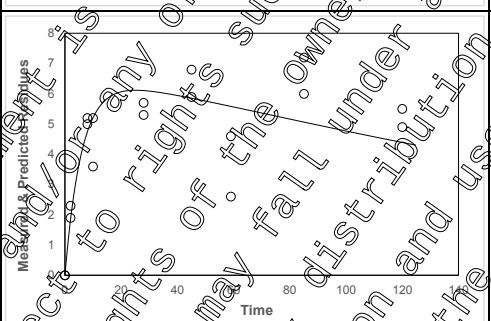
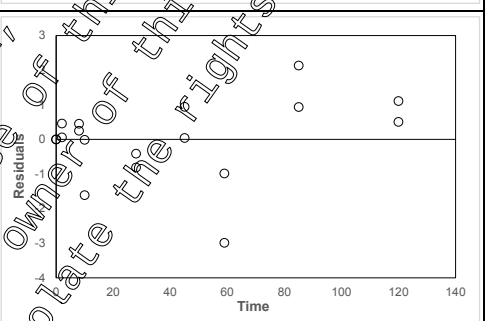
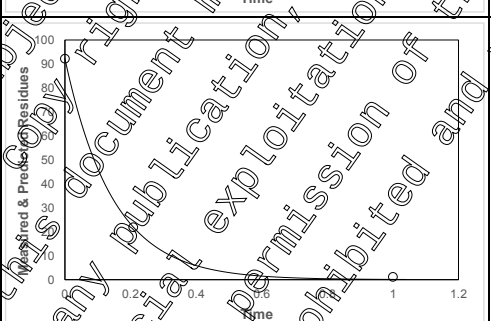
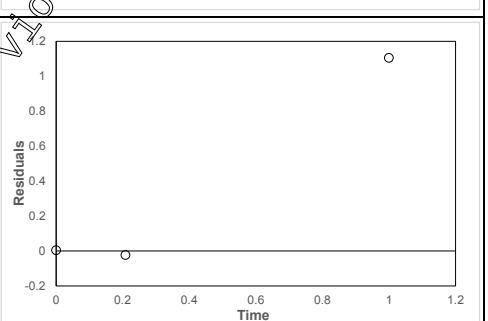
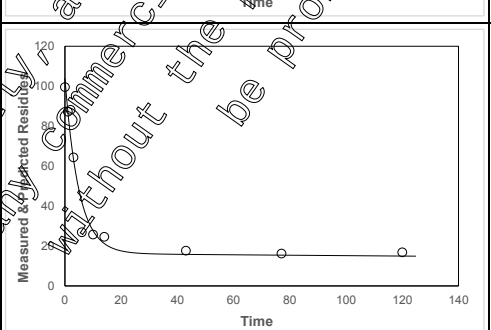
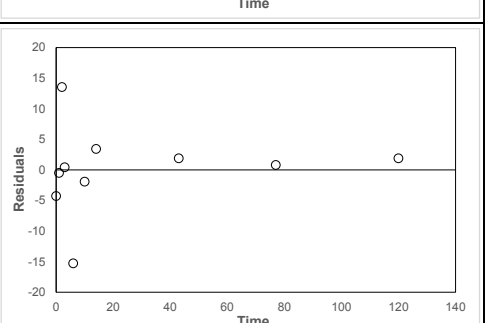
Soil Model Reference	Modelled vs observed	Residuals
L[redacted] SFO [redacted] (2017)		
L[redacted] SFO [redacted] (2017)		

Table 7.1.2.1.1- 98: Graphical representations of best fit models for modelling endpoints for M-03 (AE 0608000)

Soil Model Reference	Modelled vs observed	Residuals
Münster SFO [redacted] (2003)		
Lamberton SFO [redacted] (2003c)		
Pikeville SFO [redacted] (2019)		
Abington SFO [redacted] (2003)		
Münster DFOP [redacted] (2003)		

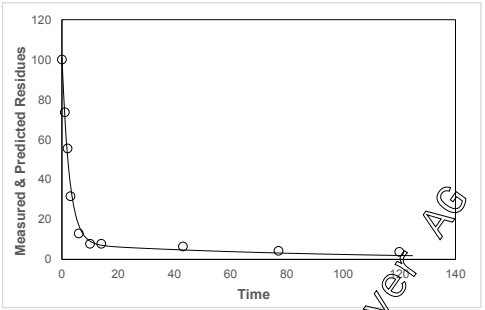
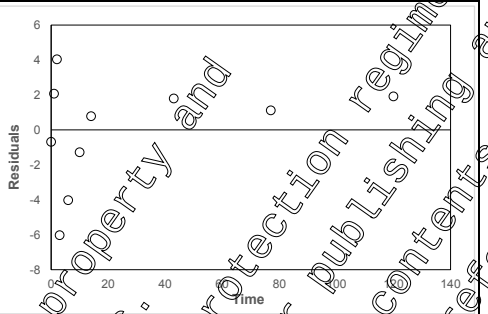
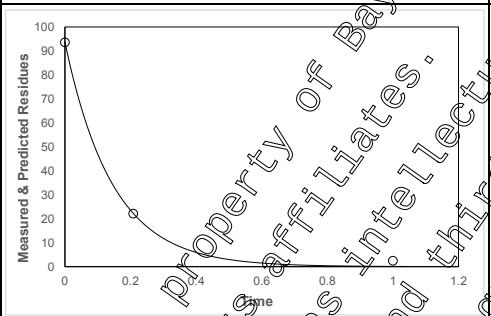
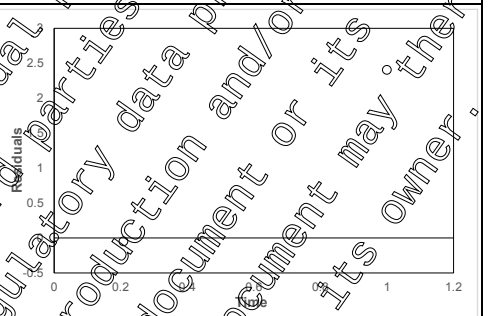
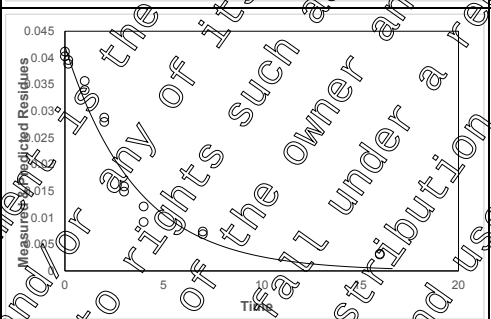
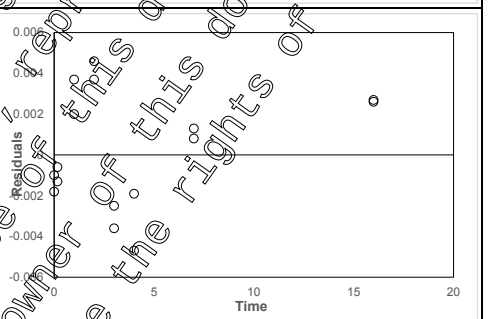
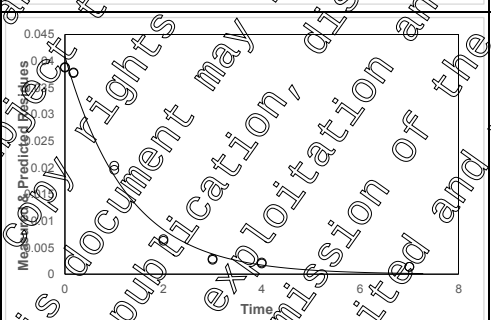
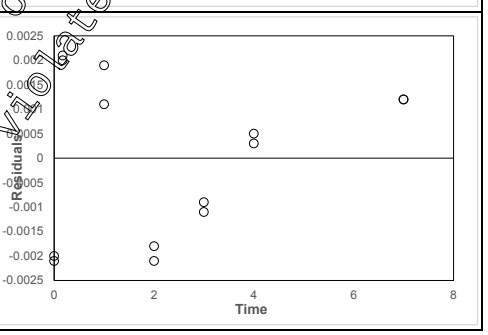
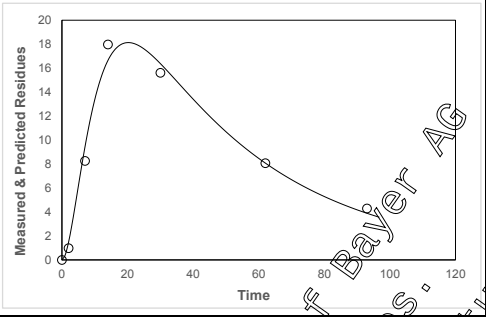
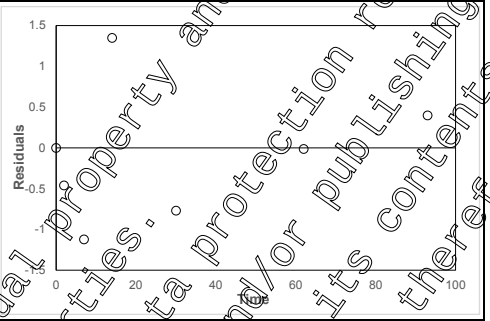
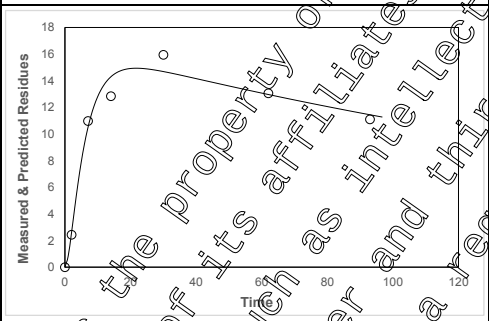
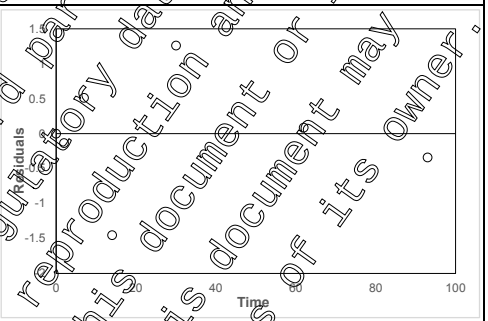
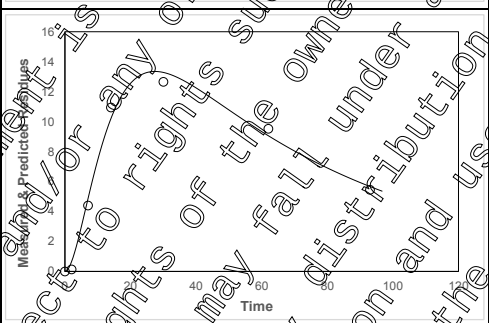
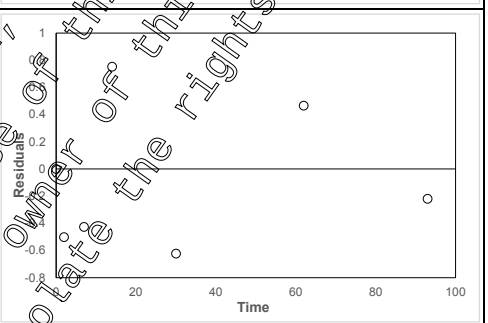
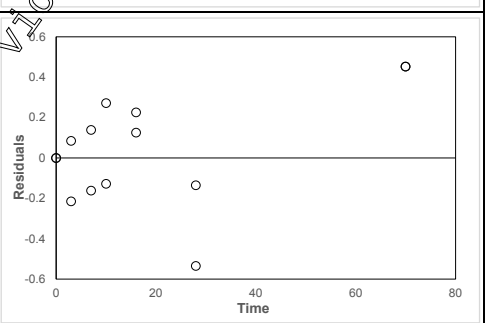
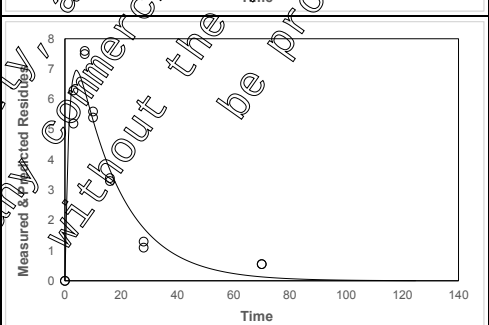
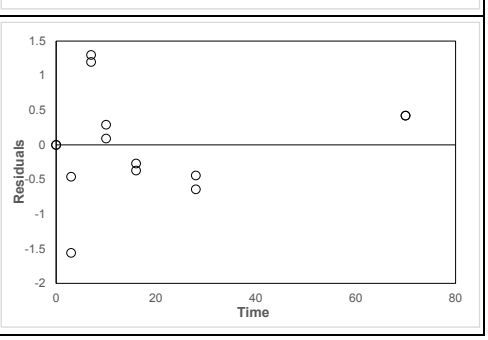
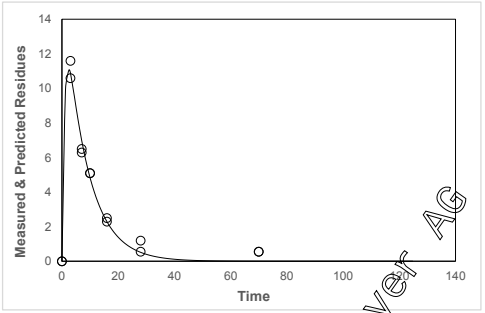
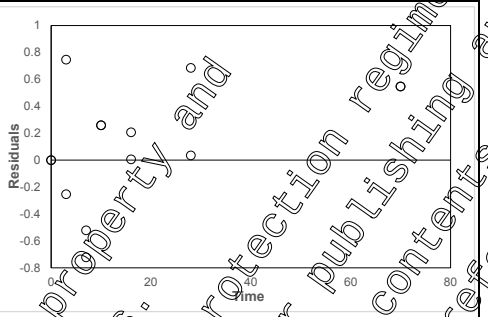
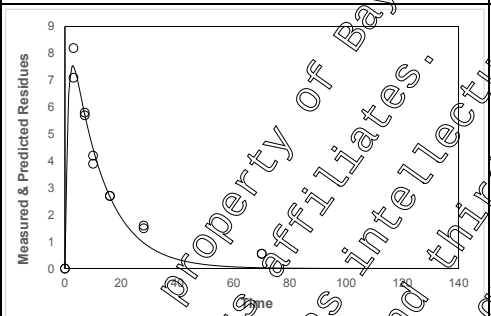
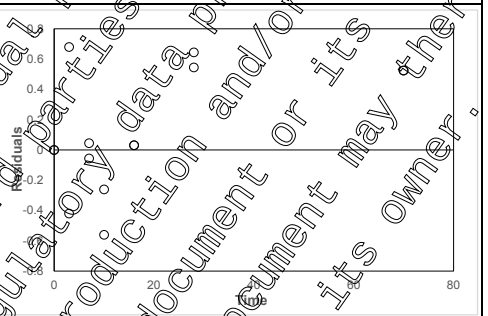
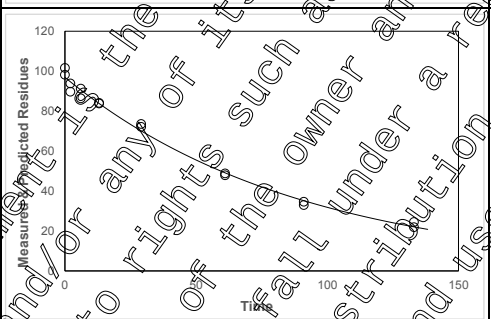
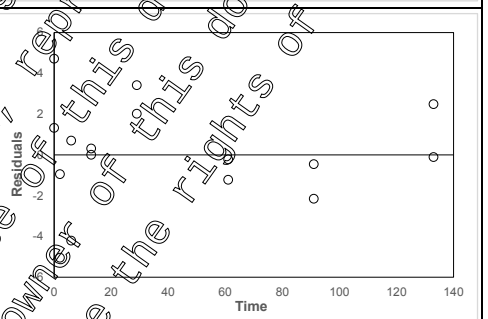
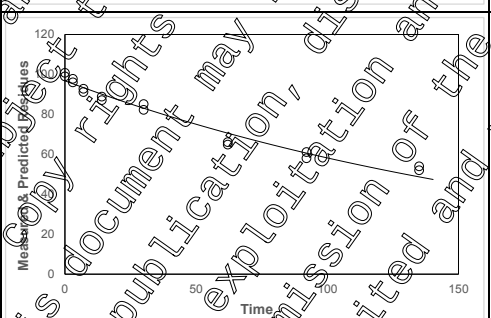
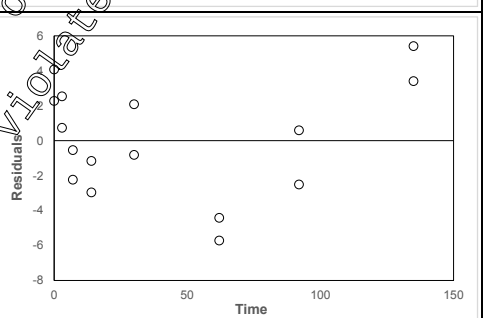
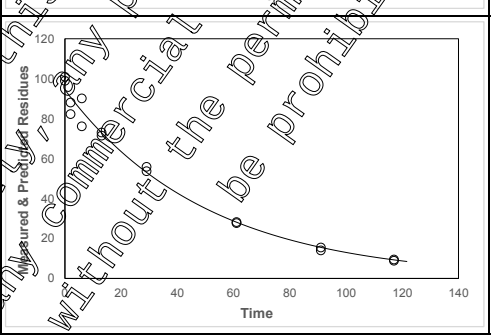
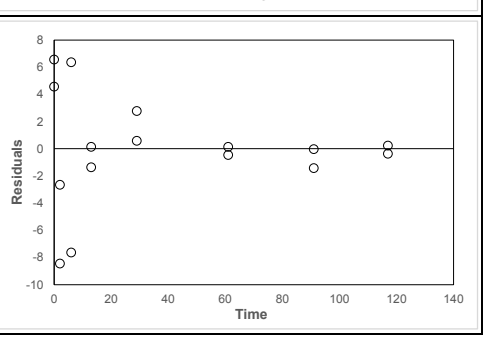
Soil Model Reference	Modelled vs observed	Residuals
Pikeville DFOP [redacted] (2003)		
Sarotti SFO [redacted] (2003)		
Brierlow (BL) SFO [redacted] (2016a)		
H [redacted] SFO [redacted] (2016a)		

Table 7.1.2.1.1- 99: Graphical representations of best fit models for modelling endpoints for M-05 (AE 1344122)

Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003)		
Münster SFO [redacted] (2003)		
Sarotti SFO [redacted] (2003)		
Dollendorf II SFO [redacted] (2017)		
H[redacted] SFO [redacted] (2017)		

Soil Model Reference	Modelled vs observed	Residuals
L[redacted] SFO [redacted] (2017)		
L[redacted] SFO [redacted] (2017)		
Abington SFO [redacted] (2003a)		
Münster SFO [redacted] (2003a)		
Sarotti SFO [redacted] (2003a)		

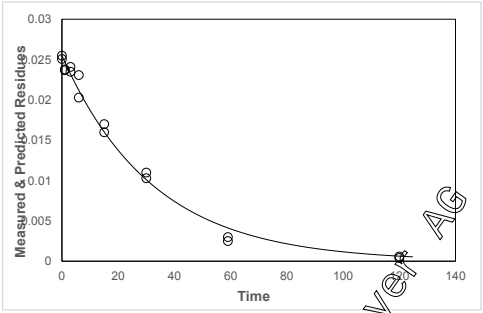
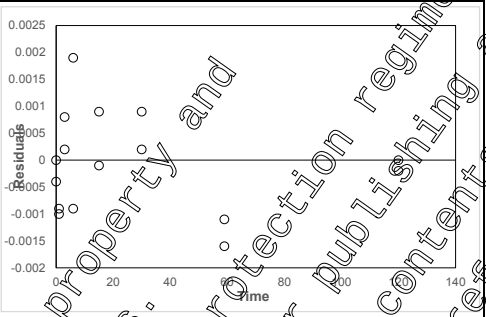
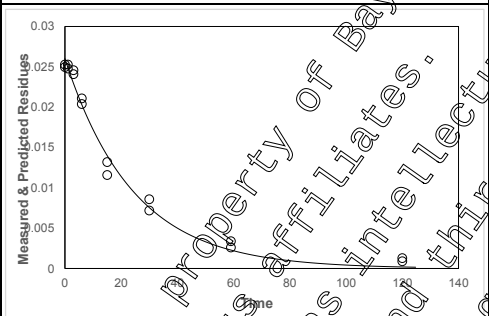
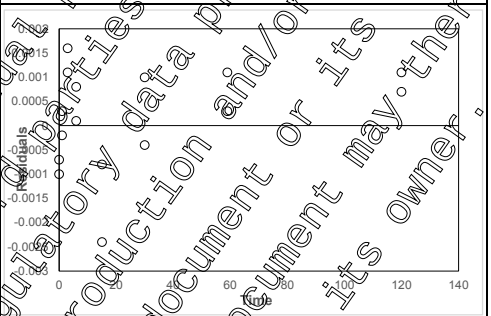
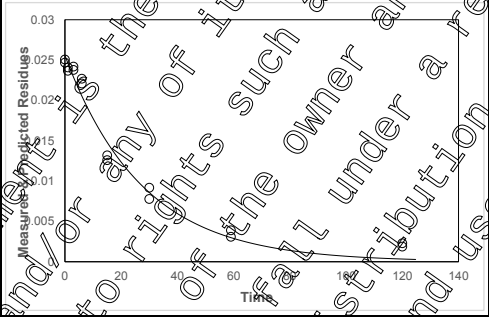
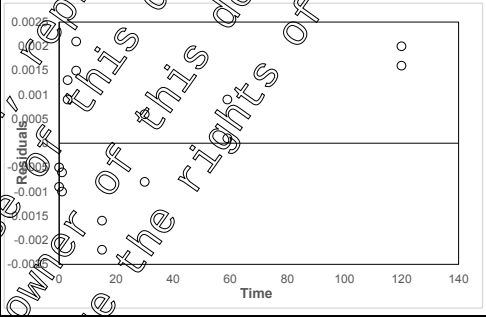
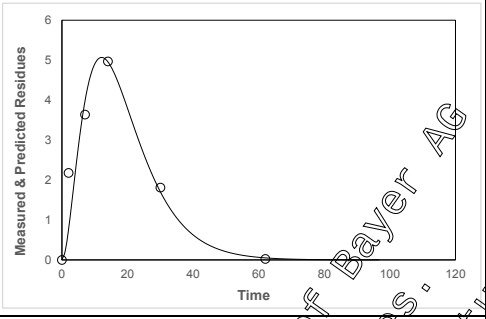
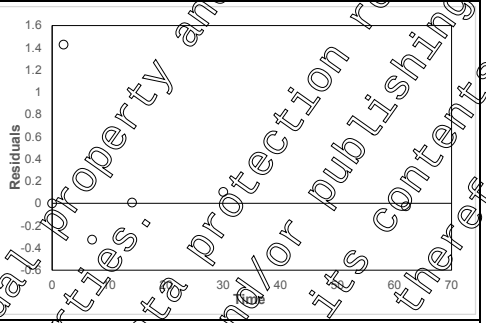
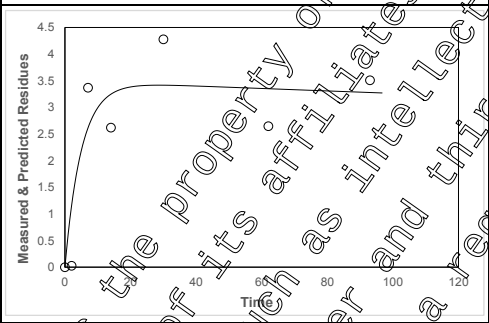
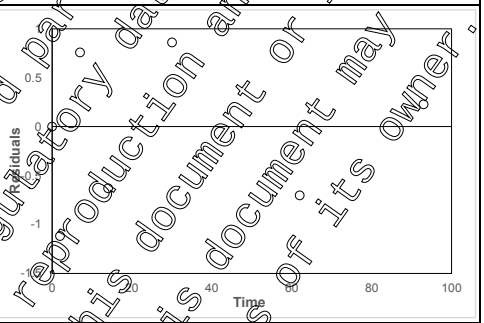
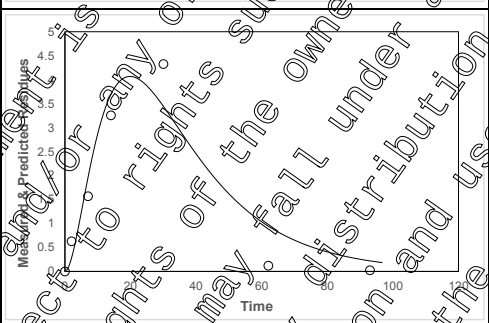
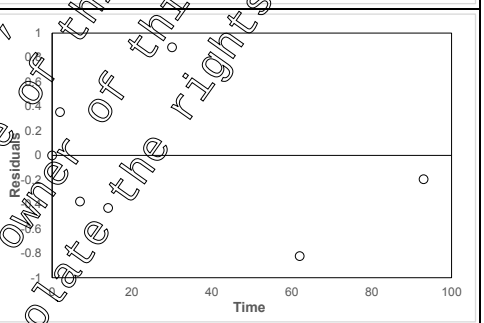
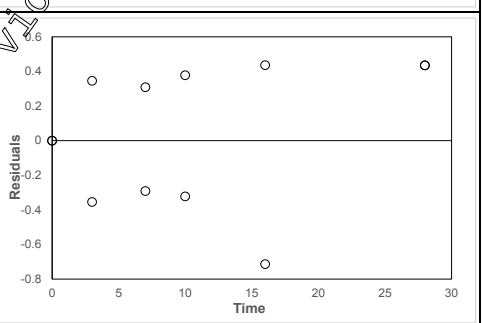
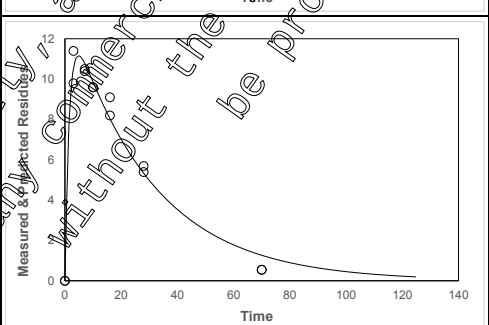
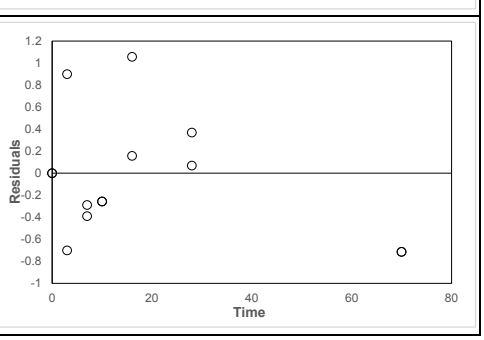
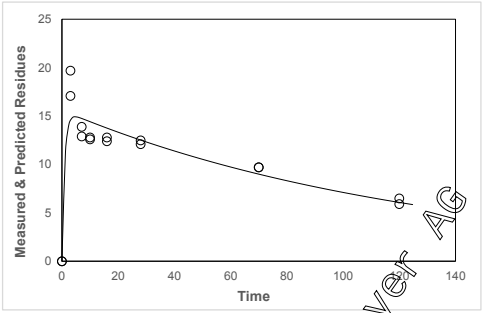
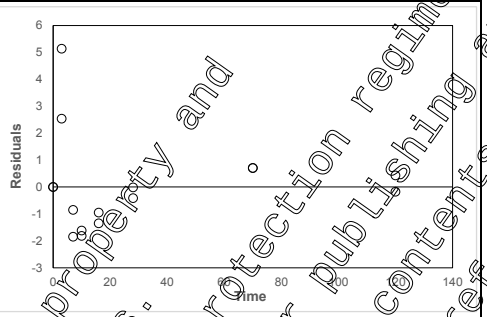
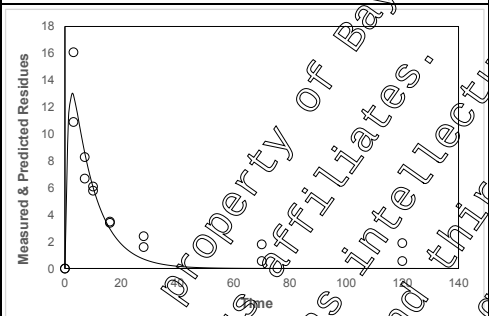
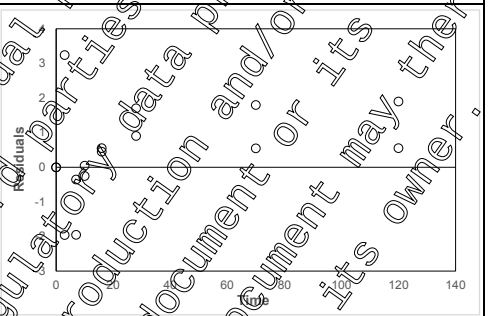
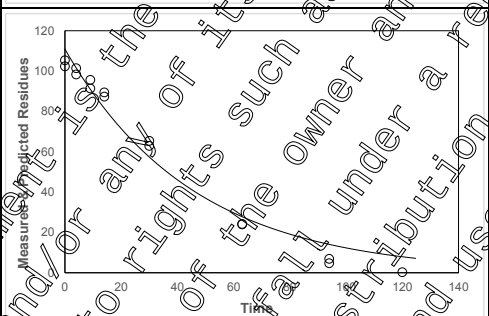
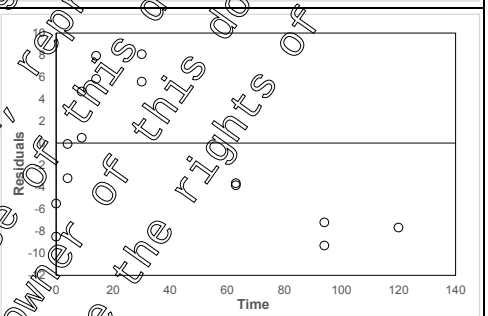
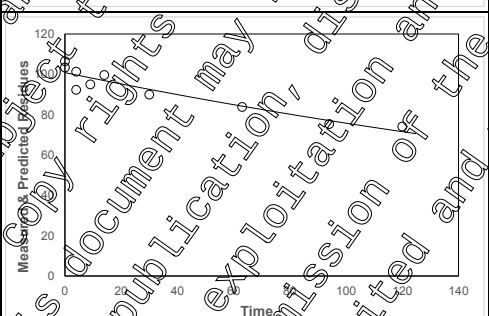
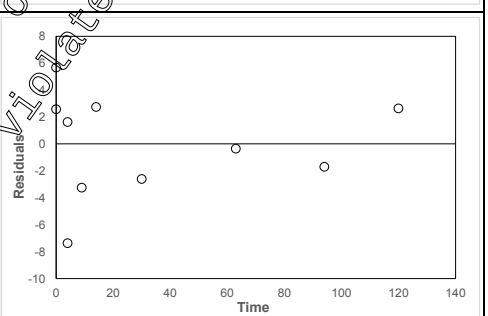
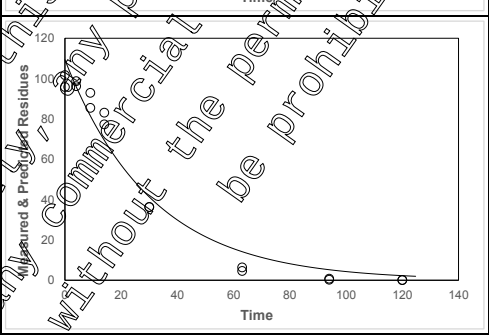
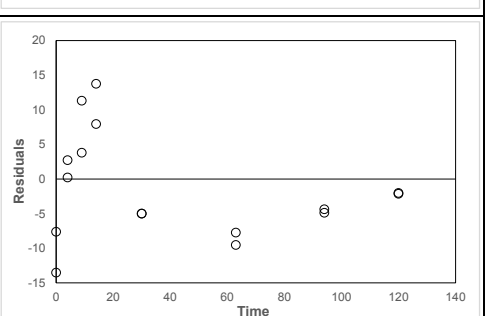
Soil Model Reference	Modelled vs observed	Residuals
H[redacted] SFO [redacted] (2016b)		
L[redacted] SFO [redacted] (2016b)		
L[redacted] SFO [redacted] (2016b)		

Table 7.1.2.1.1- 100: Graphical representations of best fit models for modelling endpoints for M-10 (AE 1344123)

Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003)		
Münster SFO [redacted] (2003)		
Sarotti SFO [redacted] (2003)		
Dollendorf II SFO [redacted] (2017)		
H[redacted] SFO [redacted] (2017)		

Soil Model Reference	Modelled vs observed	Residuals
L[redacted] SFO [redacted] (2017)		
L[redacted] SFO [redacted] (2017)		
Abington SFO [redacted] (2003b)		
Münster SFO [redacted] (2003b)		
Sarotti SFO [redacted] (2003b)		

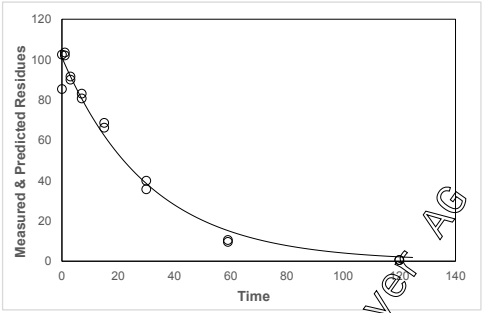
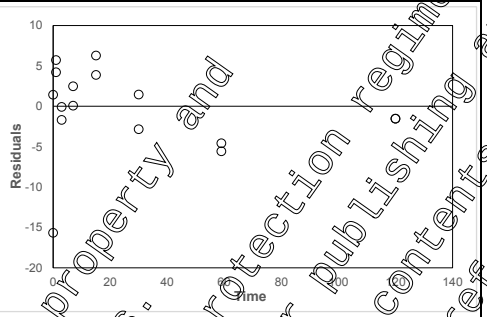
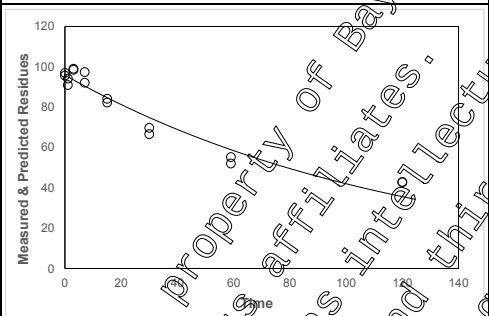
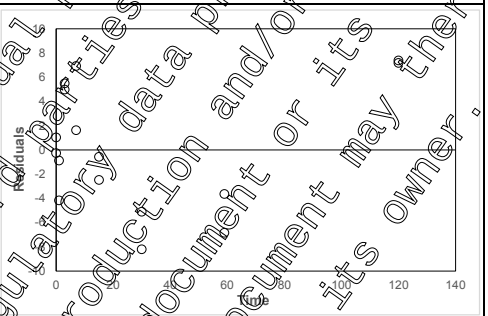
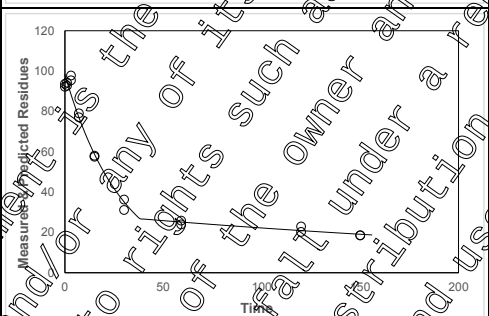
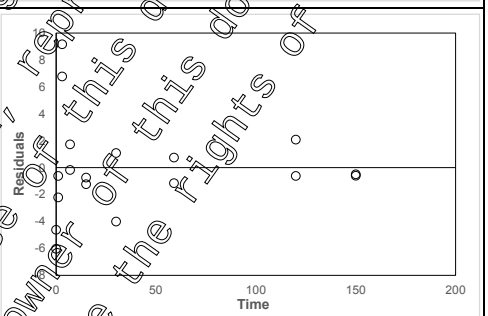
Soil Model Reference	Modelled vs observed	Residuals
H[redacted] SFO [redacted] (2016c)		
L[redacted] SFO [redacted] (2016c)		
L[redacted] HS [redacted] (2016c)		

Table 7.1.2.1.1- 101: Graphical representations of best fit models for modelling endpoints for M-11/M-12

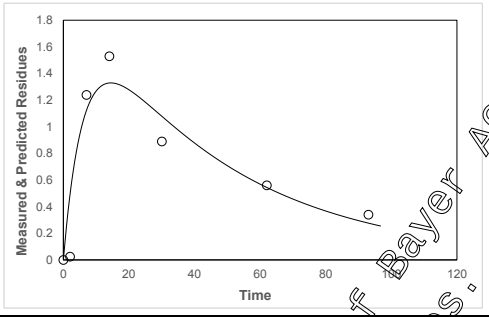
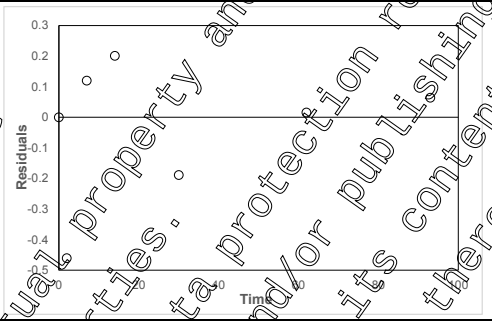
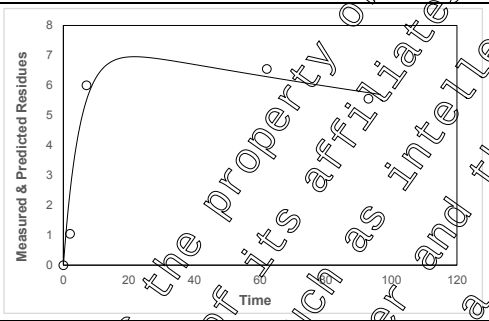
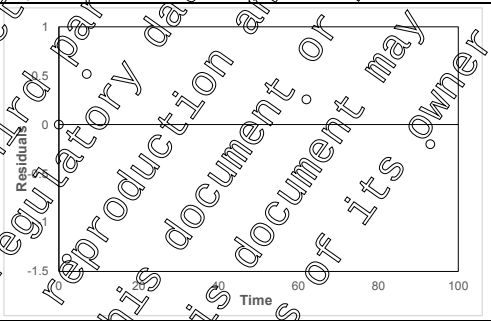
Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003)		
Münster SFO [redacted] (2003)		

Table 7.1.2.1.1- 102: Graphical representations of best fit models for modelling endpoints for M-13

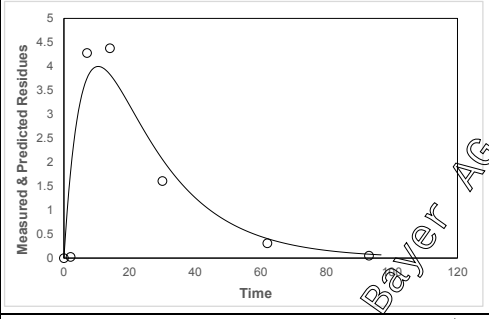
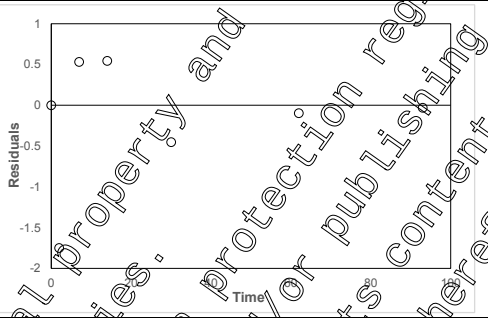
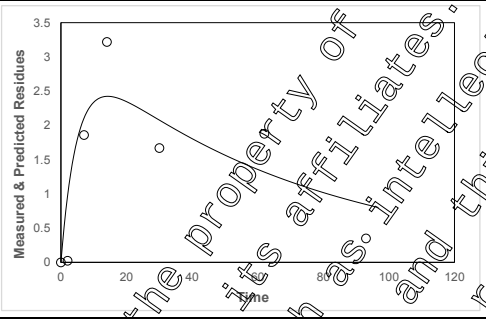
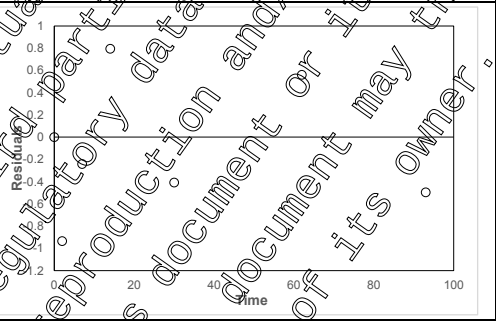
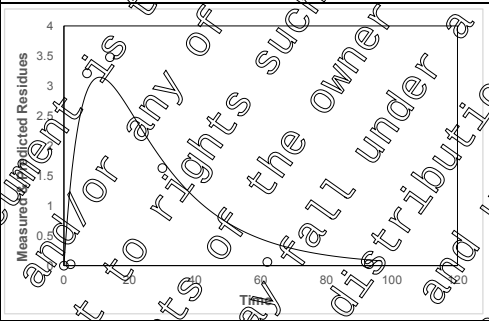
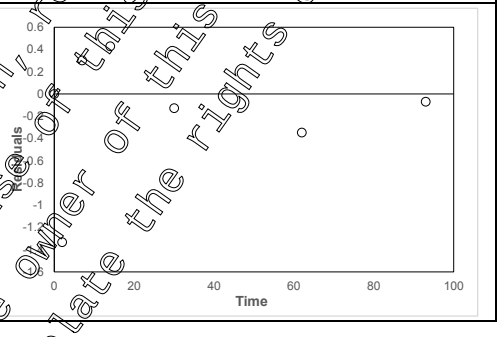
Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003)		
Münster SFO [redacted] (2003)		
Sarotti SFO [redacted] (2003)		

Table 7.1.2.1.1- 103: Graphical representations of best fit models for modelling endpoints for M-14 (AE 1388273)

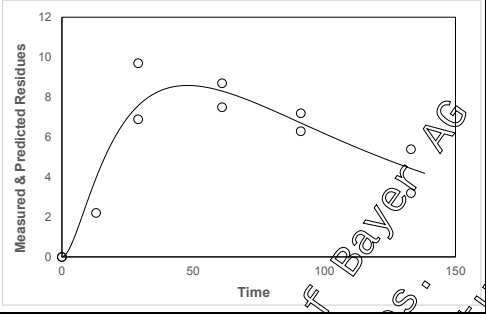
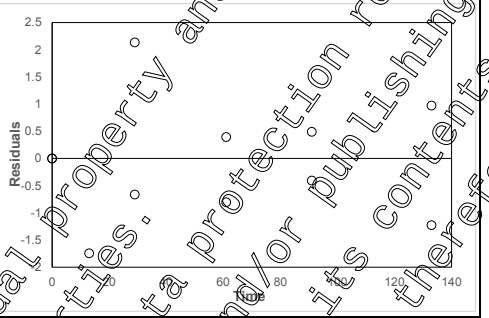
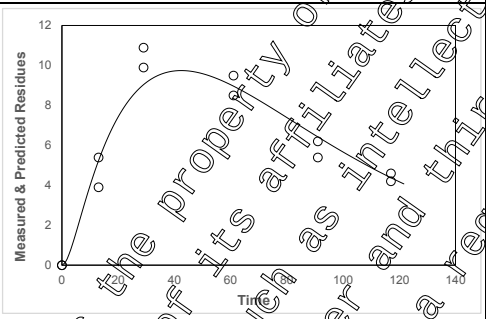
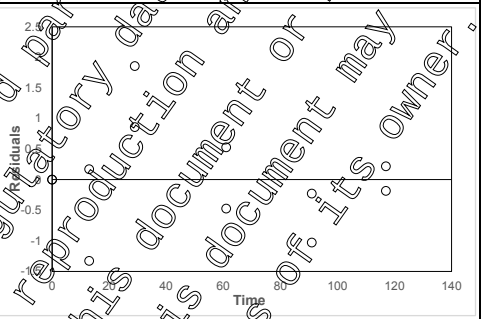
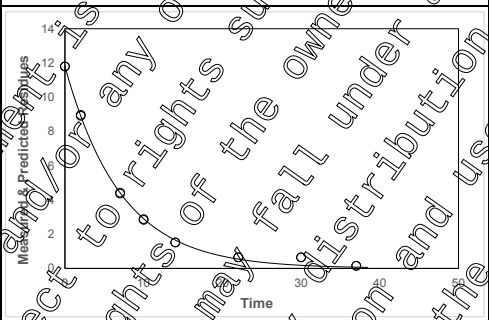
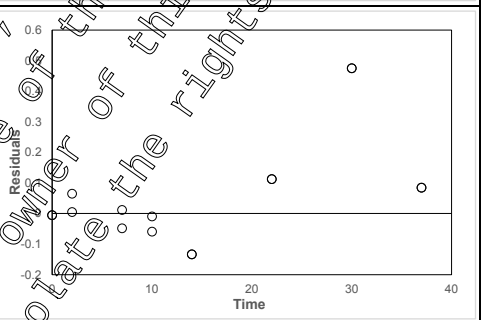
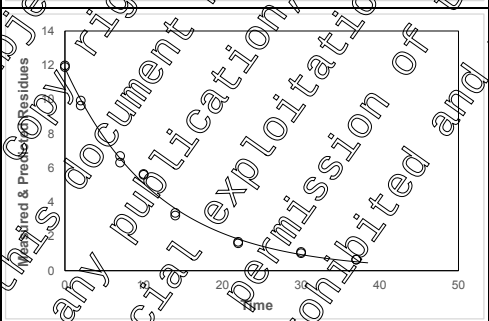
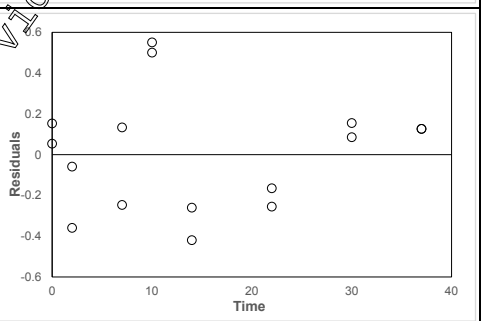
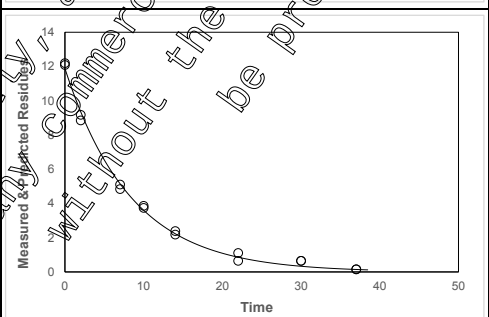
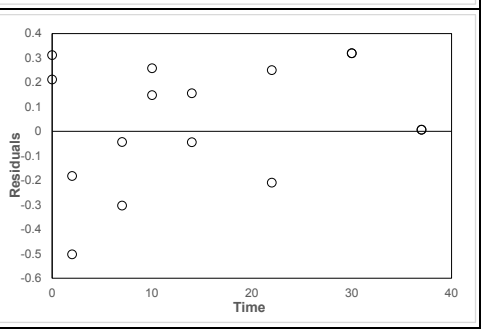
Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003a)		
Sarotti SFO [redacted] (2003a)		
Abington SFO [redacted] (2003)		
Münster SFO [redacted] (2003)		
Sarotti (SLS) SFO [redacted] (2003)		

Table 7.1.2.1.1- 104: Graphical representations of best fit models for modelling endpoints for M-15 (AE 1413903)

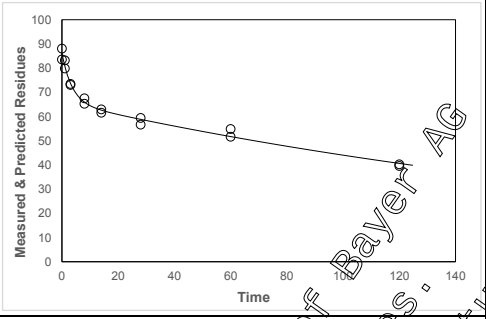
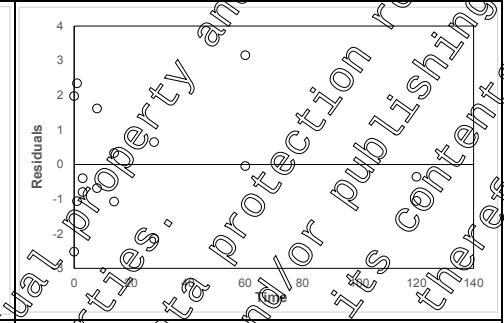
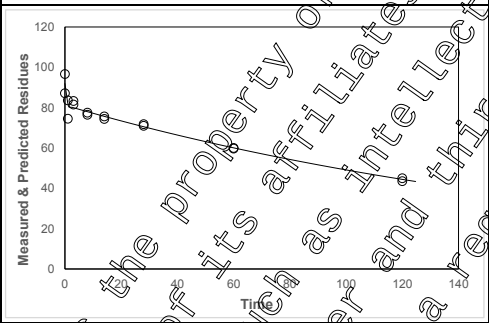
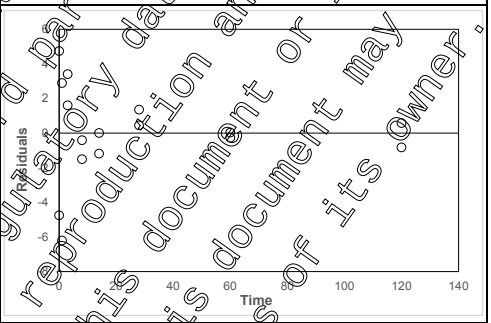
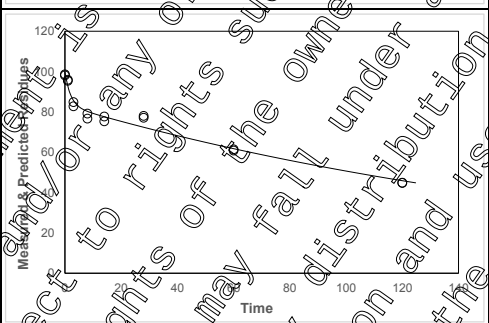
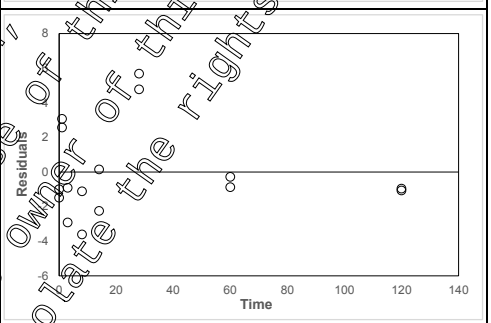
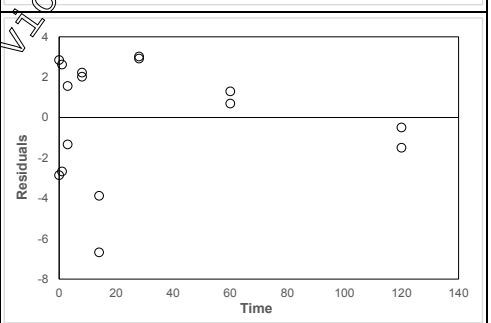
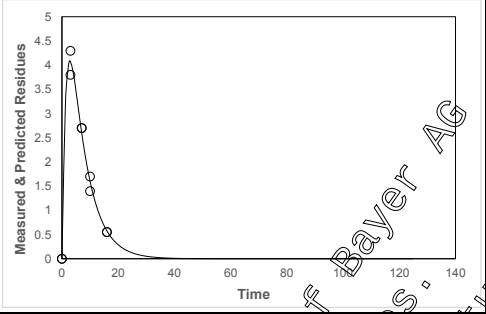
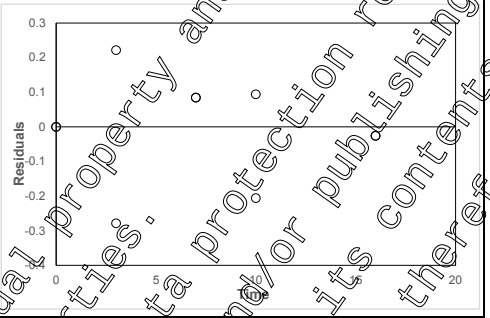
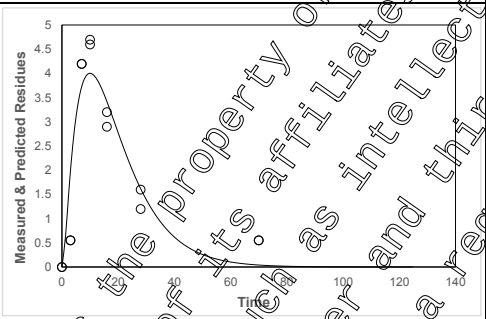
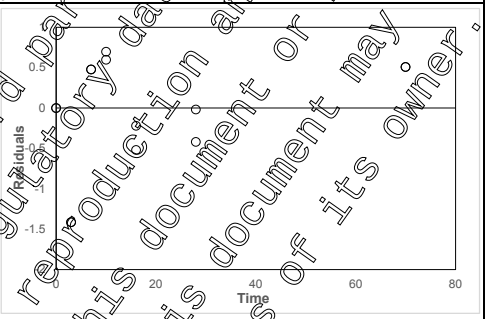
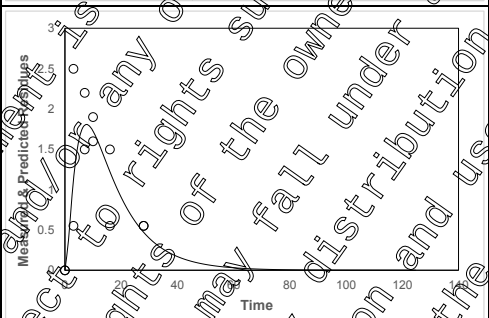
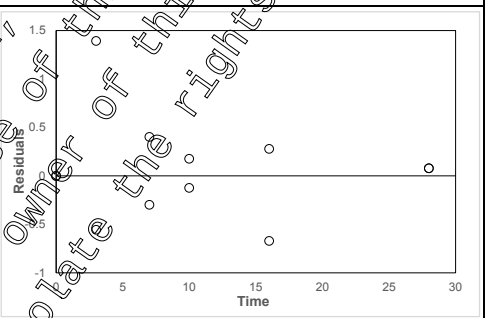
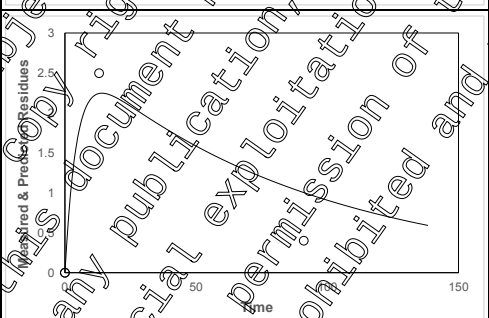
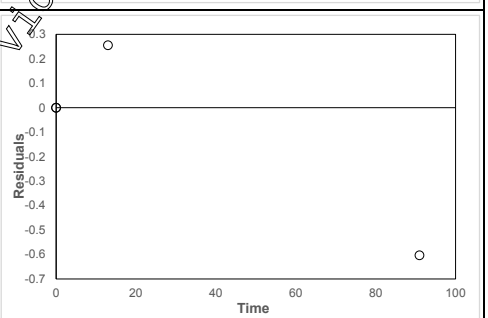
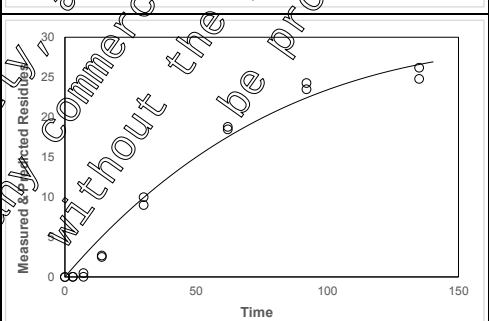
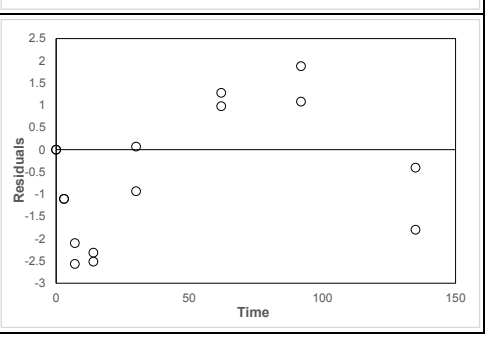
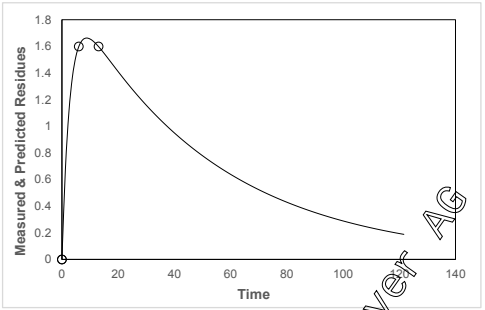
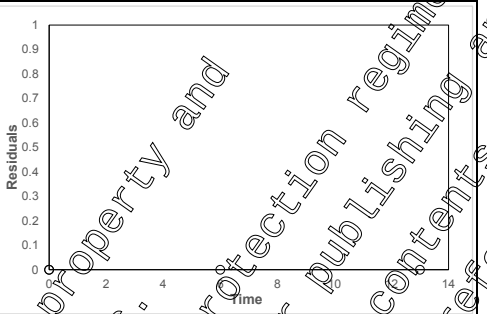
Soil Model Reference	Modelled vs observed	Residuals
Dollendorf II DFOP [redacted] (2016d)		
H[redacted] DFOP [redacted] (2016d)		
L[redacted] DFOP [redacted] (2016d)		
L[redacted] DFOP [redacted] (2016d)		

Table 7.1.2.1.1- 105: Graphical representations of best fit models for modelling endpoints for M-20 (BCS-BX16566)

Soil Model Reference	Modelled vs observed	Residuals
H SFO (2017)		
L SFO (2017)		
L SFO (2017)		
Abington SFO (2003a)		
Münster SFO (2003a)		

Soil Model Reference	Modelled vs observed	Residuals
Sarotti SFO [redacted] (2003a)		

III. Conclusion

Data from nineteen aerobic soils studies has been used in a kinetic evaluation to derive parameters suitable for use as trigger endpoints and modelling endpoints for fluopicolide and 11 of its metabolites. The metabolites included in the analysis were: M-01 (AE C653711, BAM), M-02 (AE C657188, PCA), M-03 (AE 0608000), M-05 (AE 1344122), M-10 (AE 1344123), M-11/12, M-13, M-14 (AE 1388273), M-15 (AE 1413903) and M-20 (BCS-BX10566).

Assessment and conclusion by applicant:

The modelling report was conducted according to FOCUS Degradation Kinetics (2006, 2014) and is considered valid to assess trigger and modelling endpoints for fluopicolide and its metabolites in soil under laboratory conditions.

CA 7.1.2.1.2 Aerobic degradation of metabolites, breakdown and reaction products

The aerobic degradation of the metabolites of fluopicolide was evaluated in seven separate laboratory studies during the previous EU review and these studies are still considered as reliable to assess their behaviour in soil (KCA 7.1.2.1.2/01, KCA 7.1.2.1.2/02, KCA 7.1.2.1.2/03, KCA 7.1.2.1.2/04, KCA 7.1.2.1.2/05 and KCA 7.1.2.1.2/06). A study with M-15 was subsequently supplied as confirmatory data (KCA 7.1.2.1.2/08). A superseded kinetic evaluation report (KCA 7.1.2.1.2/07) is listed. For procedural reasons, the previously submitted kinetic evaluation report has to be included under Point KCA 7.1.2.1.2 in the current dossier but this report is fully superseded by the latest kinetic evaluation report (see KCA 7.1.2.1.1/10, M-685680-01-1). Four additional aerobic soil studies, KCA 7.1.2.1.2/09, KCA 7.1.2.1.2/10, KCA7.1.2.1.2/11 and KCA 7.1.2.1.2/12 are provided as new data not yet reviewed.

Studies have been conducted for M-01 (AE C653711), M-02 (AE C657188), M-03 (AE 0608000), M-05 (AE 1344122), M-10 (AE 1344123), M-14 (AE 1388273) and M-15 (AE 1413903).

Metabolite	Report reference	Author, Year	Comment
M-01	KCA 7.1.2.1.2/01 M-234320-01-1	[redacted] 2002	Submitted and reviewed for first approval of fluopicolide, 2005. Considered as supporting data.
M-03	KCA 7.1.2.1.2/02 M-241188-01-1	[redacted] 2003	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
M-02	KCA 7.1.2.1.2/03 M-219824-01-1	[redacted] 2003	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
M-05	KCA 7.1.2.1.2/04 M-241410-01-2	[redacted] 2003	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
M-10	KCA 7.1.2.1.2/05 M-241411-01-2	[redacted] 2003	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
M-14	KCA 7.1.2.1.2/06 M-234149-01-2	[redacted] 2003	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
Modelling report	KCA 7.1.2.1.2/07 M-233743-01-1	[redacted] 2004	Submitted and reviewed for first approval of fluopicolide, 2005. Superseded by KCA 7.1.2.1.1/10 (M-685680-01-1).
M-15	KCA 7.1.2.1.2/08 M-585202-01-1	[redacted] 2017	Submitted as additional information regarding fluopicolide confirmatory data on metabolite M-15, 2018. Reviewed and accepted by RMS Austria.
M-02	KCA 7.1.2.1.2/09 M-581364-01-1	[redacted] 2017	New data not yet reviewed.
M-03	KCA 7.1.2.1.2/10 M-565219-01-1	[redacted] 2016	New data not yet reviewed.
M-05	KCA 7.1.2.1.2/11 M-565223-01-1	[redacted] 2016	New data not yet reviewed.
M-10	KCA 7.1.2.1.2/12 M-565224-01-1	[redacted] 2016	New data not yet reviewed.

Data Point:	KCA 7.1.2.1.2/01
Report Author:	[redacted]
Report Year:	2002
Report Title:	Aerobic soil metabolism of 14C-2,6-dichlorobenzamide (BAM)
Report No:	C034067
Document No:	M-234320-01-1
Guideline(s) followed in study:	USEPA (=EPA): Subdivision N, 161-1
Deviations from current test guideline:	Yes. According to OECD 307 soil laboratory studies should not normally exceed 120 days. The soils were incubated under aerobic conditions for 365 and 180 days, as required by US EPA guidelines at the time the study was conducted. Final biomass samples are low compared to initial measurements.
Previous evaluation:	yes, evaluated and accepted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The biotransformation of [^{14}C-1-phenyl]-M-01 (2,6-dichlorobenzamide, AE C653711) was investigated under aerobic laboratory conditions in the laboratory in two US soils Bethany (sandy loam) and North Dakota (sandy loam) following incubation in the dark at 25 °C at a moisture content equivalent to 75% of ⅓ bar for up to 365 days and 180 days respectively. The nominal test concentration was 1.2 mg test item/kg soil, equivalent to a field application rate of 1.6 kg/ha in Bethany soil and 1 kg/ha in North Dakota soil. The radiochemical purity and specific activity were 96.74% and 6.86 mCi/mmol (equivalent to 1.34 MBq/mg), respectively.

Soil	Texture (USDA)	pH (water)	% Organic Matter
Bethany	Sandy loam	4.8	1.6
North Dakota	Sandy loam	7.7	9.6

Samples of Bethany soil were taken for extraction and analysis immediately after treatment (day 0) and 1, 3, 7, 14, 30, 61, 91, 120, 180, 268 and 365 days of incubation and samples of North Dakota soil after 0, 7, 14, 31, 61, 119 and 180 days of incubation. Soil samples were exhaustively extracted with three successive extractions at ambient temperature with methanol, water (or methanol / water (1:1 v/v) for 0 to 7 DAT samples of Bethany soil) and finally with acetonitrile. The soil residue was then extracted by soxhlet extraction with methanol followed by a final soxhlet extraction with water. Pooled ambient soil extracts and Soxhlet extracts were analysed by reverse phase high performance liquid chromatography (HPLC).

Recovery of radioactivity was quantitative throughout the study. Overall mean mass balances were 98.8% AR for Bethany soil and 98.7% AR for North Dakota soil.

Total extractable radioactivity decreased from 99.6% AR at DAT 0 to 86.4% by DAT 365 in Bethany soil and from 103.6% AR at DAT 0 to 82.2% by DAT 180 in North Dakota soil. The amount of radioactivity extracted by ambient extraction declined from *ca*. 98% AR in both soils at DAT 0 to 69% AR by DAT 365 in Bethany soil and to 62% AR by DAT 180 in North Dakota soil. With time, increasing amounts of radioactivity could only be extracted by repeated soxhlet extraction in both soils.

In both soils, non-extractable radioactivity increased slowly but remained low, reaching maxima of 5.1% AR in Bethany soil by DAT 180 and thereafter remained at this level until DAT 365, and 4.6% AR in North Dakota soil by DAT 180, the end of the incubation period.

The maximum amount of carbon dioxide formed was 6.1% AR in Bethany soil by the end of the study (DAT 365) and 8.2% AR in North Dakota soil (DAT 180). No significant levels of organic volatiles were observed (≤ 0.1% AR).

After 365 days incubation at 25 °C in Bethany soil, M-01 degraded to 86% of the applied radioactivity while in North Dakota soil, M-01 declined to 82% AR at termination of the study after 180 days. The levels of M-01 extracted by ambient extraction decreased steadily throughout the study with significant quantities only extractable by soxhlet extraction at longer incubation intervals. M-01 was the principal radiolabelled component detected in soil extracts. In addition, a number of very minor unidentified components (maximum 0.6%) were detected in aqueous soxhlet extracts, one which was tentatively identified 2,6-dichlorobenzoic acid.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} values of 3461 and 568.8 days in Bethany and North Dakota soils, respectively. Corresponding DT_{50} values were > 10,000 and 1889 days.

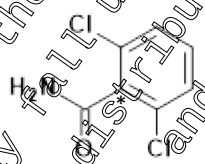
Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)
Bethany (sandy loam)	DFOP	3461	>10000	0.636
North Dakota (sandy loam)	SFO	568.8	1889	1.97

I. Materials and Methods

A. Materials

1. Test Item

[^{14}C-1-Phenyl]-M-01 (referred to as BAM, 2,6-dichlorobenzamide in the report)



* Denotes position of [^{14}C]-radiolabel

Specific Activity: 6.86 mCi/mmol

Radiochemical Purity: 96.74% (HPLC)

Sample/Batch ID: DUPH016

2. Test Soils

The study was performed using two US soils collected from the field, sieved to 2 mm and stored under refrigerated aerobic conditions for a total of 47 and 95 days prior to dispersing into incubations tubes. The physico-chemical characteristics are summarized in Table 7.1.2.1.2- 1.

Table 7.1.2.1.2- 1: Physico-chemical properties of test soils

Parameter		Soil	
Soil Designation		Bethany	North Dakota
Geographic Location			
City		Bethany, Connecticut	Northwood Township, Grand Forks County, North Dakota
Country		USA	USA
Textural Classification (USDA)		Sandy loam	Sandy loam
Sand [50 - 2000 µm]	(%)	74	62
Silt [2 – 50 µm]	(%)	20	28
Clay [< 2 µm]	(%)	6	10
pH			
in H_2O (1:1)		4.8	7.7
Organic Matter (%)		1.6	9.6
Cation Exchange Capacity (meq/100 g)		7	23.1
Calcium (ppm)		400	3100
Magnesium (ppm)		60	430
Sodium (ppm)		32	24
Potassium (ppm)		72	790
Hydrogen (ppm)		42	18
Water Holding Capacity			
Water Holding Capacity at ⅓ bar (g H_2O per 100 g DW)		15.1	45.7
Test moisture (g H_2O per 100 g DW)		11.3	34.3
Moisture Content During Incubation (%)		75% of ⅓ Bar	75% of ⅓ Bar
Soil Microbial Biomass (µg microbial C / g soil)			
Initial (Day 0)		75	1480
Mid (Day 11)		47	na
Final (Day 365)		46	na

B. Study Design

1. Experimental Conditions

Tests were performed in flow through systems consisting of a Teflon incubation tube containing 10 g soil (dry weight equivalents) and attached in series to four traps. The first trap contained ethylene glycol to collect organic volatiles, the second and third traps contained 1M sodium hydroxide to collect carbon dioxide and the fourth contained 1M sulfuric acid.

The tests were performed at a concentration of 1.2 mg/kg dry weight of soil, equivalent to a field rate of 1.6 and 1 kg/ha in Bethany and North Dakota soils respectively. The test item [^{14}C-1-phenyl]-M-01 dissolved in methanol (50 µL) was applied to the soil surface. Soil samples were adjusted to a moisture content equivalent to 75% of ⅓ bar and incubated at 25 °C several days prior to application. Treated samples were incubated at 25± 1 °C under aerobic conditions in the dark for up to 365 days (Bethany

soil) and 180 days (North Dakota soil).

The incubation temperature of the Bethany soil samples was not maintained at 25 ºC for two periods of 16 hours and 55 hours, where the temperature reached a maximum of 29.4 and 33.2 ºC respectively.

The soil moisture content of the soils was maintained at 11.3% for Bethany soil and 34.3% for North Dakota soils, by the addition of water periodically (*ca*. every 3 weeks) throughout the course of the study.

2. Sampling

Following incubation, duplicate samples were taken for analysis after 0, 1, 3, 7, 14, 30, 61, 91, 120, 180, 268 and 365 days (Bethany soil) and 0, 7, 14, 31, 61, 119 and 180 days (North Dakota soil). The microbial biomass of both soils was measured at the start of the incubation period and after 111 days and 365 days for the Bethany soil.

3. Analytical procedures

Day 0, 1, 3 and 7 samples of Bethany soil were extracted at ambient temperature twice with methanol, followed by methanol / water 1:1 and then acetonitrile. The remaining Bethany soil samples and the North Dakota soil samples were extracted at ambient temperature twice with methanol, followed by water and then acetonitrile. The ambient extracts were pooled. The soil residue was then extracted by soxhlet extraction with methanol followed by further soxhlet extraction with water.

Radioactivity extracted from soil and in the volatile traps was quantified by liquid scintillation counting (LSC). Radioactivity remaining unextracted from soil was quantified by combustion and LSC. The radioactivity contained in the sodium hydroxide traps was confirmed as $^{14}CO_2$ by precipitation as ^{14}C barium carbonate.

Quantification of the levels of M-01 and its metabolites in soil extracts was carried out by high performance liquid chromatography (HPLC). Selected samples were analysed by a second HPLC method to confirm the results obtained. Analysis by LC-MS was used to verify the most abundant radiolabelled component as M-01 (called BAM or 2,6 dichlorobenzamide in the report).

4. Determination of degradation kinetics:

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of M-01 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-01 (BAM), an initial comparison was performed for each soil between the SFO and FOMC fits. For both soils, the FOMC fit resulted in a lower χ^2 err% value and better visual fit than the SFO model, and the DFOP model was therefore also fitted. For the Bethany soil, the resulting DFOP fit was visually and statistically acceptable, providing the lowest χ^2 err% value, and was accepted. For the North Dakota soil, confidence in the k_2 rate constant for the DFOP model was low (p=0.37), and DFOP was therefore not accepted. The FOMC fit was also not accepted, as extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using this model (EFSA, 2009). The SFO model therefore provided the most appropriate description of M-01 degradation in the North Dakota soil. It is noted that the relevant trigger values are exceeded for all of the fits obtained, therefore model selection does not alter the regulatory implications of the endpoints.

II. Results and Discussion

A. Data

The results of aerobic biotransformation of [^{14}C-1-phenyl]-M-01 after incubation in two US soils are summarised in Table 7.1.2.1.2- 2 to Table 7.1.2.1.2- 3.

Table 7.1.2.1.2- 2: Degradation of M-01 in Bethany sandy loam under aerobic conditions (% AR)

Compound	Mean [A]	Incubation time (DAT)											
	SD	0	1	3	7	14	30	61	91	120	180	268	365
M-01	Mean	99.6	98.0	96.9	96.0	95.2	92.7	89.2	90.5	90.1	88.4	86.0	85.6
	SD	±0.9	±0.7	±0.5	±0.7	±0.0	±0.0	±0.1	±1.3	-	±1.3	±0.7	±2.3
Unknown RRT 0.2 [B]	Mean	nd	nd	nd	0.3	0.2	0.2	0.4	0.4	0.6	0.4	0.5	0.4
	SD	-	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.1	±0.0	±0.1	±0.0
Unknown RRT 0.4	Mean	nd	nd	nd	nd	0.0	0.0	nd	nd	nd	nd	nd	nd
	SD	-	-	-	-	±0.1	±0.0	-	-	-	-	-	-
Unknown RRT 0.7	Mean	nd	nd	nd	0.1	0.2	0.1	0.2	0.2	0.3	0.2	0.2	0.3
	SD	-	-	-	±0.0	±0.0	±0.0	±0.0	±0.1	±0.0	±0.0	±0.1	±0.0
Unknown RRT 1.2	Mean	nd	nd	nd	0.0	0.1	0.0	0.0	nd	0.0	0.2	0.0	0.2
	SD	-	-	-	±0.0	±0.0	±0.0	±0.0	-	±0.1	±0.0	±0.1	±0.0
Ambient Extracts	Mean	97.8	94.5	91.8	89.1	88.0	83.7	78.5	80.4	78.8	75.5	75.1	68.8
	SD	±1.0	±0.4	±0.6	±0.7	±0.2	±0.6	±0.5	±0.4	±0.9	±0.2	±2.1	±4.6
Soxhlet Methanol extracts	Mean	1.0	2.9	4.4	5.4	5.9	6.7	8.6	7.3	9.9	6.6	8.1	10.0
	SD	±0.0	±0.2	±0.1	±0.2	±0.0	±0.5	±0.8	±0.5	-	±0.9	±1.3	±1.5
Soxhlet Water extracts	Mean	0.7	0.7	0.8	2.0	1.8	2.7	2.7	3.5	3.0	7.1	3.6	7.7
	SD	±0.0	±0.1	±0.0	±0.1	±0.0	±0.0	±0.2	±0.2	±0.1	±0.3	±0.4	±0.8
Total Extracted	Mean	99.6	98.0	96.9	96.5	95.7	93.2	89.8	91.1	90.9	89.2	86.8	86.4
	SD	±0.9	±0.7	±0.5	±0.7	±0.1	±0.1	±0.0	±1.2	-	±1.3	±0.4	±2.3
Unextracted soil residue	Mean	0.9	1.1	1.2	1.6	1.9	2.6	3.3	4.3	3.9	5.1	4.7	4.8
	SD	±0.0	±0.0	±0.1	±0.0	±0.2	±0.1	±0.4	±0.2	±0.2	±0.3	±0.0	±0.0
$^{14}CO_2$	Mean	na	0.6	0.9	1.4	1.8	2.1	3.3	4.2	4.4	5.3	5.9	6.1
	SD		±0.0	±0.1	±0.3	±0.0	±0.6	±0.2	±0.2	±0.2	±0.3	±0.4	±1.6
Organic volatiles	Mean	na	nd	0.0	0.0	0.0	0.0	0.0	0.0	nd	nd	nd	0.1
	SD			±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	-	-	-	±0.0
Total	Mean	100.4	99.7	99.0	99.2	99.4	97.9	96.4	99.7	99.5	99.6	97.3	97.4
	SD	±0.9	±0.6	±0.5	±0.3	±0.1	±0.5	±0.6	±1.6	-	±0.8	±0.8	±0.7

Values given as percentages of initially applied radioactivity (AR)
n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[A] Mean value of two replicates
[B] RRT 0.2 was tentatively identified as 2,6-dichlorobenzoic acid

Table 7.1.2.1.2- 3: Degradation of M-01 in North Dakota sandy loam under aerobic conditions (% AR)

Compound	Mean [A] / SD	Incubation time (DAT)						
		0	7	14	31	61	119	180
M-01	Mean	103.6	100.9	96.5	92.9	88.5	84.1	81.8
	SD	±0.7	±1.6	±0.4	±1.3	-	±0.3	±0.9
Unknown RRT 0.2 [B]	Mean	nd	0.2	0.2	0.2	0.2	0.2	0.2
	SD	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0
Unknown RRT 0.4	Mean	nd	nd	nd	nd	nd	nd	nd
	SD	-	-	-	-	-	-	-
Unknown RRT 0.7	Mean	nd	0.1	0.2	0.1	0.1	0.1	0.2
	SD	-	±0.0	±0.1	±0.0	±0.1	±0.0	±0.0
Unknown RRT 1.2	Mean	nd	0.0	nd	nd	0.0	nd	nd
	SD	-	±0.0	-	-	±0.1	-	-
Ambient Extracts	Mean	98.4	89.9	85.4	79.3	72.8	67.5	62.2
	SD	±0.6	±1.0	±0.4	±0.5	±0.3	±0.8	±0.1
Soxhlet Methanol extracts	Mean	4.1	9.8	8.0	11.4	11.7	13.3	15.1
	SD	±0.2	±0.7	±0.4	±0.7	-	±0.2	±0.6
Soxhlet Water extracts	Mean	1.2	1.6	3.4	2.5	4.3	3.6	4.9
	SD	±0.2	±0.1	±0.3	±0.1	±0.3	±0.4	±0.1
Total Extracted	Mean	103.6	101.2	96.9	93.2	88.7	84.4	82.2
	SD	±0.7	±1.6	±0.5	±1.3	-	±0.2	±0.9
Unextracted soil residue	Mean	0.6	1.9	2.2	2.3	2.9	3.6	4.6
	SD	±0.1	±0.0	±0.1	±0.1	±0.2	±0.4	±0.1
$^{14}CO_2$	Mean	na	1.6	2.0	2.8	3.4	5.7	8.2
	SD	-	±0.0	±0.2	±0.2	±0.9	±1.3	±0.7
Organic volatiles	Mean	na	nd	nd	nd	nd	nd	nd
	SD	-	-	-	-	-	-	-
Total	Mean	104.2	104.7	101.0	98.3	94.4	93.7	95.0
	SD	±0.7	±1.6	±0.4	±1.4	-	±0.7	±1.5

Values given as percentages of initially applied radioactivity (AR)
n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[A] Mean value of two replicates
[B] RRT 0.2 was tentatively identified as 2,6-dichlorobenzoic acid

B. Material balance

Mean mass balances were 98.8% AR for Bethany soil (range from 96.4 to 100.4% AR) and 98.7% AR for North Dakota soil (range from 93.7 to 104.7 % AR).

The results confirm there were no significant losses of radioactivity during sample processing.

C. Non-extractable and extractable residues

Values of total extractable radioactivity decreased from 103.6% of applied at zero time to 82.2% after 180 days in North Dakota soil and from 99.6% of applied at time zero to 86.4% after 365 days in Bethany soil. The amount of radioactivity extracted by ambient extraction declined from *ca*. 98% in both soils at Day 0 to 62% by DAT 180 in North Dakota soil and 69% by DAT 365 in Bethany soil. With time increasing amounts of radioactivity could only be extracted by repeated soxhlet extraction in both soils.

The amounts of non-extractable soil bound radioactivity were low, reaching a maximum of 5% of applied radioactivity in both soils.

D. Volatile radioactivity

Radiolabelled carbon dioxide evolved accounted for a maximum of 6% of the applied radioactivity in the Bethany soil by the end of the study (DAT 365) and 8% in the North Dakota soil (DAT 180). Formation of other volatile radioactivity was insignificant (≤ 0.1% AR) at any sampling interval.

E. Transformation of test substance

M-01 was the principal radiolabelled component detected in soil extracts. Levels of M-01 accounted for 100% of extracted radioactivity at DAT 0 and declined to 86 and 82% of applied radioactivity at termination of the study in Bethany soil (DAT 365) and North Dakota soil (DAT 180) respectively. The levels of M-01 extracted by ambient extraction decreased steadily throughout the study with significant quantities only extractable by soxhlet extraction at longer incubation intervals.

The microbial activity of the Bethany soil at the start of the study was low with microbial biomass levels of <1% of the organic carbon (calculated from the reported microbial biomass and organic carbon levels for this summary). This is below the lower recommended limit of microbial activity for agricultural soils according to OECD 307 guidelines. The microbial biomass of Bethany soil was also determined after 111 and 365 days incubation and had decreased by 37% from study initiation. The microbial activity of the North Dakota soil was higher with microbial biomass levels of 3% of the organic carbon at the start of the study (calculated from reported data for this summary).

The incubation conditions were not optimal for degradation of M-01 (2,6-dichlorobenzamide). Microbial viability of soil under laboratory conditions is known to decline with long ageing periods. The soil moisture content at which Bethany soil was incubated was almost certainly too low to maintain optimal microbial viability for a sandy loam soil. This has been confirmed by microbial biomass measurements throughout the incubation period. North Dakota soil was incubated under wet conditions for a sandy loam soil although this soil would have greater water holding capacity than a typical sandy loam soil as a consequence of its high organic matter content. It is often not possible to determine realistic degradation rates for substances with a degree of persistence in the laboratory, as the incubation periods required to determine accurate half-lives are too long to maintain microbially viable soil and consequently any half-lives determined are extrapolated well beyond the duration of the incubation period and cannot be regarded as reliable. For this reason incubation periods according to EU recommendations are normally limited to 120 days. In order to measure accurate degradation half-lives for substances which are slowly degraded, it is necessary to determine these under field conditions.

Microbial populations in soil will slowly degraded 2,6-dichlorobenzamide with the initial step likely to be the formation of 2,6-dichlorobenzoic acid which will the undergo decarboxylation with the evolution of carbon dioxide. A number of very minor unidentified components (maximum 0.6%) were detected in aqueous soxhlet extracts, one of these components co-chromatographed with 2,6-dichlorobenzoic acid, but the due to the very short retention time and poor chromatographic resolution this identification was tentative. It was unclear whether this compound was a metabolic product or an impurity in the starting material. M-01 is known to be stable in sterile soil [KCA 7.1.1.1/01, [redacted] 2003, M-241049-01-1] confirming that the degradation of M-01 is microbially mediated. The subsequent degradation of the metabolism products do not accumulate implying that their degradation was significantly more rapid

than that of M-01, even under incubation conditions that were not optimum for microbial activity. The radiolabelled tracer, ^{14}C, was in carbon 1 of the phenyl ring and the presence of [^{14}C]-carbon dioxide (at up to 8%) confirmed M-01 was mineralised with complete metabolism of the phenyl ring. However the presence of two chlorine atoms in the phenyl ring will mean that this process would be expected to be relatively slow.

F. Degradation kinetics

The rate of degradation of M-01 determined in the study using first order kinetics was reported for each soil (1831 and 557 days for Bethany and North Dakota soils, respectively). It was been concluded in the report that degradation was biphasic and degradation half-lives were also quoted for the first 61 days of the incubation period (431 and 227 for Bethany and North Dakota soils, respectively).

Table 7.1.2.1.2- 4: Kinetic fits for aerobic degradation of M-01 in two US soils from EFSA conclusion

Soil	Soil type	pH	t °C / % MWHC	DT_{50} (days)	DT_{90} (days)	DT_{50} (days) 20 °C & pF2	St. (r^2)	Method of calculation
Bethany	Sandy loam	4.8	25 / 75% of ⅓ bar	1831	6083	1848	0.775	SFO
North Dakota	Sandy loam	7.7	25 / 75% of ⅓ bar	557	1850	808	0.874	SFO

The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.1.2- 5. Best fit kinetics are highlighted in bold.

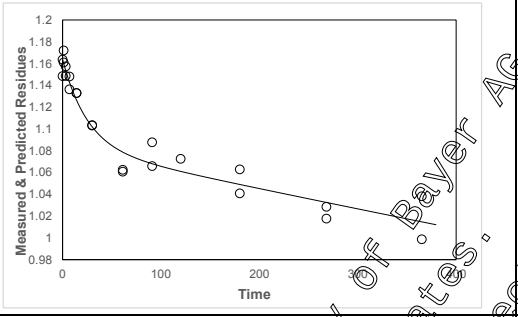
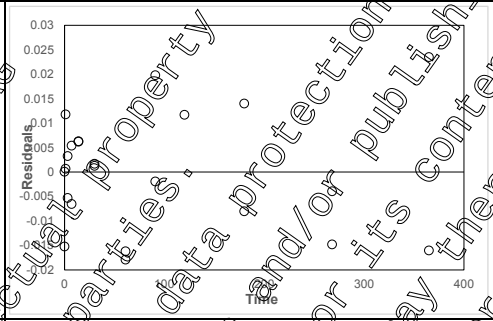
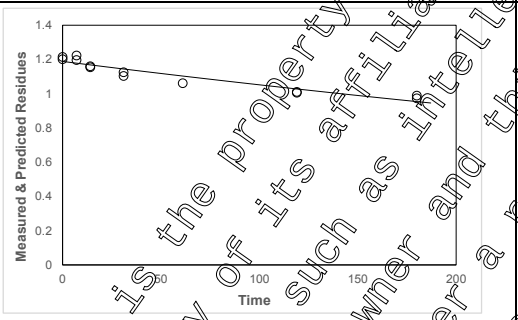
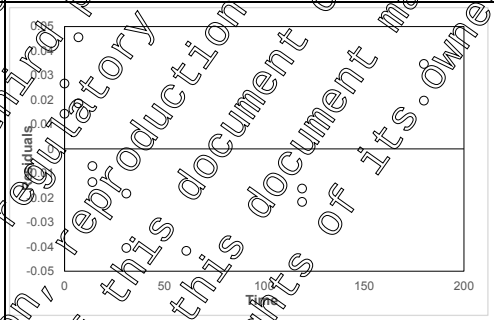
Table 7.1.2.1.2- 5: Degradation rate of M-01 under aerobic conditions at 25 ºC (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Bethany, Griffen (2002)	SFO	1.136	k 0.000373	1.66	1.42E-08	0.0002874	0	1858	6173
	FOMC	1.164	α 0.03657 β 9.731	0.684	n.r. n.r.	0.02637 0.1196	0.047 19.34	>10000	>10000
	DFOP	**1.164**	**k1 0.03968 k2 0.0001796 g 0.06902**	**0.636**	**0.00481 0.000215 n.r.**	**0.01266 9.68E-05 0.05032**	**0.067 0 0.088**	**3461**	**>10000**
North Dakota, Griffen (2002)	**SFO**	**1.189**	**k 0.001219**	**1.97**	**4.09E-07**	**0.0009774**	**0.001**	**568.8**	**1889**
	FOMC	1.22	α 0.1042 β 23.91	0.818	n.r. n.r.	0.06679 3.417	0.142 44.41	>10000	>10000
	DFOP	1.219	k1 0.01957 k2 0.0001919 g 0.1711	0.768	0.0559 0.3696 n.r.	-0.002188 -0.0009037 -0.004748	0.041 0.001 0.347	2634	>10000

Best fit model highlighted in bold

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.1- 106: Degradation of M-01 under aerobic conditions at 25 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Bethany DFOP [redacted] (2002)		
North Dakota SFO [redacted] (2002)		

III. Conclusion

M-01 is a major metabolite of fluopicolide formed in soil from degradation of the parent compound. M-01 was slowly degraded and mineralized in two tested soils; Bethany and North Dakota, under aerobic conditions at 25°C in the dark. M-01 became increasing difficult to extract from soil with time, with significant levels only being extracted under soxhlet conditions by the end of the incubation periods. Only minor components (<0.6%) other than M-01 were extracted from soil throughout the study.

Formation of carbon dioxide was significant (up to 8.2% AR) by the end of the study indicating the potential for complete mineralization of M-01.

Best fit DT_{50} values ranged from 568.8 to 3461 days in the tested soils.

Assessment and conclusion by applicant:

The study was conducted in accordance with USEPA (= EPA) N, 162-1 (1982) and has been used to provide information on the metabolism of M-01 in soil.

Data Point:	KCA 7.1.2.1.2/02
Report Author:	[redacted]
Report Year:	2003
Report Title:	The kinetics of degradation of [U-14C-phenol]-AE0608000 in four soils at 20°C under laboratory aerobic conditions
Report No:	B004233
Document No:	M-241188-01-1
Guideline(s) followed in study:	EU (=EEC): SEPAC - Europe 1.1; USEPA (=EPA): 162-1
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-03 (AE 0608000) was studied in four soils under aerobic conditions in the laboratory in the dark at 20 ± 1 °C and 40% of the maximum water holding capacity for up to 120 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
Münster	loamy sand	4.9	1.8
Pikeville	sandy loam	5.4	1.1
Abington	sandy loam	7.2	3.2
Sarotti	silt loam	7.1	2.0

[Phenyl-U-^{14}C]-labelled M-03 was applied to soil samples at an application rate of ca. 0.41 mg/kg dry weight. The radiochemical purity and specific activity were 91 to 94 % and 5.30 MBq/mg, respectively.

Samples of Münster and Pikeville soils were removed for extraction and analysis immediately after treatment (day 0) and 1, 2, 3, 6, 10, 14, 43, 77 and 120 days of incubation and samples of Abington and Sarotti soils after 0, 5 and 24 hours of incubation. Soil samples were extracted with acetonitrile/water 4/1 (v/v) at ambient temperature, followed by a soxhlet extraction with acetonitrile at later timepoints in Münster (77 and 120 DAT) and Pikeville soils (43, 77, and 120 DAT). Soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC) and the results were confirmed by mass spectral analysis.

Recovery of radioactivity was quantitative throughout the study. Overall mean mass balances were 102.0% AR for Münster soil, 101.7% AR for Pikeville soil, 99.0% AR for Abington soil and 101.5% AR for Sarotti soil.

Total extractable residues remained high in all soils, declining slightly in the very acidic soils from 104.0% AR at DAT 0 to 96.0% AR by DAT 120 in Münster soil and from 104.7 to 97.7% AR in Pikeville soil, and ranging from 96.1% to 101.4% AR in Abington soil and from100.2% to 102.9% AR in Sarotti soil over the 24 hour incubation period in neutral soils. Non-extractable residues (NER) ranged from 1.5 to 8.6% AR in Münster soil, from 1.3 to 14.8% AR in Pikeville soil, from 1.5 to 2.9% AR in Abington soil and from 1.6 to 3.5% AR in Sarotti soil. Mineralization to carbon dioxide was a minor pathway, demonstrated by the low amount of radioactivity recovered in the ethanolamine volatile traps for all soils (maximum 0.4% AR). No significant levels of organic volatiles were observed.

The degradation rate of M-03 showed a strong pH dependence with extremely rapid degradation except in soils which were highly acidic. In very acidic soils the degradation of M-03 was bi-phasic with a rapid initial degradation phase followed by a slower degradation phase. For Munster and Pikeville soils, M-

03 decreased from 99.0% and 99.2% AR at DAT 0 to 16.8% and 3.9% AR at DAT 120, respectively. For Abington and Sarotti soils, M-03 decreased from 88.9% and 91.6% AR at 0 hour to 1.2% and 2.5% AR after 24 hours, respectively. Degradation of M-03 was accompanied by the formation of the degradation product M-01 (AE C653711). M-01 levels in the soil extracts increased concurrently with the decrease of M-03. In Munster and Pikeville soils, M-01 increased to 79.2% and 93.9% by DAT 120, respectively. For Abington and Sarotti soils, M-01 increased to 95.3% and 94.3% AR by 24 hours, respectively.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} values of 4.4, 2.1, 0.1 and 0.1 days in Münster, Pikeville, Abington and Sarotti soils, respectively. Corresponding DT_{90} values were 644.9, 9.1, 0.3 and 0.3 days.

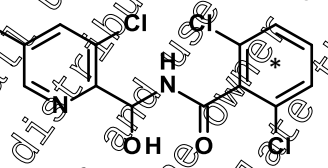
Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)
Münster (loamy sand)	DFOP	4.4	644.9	12.2
Pikeville (sandy loam)	DFOP	2.1	9.1	8.48
Abington (sandy loam)	SFO	0.1	0.3	1.43
Sarotti (silt loam)	SFO	0.1	0.3	3.11

I. Materials and Methods

A. Materials

1. Test Items

[Phenyl-U-^{14}C]-M-03 (referred to as AE 0608000 in the report)



* Denotes position of [^{14}C]-radiolabel

Specific Activity: 143.4 µCi/mg, 5.3 MBq/mg

Radiochemical Purity: 94.49% (HPLC) prior to dosing Munster and Pikeville soils

90.97% (HPLC) prior to dosing Abington and Sarotti soils

2. Test Soils

The study was performed using four soils from agricultural areas as characterized in Table 7.1.2.1.2- 6. The soils were collected from the same sites as laboratory aerobic soil studies with fluopicolide, from Abington (Cambridge, England), Munster and Sarotti (Germany) and Pikeville (North Carolina, USA).

Table 7.1.2.1.2- 6: Physico-chemical properties of test soils

Parameter	Soil			
Soil Designation	Münster	Pikeville	Abington	Sarotti
Geographic Location City Country	Münster, Northrine-Westfalia Germany	Pikeville, North Carolina, USA	Abington, Cambridgeshire UK	Hattersheim, Hesse Germany
Batch Number	EFS-132	EFS-125	EFS-128	EFS-133
Textural Classification (USDA)	Loamy sand	Sandy loam	Sandy loam	Silt loam
Sand [50 - 2000 µm] %	82	70	73	22
Silt [2 – 50 µm] %	14	22	20	62
Clay [< 2 µm] %	4	8	7	16
pH in $CaCl_2$ (1:1)	4.9	5.4	7.2	7.1
in H_2O (1:1)	5.7	6.2	7.7	7.5
Organic Matter (%) *	3.1	1.9	5.4	3.4
Organic Carbon (%)	1.8	1.1	3.2	2.0
Cation Exchange Capacity (meq/100 g)	6.1	3.9	19.4	14.5
Water Holding Capacity Maximum (g H_2O per 100 g DW)	40.6	32.3	65.4	62.4
at 1/10 bar (%)	18.2	17.8	23.2	34.0
at 1/3 bar (%)	8.8	12.6	17.9	20.9
at 15 bar (%)	4.7	3.3	14.3	11.5
Moisture Content During Incubation (%)	40% MWHC	40% MWHC	40% MWHC	40% MWHC
Bulk Density (disturbed, g/cm³)	1.34	1.45	1.20	1.16
Soil Microbial Biomass (µg microbial C/g soil)	BIO-	BIO-	BIO-	BIO-
Initial (Day 0)	73.2	100.9	228.0	419.7
Final	86.5 (DAT 136)	59.0 (DAT 129)	-	-

BIO- samples were untreated

B. Study Design

1. Experimental Conditions

Tests were performed in flow through systems consisting of an Erlenmeyer flask containing 50 g soil attached to an ethylene glycol trap to collect organic volatiles followed by an ethanolamine trap to collect carbon dioxide. Soil moisture was maintained during incubation by the periodic addition of water.

The tests were performed at a concentration of approximately 0.41 mg/kg dry weight of soil (21.58 µg/flask for Abington and Sarotti soils and 23.60 µg/flask for Münster and Pikeville soils). The test concentration was based on a field rate of 400 g a.s./ha. The test item [phenyl-U-^{14}C]-M-03, dissolved

in acetonitrile (280 µL), were applied drop wise onto the soil surface. Soil samples were adjusted to a moisture content of 40% maximum water holding capacity, at least 2 days prior to application. The samples were incubated at 20 ± 1 °C under aerobic conditions in the dark.

2. Sampling

Single samples were removed for analysis at 0, 5 and 24 hours after application for the Abington and Sarotti soils. Single samples were removed for analysis after 0, 1, 2, 3, 6, 10, 14, 43, 77 and 120 days for the Pikeville and Münster soils. Microbial soil biomass samples were analysed for all soils at the start of the experiment (Day 0) and for Pikeville and Münster soils after 129 and 136 days of incubation.

3. Analytical Procedures

For each sample analysis, the entire soil sample was extracted two to four times at ambient temperature with acetonitrile/water (4:1, v/v). After each extraction step, extract and soil were separated by centrifugation. Additionally, after ambient extraction, the Münster soil at 77 DAT and 120 DAT and the Pikeville soil at 43, 77, and 120 DAT were subjected to Soxhlet extraction with acetonitrile for 4 hours. Radioactivity extracted from soil and in the volatile traps was quantified by liquid scintillation counting (LSC).

Extracts were analyzed against authentic reference standards by reverse phase high performance liquid chromatography and the results were confirmed by mass spectral analysis.

The extracted soil at each timepoint was combusted to quantify non-extractable residue (NER).

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of M-03 and M-01 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-03, an initial comparison was performed for each soil between the SFO and FOMC fits. For the Abington and Sarotti soils, the SFO model fitted the residues appropriately, and there were insufficient time points available to fit bi-phasic models; the SFO model was therefore accepted for these soils. For the Münster and Pikeville soils, degradation appeared bi-phasic and the DFOP model was therefore also fitted. For the Münster soil, the KinGUI run for the DFOP model did not complete, while for the Pikeville soil the DFOP model provided the best fit for M-03, but there was no confidence in the resulting degradation rate constant for M-01 (BAM). In both cases, therefore, an additional fit was performed using the DFOP model for M-03, with the DT_{50} value for M-01 fixed to 1000 d. Confidence in k2 was low for both soils, however the fits were accepted as suitable and realistic; for the Münster soil, the DT_{90} will clearly exceed the relevant triggers and statistical confidence is therefore arbitrary, while for the Pikeville soil, both the DT_{50} and DT_{90} occur within the study period and the data around these endpoints are well interpolated.

Further metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soils incubated at 20 °C following application of [phenyl-U-^{14}C]-M-03 are summarized in Table 7.1.2.1.2- 7 to Table 7.1.2.1.2- 10.

Table 7.1.2.1.2- 7: Degradation of [phenyl-U-^{14}C]-M-03 in Münster soil under aerobic conditions at 20 °C [% AR]

Compound	Incubation time (DAT)									
	0	1	2	3	6	10	14	43	77	120
M-03 (AE 0608000)	99.0	87.3	88.2	64.4	26.9	25.8	24.7	17.7	16.2	16.8
M-01 (AE C653711)	5.0	8.0	11.1	32.9	72.0	70.3	73.0	75.6	81.8	79.2
Ambient Extracts	104.0	95.3	99.4	97.3	98.9	96.1	97.6	93.3	98.7	96.0
Soxhlet Extract	na	na	na	na	na	na	na	na	9.3	10.0
Total Extractable Residues	104.0	95.3	99.4	97.3	98.9	96.1	97.6	93.3	98.0	96.0
Carbon Dioxide	na	0.0	0.0	0.0	0.0	0.1	0.1	0.2	0.2	0.4
Volatile Organic Compounds	na	nd	nd	nd	nd	nd	nd	nd	nd	nd
Non-Extractable Residues	1.5	2.8	2.9	5.1	5.0	5.8	6.0	8.6	2.2	3.7
Total Recovery	105.5	98.1	102.3	102.4	103.9	101.5	103.8	102.2	100.4	100.0

n.d.: not detected, n.a.: not analysed, DAT: days after treatment
All values expressed as percentage of total applied radiolabel

Table 7.1.2.1.2- 8: Degradation of [phenyl-U-^{14}C]-M-03 in Pikeville soil under aerobic conditions at 20 °C [% AR]

Compound	Incubation time (DAT)									
	0	1	2	3	6	10	14	43	77	120
M-03 (AE 0608000)	99.2	73.8	55.5	31.7	13.1	7.8	7.9	6.5	4.3	3.9
M-01 (AE C653711)	3.6	20.4	40.1	61.2	75.3	80.3	81.1	87.7	92.6	93.9
Ambient Extracts	104.7	94.2	95.7	93.0	88.4	88.2	89.0	76.0	73.5	66.1
Soxhlet Extract	na	na	na	na	na	na	na	18.2	23.4	31.7
Total Extractable Residues	104.7	94.2	95.7	93.0	88.4	88.2	89.0	94.2	96.9	97.7
Carbon Dioxide	na	0.0	0.0	0.0	0.1	0.1	0.1	0.2	0.3	0.4
Volatile Organic Compounds	na	nd	nd	nd	nd	nd	nd	nd	nd	nd
Non-Extractable Residues	1.3	6.6	7.8	9.6	10.6	13.2	14.8	2.2	3.9	4.1
Total Recovery	106.0	100.8	103.6	102.6	99.1	101.4	103.9	96.6	101.0	102.2

n.d.: not detected, n.a.: not analysed, DAT: days after treatment
All values expressed as percentage of total applied radiolabel

Table 7.1.2.1.2- 9: Degradation of [phenyl-U-^{14}C]-M-03 in Abington soil under aerobic conditions at 20 °C [% AR]

Compound	Incubation time (HAT)		
	0	5	24
M-03 (AE 0608000)	88.9	22.0	1.2
M-01 (AE C653711)	11.1	71.6	95.3
Ambient Extracts	99.9	93.6	96.5
Soxhlet Extract	na	na	na
Total Extractable Residues	99.9	93.6	96.5
Carbon Dioxide	na	0.0	0.0
Volatile Organic Compounds	na	0.0	nd
Non-Extractable Residues	1.5	2.6	2.9
Total Recovery	101.4	96.1	99.4

n.d.: not detected, n.a.: not analysed, HAT: Hours after treatment
All values expressed as percentage of total applied radiolabel

Table 7.1.2.1.2- 10: Degradation of [phenyl-U-^{14}C]-M-03 in Sarotti soil under aerobic conditions at 20 °C [% AR]

Compound	Incubation time (HAT)		
	0	5	24
M-03 (AE 0608000)	91.6	22.2	2.5
M-01 (AE C653711)	9.8	76.3	94.5
Ambient Extracts	101.4	98.4	96.8
Soxhlet Extract	na	na	na
Total Extractable Residues	101.4	98.4	96.8
Carbon Dioxide	na	0.0	0.0
Volatile Organic Compounds	na	nd	nd
Non-Extractable Residues	1.6	2.8	3.5
Total Recovery	102.9	101.2	100.2

n.d.: not detected, n.a.: not analysed, HAT: Hours after treatment
All values expressed as percentage of total applied radiolabel

B. Material Balance

Mean mass balances were 102.0% AR for Munster soil (range from 98.1 to 105.5% AR), 101.7% AR for Pikeville soil (range from 96.6 to 106.0% AR), 99.0% AR for Abington soil (range from 96.1 to 101.4% AR) and 101.5% AR for Sarotti soil (range from 100.2 to 102.9% AR).

The results confirm there were no significant losses of radioactivity during sample processing.

C. Extractable and Non-Extractable Residues

In the very acidic soils, total extractable residues decreased slightly from 104.0% AR at DAT 0 to 96.0% AR by DAT 120 in Münster soil and from 104.7 to 97.7% AR in Pikeville soil. The amount of radioactivity extracted by ambient extraction declined from *ca*. 100% in both soils at 0 DAT to 86.0% in Münster soil and 66.1% Pikeville soil after 120 days.

With time increasing amounts of radioactivity were extracted by soxhlet extraction (up to 10.0% and 31.7% AR by 120 DAT in Münster and Pikeville soils respectively). Analysis of soxhlet extracts showed virtually all of the radioactivity was due to M-01.

For Abington and Sarotti soils, residues were fully extractable by ambient extraction over the 24 hour incubation period ranging from 96.1% to 101.4% AR in Abington soil and from100.2% to 102.9% AR in Sarotti soil.

Non-extractable residues (NER) ranged from 1.5 to 8.6% AR in Münster soil, from 1.3 to 14.8% AR in Pikeville soil, from 1.5 to 2.9% AR in Abington soil and from 1.6 to 3.5% AR in Sarotti soil.

D. Volatile Radioactivity

The presence of CO_2 in the ethanolamine traps reached maxima of 0.4% AR in Münster and Pikeville soils by DAT 120. Trace levels of CO_2 were detected in Abington and Sarotti soils over 24 hours incubation (0.02% AR) Volatilization of M-03 was not considered to be a dissipation pathway under aerobic conditions in soil as indicated by the lack of radioactivity detected (≤ 0.01% AR) in the ethylene glycol traps during the study.

E. Transformation of test substance

M-03 was rapidly degraded in soil. In very acidic soils the degradation of M-03 was bi-phasic with a rapid initial degradation phase followed by a slower degradation phase. For Munster and Pikeville soils, M-03 decreased from 99.0% and 99.2% AR at DAT 0 to 16.84% and 3.85% AR at DAT 120, respectively. For Abington and Sarotti soils, M-03 decreased from 88.9% and 91.6% AR at 0 hour to 1.2% and 2.5% AR after 24 hours, respectively.

Degradation of M-03 was accompanied by the formation of the degradation product M-01 (AE C653711). M-02 levels in the soil extracts increased concurrently with the decrease of M-03. For Munster and Pikeville soils, M-01 increased from 5.0% and 5.6% AR at DAT 0 to 79.2% and 93.9% at DAT 120, respectively. For Abington and Sarotti soils, M-01 increased from 11.1% and 9.8% AR at 0 hour to 95.3% and 94.3% AR at 24 hours, respectively.

F. Degradation Kinetics

The degradation rate of M-03 showed a strong pH dependence with extremely rapid degradation except in soils which were highly acidic. Reported SFO DT_{50} values of M-03 under aerobic conditions were 5.04 days, 2.15 days, 2.49 hours and 2.45 hours in Münster, Pikeville, Abington and Sarotti soils, respectively.

The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.1.2- 11. Best fit kinetics are highlighted in bold.

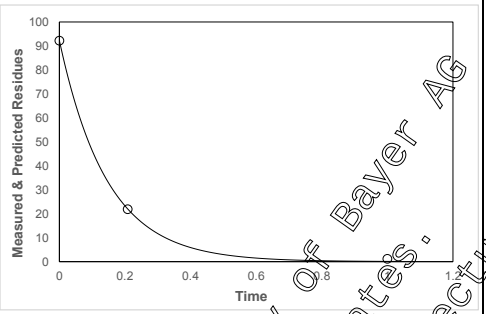
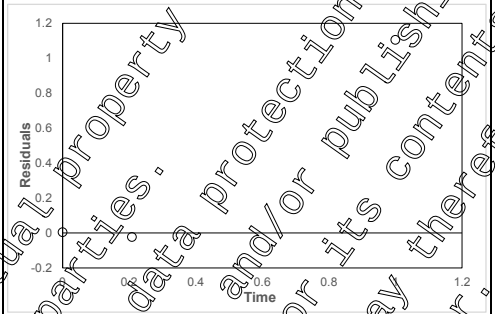
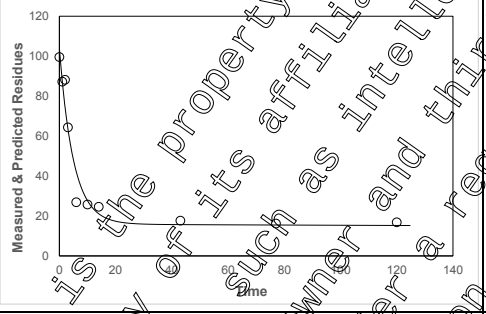
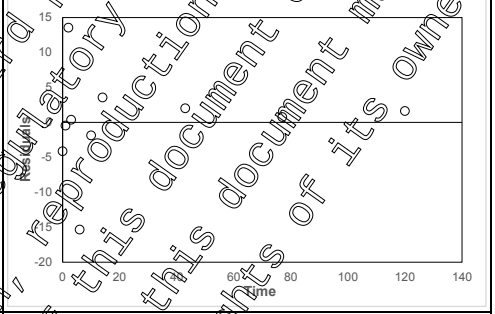
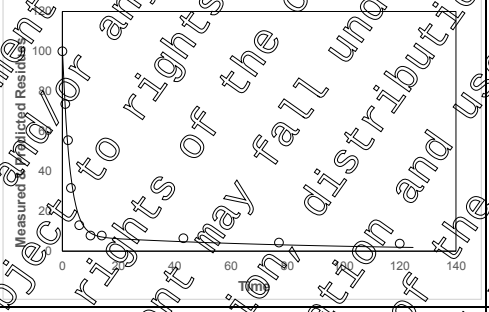
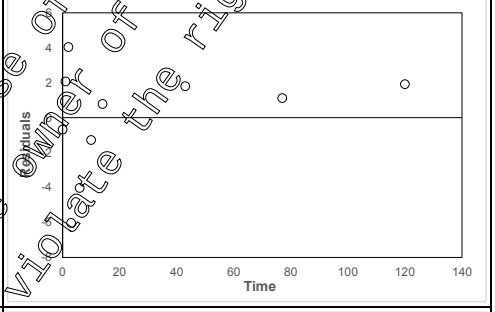
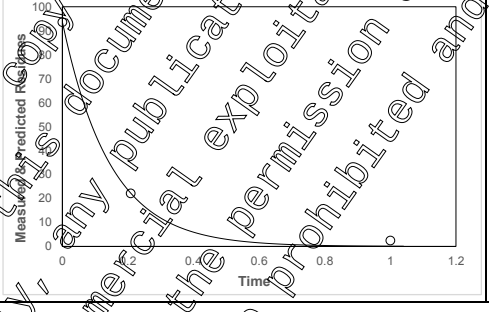
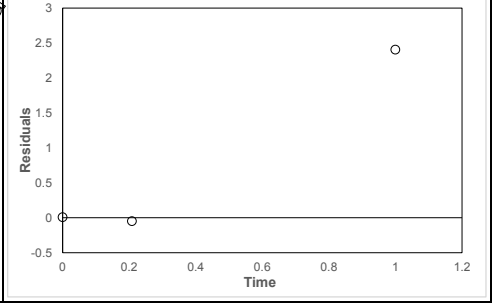
Table 7.1.2.1.2- 11: Degradation rate of M-03 under aerobic conditions at 20 ºC (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003)	**SFO**	**92.28**	**k 6.889**	**1.47**	**0.01147**	**6.402**	**7.376**	**0.1**	**0.3**
Münster, [redacted] (2003)	SFO	105.1	k 0.1641	20.5	2.06E-05	0.1066	0.222	4.2	14
	FOMC	103.4	α 1.184 β 5.945	18.3	n.r. n.r.	-0.09832 -3.203	2.467 15.09	4.7	35.6
	DFOP	103.8	k1 0.2007 k2 0.0003466 g 0.8471	12.9	7.56E-05 0.476 n.r.	0.1224 -0.04078 0.7095	0.279 0.011 0.985	4.4	1225
	DFOP (k2 fixed)	**103.9**	**k1 0.2027 k2 0.0006931 g 0.8436**	**12.2**	**1.45E-06 n.r. n.r.**	**0.1461 n.r. 0.7674**	**0.259 n.r. 0.92**	**4.4**	**644.9**
Pikeville, [redacted] (2003)	SFO	100.6	k 0.3262	11.4	1.52E-10	0.2794	0.373	2.1	7.1
	FOMC	100.9	α 4.361 β 12.08	11.7	n.r. n.r.	-1.989 -7.579	10.71 31.74	2.1	8.4
	DFOP	**100.9**	**k1 0.3746 k2 0.01155 g 0.9232**	**8.48**	**4.98E-09 0.149 n.r.**	**0.3095 -0.009459 0.8622**	**0.44 0.033 0.984**	**2.1**	**9.1**
Sarotti, [redacted] (2003)	**SFO**	**93.63**	**k 6.903**	**3.11**	**0.02458**	**5.857**	**7.95**	**0.1**	**0.3**

Best fit model highlighted in bold

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.1- 107: Degradation of M-03 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003)		
Münster DFOP [redacted] (2003)		
Pikeville DFOP [redacted] (2003)		
Sarotti SFO [redacted] (2003)		

III. Conclusion

M-03 degraded rapidly in the soils under aerobic conditions with best-fit DFOP DT_{50} values of between 2.1 and 4.4 days for the acidic soils (Pikeville and Munster soils). Corresponding DT_{90} values were 9.1 days and 644.9 days. M-03 degraded even more rapidly in the more neutral soils with best-fit SFO DT_{50} values of 0.1 days and DT_{90} values of 0.3 days for both Abington and Sarotti soils.

[^{14}C]-M-03, radiolabelled in the phenyl ring, degraded to form the cleaved metabolite M-01.

Formation of carbon dioxide was not significant (≤ 0.4% AR) and formation of non-extractable residues (NER) reached a maximum of 14.8% AR. No other degradation products were observed.

Assessment and conclusion by applicant:

The study was conducted in accordance with SETAC 1.1 (1995) and USEPA (= EPA) FN, 162-1 (1982). The study is considered valid to assess the aerobic degradation of [phenyl-U-^{14}C]-M-03 in soil.

Data Point:	KCA 7.1.2.1.2/03
Report Author:	[redacted]
Report Year:	2003
Report Title:	(14C)-AE C657188: Rate of degradation in three soils at 20 degrees C
Report No:	C036059
Document No:	M-219824-01-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EC
Deviations from current test guideline:	Yes. OECD 307 states mass balance recoveries for radiolabelled studies should range from 90 to 100%. Mass balances were not quantitative from DAT 30 to 93. The shortfalls were concluded to be due to inefficient trapping of CO2.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-02 (AE C657188) was studied in three soils under aerobic conditions in the laboratory in the dark at 20 ± 2 °C and 40% of 45% of the maximum water holding capacity for up to 93 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
Münster	loamy sand	5.4	1.1
Abington	sandy loam	7.2	2.6
Sarotti	silt loam	7.5	1.3

[2,6-pyridyl-^{14}C]-labelled M-02 was applied to soil samples at an application rate of 0.013 mg/kg dry weight, equivalent to a field application rate of 10 g/ha resulting from a conservative estimate of the maximum occurrence of this metabolite and an application rate of 400 g active substance/ha. The radiochemical purity and specific activity were > 99 % and 6.27 MBq/mg, respectively.

Samples were removed for extraction and analysis immediately after treatment (day 0) and 2, 7, 14, 30, 62 and 93 days of incubation. Soil samples were extracted at ambient temperature once with acetonitrile

followed by two further extractions with acetonitrile : water (1:1 by volume). Following extraction at ambient temperature, soil residues were extracted with acetonitrile : water (8:2 by volume) under soxhlet conditions for six hours. Soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC) and the results were confirmed by mass spectral analysis.

Recovery of radioactivity was quantitative before 30 days, the period from which all kinetic data were calculated. From DAT 30, a shortfall in mass balance was experienced. Overall mean mass balances were 91.7% AR for Münster soil, 89.0% AR for Abington soil and 90.4% AR for Sarotti soil. The losses were thought to be due to the inefficient trapping of CO_2.

Total extractable residues decreased from 94.1 to 99.0% AR at zero time to 30.1% (Münster soil), 8.6% (Abington soil) or 11.1% (Sarotti soil) by DAT 93.The levels of non-extractable radioactivity (NER) were similar in all the soils, reaching a maximum of 48.1 % by DAT 14 incubation in Munster soil, before declining to 31.8% of applied radioactivity by the end of the study. Mineralization to carbon dioxide was significant with a rapid increase in the carbon dioxide production from DAT 30, reaching a maximum of 17.1, 21.6 and 22.9% AR by DAT 93. Virtually no volatile organic products were detected throughout the study (maximum 0.5% AR).

M-02 was very rapidly metabolised in all soils declining to 5% AR or less by DAT 14. Metabolism of M-02 was accompanied by the formation of numerous metabolites including M-05, M-10, M-11, M-12, M-13 and M-14 previously identified in leachate from a lysimeter study conducted with [2,6-pyridyl-^{14}C]-labelled fluopicolide, along with a further eight unidentified metabolites.

Although the radioactive recovery of samples fell below the acceptable range of 90 to 110 % for timepoints after 30 days, only samples with acceptable recovery influenced the DT_{50} and DT_{90} values determined for M-02 as it was very rapidly degraded in soil. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} values of 4.4, 3.5 and 4.4 days in Münster, Abington and Sarotti soils, respectively.

Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)
Münster (loamy sand)	SFO	4.4	14.5	17.2
Abington (sandy loam)	SFO	3.5	11.6	8.51
Sarotti (silt loam)	SFO	4.4	14.5	9.83

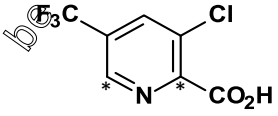
Degradation of the metabolite M-02 in aerobic soil was very rapid, with significant formation of carbon dioxide as a result of complete mineralization of the molecule. Accordingly, M-02 will not persist in the soil environment.

I. Materials and Methods

A. Materials

1. Test Items

[2,6-Pyridyl-^{14}C]-M-02 (referred to as AE C657188 in the report)



* Denotes position of [^{14}C]-radiolabel

Specific Activity:	6.27 MBq/mg
Radiochemical Purity:	99.2% (TLC) 100% (HPLC)
Sample/Batch ID:	SEL/1189

2. Test Soils

The study was performed using three different European soils from agricultural areas as characterized in Table 7.1.2.1.2- 12. The soils were collected from the same sites as laboratory aerobic soil studies with fluopicolide, from Abington (Cambridge, England) and Munster and Sarotti (Germany). The same batches of soils were used in laboratory aerobic soil studies with the other pyridyl ring metabolites M-05, M-10 and M-14. Soils were collected fresh from the field and shipped to the laboratory in Ongar, Essex, UK. On arrival the soil was sieved to 2 mm and stored for *ca* 1 month prior to use.

Table 7.1.2.1.2- 12: Physico-chemical properties of test soils

Parameter		**Soil**		
Soil Designation		Münster	Abington	Sarotti
Geographic Location				
City		Münster, Northrine-Westfalia	Abington, Cambridgeshire	Hattersheim, Hesse
Country		Germany	UK	Germany
Batch Number		03/06	03/07	03/10
Textural Classification (USDA)		Loamy sand	Sandy loam	Silt loam
Sand [50 - 2000 µm]	%	80.53	70.04	23.24
Silt [2 – 50 µm]	%	15.70	15.60	53.95
Clay [< 2 µm]	%	3.77	14.36	22.80
pH				
in Water		6.6	8.1	8.3
in KCl		5.5	7.7	7.7
in $CaCl_2$		5.4	7.2	7.5
Organic Carbon (%)		1.1	2.6	1.3
$Ca_{exchangeable}$ (meq/100 g)		1.5	19.9	34.3
$Mg_{exchangeable}$ (meq/100 g)		0.2	1.6	1.6
$Na_{exchangeable}$ (meq/100 g)		<0.05	0.1	0.1
$K_{exchangeable}$ (meq/100 g)		0.4	1.3	0.9
$Mn_{exchangeable}$ (meq/100 g)		<0.05	<0.05	<0.05
$CaCO_3$eq. (g/kg)		<0.05	73.5	13.4
Phosphorus total (mg/kg)		617.8	1586.3	728.8
Nitrogen total (mg/kg)		1077.9	2380.1	1470.2
Water Holding Capacity				
Maximum (g H_2O per 100 g DW)		46.5	57.1	52.1
Moisture Content During Incubation (%)		40% MWHC	40% MWHC	45% MWHC
Soil Microbial Biomass (µg microbial C /g soil)		BIO^+	BIO^+	BIO+
Initial		147	712	566
Interim		129	741	455
Final		110	664	290

BIO^+ samples were treated with 140 µL of acetonitrile/water (9:1 v/v)

B. Study Design

1. Experimental Conditions

Tests were performed in static systems consisting of an Erlenmeyer flask containing 100 g soil (dry weight equivalents) fitted with a trap attachment (permeable for oxygen) containing soda lime for absorption of carbon dioxide and a polyurethane foam plug for adsorption of volatile organic compounds.

The test item [2,6-pyridyl-^{14}C]-M-02, dissolved in acetonitrile (140 µL per flask), was applied dropwise onto the soil surface at an application rate of 0.013 mg/kg. The application rate was equivalent to 10 g/ha, reflecting a fluopicolide field application rate of 400 g/ha and a conservative estimate of the maximum predicted occurrence of the metabolite. The soil moisture content was adjusted to 40% MWHC (Münster and Abington soils) or 45% MWHC (Sarotti soil) by the addition of water, 36 hours prior to application. The samples were incubated at 20 ± 2 °C under aerobic conditions in the dark. Untreated soil samples were incubated under the same conditions for determination of soil microbial activity.

2. Sampling

Samples were taken for analysis after 0, 2, 7, 14, 30, 62 and 92 days of incubation. Duplicate samples were taken at Day 0 and Day 93, at other timepoints single samples were analysed. Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment.

3. Analytical procedures

Soil samples were extracted at ambient temperature once with acetonitrile followed by two further extractions with acetonitrile: water (1:1 by volume). Following extraction at ambient temperature, soil residues were extracted with acetonitrile: water (8:2 by volume) under soxhlet conditions for six hours.

Extracts were concentrated by rotary evaporation at 35 to 40 °C prior to analysis against authentic reference standards by reverse phase high performance liquid chromatography with fraction collection. In addition, selected extracts were analysed by LC/MS to provide confirmation of structural identity.

The metabolites M-05, M-10 and M-14 (called AE 1344122, AE 1344123 and AE 1388273 in the report respectively) were available as reference standards. The metabolites M-11/M-12 (called P2 in the report) and M-13 (called P3 in the report) were isolated and purified from leachate samples, in which the metabolite P2 was shown to consist of two isomers, P2a and P2b (M-11 and M-12) present in a ratio of ca 6:4. Aliquots of the radioactive isolates of M-11/M12 and M-13 were used as reference materials in this study. The limit of quantitation (LOQ) for the analytical method was 0.05% AR. The identity of M-02, M-05, M-10, M-11/M-12, M-13 and M-14 was confirmed by comparison of HPLC retention times to reference standards. In addition, MRM analysis confirmed the presence of the metabolites M-02, M-05, M-11/M-12 and M-14 by their chromatographic retention time and mass transition channel in selected extracts

The quantity of radioactive volatiles generated was determined by processing the elements that made up each volatile trap. The volatile organics were extracted from the polyurethane bung using ethyl acetate. The carbon dioxide adsorbed on the soda lime was released by digesting the soda lime with hydrochloric acid and re-trapped into a series of traps containing potassium hydroxide solution. The radioactivity contained in the potassium hydroxide traps was confirmed as $^{14}CO_2$ by precipitation as ^{14}C barium carbonate.

Radioactivity in soil extracts was determined by LSC. Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of M-02, M-05, M-10, M-11/M-12 and M-13 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-02 an initial comparison was performed for each soil between the SFO and FOMC fits. For all soils, the SFO model resulted in a comparable visual fit and lower $\chi 2$ err% value than the FOMC model, and SFO was accepted as the most appropriate model to describe M-02 degradation.

Further metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soils incubated at 20 °C following application of [2,6-pyridyl-^{14}C]- M-02 are summarized in Table 7.1.2.1.2- 13 to Table 7.1.2.1.2- 15.

Table 7.1.2.1.2- 13: Degradation of [2,6-pyridyl-^{14}C]-M-02 in Münster soil under aerobic conditions at 20 °C [% AR]

Compound	Incubation time (DAT)						
	0	2	7	14	30	62	93
M-02 (AE C657188)	94.4	78.2	32.8	25	nd	0.2	nd
M-05 (AE 1344122)	nd	2.4	11.0	12.9	16.0	13.1	11.1
M-10 (AE 1344123)	nd	nd	3.4	2.6	4.3	2.7	3.5
M-11/M-12 (Fraction 2-4)	nd	1.1	6.0	3.2	4.0	6.6	5.6
M-13 (Fraction10-12)	nd	nd	1.9	3.2	1.7	1.9	0.4
Fraction 4-6	nd	0.1	1.4	2.1	1.5	2.0	nd
Fraction 6-8	nd	nd	2.2	2.0	0.4	1.6	1.9
Fraction 8-10	nd	0.1	2.4	3.0	0.5	2.2	0.7
Fraction 12-14	nd	nd	1.3	2.5	3.0	1.5	0.2
Fraction 14-16	nd	nd	1.6	1.4	2.8	1.0	0.4
Fraction 18-20	nd	1.2	1.2	2.1	6.3	nd	0.8
Ambient Extracts	94.1 [A]	78.4	38.2	31.7	34.6	26.0	24.8 [A]
Soxhlet Extract	n/a	7.0	9.6	6.8	6.5	8.7	5.3 [A]
Total Extractable Residues	94.1 [A]	85.4	47.8	38.5	41.1	34.7	30.1 [A]
Carbon Dioxide	na	0.7	4.7	3.4	5.7	18.0	17.1 [A]
Volatile Organic Compounds	na	0.1	nd	nd	nd	nd	nd
Non-Extractable Residues	9.6 [A]	12.2	39.2	48.1	47.8	32.5	31.8 [A]
Total Recovery	103.7 [A]	98.3	91.7	89.9	94.5	85.1	79.0 [A]

n.d.: not detected, n.a.: not analysed, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] Distribution values at DAT 0 and DAT 93 for mean of duplicate flasks

Table 7.1.2.1.2- 14: Degradation of [2,6-pyridyl-^{14}C]-M-02 in Abington soil under aerobic conditions at 20 °C [% AR]

Compound	Incubation time (DAT)						
	0	2	7	14	30	62	93
M-02 (AE C657188)	93.8	79.3	34.8	4.7	2.0	2.1	3.2
M-05 (AE 1344122)	nd	1.0	8.3	18.0	15.6	8.1	4.3
M-10 (AE 1344123)	nd	2.2	3.6	5.0	1.8	nd	nd
M-14 (AE 1388273)	nd	nd	nd	0.3	1.2	1.6	0.2
M-11/M-12 (Fraction 2-4)	nd	nd	1.2	1.5	0.9	0.6	0.3
M-13 (Fraction10-12)	nd	nd	2.8	3.6	0.6	0.1	nd
Fraction 8-10	nd	nd	4.3	4.4	1.6	0.3	0.1
Fraction 12-14	nd	nd	1.3	3.2	3.7	0.4	0.1
Fraction 14-16	nd	0.7	1.5	2.2	1.7	0.1	nd
Fraction 18-20	nd	nd	0.6	1.8	2.7	nd	nd
Fraction 40-42	nd	1.4	1.1	2.9	4.1	2.3	0.8
Ambient Extracts	94.5 [A]	83	58.3	47.9	31.5	13.5	8.6 [A]
Soxhlet Extract	na	5.1	9.9	9	5.6	4.1	2.4 [A]
Total Extractable Residues	94.5 [A]	88.1	68.2	56.9	37.1	17.6	11.0 [A]
Carbon Dioxide	na	0.6	0.9	0.6	4.4	18	21.6 [A]
Volatile Organic Compounds	na	nd	nd	nd	0.1	nd	nd
Non-Extractable Residues	7.5 [A]	10.9	25.5	35.0	43.5	39.7	40.9 [A]
Total Recovery	102.1 [A]	99.5	94.7	92.5	85.1	75.3	73.5 [A]

n.d.: not detected, n.a.: not analysed, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] Distribution values at DAT 0 and DAT 93 for mean of duplicate flasks

Table 7.1.2.1.2- 15: Degradation of [2,6-pyridyl-^{14}C]-M-02 in Sarotti soil under aerobic conditions at 20 °C [% AR]

Compound	Incubation time (DAT)						
	0	2	7	14	30	62	93
M-02 (AE C657188)	95.1	81.7	32.4	5.1	2.3	2.3	3.2
M-05 (AE 1344122)	nd	0.1	4.4	11.0	12.7	9.5	5.5
M-10 (AE 1344123)	nd	0.6	1.6	3.3	4.3	0.1	nd
M-14 (AE 1388273)	nd	nd	nd	0.4	0.2	1.0	nd
M-11/M-12 (Fraction 2-4)	nd	nd	0.4	1.0	0.6	0.8	0.6
M-13 (Fraction10-12)	nd	nd	3.2	3.5	1.6	0.1	nd
Fraction 8-10	nd	0.4	0.3	3.0	0.4	0.1	nd
Fraction 12-14	nd	nd	2.2	2.2	3.0	nd	nd
Fraction 14-16	nd	nd	1.0	1.2	2.2	nd	nd
Fraction 18-20	nd	nd	3.8	2.8	3.7	nd	nd
Fraction 40-42	nd	1.7	3.0	6.1	3.7	1.7	0.8
Fraction 42-44	nd	1.3	3.0	4.9	0.6	nd	nd
Ambient Extracts	78.3	79.6	44.5	37.0	27.9	14.1	7.8
Soxhlet Extract	20.7	6.3	13.1	10.0	6.1	4.5	3.3
Total Extractable Residues	99.0 [A]	85.9	57.6	47.0	34.0	18.6	11.1 [A]
Carbon Dioxide	na	0.5	0.7	3.1	6.5	17.4	22.9 [A]
Volatile Organic Compounds	na	nd	nd	nd	0.5	nd	nd
Non-Extractable Residues	7.0 [A]	11.8	37.4	43.0	47.1	41.2	41.5 [A]
Total Recovery	106.0 [A]	98.2	95.6	92.1	88.1	77.1	75.5 [A]

n.d.: not detected, n.a.: not analysed, DAT: days after treatment
All values expressed as percentage of total applied radiolabel
[A] Distribution values at DAT 0 and DAT 93 for mean of duplicate flasks

B. Material Balance

Mean mass balances were 91.7% AR for Münster soil (range from 79.0 to 103.7% AR), 89.0% AR for Abington soil (range from 73.5 to 102.1% AR) and 90.4% AR for Sarotti soil (range from 75.5 to 106.0% AR). All mass balances were quantitative (90-110% of applied radioactivity) before 30 days, the period from which all kinetic data were calculated. From DAT 30, a shortfall in mass balance was experienced. Observed trends indicated that this may be due to the inefficient trapping of CO_2, as the decline was initiated as the formation of CO_2 increased. Experiments were conducted on some of the spare treated flasks to address the low material balances but proved unsuccessful. Other sources of potential loss, such as the possibility of radioactivity adhering to the sides of the vessels used were ruled out.

C. Extractable and Non-Extractable Residues

Values of extractable radioactivity decreased from 94.1 to 99.0% of applied at zero time to 30.1% (Münster soil), 8.6% (Abington soil) or 11.1% (Sarotti soil) by the end of the study

The levels of non-extractable radioactivity (NER) were similar in all the soils, reaching a maximum of 48.1% by DAT 14 incubation in Munster soil, before declining to 31.8% of applied radioactivity by the end of the study. In Abington soil NER reached a maximum of 43.5% AR by DAT 30, declining slightly to 40.9% AR by the end of the study. In Sarotti soil NER reached a maximum of 47.1% AR by DAT 30, declining slightly to 41.5% AR by the end of the study (DAT 93).

D. Volatile Radioactivity

There was a rapid increase in the carbon dioxide production from DAT 30, reaching a maximum of 17.1, 21.6 and 22.9% AR by DAT 93. Virtually no volatile organic products were detected throughout the study (maximum 0.5% AR).

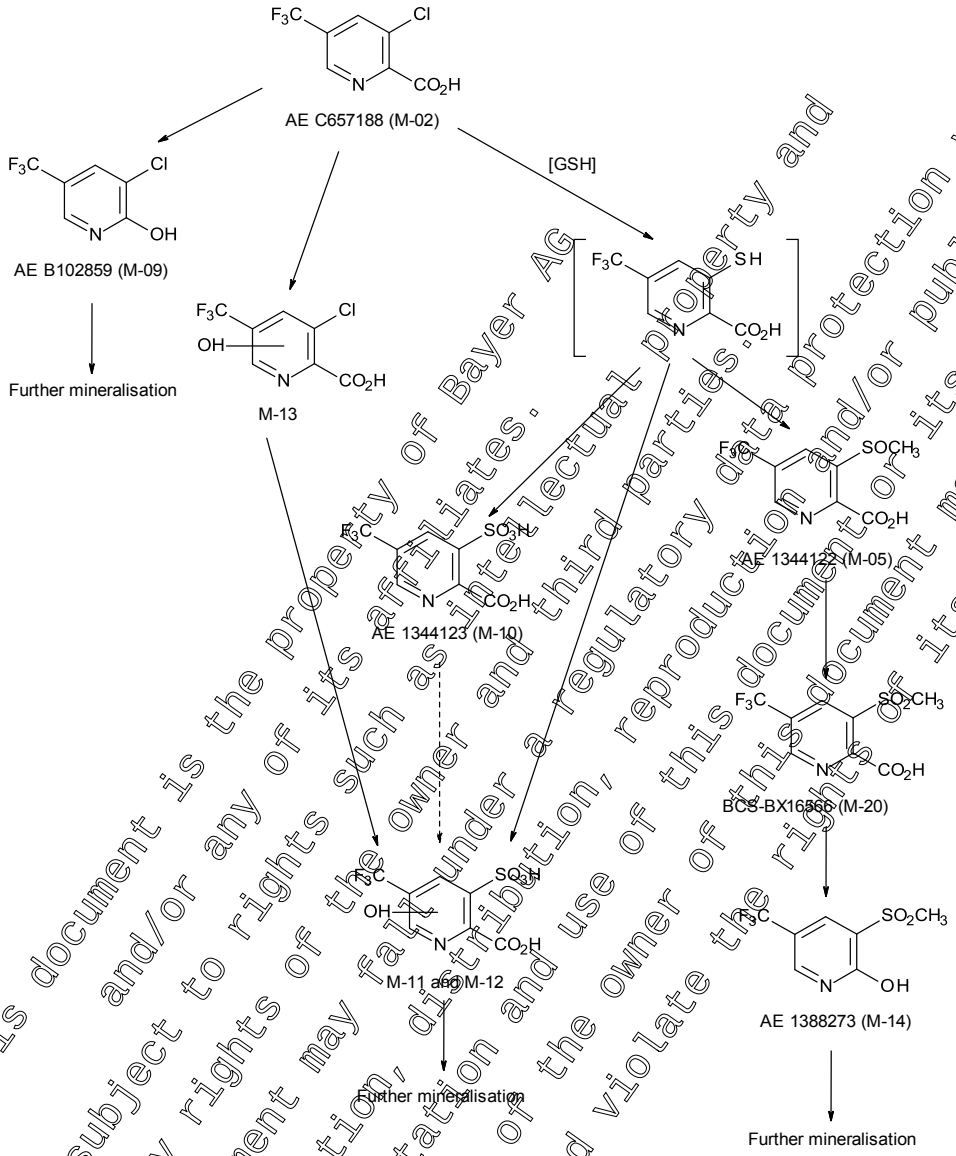
E. Transformation of test substance

M-02 was very rapidly degraded in the soils to a number of metabolites, and despite exhaustive extraction of soil samples rapidly formed significant quantities of NER. The levels of M-02 had declined to 5% AR or less in all soils by DAT 14. The metabolites M-05, M-10, M-11/M-12, M-13 and M-14 had been identified in leachate from a lysimeter study conducted with [2,6-pyridyl-^{14}C]- labelled fluopicolide [see KCA 7.1.4.2/01, M-218465-01-1].

Soil samples were incubated beyond the time period required to reach 90% degradation of M-02 to follow the formation and decline of its metabolites. M-02 was metabolised either by hydroxylation of the pyridyl ring to form M-13 or by a postulated reaction with glutathione to form a transient intermediate that can be oxidised to M-10, oxidised and hydroxylated to M-11/M-12, or methylated and oxidised to M-05. M-05 and M-10 may then be hydroxylated to M-11/M-12 or M-14 respectively. M-05 was observed at a maximum of 16.0%, 18.0% and 12.7% of applied radioactivity and had declined to 11.1%, 4.3% and 5.5% AR% in Munster, Abington and Sarotti soils respectively by the end of the study. M-10 was observed in all soils accounting for 4.3 to 5.0% of applied radioactivity. M-14 was observed at *ca.* 1% in Abington and Sarotti soils but not in Munster soil. The metabolites M-11/M-12 and M-13 were also observed. M-11/M-12 at a maximum of 6.6% AR in Münster soil and M-13 at a maximum of 3.2 to 3.6% AR in all soils. Eight unidentified metabolites were detected at levels ranging from 2.1 to 7.9% of applied radioactivity. All of these unidentified metabolites appeared to be transient in nature reaching maxima after 7 to 30 days incubation and declining or below the limit of detection by the end of the study.

The proposed route of degradation of M-02 in aerobic soil is presented in Figure 7.1.2.1.2- 1.

Figure 7.1.2.1.2- 1: Proposed metabolic pathway for M-02 in aerobic soil



M-09 and M-20 were identified in later aerobic soil study (KCA 7.1.2.1.2/09, M-581364-01-1) with M-02 (AE C657188).

F. Degradation Kinetics

Although the radioactive recovery of samples fell below the acceptable range of 90 to 110 % for timepoints after 30 days, only samples with acceptable recovery influenced the DT_{50} and DT_{90} values determined for M-02 as it was very rapidly degraded in soil. Reported SFO DT_{50} values of M-02 under aerobic conditions were 4.5, 3.2 and 4.5 days in Münster, Abington and Sarotti soils, respectively.

The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.1.2- 16. Best fit kinetics are highlighted in bold.

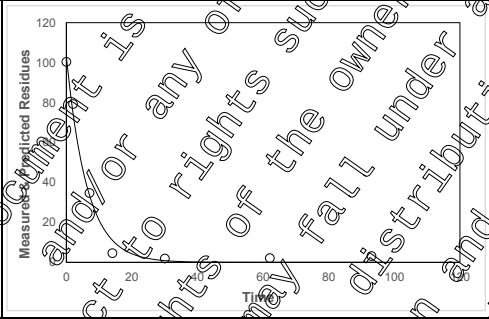
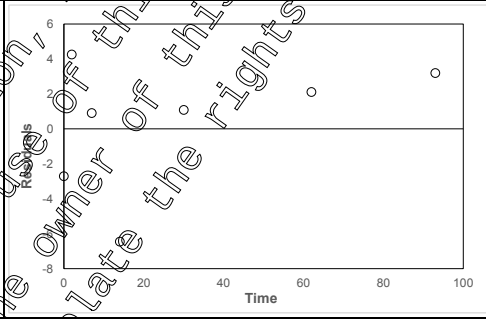
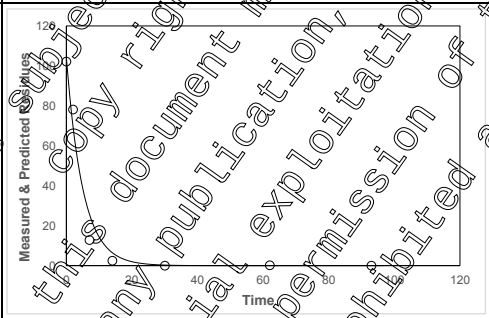
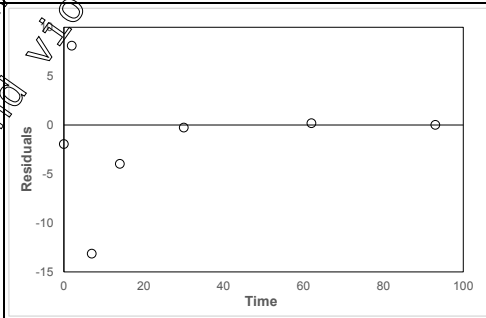
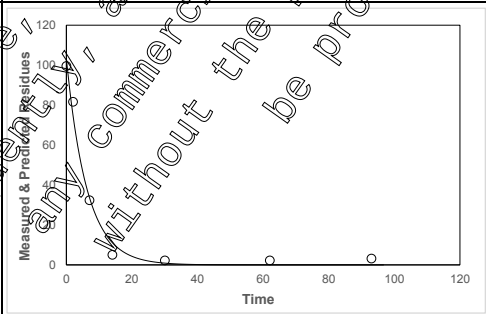
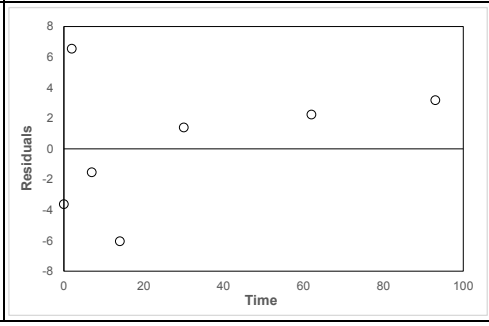
Table 7.1.2.1.2- 16: Degradation rate of M-02 under aerobic conditions at 20 ºC (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003)	**SFO**	**103.2**	**k 0.1591**	**8.51**	**3.07E-10**	**0.129**	**0.189**	**4.4**	**14.5**
	FOMC	102.4	α 2.07E+07 β 1.35E+08	9.46	n.r. n.r.	2.07E+07 1.35E+08	2.07E+07 1.35E+08	4.6	15.1
Münster, [redacted] (2003)	**SFO**	**104.2**	**k 0.1986**	**17.2**	**9.69E-08**	**0.1462**	**0.251**	**3.5**	**11.6**
	FOMC	104.7	α 182.2 β 907.5	18.5	n.r. n.r.	-20558 -1.03E+05	20910 1.05E+05	3.5	11.5
Sarotti, [redacted] (2003)	**SFO**	**103.3**	**k 0.159**	**9.83**	**2.74E-08**	**0.1239**	**0.194**	**4.4**	**14.5**
	FOMC	102.2	α 1.03E+05 β 6.87E+05	11.2	n.r. n.r.	1.03E+05 6.87E+05	1.03E+05 6.87E+05	4.6	15.3

Best fit model highlighted in bold

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.1- 108: Degradation of M-02 under aerobic conditions at 20 ºC (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003)		
Münster SFO [redacted] (2003)		
Sarotti SFO [redacted] (2003)		

III. Conclusion

M-02 is a minor metabolite of fluopicolide which was observed in laboratory soil metabolism studies conducted with the parent and reached a maximum of 7.3% of applied radioactivity. M-02 was very rapidly degraded in soil to a number of pyridyl ring metabolites, with a significant portion completely mineralized to CO_2 and, thus, it would not be expected to persist in the soil environment. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of between 3.5 and 4.4 days.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002) and SETAC 1.1 (1995). The study is considered valid to assess the aerobic degradation of 2.6-^{14}C pyridyl-[^{14}C] M-02 in soil.

Data Point:	KCA 7.1.2.1.2/04
Report Author:	[redacted]
Report Year:	2003
Report Title:	Aerobic soil kinetics of AE 1344122 [pyridine-2,6-14C] in Three Soils
Report No:	M-241410-01-2
Document No:	M-241410-01-2
Guideline(s) followed in study:	EU (=EEC): 95/36, 91/414/EEC; OECD: 307
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-03 (AE 1344122) was studied in three soils under aerobic conditions in the laboratory in the dark at 20 ± 1 °C and 40% or 45% of the maximum water holding capacity for up to 135 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
Münster	loamy sand	5.4	1.1
Abington	sandy loam	7.2	2.6
Sarotti	silt loam	7.5	1.3

[2,6-pyridyl-^{14}C]-labelled M-05 was applied to soil samples at an application rate of 0.013 mg/kg dry weight, equivalent to a field application rate of 10 g/ha resulting from a conservative estimate of the maximum occurrence of this metabolite and an application rate of 400 g active substance/ha. The radiochemical purity and specific activity were 100 % and 4.97 MBq/mg, respectively.

Samples were removed for extraction and analysis immediately after treatment (day 0) and 3, 7, 14, 30, 62, 92 and 135 days of incubation (Münster soil), 0, 2, 6, 13, 29, 61, 91 and 133 days (Abington soil) and 0, 2, 6, 13, 29, 61, 91 and 117 days (Sarotti soil). Soil samples were extracted at ambient temperature

twice with a mixture of methanol:buffer (80:20 v/v, pH 12). Starting on DAT 61/62 soil samples were first extracted with acetonitrile:water (4:1 v/v) at ambient temperature followed by methanol:buffer extraction to ensure that any potential degradates formed from M-05 were extracted. Soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC) and the results were confirmed by mass spectral analysis.

Recovery of radioactivity ranged from 95.1 to 103.5% (Münster soil), 90.0 to 103.8% (Abington) and 89.4 to 102.3% (Sarotti). Total extractable residues decreased from 97 to 99% AR at zero time to 30.4% (Münster soil), 28.3% (Abington soil) or 20.7% (Sarotti soil) by the end of the study. In all soils non-extractable radioactivity increased steadily and reached a maximum of 9.9% in Münster soil, 37.3% in Abington soil and 39.2% in Sarotti soil. Mineralization to carbon dioxide was significant reaching a maximum of 5.4% in Münster soil by DAT 135, 24.4% in Abington soil by DAT 133 and 29.5% in Sarotti soil by DAT 117. Formation of other organic volatiles was negligible (< 0.1% AR) throughout the study.

M-05 decreased from 98.4% AR at time zero to 53.1% AR by DAT 135 in Münster soil, from 98.9% to 23.2% by DAT 133 in Abington soil and from 97.4% to 9.2% by DAT 117 in Sarotti soil. The metabolism of M-05 was more extensive in Abington and Sarotti soils than in Münster soil. The degradation pathway for M-05 in soil was found to proceed to M-14 (AE 1388273) and carbon dioxide, via an intermediate metabolite M-20.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} values of 136.1, 62.2 and 34.9 days in Münster, Abington and Sarotti soils, respectively.

Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)
Münster (loamy sand)	SFO	136.1	452.2	2.85
Abington (sandy loam)	SFO	62.2	206.5	2.36
Sarotti (silt loam)	SFO	34.9	116.1	4.09

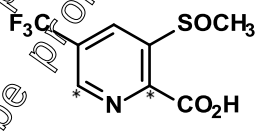
Degradation of the metabolite M-05 in aerobic soil was extensive, with significant formation of carbon dioxide as a result of complete mineralization of the molecule. Accordingly, M-05 is not expected to persist in the soil environment.

II. Materials and Methods

A. Materials

1. Test Items

[2,6-Pyridyl-^{14}C]-M-05 (referred to as AE 1344122 in the report)



* Denotes position of [^{14}C]-radiolabel

Specific Activity:	4.97 MBq/mg (298,367 dpm/µg, 34.03 mCi/mmole)
Radiochemical Purity:	100% (HPLC)
Sample/Batch ID:	SEL/1192 / C-937

2. Test Soils

The study was performed using three European soils collected fresh from the field with minimal storage time (in total 35 to 41 days) prior to sieving to 2 mm and dispersing into flasks. Once received at the test facility the soil was stored under alfalfa cover in a glasshouse. The soils were collected from the same sites as laboratory aerobic soil studies with fluopicolide, from Abington (Cambridge, England) and Münster and Sarotti (Germany). The same batches of soils were used in laboratory aerobic soil studies with the other pyridyl ring metabolites M-02, M-10 and M-14. The physico-chemical characteristics are summarized in Table 7.1.2.1.2- 17.

Table 7.1.2.1.2- 17: Physico-chemical properties of test soils

Parameter		Soil		
Soil Designation		Münster	Abington	Sarotti
Geographic Location				
City		Münster, Northrine-Westfalia	Cambridgeshire, United Kingdom	Hättersheim, Hesse
Country		Germany	UK	Germany
Textural Classification (USDA)		Loamy sand	Sandy loam	Silt loam
Sand [50 - 2000 µm]	(%)	80.5	70.0	23.2
Silt [2 – 50 µm]	(%)	15.7	15.6	54.0
Clay [< 2 µm]	(%)	3.8	14.4	22.8
pH				
in $CaCl_2$		5.4	7.2	7.5
in H_2O		6.6	8.1	8.3
in KCl		5.5	7.8	7.7
Organic Carbon (%)		1.1	2.6	1.3
$Ca_{exchangeable}$ (meq/100 g)		1.5	19.9	34.3
$Mg_{exchangeable}$ (meq/100 g)		0.2	1.6	1.6
$Na_{exchangeable}$ (meq/100 g)		0.05	0.1	0.1
$K_{exchangeable}$ (meq/100 g)		0.4	1.3	0.9
$Mn_{exchangeable}$ (meq/100 g)		<0.05	<0.05	<0.05
$CaCO_3$ equivs (g/kg)		0.05	73.5	13.4
Phosphorus total (mg/kg)		617.8	1586.3	728.8
Nitrogen total (mg/kg)		1077.9	2380.1	1470.2
Water Holding Capacity				
Maximum (g H_2O per 100 g DW)		46.5	57.1	52.1
Test moisture (g H_2O per 100 g DW)		18.6	22.8	23.5
Moisture Content During Incubation		40% MWHC	40% MWHC	45% MWHC
Soil Microbial Biomass (µg microbial C /g soil)		BIO$^-$	BIO$^-$	BIO$^-$
Initial (DAT 0)		158	848	644
Mid (DAT 62/63)		158	750	536
Final (DAT 117/118)		129	693	470

BIO$^-$ samples were treated with water

B. Study Design

1. Experimental Conditions

Tests were performed in static systems consisting of an Erlenmeyer flask containing 100 g soil (dry weight equivalents) fitted with a trap attachment (permeable for oxygen) containing soda lime for absorption of carbon dioxide and a polyurethane foam plug for adsorption of volatile organic compounds.

[2,6-pyridyl-^{14}C]-M-05 was applied at an application rate of 0.013 mg/kg, equivalent to 10 g/ha, reflecting a fluopicolide field application rate of 400 g/ha and a conservative estimate of the maximum predicted occurrence of the metabolite. The test item was dissolved in water with acetonitrile as a co-solvent (<0.1%) and applied dropwise onto the soil surface.

Soils were collected fresh from European fields and shipped to the laboratory in Stilwell, Kansas, USA. On arrival soil was maintained in a biologically active state under alfalfa cover in a greenhouse (11 to 20 days), then dried slightly and sieved to 2 mm prior to dispersing into flasks (100 g dry weight) on 8 April 2003.

Münster soil was treated with the test substance on 8 April 2003 and Abington and Sarotti soils on 9 April 2003. The soil moisture content was adjusted to 40% MWHC (Münster and Abington soils) or 45% MWHC (Sarotti soil) by the addition of water. All soils were incubated in the dark under aerobic conditions at 20 ºC. Soil samples were maintained under static conditions in flasks equipped with a combined solid phase trap for the collection of CO_2 (soda lime) and volatile organic compounds (glass wool saturated with mineral oil). Untreated soil samples were incubated under the same conditions for determination of soil microbial activity.

2. Sampling

Following incubation, duplicate samples were taken for analysis after 0, 3, 7, 14, 30, 62, 92 and 135 days (Münster soil), 0, 2, 6, 13, 29, 61, 91 and 133 days (Abington soil) and 0, 2, 6, 13, 29, 61, 91 and 117 days (Sarotti soil). Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment (Day 0, Day 62/63 and Day 117/118).

3. Analytical procedures

The entire soil sample of each test vessel was extracted twice with a mixture of methanol:buffer (80:20 v/v, pH 12) at ambient temperature. Initially, the buffer consisted of a 50:50 mixture of 1.0 M KCl and 1.0 M K_2CO_3 and resulted in quantitative extraction of M-05 on Day 0. The buffer was changed to a 50:50 mixture of 0.5M KCl and 0.5M K_2CO_3 beginning on Day 7 for Münster soil and Day 6 for Abington and Sarotti soils to avoid precipitation of solids in the methanol:buffer mixture. This mixture was also shown to result in quantitative extraction of M-05.

The use of acetonitrile:water (4:1 v/v) or acetonitrile:water (1% acetic acid) (4:1) as solvent mixture at ambient temperature did not result in quantitative extraction of M-05 immediately after treatment to soil. Starting on Day 61 (Abington and Sarotti) and Day 62 (Münster), soil samples were first extracted with acetonitrile:water (4:1 v/v) at ambient temperature followed by methanol:buffer extraction to ensure that any potential degradates formed from M-05 were extracted.

Each extraction step was followed by filtration of soil extracts. Following concentration, buffer and acetonitrile:water extracts were analysed separately by reversed phase HPLC and ^{14}C-flow-through detection techniques. The limit of quantitation (LOQ) for the analytical method was <0.2% AR. The identity of M-05 and M-14 (called AE 1388273 in the report) was confirmed by comparison of HPLC retention times to reference standards. The identity of M-05 in soil extracts was confirmed by mass spectrometry of isolated HPLC peaks. Another degradate M-20 (called Unknown 1 in the report) was also subject to LC-MS/MS investigations and established to have a molecular weight of 269 g/mole.

This metabolite was identified in a later study conducted with [2,6-pyridyl-^{14}C]-M-02 [KCA 7.1.2.1.2/09, M-581364-01-1].

^{14}C-carbon dioxide adsorbed to soda lime was released by concentrated hydrochloric acid and re-trapped in scintillant solution. For determination of other volatile radioactivity glass wool from the volatile traps was extracted with ethyl acetate. Radioactivity in samples was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics:

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of M-05, M-14 and M-20 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints for M-05 is provided below.

To derive trigger endpoints for M-05, an initial comparison was performed for each soil between the SFO and FOMC fits. For the Abington and Sarotti soils, the SFO model provided a better fit, with a lower χ^2 err% value. For the Münster soil, the FOMC model provided a better visual fit, and the DFOP model was therefore fitted as well. There was no confidence in the resulting DFOP rate constant k_2, and this fit was not accepted. The FOMC fit was also not accepted, as extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using this model (EFSA, 2009). The SFO model therefore provided the most appropriate description of M-05 degradation in the Münster soil.

Further metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The results of aerobic metabolism of [2,6-pyridyl-^{14}C]-M-05 after incubation in three European soils are summarised in Table 7.1.2.1.2- 18 to Table 7.1.2.1.2- 20.

Table 7.1.2.1.2- 18: Degradation of [2,6-pyridyl-^{14}C]-M-05 in Münster soil under aerobic conditions at 20 °C [% AR]

Compound	Mean [A] / SD	Incubation time (DAT)							
		0	3	7	14	30	62	92	135
M-05 (AE 1344122)	Mean	98.4	97.0	92.1	88.1	83.7	65.6	59.6	53.1
	SD	± 0.5	± 1.3	± 1.2	± 1.3	± 2.0	± 0.9	± 2.2	± 1.4
M-14 (AE 1388273)	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Unknown 1 / M-20 (BCS-BX16566) [B]	Mean	n.d.	n.d.	0.2	2.6	9.5	18.6	23.9	25.4
	SD			± 0.3	± 0.2	± 0.7	± 0.2	± 0.6	± 1.0
Unknown 2	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Unidentified Radioactivity [C]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.3
	SD								± 0.4
Total extractable radioactivity	Mean	98.4	97.0	92.3	90.7	93.2	84.2	83.5	80.4
	SD	± 0.5	± 1.3	± 0.8	± 1.5	± 2.8	± 1.1	± 1.6	± 2.0
Carbon dioxide	Mean	n.a.	0.1	0.1	0.4	0.7	1.9	3.2	5.4
	SD	0	± 0.0	± 0.0	± 0.2	± 0.0	± 0.2	± 0.1	± 0.0
Other volatiles	Mean	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Non-extractable radioactivity	Mean	1.6	3.4	5.2	6.9	9.6	9.0	8.4	9.9
	SD	± 0.9	± 0.3	± 0.6	± 2.0	± 0.1	± 0.3	± 1.1	± 0.0
Total radioactivity (%)	Mean	100.0	100.5	97.7	98.1	103.5	95.1	95.1	95.7
	SD	± 1.3	± 1.6	± 0.3	± 0.4	± 2.7	± 1.1	± 2.6	± 2.0

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Mean values of two replicates;
[B] Unknown 1 had a molecular weight of 269 g/mole and was subsequently identified as M-20 (BCS-BX16566) in a later aerobic soil study (KCA 7.1.2.1.2/09, M-581364-01-1) dosed with M-02 (AE C657188),
[C] No individual peak amounted to more than 1.3% of AR

Table 7.1.2.1.2- 19: Degradation of [2,6-pyridyl-^{14}C]-M-05 in Abington soil under aerobic conditions [% AR]

Compound	Mean [A] SD	Incubation time (DAT)							
		0	2	6	13	29	61	91	133
M-05 (AE 1344122)	Mean	98.9	91.9	88.9	84.0	72.9	48.4	33.9	23.2
	SD	± 2.4	± 2.9	± 3.5	± 0.2	± 1.0	± 0.7	± 1.2	± 1.8
M-14 (AE 1388273)	Mean	n.d.	n.d.	n.d.	1.1	8.3	8.1	6.7	4.3
	SD				± 1.6	± 2.0	± 0.8	± 0.7	± 1.6
Unknown 1 / M-20 (BCS-BX16566) [B]	Mean	n.d.	n.d.	n.d.	1.2	n.d.	n.d.	0.2	n.d.
	SD				± 1.7			± 0.3	
Unknown 2	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	0.4	1.3	0.6
	SD						± 0.5	± 0.4	± 0.1
Unidentified Radioactivity [C]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	0.2	0.2	0.1
	SD						± 0.2	± 0.6	± 0.2
Total extractable radioactivity	Mean	98.9	91.9	88.9	86.3	81.1	57.0	42.3	28.3
	SD	± 2.4	± 2.9	± 3.5	± 3.5	± 3.0	± 0.1	± 1.0	± 0.1
Carbon dioxide	Mean	0	0.1	0.3	0.6	3.9	11.0	17.1	24.4
	SD	0	± 0.0	± 0.0	± 0.1	± 0.8	± 0.7	± 0.9	± 0.2
Other volatiles	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Non-extractable radioactivity	Mean	1.1	5.6	10.5	10.4	18.8	25.6	31.7	37.3
	SD	± 0.0	± 1.1	± 1.0	± 0.1	± 1.2	± 0.3	± 0.5	± 0.6
Total radioactivity (%)	Mean	100.0	97.6	99.7	97.4	103.8	93.7	91.0	90.0
	SD	± 2.4	± 1.8	± 4.5	± 3.7	± 2.6	± 0.3	± 1.4	± 1.0

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel

[A)] Mean values of two replicates;

[B)] Unknown 1 had a molecular weight of 269 g/mole and was subsequently identified as M-20 (BCS-BX16566) in a later aerobic soil study (KCA 7.1.2.1.2/09, M-581364-01-1) dosed with M-02 (AE C657188),

[C)] No individual peak amounted to more than 0.2% of AR

Table 7.1.2.1.2- 20: Degradation of [2,6-pyridyl-^{14}C]-M-05 in Sarotti soil under aerobic conditions [% AR]

Compound	Mean [A] SD	Incubation time (DAT)							
		0	2	6	13	29	61	91	117
M-05 (AE 1344122)	Mean	97.4	85.2	83.2	72.4	54.8	28.0	14.8	9.2
	SD	± 3.2	± 4.1	± 9.9	± 1.1	± 1.6	± 0.5	± 0.9	± 0.5
M-14 (AE 1388273)	Mean	n.d.	n.d.	n.d.	4.6	10.4	9.0	5.8	4.4
	SD				± 1.0	± 0.7	± 0.1	± 0.5	± 0.3
Unknown 1 / M-20 (BCS-BX16566) [B]	Mean	n.d.	n.d.	0.8	0.8	0.0	0.0	0.0	0.0
	SD			± 1.1	± 1.1	0.0	0.0	0.0	0.0
Unknown 2	Mean	n.d.	n.d.	n.d.	n.d.	2.4	3.1	5.1	6.8
	SD					± 0.1	± 0.8	± 0.3	± 0.9
Unidentified Radioactivity [C]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.1
	SD								± 0.2
Total extractable radioactivity	Mean	97.4	85.2	84.0	77.8	67.6	39.2	25.7	20.6
	SD	± 3.2	± 4.1	± 11.0	± 0.9	± 2.4	± 1.8	± 0.1	± 0.1
Carbon dioxide	Mean	n.d.	0.1	0.3	1.2	6.7	18.1	26.3	29.5
	SD		± 0.0	± 0.0	± 0.2	± 0.1	± 0.4	± 0.8	± 0.8
Other volatiles	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Non-extractable radioactivity	Mean	2.6	14.0	18.0	16.3	25.3	37.2	39.7	39.2
	SD	± 1.8	± 1.4	± 1.0	± 0.6	± 2.8	± 0.1	± 1.1	± 1.2
Total radioactivity (%)	Mean	100.0	99.4	102.3	95.3	98.9	94.4	91.7	89.4
	SD	± 1.4	± 2.7	± 12.0	± 1.8	± 0.3	± 2.1	± 0.5	± 0.2

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A)] Mean values of two replicates;
[B)] Unknown 1 had a molecular weight of 269 g/mole and was subsequently identified as M-20 (BCS-BX16566) in a later aerobic soil study (KCA 7.1.2.1.2/09, M-581364-01-1) closed with M-02 (AE C657188),
[C)] No individual peak amounted to more than 0.1% of AR

B. Material balance

Mean mass balances were 98.2% AR for Münster soil (range from 95.1 to 103.5% AR), 96.7% AR for Abington soil (range from 90.0 to 103.8% AR) and 96.4% AR for Sarotti soil (range from 89.4 to 102.3% AR).

The results confirm there were no significant losses of radioactivity during sample processing. The material balance for Sarotti soil was slightly below 90% at the last sampling interval. While there were no signs of losses during work-up and processing, this was, possibly due to loss of CO_2.

C. Non-extractable and extractable residues

Values of extractable radioactivity decreased from 97 to 99% of applied at zero time to 80.4% (Münster soil), 28.3% (Abington soil) or 20.7% (Sarotti soil) by the end of the study. In all soils the amounts of non-extractable radioactivity increased steadily and reached a maximum of 39.2% in Sarotti soil, 37.3% in Abington soil and 9.9% in Münster soil by final timepoint.

D. Volatile radioactivity

Mineralisation of [2,6-pyridyl-^{14}C]-M-05 to ^{14}C-carbon dioxide was significant, reaching a maximum of 29.5% in Sarotti soil, 24.4% in Abington soil and 5.4% in Münster soil by the final timepoint (after 117 to 135 days of incubation). Formation of other volatile radioactivity was insignificant (≤ 0.1% AR) at any sampling interval.

E. Transformation of test substance

Metabolite M-05 was degraded relatively rapidly in Abington and Sarotti soils, accompanied by the formation of M-14 at a maximum of 8.3% AR (Day 29, Abington) and 10.4% (Day 29, Sarotti) during the study (Table 7.1.2.1.2- 14 to Table 7.1.2.1.2- 15). Degradation was slower in Münster soil with a metabolite M-20 observed at a maximum of 25.5% (Day 135). This was a transient molecule in the other two soils formed at a maximum of 1% of applied radioactivity. All other components were ≤ 1.3% in any soil through the course of the experiment, except for Unknown 2 which was observed at a maximum of 6.8% in Sarotti soil.

The proposed route of degradation of M-05 in aerobic soil is presented in Figure 7.1.2.1.2-2.

Figure 7.1.2.1.2- 2: Proposed metabolic pathway for M-05 in aerobic soil

M-20 (called Unknown 1, Molecular weight 269 g/mol in the report) was identified in a later aerobic soil study (KCA 7.1.2.1.2/09, M-581364-01-1) treated with M-02 (AE C657188).

F. Degradation kinetics

Reported SFO DT_{50} values of M-05 under aerobic conditions were 130.4, 60.4 and 33.5 days in Münster, Abington and Sarotti soils, respectively. DT_{90} values ranged from 111 to 433 days.

The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.1.2- 21. Best fit kinetics are highlighted in bold.

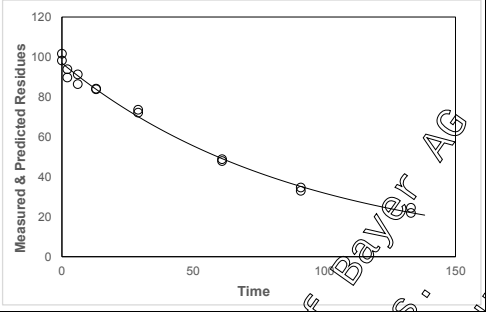
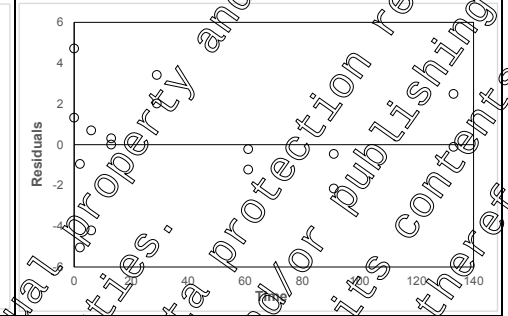
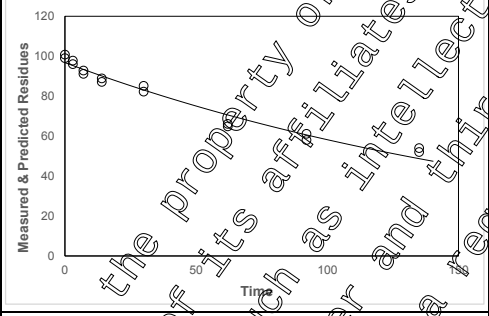
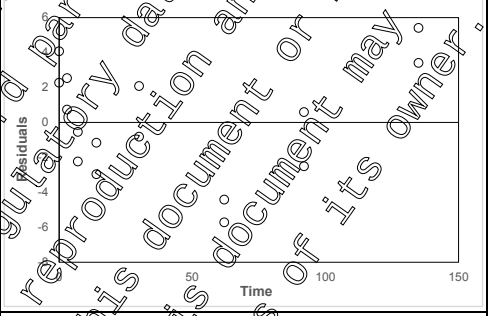
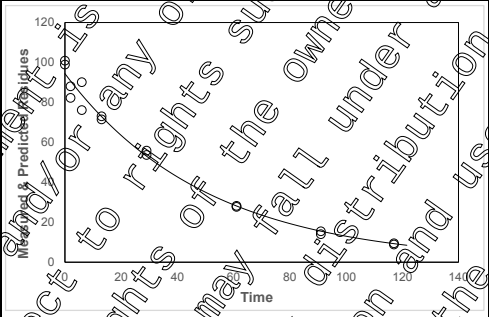
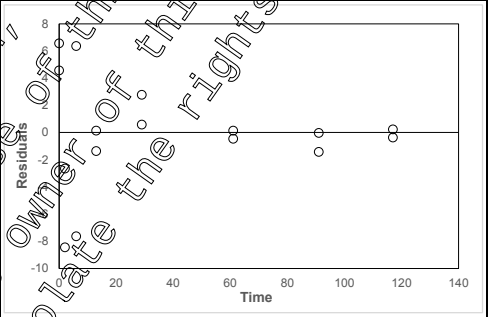
Table 7.1.2.1.2- 21: Degradation rate of M-05 under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003a)	**SFO**	**96.98**	**k 0.01115**	**2.36**	**<2e-16**	**0.01041**	**0.012**	**62.2**	**206.5**
	FOMC	96.96	α 91.57 β 8194	2.51	n.r. n.r.	-2587 -2.33E+05	2770 2.49E+05	62.3	208.6
Münster, [redacted] (2003a)	**SFO**	**96.83**	**k 0.005092**	**2.85**	**<2e-16**	**0.004512**	**0.006**	**136.1**	**452.2**
	FOMC	98.69	α 0.7676 β 101.2	1.9	n.r. n.r.	0.2406 4.019	1.295 198.5	148.5	1932
	DFOP	98.68	k1 0.0126 k2 2.28E-14 g 0.5758	1.91	0.139 0.5 n.r.	-0.009647 -0.0139 -0.7609	0.035 0.014 1.912	160.9	>10000
Sarotti, [redacted] (2003a)	**SFO**	**94.42**	**k 0.01984**	**4.09**	**<2e-16**	**0.01984**	**0.02**	**34.9**	**116.1**
	FOMC	94.57	α 1.79E+08 β 8.95E+09	4.37	n.r. n.r.	1.79E+08 8.95E+09	1.79E+08 8.95E+09	34.6	115

Best fit model highlighted in bold

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.1- 109: Degradation of M-05 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003a)		
Münster SFO [redacted] (2003a)		
Sarotti SFO [redacted] (2003a)		

III. Conclusion

M-05 is a minor metabolite of fluopicolide, which would be predicted to form in the soil environment at very low levels. It was not observed in soil laboratory metabolism studies conducted with fluopicolide. M-05 was degraded in soil, with a significant portion completely mineralized to CO_2 and, thus, it would not be expected to persist in the soil environment. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of between 34.9 and 136.1 days.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002). The study is considered valid to assess the aerobic degradation of [2,6-pyridyl-^{14}C]-M-05 (AE 1344122) in soil.

Data Point:	KCA 7.1.2.1.2/05
Report Author:	[redacted]
Report Year:	2003
Report Title:	Aerobic Soil Kinetics of AE 1344123 [pyridine-2,6-14C] in Three Soils
Report No:	M-241411-01-2
Document No:	M-241411-01-2
Guideline(s) followed in study:	EU (=EEC): 95/36; 91/414/EEC; OECD: 307
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-10 (AE 1344123) was studied in three soils under aerobic conditions in the laboratory in the dark at 20 ± 1 °C and 40% or 45% of the maximum water holding capacity for up to 120 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
Münster	loamy sand	5.4	1.1
Abington	sandy loam	7.2	2.6
Sarotti	silt loam	7.9	1.3

[2,6-pyridyl-^{14}C]-labelled M-10 was applied to soil samples at an application rate of 0.013 mg/kg dry weight, equivalent to a field application rate of 10 g/ha resulting from a conservative estimate of the maximum occurrence of this metabolite and an application rate of 400 g active substance/ha. The radiochemical purity and specific activity were 95.7 % and 3.97 MBq/mg, respectively.

Samples were removed for extraction and analysis immediately after treatment (day 0) and 4, 9, 14, 30, 63, 94 and 120 days. Soil samples were extracted with methanol : water (50 : 50 by volume) for 10 minutes using an accelerated solvent extractor (ASE). Beginning with DAT 14 samples a second extraction with a static extraction time of 20 minutes was added. Soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC).

Recovery of radioactivity ranged from 93.9 to 107.2% (Münster soil), 88.3 to 105.0% (Abington) and 89.3 to 103.8% (Sarotti). Total extractable residues decreased from 97.9 to 104.4% AR at zero time to 89.0% (Münster soil), 2.6% (Abington soil) or 2.2% (Sarotti soil) by the end of the study. Non-extractable radioactivity reached a maximum of 10.2% in Münster soil, 32.6% in Abington soil and 41.4% in Sarotti soil before declining to 6.7, 30.2 and 31.0% AR by DAT 120, respectively. Mineralization to carbon dioxide was significant reaching a maximum of 3.6% in Münster soil, 55.4% in Abington soil and 56.1% in Sarotti soil by DAT 120. Virtually no organic volatiles were detected throughout the study ≤ 0.1% AR).

M-10 decreased from 104.4% AR at time zero to 74.2% AR in Münster soil, from 102.5% to 0.3% in Abington soil and from 97.9% to 0.1% in Sarotti soil by DAT 120. The metabolism of M-10 was more extensive in Abington and Sarotti soils than in Münster soil.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} values of 241.9, 31.6 and 21.3 days in Münster, Abington and Sarotti soils, respectively.

Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)
Münster (loamy sand)	SFO	241.9	803.6	2.43
Abington (sandy loam)	SFO	31.6	104.9	8.03
Sarotti (silt loam)	SFO	21.3	70.9	5.12

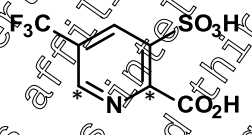
Degradation of the metabolite in aerobic soil was relatively slow, however M-10 is a minor metabolite of fluopicolide formed in the soil environment at very low levels. Should M-10 form in soil it would be steadily degraded and would not be expected to accumulate.

I. Materials and Methods

A. Materials

1. Test Items

[2,6-Pyridyl-^{14}C]-M-10 (referred to as AE 1344123 in the report)



* Denotes position of [^{14}C]-radiolabel

Specific Activity: 3.97 MBq/mg (238,487 dpm/µg, 31.49 mCi/mmole)

Radiochemical Purity: 95.7% (HPLC)

Sample/Batch ID: SEL/1191/C-941

2. Test Soils

The study was performed using three European soils collected fresh from the field with storage times of 52 to 58 days, prior to sieving to 2 mm and dispersing into flasks. Once received at the test facility the soil was stored under alfalfa cover in a glasshouse. The soils were collected from the same sites as laboratory aerobic soil studies with fluopicolide, from Abington (Cambridge, England) and Munster and Sarotti (Germany). The same batches of soils were used in laboratory aerobic soil studies with the other pyridyl ring metabolites M-05, M-10 and M-14. The physico-chemical characteristics are summarized in Table 7.1.2.1.2- 22.

Table 7.1.2.1.2- 22: Physico-chemical properties of test soils

Parameter		Soil		
Soil Designation		Münster	Abington	Sarotti
Geographic Location				
City		Münster, Northrine-Westfalia	Cambridgeshire, United Kingdom	Hättersheim, Hesse
Country		Germany	UK	Germany
Textural Classification (USDA)		Loamy sand	Sandy loam	Silt loam
Sand [50 - 2000 µm]	(%)	80.53	70.04	23.24
Silt [2 – 50 µm]	(%)	15.70	15.60	53.95
Clay [< 2 µm]	(%)	3.77	14.36	22.80
pH				
in $CaCl_2$		5.4	7.2	7.5
in H_2O		6.6	8.1	8.3
in KCl		5.5	7.7	7.7
Organic Carbon (%)		1.1	2.6	1.3
$Ca_{exchangeable}$ (meq/100 g)		1.5	19.9	34.3
$Mg_{exchangeable}$ (meq/100 g)		0.2	1.6	1.6
$Na_{exchangeable}$ (meq/100 g)		0.05	0.1	0.1
$K_{exchangeable}$ (meq/100 g)		0.4	1.3	0.9
$Mn_{exchangeable}$ (meq/100 g)		<0.05	<0.05	<0.05
$CaCO_3$ equivs (g/kg)		0.05	73.5	13.4
Phosphorus total (mg/kg)		617.8	1586.3	728.8
Nitrogen total (mg/kg)		1077.9	2380.1	1470.2
Water Holding Capacity				
Maximum (g H_2O per 100 g DW)		46.5	57.1	52.1
Moisture Content During Incubation		40% MWHC	40% MWHC	45% MWHC
Soil Microbial Biomass (µg microbial C /g soil)		BIO$^-$	BIO$^-$	BIO$^-$
Initial (DAT 0)		147	748	454
Mid (DAT 65)		135	356	716
Final (DAT 123)		92	525	369

BIO$^-$ samples were treated with water

B. Study Design

1. Experimental Conditions

Tests were performed in static systems consisting of an Erlenmeyer flask containing 100 g soil (dry weight equivalents) fitted with a trap attachment (permeable for oxygen) containing soda lime for absorption of carbon dioxide and a polyurethane foam plug for adsorption of volatile organic compounds.

[2,6-Pyridyl-^{14}C]-M-10 was applied at an application rate of 0.013 mg/kg, equivalent to 10 g/ha, reflecting a fluopicolide field application rate of 400 g/ha and a conservative estimate of the maximum predicted occurrence of the metabolite. The test item was dissolved in water and applied dropwise onto the soil surface

Soils were collected fresh from European fields and shipped to the laboratory in Stilwell, Kansas, USA. On arrival soil was maintained in a biologically active state under alfalfa cover in a greenhouse (11 to 20 days), then dried slightly and sieved to 2 mm prior to dispersing into flasks (100 g dry weight) on 25 April 2003.

Soil samples were treated with the test substance on 28 April 2003. The soil moisture content was adjusted to 40% MWHC (Münster and Abington soils) or 45% MWHC (Sarotti soil) by the addition of water. All soils were incubated in the dark under aerobic conditions at 20 °C. Soil samples were maintained under 'static' conditions in flasks equipped with a combined solid phase trap for the collection of CO_2 (soda lime) and volatile organic compounds (glass wool saturated with mineral oil). Untreated soil samples were incubated under the same conditions for determination of soil microbial activity.

2. Sampling

Following incubation, duplicate samples were taken for analysis after 0, 4, 9, 14, 30, 63, 94 and 120 days. Due to poor extractability of the radioactive residues at zero time, the Day 0 interval was repeated in all soils. Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment (Day 0, Day 65 and Day 123).

3. Analytical procedures

The soil samples were extracted with methanol : water (50 : 50 by volume) at 80 ºC and 1500 psi for a static time of 10 minutes using an accelerated solvent extractor (ASE). Beginning with DAT 14 samples a second extraction with a static extraction time of 20 minutes was added, in addition to the first extraction and the extracts were combined.

Following concentration, extracts were analysed by reversed phase HPLC and ^{14}C-flow-through detection techniques. The limit of quantitation (LOQ) for the analytical method was <2% AR. The identity of M-10 was confirmed by comparison of HPLC retention times in two different HPLC systems to a reference standard.

^{14}C-carbon dioxide adsorbed to soda lime was released by concentrated hydrochloric acid and re-trapped in scintillant solution. For determination of other volatile radioactivity glass wool from the volatile traps was extracted with ethyl acetate. Radioactivity in samples was determined by LSC.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion.

4. Determination of degradation kinetics:

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of M-10 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-10, an initial comparison was performed for each soil between the SFO and FOMC fits. For the Abington and Sarotti soils, the SFO model provided a better fit, with a lower χ^2 err% value. For the Münster soil, the FOMC model provided a slightly lower χ^2 err% value, and the DFOP model was therefore fitted as well. The resulting DFOP fit was similar visually to the SFO fit, and the degree of confidence in the DFOP rate constant k_1 could not be calculated, suggesting that the behaviour did not differ significantly from a first order decline, therefore this fit was not accepted. The FOMC fit was not accepted, as extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using this model (FOCUS, 2006) and visual inspection did not suggest a conclusive bi-phasic decline in the observed data. The SFO model therefore provided the most appropriate description of M-10 degradation in the Münster soil.

II. Results and Discussion

A. Data

The results of aerobic metabolism of [2,6-pyridyl-^{14}C]-M-10 after incubation in three European soils are summarised in Table 7.1.2.1.2- 23 to Table 7.1.2.1.2- 25.

Table 7.1.2.1.2- 23: Degradation of [2,6-pyridyl-^{14}C]-M-10 in Münster soil under aerobic conditions at 20 °C [% AR]

Compound	Mean [A] / SD	Incubation time (DAT)							
		0	4	9 [B]	14 [B]	30 [B]	63 [B]	94 [B]	120 [B]
M-05 (AE 1344122)	Mean	104.4	96.9	95.1	99.7	90	83.9	75.4	74.2
	SD	± 2.4	± 6.4	-	-	-	-	-	-
Unknown 1	Mean	nd	nd	1.3	0.5	0.7	nd	nd	1.3
	SD	± 0.0	± 0.0	-	-	-	-	-	-
Unknown 2	Mean	nd	nd	nd	nd	nd	nd	nd	nd
	SD	-	-	-	-	-	-	-	-
Unknown 3	Mean	nd	nd	nd	nd	4.3	8.6	12.6	13.4
	SD	± 0.0	± 0.0	-	-	-	-	-	-
Unknown 5	Mean	nd	nd	nd	nd	nd	nd	nd	nd
	SD	-	-	-	-	-	-	-	-
Unknown 6	Mean	nd	nd	nd	nd	nd	nd	nd	nd
	SD	-	-	-	-	-	-	-	-
Total extractable radioactivity	Mean	104.4	96.9	96.4	100.2	95.0	92.4	88.0	89.0
	SD	± 2.4	± 6.4	-	-	-	-	-	-
Carbon dioxide	Mean	nd	0.4	0.6	0.8	1.6	2.1	2.6	3.6
	SD	± 0.0	± 0.1	-	-	-	-	-	-
Other volatiles	Mean	nd	nd	nd	nd	nd	nd	nd	nd
	SD	-	-	-	-	-	-	-	-
Non-extractable radioactivity	Mean	0.7	1.5	10.2	2.2	9.2	3.5	3.3	6.7
	SD	± 0.3	± 0.0	-	-	-	-	-	-
Total radioactivity (%)	Mean	105.0	98.7	107.2	103.2	105.8	98.1	93.9	99.3
	SD	± 2.1	± 6.4	-	-	-	-	-	-

n.d.: not detected, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A)] Mean values of two replicates, [B] One replicate only for DAT 9 to 120

Table 7.1.2.1.2- 24: Degradation of [2,6-pyridyl-^{14}C]-M-10 in Abington soil under aerobic conditions [% AR]

Compound	Mean [A] / SD	Incubation time (DAT)							
		0	4	9	14	30	63	94	120 [B]
M-05 (AE 1344122)	Mean	102.5	99.9	93.6	88.5	64.3	24.1	5.9	0.3
	SD	± 2.5	± 2.2	± 3.0	± 1.5	± 1.8	± 0.2	± 1.5	-
Unknown 1	Mean	nd	0.7	nd	nd	nd	nd	nd	nd
	SD	-	± 1.0	-	-	-	-	-	-
Unknown 2	Mean	nd	nd	nd	nd	1.0	0.5	3.3	0.5
	SD	-	-	-	-	± 0.3	± 0.6	± 1.7	-
Unknown 3	Mean	nd	nd	nd	nd	nd	nd	2.4	1.9
	SD	-	-	-	-	-	-	± 3.4	-
Unknown 5	Mean	nd	nd	nd	nd	nd	nd	0.3	nd
	SD	-	-	-	-	-	-	± 0.1	-
Unknown 6	Mean	nd	nd	nd	nd	nd	nd	nd	nd
	SD	-	-	-	-	-	-	-	-
Total extractable radioactivity	Mean	102.5	100.6	93.6	88.5	65.3	24.5	12.1	2.6
	SD	± 2.5	± 3.2	± 3.0	± 1.5	± 1.5	± 0.8	± 0.6	-
Carbon dioxide	Mean	nd	1.4	2.8	4.9	14	35.4	43.5	55.4
	SD	± 0.0	± 0.5	± 0.4	± 0.0	± 0.1	± 4.7	± 1.5	-
Other volatiles	Mean	nd	nd	nd	nd	nd	0.1	0.1	0.1
	SD	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	-
Non-extractable radioactivity	Mean	1.0	2.9	6.1	3.6	16.6	31.8	32.6	30.2
	SD	± 0.4	± 0.2	± 0.1	± 4.2	± 0.5	± 0.4	± 1.9	-
Total radioactivity (%)	Mean	103.9	105.0	102.5	97.0	95.9	91.8	88.3	88.3
	SD	± 2.1	± 2.4	± 2.7	± 2.6	± 2.1	± 5.9	± 1.1	-

n.d.: not detected, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A)] Mean values of two replicates; [B] One replicate only for DAT 120

Table 7.1.2.1.2- 25: Degradation of [2,6-pyridyl-^{14}C]-M-10 in Sarotti soil under aerobic conditions [% AR]

Compound	Mean [A]	Incubation time (DAT)							
	SD	0	4	9	14	30	63	94	120
M-05 (AE 1344122)	Mean	97.9	97.5	89.3	80.3	36.3	5.3	0.6	0.1
	SD	± 4.2	± 1.8	± 5.3	± 4.1	± 0.0	± 1.3	± 0.3	± 0.2
Unknown 1	Mean	nd	nd	nd	nd	nd	nd	nd	nd
	SD	-	-	-	-	-	-	-	-
Unknown 2	Mean	nd	nd	nd	nd	0.2	0.6	1.3	nd
	SD	-	-	-	-	± 0.3	± 0.8	± 0.5	-
Unknown 3	Mean	nd	nd	nd	0.7	nd	nd	4.6	2.1
	SD	-	-	-	± 1.0	-	-	± 0.2	± 0.1
Unknown 5	Mean	nd	nd	nd	nd	nd	nd	0.5	nd
	SD	-	-	-	-	-	-	± 0.1	-
Unknown 6	Mean	nd	nd	nd	nd	nd	nd	0.2	nd
	SD	-	-	-	-	-	-	± 0.2	-
Total extractable radioactivity	Mean	97.9	97.5	89.3	81.0	36.5	6.1	7.1	2.2
	SD	± 4.2	± 1.8	± 5.3	± 3.1	± 0.3	± 2.1	± 0.0	± 0.2
Carbon dioxide	Mean	nd	1.0	3.2	5.0	16.0	43.1	53.1	56.1
	SD	-	± 0.0	± 0.1	± 0.0	± 1.1	± 0.2	± 0.5	± 0.3
Other volatiles	Mean	nd	0.3	0.3	0.3	nd	0.1	0.1	nd
	SD	-	± 0.0	± 0.0	± 0.0	-	± 0.0	± 0.0	-
Non-extractable radioactivity	Mean	0.9	4.9	5.3	13.4	38.3	41.4	36.2	31.0
	SD	± 0.0	± 0.1	± 6.3	± 1.9	± 1.6	± 4.1	± 0.7	± 1.4
Total radioactivity (%)	Mean	98.8	103.8	98.0	99.7	90.9	90.7	96.4	89.3
	SD	± 4.2	± 2.0	± 11.5	± 1.1	± 0.3	± 1.9	± 0.2	± 1.5

n.d.: not detected, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Mean values of two replicates

B. Material balance

Mean mass balances were 101.4% AR for Münster soil (range from 93.9 to 107.2% AR), 96.6% AR for Abington soil (range from 88.3 to 105.0% AR) and 96.0% AR for Sarotti soil (range from 89.3 to 103.8% AR).

The results confirm there were no significant losses of radioactivity during sample processing. The material balance at the last two intervals for Abington soil (DAT 94 and 120) and the final interval in Sarotti soil (DAT 120) were slightly below 90%. While there were no signs of losses during work-up and processing this was, possibly due to loss of CO_2.

C. Non-extractable and extractable residues

Values of extractable radioactivity decreased from 97.9 to 104.4% AR at zero time to 89.0% (Münster soil), 2.6% (Abington soil) or 2.2% (Sarotti soil) by the end of the study (DAT 120). Amounts of non-extractable radioactivity increased to a maximum of 10.2% in Münster soil by DAT 30 before declining slightly to 6.7% AR by DAT 120. In Abington soil NER increased to a maximum of 32.6% AR by DAT 94 declining to 30.2% by DAT 120 and to 41.4% AR in Sarotti soil by DAT 64 declining to 31.0% AR by DAT 120.

D. Volatile radioactivity

Mineralisation of [2,6-pyridyl-^{14}C]-M-10 to ^{14}C-carbon dioxide was significant, reaching a maximum of 56.1% AR in Sarotti soil, 55.4 % in Abington soil and 3.6 % in Munster soil by the end of the study (DAT 120). Virtually no organic volatiles were detected throughout the study in any soil.

E. Transformation of test substance

The slowest rate of degradation of pyridyl ring metabolites was observed in Münster soil. In Abington and Sarotti soils, degradation of M-10 led to the formation of four transient unidentified metabolites. In the Munster soil the extent of degradation of M-10 was less with 74% of the test item remaining at the end of the incubation period compared with <1% in the other soils. Two metabolites of M-10 were observed in Münster soil. The largest region detected was a polar degradate (or mixture of degradates) which reached a maximum of 13% AR in Münster soil by the end of the study (DAT 120). This region was also detected as a transient degradate in the other two soils at a maximum of 4.8% AR.

F. Degradation kinetics

Reported SFO DT_{50} values of M-10 under aerobic conditions were 252.5, 35.8 and 24.1 days in Münster, Abington and Sarotti soils, respectively. DT_{50} values ranged from 80.2 to 839 days.

The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.1.2- 26. Best fit kinetics are highlighted in bold.

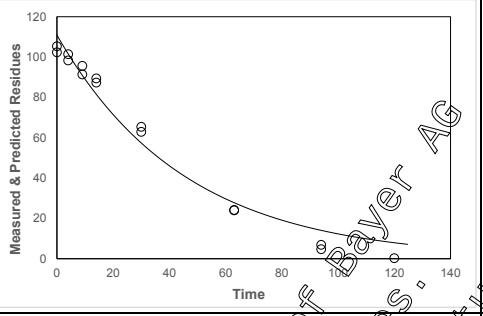
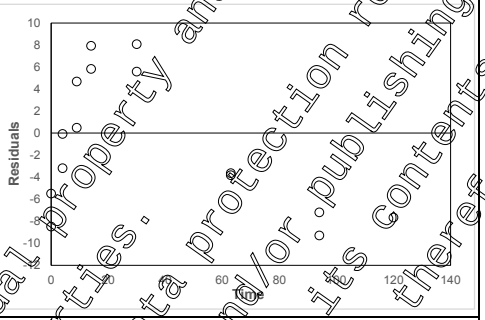
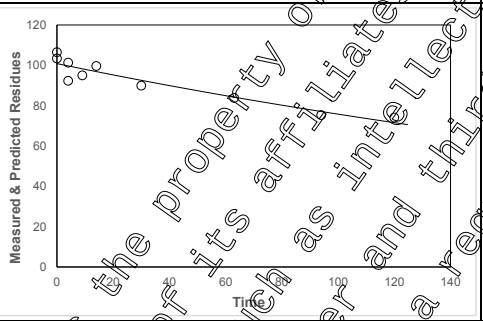
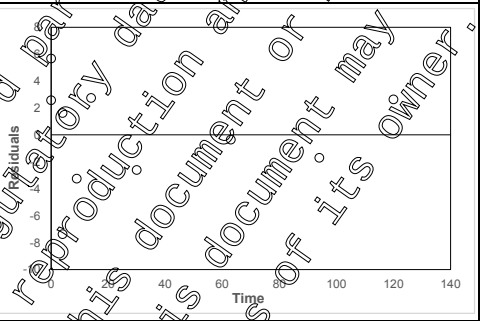
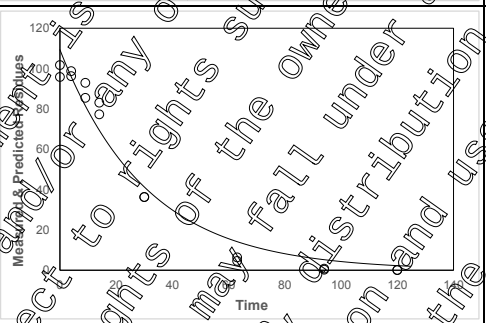
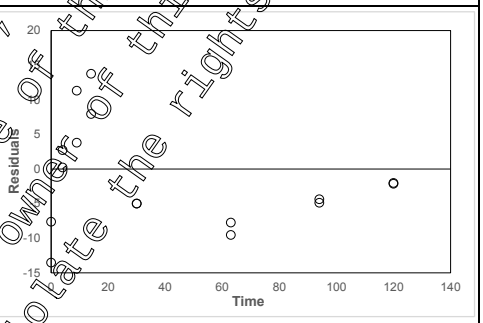
Table 7.1.2.1.2- 26: Degradation rate of M-10 under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003b)	**SFO**	**110.9**	**k 0.02195**	**8.03**	**6.09E-09**	**0.01852**	**0.025**	**31.6**	**104.9**
	FOMC	110.9	α 10150 β 4.63E+05	8.57	n.r. n.r.	8501 4.63E+05	11800 4.63E+05	31.6	104.9
Münster, [redacted] (2003b)	**SFO**	**100.9**	**k 0.002865**	**2.43**	**3.68E-05**	**0.00211**	**0.004**	**241.9**	**803.6**
	FOMC	102.5	α 0.2448 β 42.87	2.31	n.r. n.r.	-0.1525 -76.51	0.642 162.3	684.9	>10000
	DFOP	105.1	k1 1888 k2 0.002565 g 0.061	2.04	NA 0.000233 n.r.	NA 0.001834 0.02595	NA 0.003 0.096	245.7	873
Sarotti, [redacted] (2003b)	**SFO**	**109.4**	**k 0.03248**	**11.2**	**9.06E-08**	**0.02576**	**0.039**	**21.3**	**70.9**
	FOMC	109.4	α 19160 β 5.90E+05	12	n.r. n.r.	15050 5.90E+05	23270 5.90E+05	21.3	70.9

Best fit model highlighted in bold

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.1- 110: Degradation of M-10 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003b)		
Münster SFO [redacted] (2003b)		
Sarotti SFO [redacted] (2003b)		

III. Conclusion

M-10 is a minor metabolite of fluopicolide, which would be predicted to form in the soil environment at very low levels. It was not observed in soil laboratory metabolism studies conducted with fluopicolide. M-10 was degraded in soil, with a significant portion completely mineralized to CO_2 andit would be expected to steadily decline in the soil environment. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of between 21.3 and 241.9 days.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002). The study is considered valid to assess the aerobic degradation of [2,6-pyridyl-^{14}C]-M-10 (AE 1344123) in soil.

Data Point:	KCA 7.1.2.1.2/06
Report Author:	[redacted]
Report Year:	2003
Report Title:	AE 1388273 - Rate of degradation in soil under aerobic conditions
Report No:	M-234149-01-2
Document No:	M-234149-01-2
Guideline(s) followed in study:	OECD 307; SETAC-Europe, March 1995; EU 95/36/EC amending 91/414/EEC
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-14 (AE 1388273) was studied in three soils under aerobic conditions in the laboratory in the dark at 20 ± 2 °C and 55% of the maximum water holding capacity for up to 37 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
Münster	loamy sand	5.4	1.1
Abington	sandy loam	7.2	2.6
Sarotti	silt loam	7.8	1.3

M-14 was applied to soil samples at an application rate of 0.013 mg/kg dry weight, equivalent to a field application rate of 10 g/ha resulting from a conservative estimate of the maximum occurrence of this metabolite and an application rate of 400 g active substance/ha. The chemical purity of M-14was 99.9% which was considered in determining the application rate.

Samples were removed for extraction and analysis immediately after treatment (day 0) and 2, 7, 10, 14, 22, 30 and 37 days of incubation. Soil samples were extracted by microwave extraction with acetonitrile/water (4/1, *v/v*). Soil extracts were analysed by HPLC-MS/MS to quantify the amount M-14 remaining.

M-14 was rapidly degraded in all three soils. The amount in soil extracts declined from between 11.80 to 12.15 µg/kg at time zero to < LOQ (equivalent to < 7.7% of applied) by DAT 37, 22 and 30 in Münster, Abington and Sarotti soils, respectively.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} values of 8.2, 4.9 and 5.8 days in Münster, Abington and Sarotti soils, respectively.

Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)
Münster (loamy sand)	SFO	8.2	27.3	4.02
Abington (sandy loam)	SFO	4.9	16.4	3.75
Sarotti (silt loam)	SFO	5.8	19.3	4

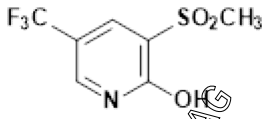
Degradation of the metabolite M-14 in aerobic soil was very rapid and it is not expected to persist in the soil environment.

I. Materials and Methods

A. Materials

1. Test Item

M-14 (referred to as AE 1388273 in the report)



Chemical Purity:	99.9% (HPLC)
Sample/Batch ID:	AE 1388273 00 1B99 0001, MGP2442A
Expiry Date:	25 March 2005

2. Test Soils

The study was performed using three European soils collected fresh from the field with storage times of 42 to 48 days, prior to sieving to 2 mm and dispersing into flasks. Once received at the test facility the soil was stored at 4°C. The soils were collected from the same sites as laboratory aerobic soil studies with fluopicolide, from Abington (Cambridge, England) and Munster and Saroth (Germany). The same batches of soils were used in laboratory aerobic soil studies with the other pyridyl ring metabolites M-02, M-05 and M-10. The physico-chemical characteristics are summarized in Table 7.1.2.1.2- 27.

Table 7.1.2.1.2- 27: Physico-chemical properties of test soils

Parameter		Soil		
Soil Designation		Münster	Abington	Saroth
Geographic Location				
City		Münster, Northrine-Westfalia	Cambridgeshire, United Kingdom	Hattersheim, Hesse
Country		Germany	UK	Germany
Textural Classification (USDA)		Loamy sand	Sandy loam	Silt loam
Sand [50 - 2000 µm]	(%)	80.53	70.04	23.24
Silt [2 – 50 µm]	(%)	15.70	15.60	53.95
Clay [< 2 µm]	(%)	3.77	14.36	22.80
pH				
in $CaCl_2$		5.4	7.2	7.5
in H_2O		6.6	8.1	8.3
in KCl		5.5	7.7	7.7
Organic Carbon (%)		1.1	2.6	1.3
$Ca_{exchangeable}$ (meq/100 g)		11.5	19.9	34.3
$Mg_{exchangeable}$ (meq/100 g)		0.2	1.6	1.6
$Na_{exchangeable}$ (meq/100 g)		<0.05	0.1	0.1
$K_{exchangeable}$ (meq/100 g)		0.4	1.3	0.9
$Mn_{exchangeable}$ (meq/100 g)		<0.05	<0.05	<0.05
$CaCO_3$ equivs (g/kg)		<0.05	73.5	13.4
Phosphorus total (mg/kg)		617.8	1586.3	728.8
Nitrogen total (mg/kg)		1077.9	2380.1	1470.2
Water Holding Capacity				
Maximum (g H_2O per 100 g DW)		46.5	57.1	52.1
Moisture Content During Incubation		40% MWHC	40% MWHC	45% MWHC
Soil Microbial Biomass (µg microbial C /g soil)		BIO-	BIO-	BIO-
Initial (DAT 0)		158	796	217
Mid (DAT 30)		136	851	306
Final (DAT 41)		151	880	319

BIO- samples were treated with water

B. Study Design

1. Experimental Conditions

Tests were performed in static systems consisting of an Erlenmeyer flask containing 25 g soil (dry weight equivalents) fitted with a polyurethane foam plug to maintain aerobic conditions and to minimise water loss.

M-14 was applied at an application rate of 0.013 mg/kg, equivalent to 10 g/ha, reflecting a fluopicolide field application rate of 400 g/ha and a conservative estimate of the maximum predicted occurrence of the metabolite. The test item was dissolved in water and applied dropwise onto the soil surface. Soil samples were adjusted to a moisture content equivalent to 40% MWHC (Münster and Abington soils) or 45% MWHC (Sarotti soil) 40, one day prior to application. The samples were incubated at 20 ± 1 °C under aerobic conditions in the dark for 37 days. Soil moisture was maintained during incubation by the weekly addition of water to the samples. Untreated soil samples were incubated under the same conditions for determination of soil microbial activity.

2. Sampling

Following incubation, duplicate samples were taken for analysis after 0, 2, 7, 10, 14, 22, 30 and 37 days. Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment (Day 0, Day 30 and Day 41).

3. Analytical procedures

The entire soil sample of each test vessel was extracted by microwave extraction with acetonitrile/water (1/4, v/v), the soil extracts centrifuged and the amount of AE 1388273 in the extracts determined by LC-MS/MS. Concurrent recoveries to demonstrate the extraction efficiency and verify the analytical method from all three soils were measured routinely in control samples fortified at 0.01 mg/kg and mean values of 105%, 101% and 99% were obtained for Abington, Sarotti and Münster, respectively. The limit of detection in soil matrix was determined as 0.3 µg/kg (2.3% of applied test substance) and the limit of quantification was determined as 1 µg/kg (7.7% of applied test substance). The analytical method was validated in two soils prior to starting the test.

Samples were usually analysed directly without storage. If storage was required, samples were stored frozen for a maximum of 10 days in a freezer. Storage stability tests established stability at <-18°C for a minimum of 13 days

4. Determination of degradation kinetics:

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of M-14 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-14, an initial comparison was performed for each soil between the SFO and FOMC fits. FOMC fits did not provide any visually discernible improvement over SFO kinetics for any soil. The SFO fits were therefore accepted for all soils.

II. Results and Discussion

A. Analytical Methodology:

A full summary of the analytical method, which is validated in a separate report (M-234149-01-2), is provided in Document MCA 4, Section 4.1.2. The method complies with all criteria according to *SANCO/3029/99 rev. 4* and is suitable for the determination of M-14 in soil samples by HPLC-MS/MS.

B. Data:

M-14 was rapidly degraded in all three soils. The amount in soil extracts declined from 11.95 µg/kg at time zero to < LOQ (equivalent to < 7.7% of applied) by DAT 37 in Münster soil, from 11.80 µg/kg at time zero to < LOQ by DAT 22 in Abington soil and from 12.15 µg/kg at time zero to < LOQ by DAT 30 in Sarotti soil.

The results for each soil are summarized in Table 7.1.2.1.2- 28 to Table 7.1.2.1.2- 30.

Table 7.1.2.1.2- 28: Degradation of M-14 in Münster soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	2	7	10	14	22	30	37
Mean	µg/kg	11.95	9.80	6.51	5.63	3.36	1.65	1.07	<LOQ
SD	µg/kg	0.05	0.15	0.19	0.03	0.08	0.04	0.04	-

DAT: days after treatment
LOQ = 1 µg/kg; LOD = 0.3 µg/kg

Table 7.1.2.1.2- 29: Degradation of M-14 in Abington soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	2	7	10	14	22	30	37
Mean	µg/kg	11.80	8.95	4.40	2.87	1.52	<LOQ	<LOQ	<LOD
SD	µg/kg	0.00	0.03	0.03	0.03	0.00	-	-	-

DAT: days after treatment
LOQ = 1 µg/kg; LOD = 0.3 µg/kg

Table 7.1.2.1.2- 30: Degradation of M-14 in Sarotti soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	2	7	10	14	22 [A]	30	37
Mean	µg/kg	12.15	9.02	4.98	3.81	2.29	0.81	<LOQ	<LOD
SD	µg/kg	0.05	0.16	0.13	0.05	0.10	0.31	-	-

DAT: days after treatment
LOQ = 1 µg/kg; LOD = 0.3 µg/kg
[A] Replicate values 1.1 µg/kg and <LOQ. Mean value calculated assuming value < LOQ = ½ LOQ

F. Degradation Kinetics

Reported SFO DT_{50} values of M-14 under aerobic conditions were 8.9, 4.9 and 5.8 days in Münster, Abington and Sarotti soils, respectively. DT_{90} values ranged from 16.2 to 27.2 days.

The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.1.2- 31. Best fit kinetics are highlighted in bold.

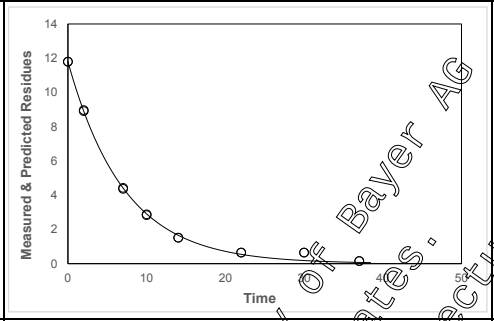
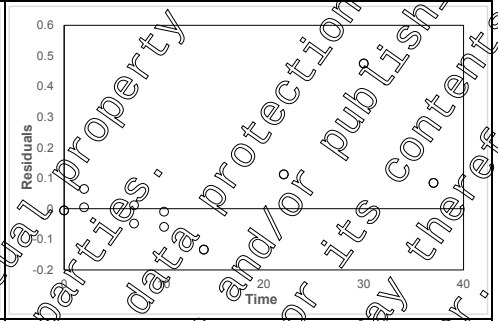
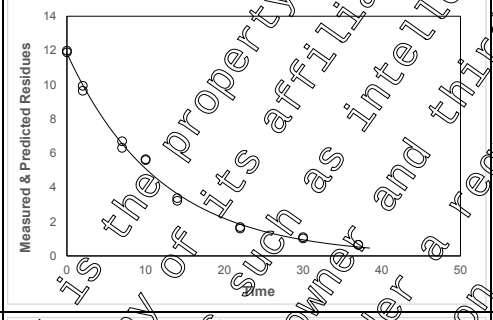
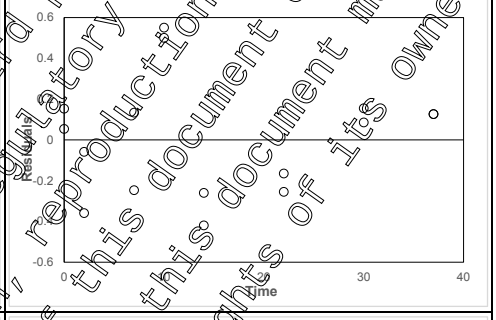
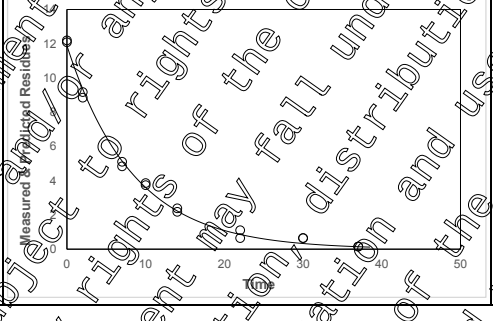
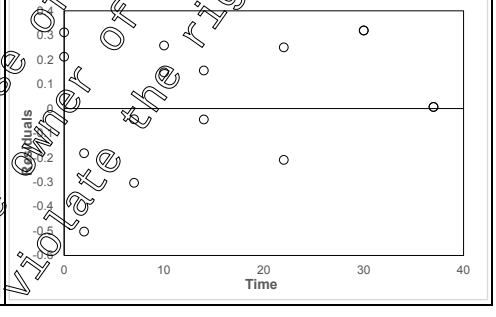
Table 7.1.2.1.2- 31: Degradation rate of M-14 under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Abington, [redacted] (2003)	**SFO**	**11.81**	**k 0.1404**	**3.75**	**<2e-16**	**0.1345**	**0.146**	**4.9**	**16.4**
	FOMC	11.88	α 10.59 β 70.34	3.58	n.r. n.r.	-0.6072 -9.509	21.78 150.2	4.8	17.1
Münster, [redacted] (2003)	**SFO**	**11.85**	**k 0.08429**	**4.02**	**3.01E-15**	**0.0795**	**0.089**	**8.2**	**27.3**
	FOMC	11.85	α 606.5 β 7187	4.29	n.r. n.r.	-55090 -6.54E+05	56310 6.68E+05	8.2	27.3
Sarotti (SLS), [redacted] (2003)	**SFO**	**11.89**	**k 0.1194**	**4**	**1.33E-15**	**0.113**	**0.126**	**5.8**	**19.3**
	FOMC	12.03	α 6.376 β 47.38	3.17	n.r. n.r.	1.655 8.062	11.1 86.69	5.4	20.6

Best fit model highlighted in bold

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.1- 111: Degradation of M-14 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Abington SFO [redacted] (2003)		
Münster SFO [redacted] (2003)		
Sarotti (SLS) SFO [redacted] (2003)		

III. Conclusion

M-14 is a minor metabolite of fluopicolide, which would be predicted to form in the soil environment at very low levels. It was not observed in soil laboratory metabolism studies conducted with fluopicolide. M-14 was rapidly degraded in soil and thus, it would not be expected to persist in the soil environment. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit un-normalised DT_{50} value of between 4.9 and 8.2 days.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002). The study is considered valid to assess the aerobic degradation of M-14 in soil.

Data Point:	KCA 7.1.2.1.2/07
Report Author:	[redacted]
Report Year:	2004
Report Title:	Kinetic evaluation of data from a PCA (AE C657188) rate of degradation study in three soils
Report No:	C037887
Document No:	M-223743-01-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EC, Ann.II, sect 7,7.1.1
Deviations from current test guideline:	Yes. The requirements of kinetic evaluations according to FOCUS kinetics have changed.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Supportive only

In the previous submission (DAR, 2005), this modelling report was evaluated and accepted as valid for risk assessment purposes. However additional studies have been conducted and the requirements of kinetic evaluations according to FOCUS kinetics have changed, thus the report is no longer considered as valid. It has been superseded by KCA 7.1.2.1.1/10 ([redacted] 2019, M-685680-01-1) and hence a summary is not presented in this dossier.

Data Point:	KCA 7.1.2.1.2/08
Report Author:	[redacted]
Report Year:	2017
Report Title:	AE1413903: Aerobic degradation in four soils
Report No:	S16-01252
Document No:	M-585202-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307, 2002 SANCO/3029/99 rev.4
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted in the Confirmatory Data (2017).
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-15 (AE 1413903) was studied in four soils under aerobic conditions in the laboratory in the dark at 20 ± 2 °C and 55% of the maximum water holding capacity for up to 120 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
L[redacted]	loamy sand	6.0	1.8
Dollendorf II	loam	7.3	5.6
L[redacted]	sandy loam	5.8	2.1
H[redacted]	silt loam	6.0	1.8

M-15 was applied to soil samples at an application rate of 0.043 mg/kg dry weight, equivalent to a field application rate of 133 g/ha of fluopicolide and a highly conservative estimate of the maximum occurrence of this metabolite. The chemical purity of M-15 was > 93.4% which was considered in determining the application rate.

Samples were removed for extraction and analysis immediately after treatment (day 0) and 1, 3, 8, 14, 28, 60 and 120 days of incubation. Soil samples were extracted at ambient temperature three times with acetonitrile/water (4/1, *v/v*) and then by microwave extraction with acetonitrile/water (4/1, *v/v*) at 60 °C. Soil extracts were analysed by HPLC-MS/MS to quantify the amount M-15 remaining.

M-15 was steadily degraded in all four soils. The amount in soil extracts declined from between 0.037 to 0.042 mg/kg at time zero to 0.017 to 0.019 mg/kg by DAT 120, equivalent to 44.9%, 40.0%, 42.1% and 44.3% of applied in soil L[redacted], Dollendorf II, L[redacted] and H[redacted] soils respectively.

The following DT_{50} and DT_{90} values were calculated for M-15 in the four soils.

Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)	Visual Assessment
L[redacted] (sandy loam)	SFO	112	371	5.6	Moderate
Dollendorf II (clay loam)	DFOP	151	n.d.	4.8	Good
L[redacted] (sandy loam)	SFO	119	394	5.3	Moderate
H[redacted] (silt loam)	SFO	126	419	3.9	Good

A re-evaluation of the degradation kinetics resulted in similar best-fit DFOP un-normalised DT_{50} values of 102.7 to 113.2 days and DT_{90} values of 410 to 506.1 days.

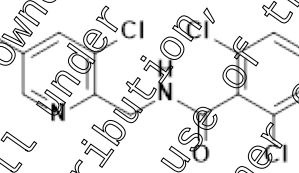
Degradation of the metabolite in aerobic soil was relatively slow, however M-15 is a minor metabolite of fluopicolide formed in the soil environment at very low levels. Should M-15 form in soil it would be steadily degraded and would not be expected to accumulate.

I. Materials and Methods

A. Materials

1. Test Item

M-15 (referred to as AE 1413903 in the report)



Chemical Purity: 93.4%

Sample/Batch ID: ME20030113-2-7B-1

Expiry Date: 18 February 2019

2. Test Soils

The study was performed using four German soils as characterized in Table 7.1.2.1.2- 32. The same soils were also used in laboratory aerobic soil studies with fluopicolide. Soils were collected fresh from the field and used with minimal storage time prior to sieving to 2 mm and dispersing into flasks.

Table 7.1.2.1.2- 32: Physico-chemical properties of test soils

Parameter	Soil							
Soil Designation	L[redacted]		Dollendorf II		[redacted]		H[redacted]	
Soil ID	[redacted]		DD		[redacted]		[redacted]	
Geographic Location City Country	Monheim, Germany		Blankenheim, Germany		Monheim, Germany		Burscheid, Germany	
GPS coordinates	[redacted]		[redacted]		[redacted]		[redacted]	
Batch Number	20160405		20160401		20160401		20160401	
Textural Classification (USDA)	Sandy loam		Clay loam		Sandy loam		Silt loam	
Sand [50 - 2000 µm] (%)	74		34		54		26	
Silt [2 – 50 µm] (%)	18		38		32		58	
Clay [< 2 µm] (%)	8		28		14		16	
pH								
in $CaCl_2$ (1:2)	6.0		7.3		5.3		6.0	
in H_2O (1:1)	6.2		7.4		5.6		6.2	
Saturated paste	6.1		7.4		5.5		6.2	
in KCl (1:1)	5.7		7.0		5.0		5.7	
Organic Matter (%) *	2.6		8.4		3.3		3.1	
Organic Carbon (%)	1.5		4.9		1.9		1.8	
Cation Exchange Capacity (meq/100 g)	8.1		18.7		9.3		10.7	
Water Holding Capacity								
Maximum (g H_2O per 100 g DW)	46.1		79.3		59.0		53.8	
at 1/3 bar (%)	10.0		31.6		16.1		18.7	
Moisture Content During Incubation (%)	55% MWHC		55% MWHC		55% MWHC		55% MWHC	
Bulk Density (disturbed, g/cm^3)	1.25		0.98		1.13		1.11	
Soil Microbial Biomass (mg microbial C /100 g soil)	BIO^-	BIO^+	BIO^-	BIO^+	BIO^-	BIO^+	BIO^-	BIO^+
Arrival	233.3		449.8		267.4		228.0	
Initial (DAT 0)	212.2		458.8		222.2		227.2	
Mid (DAT 58)	188.1	184.8	400.5	390.6	198.5	195.5	218.5	228.1
Final (DAT 120)	154.7	163.1	334.5	369.2	163.5	157.5	177.0	179.3

* Calculated by multiplying organic carbon content by 1.724
MWHC = Maximum Water Holding Capacity
BIO^- samples were untreated
BIO^+ samples were treated with 400 µL of methanol:water (1:1, v/v)

B. Study Design

1. Experimental Conditions

Samples of 100 g dry weight of soil each were filled into glass incubation flasks and pre-equilibrated prior to treatment at approximate study conditions (darkness, 20 ± 2 °C, soil moisture content equivalent to 55 ± 5% of maximum water holding capacity (MWHC)).

At the start of the test, each sample received 0.043 mg test substance/kg soil reflecting the maximum single field application rate of 133 g/ha of fluopicolide and a highly conservative formation level for the metabolite.

Samples were incubated and maintained at 20 ± 2 °C and 55 ± 5% of MWHC in the dark for a maximum of 120 days. Soil samples were maintained under static conditions. All the flasks were stoppered with cotton wool. Untreated soil samples were incubated under the same conditions for determination of soil microbial activity. Additional untreated flasks containing 100g (dry weight) equivalent soil [redacted] were used to provide fortification samples to confirm the analytical method efficiency. At each sampling interval two flasks were fortified at the LOQ level (0.002 mg/kg, 5% of the application rate) and two flasks at 22 times the LOQ (0.0473 mg/kg, 110% of the application rate).

2. Sampling

Duplicate samples were taken for analysis after 0, 1, 3, 8, 14, 28, 60 and 120 days of incubation. Microbial soil biomass samples were analysed on arrival and at the start, midpoint and end of the experiment (DAT 0, 58 and 120).

3. Analytical procedures

The entire soil sample of each test vessel was extracted three times with a mixture of acetonitrile:water (4:1, v/v) at ambient temperature. Ambient extraction was followed by an additional microwave extraction with acetonitrile:water (4:1, v/v) at 60°C. All extracts were combined, and an aliquot centrifuged and diluted with acetonitrile/water (1/1 v/v). Identification of M-15 in soil extracts was by HPLC-MS/MS. The MS analysis was performed with ESI (Electrospray Ionisation) in negative ion SRM mode (selected reaction monitoring). For each analytical run, an 8 point calibration curve using standards of 0.010 ng/mL to 10 ng/mL was constructed to allow quantification of the extracts. The analytical method was validated with all four soils prior to starting the test.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The degradation of M-15 followed single first order (SFO) kinetics in [redacted], [redacted] and [redacted] soils and double first order in parallel (DFOP) kinetics in Dollendorf soil based on lowest chi² error values and visual assessments of fits.

Additionally, modelling endpoints for the degradation of M-15 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-15, an initial comparison was performed for each soil between the SFO and FOMC fits, which suggested that decline was slightly bi-phasic. The DFOP model was therefore also fitted and was accepted for all soils. For the [redacted] soil, the t-test did not complete successfully for the DFOP rate constant k_1, however the fit was accepted as the DT_{50} and DT_{90} values derived were identical to those obtained from an additional fit of the HS model with statistically significant parameter estimates, which is provided as supporting information (note - the HS model is not recommended by FOCUS to derive trigger endpoints).

II. Results and Discussion

A. Analytical Methodology:

A full summary of the analytical method is provided in Document MCA 4, Section 4.1.2. The method complies with all criteria according to *SANCO/3029/99 rev. 4* and is suitable for the determination of M-15 in soil samples by HPLC-MS/MS.

B. Data:

M-15 was steadily degraded in all four soils. The amount in soil extracts declined from between 0.037 to 0.042 mg/kg at time zero to 0.017 to 0.019 mg/kg by DAT 120, equivalent to 44.9%, 40.0%, 42.1% and 44.3% of applied in L[redacted], Dollendorf II, [redacted] and H[redacted] soils, respectively.

The results for each soil are summarized in Table 7.1.2.1.2- 33 to Table 7.1.2.1.2- 36.

Table 7.1.2.1.2- 33: Degradation of M-15 in L[redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	1	3	8	14	28	60	120
Mean	mg/kg	0.04234	0.04118	0.03607	0.03352	0.03294	0.0334	0.02631	0.0193
SD	mg/kg	0.00016	0.00015	0.0006	0.00078	0.00071	0.00028	0.00018	0.00003
Mean	%	98.5	95.8	83.9	78.0	76.6	77.7	61.2	44.9
RSD	%	0.4	0.4	1.7	2.3	2.2	0.8	0.7	0.2

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

Table 7.1.2.1.2- 34: Degradation of M-15 in Dollendorf II soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	1	3	8	14	28	60	120
Mean	mg/kg	0.03691	0.03508	0.03153	0.02857	0.02678	0.02498	0.02291	0.01719
SD	mg/kg	0.00139	0.00103	0.00013	0.00068	0.00044	0.00087	0.00096	0.00023
Mean	%	85.8	81.6	73.3	66.4	62.3	58.1	53.3	40.0
RSD	%	3.7	2.9	0.4	2.4	1.6	3.4	4.2	1.2

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

Table 7.1.2.1.2- 35: Degradation of M-15 in L[redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	1	3	8	14	28	60	120
Mean	mg/kg	0.04057	0.03457	0.03424	0.03422	0.03001	0.03129	0.0258	0.01809
SD	mg/kg	0.00173	0.00161	0.00089	0.00005	0.00086	0.00003	0.00019	0.0003
Mean	%	94.3	80.4	79.6	79.6	69.8	72.8	60.0	42.1
RSD	%	4.3	4.7	2.6	0.2	2.8	0.1	0.7	1.7

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

Table 7.1.2.1.2- 36: Degradation of M-15 in H[redacted] [redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	1	3	8	14	28	60	120
Mean	mg/kg	0.03955	0.03405	0.03548	0.03315	0.0323	0.03072	0.02579	0.01905
SD	mg/kg	0.0029	0.00276	0.00054	0.00033	0.00035	0.0003	0.00011	0.00045
Mean	%	92.0	79.2	82.5	77.1	75.1	71.4	60.0	44.3
RSD	%	7.3	8.1	1.5	1.0	1.1	0.9	0.4	2.2

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

F. Degradation Kinetics

Best fit reported DT_{50} values of M-15 under aerobic conditions were 112, 151, 119 and 126 days in L[redacted] [redacted], Dollendorf II, L[redacted] [redacted] and H[redacted] soils, respectively. The experimental data were best described by either a simple first order (SFO) in L[redacted] [redacted], H[redacted] [redacted] and L[redacted] [redacted] soils or a double first order in parallel (DFOP) kinetic model in Dollendorf soil. Details are provided below in Table 7.1.2.1.2- 37.

Table 7.1.2.1.2- 37: Reported degradation rate of M-15 under aerobic conditions at 20 ºC (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} (days)	DT_{90} (days)
L[redacted]	SFO	0.03869	k 0.0062107	5.6	3.71e-07	0.008	0.0007368	112	371
Dollendorf II	DFOP	0.03539	k1 0.0571716 k2 0.0013922 g 0.3828566	4.8	1.52e-05 0.0279 9.78e-10	0.0398683 0.0001038 0.3357395	0.074 0.003 0.430	151	NC
L[redacted]	SFO	0.03604	k 0.0058466	5.3	6.44e-07	0.0044218	0.007	119	394
H[redacted]	SFO	0.03595	k 0.0054999	3.9	1.73e-07	0.0043000	0.007	126	419

NC.: Not calculated by KinGUII

In addition, the experimental data for the degradation of M-15 been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting DT_{50} values for trigger endpoints for M-15 are summarised below in Table 7.1.2.1.2- 38. Best fit kinetics are highlighted in bold.

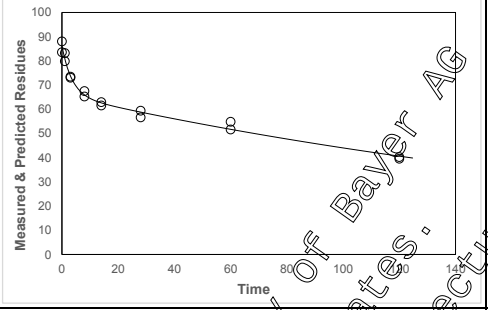
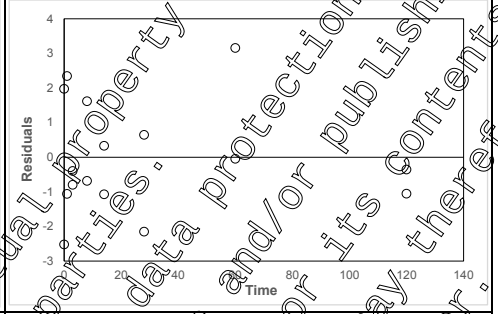
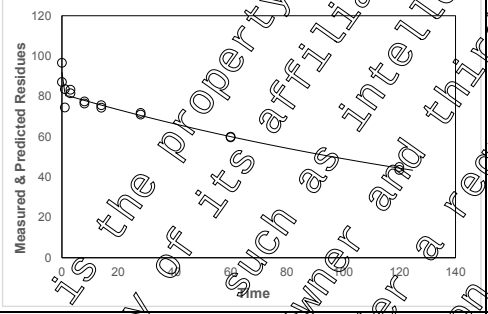
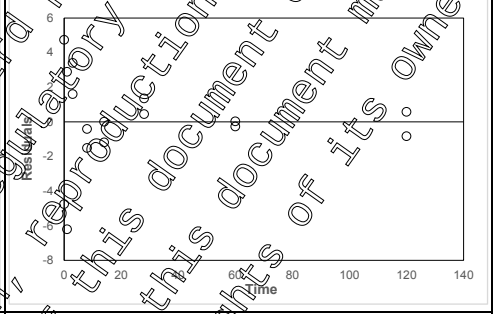
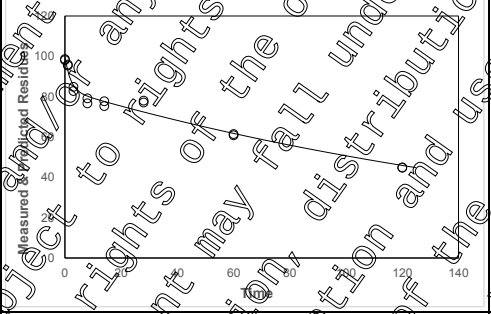
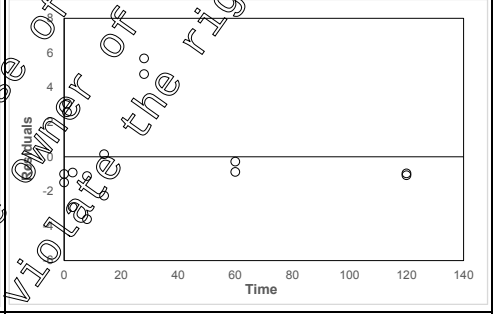
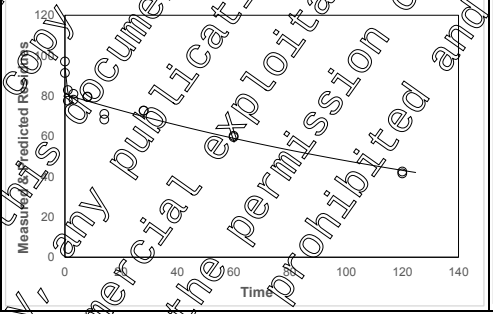
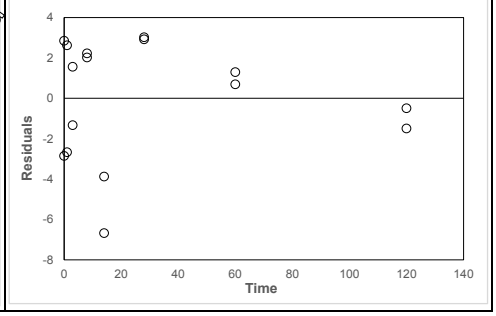
Table 7.1.2.1.2- 38: Re-evaluation of degradation rate of M-15 under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Dollendorf II, [redacted] (2016d)	SFO	76.03	k 0.00625	7.16	4.77E-06	0.004431	0.008	110.9	368.4
	FOMC	85.5	α 0.1742 β 2.727	2.92	n.r. n.r.	0.1288 0.4348	0.22 5.018	143.1	>10000
	DFOP	**86.12**	**k1 0.2768 k2 0.004019 g 0.2353**	**1.08**	**0.000202 2.90E-08 n.r.**	**0.1648 0.003352 0.2027**	**0.389 0.005 0.268**	**105.7**	**506.1**
H[redacted] 2016d)	SFO	83.59	k 0.005497	3.9	6.52E-08	0.00439	0.007	126.1	418.9
	FOMC	84.69	α 0.8885 β 116.4	3.96	n.r. n.r.	-0.8053 -176.7	2.582 409.4	137.5	1437
	DFOP	**91.95**	**k1 22 k2 0.005028 g 0.1168**	**1.49**	**<2e-16 1.07E-08 n.r.**	**22 0.004264 0.06913**	**22 0.006 0.165**	**113.2**	**433.3**
L[redacted] 016d)	SFO	89.97	k 0.006216	5.6	2.43E-07	0.004821	0.008	111.5	370.5
	FOMC	94.18	α 0.3379 β 19.77	5.28	n.r. n.r.	0.08529 -9.301	0.59 48.83	134	>10000
	DFOP	**99.69**	**k1 0.4867 k2 0.004965 g 0.1652**	**2.94**	**0.0123 4.13E-08 n.r.**	**0.1157 0.004114 0.12**	**0.858 0.006 0.21**	**103.2**	**427.4**
L[redacted] (2016d)	SFO	83.81	k 0.005842	5.32	3.38E-07	0.004494	0.007	118.7	394.2
	FOMC	85.02	α 0.8946 β 108.5	5.54	n.r. n.r.	-0.9788 -194.8	2.768 411.9	127	1315
	DFOP	**94.35**	**k1 22.61 k2 0.005237 g 0.1437**	**2.95**	**NA 1.58E-08 n.r.**	**NA 0.004414 0.09579**	**NA 0.006 0.192**	**102.7**	**410**

Best fit kinetics are highlighted in bold

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.2- 39: Degradation of M-15 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Dollendorf II DFOP [redacted] (2016d)		
H[redacted] DFOP [redacted] (2016d)		
L[redacted] DFOP [redacted] (2016d)		
L[redacted] DFOP [redacted] (2016d)		

III. Conclusion

M-15 is a minor metabolite of fluopicolide, which would be predicted to form in the soil environment at very low levels. It was not observed in soil laboratory metabolism studies conducted with fluopicolide. Should M-15 form in soil it would be steadily degraded with reported half-lives of between 112 and 151 days. Re-evaluated best fit DT_{50} values were similar ranging from 102.7 to 113.2 days.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002). The study is considered valid to assess the aerobic degradation of M-15 in soil.

Data Point:	KCA 7.1.2.1.2/09
Report Author:	[redacted]
Report Year:	2017
Report Title:	[pyridyl-2,6-14C] AE C657188: Aerobic soil metabolism in four European soils
Report No:	EnSa-16-0574
Document No:	M-581364-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307 Commission Regulation (EU) No 283/2013 / DRAFT SANCO 11802/2010 rev 7 in accordance with Regulation (EC) No 1107/2009 US EPA OCSPP Test Guideline No. 835.4100 / 835.4200
Deviations from current test guideline:	none
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-02 (AE C657188) was studied in four soils under aerobic conditions in the laboratory in the dark at 20 ± 2 °C and 55% of the maximum water holding capacity for up to 120 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
L[redacted]	loamy sand	5.2	1.8
Dollendorf II	loam	6.9	5.6
L[redacted]	sandy loam	4.9	2.1
H[redacted]	silt loam	5.9	1.8

[2,6-pyridyl-^{14}C]-labelled M-02 was applied to soil samples at an application rate of 0.013 mg/kg dry weight, equivalent to a field application rate of 10 g/ha resulting from a conservative estimate of the maximum occurrence of this metabolite and an application rate of 400 g active substance/ha. The radiochemical purity and specific activity were > 98 % and 4.33 MBq/mg, respectively.

Samples were removed for extraction and analysis immediately after treatment (day 0) and 3, 7, 10, 16, 28, 70 and 120 days of incubation. Soil samples were extracted at ambient temperature three times with acetonitrile/water (4/1, *v/v*) and then by two microwave extraction, first with acetonitrile/water (4/1, *v/v*) at 70 °C and then with acetone at 50 °C. Concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC). Selected extracts were analysed by normal phase thin layer chromatography (TLC) to confirm the results obtained by HPLC. LC/MS/MS including accurate mass determination was used to identify metabolites isolated from selected soil extracts.

Recovery of radioactivity was quantitative throughout the study. Overall mean mass balances were 97.3% AR for L[redacted] soil, 96.3% AR for Dollendorf II soil, 95.7% AR for L[redacted] soil and 96.5% AR for H[redacted] soil.

Total extractable residues decreased from 91.5 to 98.1% of applied at zero time to 7.4% (L[redacted] soil), 3.6% (Dollendorf II soil), 2.4% (L[redacted] soil) or 1.8% (H[redacted] soil) by DAT 120. Levels of non-extractable radioactivity (NER) increased rapidly in all soils peaking at DAT 3 or DAT 7 and then declining gradually by DAT 120. Overall the maximum amount of NER observed was 42.7% AR in L[redacted] soil at DAT 3, which declined to 23.5% AR by DAT 120. Mineralization to carbon dioxide was significant with a rapid increase in the carbon dioxide production, reaching a maximum of 56.1, 64.0, 67.7 and 65.9% by DAT-120 in L[redacted], Dollendorf II, L[redacted] and H[redacted] soils. Virtually no volatile organic products were detected throughout the study (maximum 1.8% AR).

M-02 was very rapidly metabolised in all soils declining to below the LOD (< 1.1% AR) by DAT 7 in L[redacted] and L[redacted] soils, and by DAT 16 in Dollendorf II and H[redacted] soils. Metabolism of M-02 was accompanied by the formation of numerous metabolites including M-05, M-09, M-10 and M-20 (called AE 1344122, AE 1344123, AE B102859 and BCS-BX16566 in the report), along with a further three unidentified metabolites.

Degradation kinetics for M-02 provided in the report were conducted in accordance with FOCUS guidance document on degradation kinetics (2014). The best-fit DT_{50} values were 0.67, 0.85, 0.73 and 1.01 days in L[redacted], Dollendorf II, L[redacted] and H[redacted] soils, respectively.

Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)	Visual Assessment
L[redacted] (loamy sand)	SFO	0.67	2.22	0.7	Good
Dollendorf II (loam)	FOMC	0.85	4.06	0.7	Good
L[redacted] (sandy loam)	SFO	0.73	2.43	0.6	Good
H[redacted] (silt loam)	FOMC	1.01	3.66	1.7	Good

A re-evaluation of the degradation kinetics resulted in similar best-fit un-normalised ranging from 0.7 to 1.1 days and DT_{90} values of 2.2 to 4.0 days.

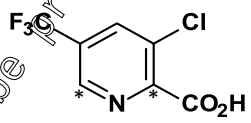
Degradation of the metabolite M-02 in aerobic soil was very rapid, with significant formation of carbon dioxide as a result of complete mineralization of the molecule. Accordingly, M-02 will not persist in the soil environment.

I. Materials and Methods

A. Materials

1. Test Item

[2,6-Pyridyl-^{14}C]-M-02 (referred to as AE C657188 in the report)



* Denotes position of [^{14}C]-radiolabel

Specific Activity:	4.33 MBq/mg
Radiochemical Purity:	>98% (HPLC)
Chemical Purity:	>99% (HPLC)
Sample/Batch ID:	KML 9974

2. Test Soils

The study was performed using four German soils as characterized in Table 7.1.2.1.2- 40. The same soils were also used in laboratory aerobic soil studies with fluopicolide. Soils were collected fresh from the field and used with minimal storage time prior to sieving to 2 mm and dispersing into flasks.

Table 7.1.2.1.2- 40: Physico-chemical properties of test soils

Parameter	Soil							
Soil Designation	L[redacted]		Dollendorf II		L[redacted]		H[redacted]	
Soil ID	[redacted]		DD		[redacted]		[redacted]	
Geographic Location								
City	Monheim,		Blankenheim,		Monheim,		Burscheid,	
Country	Germany		Germany		Germany		Germany	
Batch Number	20150803		20150803		20150803		20150803	
Soil Taxonomic Classification (USDA)	Sandy-mixed mesic Typic Cambudoll		Fine-loamy, mixed, active, frigid Typic Eutrudept		No information		No information	
Textural Classification (USDA)	Loamy sand		Loam		Sandy loam		Silt loam	
Sand [50 - 2000 µm] (%)	80		34		58		28	
Silt [2 – 50 µm] (%)	16		40		32		60	
Clay [< 2 µm] (%)	4		26		10		12	
pH								
in $CaCl_2$ (1:2)	5.2		6.9		4.9		5.9	
in H_2O (1:1)	5.4		7.0		5.1		6.1	
Saturated paste	5.5		7.0		5.1		6.1	
in KCl (1:1)	5.0		6.7		4.6		5.6	
Organic Matter (%) *	3.1		9.7		3.6		3.1	
Organic Carbon (%)	1.8		5.6		2.1		1.8	
Cation Exchange Capacity (meq/100 g)	8.2		18.8		9.7		10.5	
Water Holding Capacity								
Maximum (g H_2O per 100 g DW)	52.4		83.6		59.3		54.9	
at 1/10 bar (%)	13.9		45.7		28.2		38.6	
Moisture Content During Incubation (%)	54.0% MWHC		54.6% MWHC		54.1% MWHC		54.0% MWHC	
Bulk Density (disturbed, g/cm³)	1.21		0.96		1.14		1.11	
Soil Microbial Biomass (µg microbial C /g soil)	BIO⁻	BIO⁺	BIO⁻	BIO⁺	BIO⁻	BIO⁺	BIO⁻	BIO⁺
Initial (DAT 4)	801		2715		979		831	
Mid (DAT 80)	466	468	2235	2167	643	628	573	575
Final (DAT 126)	365	355	1939	1880	424	413	477	472

* Calculated by multiplying organic carbon content by 1.724
MWHC = Maximum Water Holding Capacity
BIO⁻ samples were untreated
BIO⁺ samples were treated with 400 µL of water

B. Study Design

1. Experimental Conditions

Tests were performed in static systems consisting of an Erlenmeyer flask containing 100 g soil (dry weight equivalents) fitted with a trap attachment (permeable for oxygen) containing soda lime for absorption of carbon dioxide and a polyurethane foam plug for adsorption of volatile organic compounds.

The test item [2,6-pyridyl-^{14}C]-M-02 was dissolved in water with methanol as a co-solvent (0.3%) and applied dropwise onto the soil surface (400 µL per flask) at an application rate of 0.013 mg/kg. The application rate was equivalent to 10 g/ha, reflecting a fluopicolide field application rate of 400 g/ha and a conservative estimate of the maximum predicted occurrence of the metabolite. The soil moisture content was adjusted to 54.2% MWHC 3 days prior to application. The samples were incubated at 20 ± 2 °C under aerobic conditions in the dark. Untreated soil samples were incubated under the same conditions for determination of soil microbial activity.

2. Sampling

Duplicate samples were taken for analysis after 0, 3, 7, 10, 16, 28, 70 and 120 days of incubation. Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment (DAT 4, 80 and 126).

3. Analytical procedures

Soil samples were first extracted three times with acetonitrile/water (4/1, *v/v*) at ambient temperature followed by two microwave extractions, first with acetonitrile/water (4/1, *v/v*) at 70 °C and then with acetone at 50 °C. After each extraction step, extract and soil were separated by centrifugation.

Radioactivity in extracts was determined by liquid scintillation counting (LSC). Ambient and microwave soil extracts were pooled and concentrated prior to analysis by HPLC. The HPLC LOD and LOQ were determined as 1.1% and 3.3% AR. The mean recovery of the concentration procedure for the combined soil extracts was between 88.9 and 96.1% for all soils. The mean HPLC column recovery was 110%. The primary chromatographic method for analysis of soil extracts was a reverse phase C18 HPLC method. Selected extracts were analysed by a second confirmatory normal phase TLC method. Four degradation products (M-05, M-09, M-10 and M-20, called AE 1344122, AE 1344123, AE B102859 and BCS-BX16566 in the report) were identified by LC/MS/MS including accurate mass determination after isolation of the radiopeak from selected soil extracts.

With the exception of the time zero samples, trap attachments were removed for analysis at each sampling time. The quantity of radioactive volatiles generated was determined by processing the elements that made up each volatile trap. The volatile organics were extracted from the polyurethane bung using ethyl acetate. The carbon dioxide adsorbed on the soda lime was released by digesting the soda lime with hydrochloric acid and re-trapped into a series of traps containing scintillation cocktail. The radioactivity contained in the 120 DAT traps was trapped in aqueous potassium hydroxide and confirmed as $^{14}CO_2$ by precipitation as ^{14}C barium carbonate with aqueous barium chloride.

Following homogenisation, non-extractable residues (NER) in extracted soils were determined by combustion. NER were further characterized by organic matter fractionation into humic acids, fulvic acids and humin fractions extracted soil samples from DAT-120.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The degradation of M-02 followed single first order (SFO) kinetics in soils L[redacted] and L[redacted] soils and first-order multi compartment (FOMC) kinetics in soils Dollendorf II and H[redacted] based on lowest chi^2 error values and visual assessments of fits.

Additionally, modelling endpoints for the degradation of M-02, M-05, M-10 and M-20 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-02 (PCA), an initial comparison was performed for each soil between the SFO and FOMC fits. For the Dollendorf II soil, the FOMC model resulted in a better visual fit, and the DFOP model was therefore also fitted. DFOP provided the best fit to the data, with the lowest χ^2 err% value, and was accepted as the most appropriate model to describe M-02 degradation. For the remaining soils, SFO resulted in a more appropriate fit than FOMC for M-02 degradation.

Further metabolite optimisations were performed using the best-fit model for the applied compound and the SFO model for metabolites.

II. Results and Discussion

A. Data

The distribution and characterisation of radioactivity for the test soils incubated at 20 °C following application of [2,6-pyridyl-^{14}C]- M-02 are summarized in Table 7.1.2.1.2- 41 to Table 7.1.2.1.2- 44.

Table 7.1.2.1.2- 41: Degradation of [2,6-pyridyl-^{14}C]-M-02 in L[redacted] soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)							
		0	3	7	10	16	28	70	120
M-02 (AE C657188)	Mean	97.3	4.6	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.
	SD	±0.3	±1.0						
M-05 (AE 1344122)	Mean	n.d.	11.1	6.4	5.1	2.4	< LOD	n.d.	n.d.
	SD		±0.5	±0.1	±0.0	±0.1			
M-09 (AE B102859)	Mean	n.d.	< LOD	< LOD	< LOD	n.d.	n.d.	n.d.	n.d.
	SD								
M-10 (AE 1344123)	Mean	n.d.	18.4	13.4	12.7	12.6	12.3	9.7	6.2
	SD		±1.3	±0.5	±0.1	±0.2	±0.2	±0.0	±0.3
M-20 (BCS-BX16566)	Mean	n.d.	n.d.	4.2	4.7	3.1	1.4	n.d.	< LOD
	SD			±0.0	±0.0	±0.1	±0.2		
u4	Mean	n.d.	n.d.	< LOD	2.2	2.4	< LOD	n.d.	< LOD
	SD				±0.3	±0.1			
u6	Mean	n.d.	1.9	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.
	SD		±0.0						
u7	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Sum of Unid./Diff. Residues [A]	Mean	n.d.	1.9	1.5	2.2	2.4	< LOD	n.d.	< LOD
	SD		±0.0	±0.0	±0.3	±0.1			
Ambient Extract	Mean	91.2	31.5	23.9	21.5	17.9	13.4	8.4	6.5
	SD	±0.2	±0.1	±0.2	±0.2	±0.0	±0.2	±0.0	±0.2
Microwave Extract 1	Mean	6.0	5.2	3.8	3.3	2.5	2.0	1.1	0.8
	SD	±0.0	±0.1	±0.1	±0.1	±0.1	±0.1	±0.0	±0.0
Microwave Extract 2	Mean	0.2	0.2	0.3	0.2	0.1	0.2	0.1	0.2
	SD	±0.1	±0.0	±0.1	±0.1	±0.0	±0.0	±0.1	±0.0
Total Extractable Residues	Mean	97.3	36.9	28.0	25.0	20.5	15.7	9.7	7.4
	SD	±0.3	±0.1	±0.3	±0.1	±0.0	±0.2	±0.0	±0.2
Carbon Dioxide	Mean	n.a.	24.3	32.8	35.7	41	45.4	52.1	56.1
	SD		±0.4	±0.4	±0.4	±0.1	±1.0	±1.1	±0.0
Volatile Organic Compounds	Mean	n.a.	0.3	0.1	0.1	0.1	0.4	0.1	0.1
	SD		±0.3	±0.1	±0.0	±0.0	±0.4	±0.0	±0.0
Non-Extractable Residues	Mean	5.7	37.9	35.7	36.4	36.1	35.0	31.1	31.0
	SD	±0.3	±0.3	±0.8	±0.3	±0.1	±0.4	±0.1	±0.4
Total Recovery	Mean	103.1	99.3	96.7	97.2	97.7	96.5	93.0	94.6
	SD	±0.5	±0.4	±1.1	±0.1	±0.1	±1.2	±1.1	±0.2

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.2- 42: Degradation of [2,6-pyridyl-^{14}C]-M-02 in Dollendorf II soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)							
		0	3	7	10	16	28	70	120
M-02 (AE C657188)	Mean	98.1	15.9	4.1	2.5	< LOD	< LOD	< LOD	n.d.
	SD	±0.3	±0.2	±0.3	±0.0				
M-05 (AE 1344122)	Mean	n.d.	11.5	10.4	8.6	5.7	1.9	n.d.	n.d.
	SD		±0.1	±0.2	±0.2	±0.0	±0.2		
M-09 (AE B102859)	Mean	n.d.	8.6	9.4	9.5	10.5	8.3	5.4	3.6
	SD		±0.8	±0.1	±0.3	±1.3	±0.8	±0.1	±0.0
M-10 (AE 1344123)	Mean	n.d.	11.9	7.2	4.1	< LOD	< LOD	n.d.	n.d.
	SD		±0.3	±0.3	±0.3				
M-20 (BCS-BX16566)	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
u4	Mean	n.d.	n.d.	3.4	3.7	4.1	3.2	n.d.	n.d.
	SD			±0.2	±0.2	±0.0	±0.1		
u6	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
u7	Mean	n.d.	2.1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD		±0.3						
Sum of Unid./Diff. Residues [A]	Mean	n.d.	2.1	3.4	3.7	4.1	3.2	n.d.	n.d.
	SD		±0.3	±0.2	±0.2	±0.0	±0.1		
Ambient Extract	Mean	92.2	45.4	31.2	25.6	19.6	12.3	4.7	3.0
	SD	±0.6	±0.2	±0.4	±0.2	±0.3	±0.1	±0.1	±0.0
Microwave Extract 1	Mean	4.6	4.3	2.8	2.4	1.8	1.4	0.7	0.4
	SD	±0.3	±0.3	±0.1	±0.1	±0.0	±0.0	±0.0	±0.1
Microwave Extract 2	Mean	0.3	0.4	0.4	0.4	0.4	0.4	0.3	0.2
	SD	±0.0	±0.1	±0.0	±0.1	±0.0	±0.0	±0.0	±0.0
Total Extractable Residues	Mean	98.2	50.1	34.4	28.4	21.8	14.1	5.6	3.7
	SD	±0.4	±0.2	±0.4	±0.2	±0.3	±0.1	±0.1	±0.1
Carbon Dioxide	Mean	n.a.	8.6	21.1	28.8	38.3	45.8	58.0	64.0
	SD		±0.1	±0.4	±0.7	±0.8	±0.2	±0.3	±0.7
Volatile Organic Compounds	Mean	n.a.	0.1	< 0.1	1.8	0.3	< 0.1	0.1	0.4
	SD		±0.0	±0.0	±0.9	±0.3	±0.0	±0.0	±0.3
Non-Extractable Residues	Mean	5.8	38.5	39.5	38.9	36.8	33.4	27.9	26.5
	SD	±0.4	±0.0	±0.5	±0.0	±0.6	±0.3	±0.1	±0.1
Total Recovery	Mean	103.9	97.2	95.1	97.8	97.2	93.3	91.6	94.5
	SD	±0.1	±0.1	±0.5	±1.5	±1.5	±0.3	±0.4	±1.1

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.2- 43: Degradation of [2,6-pyridyl-^{14}C]-M-02 in L[redacted] [redacted] soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)							
		0	3	7	10	16	28	70	120
M-02 (AE C657188)	Mean	91.5	6.0	< LOD	< LOD	n.d.	n.d.	n.d.	< LOD
	SD	±0.8	±0.8						
M-05 (AE 1344122)	Mean	n.d.	7.6	5.7	4.0	2.7	1.6	n.d.	n.d.
	SD		±0.6	±0.1	±0.1	±0.0	±0.0		
M-09 (AE B102859)	Mean	n.d.	1.9	2.1	1.6	< LOD	< LOD	< LOD	< LOD
	SD		±0.0	±0.6	±0.1				
M-10 (AE 1344123)	Mean	n.d.	13.5	7.5	6.0	3.5	2.0	< LOD	< LOD
	SD		±2.6	±0.8	±0.1	±0.0	±0.4		
M-20 (BCS-BX16566)	Mean	n.d.	< LOD	1.9	1.7	< LOD	< LOD	n.d.	n.d.
	SD			±0.4	±0.2				
u4	Mean	n.d.	n.d.	2.2	2.5	1.8	1.5	n.d.	n.d.
	SD			±0.1	±0.3	±0.1	±0.1		
u6	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
u7	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Sum of Unid./Diff. Residues [A]	Mean	n.d.	n.d.	2.2	2.5	1.8	1.5	1.6	n.d.
	SD			±0.1	±0.3	±0.1	±0.1	±0.0	
Ambient Extract	Mean	82.0	26.0	17.2	13.6	9.0	5.2	2.5	1.9
	SD	±0.6	±0.4	±0.0	±0.0	±0.2	±0.0	±0.1	±0.2
Microwave Extract 1	Mean	8.1	4.0	2.6	2.2	1.6	0.9	0.5	0.3
	SD	±0.3	±0.2	±0.0	±0.2	±0.1	±0.0	±0.0	±0.0
Microwave Extract 2	Mean	0.4	0.3	0.3	0.3	0.1	0.3	0.2	0.1
	SD	±0.0	±0.1	±0.0	±0.1	±0.0	±0.1	±0.1	±0.0
Total Extractable Residues	Mean	91.5	30.3	20.1	16.0	10.7	6.3	3.1	2.4
	SD	±0.9	±0.3	±0.0	±0.2	±0.1	±0.1	±0.0	±0.2
Carbon Dioxide	Mean	n.a.	23.8	36.1	42.9	50.1	57.7	63.3	67.7
	SD		±0.6	±0.4	±0.0	±1.4	±0.4	±0.6	±1.0
Volatile Organic Compounds	Mean	n.a.	0.1	0.2	0.1	0.6	0.1	0.5	< 0.1
	SD		±0.0	±0.1	±0.0	±0.4	±0.0	±0.4	±0.0
Non-Extractable Residues	Mean	12.6	42.7	39.1	36.5	32.8	30	25.1	23.5
	SD	±0.8	±0.1	±1.3	±0.1	±0.1	±1.3	±0.3	±0.8
Total Recovery	Mean	104.1	96.9	95.5	95.5	94.2	94.2	92.0	93.6
	SD	±0.1	±0.8	±0.8	±0.3	±1.8	±1.8	±1.3	±0.0

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

Table 7.1.2.1.2- 44: Degradation of [2,6-pyridyl-^{14}C]-M-02 in H[redacted] soil under aerobic conditions at 20 °C [% AR]

Compound	Mean SD	Incubation time (DAT)							
		0	3	7	10	16	28	70	120
M-02 (AE C657188)	Mean	97.5	15.3	1.4	1.2	n.d.	n.d.	n.d.	n.d.
	SD	±0.4	±0.8	±0.0	±0.0				
M-05 (AE 1344122)	Mean	n.d.	5.8	7.6	5.5	3.3	1.2	n.d.	n.d.
	SD		±0.6	±0.0	±0.1	±0.0	±0.1		
M-09 (AE B102859)	Mean	n.d.	5.1	4.6	3.4	2.6	1.9	1.3	n.d.
	SD		±0.6	±0.1	±0.1	±0.1	±0.3	±0.0	
M-10 (AE 1344123)	Mean	n.d.	10.6	10.4	9.6	8.6	5.6	n.d.	n.d.
	SD		±0.8	±0.1	±0.0	±0.5	±0.1		
M-20 (BCS-BX16566)	Mean	n.d.	4.1	2.7	1.6	< LOD	n.d.	n.d.	n.d.
	SD		±0.2	±0.0	±0.1				
u4	Mean	n.d.	n.d.	2.2	3.1	3.1	2.3	n.d.	n.d.
	SD			±0.1	±0.0	±0.3	±0.1		
u6	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
u7	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Sum of Unid./Diff. Residues [A]	Mean	n.d.	n.d.	2.2	3.1	3.1	2.3	1.3	1.8
	SD			±0.1	±0.0	±0.3	±0.1	±0.0	±0.2
Ambient Extract	Mean	90.9	35.9	24.4	21.4	16.2	9.4	2.0	1.3
	SD	±0.4	±0.1	±0.2	±0.2	±0.0	±0.1	±0.1	±0.1
Microwave Extract 1	Mean	6.4	4.6	3.5	2.7	2.2	1.4	0.5	0.4
	SD	±0.0	±0.1	±0.2	±0.0	±0.0	±0.0	±0.0	±0.1
Microwave Extract 2	Mean	0.2	0.4	1.0	0.3	0.2	0.3	0.2	0.2
	SD	±0.1	±0.1	±0.4	±0.0	±0.0	±0.1	±0.0	±0.1
Total Extractable Residues	Mean	97.5	40.8	28.9	24.4	18.6	11.0	2.6	1.8
	SD	±0.4	±0.3	±0.4	±0.2	±0.1	±0.1	±0.1	±0.3
Carbon Dioxide	Mean	n.a.	14.5	26.5	29.4	37.0	46.6	59.4	65.9
	SD		±0.1	±0.3	±0.3	±0.2	±1.1	±0.7	±0.2
Volatile Organic Compounds	Mean	n.a.	0.1	0.1	< 0.1	0.1	0.5	0.5	0.4
	SD		±0.0	±0.0	±0.0	±0.0	±0.4	±0.3	±0.3
Non-Extractable Residues	Mean	6.6	42.3	41.5	41.3	38.5	36.5	30.3	28.3
	SD	±0.2	±0.3	±0.4	±0.6	±0.2	±0.9	±0.9	±0.8
Total Recovery	Mean	104.1	97.8	97.0	95.2	94.1	94.6	92.8	96.4
	SD	±0.2	±0.4	±1.2	±0.4	±0.4	±2.3	±1.9	±0.1

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
All values expressed as percentage of total applied radiolabel
[A] Sum of minor unidentified components and diffuse residues.

B. Material Balance

Mean mass balances were 97.3% AR for L[redacted] soil (range from 93.0 to 103.0% AR), 96.3% AR for Dollendorf II soil (range from 91.6 to 103.9% AR), 95.7% AR for L[redacted] soil (range from 92.0 to 104.1% AR) and 96.5% AR for H[redacted] soil (range from 92.8 to 104.1% AR).

C. Extractable and Non-Extractable Residues

Values of extractable radioactivity decreased from 91.5 to 98.1% of applied at zero time to 7.4% (L[redacted] soil), 3.6% (Dollendorf II soil), 2.4% (L[redacted] soil) or 1.8% (H[redacted] soil) by the end of the study (DAT 120).

In all of the soils non-extractable residues (NER) increased rapidly peaking at DAT 3 (or DAT 7 in Dollendorf II soil) and then declining gradually by DAT 120. NER increased in L[redacted] soil from DAT 0 to DAT 3 from 5.7 to 37.9% AR and then decreased slightly to 31.0% AR by DAT 120. In Dollendorf II soil, NER increased from DAT 0 to DAT 7 from 5.9 to 39.5% AR and then decreased to 26.5% AR by DAT 120. NER increased in L[redacted] soil from DAT 0 to DAT 3 from 12.6 to 42.7% AR and then decreased to 23.5% AR by DAT 120. In H[redacted] soil NER increased from DAT 0 to DAT 3 from 6.6 to 42.3% AR and then decreased to 28.3% AR at DAT 120.

The distribution of the NER in different humic substance fractions at DAT 120 is shown in the table below.

Table 7.1.2.1.2- 45: Humic substance fractionation (as % applied radioactivity)

Soil	Humin fraction [% AR]	Humic acid fraction [% AR]	Fulvic acid fraction [% AR]	Total [% AR]
L[redacted]	9.0	7.5	15.3	31.8
Dollendorf II	15.1	5.9	5.1	26.0
L[redacted]	9.2	6.4	7.9	23.5
H[redacted]	11.0	5.6	10.5	27.2

D. Volatile Radioactivity

There was a rapid increase in the carbon dioxide production from the start of the study, reaching a maximum of 56.1, 64.0, 67.7 and 65.9% by DAT 120 in L[redacted], Dollendorf II, [redacted] and H[redacted] soils. Formation of volatile organic compounds (VOC) was not significant at ≤ 1.8% AR at all sampling intervals for all soils.

E. Transformation of test substance

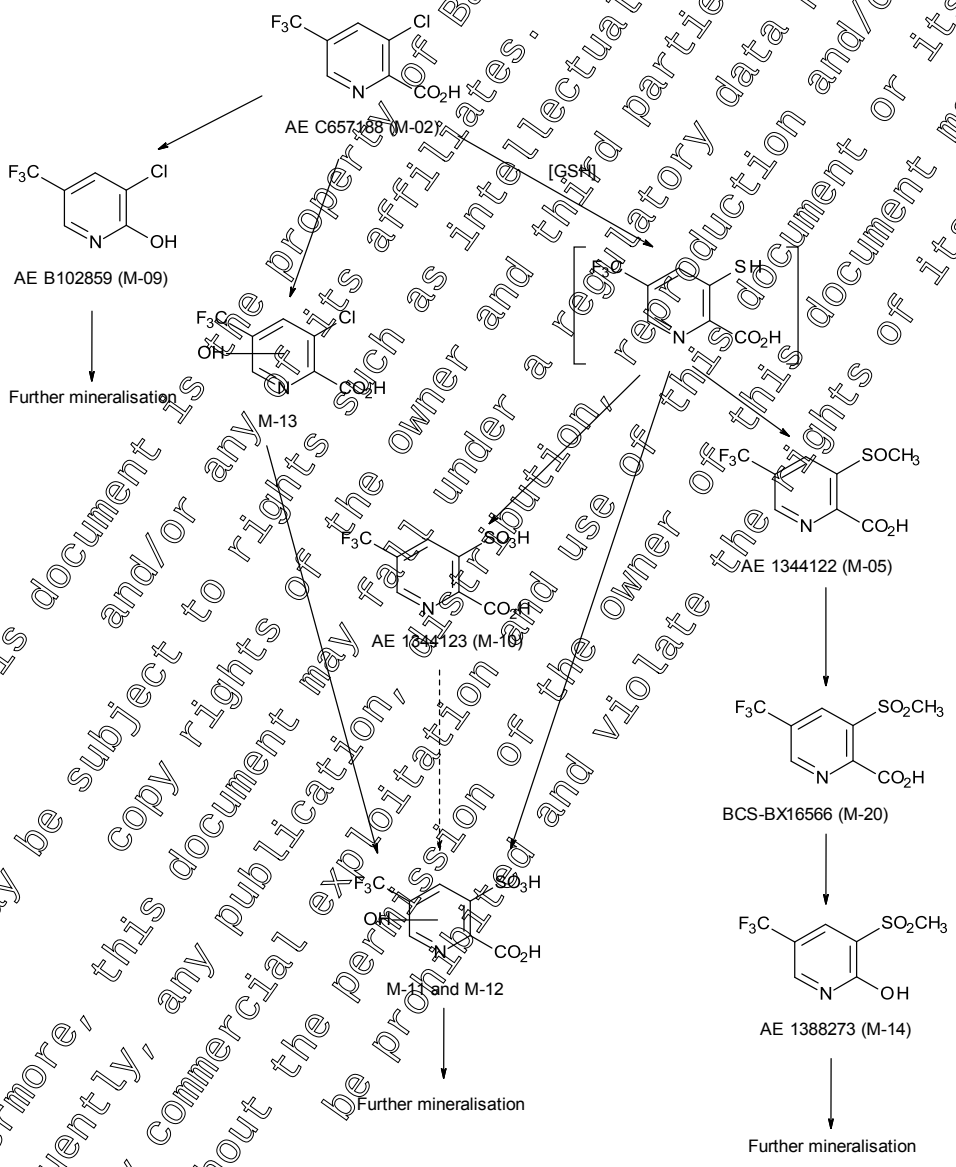
M-02 was very rapidly degraded in the soils to a number of metabolites, and despite exhaustive extraction of soil samples rapidly formed significant quantities of NER. The levels of M-02 declined from 97.3 and 91.5% AR at DAT 0 to below the LOD (< 1.1% AR) by DAT 7 in L[redacted] and L[redacted] soils, and from 98.1 and 97.5% AR at DAT 0 to below the LOD by DAT 16 in Dollendorf II and H[redacted] soils.

Degradation of M-02 was accompanied by the formation of four metabolites M-05, M-09, M-10 and M-20, along with a further three unidentified degradation products. M-05 was detected in all 4 soils and found at a maximum of 11.5% AR at DAT 3 in Dollendorf II soil. M-09 was also detected in all 4 soils and formed at a maximum of 10.5% AR at DAT 16 in Dollendorf II soil. M-10 was identified in all 4 soils at a maximum of 18.4% AR at DAT 3 in L[redacted] soil. M-20 was identified in 3 soils at a maximum of 4.7%AR at DAT 10 in L[redacted] soil. The metabolites M-05 and M-10 had

previously been identified in leachate from a lysimeter study conducted with [2,6-pyridyl-^{14}C]- labelled fluopicolide [see KCA7.1.4.2/01, M-218465-01-1]. The metabolite M-20 has a molecular weight of 269 g/mole and was previously observed in an aerobic soil study conducted with [2,6-pyridyl-^{14}C]- labelled M-05 [see KCA 7.1.2.1.2/04, M-241410-01-2], where it was not fully identified but its molecular weight was established as 269 g/mole. The metabolite M-09 had not been observed in soil or leachate before but was identified in crops from a confined crop rotation study following soil application with [2,6-pyridyl-^{14}C]- labelled fluopicolide [see KCA 6.6.1/01, M-240707-03-1] as were M-02 and M-05. The unidentified degradation products u4, u6 and u7 were observed at maxima of 4.1, 1.9 and 2.1% AR respectively.

The proposed route of degradation of M-02 in aerobic soil is presented in Figure 7.1.2.1.2-3.

Figure 7.1.2.1.2- 3: Proposed metabolic pathway for M-02 in aerobic soil



M-11/M-12, M-13 and M-14 were observed in an earlier aerobic soil study (KCA 7.1.2.1.2/03, M-219824-01-1) treated with M-02 (AE C657188) and previously identified in leachate from a lysimeter study conducted with [2,6-pyridyl-^{14}C]-labelled fluopicolide (KCA 7.1.4.2/01, M-218465-01-1)

F. Degradation Kinetics

Reported DT_{50} values of M-02 under aerobic conditions were 0.67, 0.85, 0.73 and 1.01 days in L[redacted], Dollendorf II, L[redacted] and H[redacted] soils, respectively. The experimental data were best described by either a simple first order (SFO) or a first order multi compartment (FOMC) kinetic model. Details are provided below in Table 7.1.2.1.2-46.

Table 7.1.2.1.2- 46: Reported degradation rate of M-02 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} (days)	DT_{90} (days)
[redacted]	SFO	103.0	k 1.03861	0.7	1.28e-05	-	-	0.67	2.22
Dollendorf II	FOMC	103.9	α 2.4525 β 2.6091	0.7	-	2.1712 2.1587	2.734 3.060	0.85	4.06
[redacted]	SFO	104.1	k 0.9473	0.6	1.86e-06	-	-	0.73	2.43
H[redacted]	FOMC	104.1	α 9.7084 β 13.6868	1.7	-	-2.1993 4.9493	21.62 32.32	1.01	3.66

In addition, the experimental data for the degradation of M-02 and its further metabolites has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting DT_{50} values for trigger endpoints for M-02 are summarised below in Table 7.1.2.1.2- 47. Best fit kinetics are highlighted in bold.

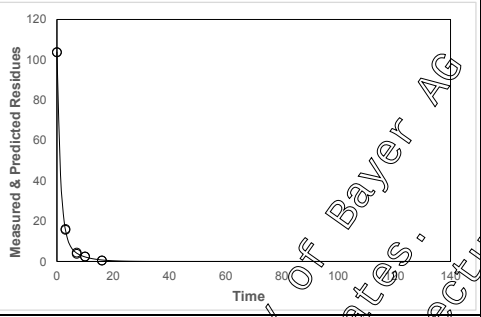
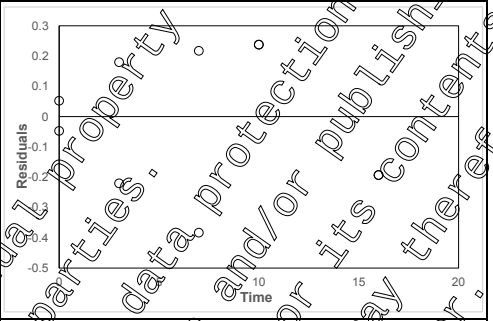
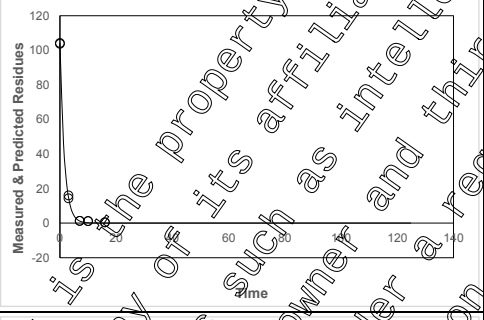
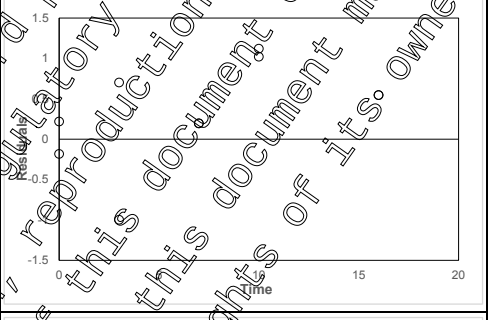
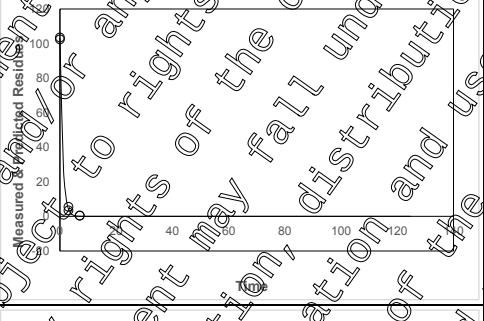
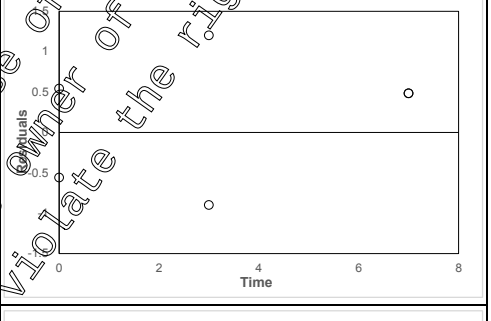
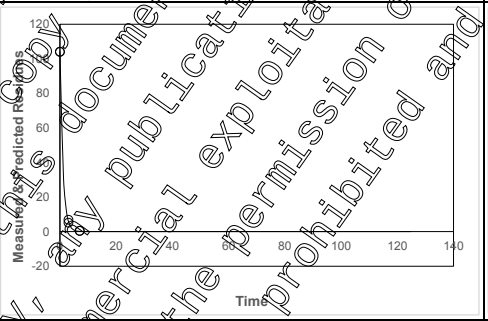
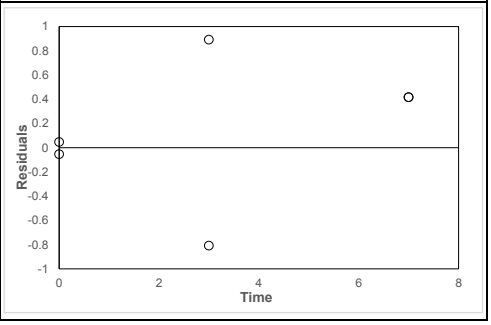
Table 7.1.2.1.2- 47: Re-evaluation of degradation rate of M-02 under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Dollendorf II, [redacted] (2017)	SFO	103.7	k 0.6056	5.1	<2e-16	0.5658	0.645	1.1	3.8
	FOMC	103.8	α 2.494 β 2.678	0.736	n.r. n.r.	2.21 2.223	2.778 3.134	0.9	4.1
	DFOP	**103.8**	**k1 0.8133 k2 0.1837 g 0.8648**	**0.639**	**<2e-16 2.32E-08 n.r.**	**0.7319 0.1351 0.8056**	**0.895 0.232 0.924**	**1**	**4**
H[redacted] (2017)	**SFO**	**104.1**	**k 0.637**	**1.79**	**<2e-16**	**0.6172**	**0.657**	**1.1**	**3.6**
	FOMC	104.1	α 10.85 β 15.51	1.71	n.r. n.r.	-2.618 -5.618	24.31 36.63	1	3.7
L[redacted] (2017)	**SFO**	**103.1**	**k 1.051**	**0.719**	**<2e-16**	**0.9643**	**1.138**	**0.7**	**2.2**
	FOMC	103.1	α 4.003 β 2.504	NaN	n.r. n.r.	-4.513 -5.239	12.52 10.25	0.5	1.9
L[redacted] (2017)	**SFO**	**104.2**	**k 0.9509**	**0.578**	**<2e-16**	**0.9026**	**0.999**	**0.7**	**2.4**
	FOMC	104.2	α 5.202 β 4.103	NaN	n.r. n.r.	-2.89 -4.23	13.29 12.44	0.6	2.3

Best fit kinetics are highlighted in bold
NaN – Not a number (value not calculated by KinGUI)

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.2- 48: Degradation of M-02 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Dollendorf II DFOP [redacted] (2017)		
H[redacted] SFO [redacted] (2017)		
L[redacted] SFO [redacted] (2017)		
L[redacted] SFO [redacted] (2017)		

III. Conclusion

M-02 is a minor metabolite of fluopicolide which was observed in laboratory soil metabolism studies conducted with the parent and reached a maximum of 7.3% of applied radioactivity. M-02 was very rapidly degraded in soil to a number of pyridyl ring metabolites, with a significant portion completely mineralized to CO_2 and, thus, it would not be expected to persist in the soil environment. Best fit DT_{50} values ranged from 0.67 to 1.01 days in the tested soils. Re-evaluated best fit DT_{50} values were similar ranging from 0.7 to 1.1 days.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002). The study is considered valid to assess the aerobic degradation of [2,6-^{14}C pyridyl-^{14}C] M-02 in soil.

Data Point:	KCA 7.1.2.1.2/10
Report Author:	[redacted]
Report Year:	2016
Report Title:	AE0608000: Aerobic degradation in two soils
Report No:	S15-04154
Document No:	M-565219-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307, 2002; SANCO/3029/99 rev.4
Deviations from current test guideline:	none
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-03 (AE0608000) was studied in two acidic soils under aerobic conditions in the laboratory in the dark at 20 ± 2 °C and 55% of the maximum water holding capacity for up to 16 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
Brierlow	silt loam	5.3	4.5
H[redacted]	silt loam	6.0	1.8

M-03 was applied to soil samples at an application rate of 0.040 mg/kg dry weight, reflecting the maximum single field application rate of 133 g/ha for parent fluopicolide and the maximum occurrence of this metabolite. The chemical purity of M-03 was 96.9% which was considered in determining the application rate.

Samples were removed for extraction and analysis immediately after treatment (day 0) and 0.17, 1, 2, 3, 4, 7 and 16 days (Brierlow only) of incubation. Soil samples were extracted at ambient temperature three times with acetonitrile:water (4:1, v/v) with 1% formic acid and then by microwave extraction with acetonitrile:water (4:1, v/v) with 1% formic acid at 60 °C. Soil extracts were analysed by HPLC-MS/MS to quantify the amount M-03 remaining.

M-03 was rapidly degraded in both acidic soils. The amount of the metabolite in soil extracts declined from 0.04073 mg/kg at time zero to 0.00318 mg/kg by DAT 16, equivalent to 8% of applied, in Brierlow soil, and from 0.03887 mg/kg at time zero to 0.0014 by DAT 7, equivalent to 3.5% of applied in H[redacted] soil.

The following DT_{50} and DT_{90} values were calculated for M-03 in the two soils.

Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)	Visual Assessment
Brierlow (silt loam)	SFO	2.5	8.4	9.5	Moderate
H[redacted] (silt loam)	SFO	0.9	2.9	8.0	Good

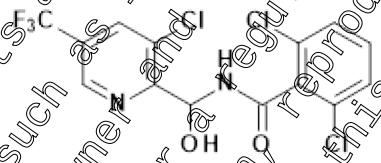
Degradation of the metabolite M-03 in aerobic acidic soils was very rapid and it is not expected to persist in the soil environment.

I. Materials and Methods

A. Materials

1. Test Item

M-03 (referred to as AE 0608000 in the report)



Chemical Purity: 96.9%

Sample/Batch ID: MOY4622M

Expiry Date: 06 May 2017

2. Test Soils

The study was performed using two acidic soils as characterized in Table 7.1.2.1.2- 49. Soils were collected fresh from the field and used with minimal storage time (27 to 70 days) prior to sieving to 2 mm and dispersing into flasks.

Table 7.1.2.1.2- 49: Physico-chemical properties of test soils

Parameter		Soil			
Soil Designation		Brierlow		H[redacted]	
Soil ID		BL		[redacted]	
Geographic Location					
City		Brierlow, Derbyshire		Burscheid,	
Country		United Kingdom		Germany	
GPS coordinates		[redacted]		[redacted]	
Batch Number		20151201		20160113	
Textural Classification (USDA)		Silt loam		Silt loam	
Sand [50 - 2000 µm]	(%)	31		22	
Silt [2 – 50 µm]	(%)	58		64	
Clay [< 2 µm]	(%)	11		14	
pH					
in $CaCl_2$ (1:2)		5.3		6.0	
in H_2O (1:1)		5.5		6.3	
Saturated paste		5.5		6.3	
in KCl (1:1)		5.0		5.8	
Organic Matter (%) *		7.8		3.4	
Organic Carbon (%)		4.5		2.0	
Cation Exchange Capacity (meq/100 g)		12.4		11.5	
Water Holding Capacity					
Maximum (g H_2O per 100 g DW)		75.1		62.2	
at 1/3 bar (%)		29.5		22.0	
Moisture Content During Incubation (%)		55% MWHC		55% MWHC	
Bulk Density (disturbed; g/cm³)		0.96		1.03	
Soil Microbial Biomass (mg microbial C /100 g soil)		BIO⁻	BIO⁺	BIO⁻	BIO⁺
Arrival		251		261.6	
Initial (DAT 0)		173.5		262.6	
Final (DAT 21)		163.8	162.3	231.9	245.5

* Calculated by multiplying organic carbon content by 1.724
MWHC = Maximum Water Holding Capacity
BIO⁻ samples were untreated
BIO⁺ samples were treated with 200 µL of acetonitrile:water (4:1, v/v) containing 0.5% formic acid

B. Study Design

1. Experimental Conditions

Tests were performed in static systems consisting of a glass incubation flask containing 100 g soil (dry weight equivalents) stoppered with cotton wool to maintain aerobic conditions and to minimise water loss.

M-03 was applied at an application rate of 0.040 mg/kg, reflecting the maximum single field application rate of 133 g/ha for parent fluopicolide and the maximum occurrence of this metabolite. The test item was dissolved in acetonitrile:water (4:1, v/v) containing 0.5% formic acid and applied dropwise onto the soil surface. Soil samples were adjusted to a moisture content equivalent to 55% ± 5% of MWHC 3 days prior to application. The samples were incubated at 20 ± 2 °C under aerobic conditions in the dark for up to 16 days after application. Soil moisture was maintained during incubation by addition of water to the samples. Untreated soil samples were incubated under the same conditions for determination of soil microbial activity. Additional untreated flasks containing 100g (dry weight) equivalent of each soil were used to provide fortification samples to confirm the analytical method efficiency. At each sampling interval a flask of each soil was fortified at the LOQ level (0.00040 mg/kg, 10% of the application rate) and at 110 times the LOQ (0.044 mg/kg, 1100% of the application rate).

2. Sampling

Duplicate samples were taken for analysis after 0, 0.17, 1, 2, 3, 4, 7 and 16 days of incubation in Brierlow soil and 0, 0.17, 1, 2, 3, 4 and 7 days of incubation in H[redacted] soil. Microbial soil biomass samples were analysed on arrival and at the start and end of the experiment (DAT 0 and 21).

3. Analytical procedures

The entire soil sample of each test vessel was extracted three times with a mixture of acetonitrile:water (4:1, v/v) with 1% formic acid at ambient temperature. Ambient extraction was followed by an additional microwave extraction with acetonitrile:water (4:1, v/v) with 1% formic acid at 60°C. All extracts were combined and an aliquot centrifuged prior to analysis. Identification of M-03 in soil extracts was by HPLC-MS/MS. M-03 was quantified using the transition of m/z 399.0 to m/z 173.0 and in addition by the transition m/z 399.0 to m/z 109.1. Matrix-matched calibration curves were used for the quantification of M-03. The analytical method was validated with both soils prior to starting the test.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The degradation of M-03 followed single first order (SFO) kinetics in Brierlow and H[redacted] soils based on lowest chi^2 error values and visual assessments of fits.

Additionally, modelling endpoints for the degradation of M-03 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-03, an initial comparison was performed for each soil between the SFO and FOMC fits. The SFO model provided a better fit for both soils, with a lower χ^2 err% value, and was therefore accepted.

II. Results and Discussion

A. Analytical Methodology:

A full summary of the analytical method is provided in Document MCA 4, Section 4.1.2. The method complies with all criteria according to *SANCO/3029/99 rev. 4* and is suitable for the determination of M-03 in soil samples by HPLC-MS/MS.

B. Data:

M-03 was rapidly degraded in both soils. The amount of the metabolite in soil extracts declined from 0.04073 mg/kg at time zero to 0.00318 mg/kg by DAT 16, equivalent to 8% of applied, in Brierlow soil, and from 0.03887 mg/kg at time zero to 0.0014 by DAT 7, equivalent to 3.5% of applied in H[redacted] soil.

The results for each soil are summarized in Table 7.1.2.1.2- 50 to Table 7.1.2.1.2- 51.

Table 7.1.2.1.2- 50: Degradation of M-03 in Brierlow soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	0.17	1	2	3	4	7	16
Mean	mg/kg	0.04073	0.03922	0.03485	0.02842	0.01538	0.01063	0.00776	0.00318
SD	mg/kg	0.00061	0.00051	0.00118	0.00057	0.00074	0.00202	0.00041	0.00005
Mean	%	101.8	98.1	87.1	71.1	38.5	26.7	17.9	8.0
RSD	%	1.5	1.3	3.4	2.1	4.8	19.1	5.5	0.9

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

Table 7.1.2.1.2- 51: Degradation of M-03 in H[redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)						
		0	0.17	1	2	3	4	7
Mean	mg/kg	0.03887	0.03783	0.02003	0.00643	0.00282	0.00211	0.0014
SD	mg/kg	0.00004	0.00006	0.00055	0.00023	0.00016	0.0001	0.00001
Mean	%	97.2	94.6	50.1	16.1	7.1	5.3	3.5
RSD	%	0.1	0.1	2.8	3.5	5.0	5.3	0.0

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

F. Degradation Kinetics

Best fit reported DT_{50} values of M-03 under aerobic conditions were 2.5 and 0.9 days in Brierlow and H[redacted] soils, respectively. The experimental data were best described by a simple first order (SFO) kinetic model in both soils. Details are provided below in Table 7.1.2.1.2- 52.

Table 7.1.2.1.2- 52: Reported degradation rate of M-03 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} (days)	DT_{90} (days)
Brierlow	SFO	0.04213	k 0.27557	9.5	1.14-09	0.23522	0.316	2.5	8.4
H[redacted]	SFO	0.0409238	k 0.7929048	8.0	3.39e-11	0.7198577	0.866	0.9	8.0

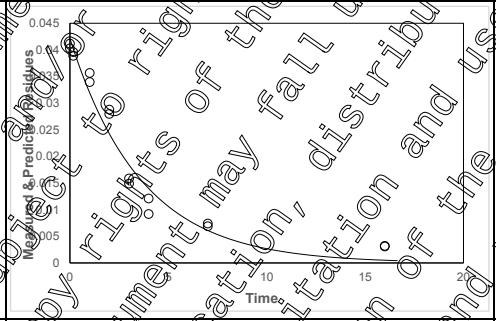
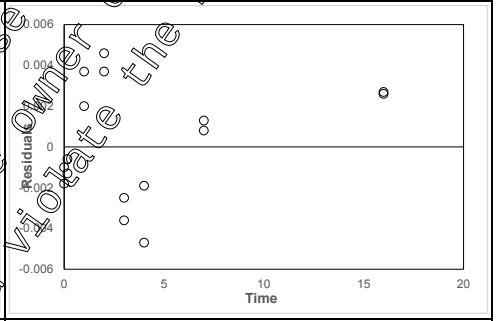
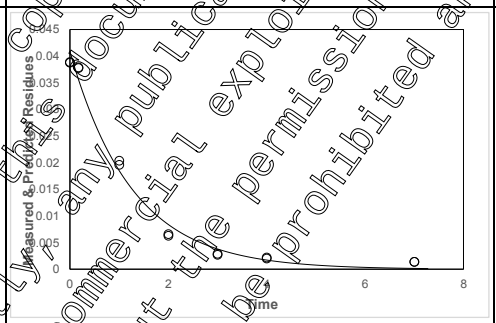
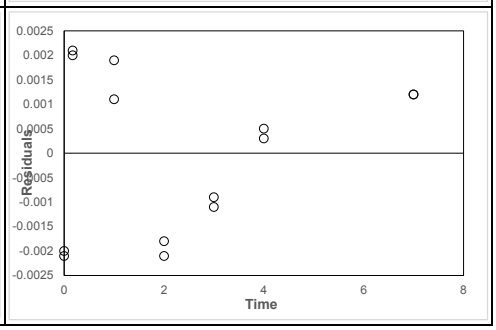
In addition, the experimental data for the degradation of M-03 been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.2/10. The resulting DT_{50} values for trigger endpoints for M-03 are summarised below in Table 7.1.2.1.2- 53. Best fit kinetics are highlighted in bold (and are the same as the reported values).

Table 7.1.2.1.2- 53: Re-evaluation of degradation rate of M-03 under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2 %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Brierlow (BL), [redacted] (2016a)	**SFO**	**0.04213**	**k 0.2757**	**9.46**	**6.21E-09**	**0.2297**	**0.322**	**2.5**	**8.4**
	FOMC	0.04213	α 4211 β 15270	10.1	n.r. n.r.	4211 15270	4211 15270	2.5	8.4
H[redacted] [redacted] (2016a)	**SFO**	**0.04089**	**k 0.7914**	**7.98**	**4.53E-10**	**0.7003**	**0.882**	**0.9**	**2.9**
	FOMC	0.04089	α 9949 β 12570	8.62	n.r. n.r.	9949 12570	9949 12570	0.9	2.9

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.2- 54: Degradation of M-03 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
Brierlow (BL) SFO [redacted] (2016a)		
H[redacted] SFO [redacted] (2016a)		

III. Conclusion

M-03 is a major metabolite of fluopicolide formed in soil from degradation of the parent compound. M-03 degraded rapidly in acidic soils under aerobic conditions with best-fit DT_{50} SFO values of between 2.5 and 0.9 days for Brierlow and H[redacted] soils.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002). The study is considered valid to assess the aerobic degradation of M-03 in acidic soils.

Data Point:	KCA 7.1.2.1.2/11
Report Author:	[redacted]
Report Year:	2016
Report Title:	AE1344122: Aerobic degradation in three soils
Report No:	S15-04140
Document No:	M-565223-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307, 2002; SANCO/3029/99 rev.4
Deviations from current test guideline:	none
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-05 (AE 1344122) was studied in three soils under aerobic conditions in the laboratory in the dark at 20 ± 2 °C and 55% of the maximum water holding capacity for up to 120 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
L[redacted]	loamy sand	5.3	1.5
L[redacted]	sandy loam	5.1	1.9
H[redacted]	silt loam	5.8	1.9

M-05 was applied to soil samples at an application rate of 0.026 mg/kg dry weight, equivalent to a field application rate of 133 g/ha of fluopicolide and a highly conservative estimate of the maximum occurrence of this metabolite. The chemical purity of M-05 was 98.8% which was considered in determining the application rate.

Samples were removed for extraction and analysis immediately after treatment (day 0) and 1, 3, 6, 15, 30, 59 and 120 days of incubation. Soil samples were extracted at ambient temperature three times with acetonitrile/0.05 M ammonium carbonate (80:20, v/v)and then by microwave extraction with acetonitrile/0.05 M ammonium carbonate (80:20, v/v) at 60 °C. Soil extracts were analysed by HPLC-MS/MS to quantify the amount M-05 remaining.

M-05 was readily degraded in all three soils. The amount in soil extracts declined from between 0.0249 to 0.0253 mg/kg at time zero to 0.0005 to 0.0021 mg/kg by DAT 120, equivalent to 4.2%, 8.1% and 1.8% of applied in L[redacted], Wurmwiese and H[redacted] soils, respectively.

The following DT_{50} and DT_{90} values were calculated for M-05 in the three soils.

Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)	Visual Assessment
L[redacted] (loamy sand)	SFO	16.8	55.9	4.8	Moderate
L[redacted] (sandy loam)	SFO	19.0	63.2	6.6	Moderate
H[redacted] (silt loam)	SFO	22.5	74.6	3.6	Good

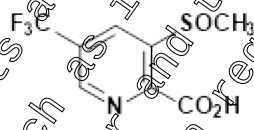
Degradation of the metabolite M-05 in aerobic acidic soils was relatively rapid and it is not expected to persist in the soil environment.

I. Materials and Methods

A. Materials

1. Test Item

M-05 (referred to as AE 1344122 in the report)



Chemical Purity: 98.8 % w/w

Sample/Batch ID: AE 1344122.00 1B99 0001, YG3228

Expiry Date: 26 January 2016

2. Test Soils

The study was performed using three German soils as characterized in Table 7.1.2.1.2- 55. The same soils were also used in laboratory aerobic soil studies with fluopicolide. Soils were collected fresh from the field and used with minimal storage time (49 days) prior to sieving to 2 mm and dispersing into flasks.

Table 7.1.2.1.2- 55: Physico-chemical properties of test soils

Parameter		Soil					
Soil Designation		L[redacted]		L[redacted]		H[redacted]	
Soil ID		[redacted]		[redacted]		[redacted]	
Geographic Location							
City		Monheim		Monheim		Burscheid	
Country		Germany		Germany		Germany	
GPS coordinates		[redacted]		[redacted]		[redacted]	
Batch Number		20150811-A		20150811-A		20150811-A	
Textural Classification (USDA)		Loamy sand		Sandy loam		Silt loam	
Sand [50 - 2000 µm]	(%)	78		58		26	
Silt [2 – 50 µm]	(%)	16		30		60	
Clay [< 2 µm]	(%)	6		12		14	
pH							
in $CaCl_2$ (1:2)		5.3		5.1		5.8	
in H_2O (1:1)		5.5		5.3		5.9	
Saturated paste		5.5		5.3		5.9	
in KCl (1:1)		5.0		4.8		5.5	
Organic Matter (%) *		2.6		3.3		3.3	
Organic Carbon (%)		1.5		1.9		1.9	
Cation Exchange Capacity (meq/100 g)		8.2		10.6		12.2	
Water Holding Capacity							
Maximum (g H_2O per 100 g DW)		31.8		55.5		58.3	
at 1/3 bar (%)		10.8		15.8		20.7	
Moisture Content During Incubation (%)		55% MWHC		55% MWHC		55% MWHC	
Bulk Density (disturbed, g/cm³)		1.22		1.13		1.08	
Soil Microbial Biomass (mg microbial C /100 g soil)		BIO^-	BIO^+	BIO^-	BIO^+	BIO^-	BIO^+
Initial (DAT 1)		250.1		244.7		229.7	
Mid (DAT 29)		189.1	158.5	215.6	222.9	233.6	302.3
Final (DAT 122)		148.7	113.6	190.7	181.2	191.0	256.8

* Calculated by multiplying organic carbon content by 1.724
MWHC = Maximum Water Holding Capacity
BIO^- samples were untreated
BIO^+ samples were treated with 400 µL of methanol:water (1:1, v/v)

B. Study Design

1. Experimental Conditions

Samples of 100 g dry weight of soil each were filled into glass incubation flasks and pre-equilibrated for 4 days prior to treatment at approximate study conditions (darkness, 20 ± 2 °C, soil moisture content equivalent to 55 ± 5% of maximum water holding capacity (MWHC)).

At the start of the test, each sample received 0.026 mg test substance/kg soil reflecting the maximum single field application rate of 133 g/ha of fluopicolide and a highly conservative formation level for the metabolite.

Samples were incubated and maintained at 20 ± 2 °C and 55 ± 5% of MWHC in the dark for a maximum of 120 days. Soil samples were maintained under static conditions. All the flasks were stoppered with cotton wool. Untreated soil samples were incubated under the same conditions for determination of soil microbial activity. Additional untreated flasks containing 100g (dry weight) equivalent soil [redacted] were used to provide fortification samples to confirm the analytical method efficiency. At each sampling interval two flasks were fortified at the LOQ level (0.0013 mg/kg, 5% of the application rate) and two flasks at 22 times the LOQ (0.0286 mg/kg, 110% of the application rate).

2. Sampling

Duplicate samples were taken for analysis after 0, 1, 3, 6, 15, 30, 59 and 120 days of incubation. Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment (DAT 0, 29 and 122).

3. Analytical procedures

The entire soil sample of each test vessel was extracted three times with a mixture of acetonitrile:0.05 M ammonium carbonate (80:20, v/v) at ambient temperature. Ambient extraction was followed by an additional microwave extraction with acetonitrile:0.05 M ammonium carbonate (80:20, v/v) at 60°C. All extracts were combined and an aliquot centrifuged prior to analysis. Identification of M-05 in soil extracts was by HPLC-MS/MS. M-05 was quantified using the transition of m/z 252.0 to m/z 61.0 and in addition by the transition m/z 252.0 to m/z 146.0. Matrix-matched calibration curves were used for the quantification of M-05. The analytical method was validated with all three soils prior to starting the test.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The degradation of M-05 followed single first order (SFO) kinetics in L[redacted], H[redacted] and L[redacted] soils based on lowest chi^2 error values and visual assessments of fits.

Additionally, modelling endpoints for the degradation of M-05 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-05, an initial comparison was performed for each soil between the SFO and FOMC fits. The FOMC fits did not provide a significant improvement on the SFO fits, and the SFO model was therefore accepted for all soils.

II. Results and Discussion

A. Analytical Methodology:

A full summary of the analytical method is provided in Document MCA 4, Section 4.1.2. The method complies with all criteria according to *SANCO/3029/99 rev. 4* and is suitable for the determination of M-05 in soil samples by HPLC-MS/MS.

B. Data:

M-05 was readily degraded in all three soils. The amount in soil extracts declined from between 0.0249 to 0.0253 mg/kg at time zero to 0.0005 to 0.0021 mg/kg by DAT 120, equivalent to 4.2%, 8.1% and 1.8% of applied in L[redacted], L[redacted], and H[redacted] soils, respectively.

The results for each soil are summarized in Table 7.1.2.1.2- 56 to Table 7.1.2.1.2- 58.

Table 7.1.2.1.2- 56: Degradation of M-05 in L[redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	1	3	6	15	30	59	120
Mean	mg/kg	0.0252	0.025	0.0243	0.0208	0.0124	0.0079	0.003	0.0011
SD	mg/kg	0.0002	0.0003	0.0004	0.0005	0.001	0.001	0.0006	0.0003
Mean	%	96.9	96.2	93.5	79.8	47.8	30.4	11.5	4.2
RSD	%	0.9	1.3	1.4	2.3	9.2	12.6	20.9	25.6

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

Table 7.1.2.1.2- 57: Degradation of M-05 in L[redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	1	3	6	15	30	59	120
Mean	mg/kg	0.0249	0.0239	0.0240	0.0224	0.0129	0.0085	0.0035	0.0021
SD	mg/kg	0.0003	0.0003	0.0003	0.0004	0.0004	0.0009	0.0006	0.0003
Mean	%	95.7	91.9	92.4	86.2	49.5	32.7	13.4	8.1
RSD	%	1.1	1.4	1.1	1.9	3.4	11.0	17.3	14.0

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

Table 7.1.2.1.2- 58: Degradation of M-05 in H[redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	1	3	6	15	30	59	120
Mean	mg/kg	0.0253	0.0238	0.0238	0.0217	0.0165	0.0107	0.0027	0.0005
SD	mg/kg	0.0003	0.0001	0.0005	0.002	0.0007	0.0004	0.0003	0.0001
Mean	%	97.2	91.4	91.5	83.4	63.4	41.0	10.5	1.8
RSD	%	1.1	0.3	1.9	9.2	4.6	4.1	11.4	31.4

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

F. Degradation Kinetics

Best fit reported DT_{50} values of M-05 under aerobic conditions were 16.8, 19.0 and 22.5 days in L[redacted], L[redacted] and H[redacted] soils, respectively. The experimental data were best described by a simple first order (SFO) kinetic model in each soil. Details are provided below in Table 7.1.2.1.2- 59.

Table 7.1.2.1.2- 59: Reported degradation rate of M-05 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} (days)	DT_{90} (days)
L[redacted]	SFO	0.0260182	k 0.0411709	4.8	1.99e-10	0.0358906	0.046	16.8	55.9
L[redacted]	SFO	0.0255707	k 0.0364451	6.6	5.72e-09	0.0303984	0.042	19.0	63.2
H[redacted]	SFO	0.0255028	k 0.0308704	3.6	3.77e-11	0.0273779	0.034	22.5	74.6

In addition, the experimental data for the degradation of M-05 been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting DT_{50} values for trigger endpoints for M-15 are summarised below in Table 7.1.2.1.2- 60. Best fit kinetics are highlighted in bold (and are the same as the reported values).

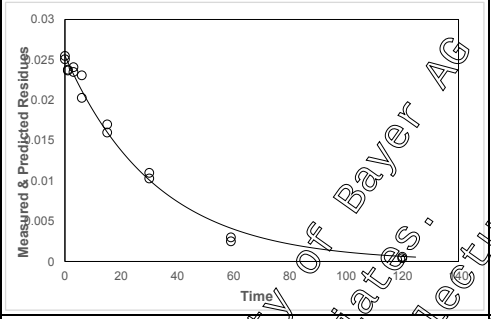
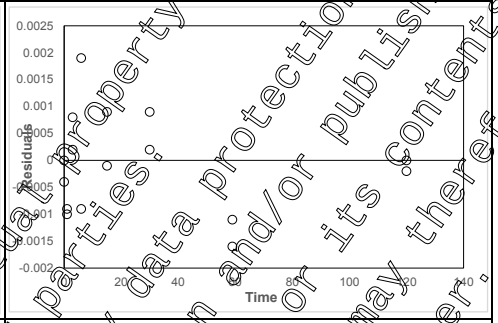
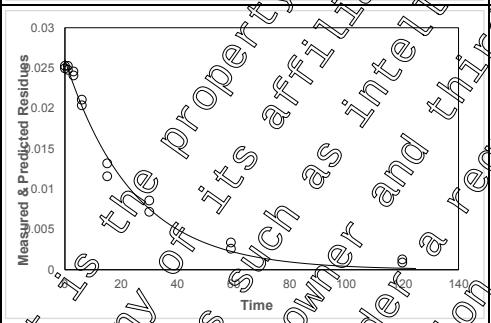
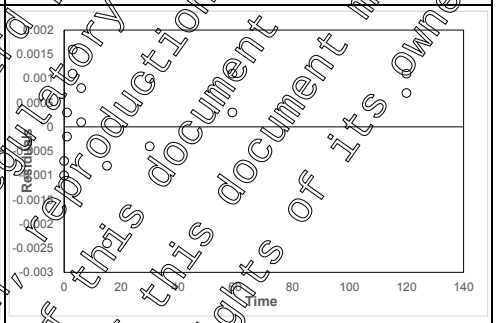
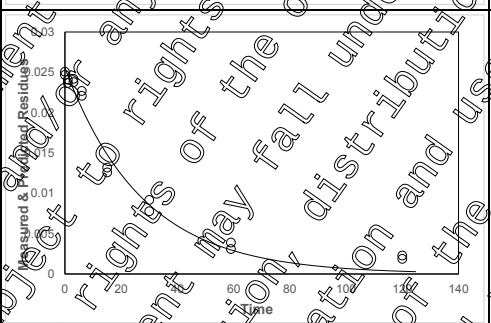
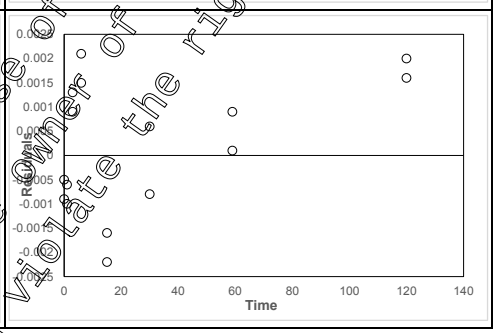
Table 7.1.2.1.2- 60: Re-evaluated degradation rate of M-05 under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
H[redacted] (2016b)	**SFO**	**0.02551**	**k 0.03086**	**3.6**	**1.21E-11**	**0.02765**	**0.034**	**22.5**	**74.6**
	FOMC	0.02551	α 1.01E+05 β 3.28E+06	3.84	n.r. n.r.	1.01E+05 3.28E+06	1.01E+05 3.28E+06	22.5	74.6
L[redacted] (2016b)	**SFO**	**0.02603**	**k 0.04124**	**4.94**	**7.06E-11**	**0.03635**	**0.046**	**16.8**	**55.8**
	FOMC	0.0263	α 4.8 β 102.9	4.84	n.r. n.r.	-2.268 -68.98	11.87 274.7	16	63.3
[redacted] (2016b)	**SFO**	**0.02557**	**k 0.03642**	**6.57**	**1.83E-09**	**0.03089**	**0.042**	**19.0**	**63.2**
	FOMC	0.02584	α 4.075 β 97.64	6.6	n.r. n.r.	-2.693 -90	10.84 285.3	18.1	74.2

Best fit model highlighted in bold

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.1- 112: Degradation of M-05 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
H[redacted] SFO [redacted] (2016b)		
L[redacted] SFO [redacted] (2016b)		
L[redacted] SFO [redacted] (2016b)		

III. Conclusion

M-05 is a minor metabolite of fluopicolide which would be predicted to form in the soil environment at very low levels. It was not observed in soil laboratory metabolism studies conducted with fluopicolide. M-05 was readily degraded in soil with half-lives of between 16.8 and 22.5 days and thus, it would not be expected to persist in the soil environment.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002). The study is considered valid to assess the aerobic degradation of M-05 in soil.

Data Point:	KCA 7.1.2.1.2/12
Report Author:	[redacted]
Report Year:	2016
Report Title:	AE1344123: Aerobic degradation in three soils
Report No:	S15-04155
Document No:	M-565224-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307, 2002; SANCO/3029/99 rev.4
Deviations from current test guideline:	none
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The degradation of M-10 (AE 1344123) was studied in three soils under aerobic conditions in the laboratory in the dark at 20 ± 2 °C and 55% of the maximum water holding capacity for up to 150 days.

Soil	Texture (USDA)	pH ($CaCl_2$)	% Organic Carbon
L[redacted]	loamy sand	5.3	1.5
L[redacted]	sandy loam	5.1	1.9
H[redacted]	silt loam	5.8	1.9

M-10 was applied to soil samples at an application rate of 0.026 mg/kg dry weight, equivalent to a field application rate of 133 g/ha of fluopicolide and a highly conservative estimate of the maximum occurrence of this metabolite. The chemical purity of M-10 was 98.5% which was considered in determining the application rate.

Samples were removed for extraction and analysis immediately after treatment (day 0) and 1, 3, 7, 15, 30, 59, 120 and 150 ([redacted] soil only) days of incubation. Soil samples were extracted at ambient temperature three times with acetonitrile/0.05 M ammonium carbonate (80:20, v/v) and then by microwave extraction with acetonitrile/0.05 M ammonium carbonate (80:20, v/v) at 60 °C. Soil extracts were analysed by HPLC-MS/MS to quantify the amount M-10 remaining.

M-10 was rather slowly degraded steadily degraded in L[redacted] soil, more readily degraded in L[redacted] soil and most readily degraded in H[redacted] soil. The amount of the metabolite in soil extracts declined from 0.0251 mg/kg at time zero to 0.0112 mg/kg by DAT 120, equivalent to 43.0% of applied in L[redacted] soil, from 0.0242 mg/kg at time zero to 0.048 mg/kg by DAT 150, equivalent to 18.6% of applied in L[redacted] soil and from 0.0244 mg/kg at time zero to 0.0026 mg/kg, equivalent to 10.1% of applied by DAT 59 in H[redacted] soil.

The following DT_{50} and DT_{90} values were calculated for M-10 in the three soils.

Soil (USDA texture)	Best Fit Kinetic Model	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)	Visual Assessment
L[redacted] (loamy sand)	DFOP	80.2	681.7	3.6	Good
L[redacted] (sandy loam)	FOMC	20.9	237.5	8.1	Moderate
H[redacted] (silt loam)	SFO	21.6	71.9	5.3	Good

A re-evaluation of the degradation kinetics resulted in similar best-fit un-normalised DT_{50} values of 20.2 to 77.3 days but overall longer DT_{90} values ranging from 71.6 days to >10000 days.

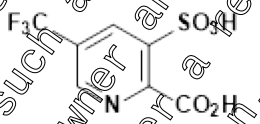
Degradation of the metabolite in aerobic soil was relatively slow, however M-10 is a minor metabolite of fluopicolide formed in the soil environment at very low levels. Should M-10 form in soil it would be steadily degraded and would not be expected to accumulate.

I. Materials and Methods

A. Materials

1. Test Item

M-10 (referred to as AE 1344123 in the report)



Chemical Purity: 98.5 % w/w

Sample/Batch ID: AE 1344123 00-1B99 0001, NLL7333-9a

Expiry Date: 09 March 2020

2. Test Soils

The study was performed using three German soils as characterized in Table 7.1.2.1.2- 61. The same soils were also used in laboratory aerobic soil studies with fluopicolide. Soils were collected fresh from the field and used with minimal storage time (44 days) prior to sieving to 2 mm and dispersing into flasks.

Table 7.1.2.1.2- 61: Physico-chemical properties of test soils

Parameter		Soil					
Soil Designation		L[redacted]		L[redacted]		H[redacted]	
Soil ID		[redacted]		[redacted]		[redacted]	
Geographic Location							
City		Monheim		Monheim		Burscheid	
Country		Germany		Germany		Germany	
GPS coordinates		[redacted]		[redacted]		[redacted]	
Batch Number		20150811-A		20150811-A		20150811-A	
Textural Classification (USDA)		Loamy sand		Sandy loam		Silt loam	
Sand [50 - 2000 µm]	(%)	78		58		26	
Silt [2 – 50 µm]	(%)	16		30		60	
Clay [< 2 µm]	(%)	6		12		14	
pH							
in $CaCl_2$ (1:2)		5.3		5.1		5.8	
in H_2O (1:1)		5.5		5.3		5.9	
Saturated paste		5.5		5.3		5.9	
in KCl (1:1)		5.0		4.8		5.5	
Organic Matter (%) *		2.6		3.3		3.3	
Organic Carbon (%)		1.5		1.9		1.9	
Cation Exchange Capacity (meq/100 g)		8.2		10.6		12.2	
Water Holding Capacity							
Maximum (g H_2O per 100 g DW)		31.8		55.5		58.3	
at 1/3 bar (%)		10.8		15.8		20.7	
Moisture Content During Incubation (%)		55% MWHC		55% MWHC		55% MWHC	
Bulk Density (disturbed, g/cm³)		1.22		1.13		1.08	
Soil Microbial Biomass (mg microbial C /100 g soil)		BIO^-	BIO^+	BIO^-	BIO^+	BIO^-	BIO^+
Initial (DAT 2)		152.6		162.2		185.3	
Mid (DAT 30)		179.8	199.3	206.9	222.4	240.3	235.5
Final (DAT 120)		161.3	160.0	176.6	197.9	238.9	206.5
Final (DAT 156)		-	-	170.3	-	-	-

* Calculated by multiplying organic carbon content by 1.724
MWHC = Maximum Water Holding Capacity
BIO^- samples were untreated
BIO^+ samples were treated with 400 µL of methanol:water (1:1, v/v)

B. Study Design

1. Experimental Conditions

Samples of 100 g dry weight of soil each were filled into glass incubation flasks and pre-equilibrated for 4 days prior to treatment at approximate study conditions (darkness, 20 ± 2 °C, soil moisture content equivalent to 55 ± 5% of maximum water holding capacity (MWHC)).

At the start of the test, each sample received 0.026 mg test substance/kg soil reflecting the maximum single field application rate of 133 g/ha of fluopicolide and a highly conservative formation level for the metabolite.

Samples were incubated and maintained at 20 ± 2 °C and 55 ± 5% of MWHC in the dark for a maximum of 120 days in L[redacted] [redacted] and H[redacted] [redacted] soils and 150 days in [redacted] soil. Soil samples were maintained under static conditions. All the flasks were stoppered with cotton wool. Untreated soil samples were incubated under the same conditions for determination of soil microbial activity. Additional untreated flasks containing 100g (dry weight) equivalent soil L[redacted] [redacted] were used to provide fortification samples to confirm the analytical method efficiency. At each sampling interval two flasks were fortified at the LOQ level (0.0013 mg/kg, 5% of the application rate) and two flasks at 22 times the LOQ (0.0286 mg/kg, 110% of the application rate).

2. Sampling

Duplicate samples were taken for analysis after 0, 1, 3, 7, 15, 30, 59 and 120 days of incubation in L[redacted] [redacted] and H[redacted] soils, and 0, 1, 3, 7, 15, 30, 59, 120 and 150 days of incubation in Wurmwiese soil. Microbial soil biomass samples were analysed at the start, midpoint and end of the experiment (DAT 2, 30 and 120) in each soil and an additional sample was taken at DAT 150 in [redacted] soil.

3. Analytical procedures

The entire soil sample of each test vessel was extracted three times with a mixture of acetonitrile:0.05 M ammonium carbonate (80:20, v/v) at ambient temperature. Ambient extraction was followed by an additional microwave extraction with acetonitrile:0.05 M ammonium carbonate (80:20, v/v) at 60°C. All extracts were combined and an aliquot centrifuged prior to analysis. Identification of M-10 in soil extracts was by HPLC-MS/MS. M-10 was quantified using the transition of m/z 270.0 to m/z 162.0 and in addition by the transition m/z 270.0 to m/z 74.1. Matrix-matched calibration curves were used for the quantification of M-10. The analytical method was validated with all three soils prior to starting the test.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The degradation of M-10 followed double first order in parallel (DFOP) kinetics in L[redacted] [redacted], first order multiple compartment (FOMC) kinetics in L[redacted] [redacted] soil and simple first order (SFO) kinetics in H[redacted] [redacted] [redacted] soil based on lowest chi^2 error values and visual assessments of fits.

Additionally, modelling endpoints for the degradation of M-10 have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/10 (M-685680-01-1). A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints for M-10, an initial comparison was performed for each soil between the SFO and FOMC fits. The SFO model provided a better fit for the H[redacted] soil, with a lower χ^2 err% value. For the L[redacted] [redacted] and L[redacted] [redacted] soils, the FOMC model provided a better visual fit, and the DFOP model was therefore fitted as well. DFOP provided the best

visual fit for both soils, with the lowest χ^2 err% value. The DFOP model was accepted for both soils despite a lack of confidence in the optimised rate constants, as the estimated DT_{90} exceeded the relevant regulatory triggers with either bi-phasic model and DFOP kinetics provided the best visual description of the decline.

II. Results and Discussion

A. Analytical Methodology:

A full summary of the analytical method is provided in Document MCA 4, Section 4.1.2. The method complies with all criteria according to *SANCO/3029/99 rev. 4* and is suitable for the determination of M-10 in soil samples by HPLC-MS/MS.

B. Data:

M-10 was rather slowly degraded steadily degraded in L[redacted] soil, more readily degraded in L[redacted] soil and most readily degraded in H[redacted] soil. The amount of the metabolite in soil extracts declined from 0.0251 mg/kg at time zero to 0.0112 mg/kg by DAT 120, equivalent to 43.0% of applied, in L[redacted] soil from 0.0242 mg/kg at time zero to 0.048 mg/kg by DAT 150, equivalent to 18.6% of applied in L[redacted] soil and from 0.0244 mg/kg at time zero to 0.0026 mg/kg, equivalent to 10.1% of applied, by DAT 59 in L[redacted] soil.

The results for each soil are summarized in Table 7.1.2.1.2- 62 to Table 7.1.2.1.2- 64.

Table 7.1.2.1.2- 62: Degradation of M-10 in L[redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	1	3	7	15	30	59	120
Mean	mg/kg	0.0251	0.0241	0.0257	0.0247	0.0217	0.0178	0.0140	0.0112
SD	mg/kg	0.0002	0.0006	0.0001	0.0010	0.0003	0.0006	0.0006	0.0000
Mean	%	96.3	92.7	98.8	94.8	83.3	68.3	53.7	43.0
RSD	%	1.0	2.5	0.4	4.0	1.6	3.2	4.2	0.3

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

Table 7.1.2.1.2- 63: Degradation of M-10 in L[redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)								
		0	1	3	7	15	30	59	120	150
Mean	mg/kg	0.0242	0.0244	0.0251	0.0203	0.0150	0.0088	0.0065	0.0056	0.0048
SD	mg/kg	0.0003	0.0003	0.0004	0.0004	0.0001	0.0009	0.0004	0.0005	0.0000
Mean	%	93.0	93.7	96.7	78.2	57.8	33.8	25.0	21.7	18.6
RSD	%	1.1	1.2	1.8	1.7	0.6	10.7	5.4	8.8	0.4

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation

Table 7.1.2.1.2- 64: Degradation of M-10 in H[redacted] soil under aerobic conditions at 20 °C

Parameter	Units	Incubation time (DAT)							
		0	1	3	7	15	30	59	120
Mean	mg/kg	0.0244	0.0267	0.0236	0.0213	0.0176	0.0099	0.0026	<LOD
SD	mg/kg	0.0031	0.0003	0.0003	0.0005	0.0004	0.0008	0.0002	<LOD
Mean	%	94.0	102.8	90.9	82.0	67.5	37.9	10.1	<LOD
RSD	%	12.9	1.0	1.2	2.1	2.5	8.0	7.0	<LOD

DAT: days after treatment
SD Standard deviation, RSD Relative standard deviation
LOQ = 0.0013 mg/kg; LOD = 0.0003 mg/kg

F. Degradation Kinetics

Best fit reported DT_{50} values of M-10 under aerobic conditions were 80.2, 20.9 and 21.6 days in L[redacted], L[redacted] and H[redacted] soils, respectively. The experimental data were best described by a double first order in parallel (DFOP) kinetic model in L[redacted] soil, a first order multiple compartment (FOMC) model in L[redacted] soil and a simple first order (SFO) model in H[redacted] soil. Details are provided below in Table 7.1.2.1.2- 65.

Table 7.1.2.1.2- 65: Reported degradation rate of M-10 under aerobic conditions at 20 ºC (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} (days)	DT_{90} (days)
L[redacted]	DFOP	0.0256765	k1 0.0265468 k2 0.0024850 g 0.4558428	3.6	1.85e-05 0.000549 < 2e-16	0.0183432 0.0014082 0.4479970	0.035 0.004 0.464	80.2	681.7
L[redacted]	FOMC	2.603e-02	α 8.385e-01 β 1.627e+01	8.1	1.97e-06 0.000326	6.054e-01 8.836e+00	1.072 23.739	20.9	237.5
H[redacted]	SFO	0.0262604	k 0.0320407	5.3	5.5e-10	0.0276025	0.036	21.6	71.9

In addition, the experimental data for the degradation of M-10 been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/10. The resulting DT_{50} values for trigger endpoints for M-15 are summarised below in Table 7.1.2.1.2- 66. Best fit kinetics are highlighted in bold.

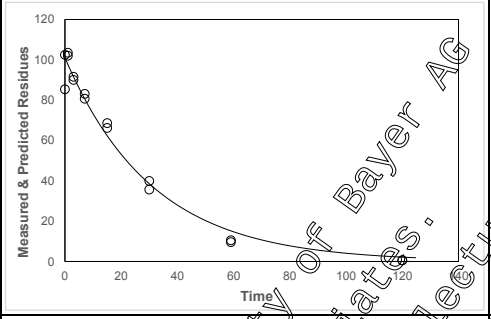
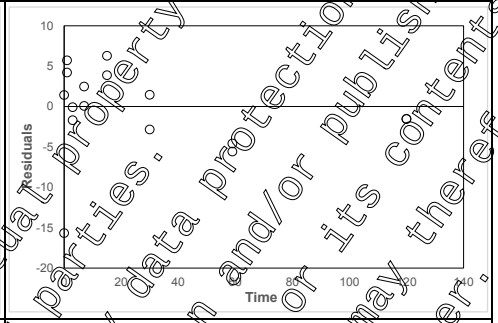
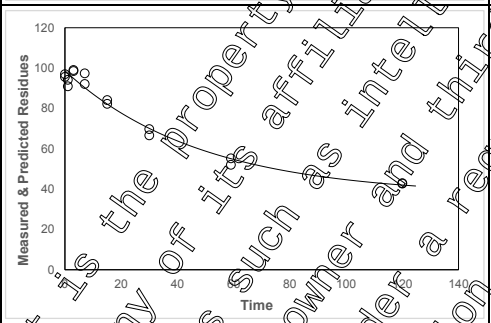
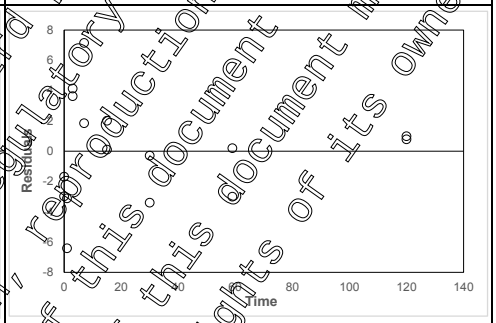
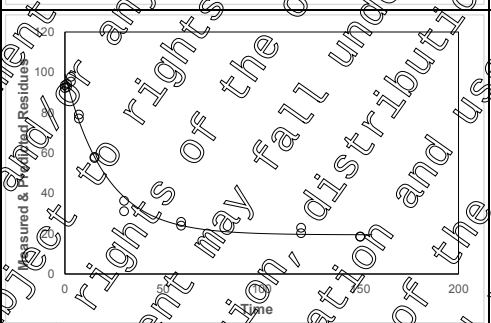
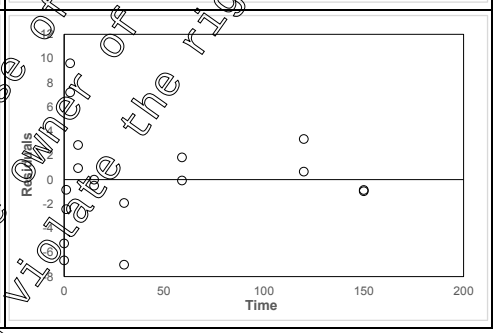
Table 7.1.2.1.2- 66: Re-evaluated degradation rate of M-10 under aerobic conditions at 20 °C (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
H [redacted] 2016c)	**SFO**	**101.1**	**k 0.03214**	**5.34**	**1.61E-09**	**0.0273**	**0.037**	**21.6**	**71.6**
	FOMC	101.1	α 2.33E+05 β 7.26E+06	5.7	n.r. n.r.	2.33E+05 7.26E+06	2.33E+05 7.26E+06	21.6	71.6
L [redacted] (2016c)	SFO	95.96	k 0.008261	4.79	7.25E-09	0.006864	0.01	83.9	278.7
	FOMC	98.66	α 0.762 β 54.72	3.57	n.r. n.r.	0.2004 -5.75	1.324 115.2	84.3	1068
	DFOP	**98.68**	**k1 0.02068 k2 2.34E-14 g 0.6267**	**3.47**	**0.171 0.5 n.r.**	**-0.0202 -0.02341 -0.7739**	**0.062 0.023 2.027**	**77.3**	**>10000**
L [redacted] (2016c)	SFO	94.31	k 0.02498	13.3	2.37E-06	0.01772	0.032	27.8	92.2
	FOMC	99.46	α 0.8631 β 17.73	8.12	n.r. n.r.	0.4364 -1.976	1.29 33.48	21.8	284.7
	DFOP	**98.97**	**k1 0.04804 k2 2.34E-14 g 0.8041**	**6.14**	**0.000137 0.5 n.r.**	**-0.02849 -0.007782 0.6095**	**0.068 0.008 0.999**	**20.2**	**>10000**
	HS	98.33	k1 0.03424 k2 0.003029 tb 37.96	5.18	2.30E-10 0.9688 n.r.	0.0298 -0.0007402 27.81	0.039 0.007 48.11	20.2	369.1

Best fit model highlighted in bold

Graphical representations of the final kinetic fits are shown below.

Table 7.1.2.1.1- 113: Degradation of M-10 under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil Model Reference	Modelled vs observed	Residuals
H SFO (2016c)		
L DFOP (2016c)		
L DFOP (2016c)		

III. Conclusion

M-10 is a minor metabolite of fluopicolide which would be predicted to form in the soil environment at very low levels. It was not observed in soil laboratory metabolism studies conducted with fluopicolide. Should M-10 form in soil it would be steadily degraded with reported half-lives of between 21.6 and 80.2 days. Re-evaluated best fit DT_{50} values were similar ranging from 21.6 to 77.3 days.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002). The study is considered valid to assess the aerobic degradation of M-10 in soil.

CA 7.1.2.1.3 Anaerobic degradation of the active substance

Data Point:	KCA 7.1.2.1.3/01
Report Author:	[redacted]
Report Year:	2003
Report Title:	Route and rate of degradation of [2,6-14C-pyridinyl] and [U-14C-benzoyl]-AE C638206[in a European sandy loam under laboratory anaerobic conditions at 20 degrees C
Report No:	B004072
Document No:	M-241050-01-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EC of July 1995
Deviations from current test guideline:	Yes. The soil was not incubated under aerobic conditions for 30 days prior to flooding. Due to the rate of degradation of fluopicolide in aerobic soil this will not impact significantly on the results of the study.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The rate of degradation of fluopicolide under anaerobic conditions is summarised under point CA 7.1.1.2.

CA 7.1.2.1.4 Anaerobic degradation of metabolites, breakdown and reaction products

The fate of metabolites of fluopicolide in soil under anaerobic conditions was investigated as part of the study on the anaerobic degradation and metabolism of the active substance. The details of this study are summarised under point 7.1.1.2. The route and rate of degradation of fluopicolide, labelled in the phenyl or in the pyridyl ring, was investigated in one soil under anaerobic conditions at 20°C and at an application rate equivalent to 400 g/ha.

No major metabolites were detected in either the water or soil phase.

CA 7.1.2.2 Field studies

CA 7.1.2.2.1 Soil dissipation studies

The field dissipation of fluopicolide has been investigated at a total of 17 locations; six 'legacy' European trials, five North American trials, two of which were identified as representative for European climate conditions and soil properties, and six new European trials where the design minimized soil surface processes as required by EFSA (2014). Separate trials were run concurrently at the latter six European sites with the metabolite M-01 (AE C653711) using a similar study design to minimized soil surface processes and included sampling of soil to a depth of 110 cm.

Four field dissipation studies (KCA 7.1.2.2.1/01, KCA 7.1.2.2.1/02, KCA 7.1.2.2.1/03, KCA 7.1.2.2.1/04) and two dissipation and accumulation studies (KCA 7.1.2.2.1/08 and KCA 7.1.2.2.1/09), conducted at six locations in Europe during 1999 to 2003, were evaluated during the previous EU review and are still considered as reliable to assess the rate of fluopicolide degradation in soil. A third accumulation study, where the trial was a continuation of the field dissipation trial conducted at the same site in previous years, was also evaluated during the previous EU review and is summarised under Point KCA 7.1.2.2.2 (KCA 7.1.2.2.2/01). North American field dissipation studies (KCA 7.1.2.2.1/16 and KCA 7.1.2.2.1/17) were conducted during 2001 to 2003 but have not yet been reviewed. A report assessing the relevance of North American trials for Europe is also provided (KCA 7.1.2.2.1/15).

In addition, new field studies KCA 7.1.2.2.1/12 and KCA 7.1.2.2.1/13 with fluopicolide, and KCA 7.1.2.2.1/18, KCA 7.1.2.2.1/19 and KCA 7.1.2.2.1/20 with M01 (AE C653711), plus their corresponding kinetic evaluation reports to derive DT_{50} and DT_{90} values (KCA 7.1.2.2.1/14 and KCA 7.1.2.2.1/21) are provided as new data not yet reviewed.

For procedural reasons five previously submitted reports also have to be included under Point KCA 7.1.2.2.1 in the current dossier (KCA 7.1.2.2.1/05, KCA 7.1.2.2.1/06, KCA 7.1.2.2.1/07, KCA 7.1.2.2.1/10 and KCA 7.1.2.2.1/11) but each has been fully superseded as described later.

Finally, new three kinetic evaluation reports (KCA 7.1.2.2.1/22, KCA 7.1.2.2.1/23 and KCA 7.1.2.2.1/24) are provided. In KCA 7.1.2.2.1/22 and KCA 7.1.2.2.1/23 $DegT50_{matrix}$ values, normalised to 20°C and pF2, were derived from field data for use as modelling endpoints. KCA 7.1.2.2.1/22 evaluates the data from the six 'legacy' field dissipation trials in which data points before 10 mm rainfall and irrigation were eliminated to minimise any influence from surface processes as advised by EFSA, 2014. KCA 7.1.2.2.1/23 evaluates the data from new field dissipation trials run concurrently with fluopicolide and M-01, in which the test item was incorporated into the soil immediately after application to eliminate processes potentially occurring at the soil surface such as photodegradation or volatilisation. KCA 7.1.2.2.1/24 evaluates the data from eight field dissipation trials to derive DT_{50} and DT_{90} values for fluopicolide (un-normalised) for use as trigger endpoints.

Report reference	Author, Year	Test item	Comment
KCA 7.1.2.2.1/01 M-218672-01-1	[redacted] 2003	Fluopicolide	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.2.2.1/02 M-218667-01-1	[redacted] 2003	Fluopicolide	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.2.2.1/03 M-234424-01-1	[redacted] 2004	Fluopicolide	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.2.2.1/04 M-220477-02-1	[redacted] 2003	Fluopicolide	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.2.2.1/05 M-223191-01-1	[redacted] 2003	Fluopicolide	Submitted and reviewed for first approval of fluopicolide, 2005. Interim report superseded by KCA 7.1.2.2.1/08 (M-251338-01-1).
KCA 7.1.2.2.1/06 M-223195-01-1	[redacted] 2003	Fluopicolide	Submitted and reviewed for first approval of fluopicolide, 2005. Interim report superseded by KCA 7.1.2.2.1/09 (M-247945-01-1).
KCA 7.1.2.2.1/07 M-234722-01-1	[redacted] 2003	Modelling report	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable for the initial submission. Superseded by M-685676-01-1.
KCA 7.1.2.2.1/08 M-251338-01-1	[redacted] 2005	Fluopicolide	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.2.2.1/09 M-247945-01-1	[redacted] 2005	Fluopicolide	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.
KCA 7.1.2.2.1/10 M-294400-01-1	[redacted] 2007	Modelling report	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable for the initial submission. Superseded by M-685682-01-1
KCA 7.1.2.2.1/11 M-294399-01-1	[redacted] 2007	Modelling report	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable for the initial submission. Superseded by M-685676-01-1.
KCA 7.1.2.2.1/12 M-651181-01-1	[redacted] 2019	Fluopicolide	New data not yet reviewed.
KCA 7.1.2.2.1/13 M-651179-01-1	[redacted] 019	Fluopicolide	New data not yet reviewed.
KCA 7.1.2.2.1/14 M-651636-01-1	[redacted] 019	Fluopicolide modelling report	New data not yet reviewedö
KCA 7.1.2.2.1/15 M-592872-01-1	[redacted] 2017	Fluopicolide	New data not yet reviewed.
KCA 7.1.2.2.1/16 M-248833-01-1	[redacted] 2004	Fluopicolide	New data not yet reviewed.
KCA 7.1.2.2.1/17 M-251292-01-1	[redacted] 2005	Fluopicolide	New data not yet reviewed.

Report reference	Author, Year	Test item	Comment
KCA 7.1.2.2.1/18 M-647366-03-1	[redacted] 2019	M-01	New data not yet reviewed.
KCA 7.1.2.2.1/19 M-647370-02-1	[redacted] 2019	M-01	New data not yet reviewed.
KCA 7.1.2.2.1/20 M-647363-02-1	[redacted] 2019	M-01	New data not yet reviewed.
KCA 7.1.2.2.1/21 M-650733-02-1	[redacted] 019	M-01 modelling report	New data not yet reviewed.
KCA 7.1.2.2.1/22 M-685676-01-1	[redacted] 2020	Modelling report	New data not yet reviewed.
KCA 7.1.2.2.1/23 M-685675-01-1	[redacted] 2020	Modelling report	New data not yet reviewed.
KCA 7.1.2.2.1/24 M-685682-01-1	[redacted] 2020	Modelling report	New data not yet reviewed.

Additional field studies are ongoing to investigate the degradation of M-01 (*Study Director:* [redacted] *Study Number: 18-2700, Title: Terrestrial Field Dissipation Study with BAM SC 125 in France (North); Study Director:* [redacted]*, Study Number: 18-2701, Title: Terrestrial Field Dissipation Study with BAM SC 125 in France (North); Study Director:* [redacted]*, Study Number: 18-2702, Title: Terrestrial Field Dissipation Study with BAM SC 125 in Germany and Study Director:* [redacted] *Study Number: 18-2703, Title: Terrestrial Field Dissipation Study with BAM SC 125 in Spain*). Final reports for these studies were not available in time to be included in this dossier. As agreed with the RMS, an updated dossier will be submitted by the notifier which will include the final report and its OECD summary.

Data Point:	KCA 7.1.2.2.1/01
Report Author:	[redacted]
Report Year:	2003
Report Title:	Aerobic outdoor field dissipation of the fungicide AE C638206 in a clay loam soil
Report No:	C035563
Document No:	M-218672-01-1
Guideline(s) followed in study:	BBA: IV, 4-1; SETAC: ; USEPA (=EPA): 164-1
Deviations from current test guideline:	Yes. Report meets the requirement for field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013 and the requirements for assessing parent soil DegT50matrix values as required by EFSA (2014) for legacy field studies. Report does not meet the requirement for assessing metabolite soil DegT50matrix values as required by EFSA (2014) for field studies.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

Soil dissipation of fluopicolide was studied after application as a suspo-emulsion formulation containing 97.9 g/L to bare soil plots under field conditions for 720 days at the trial site in Rödelsee (Germany).

A nominal application rate was 400 g fluopicolide /ha was applied in June 2000.

The initial dissipation of fluopicolide was rapid followed by a slower dissipation phase. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. In samples taken 14 to 360 days after application low residues were detected in the 10-20 cm horizon at concentrations ranging from 0.004 to 0.041 mg/kg (mean values) and once in the 20-30 cm horizon at a mean concentration of 0.005 mg/kg. In deeper depths no residues of fluopicolide were found above the LOQ.

The concentration of M-01 (AE C653711) in the soil profile varied with the degradation rate of fluopicolide. During the summer months when the degradation rate of the parent compound was relatively rapid, M-01 concentrations increased steadily reaching a peak at days 120 and declined thereafter as the degradation rate of fluopicolide slowed. The metabolite M-01 was detected in 0-10 cm, 10-20 cm, and once in 20-30 cm soil depths. The maximum residue level in the 0-10 cm horizon was observed 120 days after application at 0.025 mg/kg (mean of three replicates). The maximum residue in the underlying 10-20 cm horizon was also detected at 120 days after application at 0.010 mg/kg (mean value). In the 20-30 cm horizon residue levels were below the LOQ except for one replicate at 540 days in which M-01 residues were detected at the LOQ (0.005 mg/kg).

M-02 (AE C657188) and M-03 (AE 0608000) were very rapidly degraded in soil. M-02 was detected in the 0-10 cm depth in soil samples taken 5, 14 and 30 days after application at concentrations ranging from 0.009 to 0.013 mg/kg (mean values). One month after application no further residues of M-02 were detected. No residues of M-03 were found above the LOQ throughout the study. The degradation of M-03 is known to be pH dependant and is very rapidly degraded in neutral to alkaline soils such as the soil at the Rödelsee trial site.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit SFO un-normalised DT_{50} value of 256.9 days and DT_{90} of 853.5 days for fluopicolide.

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide formulated as liquid suspo-emulsion (97.9 g/L fluopicolide)

Certificate of Analysis: AGF2000-0106-01

Lot No: OP200271

2. Trial Locations & Soils

A single site was selected east of Würzburg (region "Unterfranken") near the village Rodelsee, in Southern Germany (see Table 7.1.2.2.1- 1). The test site had no significant slope and the top soil was a heavy, fine texture sandy clay loam soil (USDA classification) mainly composed of clayey silty sediments with carbonate and gypsum layers between. The field soil dissipation trial consisted of three treated plots, each measuring 5 metres by 36 metres (540 m² in total), and an untreated plot (180 m²) which served as a control. No dichlobenil containing pesticides were used during the study and two years before, according to personal communication.

Table 7.1.2.2.1- 1: Location, site description and climatic data of test site

Characteristic	Units	Rödelsee D-97348, Southern Germany					
		Horizon 1	Horizon 2	Horizon 3	Horizon 1 - 3	Horizon 4	Horizon 5
Sampling depth	cm	0 - 10	10 - 20	20 - 30	0 - 30	30 - 50	50 - 90
Date of sampling		10.04.00	10.04.00	10.04.00	28.11.01	28.11.01	28.11.01
pH	$CaCl_2$	7.4	7.5	7.8	7.3	7.5	7.6
Cation exchange capacity	meq/100 g	16	17	16	7.1	7.6	7.2
Total organic carbon (TOC)	%	1.7	1.4	1.1	1.4	1.0	0.3
Organic matter	%	2.9	2.4	1.9	2.4	1.7	0.6
Biomass	mg C/100 g	7.09	7.37	6.61	6.58	3.91	0.39
Soil water content	weight-%	18.8	9.9	16.0	n.d.	n.d.	n.d.
Particle density	g/cm³	n.d.	2.59	n.d.	2.60	2.66	2.69
Dry (bulk) density	g/cm³	1.53	1.54	1.60	1.45	1.49	1.64
Pore volume							
Fine pores (<0.2 µm)	Vol.-%	n.d.	22.6	n.d.	24.3	24.8	18.3
Medium pores + small coarse pores (0.2-30 µm)	Vol.-%	n.d.	13.3	n.d.	16.2	12.7	11.4
Coarse pores (>30 µm)	Vol.-%	n.d.	8.1	n.d.	4.2	6.5	9.3
Total pore volume (calc.)	Vol.-%	n.d.	44.0	n.d.	44.7	44.0	39.0
Field capacity (≥pF 2.0)	Vol.-%	n.d.	35.9	n.d.	40.5	37.5	29.7
Available water storage capacity (pF 2.0-4.2)	Vol.-%	n.d.	13.3	n.d.	16.2	12.7	11.4
Textural class	DIN	slight clayey loam	sandy clayey loam	sandy clayey loam	sandy clayey loam	sandy clayey loam	strong sandy loam
Particle size distribution (USDA)							
Clay < 0.002 mm	%	30.4	33.1	33.6	39.0	38.9	13.4
Total silt 0.002 - 0.050 mm	%	20.4	20.4	20.0	15.1	15.1	10.4
Total sand 0.050 - 2 mm	%	45.7	44.8	45.2	40.7	41.2	51.8
Gravel > 2 mm	%	2.4	1.2	0.9	3.6	3.4	17.0
Textural class	USDA	sandy clay loam	sandy clay loam	sandy clay loam	clay	clay	sandy loam

n.d: not determined

B. Study Design

1. Experimental Conditions

Fluopicolide was applied once as a suspo-emulsion containing 97.9 g/L at a nominal application rate of 400 g/ha on 7 June 2000. The nominal application rate was confirmed by measuring the unused formulation remaining in the spray tank to calibrate the amount applied (403 g/ha).

Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of the herbicide glyphosate to control weeds.

The weather conditions and the soil hydrology were monitored in an adjacent site at a distance of approximately 200 metres from the test site. On the adjacent site, rainfall, air temperature, soil temperature, wind speed, wind direction, relative air humidity, global radiation and soil moisture contents at depths of 30, 50, 80 and 120 cm were measured continuously. The total rainfall at the trial site amounted to 512 mm in 2000, 598 mm in 2001 and during the months January to May 2002 to 256 mm. The long term average rainfall for this region amounts to 594 mm/year.

Soil dissipation of fluopicolide was studied for 720 days.

2. Sampling

Soil cores were taken to a depth of 50 cm during the first year and to a depth of 90 cm during the second year, covering horizons of 0-10, 10-20, 20-30, 30-50, 50-70 and 70-90 cm. The sampling spots were equally distributed over sampling rows from each plot to obtain representative samples. At each sampling date 10 samples from each plot and each depth were taken using a Humax soil corer. In the first year it was not possible to sample to 50 cm depth at most sampling points as the soil was dry and hard and consequently samples were taken to a depth of 30 cm. This was not thought to have a detrimental effect on the study as the soil conditions and absence of water indicated that movement of residues to depth was unlikely.

Samples were taken, 1 day before application, directly after application (day 0) as well as 1, 5, 14, 30, 60, 120, 180, 270, 360, 450, 540 and 720 days after treatment (DAT).

The soil cores were frozen immediately after sampling. The soil samples from the same horizon of each subplot were thawed and blended in Germany and a subsample dispatched frozen to the analytical laboratory in France. The samples were then stored at -18 °C until required for analysis.

3. Analytical Procedures

The analytical method AR 265-01 was used to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000 referred to in the report as RPA 427867, fluopicolide-hydroxy). Soil samples of 20 g were extracted twice at ambient temperature for 5 minutes by mechanical agitation using acetonitrile/water (70/30, *v/v*) acidified with 0.1% formic acid. After each extraction step, extract and soil were separated by centrifugation and decantation. The soil extracts were combined and diluted with acidified water to result in a final solvent of acetonitrile/water (30/70) with 0.1% formic acid. Quantification was carried out by LC-MS/MS using external standardisation for the parent compound and its metabolites. The limit of quantification (LOQ) was 0.005 mg/kg for each analyte.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide and reference items M-01, M-02 and M-03 at levels of LOQ and 100 x LOQ and processed in parallel to the dissipation samples. The mean recoveries of LOQ and 100 x LOQ were 94 and 99% (RSD 6.4 and 5.5%) for fluopicolide, 96 and 102% (RSD 9.6 and 10.5%) for M-01, 95 and 93% (RSD 7.8 and 10.5%) for M-02 and 83 and 100% (RSD 12.0 and 8.7%) for M-03.

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.2.1/24. A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each site between the SFO and FOMC fits for fluopicolide. For the Rödelsee site, the FOMC fit provided no significant improvement, and the SFO fit was therefore accepted.

II. Results and Discussion

A. Analytical Methodology

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) in soil samples by HPLC-MS/MS.

B. Data

The results for fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots in Table 7.1.2.2.1- 2 to Table 7.1.2.2.1- 5.

Table 7.1.2.2.1- 2: Residues of fluopicolide in different depths of soil at Rödelsee (Germany), values expressed as mg/kg

Depth [cm]	Sub plot	DAT													
		-1	0	1	5	14	30	60	120[1]	180	270	360	450	540	720
0-10	1	<LOQ	0.460	0.470	0.454	0.259	0.273	0.200	0.312	0.199	0.225	0.141	0.082	0.050	0.043
	2	<LOQ	0.455	0.412	0.369	0.237	0.291	0.286	0.339	0.315	0.239	0.138	0.086	0.047	0.050
	3	<LOQ	0.484	0.454	0.378	0.256	0.288	0.273	0.307	0.263	0.137	0.117	0.072	0.045	0.046
	mean	**<LOQ**	**0.466**	**0.445**	**0.400**	**0.251**	**0.284**	**0.253**	**0.319**	**0.259**	**0.214**	**0.132**	**0.080**	**0.047**	**0.046**
10-20	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.058	0.014	0.028	0.011	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	0.007	<LOQ	0.039	0.007	0.009	0.016	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	0.006	0.054	<LOQ	0.027	0.014	0.019	0.012	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**0.004**	**0.021**	**<LOQ**	**0.041**	**0.012**	**0.019**	**0.013**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	0.009	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**0.005**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**-**	**-**

DAT: days after treatment
LOQ (limit of quantitation) = 0.005 mg/kg
[1] average of two determinations per subplot

Table 7.1.2.2.1- 3: Residues of M-01 (AE C653711) in different depths of soil at Rödelsee (Germany), values expressed as mg/kg

Depth [cm]	Sub plot	DAT													
		-1	0	1	5	14	30	60	120	180	270	360	450	540	720
0-10	1	<LOQ	<LOQ	<LOQ	0.013	0.012	0.017	0.017	0.023	0.014	0.008	0.010	0.012	0.009	0.006
	2	<LOQ	<LOQ	<LOQ	0.008	0.010	0.018	0.016	0.024	0.018	0.009	0.010	0.014	0.008	0.005
	3	<LOQ	<LOQ	<LOQ	0.009	0.012	0.017	0.021	0.029	0.019	0.009	0.011	0.011	0.009	0.005
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**0.010**	**0.011**	**0.017**	**0.018**	**0.025**	**0.017**	**0.009**	**0.010**	**0.012**	**0.009**	**0.005**
10-20	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.013	0.009	0.008	0.008	<LOQ	0.006	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.009	0.008	0.008	0.007	<LOQ	0.008	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.008	0.007	0.010	0.009	<LOQ	0.008	0.006
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.010**	**0.008**	**0.009**	**0.008**	**<LOQ**	**0.007**	**0.004**
20-30	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.005	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.003**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**-**	**-**

DAT: days after treatment

LOQ (limit of quantitation) = 0.005 mg/kg

[1] average of two determinations per subplot

Table 7.1.2.2.1- 4: Residues of M-03 (AE 0608000) in different depths of soil at Rödelsee (Germany), values expressed as mg/kg

Depth [cm]	Sub plot	DAT													
		-1	0	1	5	14	30	60	120	180	270	360	450	540	720
0-10	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**-**	**-**

DAT: days after treatment
LOQ (limit of quantitation) = 0.005 mg/kg

Table 7.1.2.2.1- 5: Residues of M-02 (AE C657188) different depths of soil at Rödelsee (Germany), values expressed as mg/kg

Depth [cm]	Sub plot	DAT													
		-1	0	1	5	14	30	60	120	180	270	360	450	540	720
0-10	1	<LOQ	<LOQ	<LOQ	0.011	0.012	0.012	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	0.008	0.011	0.013	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	0.009	0.014	0.013	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**0.009**	**0.012**	**0.013**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	-	-	-	-	-	<LOQ	-	-
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	-	-	-	-	<LOQ	-	-
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**-**	**-**

DAT: days after treatment
LOQ (limit of quantitation) = 0.005 mg/kg

C. Residues

Analysis of control samples showed no residues above the limit of quantification (LOQ) of 0.005 mg/kg.

The average initial concentration of fluopicolide in soil samples taken immediately after application was 0.466 mg/kg (range 0.455 to 0.484 mg/kg). This corresponds to an apparent application rate of 699 g/ha compared to the nominal application rate of 400 g/ha. This difference was concluded to be due to insufficient soil homogenisation, soil bulk density of less than 1.5 g/cm^3 (assumed throughout the study) at sampling or difficulties during sampling.

The initial dissipation of fluopicolide was rapid followed by a slower dissipation phase. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. In samples taken 14 to 360 days after application low residues were detected in the 10-20 cm horizon at concentrations ranging from 0.004 to 0.041 mg/kg (mean values) and once in the 20-30 cm horizon at a mean concentration of 0.005 mg/kg. In deeper depths no residues of fluopicolide were found above the LOQ.

The concentration of M-01 (AE C653711) in the soil profile varied with the degradation rate of fluopicolide. During the summer months, when the degradation rate of the parent compound was relatively rapid, M-01 concentrations increased steadily reaching a peak at DAT-120 and declined thereafter as the degradation rate of fluopicolide slowed. The metabolite M-01 was detected in 0-10 cm, 10-20 cm, and once in 20-30 cm soil depths. The maximum residue level in the 0-10 cm horizon was observed 120 days after application at 0.025 mg/kg (mean of three replicates). The maximum residue in the underlying 10-20 cm horizon was also detected at 120 days after application at 0.010 mg/kg (mean value). In the 20-30 cm horizon residue levels were below the LOQ except for one replicate at 540 days in which M-01 residues were detected at the LOQ (0.005 mg/kg).

M-02 (AE C657188) and M-03 (AE 0608000) were very rapidly degraded in soil. M-02 was detected in the 0-10 cm depth in soil samples taken 5, 14 and 30 days after application at concentrations ranging from 0.009 to 0.013 mg/kg (mean values). One month after application no further residues of M-02 were detected. No residues of M-03 (AE 0608000) were found above the LOQ throughout the study. The degradation of M-03 is known to be pH dependant and is very rapidly degraded in neutral to alkaline soils such as the soil at the Rödelsee trial site.

Based on these findings soil samples from the deeper soil layers (50 to 90 cm) taken in the second year were not analysed.

D. Kinetic Analysis

The half-life of fluopicolide included in the report was calculated using a bi-phasic first-order kinetic model (Hockey Stick) as 132.5 days. The DT$_{90}$ was 863.1 days and the r² was 0.987. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for Document KCA 7.1.2.2.1/24. The resulting best-fit DT$_{50}$ values for trigger endpoints are summarised below in Table 7.1.2.2.1- 6. Best fit kinetics are highlighted in bold.

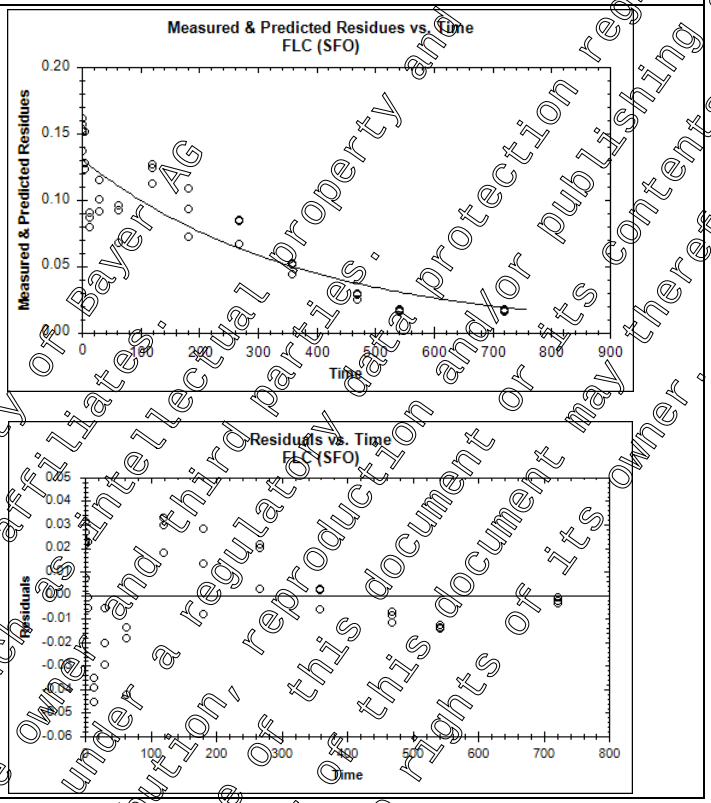
Table 7.1.2.2.1- 6; Degradation rate of fluopicolide under field conditions (DT$_{50}$ values for trigger endpoints)

Soil	Kinetic model	M$_0$	Parameter (k, k1, k2, g, tb, α, β)	χ², %-error	Prob >t	Lower CI	Upper CI	DT$_{50}$ [days]	DT$_{90}$ [days]
Rödelsee (Germany), CA 7.1.2.2.1/01, [redacted] 003)	**SFO**	**0.1302**	**k 0.002698**	**18.5**	**1.10E-09**	**0.002024**	**0.003**	**256.9**	**853.5**
	FOMC	0.1302	α 7858 β 2.91E+06	19.2	n.r. n.r.	7858 2.91E+06	7858 2.91E+06	256.9	853.6

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.2.1- 7: Graphical representations of best fit model

Trial / Best Fit Model	Graphical Representations
Rödelsee (Germany) / SFO	

III. Conclusion

Following a single application of fluopicolide at a rate of nominal rate of 400 g/ha to bare soil in summer 2000, the decline of fluopicolide and the formation and decline of its metabolites M-01, M-02 and M-03 was followed for up to 721 days after application at a trial site in Rödelsee, Germany. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit SFO un-normalised DT_{50} value of 256.9 days and DT_{90} value of 853.59 days for fluopicolide.

The metabolite M-01 was detected in 0-10 cm, 10-20 cm, and once in 20-30 cm soil depths. No residues of M-03 were found above LOQ (0.005 mg/kg) throughout the study. M-02 was detected in the 0-10 cm soil depth in early timepoints at low concentrations. One month after application no further residues of M-02 were detected.

Assessment and conclusion by applicant:

The study is considered valid to assess the dissipation of fluopicolide under field conditions in soil. The study meets the requirements to assess field persistence of fluopicolide and its metabolites, and to derive parent soil $DegT_{50matrix}$ values for legacy field studies as defined by EFSA (2014). It is not suitable for assessing metabolite soil $DegT_{50matrix}$ values as the design did not minimise soil surface processes immediately after application as required by EFSA (2014).

Data Point:	KCA 7.1.2.2.1/02
Report Author:	[redacted]
Report Year:	2003
Report Title:	Field soil dissipation of AE C638206 following single application to bare soil in Northern Germany
Report No:	C035562
Document No:	M-218667-01-1
Guideline(s) followed in study:	BBA: IV, 4-1; SETAC: ; USEPA (=EPA): 164-1
Deviations from current test guideline:	Yes. Report meets the requirement for field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013 and the requirements for assessing parent soil DegT50matrix values as required by EFSA (2014) for legacy field studies. Report does not meet the requirement for assessing metabolite soil DegT50matrix values as required by EFSA (2014) for field studies.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities.
Acceptability/Reliability:	Yes

Executive Summary

Soil dissipation of fluopicolide was studied after application as a suspo-emulsion formulation containing 97.9 g/L to bare soil plots under field conditions for 720 days at a site at the trial site in Huntlosen (Germany).

A nominal application rate of 400 g fluopicolide/ha was applied in May 2000.

The initial dissipation of fluopicolide was rapid followed by a slower dissipation phase. Residues of fluopicolide were only detected in the 0-10 cm soil horizon throughout the trial at concentrations ranging from 0.040 to 0.250 mg/kg (mean values). In deeper depths no residues of fluopicolide were found above the LOQ.

The concentration of M-01 (AE C653711) in the soil profile varied with the degradation rate of fluopicolide. During the summer months when the degradation rate of the parent compound was relatively rapid, M-01 concentrations increased steadily reaching a peak at days 120 and declined thereafter as the degradation rate of fluopicolide slowed. The metabolite M-01 was detected in 0-10 cm, 10-20 cm, and once in 20-30 cm soil depths. The maximum residue level in the 0-10 cm horizon was observed 120 days after application at 0.018 mg/kg (mean of three replicates). The maximum residue in the underlying 10-20 cm horizon was also detected at 270 days after application at 0.013 mg/kg (mean value). In the 20-30 cm horizon the maximum residue levels was detected at 0.006 mg/kg (mean value) at 360 days.

M-02 (AE C657188) was very rapidly degraded in soil. The meatobilite was only detected once in the 0-10 cm depth soil samples taken at 14 days after application at concentration of 0.008 mg/kg (mean of three replicates). M-03 was only found in soil samples from the 0-10 cm horizon reaching a maximum of 0.016 mg/kg at 180 days after application before steadily declining to 0.003 mg/kg at the end of the study. The degradation rate of M-03 in laboratory studies has been shown to be strongly pH dependant with bi-phasic degradation observed in strongly acidic soils.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit SFO un-normalised DT_{50} value of 290.2 days and DT_{90} of 963.9 days for fluopicolide.

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide formulated as liquid suspo-emulsion (97.9 g/L fluopicolide)

Certificate of Analysis: AGF2000-0106-01

Lot No: OP200271

2. Trial Location & Soil

A single site was selected near the village of Großenkneten-Huntlosen, D-26197, in Northern Germany (see Table 7.1.2.2.1- 8). The test site was flat and the top soil was a sand (USDA classification) with silty layers in between. The field soil dissipation trial consisted of three treated plots, each measuring 5 metres by 36 metres (540 m² in total), and an untreated plot (180 m²) which served as a control. No dichlobenil containing pesticides were used during the study and two years before, according to personal communication.

Table 7.1.2.2.1- 8: Location, site description and climatic data of test site

Characteristic	Units	Großenkneten-Huntlosen, D-26197, Northern Germany			
		Horizon 1	Horizon 2	Horizon 3	Horizon 4
Sampling depth	cm	10 – 20	20 – 30	30 – 50	50 – 90
Date of sampling		13.04.00	23.08.01	23.08.01	23.08.01
pH	CaCl2	5.3	4.9	4.9	5.1
Cation exchange capacity	meq/100 g	4.5	9.3	7.7	3.9
Total organic carbon (TOC)	%	1.9	1.8	0.8	0.5
Organic matter	%	3.3	3.1	1.4	0.9
Biomass 12 April 2000	mg C/100 g	4.56	n.a.	n.a.	n.a.
23 May 2001	mg C/100 g	n.a.	10.2	0.0	0.0
23 Nov 2001	mg C/100 g	n.a.	1.52	n.a.	n.a.
Soil water content	weight-%	8.5	8.0	n.d.	n.d.
Particle density	g/cm³	2.65	2.58	2.62	2.63
Dry (bulk) density	g/cm³	1.50	1.42	1.31	1.50
Pore volume					
Fine pores (<0.2 µm)	Vol.-%	6.5	8.0	5.3	2.6
Medium pores + small coarse pores (0.2-30 µm)	Vol.-%	17.6	16.8	8.7	6.0
Coarse pores (>30 µm)	Vol.-%	21.6	20.2	36.0	34.4
Total pore volume (calc.)	Vol.-%	45.7	45.0	50.0	43.0
Field capacity (≥pF 2.0)	Vol.-%	24.1	24.8	14.0	8.6
Available water storage capacity (pF 2.0-4.2)	Vol.-%	17.6	16.8	8.7	6.0
Particle size distribution (USDA)					
Clay < 0.002 mm	%	3.2	3.9	1.2	0.7
Total silt 0.002 - 0.050 mm	%	8.9	8.7	2.8	1.1
Total sand 0.050 - 2 mm	%	86.0	87.0	94.6	97.8
Gravel > 2 mm	%	1.37	0.3	1.0	0.3
Textural class	USDA	sand	sand	sand	sand

n.d: not determined, n.a: not applicable

B. Study Design

1. Experimental Conditions

Fluopicolide was applied once as a suspo-emulsion containing 97.9 g/L at an application rate of 400 g/ha on 31 May 2000. The nominal application rate was confirmed by measuring the unused formulation remaining in the spray tank to calibrate the amount applied (399 g/ha).

Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of the herbicide glyphosate to control weeds.

The weather conditions and the soil hydrology were monitored on site. The rainfall, air temperature, soil temperature, wind speed and soil moisture contents at depths of 10 and 20 cm were measured continuously. The total rainfall at the trial site amounted to 477.6 mm in 2000, 933.6 mm in 2001 and during the months January to May 2002 to 354.1 mm. The long term average rainfall for this region amounts to 700 - 800 mm/year.

Soil dissipation of fluopicolide was studied for 720 days.

2. Sampling

Soil cores were taken to a depth of 50 cm during the first year and to a depth of 90 cm during the second year, covering horizons of 0-10, 10-20, 20-30, 30-50, 50-70 and 70-90 cm. The sampling spots were equally distributed over sampling rows from each plot to obtain representative samples. At each sampling date 10 samples from each plot and each depth were taken using a Humax soil corer.

Samples were taken, 1 day before application, directly after application (day 0) as well as 1, 5, 14, 30, 60, 120, 180, 270, 360, 450, 540 and 720 days after treatment (DAT).

The soil cores were frozen immediately after sampling. The soil samples from the same horizon of each subplot were thawed and blended in Germany and a subsample dispatched frozen to the analytical laboratory in France. The samples were then stored at -18 ºC until required for analysis.

3. Analytical Procedures

The analytical method AR 265-01 was used to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000 referred to in the report as RPA 427967, fluopicolide-hydroxy). Soil samples of 20 g were extracted twice at ambient temperature for 5 minutes by mechanical agitation using acetonitrile/water (70/30, *v/v*) acidified with 0.1% formic acid. After each extraction step, extract and soil were separated by centrifugation and decantation. The soil extracts were combined and diluted with acidified water to result in a final solvent of acetonitrile/water (30/70) with 0.1% formic acid. Quantification was carried out by LC-MS/MS using external standardisation for the parent compound and its metabolites. The limit of quantification (LOQ) was 0.005 mg/kg for each analyte.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide and reference items M-01, M-02 and M-03 at levels of LOQ and 100 x LOQ and processed in parallel to the dissipation samples. The mean recoveries of LOQ and 100 x LOQ were 92 and 94% (RSD 15.4 and 4.8%) for fluopicolide, 97 and 98% (RSD 6 and 6%) for M-01, 91 and 88% (RSD 9.4 and 10.4%) for M-02 and 96 and 104% (RSD 22.7 and 14.2%) for M-03.

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.2.1/24. A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each site between the SFO and FOMC fits for fluopicolide. For the Huntlosen site, the FOMC fit provided no significant improvement, and the SFO fit was therefore accepted.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) in soil samples by HPLC-MS/MS.

B. Data

The results for fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots in Table 7.1.2.2.1-9 to Table 7.1.2.2.1-12.

Table 7.1.2.2.1- 9: Residues of fluopicolide in different depths of soil at Huntlosen (Germany), values expressed as mg/kg

Depth [cm]	Sub plot	DAT													
		-1	0	1	5	14	30	60	120	180	270	360	450	540	720
0-10	1	<LOQ	0.247	0.202	0.185	0.167	0.106	0.122	0.120	0.108	0.109	0.099	0.038	0.037	0.033
	2	<LOQ	0.271	0.221	0.187	0.162	0.138	0.115	0.100	0.158	0.109	0.096	0.034	0.040	0.042
	3	<LOQ	0.233	0.187	0.234	0.210	0.116	0.214	0.267	0.138	0.145	0.139	0.060	0.033	0.044
	mean	**<LOQ**	**0.250**	**0.203**	**0.202**	**0.180**	**0.120**	**0.150**	**0.162**	**0.131**	**0.121**	**0.111**	**0.044**	**0.037**	**0.040**
10-20	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

DAT: days after treatment
LOQ (limit of quantitation) = 0.005 mg/kg
[1] average of two determinations per subplot

Table 7.1.2.2.1- 10: Residues of M-01 (AE C653711) in different depths of soil at Huntlosen (Germany), values expressed as mg/kg

Depth [cm]	Sub plot	DAT													
		-1	0	1	5	14	30	60	120	180	270	360	450	540	720
0-10	1	<LOQ	<LOQ	<LOQ	<LOQ	0.012	0.013	0.012	0.016	0.016	0.007	0.012	0.009	0.007	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	0.010	0.014	0.015	0.016	0.014	0.008	0.010	0.009	0.005	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	0.014	0.015	0.016[1]	0.022	0.016	0.009	0.014	0.014	0.007	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.012**	**0.014**	**0.014**	**0.018**	**0.015**	**0.008**	**0.012**	**0.011**	**0.006**	**<LOQ**
10-20	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.012	0.008	0.008	<LOQ	0.009	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.013	0.007	0.007	0.006	0.008	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.009	0.014	0.011	0.011	0.007	0.009	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.005**	**0.013**	**0.013**	**0.009**	**0.005**	**0.009**	**<LOQ**
20-30	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.007	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.006	0.008	0.006	0.007	0.007
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.004**	**0.006**	**0.004**	**0.004**	**0.004**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.005
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.003**

DAT: days after treatment

LOQ (limit of quantitation) = 0.005 mg/kg

[1] average of two determinations per subplot

Table 7.1.2.2.1- 11: Residues of M-02 (AE C657188) different depths of soil at Huntlosen (Germany), values expressed as mg/kg

Depth [cm]	Sub plot	DAT													
		-1	0	1	5	14	30	60	120	180	270	360	450	540	720
0-10	1	<LOQ	<LOQ	<LOQ	<LOQ	0.007	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	0.006	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	0.010	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.008**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

DAT: days after treatment
LOQ (limit of quantitation) = 0.005 mg/kg

Table 7.1.2.2.1- 12: Residues of M-03 (AE 0608000) in different depths of soil at Huntlosen (Germany), values expressed as mg/kg

Depth [cm]	Sub plot	DAT													
		-1	0	1	5	14	30	60	120	180[1]	270	360	450	540	720
0-10	1	<LOQ	<LOQ	<LOQ	<LOQ	0.008	0.006	0.005	0.011	0.018	0.016	0.012	<LOQ	0.007	0.005
	2	<LOQ	<LOQ	<LOQ	0.006	0.006	0.006	<LOQ	0.009	0.019	0.013	0.009	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	0.008	0.012	0.006	0.005	0.016	0.012	0.012	0.010	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**0.006**	**0.009**	**0.006**	**0.004**	**0.012**	**0.016**	**0.014**	**0.010**	**<LOQ**	**0.004**	**0.003**
10-20	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

DAT: days after treatment

LOQ (limit of quantitation) = 0.005 mg/kg

[1] average of two determinations per subplot

C. Residues

Analysis of control samples showed no residues above the limit of quantification (LOQ) of 0.005 mg/kg.

The average initial concentration of fluopicolide in soil samples taken immediately after application was 0.250 mg/kg (range 0.233 to 0.271 mg/kg). This corresponds to an apparent application rate of 375 g/ha compared to the nominal application rate of 400 g/ha.

The initial dissipation of fluopicolide was rapid followed by a slower dissipation phase. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. At all other soil depths, no residues of fluopicolide were found above the LOQ.

The concentration of M-01 (AE C653711) in the soil profile varied with the degradation rate of fluopicolide. During the summer months when the degradation rate of the parent compound was relatively rapid, M-01 concentrations increased steadily reaching a peak at DAT-120 and declined thereafter as the degradation rate of fluopicolide slowed. The metabolite M-01 was detected in 0-10 cm, 10-20 cm, and once in 20-30 cm soil horizons. The maximum residue level in the 0-10 cm horizon was observed 120 days after application at 0.018 mg/kg (mean of three replicates). The maximum residues in the underlying 10-20 cm and 20-30 cm horizons were detected at 270 and 360 days after application at 0.013 and 0.006 mg/kg (mean values), respectively. In the 30-50 cm horizon residue levels were below the LOQ except for one replicate at 720 days in which AE C653711 residues were detected at the LOQ (0.005 mg/kg. In the overlying horizons at 720 days virtually no residues of AE C653711 were detected.

M-02 (AE C657188) was very rapidly degraded in soil. The metabolite was only detected once in the 0-10 cm depth soil samples taken at 14 days after application at concentration of 0.008 mg/kg (mean of three replicates). No other residues of M-02 were detected throughout the trial. M-03 was only found in soil samples from the 0-10 cm horizon reaching a maximum of 0.016 mg/kg at 180 days after application before steadily declining to 0.003 mg/kg at the end of the study. This concentration (0.016 mg/kg) represents the maximum formation of M-03 in terrestrial field dissipation studies (6.1%**).** The degradation rate of M-03 in laboratory studies has been shown to be strongly pH dependant with bi-phasic degradation observed in strongly acidic soils.

Based on these findings soil samples from the deeper soil layers (50 to 90 cm) taken in the second year were not analysed.

D. Kinetic Analysis

The half-life of fluopicolide included in the report was calculated using a bi-phasic first-order kinetic model (Hockey Stick) as 121.8 days. The DT_{90} was 892.8 days and the r^2 was 0.959. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for Document KCA 7.1.2.2.1/24. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.2.1-13. Best fit kinetics are highlighted in bold.

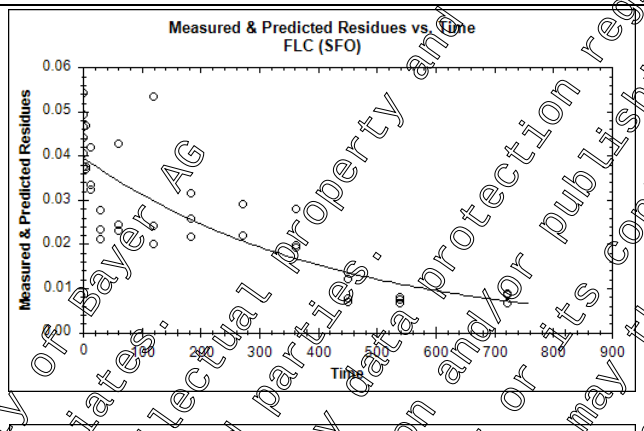
Table 7.1.2.2.1- 13: Degradation rate of fluopicolide under field conditions (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Huntlosen (Germany), CA 7.1.2.2.1/02, ([redacted] 2003)	**SFO**	**0.03948**	**k 0.002389**	**16.5**	**1.13E-07**	**0.001649**	**0.003**	**290.2**	**963.9**
	FOMC	0.03948	α 2442 β 1.02E+06	17.1	n.r. n.r.	2442 1.02E+06	2442 1.02E+06	290.2	964.2

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.2.1- 14: Graphical representations of best fit model

Trial / Best Fit Model	Graphical Representations
Huntlosen (Germany) / SFO	

III. Conclusion

Following a single application of fluopicolide at a nominal application rate of 400 g/ha to bare soil in summer 2000, the decline of fluopicolide and the formation and decline of its metabolites M-01, M-02 and M-03 was followed for up to 720 days after application at a trial site in Huntlosen, Germany. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit SFO un-normalised DT_{50} value of 290.2 days and DT_{90} value of 963.9 days for fluopicolide.

The metabolite M-01 was detected in 0-10 cm, 10-20 cm, and once in 20-30 cm soil depths. M-02 was only detected once at 14 days in the 0-10 cm soil horizon at 0.008 mg/kg while M-03 were found in the 0-10 cm soil horizon throughout the study from 5 days after application.

Assessment and conclusion by applicant:

The study is considered valid to assess the dissipation of fluopicolide under field conditions in soil. The study meets the requirements to assess field persistence of fluopicolide and its metabolites, and to derive parent soil $DegT_{50matrix}$ values for legacy field studies as defined by EFSA (2014). It is not suitable for assessing metabolite soil $DegT_{50matrix}$ values as the design did not minimise soil surface processes immediately after application as required by EFSA (2014).

Data Point:	KCA 7.1.2.2.1/03
Report Author:	[redacted]
Report Year:	2004
Report Title:	Field soil dissipation of AE C638206 following a single application to bare soil plots at 1 location in Spain, 2001
Report No:	C034123
Document No:	M-234424-01-1
Guideline(s) followed in study:	BBA: Part IV, 4-1; EU (=EEC): Anonymus, (1997) ; JVA: Beutel, (1993); SETAC: Lynch, (1995)
Deviations from current test guideline:	Yes. Report meets the requirement for field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013 and the requirements for assessing parent soil DegT50matrix values as required by EFSA (2014) for legacy field studies. Report does not meet the requirement for assessing metabolite soil DegT50matrix values as required by EFSA (2014) for field studies.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities.
Acceptability/Reliability:	Yes

Executive Summary

Soil dissipation of fluopicolide was studied after application as a suspo-emulsion formulation containing 90.1 g/L to bare soil plots under field conditions for 708 days at the trial site in Alboraya, Valencia, Eastern Spain.

A nominal application rate was 400 g fluopicolide/ha was applied in July 2001.

The initial dissipation of fluopicolide was rapid followed by a slower dissipation phase. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. Low residues were detected in the 10-20 cm horizon at concentrations ranging from 0.002 to 0.018 mg/kg (mean values). In the deeper depths no residues of fluopicolide were found above the LOQ.

The concentration of M-01 (AE C653711) in soil increased rapidly in the first month after application and then remained fairly constant throughout the study due to its constant formation from the degradation of parent, before declining at the end of the study period. M-01 was detected in 0-10 cm soil depth and at low levels in 10-20 cm depth from 128 days after application (November 2001). The maximum residue level in the 0-10 cm horizon was observed 36 days after application at 0.021 mg/kg (mean of four replicates). The maximum residue in the underlying 10-20 cm horizon was detected in January 2003, 120 days after application at 0.009 mg/kg (mean value). In the 20-30 cm horizon and deeper soil layers residue levels were below the LOQ (0.005 mg/kg).

M-02 (AE C657188) was detected at timepoints up to 247 days after application in the 0-10 cm layer at a maximum concentration of 0.017 mg/kg (mean value) 36 days after application. No residues of M-02 were detected in deeper soil depths above the LOQ.

No residues of M-03 (AE 0608000) were found above the LOQ throughout the study. The degradation of M-03 is known to be pH dependent and is very rapidly degraded in neutral to alkaline soils such as the soil at the Valencia trial site.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit DFOP un-normalised DT_{50} value of 53.9 days and DT_{90} of 987.5 days for fluopicolide

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide formulated as liquid suspo-emulsion (90.1 g/L fluopicolide)

Certificate of Analysis: AGF2001-0174-01

Lot No: OP210510

2. Trial Location & Soil

A single site was selected near the village of Alboraya, Valencia, Eastern Spain (see Table 7.1.2.2.1-15). The soil at the test site was a loamy sand (DIN classification) which was relatively uniform with depth. The field soil dissipation trial consisted of four treated plots, each measuring 3 metres by 20 metres (240 m² in total), and an untreated plot (36 m²) which served as a control.

Table 7.1.2.2.1- 15: Location, site description and climatic data of test site

Characteristic	Units	Alboraya, Valencia, Spain		
		Horizon 1	Horizon 2	Horizon 3
Sampling depth	cm	0 - 20	20 - 50	50 - 90
Sampling date		3 July 2001	3 July 2001	3 July 2001
pH	$CaCl_2$	7.3	7.4	7.6
Cation exchange capacity	mval/100 g	11.5	11.0	12.9
Total organic carbon (TOC)	%	1.87	1.53	1.58
Biomass	mg C/100 g	18.48	n.d.	n.d.
Soil Density	g/L	1530	1410	1300
Particle size distribution (DIN)				
Clay < 0.002 mm	%	33.0	75.0	60.1
Total silt 0.002 - 0.063 mm	%	16.1	16.2	23.4
Total sand 0.063 - 2 mm	%	10.9	8.8	16.5
Textural class	DIN	Loamy sand	Loamy sand	Loamy sand
Water Holding Capacity	Vol % at 1/10 bar (pF2)	24.6	24.2	n.d.
	Vol % at 15 bar (pF4.2)	10.6	11.2	n.d.

n.d. = not determined

B. Study Design

1. Experimental Conditions

Fluopicolide was applied once as a suspo-emulsion containing 90.1 g/L at a nominal application rate of 400 g/ha on 04 July 2001. The nominal application rate was confirmed by measuring the unused formulation remaining in the spray tank to calibrate the amount applied (423 g/ha).

Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of the herbicide glyphosate to control weeds.

The weather conditions and the soil hydrology were monitored on site. The rainfall, air temperature, soil temperature, relative air humidity, global radiation and the soil moisture content at a depth of 10 cm were measured continuously. The total rainfall at the trial site amounted to 156 mm in July to December 2001, 503 mm in 2002 and during the months January to June 2003 to 192 mm. The corresponding long term average rainfall for this region over these time periods amounted to 240 mm in 2001, 403 mm in 2002 and 164 mm in 2003. Supplemental irrigation was applied to the soil surface in 2001.

Soil dissipation of fluopicolide was studied for 708 days.

2. Sampling

Soil cores were taken to a depth of 30 cm during the first two weeks, to a depth of 50 cm at time-points up to two months and to a depth of 90 cm during the remainder of the study (23 months). At each sampling date 5 samples from each plot were taken (20 cores in total).

Samples were taken, 1 day before application, directly after application (day 0) as well as 1, 3, 14, 36, 65, 128, 194, 247, 315, 373, 460, 553 and 708 days after treatment (DAT).

The soil cores were frozen immediately after sampling. The soil samples from the same horizon of each subplot were thawed and blended in Germany and a subsample dispatched frozen to the analytical laboratory in France. The samples were then stored at -18 ºC until required for analysis.

3. Analytical Procedures

The analytical method AR 265-01 was used to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608009 referred to in the report as hydroxy RPA 427967). Soil samples of 20 g were extracted twice at ambient temperature for 5 minutes by mechanical agitation using acetonitrile/water (70/30, v/v) acidified with 0.1% formic acid. After each extraction step, extract and soil were separated by centrifugation and decantation. The soil extracts were combined and diluted with acidified water to result in a final solvent of acetonitrile/water (30/70) with 0.1% formic acid. Quantification was carried out by LC-MS/MS using external standardisation for the parent compound and its metabolites. The limit of quantification (LOQ) was 0.005 mg/kg for each analyte.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide and reference items M-01, M-02 and M-03 at levels of LOQ and 100 x LOQ and processed in parallel to the dissipation samples. The mean recoveries of LOQ and 100 x LOQ were 92 and 94% (RSD 9.1 and 7.2%) for fluopicolide, 96 and 100% (RSD 10.3 and 9.0%) for M-01, 85 and 98% (RSD 11.1 and 10.2%) for M-02 and 82 and 77% (RSD 14.1 and 14.2%) for M-03.

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.2.1/24. A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each site between the SFO and FOMC fits for fluopicolide. The comparison of the SFO and FOMC fits suggested bi-phasic decline, and the DFOP model was therefore also fitted. For the Valencia site, DFOP provided the best fit to the residues, with the lowest χ^2 err% value, and was therefore accepted.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) in soil samples by HPLC-MS/MS.

B. Data

The results for fluopicolide and its metabolites M-01 (AE C653711) and M-02 (AE C657188) are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots in Table 7.1.2.2.1- 16 to Table 7.1.2.2.1- 18. No residues of M-03 (AE 0608000) were detected throughout the trial.

Table 7.1.2.2.1- 16: Residues of fluopicolide in different depths of soil at Valencia (Spain), values expressed as mg/kg

Depth [cm]	Sub plot	DAT														
		-1	0	1	3	14	36	65	128	194	247	315	373	460	553	708
0-10	1	<LOQ	0.339	0.255	0.307	0.268	0.282	0.073	0.083	0.102	0.125	0.141	0.073	0.050	0.050	0.040
	2	<LOQ	0.446	0.220	0.237	0.240	0.166	0.099	0.087	0.084	0.139	0.105	0.074	0.071	0.054	0.037
	3	<LOQ	0.304	0.236	0.198	0.228	0.187	0.190	0.099	0.132	0.161	0.084	0.053	0.043	0.053	0.050
	4	<LOQ	0.245	0.303	0.371	0.213	0.123	0.129	0.109	0.103	0.132	0.082	0.055	0.051	0.038	0.035
	mean	**<LOQ**	**0.334**	**0.254**	**0.278**	**0.237**	**0.191**	**0.123**	**0.095**	**0.105**	**0.139**	**0.103**	**0.064**	**0.054**	**0.049**	**0.041**
10-20	1	<LOQ	0.009	0.008	0.024	0.010	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.007	<LOQ
	2	<LOQ	0.008	0.007	0.012	<LOQ	<LOQ	<LOQ	0.009	<LOQ	<LOQ	0.007	<LOQ	<LOQ	0.016	<LOQ
	3	<LOQ	0.008	0.009	0.017	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.008	<LOQ	<LOQ	<LOQ	0.012	<LOQ
	4	<LOQ	<LOQ	0.011	0.019	0.008	<LOQ	<LOQ	0.023	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.007	<LOQ
	mean	**<LOQ**	**0.006**	**0.009**	**0.018**	**0.005[1]**	**<LOQ**	**<LOQ**	**0.008[1]**	**<LOQ**	**0.002[1]**	**0.002[1]**	**<LOQ**	**<LOQ**	**0.011**	**<LOQ**
20-30	1	<LOQ	**<LOQ**	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	<LOQ	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	4	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	n.a.	n.a.
50-70	1	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	2	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	3	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	4	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	n.a.	n.a.

Depth [cm]	Sub plot	DAT														
		-1	0	1	3	14	36	65	128	194	247	315	373	460	553	708
70-90	1	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	2	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	3	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	4	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	n.a.	n.a.

DAT: days after treatment

LOQ (limit of quantitation) = 0.005 mg/kg, n.a. = not analysed, [1] Replicate value > LOQ

Table 7.1.2.2.1- 17: Residues of M-01 (AE C653711) in different depths of soil at Valencia (Spain), values expressed as mg/kg

Depth [cm]	Sub plot	DAT														
		-1	0	1	3	14	36	65	128	194	247	315	373	460	553	708
0-10	1	<LOQ	<LOQ	0.005	0.013	0.021	0.029	0.016	0.012	0.012	0.020	0.011	0.014	0.015	0.015	0.009
	2	<LOQ	<LOQ	<LOQ	0.007	0.018	0.018	0.017	0.020	0.007	0.020	0.009	0.015	0.020	0.015	0.009
	3	<LOQ	<LOQ	0.006	0.008	0.020	0.015	0.023	0.015	0.008	0.017	0.010	0.012	0.011	0.016	0.012
	4	<LOQ	<LOQ	0.007	0.015	0.016	0.022	0.021	0.018	<LOQ	0.021	0.012	0.013	0.012	0.011	0.008
	mean	**<LOQ**	**<LOQ**	**0.005**	**0.011**	**0.019**	**0.021**	**0.019**	**0.016**	**0.007**	**0.020**	**0.011**	**0.014**	**0.015**	**0.014**	**0.010**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.009	<LOQ	<LOQ	<LOQ	<LOQ	0.007	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.009	<LOQ	<LOQ	0.008	0.007	0.007	0.010	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.007	0.008	<LOQ	<LOQ	<LOQ	0.012	0.008
	4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.008	<LOQ	<LOQ	0.007	<LOQ	<LOQ	0.007	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.002[1]**	**0.004[1]**	**0.002[1]**	**0.004[1]**	**0.002[1]**	**0.002[1]**	**0.009**	**0.002[1]**

Depth [cm]	Sub plot	DAT														
		-1	0	1	3	14	36	65	128	194	247	315	373	460	553	708
20-30	1	<LOQ	**<LOQ**	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	<LOQ	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	4	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	n.a.	n.a.
50-70	1	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	2	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	3	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	4	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	n.a.	n.a.
70-90	1	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	2	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	3	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	4	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	n.a.	n.a.

DAT: days after treatment

LOQ (limit of quantitation) = 0.005 mg/kg, n.a. = not analysed, | Replicate value > LOQ

Table 7.1.2.2.1- 18: Residues of M-02 (AE C657188) different depths of soil at Valencia (Spain), mean values expressed as mg/kg

Depth [cm]	Sub plot	DAT														
		-1	0	1	3	14	36	65	128	194	247	315	373	460	553	708
0-10	1	<LOQ	<LOQ	<LOQ	<LOQ	0.017	0.021	0.015	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	0.015	0.015	0.011	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	0.006	0.015	0.013	0.022	<LOQ	<LOQ	0.007	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	4	<LOQ	<LOQ	<LOQ	0.009	0.014	0.018	0.018	0.006	<LOQ	0.005	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**0.004[1]**	**0.015**	**0.017**	**0.017**	**0.002[1]**	**<LOQ**	**0.003[1]**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	**<LOQ**	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	<LOQ	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	2	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	3	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	4	<LOQ	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	n.a.	n.a.
50-70	1	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	2	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	3	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	4	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	n.a.	n.a.

Depth [cm]	Sub plot	DAT													
		-1	0	1	3	14	36	65	128	194	247	315	373	460	553	708
70-90	1	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	2	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	3	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	4	<LOQ	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a.	n.a.
	mean	**<LOQ**	**-**	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	n.a.	n.a.

DAT: days after treatment

LOQ (limit of quantitation) = 0.005 mg/kg, n.a. = not analysed, [1] Replicate value > LOQ

C. Residues

Analysis of control samples showed no residues above the limit of quantification (LOQ) of 0.005 mg/kg.

The average initial concentration of fluopicolide in soil samples taken immediately after application was 0.334 mg/kg (range 0.245 to 0.446 mg/kg) in 0-10 cm layer directly after application plus 0.006 mg/kg (range < LOQ to 0.009 mg/kg) in the 10-20 cm layer. This corresponds to an apparent application rate of 509.6 g/ha compared to the nominal application rate of 400 g/ha.

The initial dissipation of fluopicolide was relatively rapid followed by a slower dissipation phase during the winter months due to the cold climate and possibly reduced availability of fluopicolide due to increased adsorption to soil with ageing. The initial dissipation of fluopicolide was relatively rapid followed by a slower dissipation phase during the winter months due to the cold climate and possibly reduced availability of fluopicolide due to increased adsorption to soil with ageing. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. Low residues were detected in the 10-20 cm horizon at concentrations ranging from 0.002 to 0.018 mg/kg (mean values). In the deeper depths no residues of fluopicolide were found above the LOQ.

The concentration of M-01 (AE C653711) in the soil profile in soil increased rapidly in the first month after application and then remained fairly constant throughout the study due to its constant formation from the degradation of parent, before declining at the end of the study period. M-01 was detected in 0-10 cm soil depth and at low levels in 10-20 cm depth from 128 days after application (November 2001). The maximum residue level in the 0-10 cm horizon was observed 30 days after application at 0.021 mg/kg (mean of four replicates). The maximum residue in the underlying 10-20 cm horizon was detected in January 2003, 120 days after application at 0.009 mg/kg (mean value). In the 20-30 cm horizon and deeper soil layers residue levels were below the LOQ (0.005 mg/kg).

M-02 (AE C657188) was detected at timepoints up to 247 days after application in the 0-10 cm layer at a maximum concentration of 0.017 mg/kg (mean value) 36 days after application. No residues of M-02 were detected in deeper soil depths above the LOQ.

No residues of M-03 (AE 0608000) were found above the LOQ throughout the study. The degradation of M-03 is known to be pH dependent and is very rapidly degraded in neutral to alkaline soils such as the soil at the Valencia trial site.

D. Kinetic Analysis

The half-life of fluopicolide included in the report was calculated using a bi-phasic first-order kinetic model (Hockey Stick) as 50 days. The DT_{90} was 973 days and the r^2 was 0.953. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for Document KCA 7.1.2.2.1-24. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.2.1- 19. Best fit kinetics are highlighted in bold.

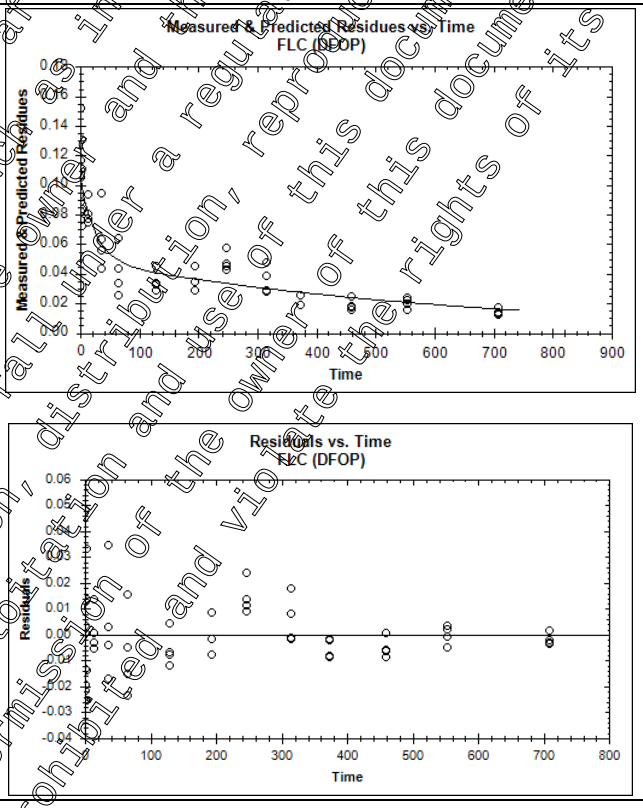
Table 7.1.2.2.1- 19: Degradation rate of fluopicolide under field conditions (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Valencia (Spain), CA 7.1.2.2.1/03, [redacted] (2004)	SFO	0.08862	k 0.003908	21.5	1.92E-10	0.002905	0.005	177.4	589.3
	FOMC	0.1048	α 0.4169 β 14	12.7	n.r. n.r.	0.2407 -3.434	0.593 31.43	39.8	3490
	DFOP	**0.104**	**k1 0.03872 k2 0.001574 g 0.5271**	**12**	**0.01124 0.00433 n.r.**	**0.006458 0.000443 0.3584**	**0.071 0.003 0.696**	**53.9**	**987.5**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.2.1- 20: Graphical representations of best fit model

Trial / Best Fit Model	Graphical Representations
Valencia (Spain) / DFOP	

III. Conclusion

Following a single application of fluopicolide at a nominal application rate of 400 g/ha to bare soil in summer 2001, the decline of fluopicolide and the formation and decline of its metabolites M-01, M-02 and M-03 was followed for up to 708 days after application at a trial site in Valencia, Spain. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit DFOP un-normalised DT_{50} value of 53.9 days and DT_{90} value of 987.5 days for fluopicolide.

The metabolites M-01 (AE C653711) and M-02 (AE C657188) were detected in Valencia soil. M-01 was detected in the 0-10 cm soil depth and at low concentrations in the 10-20 cm depth. M-02 was detected only in the 0-10 cm depth. No residues of M-03 were detected throughout the study.

Assessment and conclusion by applicant:

The study is considered valid to assess the dissipation of fluopicolide under field conditions in soil. The study meets the requirements to assess field persistence of fluopicolide and its metabolites, and to derive parent soil $DegT_{50matrix}$ values for legacy field studies as defined by EFSA (2014). It is not suitable for assessing metabolite soil $DegT_{50matrix}$ values as the design did not minimise soil surface processes immediately after application as required by EFSA (2014).

Data Point:	IIIA 7.1.2.2.1/04
Report Author:	[redacted]
Report Year:	2003
Report Title:	Field soil dissipation of AE C638206 following single application to bare soil plots at two locations in Europe in 1999
Report No:	C036344
Document No:	M-220477-02-1
Guideline(s) followed in study:	BBA: part IV, 4-1; SETAC: ; USEPA (EPA): 164-1
Deviations from current test guideline:	Yes. Report meets the requirement for field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013 and the requirements for assessing parent soil DegT50matrix values as required by EFSA (2014) for legacy field studies. Report does not meet the requirement for assessing metabolite soil DegT50matrix values as required by EFSA (2014) for field studies.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The soil dissipation of fluopicolide, formulated as a suspension concentrate containing 20.4 % w/w fluopicolide, was initiated at two trial sites at sites near Plusha, Cornwall, UK and Senas, Southern France. The trial at Plusha was terminated and no analytical data was reported for this site.

A nominal application rate was 500 g fluopicolide /ha was applied in June 1999.

The initial dissipation of fluopicolide was relatively rapid followed by a slower dissipation phase during the winter months due to the cold climate. Dissipation continued the following summer at a slower rate than the initial rapid phase possibly due to the reduced availability of fluopicolide due to increased adsorption to soil with ageing. Residues of fluopicolide were detected only in the 0-10 cm soil horizon throughout the trial at concentrations ranging from 0.018 to 0.218 mg/kg (mean values). In the deeper depths no residues of fluopicolide were found above the LOQ.

The concentration of M-01 (AE C653711) in soil increased rapidly in the two months after application and then remained fairly constant throughout the first year due to its constant formation from the degradation of parent, before declining rapidly in the second year. The maximum residue level in the 0-10 cm horizon was observed 60 days after application at 0.026 mg/kg (mean of three replicates). The maximum residue in the underlying 10-20 cm was detected at 240 days after application at 0.008 mg/kg (mean values). In the 20-30 cm horizon residue levels were below the LOQ except for one replicate at 300 days in which AE C653711 residues were detected at 0.006 mg/kg. No residues were detected in 30-45 cm depth above the LOQ. By the end of the study after two years, residue levels had declined to the LOQ.

M-02 (AE C657188) was detected at timepoints up to 60 days after application in the 0-10 cm layer at a maximum concentration of 0.021 mg/kg (mean value) 28 days after application. No residues of M-02 were detected in deeper soil depths above the LOQ.

No residues of M-03 (AE 0608000) were found above the LOQ throughout the study. The degradation of M-03 is known to be pH dependent and is very rapidly degraded in neutral to alkaline soils such as the soil at the Valencia trial site.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit DFOP un-normalised DT_{50} value of 109.8 days and DT_{90} of 627.2 days for fluopicolide

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide formulated as a suspension concentrate (20.4 % w/w fluopicolide)

Lot No: TM99000750

2. Trial Location & Soil

A terrestrial field dissipation study with fluopicolide was initiated at two trial sites at sites near Plusha, Cornwall, UK and Senas, Southern France.

The Senas test site was selected to be representative of a vine growing region. The top soil was a sandy silt loam / clay loam soil (ADAS classification). The characteristics of the soil are summarised in Table 7.1.2.2.1- 21. Three experimental plots, each measuring 2.5 metres by 26 metres (65 m² in total), were treated with the test substance. Each plot was divided into two strips 13 metres long and one of these strips from each plot was sampled and reported in this study. The other strip has been sampled and reported separately as an accumulation trial (KCA 7.1.2.2.2/01, M-223186-01-1). A fourth identical plot was left untreated to provide control samples.

The trial at Plusha was terminated after 1 year as the high organic matter and biomass at this site, which had previously been pasture land, led to high variations in the soil residues and no analytical data was reported for this site.

Table 7.1.2.2.1- 21: Location, site description and climatic data of test site

Characteristic	Units	Senas, Provence, France .
Sampling date		6 December 1999
pH	Water	7.6
Cation exchange capacity	meq/100 g	11.8
Organic carbon	%	1.6
Biomass	µg C/g	553.5
Dry matter content (of air-dried soil)	% (m/m)	99.0
Water content (of air-dried soil)	% (m/m)	1.0
Bulk density (disturbed sample)	g/cm³	1.2
Particle size distribution (ADAS)		
Clay < 0.002 mm	%	16.76
Total silt 0.002 - 0.063 mm	%	44.74
Total sand 0.063 - 2 mm	%	38.50
Textural class	ADAS	Sandy silt loam / Clay loam
Loss on ignition	%	1.8
WHC_{max}	% (m/m)	47.6
$WHC_{0.05bar}$	% (m/m)	32.3
WHC_{15bar}	% (m/m)	7.2
Dry matter content (of field-moist soil)	% (m/m)	91.4
Water content (of field-moist soil)	% (m/m)	9.4

* Calculated from data reported for this summary

B. Study Design

1. Experimental Conditions

Fluopicolide applied as a suspension concentrate containing 20.4 % w/w fluopicolide, was initiated at two trial sites at sites near Plusha, Cornwall, UK and Senas, Southern France. The formulated material was applied once, at the rate required to achieve an application of 500 g/ha of fluopicolide. The nominal application rate was confirmed by measuring the unused formulation remaining in the spray tank to calibrate the amount applied (500 g/ha). The test item was applied on the 24 June 1999 at Senas and on the 7 July 1999 at Plusha (as already stated the trial at Plusha was terminated and no analytical data was reported for this site).

Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of the herbicides glyphosate and simazine to control weeds.

The weather conditions were monitored in a secure location at a distance of approximately 2 kilometres from the test site. Rainfall, air temperature and soil temperature were measured daily.

Soil dissipation of fluopicolide was studied for 716 days.

2. Sampling

Soil cores (5 cm diameter) were taken to a depth of 10 cm and 30 cm during the first year. It was not possible to sample to 30 cm depth for the first 6 months as the soil was dry and hard. This was not thought to have a detrimental effect on the study as the soil conditions indicated that due to the lack of water, movement of residues to depth was unlikely. After 6 months soil cores (2.5 cm diameter) were taken to a depth of 30 cm until the end of the study. At the final two sampling points 21 and 24 months additional soil cores (2.5 cm diameter) were taken to a depth of 45 cm. Field samples were frozen after

sampling and dispatched frozen to the analytical laboratories in the UK and France. The samples were then stored at -18 ºC until required for analysis.

3. Analytical Procedures

The analytical method AR 265-01 was used to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000, referred to in the report as RPA 427967). Soil samples of 20 g were extracted twice at ambient temperature for 5 minutes by mechanical agitation using acetonitrile/water (70/30, *v/v*) acidified with 0.1% formic acid. After each extraction step, extract and soil were separated by centrifugation and decantation. The soil extracts were combined and diluted with acidified water to result in a final solvent of acetonitrile/water (30/70) with 0.1% formic acid. Quantification was carried out by LC-MS/MS using external standardisation for the parent compound and its metabolites. The limit of quantification (LOQ) was 0.005 mg/kg for each analyte.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide and reference items M-01, M-02 and M-03 at levels of LOQ and 100 x LOQ and processed in parallel to the dissipation samples. The mean recoveries of LOQ and 100 x LOQ were 97 and 99% (RSD 14.9 and 10.6%) for fluopicolide, 106 and 105% (RSD 5.9 and 9.6%) for M-01, 92 and 96% (RSD 8.9 and 4.4%) for M-02 and 95 and 91% (RSD 15.7 and 8.4%) for M-03.

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.2.1/24. A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each site between the SFO and FOMC fits for fluopicolide. The comparison of the SFO and FOMC fits suggested bi-phasic decline, and the DFOP model was therefore also fitted. For the Senas site, DFOP provided the best fit to the residues, with the lowest χ^2 error% value, and was therefore accepted.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) in soil samples by HPLC-MS/MS.

B. Data

The results for fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots in Table 7.1.2.2.1- 22 to Table 7.1.2.2.1- 25.

Table 7.1.2.2.1- 22: Residues of fluopicolide in different depths of soil at Senas, Provence, France values expressed as mg/kg

Depth [cm]	Sub plot	DAT															
		-1	0	1	3	14	28	60	130	181	231	300	368	459	554	630[1]	716
0-10	1	<LOQ	0.189	0.242	0.195	0.174	0.151	0.152	0.095	0.104	0.091	0.076	0.043	0.040	0.032	0.017	0.017
	2	<LOQ	0.239	0.168	0.171	0.121	0.161	0.095	0.061	0.101	0.073	0.055	0.050	0.040	0.021	0.012	0.014
	3	<LOQ	0.226	0.193	0.161	0.150	0.150	0.117	0.069	0.087	0.080	0.067	0.045	0.031	0.022	0.014	0.024
	mean	**<LOQ**	**0.218**	**0.201**	**0.176**	**0.148**	**0.154**	**0.121**	**0.075**	**0.097**	**0.081**	**0.066**	**0.046**	**0.037**	**0.025**	**0.015**	**0.018**
10-20	1	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-45	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	mean	-	-	-	-	-	-	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**

DAT: days after treatment
LOQ (limit of quantitation) = 0.005 mg/kg,
[1]Cores were analysed as 0-15 cm, 15-25 cm, 25-35 cm and 35-45 cm horizons

Table 7.1.2.2.1- 23: Residues of M-01 (AE C653711) in different depths of soil at Senas, Provence, France values expressed as mg/kg

Depth [cm]	Sub plot	DAT															
		-1	0	1	3	14	28	60	130	181	231	300	368	459	554	630[1]	716
0-10	1	<LOQ	<LOQ	0.015	0.014	0.022	0.018	0.029	0.016	0.016	0.017	0.011	0.016	0.017	0.005	<LOQ	0.006
	2	<LOQ	<LOQ	0.010	0.013	0.012	0.023	0.021	0.015	0.017	0.017	0.014	0.020	0.020	0.003	<LOQ	0.003
	3	<LOQ	<LOQ	0.011	0.011	0.015	0.021	0.027	0.017	0.017	0.019	0.014	0.019	0.020	0.003	**<LOQ**	0.006
	mean	**<LOQ**	**<LOQ**	**0.012**	**0.013**	**0.016**	**0.021**	**0.026**	**0.016**	**0.017**	**0.018**	**0.013**	**0.018**	**0.019**	**0.004**	<LOQ	**0.005**
10-20	1	<LOQ	-	-	-	-	-	-	-	-	0.008	0.008	<LOQ	<LOQ	0.007	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	-	-	-	-	0.007	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	-	-	-	-	0.009	0.009	0.007	<LOQ	0.005	0.005	<LOQ
	mean	**<LOQ**	-	-	-	-	-	-	-	-	**0.008**	**0.006**	**0.004**	**<LOQ**	**0.005**	**0.004**	**<LOQ**
20-30	1	<LOQ	-	-	-	-	-	-	-	-	<LOQ	0.006	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	-	-	-	-	-	-	-	-	**<LOQ**	**0.004**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-45	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	mean	-	-	-	-	-	-	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**

DAT: days after treatment
LOQ (limit of quantitation) = 0.005 mg/kg,
[1]Cores were analysed as 0-15 cm, 15-25 cm, 25-35 cm and 35-45 cm horizons

Table 7.1.2.2.1- 24: Residues of M-02 (AE C657188) in different depths of soil at Senas, Provence, France values expressed as mg/kg

Depth [cm]	Sub plot	DAT															
		-1	0	1	3	14	28	60	130	181	231	300	368	459	554	630[1]	716
0-10	1	<LOQ	<LOQ	0.007	0.010	0.026	0.020	0.017	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	0.007	0.009	0.013	0.020	0.017	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	0.008	0.008	0.016	0.023	0.016	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**0.007**	**0.009**	**0.018**	**0.021**	**0.017**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-45	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	mean	-	-	-	-	-	-	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**

DAT: days after treatment
LOQ (limit of quantitation) = 0.005 mg/kg
[1]Cores were analysed as 0-15 cm, 15-25 cm, 25-35 cm and 35-45 cm horizons

Table 7.1.2.2.1- 25: Residues of M-03 (AE 0608000) in different depths of soil at Senas, Provence, France values expressed as mg/kg

Depth [cm]	Sub plot	DAT															
		-1	0	1	3	14	28	60	130	181	231	300	368	459	554	630[1]	716
0-10	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-45	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ
	mean	-	-	-	-	-	-	-	-	-	-	-	-	-	-	**<LOQ**	**<LOQ**

DAT: days after treatment
LOQ (limit of quantitation) = 0.005 mg/kg
[1]Cores were analysed as 0-15 cm, 15-25 cm, 25-35 cm and 35-45 cm horizons

C. Residues

Analysis of control samples showed no residues above the limit of quantification (LOQ) of 0.005 mg/kg.

The average initial concentration of fluopicolide in soil samples taken immediately after application was 0.218 mg/kg (range 0.189 to 0.239 mg/kg). This corresponds to an apparent application rate of 327 g/ha which is lower than the nominal application rate of 500 g/ha.

The initial dissipation of fluopicolide was relatively rapid followed by a slower dissipation phase during the winter months due to the cold climate. Dissipation continued the following summer at a slower rate than the initial rapid phase possibly due to the reduced availability of fluopicolide due to increased adsorption to soil with ageing. Residues of fluopicolide were detected only in the 0-10 cm soil horizon throughout the trial. At all other soil depths no residues of fluopicolide were found above the LOQ.

The concentration of the metabolite AE C653711 in soil increased rapidly in the two months after application and then remained fairly constant throughout the first year due to its constant formation from the degradation of parent, before declining rapidly in the second year. The maximum residue level in the 0-10 cm horizon was observed 60 days after application at 0.026 mg/kg (mean of three replicates). This concentration represents the maximum formation of M-01 in terrestrial field dissipation studies (24.1%**).** The maximum residue in the underlying 10-20 cm was detected at 240 days after application at 0.008 mg/kg (mean values). In the 20-30 cm horizon residue levels were below the LOQ except for one replicate at 300 days in which AE C653711 residues were detected at 0.006 mg/kg. No residues were detected in 30-45 cm depth above the LOQ. By the end of the study after two years, residue levels had declined to the LOQ.

M-02 (AE C657188) was detected at timepoints up to 60 days after application in the 0-10 cm layer at a maximum concentration of 0.021 mg/kg (mean value) 28 days after application. This concentration represents the maximum formation of M-02 in terrestrial field dissipation studies (16.4%**).** No residues of M-02 were detected in deeper soil depths above the LOQ.

No residues of M-03 (AE 0608000) were found above the LOQ throughout the study. The degradation of M-03 is known to be pH dependent and is very rapidly degraded in neutral to alkaline soils such as the soil at the Senas trial site.

D. Kinetic Analysis

The half-life of fluopicolide included in the report was calculated using a simple first-order kinetic model (SFO) as 131 days. The DT_{50} was 434 days. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for Document KCA 7.1.2.2.1/24. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.2.1- 6. Best fit kinetics are highlighted in bold.

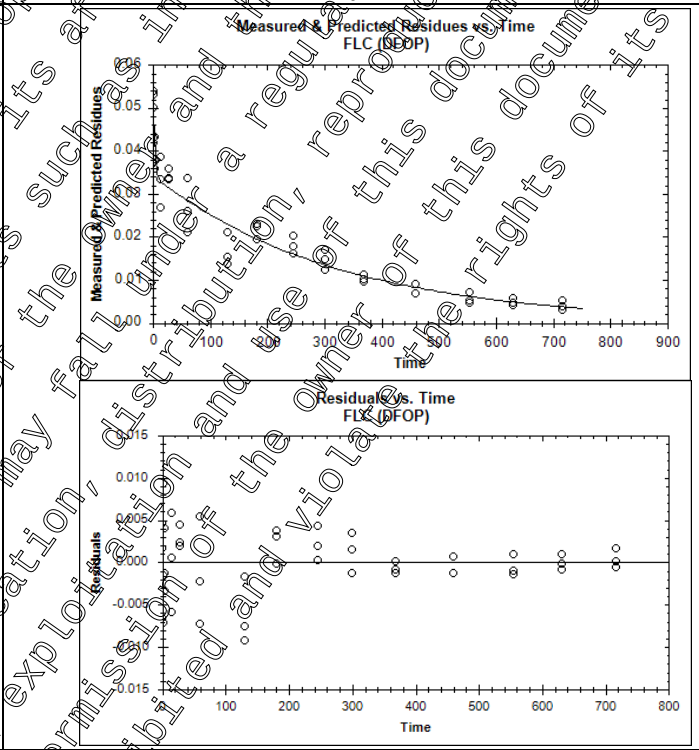
Table 7.1.2.2.1- 26: Degradation rate of fluopicolide under field conditions (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Senas (France), CA 7.1.2.2.1/04, ([redacted] 2003)	SFO	0.04026	k 0.003881	14.3	1.18E-14	0.003203	0.005	178.6	593.3
	FOMC	0.0436	α 0.8025 β 76.06	11.8	n.r. n.r.	0.3857 -1.463	1.219 153.6	104.4	1264
	DFOP	**0.04855**	**k1 0.3279 k2 0.00311 g 0.2965**	**7.9**	**0.0207 4.90E-14 n.r.**	**0.02257 0.002553 0.2113**	**0.633 0.004 0.382**	**109.8**	**627.2**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.2.1- 27: Graphical representations of best fit model

Trial / Best Fit Model	Graphical Representations
Senas (France), DFOP	

III. Conclusion

Following a single application of fluopicolide at a nominal application rate of 500 g/ha to bare soil in summer 1999, the decline of fluopicolide and the formation and decline of its metabolites M-01, M-02 and M-03 was followed for up to 740 days after application at a site in Senas, Southern France. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit DFOP un-normalised DT_{50} value of 109.8 days and DT_{90} value of 627.2 days for fluopicolide.

The metabolites M-01 (AE C653711) and M-02 (AE C657188) were detected in Senas soil. M-01 was detected at low concentrations in 0-10 cm, 10-20 cm and once in the 20-30 cm soil depths. M-02 was detected only at early timepoints in the 0-10 cm depth. No residues of M-03 were detected throughout the study.

Assessment and conclusion by applicant:

The study is considered valid to assess the dissipation of fluopicolide under field conditions in soil. The study meets the requirements to assess field persistence of fluopicolide and its metabolites, and to derive parent soil $DegT_{50matrix}$ values for legacy field studies as defined by EFSA (2014). It is not suitable for assessing metabolite soil $DegT_{50matrix}$ values as the design did not minimise soil surface processes immediately after application as required by EFSA (2014).

Data Point:	KCA 7.1.2.2.1/05
Report Author:	[redacted]
Report Year:	2003
Report Title:	Field soil dissipation of AE C638206 following a single application and multiple applications to bare soil plots at 1 location in France, 2000 Interim report
Report No:	C037583
Document No:	M-223191-01-1
Guideline(s) followed in study:	BBA: Part IV-4-1; EU (=EEC): Anonymous, 1997; IVA: (1993); SETAC: (1995)
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

For procedural reasons the previously submitted interim report is included under Point KCA 7.1.2.2.1 in the current dossier (KCA 7.1.2.2.1/05). However the interim report has been fully superseded by the final report which was also previously submitted as part of the original EU approval (KCA 7.1.2.2.1/08). Consequently no summary of the interim report has been included in this dossier. A summary of the final report has been provided under KCA 7.1.2.2.1/08.

Data Point:	KCA 7.1.2.2.1/06
Report Author:	[redacted]
Report Year:	2004
Report Title:	Field soil dissipation of AE C638206 following a single application and multiple applications to bare soil plots at 1 location in Germany, 2000
Report No:	C037584
Document No:	M-223195-01-1
Guideline(s) followed in study:	BBA: Part IV, 4-1; EU (=EEC): Anonymous, 1997; IVA: (1993); SETAC: (1995)
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

For procedural reasons the previously submitted interim report is included under Point KCA 7.1.2.2.1 in the current dossier (KCA 7.1.2.2.1/06). However the interim report has been fully superseded by the final report which was also previously submitted as part of the original EU approval (KCA 7.1.2.2.1/09). Consequently no summary of the interim report has been included in this dossier. A summary of the final report has been provided under KCA 7.1.2.2.1/09.

Data Point:	KCA 7.1.2.2.1/07
Report Author:	[redacted]
Report Year:	2003
Report Title:	Kinetic evaluation of field dissipation studies after application of AE C638206 using ModelMaker, Pearl and Pest Codes: AE C638206, AE 0608000, AE C653711, AE C657188
Report No:	C034324
Document No:	M-234722-01-1
Guideline(s) followed in study:	-
Deviations from current test guideline:	none
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

For procedural reasons the previously submitted modelling report is included under Point KCA 7.1.2.2.1 in the current dossier (KCA 7.1.2.2.1/07). However the report has been fully superseded by two new kinetic evaluation reports of the original field dissipation trials (KCA 7.1.2.2.1/22, M-685676-01-1 and KCA 7.1.2.2.1/24, M-685682-01-1). Consequently no summary of this report has been included in this dossier.

Data Point:	KCA 7.1.2.2.1/08
Report Author:	[redacted]
Report Year:	2005
Report Title:	Field soil dissipation of AE C638206 following a single application and multiple applications to bare soil plots at 1 location in France, 2000
Report No:	C048340
Document No:	M-251338-01-1
Guideline(s) followed in study:	BBA: 1986; EU (=EEC): 1999; IVA: 1993; SETAC: 1995
Deviations from current test guideline:	Yes. Report meets the requirement for field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013 and the requirements for assessing parent soil DegT50matrix values as required by EFSA (2014) for legacy field studies. Report does not meet the requirement for assessing metabolite soil DegT50matrix values as required by EFSA (2014) for field studies.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities.
Acceptability/Reliability:	Yes

Executive Summary

A four year terrestrial field dissipation and accumulation study with fluopicolide, formulated as a suspo-emulsion containing 97.9 g/L fluopicolide (AE C638206.00 SE10 A3), has been conducted at a site at Appilly in the Picardie region in Northern France. The top soil at the test site was a sandy silt soil. The formulated material was applied once a year, at the rate required to achieve an annual application of 400 g/ha of fluopicolide using a calibrated boom sprayer to bare soil. Nominal application rates were confirmed by measuring the unused formulation remaining in the spray tank to calibrate the amount applied. The initial application was on 16 June 2000 with subsequent applications on the 27 August 2001, 17 July 2002, 18 June 2003 and 30 June 2004. Samples of soil have been taken at intervals over a four year period and analysed by an LC/MS/MS method to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) present in the samples.

The initial dissipation of fluopicolide was relatively rapid followed by a slower dissipation phase during the winter months due to the cold climate and possibly reduced availability of fluopicolide due to increased adsorption to soil with ageing. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. Low residues were detected in the 10-20 cm horizon at concentrations ranging from 0.002 to 0.008 mg/kg (mean values). Residue levels of parent declined to 76 g/ha two years after the first treatment which represented approximately 20 % of the initial concentration. Throughout the dissipation phase no residues of fluopicolide were detected above the LOQ below 20 cm depth.

The metabolite M-01 (AE C653711) was detected in 0-10 cm soil depth and occasionally at low levels in 10-20 cm depth in the dissipation plot. The maximum residue level was observed 31 days after application at 0.017 mg/kg (mean value) equivalent to 25 g/ha. In the 20-30 cm horizon and deeper soil layers residue levels were below the LOQ throughout the dissipation phase.

M-02 (AE C657188) and M-03 (AE 0608000) were rapidly degraded in the trial. Residues of M-02 were only detected at two early time points in 0-10 cm soil depth in the dissipation plot. The maximum residue observed was 0.017 mg/kg (mean value) equivalent to 25.5 g/ha 14 days after the initial application. No residues of M-02 were detected above the LOQ below 10 cm depth. No residues of M-03 were found above the LOQ throughout the study. The degradation of M-03 is known to be pH dependent and is very rapidly degraded in neutral to alkaline soils such as the soil at the Appilly trial site.

A re-evaluation of the degradation kinetics of the dissipation phase of the trial in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit DFOP un-normalised DT_{50} value of 143.4 days and DT_{90} of 1695.0 days for fluopicolide

The plateau concentrations of fluopicolide and M-01 after four years are summarised below.

Plateau concentration	Time-point	Fluopicolide (mg/kg)		Time-point	M-01 (mg/kg)	
		0-10 cm	0-20 cm		0-10 cm	0-20 cm
High[1]	Day 0 5th Application	0.387	0.199	Day 120 after 4th Application	0.036	0.026
Low[2]	Day 378 after 4th Application	0.144	0.080	Day 0 after 5th Application	0.034	0.025

[1] maximum of the high values of the "saw teeth" curve
[2] maximum of the low values of the "saw teeth" curve

It was stated in Addendum 1 to the DAR (2007) that it was inconclusive whether fluopicolide residues had reached plateau concentrations in the Appilly trial. For M-01 residues were lower but did not appear to reach a plateau either.

Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the accumulation phase. Low residues were detected in the 10-20 cm soil depth at concentrations ranging from 0.006 to 0.019 mg/kg (mean values) throughout the accumulation phase and in the 20-30 cm depth at concentrations ranging from 0.005 to 0.013 mg/kg (mean values). In the 30-50 cm soil depth fluopicolide was detected above the LOQ at one time-point at a concentration of 0.010 mg/kg which was possibly the consequence of smearing during sampling.

The metabolite M-01 was detected in 0-10 cm soil depth at a maximum concentration of 0.036 mg/kg (mean value). Levels in the 10-20 and 20-30 cm soil depths were lower with maximum values of 0.020 mg/kg (mean value) and 0.011 mg/kg (mean value), respectively. In deeper soil layers residue levels of M-01 were below the LOQ, except in a single time-point in the 30-50 cm soil layer in which a mean concentration of 0.006 mg/kg was detected.

No residues of M-02 or M-03 were detected throughout the accumulation phase.

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide formulated as liquid suspo-emulsion (fluopicolide)

Year of application:	2000 and 2001	2002 and 2003	2004
Content of a.s.:	97.9 g/L	97.5 g/L	100 g/L
Certificate of Analysis:	AGF2000-0106-01	AGF2001-0227-01	AGF2003-0035-01
Lot No:	OP200271	OP210914	OP220828

2. Trial Location & Soil

A four year terrestrial field dissipation and accumulation study with fluopicolide, formulated as a suspo-emulsion containing 97.9 g/L fluopicolide (AE C638206 00 SE10 A3), has been conducted at a site at Appilly in the Picardie region in Northern France. The top soil was a sandy silt soil (DIN classification) with sandy silt / loamy silt subsoil. The characteristics of the soil are summarised in Table 7.1.2.2.1- 28. Three experimental plots, each measuring 3 metres by 26 metres (234 m² in total), were treated with the test substance. A fourth plot measuring 3 metres by 12 metres was left untreated to provide control samples. The treated plots were subdivided into separate areas for the dissipation phase treated once in 2000 and for the accumulation phase treated up to five times in 2000, 2001, 2002, 2003 and 2004.

Table 7.1.2.2.1- 28: Location, site description and climatic data of test site

Characteristic	Units	Appilly, Picardie, France		
		Horizon 1	Horizon 2	Horizon 3
Sampling depth	cm	0 - 20	20 - 50	50 - 90
pH	$CaCl_2$	7.1	7.2	6.8
Cation exchange capacity	mval/100g	19.7	18.8	20.4
Total organic carbon (TOC)	%	1.51	1.41	0.44
Biomass 2000	mg C/100 g	24.4	n.d.	n.d.
2002	mg C/100 g	25.2	n.d.	n.d.
2003	mg C/100 g	15.5	n.d.	n.d.
2004	mg C/100 g	30.0	n.d.	n.d.
Soil Density	g/L	1200	1480	1410
Particle size distribution (DIN)				
Clay < 0.002 mm	%	32.0	23.2	21.0
Total silt 0.002 - 0.063 mm	%	62.6	69.4	69.6
Total sand 0.063 - 2 mm	%	5.4	7.4	9.4
Textural class	DIN	Sandy silt	Sandy silt	Loamy silt
Water Holding Capacity	Vol % at 1/10 bar (pF2)	34.70	38.96	35.30
	Vol % at 15 bar (pF4.2)	11.77	14.56	17.84

n.d. = not determined

B. Study Design

1. Experimental Conditions

The formulated material was applied once a year, at the rate required to achieve an annual application of 400 g/ha of fluopicolide using a calibrated boom sprayer. Nominal application rates were confirmed by measuring the unused formulation remaining in the spray tank to calibrate the amount applied (397 g/ha for the first application). The initial application was on 16 June 2000 with subsequent applications on the 27 August 2001, 17 July 2002, 18 June 2003 and 30 June 2004.

All applications were made to bare soil. Throughout the study the plots were maintained as bare soil by the periodic application of the herbicide glyphosate to control weeds.

The following weather conditions were monitored continuously at the test site; rainfall, air temperature, relative air humidity, soil temperature and soil moisture content at 10 cm depth. Wind speed and global radiation were taken from the regional official weather service (Chauny, [redacted] or St. Quentin, [redacted]).

2. Sampling

Soil cores (5 cm diameter) for the dissipation phase were taken immediately after treatment, 1 day, 3 days , 14 days and 1, 2, 4, 6, 8, 10, 12, 15, 18 and 24 months after the first application. Samples for the accumulation phase were taken immediately after each treatment and additionally approximately 4 and 12 months after the second, third and fourth applications in 2001, 2002 and 2003. The final sample was taken immediately after the fifth application in 2004. Soil cores for the dissipation phase were taken to a depth of 30 cm during the first month and to a depth of 50 cm at time-points up to two years. Soil cores for the accumulation phase were taken to a depth of 50 cm following the second application and up to 4 months after the third. At timepoints after this, soil cores were taken to a depth of 90 cm. At each sampling date 7 samples from each plot were taken (21 cores in total). Field samples were frozen immediately after sampling and shipped frozen to GAB Biotechnologie GmbH, Germany. The soil samples from the same horizon of each plot were blended in Germany and a subsample dispatched frozen to the Bayer CropScience analytical laboratory in France. The samples were then stored at -18 ºC until required for analysis.

3. Analytical Procedures

The analytical method AR 265-01 was used to determine levels of fluopicolide and its metabolites M-01 (AE C653712), M-02 (AE C657188), and M-03 (AE 0608000 referred to in the report as RPA 427967). Soil samples of 20 g were extracted twice at ambient temperature for 5 minutes by mechanical agitation using acetonitrile/water (70/30, v/v) acidified with 0.1% formic acid. After each extraction step, extract and soil were separated by centrifugation and decantation. The soil extracts were combined and diluted with acidified water to result in a final solvent of acetonitrile/water (30/70) with 0.1% formic acid. Quantification was carried out by LC-MS/MS using external standardisation for the parent compound and its metabolites. The limit of quantification (LOQ) was 0.005 mg/kg for each analyte.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide and reference items M-01, M-02 and M-03 at levels of LOQ and 100 x LOQ and processed in parallel to the dissipation samples. The mean recoveries of LOQ and 100 x LOQ were 100 and 96% (RSD 12.2 and 5.5%) for fluopicolide, 104 and 98% (RSD 10.4 and 80.5%) for M-01, 95 and 91% (RSD 10.6 and 8.5%) for M-02 and 96 and 88% (RSD 10.1 and 10.5%) for M-03. No residues of fluopicolide or its metabolites were found above the LOQ in the analysed untreated samples.

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.2.1/24. A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each site between the SFO and FOMC fits for fluopicolide. The comparison of the SFO and FOMC fits suggested bi-phasic decline, and the DFOP model was therefore also fitted. For the Appilly site, the DFOP model was accepted the as best fit to the residues, despite a lack of confidence in the optimised rate constants, as DFOP kinetics provided the best visual description of the decline. The FOMC fit was not accepted, as extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using this model (EFSA, 2009). It is noted that the estimated DT_{90} exceeded the relevant regulatory triggers for all models.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) in soil samples by HPLC-MS/MS.

B. Data

The results for fluopicolide and its metabolites M-01 (AE C653711) and M-02 (AE C657188) are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots in Table 7.1.2.2.1- 29 to Table 7.1.2.2.1- 34. No residues of M-03 (AE 0608000) were detected throughout the trial.

The levels of residues for each analyte in the three replicate treated plots were generally in good agreement throughout the trial except after the third application on 17 July 2002 and also to some extent after the fourth and fifth applications in 2003 and 2004. Fluopicolide residue levels immediately after the third application on 17 July 2002 were T1=0.278 mg/kg, T2=0.338 mg/kg and T3=0.602 mg/kg. The Plot T3 replicate was discarded as an outlier as the concentration was significantly higher than in the other plots. The dissipation and accumulation of fluopicolide (mean values and individual plots) and M-01 (mean values) at Appilly are presented in Figure 7.1.2.2.1- 1 and Figure 7.1.2.2.1- 2. In order to calculate mean values, concentrations <LOQ (0.005 mg/kg) were assumed to be 0 mg/kg. Where individual replicate values exceeded the LOQ the calculated mean concentration has been reported, including mean values that are below the LOQ. For the conversion of mg/kg into g/ha a soil density of 1.5 g/cm^3 was used.

Table 7.1.2.2.1- 29: Residues of fluopicolide in soil after an application of 400 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Depth [cm]	Sub plot	DAA1													
		0	1	3	14	31	62	136	187	245	309	370	483	584	735
0-10	1	0.291	0.273	0.207	0.377	0.242	0.162	0.102	0.095	0.088	0.078	0.087	0.011	0.037	0.043
	2	0.232	0.203	0.255	0.136	0.273	0.136	0.108	0.073	0.075	0.098	0.045	0.037	0.039	0.037
	3	0.244	0.161	0.171	0.162	0.279	0.156	0.084	0.090	0.093	0.087	0.078	0.042	0.037	0.050
	mean	**0.256**	**0.212**	**0.211**	**0.225**	**0.265**	**0.151**	**0.098**	**0.086**	**0.085**	**0.088**	**0.070**	**0.041**	**0.038**	**0.043**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.011	<LOQ	0.006	<LOQ	<LOQ	0.008
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.006	0.008	<LOQ	<LOQ	<LOQ	0.006
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.006	<LOQ	<LOQ	0.006	0.011	<LOQ	<LOQ	<LOQ	0.008
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.002[1]**	**<LOQ**	**<LOQ**	**0.008**	**0.006[1]**	**0.002[1]**	**<LOQ**	**<LOQ**	**0.007**
20-30	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**<LOQ**
30-50	1	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**<LOQ**

DAA: days after application
n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value <LOQ

Table 7.1.2.2.1- 30: Residues of fluopicolide in soil after annual applications of 400 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3			DAA4			DAA5
		0	147	361	0	128	336	0	120	378	0
0-10	1	0.196	0.122	0.087	0.278	0.186	0.129	0.408	0.185	0.125	0.457
	2	0.245	0.192	0.088	0.338[2]	0.190	0.140	0.293	0.190	0.143	0.235
	3	0.222	0.165	0.101	0.602	0.172	0.145	0.312	0.163	0.164	0.469
	mean	**0.221**	**0.160**	**0.092**	**0.308**	**0.183**	**0.138**	**0.338**	**0.179**	**0.144**	**0.387**
10-20	1	0.040	0.018	0.012	0.012	0.015	0.019	0.017	0.031	0.023	<LOQ
	2	0.080	0.020	0.008	0.009	0.020	0.020	0.012	0.011	0.007	0.011
	3	0.010	0.020	0.012	0.023	0.009	0.016	0.011	0.010	0.017	0.008
	mean	**0.019**	**0.019**	**0.011**	**0.015**	**0.015**	**0.018**	**0.013**	**0.017**	**0.016**	**0.006**
20-30	1	<LOQ	0.015	0.007	0.006	0.009	0.006	0.010	<LOQ	<LOQ	<LOQ
	2	<LOQ	0.009	0.007	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	0.014	0.008	0.012	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**0.013**	**0.007**	**0.006**	**0.005**	**0.007**	**0.005**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	<LOQ	0.010	<LOQ	<LOQ	<LOQ	0.006	0.006	<LOQ	<LOQ	<LOQ
	2	<LOQ	0.007	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	0.012	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**0.010**	**<LOQ**	**<LOQ**	**<LOQ**	**0.002**	**0.002**	**<LOQ**	**<LOQ**	**<LOQ**
50-70	1	-	-	-	-	-	<LOQ	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	<LOQ	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	<LOQ	n.a	n.a	n.a	n.a
	mean	-	-	-	-	-	**<LOQ**	**n.a**	**n.a**	**n.a**	**n.a**
70-90	1	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	mean	-	-	-	-	-	**n.a**	**n.a**	**n.a**	**n.a**	**n.a**

DAA: days after application, n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value > LOQ, [2] mean of two replicates

Table 7.1.2.2.1- 31: Residues of M-01 (AE C653711) in soil after an application of 400 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Depth [cm]	Sub plot	DAA1													
		0	1	3	14	31	62	136	187	245	309	370	483	584	735
0-10	1	<LOQ	<LOQ	0.008	0.021	0.018	0.013	0.009	0.008	0.007	0.006	0.011	0.010	0.006	0.009
	2	<LOQ	<LOQ	0.009	0.010	0.018	0.012	0.009	0.006	0.006	0.003	0.007	0.008	0.006	0.008
	3	<LOQ	<LOQ	0.007	0.012	0.014	0.0013	0.008	0.006	0.006	0.007	0.011	0.008	0.006	0.010
	mean	**<LOQ**	**<LOQ**	**0.008**	**0.014**	**0.017**	**0.013**	**0.009**	**0.007**	**0.006**	**0.006**	**0.010**	**0.009**	**0.006**	**0.009**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.006	<LOQ	<LOQ	<LOQ	<LOQ	0.008
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.006
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.008
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.002[1]**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.007**
20-30	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**<LOQ**
30-50	1	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**<LOQ**

DAA: days after application
n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value < LOQ

Table 7.1.2.2.1- 32: Residues of M-01 (AE C653711) in soil after annual applications of 400 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3			DAA4			DAA5
		0	147	361	0	128	336	0	120	378	0
0-10	1	0.013	0.013	0.021	0.017	0.027	0.022	0.015	0.044	0.029	0.036
	2	0.013	0.014	0.018	0.018	0.029	0.025	0.012	0.030	0.032	0.031
	3	0.012	0.014	0.018	0.023	0.032	0.022	0.015	0.034	0.034	0.034
	mean	**0.013**	**0.014**	**0.019**	**0.019**	**0.029**	**0.023**	**0.014**	**0.036**	**0.032**	**0.034**
10-20	1	<LOQ	0.006	0.010	0.007	0.012	0.016	0.011	0.019	0.023	0.015
	2	<LOQ	0.008	0.006	0.006	0.015	0.018	0.010	0.013	0.018	0.017
	3	<LOQ	<LOQ	0.0008	0.009	0.012	0.015	0.011	0.013	0.018	0.015
	mean	**<LOQ**	**0.005[1]**	**0.008**	**0.007**	**0.013**	**0.016**	**0.011**	**0.015**	**0.020**	**0.016**
20-30	1	<LOQ	<LOQ	0.006	0.006	0.009	0.010	0.007	0.010	0.011	0.007
	2	<LOQ	<LOQ	<LOQ	<LOQ	0.007	0.013	0.006	0.006	0.009	0.010
	3	<LOQ	<LOQ	<LOQ	0.006	0.006	0.009	0.007	0.007	0.009	0.007
	mean	**<LOQ**	**<LOQ**	**0.002[1]**	**0.004[1]**	**0.007**	**0.011**	**0.007**	**0.008**	**0.010**	**0.008**
30-50	1	-	<LOQ	<LOQ	<LOQ	<LOQ	0.011	0.006	<LOQ	<LOQ	<LOQ
	2	-	<LOQ	<LOQ	<LOQ	<LOQ	0.007	<LOQ	<LOQ	<LOQ	<LOQ
	3	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.006[1]**	**0.002[1]**	**<LOQ**	**<LOQ**	**<LOQ**
50-70	1	-	-	-	-	-	<LOQ	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	<LOQ	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	<LOQ	n.a	n.a	n.a	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**n.a**	**n.a**	**n.a**	**n.a**
70-90	1	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**n.a**	**n.a**	**n.a**	**n.a**	**n.a**

DAA: days after application, n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value > LOQ

Table 7.1.2.2.1- 33: Residues of M-02 (AE C657188) in soil after an application of 400 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Depth [cm]	Sub plot	DAA1													
		0	1	3	14	31	62	136	187	245	309	370	483	584	735
0-10	1	<LOQ	<LOQ	0.009	0.023	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	0.010	0.014	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	0.008	0.014	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**0.009**	**0.017**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**<LOQ**
30-50	1	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.a	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**<LOQ**

DAA: days after application
n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value < LOQ

Table 7.1.2.2.1- 34: Residues of M-02 (AE C657188) in soil after annual applications of 400 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3			DAA4			DAA5
		0	147	361	0	128	336	0	120	378	0
0-10	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
50-70	1	-	-	-	-	-	<LOQ	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	<LOQ	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	<LOQ	n.a	n.a	n.a	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**n.a**	**n.a**	**n.a**	**n.a**
70-90	1	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**n.a**	**n.a**	**n.a**	**n.a**	**n.a**

DAA: days after application
n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, - Replicate value > LOQ

C. Residues

Dissipation phase (2000 – 2002)

The theoretical initial concentration of fluopicolide in the 0-10 cm layer was 0.265 mg/kg based on the calibrated application rate of 397 g/ha, assuming a soil density of 1.5 g/cm^3. The average initial concentration of fluopicolide in the soil samples taken immediately after application was 0.256 mg/kg (range 0.232 to 0.291 mg/kg). This corresponds to an apparent application rate of 383.5 g/ha which is in good agreement with the nominal application rate of 400 g/ha. The initial dissipation of fluopicolide was relatively rapid followed by a slower dissipation phase during the winter months due to the cold climate and possibly reduced availability of fluopicolide due to increased adsorption to soil with ageing. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. Low residues were detected in the 10-20 cm horizon at concentrations ranging from 0.002 to 0.008 mg/kg (mean values). Residue levels of parent declined to 76 g/ha two years after the first treatment which represented approximately 20 % of the initial concentration. Throughout the dissipation phase no residues of fluopicolide were detected above the LOQ below 20 cm depth.

The metabolite M-01 (AE C653711) was detected in 0-10 cm soil depth and occasionally at low levels in 10-20 cm depth. The maximum residue level was observed 31 days after application at 0.017 mg/kg (mean value) equivalent to 25 g/ha. In the 20-30 cm horizon and deeper soil layers residue levels were below the LOQ throughout the dissipation phase.

M-02 (AE C657188) and M-03 (AE 0608000) were rapidly degraded in the trial. Residues of M-02 were only detected at two early time-points in 0-10 cm soil depth. The maximum residue observed was 0.017 mg/kg (mean value) equivalent to 25.5 g/ha 14 days after the initial application. No residues of M-02 were detected above the LOQ below 10 cm depth. No residues of M-03 were found above the LOQ throughout the study. The degradation of M-03 is known to be pH dependent and is very rapidly degraded in neutral to alkaline soils such as the soil at the Appilly trial site.

Accumulation:

The maximum average concentration of fluopicolide in soil was 0.387 mg/kg in 0 to 10 cm soil depth immediately after the fifth application in 2004. Although the upper limit of the 'saw teeth' curve still appears to increase the plateau concentration at the lower limit appears to have been reached (see Figure 7.1.2.2.1-1). The apparent increase in the upper limit could be a result of variations in concentration immediately after application. Variation between replicate plots is very much less for the lower limit of the 'saw teeth' curve.

Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the accumulation phase. Low residues were detected in the 10-20 cm soil depth at concentrations ranging from 0.006 to 0.019 mg/kg (mean values) throughout the accumulation phase and in the 20-30 cm depth at concentrations ranging from 0.005 to 0.013 mg/kg (mean values). In the 30-50 cm soil depth fluopicolide was detected above the LOQ at one time-point at a concentration of 0.010 mg/kg which was possibly the consequence of smearing during sampling.

The metabolite M-01 was detected in 0-10 cm soil depth at a maximum concentration of 0.036 mg/kg (mean value). Levels in the 10-20 and 20-30 cm soil depths were lower with maximum values of 0.020 mg/kg (mean value) and 0.011 mg/kg (mean value), respectively. In deeper soil layers residue levels of M-01 were below the LOQ, except in a single time-point in the 30-50 cm soil layer in which a mean concentration of 0.006 mg/kg was detected.

M-02 was detected only at early time-points after the initial application during the dissipation phase of the trial. No residues of M-02 were detected throughout the accumulation phase. No residues of M-03 were detected throughout the dissipation or accumulation phases of the trial.

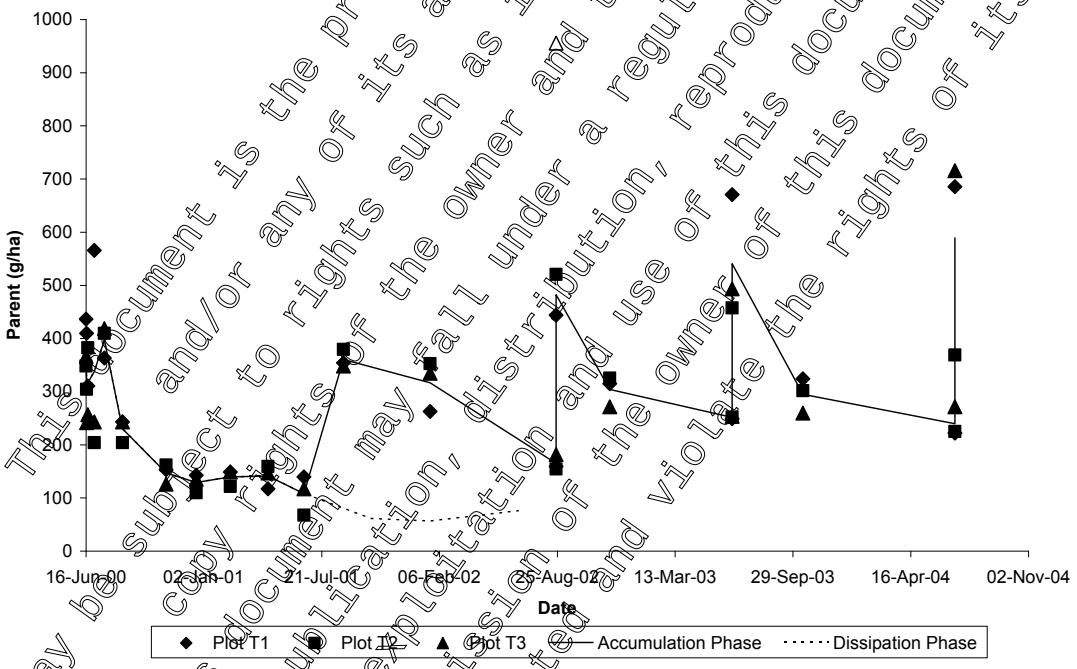
The plateau concentrations of fluopicolide and M-01 after four years are summarised below.

Plateau concentration	Time-point	Fluopicolide (mg/kg)		Time-point	M-01 (mg/kg)	
		0-10 cm	0-20 cm		0-10 cm	0-20 cm
High[1]	Day 0 5th Application	0.387	0.199	Day 120 after 4th Application	0.036	0.026
Low[2]	Day 378 after 4th Application	0.144	0.080	Day 0 after 5th Application	0.034	0.025

[1] maximum of the high values of the "saw teeth" curve
[2] maximum of the low values of the "saw teeth" curve

It was stated in Addendum 1 to the DAR (2007) that it was inconclusive whether fluopicolide residues had reached plateau concentrations in the Appoly trial. For M-01, residues were lower but did not appear to reach a plateau either.

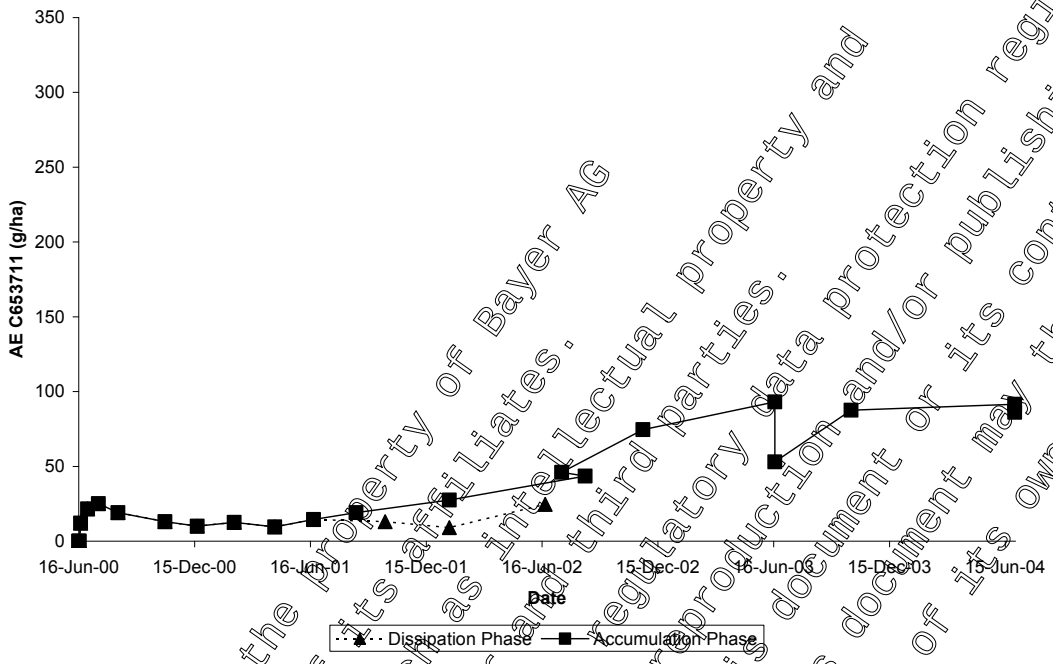
Figure 7.1.2.2.1- 1: Dissipation and Accumulation of Fluopicolide at Appilly



Mean values are given as solid and dashed lines for the accumulation phase and dissipation phase as indicated in the key. The results for individual plots are also given for fluopicolide as solid symbols. Outliers are indicated by an open symbol.

Figure 7.1.2.2.1- 2: Dissipation and Accumulation of M-01 (AE C653711) at Appilly

NB Scale different



D. Kinetic Analysis

The half-life of fluopicolide included in the report was calculated using a bi-phasic first-order kinetic model (Hockey Stick) as 99 days. The DT_{90} was 953 days and the r^2 was 0.891. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for Document KCA 7.1.2.2.1/24. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.2.1-35. Best fit kinetics are highlighted in bold.

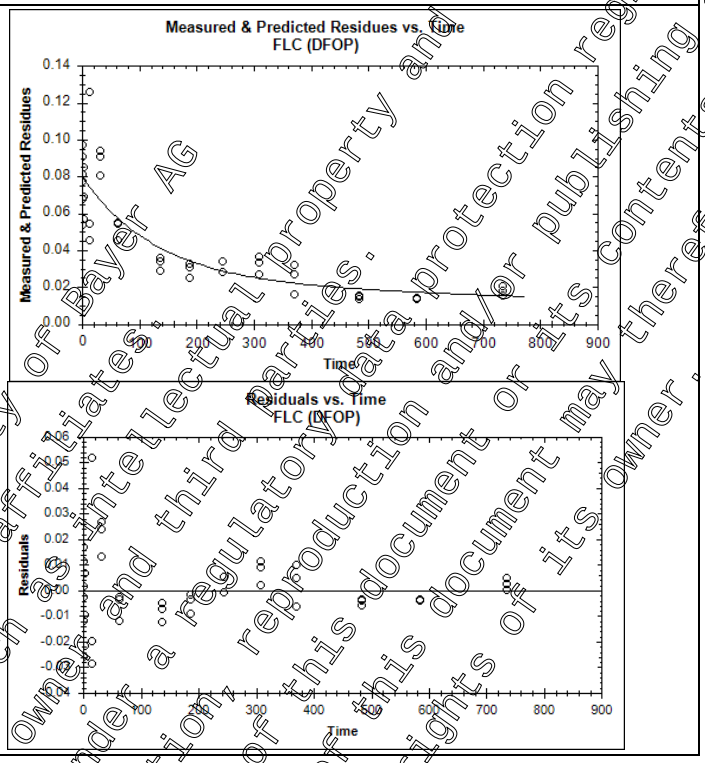
Table 7.1.2.2.1- 35: Degradation rate of fluopicolide under field conditions (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Appilly (France), CA 7.1.2.2.1/08, (Pollmann, 2005b)	SFO	0.07668	k 0.003566	16.3	7.97E-09	0.002575	0.005	194.4	645.7
	FOMC	0.07892	α 1.116 β 175.1	14.8	n.r. n.r.	-0.3699 -197.3	2.603 547.4	150.7	1202
	DFOP	**0.0799**	**k1 0.007931 k2 0.0006561 g 0.6959**	**15**	**0.1236 0.3988 n.r.**	**-0.005294 -0.004323 -0.2273**	**0.021 0.006 1.619**	**143.4**	**1695**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.2.1- 36: Graphical representations of best fit model

Trial / Best Fit Model	Graphical Representations
Appilly (France) / DFOP	

III. Conclusion

Following a single application of fluopicolide at a nominal application rate of 400 g/ha to bare soil in summer 2000, the decline of fluopicolide and the formation and decline of its metabolites M-01, M-02 and M-03 was followed for up to 735 days after application at a trial site in Appilly, Northern France. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit DFOP un-normalised DT_{50} value of 143.4 days and DT_{90} value of 1695 days for fluopicolide.

The accumulation potential of fluopicolide and its metabolites M-01, M-02 and M-03 was assessed at the same site for up to four years after repeated application to bare soil. It was concluded during the previous evaluation that it was not clear that fluopicolide residues had reached plateau concentrations in the Appilly trial. For M-01, residues were lower but also did not appear to reach a plateau. The metabolites M-02 and M-03 were rapidly degraded in soil and were either not detected or disappeared completely within one month. In this submission definitive assessment of the accumulation of fluopicolide and its metabolites in soil is addressed in Document MCP-9 by calculation.

Assessment and conclusion by applicant:

The study is considered valid to assess the dissipation of fluopicolide under field conditions in soil. The study meets the requirements to assess field persistence of fluopicolide and its metabolites under EU 283/2013, and to derive parent soil $DegT_{50matrix}$ values for legacy field studies as defined by EFSA (2014). It is not suitable for assessing metabolite soil $DegT_{50matrix}$ values as the design did not minimise soil surface processes immediately after application as required by EFSA (2014).

The study is considered as supportive information to assess the possibility of accumulation of residues in soil. Definitive assessment of the accumulation of fluopicolide and its metabolites in soil is addressed in Document MCP-9 by calculation.

Data Point:	KCA 7.1.2.2.1/09
Report Author:	[redacted]
Report Year:	2005
Report Title:	Field soil dissipation of AE C638206 following a single application and multiple applications to bare soil plots at 1 location in Germany, 2000
Report No:	C047266
Document No:	M-247945-01-1
Guideline(s) followed in study:	BBA: Part IV, 4-1; EU (=EEC): Anonymous 1999; IVA: Beutel et al. (1993); SETAC: Lynch, (1995)
Deviations from current test guideline:	Yes. Report meets the requirement for field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013 and the requirements for assessing parent soil DegT50matrix values as required by EFSA (2014) for legacy field studies. Report does not meet the requirement for assessing metabolite soil DegT50matrix values as required by EFSA (2014) for field studies.
Previous evaluation:	yes, evaluated and accepted DAR (2005)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities.
Acceptability/Reliability:	Yes

Executive Summary

A four year terrestrial field dissipation and accumulation study with fluopicolide, formulated as a suspo-emulsion containing 97.9 g/L fluopicolide (AE C638206.00 SE10 A3), has been conducted at a site at Philippsburg, Southern Germany. The top soil at the test site was a loamy sand soil. The formulated material was applied once a year, at the rate required to achieve an annual application of 400 g/ha of fluopicolide using a calibrated boom sprayer to bare soil. Nominal application rates were confirmed by measuring the unused formulation remaining in the spray tank to calibrate the amount applied. The initial application was on 20 June 2000 with subsequent applications on the 24 July 2001, 26 June 2002, 5 June 2003 and 6 July 2004. Samples of soil have been taken at intervals over a four year period and analysed by an LC/MS/MS method to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) present in the samples.

The initial dissipation of fluopicolide was rapid followed by a slower dissipation phase during the winter months due to the cold climate. Dissipation continued the following summer at a slower rate than the initial rapid phase possibly due to the reduced availability of fluopicolide due to increased adsorption to soil with ageing. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. Lower residues were detected in the 10-20 cm and 20-30 cm horizons in the dissipation phase at concentrations ranging from 0.073 to 0.002 mg/kg and 0.023 to 0.002 mg/kg, respectively (mean values). Residue levels of parent declined to 85.5 g/ha two years after the first treatment which represented less than 20% of the measured (485.5 g/ha) initial concentration. Throughout the dissipation phase no residues of fluopicolide were detected above the LOQ below 30 cm depth.

The concentration of M-01 (AE C653711) in the soil profile varied with the degradation rate of fluopicolide. During the summer months when the degradation rate of the parent compound was relatively rapid, M-01 concentrations were highest and declined during the winter as the degradation rate of fluopicolide slowed. Residue levels of M-01 reached a maximum two months after the first application at a concentration equivalent to 35.5 g/ha in August 2000. The metabolite was detected at soil depths down to 50 cm. The maximum residue level in the 0-10 cm horizon was observed 62 days after application at 0.015 mg/kg (mean of three replicates). The maximum residue in the underlying 10-20 cm was detected at 120 days after application at 0.011 mg/kg (mean values) during the dissipation phase. In the 20-30 cm and 30-50 cm horizons residue levels were generally at or below the LOQ (maximum in dissipation phase 0.007 mg/kg). Overall residue levels of M-01 declined to a concentration equivalent to 2.5 g/ha the end of the dissipation phase.

M-02 (AE C657188) and M-03 (AE 0608000) were rapidly degraded in the trial. Residues of M-02 were only detected at early timepoints in the 0-10 cm soil depth up to 62 days after application at a maximum concentration of 0.009 mg/kg (mean value), equivalent to 13.4 g/ha. Residues of M-03 were detected one and three days after application in the 0-10 cm soil layer at the LOQ (0.005 mg/kg) in single replicate samples. The degradation of AE 0608000 is known to be pH dependant and is very rapidly degraded in slightly acidic soils such as the soil at the Philippsburg trial site. No residues of M-02 and M-03 were detected above the LOQ below 10 cm depth.

A re-evaluation of the degradation kinetics of the dissipation phase of the trial in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit DFOP un-normalised DT_{50} value of 133.0 days and DT_{90} of 1417.0 days for fluopicolide

The plateau concentrations of fluopicolide and M-01 after four years are summarised below.

Plateau concentration	Time-point	Fluopicolide (mg/kg)		Time-point	M-01 (mg/kg)	
		0-10 cm	0-20 cm		0-10 cm	0-20 cm
High[1]	Day 0 2nd Application	0.341	0.191	Day 123 after 4th Application	0.070	0.042
Low[2]	Day 368 after 4th Application	0.094	0.064	Day 0 after 5th Application	0.024	0.021

[1] maximum of the high values of the "saw teeth" curve
[2] maximum of the low values of the "saw teeth" curve

It was stated in Addendum 1 to the DAR (2007) that fluopicolide residues had reached a plateau concentration in the accumulation phase of the Philippsburg trial. For M-01, residues levels were lower but did not appeared to reach a plateau.

Residues of fluopicolide were detected mainly in the 0-10 cm and 10-20 cm soil horizons throughout the accumulation phase. Residues in the 10-20cm soil depth reached a maximum of 0.073 mg/kg one day after the first application. Low residues were detected in the 20-30 cm soil depth at concentrations ranging from 0.003 to 0.033 mg/kg (mean values) throughout the accumulation phase. In the 30-50 cm soil depth fluopicolide was detected on occasions at concentrations ≥ 0.005 mg/kg (mean values) except for one replicate at 0.045 mg/kg immediately.

The metabolite M-01 was detected in 0-10 cm and 10-20 cm soil depth at maximum concentrations of 0.070 mg/kg and 0.024 mg/kg (mean values), respectively. In the 20-30 cm layer residues reached a maximum of 0.012 mg/kg and did not exceed 0.006 mg/kg in 30-50 cm depth.

No residues of M-02 or M-03 were detected throughout the accumulation phase.

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide formulated as liquid suspo-emulsion (fluopicolide)

Year of application:	2000 and 2001	2002 and 2003	2004
Content of a.s.:	97.9 g/L	97.5 g/L	100 g/L
Certificate of Analysis:	AGF2000-0106-01	AGF2001-0227-01	AGF2003-0035-01
Lot No:	OP200271	OP210914	OP220828

2. Trial Location & Soil

A four year terrestrial field dissipation and accumulation study with fluopicolide, formulated as a suspo-emulsion containing 97.9 g/L fluopicolide (AE C638206 00 SE10 A3), has been conducted at a site at Philippsburg, Southern Germany. The top soil was a low organic carbon silty sand (DIN classification) overlying sand subsoil. The characteristics of the soil are summarised in Table 7.1.2.2.1- 37. Three experimental plots, each measuring 3 metres by 26 metres (234 m² in total), were treated with the test substance. A fourth plot measuring 3 metres by 12 metres was left untreated to provide control samples. The treated plots were subdivided into separate areas for the dissipation phase treated once in 2000 and for the accumulation phase treated up to five times in 2000, 2001, 2002, 2003 and 2004.

Table 7.1.2.2.1- 37: Location, site description and climatic data of test site

Characteristic	Units	Philippsburg, Baden-Württemberg, Germany		
		Horizon 1	Horizon 2	Horizon 3
Sampling depth	cm	0 - 20	20 - 50	50 - 90
pH	$CaCl_2$	6.4	5.4	7.3
Cation exchange capacity	mval/100 g	5.59	3.90	3.50
Total organic carbon (TOC)	%	0.27	0.53	0.11
Biomass 2000	mg C/100 g	11.48	nd	nd
2002	mg C/100 g	10.00	nd	nd
2003	mg C/100 g	9.35	nd	nd
2004	mg C/100 g	13.2	nd	nd
Soil Density	g/L	1950	1650	1416
Particle size distribution (DIN)				
Clay < 0.002 mm	%	81.7	83.8	89.0
Total silt 0.002 - 0.063 mm	%	12.8	11.4	7.0
Total sand 0.063 - 2 mm	%	5.5	4.8	4.0
Textural class	DIN	Loamy sand	Silty sand	Sand
Water Holding Capacity	Vol % at 1/10 bar (pF2)	15.15	11.99	12.00
	Vol % at 15 bar (pF4.2)	3.32	2.64	nd

n.d. = not determined

B. Study Design

1. Experimental Conditions

The formulated material was applied once a year, at the rate required to achieve an annual application of 400 g/ha of fluopicolide using a calibrated boom sprayer. Nominal application rates were confirmed by measuring the unused formulation remaining in the spray tank to determine the actual amount applied (411 g/ha for the first application). The initial application was on 20 June 2000 with subsequent applications on the 24 July 2001, 26 June 2002, 5 June 2003 and 6 July 2004.

All applications were made to bare soil. Throughout the study the plots were maintained as bare soil by the periodic application of the herbicide glyphosate to control weeds.

The weather conditions were monitored at a distance of 1 km from the test site. Wind speed, global radiation, rainfall, air temperature, relative air humidity, soil temperature at 10 cm depth and soil moisture at 5 different depths were monitored continuously).

2. Sampling

Soil cores (5 cm diameter) for the dissipation phase were taken immediately after treatment, 1 day, 3 days , 14 days and 1, 2, 4, 6, 8, 10, 12, 15, 18 and 24 months after the first application. Samples for the accumulation phase were taken immediately after each treatment and additionally approximately 4 and 12 months after the second, third and fourth applications in 2001, 2002 and 2003. The final sample was taken immediately after the fifth application in 2004. Soil cores for the dissipation phase were taken to a depth of 30 cm during the first month and to a depth of 50 cm at time-points up to two years. Soil cores for the accumulation phase were taken to a depth of 50 cm following the second application and up to 4 months after the third. At timepoints after this, soil cores were taken to a depth of 90 cm. At each sampling date 7 samples from each plot were taken (21 cores in total). Field samples were frozen immediately after sampling and shipped frozen to GAB Biotechnologie GmbH, Germany. The soil samples from the same horizon of each plot were blended in Germany and a subsample dispatched frozen to the Bayer CropScience analytical laboratory in France. The samples were then stored at -18 ºC until required for analysis.

3. Analytical Procedures

The analytical method AR 265-01 was used to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000 referred to in the report as RPA 427967). Soil samples of 20 g were extracted twice at ambient temperature for 5 minutes by mechanical agitation using acetonitrile/water (70/30, *v/v*) acidified with 0.1% formic acid. After each extraction step, extract and soil were separated by centrifugation and decantation. The soil extracts were combined and diluted with acidified water to result in a final solvent of acetonitrile/water (30/70) with 0.1% formic acid. Quantification was carried out by LC-MS/MS using external standardisation for the parent compound and its metabolites. The limit of quantification (LOQ) was 0.005 mg/kg for each analyte.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide and reference items M-01, M-02 and M-03 at levels of LOQ and 100 x LOQ and processed in parallel to the dissipation samples. The mean recoveries of LOQ and 100 x LOQ were 99 and 96% (RSD 7.7 and 11.2%) for fluopicolide, 100 and 94% (RSD 5.6 and 8.3%) for M-01, 97 and 95% (RSD 7.8 and 8.7%) for M-02 and 102 and 91% (RSD 6.0 and 11.0%) for M-03. No residues of fluopicolide or its metabolites were found above the LOQ in the analysed untreated samples.

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.2.1/24. A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each site between the SFO and FOMC fits for fluopicolide. The comparison of the SFO and FOMC fits suggested bi-phasic decline, and the DFOP model was therefore also fitted. For the Philippsburg site, confidence in the k1 DFOP rate constant was slightly low (p=0.064), however the DFOP fit provided the best visual description of the residues, and was accepted.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) in soil samples by HPLC-MS/MS.

B. Data

The results for fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots in Table 7.1.2.2.1- 38 to Table 7.1.2.2.1- 45. The fluopicolide concentrations for the individual plots are also shown in Figure 7.1.2.2.1- 3 and Figure 7.1.2.2.1- 4.

The levels of residues for each analyte in the three replicate treated plots were generally in good agreement throughout the trial. The only exceptions to this were fluopicolide residue levels in Plot T1 on 23 June 2000 in the 0-10 cm soil layer and on 26 June 2002 in the 10-20 cm soil layer, where the concentrations were significantly higher than in the other plots. In order to calculate mean values, concentrations <LOQ (0.005 mg/kg) were assumed to be 0 mg/kg. Where individual replicate values exceeded the LOQ the calculated mean concentration has been reported, including mean values that are below the LOQ. For the conversion of mg/kg into g/ha a soil density of 1.5 g/cm^3 was used.

Table 7.1.2.2.1- 38: Residues of fluopicolide in soil after an application of 400 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Depth [cm]	Sub plot	DAA1													
		0	**1**	**3**	**14**	**36**	**62**	**120**	**181**	**244**	**308**	**367**	**461**	**546**	**735**
0-10	1	0.242	0.210	0.970	0.150	0.172	0.162	0.104	0.096	0.107	0.082	0.070	0.061	0.040	0.037
	2	0.183	0.333	0.145	0.193	0.156	0.110	0.075	0.093	0.047	0.060	0.055	0.033	0.026	0.021
	3	0.195	0.210	0.291	0.169	0.111	0.137	0.080	0.117	0.082	0.073	0.053	0.043	0.036	0.028
	mean	**0.207**	**0.251**	**0.218[1]**	**0.171**	**0.146**	**0.136**	**0.086**	**0.102**	**0.082**	**0.072**	**0.059**	**0.046**	**0.034**	**0.029**
10-20	1	0.007	0.121	<LOQ	0.012	0.010	0.013	0.020	0.029	0.040	0.066	0.030	<LOQ	0.014	0.035
	2	0.009	0.048	0.085	0.016	0.016	0.016	0.014	0.021	0.023	0.029	0.018	<LOQ	0.012	0.009
	3	<LOQ	0.049	<LOQ	0.038	0.005	0.012	<LOQ	0.025	0.064	0.035	0.033	0.006	0.029	0.022
	mean	**0.005[2]**	**0.073**	**0.028**	**0.022**	**0.010**	**0.013**	**0.011[2]**	**0.025**	**0.042**	**0.043**	**0.027**	**0.002[2]**	**0.018**	**0.022**
20-30	1	<LOQ	<LOQ	0.062	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.010	0.005	0.030	<LOQ	0.012
	2	<LOQ	<LOQ	<LOQ	0.009	<LOQ	<LOQ	<LOQ	<LOQ	0.007	<LOQ	<LOQ	0.017	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.008	0.005	0.023	0.009	0.007
	mean	**<LOQ**	**<LOQ**	**0.021[2]**	**0.003[2]**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.002[2]**	**0.009[2]**	**0.003[2]**	**0.023**	**0.003[2]**	**0.006[2]**
30-50	1	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

DAA: days after application
LOQ (limit of quantitation) = 0.005 mg/kg, 1 mean of two replicates, 2 Replicate value > LOQ

Table 7.1.2.2.1- 39: Residues of fluopicolide in soil after annual applications of 400 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3			DAA4			DAA5
		0	126	363	0	121	342	0	123	368	0
0-10	1	0.261	0.0093	0.053	0.345	0.126	0.087	0.240	0.150	0.080	0278
	2	0.407	0.097	0.085	0.276	0.129	0.086	0.276	0.129	0.086	0.249
	3	0.354	0.128	0.059	0.196	0.137	0.096	0.215	0.149	0.108	0.233
	mean	**0.341**	**0.106**	**0.066**	**0.272**	**0.131**	**0.090**	**0.235**	**0.163**	**0.094**	**0.252**
10-20	1	0.048	0.032	0.038	0.0286	0.049	0.052	0.091	0.044	0.027	0.039
	2	0.042	0.027	0.063	0.031	0.040	0.044	0.060	0.030	0.044	0.023
	3	0.031	0.034	0.037	0.053	0.043	0.050	0.056	0.019	0.028	0.033
	mean	**0.040**	**0.031**	**0.046**	**0.042[1]**	**0.044**	**0.049**	**0.069**	**0.031**	**0.033**	**0.032**
20-30	1	<LOQ	<LOQ	0.012	<LOQ	0.015	0.010	0.024	0.013	<LOQ	0.015
	2	0.009	0.006	0.023	0.078	0.020	0.013	0.016	<LOQ	0.013	0.005
	3	<LOQ	0.008	0.0009	0.022	0.012	0.013	0.009	<LOQ	<LOQ	<LOQ
	mean	**0.003[2]**	**0.005[2]**	**0.015**	**0.033[2]**	**0.016**	**0.012**	**0.016**	**0.004[2]**	**0.004[2]**	**0.007[2]**
30-50	1	0.045	<LOQ	0.009	<LOQ	0.005	<LOQ	0.005	<LOQ	<LOQ	<LOQ
	2	0.008	<LOQ	0.007	<LOQ	0.009	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	0.013	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**0.018[3]**	**<LOQ**	**0.005[2]**	**0.004[2]**	**0.005[2]**	**<LOQ**	**0.002[2]**	**<LOQ**	**<LOQ**	**<LOQ**
50-70	1	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	2	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	3	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**n.a**
70-90	1	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**n.a**	**n.a**	**n.a**	**n.a**	**n.a**

DAA: days after application
n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value > LOQ, [2] mean of two replicates, [3] Replicate values 0.045 mg/kg, 0.008 mg/kg, <LOQ (0.005 mg/kg)

Table 7.1.2.2.1- 40: Residues of M-01 (AE C6563711) in soil after an application of 400 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Depth [cm]	Sub plot	DAA1													
		0	1	3	14	36	62	120	181	244	308	367	461	546	735
0-10	1	<LOQ	0.007	0.017	0.013	0.017	0.018	0.009	<LOQ	<LOQ	<LOQ	0.006	0.011	<LOQ	<LOQ
	2	<LOQ	0.013	0.007	0.013	0.013	0.013	0.009	<LOQ	0.004	<LOQ	0.007	0.007	<LOQ	<LOQ
	3	<LOQ	0.007	0.015	0.012	0.011	0.015	0.006	0.006	<LOQ	<LOQ	0.005	0.007	<LOQ	<LOQ
	mean	**<LOQ**	**0.009**	**0.013**	**0.013**	**0.014**	**0.015**	**0.008**	**0.002[1]**	**0.001[1]**	**<LOQ**	**0.006**	**0.008**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	0.006	<LOQ	<LOQ	0.008	0.009	0.014	0.008	0.006	<LOQ	<LOQ	<LOQ	0.006	0.005
	2	<LOQ	<LOQ	<LOQ	<LOQ	0.005	0.008	0.010	0.007	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	0.007	0.008	0.008	0.009	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**0.002[1]**	**<LOQ**	**<LOQ**	**0.007**	**0.008**	**0.010**	**0.008**	**0.002[1]**	**<LOQ**	**<LOQ**	**<LOQ**	**0.002[1]**	**0.002[1]**
20-30	1	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.007	0.008	0.006	<LOQ	<LOQ	0.010	<LOQ	<LOQ
	2	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.006	<LOQ	<LOQ	<LOQ	0.004	<LOQ	<LOQ
	3	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.008	<LOQ	<LOQ	<LOQ	0.006	<LOQ	<LOQ
	mean	**n.a**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.002[1]**	**0.007**	**0.002[1]**	**<LOQ**	**<LOQ**	**0.007**	**<LOQ**	**<LOQ**
30-50	1	-	-	-	-	-	<LOQ	<LOQ	0.005	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**0.002[1]**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

DAA: days after application
LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value > LOQ

Table 7.1.2.2.1- 41: Residues of M-01 (AE C6563711) in soil after annual applications of 400 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3			DAA4			DAA5
		0	126	363	0	121	342	0	123	368	0
0-10	1	0.010	0.013	0.006	0.008	0.013	0.008	0.011	0.063	0.015	0.023
	2	0.013	0.011	0.010	0.010	0.016	0.010	0.012	0.081	0.025	0.025
	3	0.011	0.010	0.002	0.011	0.013	0.008	0.010	0.067	0.030	0.025
	mean	**0.011**	**0.011**	**0.007**	**0.010**	**0.014**	**0.009**	**0.011**	**0.070**	**0.023**	**0.024**
10-20	1	0.007	0.018	0.010	0.009	0.017	0.009	0.011	0.017	0.021	0.016
	2	0.007	0.015	0.011	0.007	0.018	0.010	0.009	0.013	0.025	0.018
	3	0.050	0.018	0.007	0.008	0.018	0.010	0.011	0.011	0.026	0.018
	mean	**0.021**	**0.017**	**0.009**	**0.008**	**0.018**	**0.010**	**0.010**	**0.014**	**0.024**	**0.017**
20-30	1	<LOQ	0.012	0.006	<LOQ	0.013	0.005	0.005	0.007	0.010	0.008
	2	<LOQ	0.008	0.008	0.005	0.010	0.007	0.007	0.006	0.014	0.008
	3	<LOQ	0.015	<LOQ	0.005	0.013	0.008	0.005	0.006	0.008	0.010
	mean	**<LOQ**	**0.012**	**0.005[1]**	**0.003[1]**	**0.012**	**0.007**	**0.006**	**0.006**	**0.011**	**0.009**
30-50	1	<LOQ	<LOQ	<LOQ	<LOQ	0.007	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	0.005	0.005	<LOQ	0.007	0.005	0.009	<LOQ	0.006	<LOQ
	3	<LOQ	0.005	<LOQ	<LOQ	0.005	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**0.003[1]**	**0.002[1]**	**<LOQ**	**0.006**	**0.002[1]**	**0.003[1]**	**<LOQ**	**0.002[1]**	**<LOQ**
50-70	1	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	2	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	3	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	mean	-	-	-	-	-	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**n.a**
70-90	1	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	mean	-	-	-	-	-	**n.a**	**n.a**	**n.a**	**n.a**	**n.a**

DAA: days after application, n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value > LOQ

Table 7.1.2.2.1- 42: Residues of M-02 (AE C657188) in soil after an application of 400 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Depth [cm]	Sub plot	DAA1													
		0	1	3	14	36	62	120	181	244	308	367	461	546	735
0-10	1	<LOQ	0.007	0.010	0.007	<LOQ	0.005	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	0.009	0.009	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	0.006	0.008	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**0.007**	**0.009**	**0.002[1]**	**<LOQ**	**0.002**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**n.a**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

DAA: days after application
n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value < LOQ

Table 7.1.2.2.1- 43: Residues of M-02 (AE C657188) in soil after annual applications of 400 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3			DAA4			DAA5
		0	147	361	0	128	336	0	120	378	0
0-10	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**n.a**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
50-70	1	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	2	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	3	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**n.a**
70-90	1	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**n.a**	**n.a**	**n.a**	**n.a**	**n.a**

DAA: days after application
n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, - Replicate value > LOQ

Table 7.1.2.2.1- 44: Residues of M-03 (AE 0608000) in soil after an application of 400 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Depth [cm]	Sub plot	DAA1													
		0	1	3	14	36	62	120	181	244	308	367	461	546	735
0-10	1	<LOQ	<LOQ	0.006	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	0.005	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**0.002[1]**	**0.002[1]**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**n.a**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

DAA: days after application
n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, [1] Replicate value > LOQ

Table 7.1.2.2.1- 45: Residues of M-03 (AE 0608000) in soil after annual applications of 400 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3			DAA4			DAA5
		0	147	361	0	128	336	0	120	378	0
0-10	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	1	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	3	n.a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**n.a**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
50-70	1	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	2	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	3	-	-	-	-	-	<LOQ	<LOQ	n.a	<LOQ	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**n.a**	**<LOQ**	**n.a**
70-90	1	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	2	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	3	-	-	-	-	-	n.a	n.a	n.a	n.a	n.a
	mean	**-**	**-**	**-**	**-**	**-**	**n.a**	**n.a**	**n.a**	**n.a**	**n.a**

DAA: days after application
n.a not analysed, LOQ (limit of quantitation) = 0.005 mg/kg, - Replicate value > LOQ

C. Residues

Dissipation phase (2000 – 2002)

The theoretical initial concentration of fluopicolide in the 0-10 cm layer was 0.274 mg/kg based on the calibrated application rate of 411 g/ha, assuming a soil density of 1.5 g/cm³. The measured initial concentration of fluopicolide in 0-10 cm was 0.207 mg/kg (mean value) immediately after application and 0.251 mg/kg (mean value) three days later with an additional 0.073 mg/kg detected in the 10-20 cm depth, equivalent to 318 g/ha and 485.5 g/ha, respectively. These deviations to the nominal application rate of 400 g/ha appear to be acceptable taking into account the uncertainties (e.g. soil density, homogenisation, application) related to the samples collected soon after treatment.

The initial dissipation of fluopicolide was rapid followed by a slower dissipation phase during the winter months due to the cold climate. Dissipation continued the following summer at a slower rate than the initial rapid phase possibly due to the reduced availability of fluopicolide due to increased adsorption to soil with ageing. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. Lower residues were detected in the 10-20 cm and 20-30 cm horizons in the dissipation phase at concentrations ranging from 0.073 to 0.002 mg/kg and 0.023 to 0.002 mg/kg, respectively (mean values). Residue levels of parent declined to 85.5 g/ha two years after the first treatment which represented less than 20 % of the measured (485.5 g/ha) initial concentration. Throughout the dissipation phase no residues of fluopicolide were detected above the LOQ below 30 cm depth.

The concentration of M-01 (AE C653711) in the soil profile varied with the degradation rate of fluopicolide. During the summer months when the degradation rate of the parent compound was relatively rapid, M-01 concentrations were highest and declined during the winter as the degradation rate of fluopicolide slowed. Residue levels of M-01 reached a maximum two months after the first application at a concentration equivalent to 35.5 g/ha in August 2000. The metabolite was detected at soil depths down to 50 cm. The maximum residue level in the 0-10 cm horizon was observed 62 days after application at 0.015 mg/kg (mean of three replicates). The maximum residue in the underlying 10-20 cm was detected at 120 days after application at 0.011 mg/kg (mean values) during the dissipation phase. In the 20-30 cm and 30-50 cm horizons residue levels were generally at or below the LOQ (maximum in dissipation phase 0.007 mg/kg). Overall residue levels of M-01 declined to a concentration equivalent to 2.5 g/ha the end of the dissipation phase.

M-02 (AE C657188) and M-03 (AE 0608000) were rapidly degraded in the trial. Residues of M-02 were only detected at early timepoints in the 0-10 cm soil depth up to 62 days after application at a maximum concentration of 0.009 mg/kg (mean value), equivalent to 13.4 g/ha. Residues of M-03 were detected one and three days after application in the 0-10 cm soil layer at the LOQ (0.005 mg/kg) in single replicate samples. The degradation of M-03 is known to be pH dependant and is very rapidly degraded in slightly acidic soils such as the soil at the Philippsburg trial site. No residues of M-02 and M-03 were detected above the LOQ below 10 cm depth.

Accumulation:

The maximum average concentration of fluopicolide in soil was 0.341 mg/kg in 0 to 10 cm soil depth immediately after the second application in 2001.

Residues of fluopicolide were detected mainly in the 0-10 cm and 10-20 cm soil horizons throughout the accumulation phase. Residues in the 10-20 cm soil depth reached a maximum of 0.073 mg/kg one day after the first application. Low residues were detected in the 20-30 cm soil depth at concentrations ranging from 0.003 to 0.033 mg/kg (mean values) throughout the accumulation phase. In the 30-50 cm soil depth fluopicolide was detected on occasions at concentrations ≤ 0.005 mg/kg (mean values) except for one replicate at 0.045 mg/kg immediately after the second application on 24 July 2001 which was concluded to be a result of contamination during sampling.

The metabolite M-01 was detected in 0-10 cm and 10-20 cm soil depth at maximum concentrations of 0.070 mg/kg and 0.024 mg/kg (mean values), respectively. In the 20-30 cm layer residues reached a maximum of 0.012 mg/kg and did not exceed 0.006 mg/kg in 30-50 cm depth.

M-02 and M-03 were detected only at early time-points after the initial application during the dissipation phase of the trial. No residues of either metabolite were detected throughout the accumulation phase.

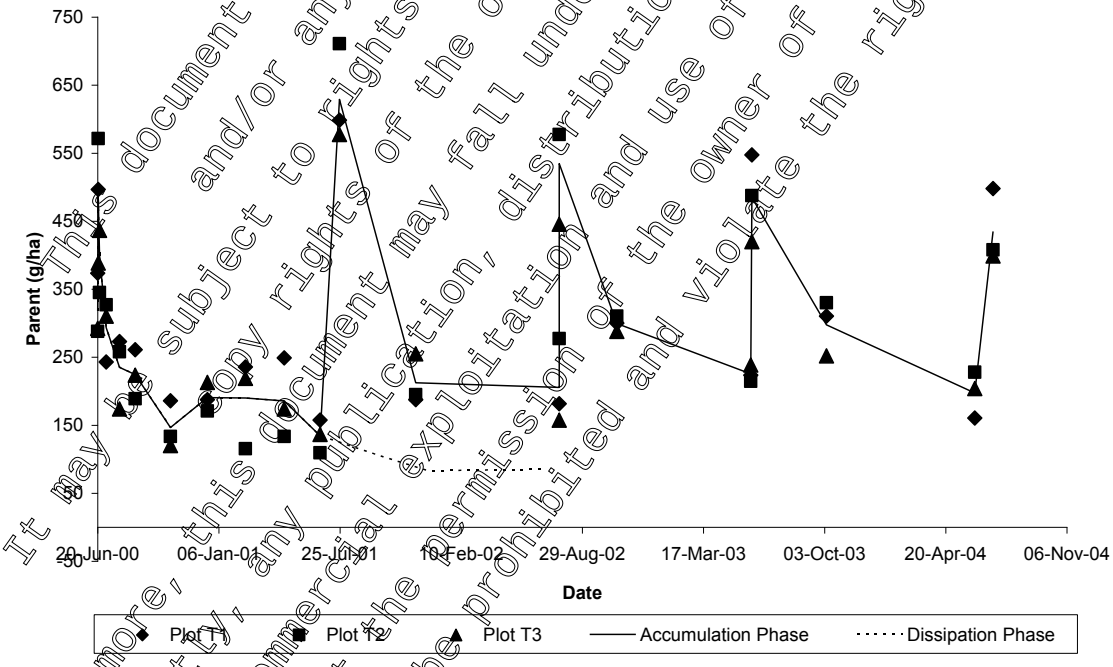
The plateau concentrations of fluopicolide and M-01 after four years are summarised below.

Plateau concentration	Time-point	Fluopicolide (mg/kg)		Time-point	M-01 (mg/kg)	
		0-10 cm	0-20 cm		0-10 cm	0-20 cm
High[1]	Day 0 2nd Application	0.341	0.191	Day 123 after 4th Application	0.070	0.042
Low[2]	Day 368 after 4th Application	0.094	0.064	Day 0 after 5th Application	0.024	0.021

[1] maximum of the high values of the "saw teeth" curve
[2] maximum of the low values of the "saw teeth" curve

It was stated in Addendum 1 to the DAR (2007) that fluopicolide residues had reached a plateau concentration in the accumulation phase of the Philippsburg trial. For M-01, residues levels were lower but did not appeared to reach a plateau.

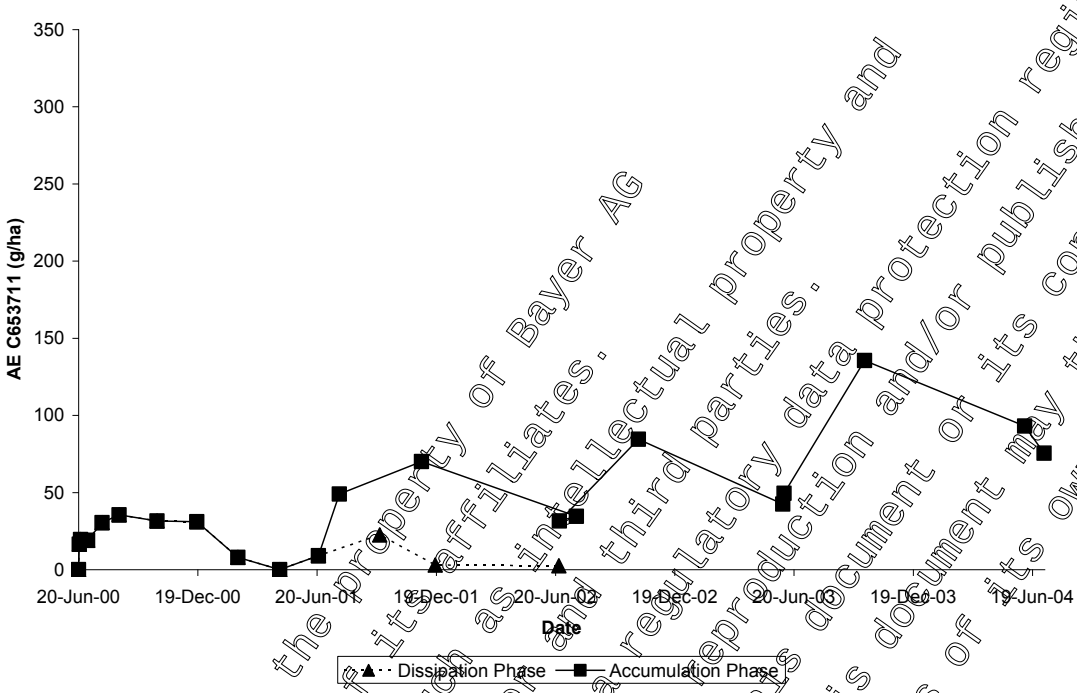
Figure 7.1.2.2.1- 3: Dissipation and Accumulation of Fluopicolide at Philippsburg



Mean values are given as solid and dashed lines for the accumulation phase and dissipation phase as indicated in the key. The results for individual plots are also given for fluopicolide as solid symbols.

Figure 7.1.2.2.1- 4: Dissipation and Accumulation of M-01 (AE C653711) at Philippsburg

NB Scale different



D. Kinetic Analysis

The half-life of fluopicolide included in the report was calculated using a bi-phasic first-order kinetic model (Hockey Stick) as 99 days. The DT_{90} was 1184 days and the r^2 was 0.879. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for Document KCA 7.1.2.2.1/24. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.2.1- 46. Best fit kinetics are highlighted in bold.

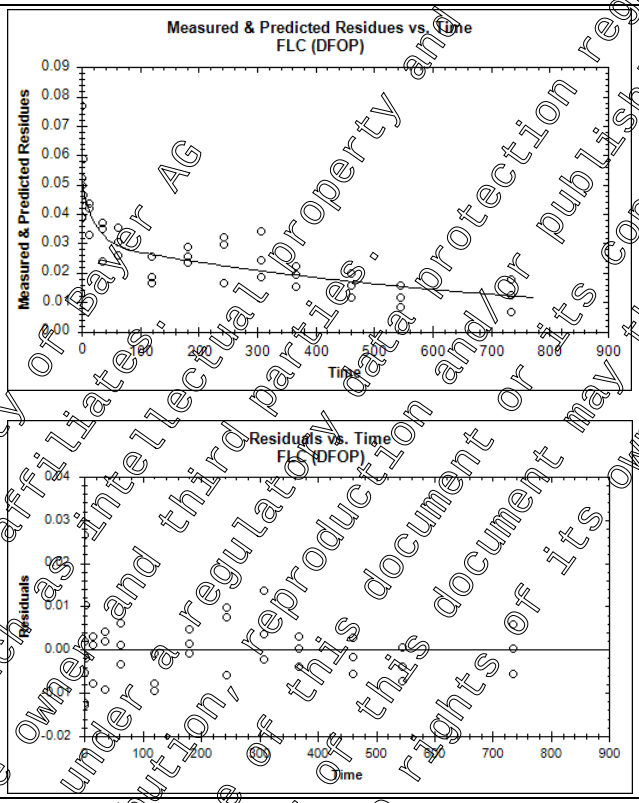
Table 7.1.2.2.1- 46: Degradation rate of fluopicolide under field conditions (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Philippsburg (Germany), CA 7.1.2.2.1/09 (Pollmann, 2005a)	SFO	0.04317	k 0.002404	18.8	6.53E-08	0.001671	0.003	288.3	957.8
	FOMC	0.05099	α 0.3088 β 13.91	13.5	n.r. n.r.	0.1372 -10.57	0.48 38.38	117.3	>10000
	DFOP	**0.05131**	**k1 0.04642 k2 0.001252 g 0.4104**	**12.7**	**0.06404 0.00473 n.r.**	**-0.01203 0.0003557 0.2359**	**0.105 0.002 0.585**	**133**	**1417**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.2.1- 47: Graphical representations of best fit model

Trial / Best Fit Model	Graphical Representations
Philippsburg (Germany) / DFOP	

III. Conclusion

Following a single application of fluopicolide at a nominal application rate of 400 g/ha to bare soil in summer 2000, the decline of fluopicolide and the formation and decline of its metabolites M-01, M-02 and M-03 was followed for up to 735 days after application at a trial site in Philippsburg, Germany. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit DFOP un-normalised DT_{50} value of 133 days and DT_{90} value of 1417 days for fluopicolide.

The accumulation potential of fluopicolide and its metabolites M-01, M-02 and M-03 was assessed at the same site for up to four years after repeated application to bare soil. It was concluded during the previous evaluation that fluopicolide residues had reached plateau concentrations in the Philippsburg trial. For M-01, residues were lower but also did not appear to reach a plateau. In this submission definitive assessment of the accumulation of fluopicolide and its metabolites in soil is addressed in Document MCP-9 by calculation.

Assessment and conclusion by applicant:

The study is considered valid to assess the dissipation of fluopicolide under field conditions in soil. The study meets the requirements to assess field persistence of fluopicolide and its metabolites under EU 283/2013, and to derive parent soil $DegT_{50matrix}$ values for legacy field studies as defined by EFSA (2014). It is not suitable for assessing metabolite soil $DegT_{50matrix}$ values as the design did not minimise soil surface processes immediately after application as required by EFSA (2014).

The study is considered as supportive information to assess the possibility of accumulation of residues in soil. Definitive assessment of the accumulation of fluopicolide and its metabolites in soil is addressed in Document MCP-9 by calculation.

Data Point:	KCA 7.1.2.2.1/10
Report Author:	[redacted]
Report Year:	2007
Report Title:	Evaluation of soil degradation parameters for fluopicolide (AE C638206) for use as trigger values
Report No:	MEF-07/265
Document No:	M-294300-01-1
Guideline(s) followed in study:	not specified
Deviations from current test guideline:	Not applicable
Previous evaluation:	yes, evaluated and accepted in the Addendum I to the DAR (2007)
GLP/Officially recognised testing facilities:	not applicable
Acceptability/Reliability:	No

For procedural reasons the previously submitted report is included under Point KCA 7.1.2.2.1 in the current dossier (KCA 7.1.2.2.1/10). However, the report has been fully superseded by two new kinetic evaluation reports of the laboratory data (KCA 7.1.2.1.1/10, M-685680-01-1) and original field dissipation trials (KCA 7.1.2.2.1/24, M-685682-01-1). Consequently, no summary of this report has been included in this dossier.

Data Point:	KCA 7.1.2.2.1/11
Report Author:	[redacted]
Report Year:	2007
Report Title:	Evaluation of soil degradation parameters for fluopicolide and its metabolites from laboratory and field trials for modelling purposes
Report No:	MEF07/266
Document No:	M-294399-01-1
Guideline(s) followed in study:	not specified
Deviations from current test guideline:	Not applicable
Previous evaluation:	yes, evaluated and accepted in the Addendum 1 to the DAR (2007)
GLP/Officially recognised testing facilities:	not applicable
Acceptability/Reliability:	No

For procedural reasons the previously submitted report is included under Point KCA 7.1.2.2.1 in the current dossier (KCA 7.1.2.2.1/11). However, the report has been fully superseded by two new kinetic evaluation reports of the laboratory data (KCA 7.1.2.1.1/10, M-685680-01-1) and original field dissipation trials (KCA 7.1.2.2.1/22; M-685676-01-1). Consequently, no summary of this report has been included in this dossier.

Data Point:	KCA 7.1.2.2.1/12
Report Author:	[redacted]
Report Year:	2019
Report Title:	Terrestrial field dissipation study with fluopicolide & propamocarb-hydrochloride SC 687.5 in Germany, United Kingdom, and France (North)
Report No:	M-651181-01-1
Document No:	M-651181-01-1
Guideline(s) followed in study:	Regulation (EC) No. 1107/2009 of the European Parliament and of the Council of 21 October 2009 including Data Requirements SANCO/11803/2010 Rev. 7 and Test Methods SANCO/11843/2010 Rev. 4 EFSA Guidance Document for Evaluating Laboratory and Field Dissipation Studies to obtain DegT50 Values of Active Substances of Plant Protection Products and Transformation Products of these Active Substances in Soil, EFSA Journal 2014; 12(5):3662, 2014
Deviations from current test guideline:	Yes. Report meets the requirement for assessing test substance soil DegT50matrix values as required by EFSA (2014) for field studies. The endpoints may be too conservative for comparison to field persistence criteria and ecotoxicological risk assessment as required by EU 283/201.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

Soil degradation of fluopicolide under Northern European field conditions was investigated after application of Fluopicolide and Propamocarb-hydrochloride SC 687.5 onto bare soil plots in Burscheid (Germany), Great Chishill (United Kingdom), and Lignieres de Touraine (France).

Fluopicolide and Propamocarb-hydrochloride SC 687.5 was sprayed once onto 400 sqm to 564 sqm plots at a rate of 6.40 L/ha, corresponding to nominal 400 g/ha fluopicolide. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface.

Soil cores were taken 0 days before up to 751 days post-application to a maximum depth of 110 cm. The soil cores were cut into 10-cm soil layers, bulked soil layers were homogenised and finally analysed for fluopicolide.

Sub-samples of homogenised soil were extracted in a microwave extractor with organic solvent. Potential matrix effects were eliminated by using an internal standard solution of isotopically labeled reference items added to sample extracts. Following separation of fine particles from soil extracts by centrifugation the identification and quantitation of the analytes was performed by high-performance liquid chromatography using MS/MS detection in the multiple reaction monitoring mode. The analytical method was validated using three different soils. The limit of quantitation (LOQ) was 5.0 µg/kg and the limit of detection (LOD) was 1.5 µg/kg for fluopicolide.

At Burscheid (Germany), the mean amount of fluopicolide at day 0 was 451 g/ha, representing 113% of the nominal application rate. Fluopicolide declined from 451 g/ha in soil at day 0 to 68.8 g/ha at day 701.

At Great Chishill (United Kingdom), the mean amount of fluopicolide at day 0 was 312 g/ha, representing 78.0% of the nominal application rate. Fluopicolide declined from 312 g/ha in soil at day 0 to 111 g/ha at day 751.

At Lignieres de Touraine (France), the mean amount of fluopicolide at day 0 was 356 g/ha, representing 89% of the nominal application rate. Fluopicolide declined from 356 g/ha in soil at day 0 to 58.5 g/ha at day 700.

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide + propamocarb-hydrochloride formulated as a suspension concentrate (62.5g/L fluopicolide + 625 g/L of propamocarb-hydrochloride)

Certificate of Analysis: 01860-00

Lot No.: 2015-00846-01

2. Trial Location & Soil

A terrestrial field dissipation with Fluopicolide & Propamocarb-hydrochloride (SC 687.5), a suspension concentrate formulation containing 62.5 g/L fluopicolide and 625 g/L propamocarb-hydrochloride, was conducted at three locations in Northern Europe. The three locations were Burscheid (Germany), Great Chishill (United Kingdom) and Lignieres de Touraine (France). The sites were fully characterised, and the results summarised in Table 7.1.2.2.1- 48. The plot sizes ranged from 400 sqm to 654 sqm. The control plot was prepared at least 5 m away from the treated plots.

Soil from the three test sites have been used in OECD 307 time dependent sorption and OECD 106 adsorption desorption studies.

Table 7.1.2.2.1- 48: Location, site description and climatic data of test sites

Characteristic	Units	Sampling depth [cm]			
		0-30	30-50	50-75	75-100
Soil Designation	-	**Burscheid (Germany)**			
Soil ID	-	VG08			
Geographic Location City Country	-	Burscheid [redacted] Germany			
pH	$CaCl_2$	5.3	5.6	5.6	5.6
Organic carbon	[% Carbon]	1.2	0.4	0.1	0.1
CEC	[meq/100 g]	12.8	11.8	12.4	11.9
Chalk	[% $CaCO_3$]	12.8	11.8	12.4	11.9
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 21 61 18	 23 57 20	 19 43 38	 13 37 50
Textural class	USDA	silt loam	silt loam	loam	loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 49.3 35.2	 50.0 31.9	 45.9 30.3	 39.4 25.9
Soil Designation	-	**Great Chishill (United Kingdom)**			
Soil ID	-	ENG8			
Geographic Location City Country	-	[redacted] Great Chishill, Cambridgeshire United Kingdom			
pH	$CaCl_2$	7.2	7.5	7.7	7.6
Organic carbon	[% Carbon]	2.1	1.1	0.5	0.5
CEC	[meq/100 g]	28.9	26.1	19.9	17.4
Chalk	[% $CaCO_3$]	1.3	5.8	37.9	43.1
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 41 23 36	 45 21 34	 53 23 24	 51 23 26
Textural class	USDA	clay	clay	clay	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 60.8 29.2	 57.2 27.9	 52.6 25.3	 49.5 25.9
Soil Designation	-	**Lignieres de Touraine (France)**			
Soil ID	-	FR09			
Geographic Location City Country	-	[redacted] 37130 Lignieres de Touraine, Central Region France			
pH	$CaCl_2$	5.9	6.5	6.8	6.8
Organic carbon	[% Carbon]	0.8	0.4	0.3	0.5
CEC	[meq/100 g]	12.2	13.2	14.7	21.8
Chalk	[% $CaCO_3$]	0.2	0.3	0.3	0.0
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 15 15 70	 15 19 66	 19 25 56	 37 33 30
Textural class	USDA	sandy loam	sandy loam	sandy loam	clay loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 44.4 17.5	 47.7 20.6	 52.3 26.1	 61.5 33.1

1. Experimental Conditions

Fluopicolide & Propamocarb-hydrochloride SC 687.5 is a suspension concentrate formulation, containing 62.5 g/L fluopicolide and 625 g/L propamocarb-hydrochloride. The product was sprayed onto bare earth once at each site at an application rate of 6.40 L/ha and 600 L/ha water, corresponding to 400 g/ha fluopicolide during May 2015. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface. Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of a nonselective herbicide to control weeds.

Air temperature, precipitation including irrigation and sunshine data were recorded on site during the field tests.

Soil dissipation of fluopicolide was studied for 751 days.

2. Sampling

The treated plot of each trial was divided into three sub-plots. From each sub-plot 10 soil cores were taken and combined together at each sampling interval (30 cores in total).

Samples were taken from treated plots on following occasions: 0 (post-application, each 0-10 cm depth), 7, 14-15, 21-22, 28-29 (each 0-40 cm depth), 56-68, 120-134 (0-60 cm) and 166-194, 252-301, 348-398, 435-468, 519-554, 605-667, 700-751 (each 0-85 cm depth) after treatment. Samples were taken from the control plot on the following occasions: 0 days before application, 348-398 and 700-751 days after application.

Soil cores taken from the sites in France and the United Kingdom were deep frozen to -18°C within twenty four hours after sampling, then shipped frozen to the analytical laboratory in Germany. Cores taken from the site in Germany were shipped fresh to the analytical laboratory where they were deep frozen to -18°C within twenty four hours after sampling.

3. Analytical Procedures

The analytical method 01445 used to determine levels of fluopicolide. Soil samples of 5 g were extracted in a microwave extractor with a mixture of acetonitrile/water (4/1, v/v). The extracts were centrifuged to remove fine particles of the soil. Possible matrix effects of fluopicolide were eliminated by using an internal standard solution of isotopic labelled reference items. Quantification was carried out by LC-MS/MS. The limit of quantitation (LOQ) for fluopicolide was 5.0 µg/kg in soil. The limit of determination (LOD) for fluopicolide was 1.5 µg/kg.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide at levels of LOQ, 10 x LOQ and 1000 x LOQ and processed in parallel to the dissipation samples throughout the study. The results are summarised in the table below.

Single Values [%]	No of Recoveries	Fortification Level [µg/kg]	Mean [%]	RSD [%]
90, 90, 91, 92, 93, 93, 93, 93, 93, 94, 94, 94, 94, 94, 95, 95, 95, 95, 95, 95, 96, 96, 96, 96, 96, 96, 96, 97, 97, 97, 97, 97, 97, 97, 98, 98, 98, 98, 98, 98, 98, 98, 98, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 101, 101, 101, 101, 101, 101, 101, 101, 101, 101, 101, 101, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 103, 103, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 106, 106, 106, 106, 106, 106, 106, 106, 106, 106, 107, 107, 107, 107, 107, 107, 107, 107, 107, 107, 107, 107, 108, 108, 108, 108, 108, 108, 108, 109, 109, 109, 110, 110, 110, 110, 110, 111, 112, 112, 112, 112, 112, 114, 114, 114	140	5	100	4.6
61, 73, 84, 85, 86, 90, 91, 91, 92, 93, 94, 94, 95, 95, 96, 97, 97, 97, 97, 98, 98, 98, 99, 99, 99, 99, 100, 100, 100, 101, 101, 101, 101, 101, 101, 101, 101, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 106, 106, 106, 106, 106, 106, 106, 106, 106, 106, 107, 107, 107, 107, 107, 107, 107, 107, 107, 107, 107, 107, 108, 108, 108, 108, 108, 108, 108, 109, 109, 109, 110, 110, 110, 110, 110, 111, 112, 112, 112, 112, 112, 114, 114, 114	433	50	103	6.9
86, 92	2	5000	89	-
Overall recovery	575		101	6.1

RSD = Relative standard deviation, LOQ = Limit of quantification

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

Additional 10 g soil samples were extracted by shaking with 0.01M calcium chloride at selected timepoints throughout the course of the study. An aliquot of the resultant extractant was mixed with an internal standard solution and the concentration of fluopicolide in the extract determined by LC-MS/MS using the same conditions described in the method above. No validation procedures were carried out for these samples.

4. Evaluation of the Data and Kinetic Calculations

For evaluation of degradation kinetics of fluopicolide according to the FOCUS guidance document on degradation kinetics, the total residue of the test item in the soil profile covering all soil horizons was calculated according to the following procedure:

- values between LOD and LOQ were set to the measured values.
- values < LOD were set to 0.5 LOD for samples after, before or deeper as a value > LOD or for samples between (> LOD and <LOQ). The curve was cut off after the first non-detect (< LOD), if no later value > LOQ followed.
- at day 0, values < LOD in deep horizons were set to 0.

The results in [µg/kg] were converted to [g/ha] considering the actual soil density of the corresponding soil layer.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide in soil samples by HPLC-MS/MS.

B. Data:

The results for residues of fluopicolide in different soil depths are presented below (expressed as g/ha) in Table 7.1.2.2.1- 49 to Table 7.1.2.2.1- 51.

Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the Burscheid trial (Table 7.1.2.2.1- 49). In soil depths below 10 cm no residues of fluopicolide were found above the LOQ except for one timepoint in one of three replicate subplots. In samples taken 121 to 701 days after application low residues were detected in the 10-20 cm horizon at concentrations ranging from <LOD to 10.2 g/ha and at two timepoints in the 20-30 cm horizon at concentrations <LOQ. No residues were detected in lower soil depths.

Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the Great Chishill trial (Table 7.1.2.2.1- 50). In samples taken 68 to 751 days after application low residues were detected in the 10-20 cm horizon at concentrations ranging from 3.36 to 16.8 g/ha. In deeper depths no residues of fluopicolide were found above the LOQ. In samples taken from 301 days occasional residues were detected at concentrations <LOQ in deeper soil horizons.

Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the Lignieres de Touraine trial (Table 7.1.2.2.1- 51). In samples taken 277 to 700 days after application low residues were detected in the 10-20 cm horizon at concentrations below the LOQ, ranging from <LOD to 7.43 g/ha. In deeper depths no residues of fluopicolide were found above the LOD.

Table 7.1.2.2.1- 49: Residues of fluopicolide in different soil depths at Burscheid trial after an application of 400 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	7	15	21	28	63	121	172	252	395	437	519	605	701
0-10	T1	499	435	393	418	390	363	136	197	161	121	94.9	73.4	51.3	60.1
	T2	488	418	530	463	467	452	183	231	206	167	143	116	73.4	76.4
	T3	366	534	668	502	369	387	245	194	131	136	110	85.6	99.7	66.5
	mean	451	462	530	461	409	401	188	206	166	141	116	91.7	74.8	67.7
10-20	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	[3.64]	10.3	[6.40]	[6.55]	[2.82]	<LOD	<LOD	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	[6.61]	[5.33]	[5.08]	<LOD	[5.22]	<LOD	[4.92]	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	[6.53]	[4.08]	<LOD	[3.89]	[4.17]	[3.04]	<LOD	[3.48]
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	[5.59]	[6.54]	[3.83]	[3.48]	[4.07]	[1.01]	[1.64]	[1.16]
20-30	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[3.53]	<LOD	<LOD	<LOD	[3.11]	<LOD	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[4.45]	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[2.66]	<LOD	<LOD	<LOD	[1.04]	<LOD	<LOD
30-40	T1	-	-	-	-	-	-	-	<LOD	<LOD	-	-	<LOD	-	-
	T2	-	-	-	-	-	-	-	<LOD	<LOD	-	-	-	-	-
	T3	-	-	-	-	-	-	-	-	<LOD	-	-	-	-	-
	mean	-	-	-	-	-	-	-	<LOD	<LOD	-	-	<LOD	-	-
40-50	T1	-	-	-	-	-	-	-	<LOD	<LOD	-	-	<LOD	-	-
	T2	-	-	-	-	-	-	-	<LOD	<LOD	-	-	-	-	-
	T3	-	-	-	-	-	-	-	-	<LOD	-	-	-	-	-
	mean	-	-	-	-	-	-	-	<LOD	<LOD	-	-	<LOD	-	
Sum	T1	499	435	393	418	390	363	140	211	167	128	97.7	76.5	51.3	60.1
	T2	488	418	530	463	467	452	190	241	211	167	148	116	78.3	76.4
	T3	366	534	668	502	369	387	252	195	131	140	114	88.6	99.7	70.0
	mean	451	462	530	461	409	401	194	216	170	145	120	93.7	76.4	68.8

LOD = 1.5 µg/kg equivalent to *ca.* 2.8 g/ha depending on soil moisture and density, LOQ = 5 µg/kg equivalent to *ca.* 9 g/ha depending on soil moisture and density,
Values in square brackets are values < LOD but > LOQ

Table 7.1.2.2.1- 50: Residues of fluopicolide in different soil depths at Great Chishill trial after an application of 400 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	7	15	22	29	68	134	194	301	398	468	554	667	751
0-10	T1	310	319	346	232	269	350	157	157	175	149	162	125	105	81.9
	T2	331	333	266	259	293	325	212	209	156	155	148	128	107	87.1
	T3	296	375	471	324	357	400	200	202	181	150	148	179	167	120
	mean	312	342	361	272	306	358	190	189	171	151	153	143	126	96.3
10-20	T1	-	<LOD	<LOD	<LOD	<LOD	[7.78]	14.4	[6.21]	[7.96]	11.1	15.0	10.9	[7.32]	12.7
	T2	-	<LOD	<LOD	<LOD	<LOD	16.8	14.1	14.7	15.8	24.0	[9.30]	14.0	13.6	10.8
	T3	-	<LOD	<LOD	<LOD	<LOD	[3.36]	11.2	11.8	[7.99]	13.9	[7.89]	11.5	12.5	12.3
	mean	-	<LOD	<LOD	<LOD	<LOD	[9.31]	13.2	[10.9]	[10.6]	16.3	[10.7]	12.1	[11.1]	11.9
20-30	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[2.81]	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[0.94]	<LOD
30-40	T1	-	-	-	-	-	-	-	<LOD	[4.49]	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	-	<LOD	<LOD	[2.83]	<LOD	<LOD	<LOD	[4.14]
	T3	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	-	<LOD	[1.50]	[0.94]	<LOD	<LOD	<LOD	[1.38]
40-50	T1	-	-	-	-	-	-	-	-	<LOD	-	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	[3.90]	-	-	-	[3.79]
	T3	-	-	-	-	-	-	-	-	-	-	-	-	-	<LOD
	mean	-	-	-	-	-	-	-	-	<LOD	[1.30]	-	-	-	[1.26]
Sum	T1	310	319	346	232	269	358	171	163	187	160	177	134	112	94.6
	T2	331	333	266	259	293	342	226	224	172	186	157	142	123	106
	T3	296	375	471	324	357	403	211	214	189	164	156	191	180	132
	mean	312	342	361	272	306	368	203	200	183	170	163	156	138	111

LOD = 1.5 µg/kg equivalent to *ca.* 2.8 g/ha depending on soil moisture and density, LOQ = 5 µg/kg equivalent to *ca.* 9 g/ha depending on soil moisture and density
Values in square brackets are values < LOD but > LOQ

Table 7.1.2.2.1- 51: Residues of fluopicolide in different soil depths at Lignieres de Touraine trial after an application of 400 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	7	14	21	28	56	120	166	277	348	435	530	645	700
0-10	T1	399	323	309	311	326	291	198	195	175	147	96.9	82.9	87.4	65.7
	T2	327	318	293	306	307	283	205	227	174	151	97.8	89.0	79.8	54.6
	T3	342	305	310	280	293	259	174	200	178	143	92.1	69.7	70.1	45.1
	mean	356	315	304	299	309	278	192	207	176	147	95.6	80.5	79.1	55.1
10-20	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[6.08]	[6.97]	<LOD	<LOD	[4.01]	[5.47]
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[4.34]	[5.29]	<LOD	<LOD	<LOD	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[4.71]	[7.43]	[4.41]	<LOD	<LOD	[4.58]
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[5.04]	[6.56]	[1.47]	<LOD	[1.34]	[3.35]
20-30	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
30-40	T1	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	T3	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
40-50	T1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	T3	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Sum	T1	399	323	309	311	326	291	198	195	181	154	96.9	82.9	91.4	71.2
	T2	327	318	293	306	307	283	205	227	178	156	97.8	89.0	79.8	54.6
	T3	342	305	310	280	293	259	174	200	183	150	96.5	69.7	70.1	49.7
	mean	356	315	304	299	309	278	192	207	181	153	97.1	80.5	80.4	58.5

LOD = 1.5 µg/kg equivalent to *ca.* 2.8 g/ha depending on soil moisture and density, LOQ = 5 µg/kg equivalent to *ca.* 9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ

The dissipation of fluopicolide with time is presented in Table 7.1.2.2.1- 52 to Table 7.1.2.2.1- 54. The values have been pre-processed according to the procedure described in FOCUS kinetics guidance (as described earlier). Actual values are given in brackets.

At Burscheid (Germany), the mean amount of fluopicolide at day 0 was 451 g/ha, representing 113% of the nominal application rate. Fluopicolide declined from 451 g/ha in soil at day 0 to 68.8 g/ha at day 701. At Great Chishill (United Kingdom), the mean amount of fluopicolide at day 0 was 312 g/ha, representing 78.0% of the nominal application rate. Fluopicolide declined from 312 g/ha in soil at day 0 to 111 g/ha at day 751. At Lignieres de Touraine (France), the mean amount of fluopicolide at day 0 was 356 g/ha, representing 89% of the nominal application rate. Fluopicolide declined from 356 g/ha in soil at day 0 to 58.5 g/ha at day 700. The dissipation of fluopicolide showed biphasic behaviour. After treatment, fluopicolide dissipated initially very rapidly within a couple of days followed by a second slower phase until study termination. Residues of fluopicolide in control samples were < LOD for all samples taken.

Table 7.1.2.2.1- 52: Residues of fluopicolide in soil from the Burscheid trial after an application of 400 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Burscheid (Germany)	DAT													
	0	**7**	**15**	**21**	**28**	**63**	**121**	**172**	**252**	**395**	**437**	**519**	**605**	**701**
T1	499 (499)	438 (435)	396 (393)	421 (418)	393 (390)	366 (363)	143 (140)	214 (211)	170 (167)	130 (128)	100 (97.7)	82.4 (76.5)	57.3 (51.3)	63.0 (60.1)
T2	488 (488)	421 (418)	533 (530)	466 (463)	470 (467)	455 (452)	193 (190)	244 (241)	214 (211)	170 (167)	151 (148)	119 (116)	81.2 (78.3)	79.4 (76.4)
T3	366 (366)	537 (534)	671 (668)	505 (502)	372 (369)	390 (387)	254 (252)	198 (195)	134 (131)	143 (140)	117 (114)	91.6 (88.6)	103 (99.7)	72.8 (70)
Mean	**451 (451)**	**465 (462)**	**533 (530)**	**464 (461)**	**412 (409)**	**404 (401)**	**197 (194)**	**219 (216)**	**173 (170)**	**148 (145)**	**123 (120)**	**97.7 (93.7)**	**80.5 (76.4)**	**71.7 (68.8)**
Min	366 (366)	421 (418)	396 (393)	421 (418)	372 (369)	366 (363)	143 (140)	198 (195)	134 (131)	130 (128)	100 (97.7)	82.4 (76.5)	57.3 (51.3)	63.0 (60.1)
Max	499 (499)	537 (534)	671 (668)	505 (502)	470 (467)	455 (452)	254 (252)	244 (241)	214 (211)	170 (167)	151 (148)	119 (116)	103 (99.7)	79.4 (76.4)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	103 (102)	118 (118)	103 (102)	91 (91)	90 (89)	44 (43)	49 (48)	38 (38)	33 (32)	27 (27)	22 (21)	18 (17)	16 (15)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3
The actual values are given in brackets (expressed as g/ha)

Table 7.1.2.2.1- 53: Residues of fluopicolide in soil from the Great Chishill trial after an application of 400 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Great Chishill (UK)	DAT													
	0	**7**	**15**	**22**	**29**	**68**	**134**	**194**	**301**	**398**	**468**	**554**	**667**	**751**
T1	310 (310)	322 (319)	349 (346)	235 (232)	272 (269)	360 (358)	174 (171)	169 (163)	192 (187)	166 (160)	180 (177)	137 (134)	115 (112)	97.4 (94.6)
T2	331 (331)	336 (333)	269 (266)	262 (259)	296 (293)	345 (342)	229 (226)	227 (224)	175 (172)	191 (186)	160 (157)	145 (142)	126 (123)	111 (106)
T3	296 (296)	378 (375)	474 (471)	327 (324)	360 (357)	406 (403)	214 (211)	217 (214)	192 (189)	167 (164)	159 (156)	193 (191)	182 (180)	135 (132)
Mean	**312 (312)**	**345 (342)**	**364 (361)**	**275 (272)**	**309 (304)**	**370 (368)**	**206 (203)**	**204 (200)**	**186 (183)**	**175 (170)**	**166 (163)**	**158 (156)**	**141 (138)**	**114 (111)**
Min	296 (296)	322 (319)	269 (266)	235 (232)	272 (269)	345 (342)	174 (171)	169 (163)	175 (172)	166 (160)	159 (156)	137 (134)	115 (112)	97.4 (94.6)
Max	331 (331)	378 (375)	474 (471)	327 (324)	360 (357)	406 (403)	229 (226)	227 (224)	192 (189)	191 (186)	180 (177)	193 (191)	182 (180)	135 (132)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	111 (110)	117 (116)	88 (87)	99 (97)	119 (118)	66 (65)	65 (64)	60 (59)	56 (54)	53 (52)	51 (50)	45 (44)	37 (36)

DAT = Days after treatment; TX = Treated Subalot X with X = 1 to 3.
The actual values are given in brackets (expressed as g/ha)

Table 7.1.2.2.1- 54: Residues of fluopicolide in soil from the Lignieres de Touraine trial after an application of 400 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Lignieres de Touraine (France)	DAT (Days)													
	0	**7**	**14**	**21**	**28**	**56**	**120**	**166**	**277**	**348**	**435**	**530**	**645**	**700**
T1	399 (399)	326 (323)	312 (309)	314 (311)	329 (326)	294 (291)	201 (198)	198 (195)	184 (181)	157 (154)	99.9 (96.9)	86.0 (82.9)	94.3 (91.4)	74.0 (71.2)
T2	327 (327)	321 (318)	296 (293)	309 (306)	310 (307)	286 (283)	208 (205)	230 (227)	181 (178)	159 (156)	101 (97.8)	92.0 (89.0)	82.9 (79.8)	57.6 (54.6)
T3	342 (342)	308 (305)	313 (310)	283 (280)	296 (293)	262 (259)	177 (174)	203 (200)	186 (183)	153 (150)	99.4 (96.5)	72.9 (69.7)	73.4 (70.1)	52.5 (49.7)
Mean	**356 (356)**	**318 (315)**	**307 (304)**	**302 (299)**	**312 (309)**	**281 (278)**	**195 (192)**	**210 (207)**	**184 (181)**	**156 (153)**	**100 (97.1)**	**83.6 (80.5)**	**83.5 (80.4)**	**61.4 (58.5)**
Min	327 (327)	308 (305)	296 (293)	283 (280)	296 (293)	262 (259)	177 (174)	198 (195)	181 (178)	153 (150)	99.4 (96.5)	72.9 (69.7)	73.4 (70.1)	52.5 (49.7)
Max	399 (399)	326 (323)	313 (310)	314 (311)	329 (326)	294 (291)	208 (205)	230 (227)	186 (183)	159 (156)	101 (97.8)	92.0 (89.0)	94.3 (91.4)	74.0 (71.2)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	89 (88)	86 (85)	85 (84)	88 (87)	79 (78)	55 (54)	59 (58)	52 (51)	44 (43)	28 (27)	23 (23)	23 (23)	17 (16)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3.
The actual values are given in brackets (expressed as g/ha)

The concentration of fluopicolide in the 0.01M calcium chloride extracts are shown in Table 7.1.2.2.1-55 to Table 7.1.2.2.1- 57.

Table 7.1.2.2.1- 55: Residues of fluopicolide in the aqueous $CaCl_2$ extracts from soil from the Burscheid trial after an application of 400 g a.s./ha (expressed as g/ha)

Burscheid (Germany)	DAT				
	0	**63**	**172**	**395**	**701**
T1	154	93.1	41.1	[29.1]	[17.8]
T2	164	106	49.7	39.4	[21.2]
T3	112	99.7	46.3	[31.2]	[20.3]
Mean	**143.3**	**69.6**	**45.7**	**33.2**	**19.8**
Min	112	93.1	41.1	29.1	17.8
Max	164	106	49.7	39.4	21.2
n	3	3	3	3	3

DAT = Days after Treatment, LOQ = 15 µg/kg, LOD = 5 µg/kg, [x] = LOD >< LOQ

Table 7.1.2.2.1- 56: Residues of fluopicolide in the aqueous $CaCl_2$ extracts from soil from the Great Chishill trial after an application of 400 g a.s./ha (expressed as g/ha)

Great Chishill (United Kingdom)	DAT (Days)				
	0	**68**	**194**	**398**	**751**
T1	85.5	87.4	41.9	[33.9]	[20.7]
T2	101	84.5	49.8	[34.8]	<LOD
T3	81.0	81.4	38.3	[27.5]	[22.1]
Mean	**89.2**	**84.4**	**43.3**	**32.0**	**14.3**
Min	81.0	81.4	38.3	[27.5]	<LOD
Max	101	87.4	49.8	[34.8]	[22.1]
n	3	3	3	3	3

DAT = Days after Treatment, LOQ = 15 µg/kg, LOD = 5 µg/kg, [x] = LOD >< LOQ

Table 7.1.2.2.1- 57: Residues of fluopicolide in the aqueous $CaCl_2$ extracts from soil from the Lignieres de Touraine trial after an application of 400 g a.s./ha (expressed as g/ha)

Lignieres de Touraine (France)	DAT (Days)				
	0	**56**	**166**	**348**	**700**
T1	130	82.4	62.5	39.2	[22.2]
T2	103	79.4	[35.9]	40.5	[19.2]
T3	110	74.7	59.6	[38.7]	<LOD
Mean	**114.3**	**78.8**	**52.7**	**39.5**	**13.8**
Min	103	74.7	[35.9]	[38.7]	<LOD
Max	130	82.4	62.5	40.5	[22.2]
n	3	3	3	3	3

DAT = Days after Treatment, LOQ = 15 µg/kg, LOD = 5 µg/kg, [x] = LOD >< LOQ

III. Conclusion

Under field conditions fluopicolide declined moderately and residues were translocated up to 30 cm (Germany), 50 cm (United Kingdom), and 20 cm (France) depth, respectively. Un-normalised DT_{50} values for the degradation of fluopicolide calculated from the reported data following the recommendations of the FOCUS work group details are provided in Document KCA 7.1.2.2.1/14.

Assessment and conclusion by applicant:

The study is considered valid to assess fluopicolide soil $DegT_{50matrix}$ values for field studies as defined by EFSA (2014). The endpoints may be too conservative to assess persistence as the design minimized soil surface processes as required by EFSA (2014) and such processes may contribute to dissipation.

Data Point:	KCA 7.1.2.2.1/13
Report Author:	[redacted]
Report Year:	2019
Report Title:	Terrestrial field dissipation study with fluopicolide & propamocarb-hydrochloride SC 687.5 in France (South), Italy and Spain
Report No:	M-651179-01-1
Document No:	M-651179-01-1
Guideline(s) followed in study:	Regulation (EC) No. 1107/2009 of the European Parliament and of the Council of 21 October 2009 including Data Requirements SANCO/11803/2010 Rev. 7 and Test Method SANCO/11843/2010 Rev. 4 EFSA Guidance Document for Evaluating Laboratory and Field Dissipation Studies to obtain DegT50 Values of Active Substances of Plant Protection Products and Transformation Products of these Active Substances in Soil, EFSA Journal 2014; 12(5):3662, 2014
Deviations from current test guideline:	Yes. Report meets the requirement for assessing test substance soil DegT50matrix values as required by EFSA (2014) for field studies. The endpoints may be too conservative for comparison to field persistence criteria and ecotoxicological risk assessment as required by EU 283/201.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

Soil degradation of fluopicolide under Southern European field conditions was investigated after application of the formulation Fluopicolide and Propamocarb-hydrochloride SC 687.5 onto bare soil plots in St. Etienne du Gres (France), Albaro (Italy) and Vilobi d'Onyar (Spain).

Fluopicolide and Propamocarb-hydrochloride SC 687.5 was sprayed once onto 400 sqm to 600 sqm plots at a rate of 6.40 L/ha, corresponding to nominal 400 g/ha fluopicolide. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface.

Soil cores were taken 0 days before up to 722 days post-application to a maximum depth of 110 cm. The soil cores were cut into 10-cm soil layers, bulked soil layers were homogenised and finally analysed for fluopicolide.

Sub-samples of homogenised soil were extracted in a microwave extractor with organic solvent. Potential matrix effects were eliminated by using an internal standard solution of isotopically labeled reference items added to sample extracts. Following separation of fine particles from soil extracts by centrifugation the identification and quantitation of the analytes was performed by high performance liquid chromatography using MS/MS detection in the multiple reaction monitoring mode. The analytical method was validated using three different soils. The limit of quantitation (LOQ) was 5.0 µg/kg and the limit of detection (LOD) was 1.5 µg/kg for fluopicolide.

At St. Etienne du Grès (France South), the mean amount of Fluopicolide at day 0 was 381 g/ha, representing 95% of the nominal application rate. Fluopicolide declined from 381 g/ha in soil at day 0 to 118 g/ha at day 714.

At Albaro (Italy), the mean amount of Fluopicolide at day 0 was 412 g/ha, representing 103% of the nominal application rate. Fluopicolide declined from 412 g/ha in soil at day 0 to 71.2 g/ha at day 722.

At Vilobi d'Onyar (Spain), the mean amount of Fluopicolide at day 0 was 391 g/ha, representing 97.8% of the nominal application rate. Fluopicolide declined from 391 g/ha in soil at day 0 to 35.7 g/ha at day 714.

1. Materials and Methods

A. Materials

1. Test Item

Fluopicolide + propamocarb-hydrochloride formulated as a suspension concentrate (62.5g/L fluopicolide + 625 g/L of propamocarb-hydrochloride)

Certificate of Analysis: 01860-00

Lot No: 2015-000846-01

2. Trial Location & Soil

A terrestrial field dissipation with Fluopicolide & Propamocarb-hydrochloride (SC 687.5) is a suspension concentrate formulation containing 62.5 g/L fluopicolide and 625 g/L propamocarb-hydrochloride was conducted at three locations in Southern Europe. The three locations were St. Etienne du Grès (France), Albaro (Italy) and Vilobi d'Onyar (Spain). The sites were fully characterised, and the results summarised in Table 7.1.2.2.1-58. The plot sizes ranged from 400 sqm to 600 sqm. The control plot was prepared at least 5 m away from the treated plots.

Soil from the three test sites have been used in OECD 307 time dependent sorption and OECD 106 adsorption desorption studies.

Table 7.1.2.2.1- 58: Location, site description and climatic data of test sites

Characteristic	Units	Sampling depth [cm]			
		0-30	30-50	50-75	75-100
Soil Designation	-	**St. Etienne du Grès (France)**			
Soil ID	-	FR08			
Geographic Location City Country	-	[redacted] 13103 St. Etienne du Grès, Provence-Alpes-Côte d'Azur France			
pH	$CaCl_2$	7.7	7.7	7.8	7.8
Organic carbon	[% Carbon]	0.8	0.6	0.6	0.4
CEC	[meq/100 g]	11.7	11.7	13.2	14.0
Chalk	[% $CaCO_3$]	42.1	42.1	42.5	43.7
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 29 45 26	 31 47 22	 39 41 20	 43 39 18
Textural class	USDA	clay loam	clay loam	clay loam	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 46.2 29.5	 43.4 26.8	 48.3 29.3	 51.4 25.0
Soil Designation	-	**Albaro (Italy)**			
Soil ID	-	IT23			
Geographic Location City Country		[redacted] 37055 Albaro di Ronco, all'Adige, Veneto Italy			
pH	$CaCl_2$	7.3	7.4	7.5	7.4
Organic carbon	[% Carbon]	1.8	1.5	0.7	0.6
CEC	[meq/100 g]	19.7	20.1	17.9	17.1
Chalk	[% $CaCO_3$]	10.6	12.0	14.1	11.8
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 35 43 22	 33 45 22	 35 45 20	 41 39 20
Textural class	USDA	clay loam	clay loam	clay loam	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 61.5 35.5	 62.7 35.9	 67.8 41.1	 65.5 37.6
Soil Designation	-	**Vilobi d'Onyar (Spain)**			
Soil ID	-	SPA1			
Geographic Location City Country		[redacted] 17185 Vilobi d'Onyar, Catalonia Spain			
pH	$CaCl_2$	6.0	6.1	6.6	7.0
Organic carbon	[% Carbon]	0.8	0.3	0.1	0.1
CEC	[meq/100 g]	9.6	11.9	13.5	14.2
Chalk	[% $CaCO_3$]	0.3	0.2	0.1	0.2
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 17 33 50	 27 23 50	 29 15 56	 27 15 58
Textural class	USDA	loam	sandy clay loam	sandy clay loam	sandy clay loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 41.4 21.5	 44.5 20.7	 47.9 23.9	 47.3 22.4

B. Study Design

1. Experimental Conditions

Fluopicolide & Propamocarb-hydrochloride SC 687.5 is a suspension concentrate formulation, containing 62.5 g/L fluopicolide and 625 g/L propamocarb-hydrochloride. The product was sprayed onto bare earth once at each site at an application rate of 6.40 L/ha and 600 L/ha water, corresponding to 400 g/ha fluopicolide during May and June 2015. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface. Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of a nonselective herbicide to control weeds.

Air temperature, precipitation including irrigation and sunshine data were recorded on site during the field tests.

Soil dissipation of fluopicolide was studied for 722 days.

2. Sampling

The treated plot of the trial was divided into three sub-plots. From each sub-plot 10 soil cores were taken and combined together at each sampling interval (30 cores in total).

Samples were taken from treated plots on following occasions: 0 (post-application; 0-10 cm depth), 6-8, 13-15, 21-22, 27-28 (each 0-40 cm depth), 56-58, 118-128 (each 0-60 cm depth) and 167-175, 279-283, 352-370, 450-489, 519-546, 618-646, 714-722 (each 0-85 cm depth) after treatment. From the control plot samples were taken on the following occasions: 0 days before application, 352-369 and 714-722 days after application.

Soil cores taken from the three sites were deep frozen to -18°C within twenty four hours after sampling, then shipped frozen to the analytical laboratory in Germany.

3. Analytical Procedures

The analytical method 01445 was used to determine levels of fluopicolide. Soil samples of 5 g were extracted in a microwave extractor with a mixture of acetonitrile/water (4/1, v/v). The extracts were centrifuged to remove fine particles of the soil. Possible matrix effects of fluopicolide were eliminated by using an internal standard solution of isotopic labelled reference items. Quantification was carried out by LC-MS/MS. The limit of quantitation (LOQ) for fluopicolide was 5.0 µg/kg in soil. The limit of determination (LOD) for fluopicolide was 1.5 µg/kg.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide at levels of LOQ, 10 x LOQ and 1000 x LOQ and processed in parallel to the dissipation samples throughout the study. The results are summarised in the table below.

Single Values [%]	No of Recoveries	Fortification level [µg/kg]	Mean [%]	RSD [%]
81, 84, 84, 85, 88, 88, 90, 90, 92, 92, 93, 93, 93, 93, 93, 94, 94, 95, 96, 96, 96, 96, 96, 96, 96, 96, 96, 96, 97, 97, 97, 97, 97, 98, 98, 98, 98, 98, 98, 98, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 101, 101, 101, 101, 101, 101, 101, 101, 101, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 105, 106, 106, 106, 106, 107, 108, 109, 109, 109, 109	125	5	100	5.2
92, 93, 93, 94, 94, 94, 94, 95, 95, 95, 96, 96, 96, 96, 96, 97, 97, 97, 98, 98, 99, 99, 99, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 101, 101, 101, 101, 101, 101, 101, 101, 101, 101, 101, 102, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 106, 106, 106, 106, 106, 106, 106, 106, 106, 106, 107, 107, 107, 107, 107, 107, 107, 107, 108, 108, 108, 108, 108, 108, 108, 109, 109, 109, 109, 109, 109, 109, 110, 110, 110, 110, 110, 111, 112, 112, 112, 112, 113, 113, 113, 114	119	50	103	4.9
76, 76, 78, 78, 79, 85	6	5000	79	4.2
Overall recovery	250		101	6.4

RSD = Relative standard deviation, LOQ = Limit of quantification

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

Additional 10 g soil samples were extracted by shaking with 0.01M calcium chloride at selected timepoints throughout the course of the study. An aliquot of the resultant extractant was mixed with an internal standard solution and the concentration of fluopicolide in the extract determined by LC-MS/MS using the same conditions described in the method above. No validation procedures were carried out for these samples.

4. Evaluation of the Data and Kinetic Calculations

For evaluation of degradation kinetics of the test item according to the FOCUS guidance document on degradation kinetics, the total residue of the test item in the soil profile covering all soil horizons was calculated according to the following procedure:

- values between LOD and LOQ were set to the measured values.
- values < LOD were set to 0.5 LOD for samples after, before or deeper as a value > LOD or for samples between (> LOD and <LOQ). The curve was cut off after the first non-detect (< LOD), if no later value > LOQ followed.
- at day 0, values < LOD in deep horizons were set to 0.

The results in [µg/kg] were converted to [g/ha] considering the actual soil density of the corresponding soil layer.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide in soil samples by HPLC-MS/MS.

B. Data:

The results for residues of fluopicolide in different soil depths are presented below (expressed as g/ha) in Table 7.1.2.2.1- 59 to Table 7.1.2.2.1- 61.

Residues of fluopicolide were detected largely in the 0-10 cm soil horizon throughout the trial at St. Etienne du Grès. In samples taken 7 to 714 days after application low residues were detected in the 10-20 cm horizon at concentrations ranging from 2.84 to 36.8 g/ha. In soil depths below 20 cm no residues of fluopicolide were found above the LOQ except for one timepoint in one of three replicate subplots. In samples taken 28 to 714 days after application low residues were detected in the 20-30 cm horizon at concentrations ranging from <LOD to 9.20 g/ha and at two timepoints in the 30-40 cm horizon at concentrations <LOQ.

Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the Albaro trial. In samples taken at 13 days and from 175 to 722 days after application low residues were detected in the 10-20 cm horizon at concentrations ranging from <LOD to 18.2 g/ha. In deeper depths no residues of fluopicolide were found above the LOQ. In samples taken at 364 and 405 days residues were detected at concentrations <LOQ in the 20-30 cm soil layer but not in deeper layers.

Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the Vilobi d'Onyar trial. In samples taken 279 to 546 days after application low residues were detected in the 10-20 cm horizon at concentrations below the LOQ, ranging from <LOD to 6.84 g/ha. In deeper depths no residues of fluopicolide were found above the LOD.

Table 7.1.2.2.1- 59: Residues of fluopicolide in different soil depths from the St. Etienne du Grès trial after an application of 400 g a.s./ha (g/ha)

Depth [cm]	Sub plot	DAT													
		0	7	14	21	28	58	118	167	280	352	451	519	646	714
0-10	T1	380	360	319	321	302	255	219	207	170	143	136	119	105	96.3
	T2	332	359	365	334	313	279	239	209	190	169	148	123	103	95.7
	T3	432	383	333	340	293	263	197	181	167	181	154	129	147	122
	mean	381	367	339	332	303	266	218	199	176	164	146	122	116	105
10-20	T1	-	[9.12]	14.2	25.6	10.4	12.5	24.0	18.2	10.0	26.6	9.27	15.0	11.9	11.6
	T2	-	16.1	[8.42]	19.5	[5.80]	36.8	[6.15]	19.8	13.8	18.5	[8.68]	14.5	13.4	11.6
	T3	-	17.6	33.0	21.9	24.2	28.0	10.5	24.7	17.3	20.1	[2.84]	15.0	14.7	17.7
	mean	-	14.3	18.5	22.3	13.5	25.8	13.6	20.9	13.7	21.7	[6.9]	14.8	13.3	13.6
20-30	T1	-	<LOD	<LOD	<LOD	<LOD	[4.15]	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	9.20	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	<LOD	<LOD	<LOD	[4.53]	<LOD	<LOD	[3.20]	[4.11]	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	<LOD	<LOD	<LOD	[1.51]	[4.38]	<LOD	[1.07]	[1.37]	<LOD	<LOD	<LOD	<LOD	<LOD
30-40	T1	-	-	<LOD	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	-	-	-	-
	T2	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	-	-	-	-
	T3	-	-	-	-	[3.38]	-	<LOD	[3.74]	<LOD	<LOD	-	-	-	-
	mean	-	-	<LOD	-	[1.13]	<LOD	<LOD	[1.25]	<LOD	<LOD	-	-	-	-
40-50	T1	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	-	-	-	-
	T2	-	-	-	-	-	<LOD	-	<LOD	<LOD	<LOD	-	-	-	-
	T3	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	-	-	-	-
	mean	-	-	-	-	-	<LOD	-	<LOD	<LOD	<LOD	-	-	-	-

Depth [cm]	Sub plot	DAT													
		0	7	14	21	28	58	118	167	280	352	451	519	646	714
Sum	T1	380	369	333	347	312	272	243	225	180	170	145	134	117	108
	T2	332	375	373	354	319	325	245	229	204	188	157	138	116	107
	T3	432	401	366	362	325	291	208	213	188	201	152	138	136	140
	mean	381	382	357	354	319	296	232	222	191	186	153	137	130	118

LOD = 1.5 µg/kg equivalent to *ca.* 2.8 g/ha depending on soil moisture and density
LOQ = 5 µg/kg equivalent to *ca.* 9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ

Table 7.1.2.2.1- 60: Residues of fluopicolide in different soil depths from the Albaro trial after an application of 400 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	6	13	21	27	57	121	175	283	364	450	541	618	722
0-10	T1	427	322	392	248	230	274	201	180	189	147	102	65.1	53.9	51.4
	T2	385	407	356	329	323	236	269	175	337	174	100	93.6	64.7	64.3
	T3	425	362	455	304	327	293	230	197	182	154	122	92.9	79	73.4
	mean	412	364	401	294	293	268	233	184	203	158	108	83.9	65.9	63.0
10-20	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[7.37]	13.5	12.2	[4.05]	[3.99]	[7.79]	[8.49]
	T2	-	<LOD	[4.20]	<LOD	<LOD	<LOD	<LOD	[4.59]	16.8	11.1	[6.90]	8.9	10.8	[9.35]
	T3	-	<LOD	18.2	<LOD	<LOD	<LOD	<LOD	<LOD	11.5	16.4	[5.09]	[6.02]	10.0	[6.60]
	mean	-	<LOD	[7.47]	<LOD	<LOD	<LOD	<LOD	[3.99]	13.9	13.2	[5.35]	[6.30]	9.53	[8.15]
20-30	T1	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[3.07]	<LOD	<LOD	<LOD	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[3.58]	<LOD	<LOD	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[3.61]	<LOD	<LOD	<LOD	<LOD
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[2.23]	[1.19]	<LOD	<LOD	<LOD
30-40	T1	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	-	<LOD	-	-	-
	T3	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-

Depth [cm]	Sub plot	DAT													
		0	6	13	21	27	57	121	175	283	364	450	541	618	722
	mean	-	-	-	-	-	-	-	-	-	<LOD	<LOD			-
40-50	T1	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	-	<LOD	-	-	-
	T3	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	<LOD	<LOD			-
Sum	T1	427	322	392	248	230	274	201	187	203	162	106	69.1	61.7	59.9
	T2	385	407	360	329	323	236	269	180	254	185	110	103	75.5	73.7
	T3	425	362	473	304	327	293	230	197	194	174	127	98.9	89.0	80.0
	mean	412	364	408	294	293	268	233	188	217	174	114	90.3	75.4	71.2

LOD = 1.5 µg/kg equivalent to *ca.* 2.8 g/ha depending on soil moisture and density
LOQ = 5 µg/kg equivalent to *ca.* 9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ

Table 7.1.2.2.1- 61: Residues of fluopicolide in different soil depths from the Vilobi d'Onyar trial after an application of 400 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	8	15	22	28	56	128	167	279	370	489	546	628	714
0-10	T1	388	363	323	257	303	293	203	160	174	111	62.2	58.9	39.3	38.7
	T2	381	367	332	310	294	272	176	151	153	87.9	67.3	65.1	52.0	32.8
	T3	403	339	319	302	318	263	195	188	169	116	65.8	59.8	51.3	35.7
	mean	391	356	325	290	305	276	191	166	165	105	65.1	61.3	47.5	35.7
10-20	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[6.34]	[6.84]	<LOD	[4.23]	<LOD	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[5.80]	<LOD	<LOD	[3.27]	<LOD	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[4.05]	[2.28]	<LOD	[2.50]	<LOD	<LOD

Depth [cm]	Sub plot	DAT													
		0	8	15	22	28	56	128	167	279	370	489	546	628	714
20-30	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
30-40	T1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	T3	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	-	-	-	-	-
40-50	T1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	T3	-	-	-	-	-	-	-	-	-	-	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Sum	T1	388	363	323	257	303	293	203	160	180	118	62.2	63.1	39.3	38.7
	T2	381	367	332	310	294	272	176	151	159	87.9	67.3	68.4	52.0	32.8
	T3	403	339	319	302	318	263	195	188	169	116	65.8	59.8	51.3	35.7
	mean	391	356	325	290	305	276	191	166	169	107	65.1	63.8	47.5	35.7

LOD = 1.5 µg/kg equivalent to *ca.* 2.8 g/ha depending on soil moisture and density
LOQ = 5 µg/kg equivalent to *ca.* 9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ

The dissipation of fluopicolide with time is presented in Table 7.1.2.2.1- 62 to Table 7.1.2.2.1- 64. The values have been pre-processed according to the procedure described in FOCUS kinetics guidance (as described earlier). Actual values are given in brackets.

At St. Etienne du Grès (France), the mean amount of fluopicolide at day 0 was 381 g/ha, representing 95.3% of the nominal application rate. Fluopicolide declined from 381 g/ha in soil at day 0 to 118 g/ha at day 714. At Albaro (Italy), the mean amount of fluopicolide at day 0 was 412 g/ha, representing 103% of the nominal application rate. Fluopicolide declined from 412 g/ha in soil at day 0 to 71.2 g/ha at day 722. At Vilobi d'Onyar (Spain), the mean amount of fluopicolide at day 0 was 391 g/ha, representing 97.8% of the nominal application rate. Fluopicolide declined from 391 g/ha in soil at day 0 to 35.7 g/ha at day 714.

The dissipation of fluopicolide showed biphasic behaviour. After treatment, fluopicolide dissipated initially very rapidly within a couple of days followed by a second slower phase until study termination. Residues of fluopicolide in control samples were < LOD for all samples taken.

Table 7.1.2.2.1- 62: Residues of fluopicolide in soil from the St. Etienne du Grès trial after an application of 400 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

St. Etienne du Grès (France)	DAT													
	0	**7**	**14**	**21**	**28**	**58**	**118**	**167**	**280**	**352**	**451**	**519**	**646**	**714**
T1	380 (380)	372 (369)	336 (333)	349 (347)	315 (312)	274 (272)	246 (243)	228 (225)	183 (180)	172 (170)	148 (145)	137 (134)	120 (117)	110 (108)
T2	332 (332)	378 (375)	376 (373)	356 (354)	322 (319)	328 (325)	248 (245)	232 (229)	206 (204)	190 (188)	159 (157)	140 (138)	119 (116)	110 (107)
T3	432 (432)	403 (401)	369 (366)	365 (362)	325 (325)	294 (291)	213 (208)	215 (213)	191 (188)	204 (201)	159 (157)	141 (138)	158 (156)	142 (140)
Mean	**381 (381)**	**384 (382)**	**360 (358)**	**357 (354)**	**321 (319)**	**299 (296)**	**236 (232)**	**225 (222)**	**193 (191)**	**189 (186)**	**155 (153)**	**139 (137)**	**132 (130)**	**121 (118)**
Min	332 (332)	372 (369)	336 (333)	349 (347)	315 (312)	274 (272)	213 (208)	215 (213)	183 (180)	172 (170)	148 (145)	137 (134)	119 (116)	110 (107)
Max	432 (432)	403 (401)	376 (373)	365 (362)	325 (325)	328 (325)	248 (245)	232 (229)	206 (204)	204 (201)	159 (157)	141 (138)	158 (156)	142 (140)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	101 (100)	94 (94)	94 (93)	84 (84)	78 (78)	62 (61)	59 (58)	51 (50)	50 (49)	41 (40)	36 (36)	35 (34)	32 (31)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3.
The actual values are given in brackets (expressed as g/ha)

Table 7.1.2.2.1- 63: Residues of fluopicolide in soil from the Albaro trial after an application of 400 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Albaro (Italy)	DAT													
	0	**6**	**13**	**21**	**27**	**57**	**121**	**175**	**283**	**364**	**450**	**541**	**618**	**722**
T1	427 (427)	325 (322)	395 (392)	250 (248)	233 (230)	277 (274)	204 (201)	190 (187)	205 (203)	165 (162)	109 (106)	71.7 (69.1)	64.2 (61.7)	62.6 (59.9)
T2	385 (385)	410 (407)	363 (360)	332 (329)	326 (323)	239 (236)	272 (269)	182 (180)	256 (254)	188 (185)	113 (110)	105 (103)	78.0 (75.5)	76.4 (73.7)
T3	425 (425)	365 (362)	476 (473)	307 (304)	329 (327)	296 (293)	233 (230)	200 (197)	196 (194)	177 (174)	130 (127)	102 (98.9)	91.5 (89.0)	82.6 (80.0)
Mean	**412 (412)**	**367 (363)**	**411 (408)**	**296 (293)**	**296 (293)**	**271 (267)**	**236 (233)**	**191 (188)**	**219 (216)**	**177 (173)**	**117 (115)**	**93.0 (90.1)**	**78.0 (75.4)**	**74.0 (71.2)**
Min	385 (385)	325 (322)	363 (360)	250 (248)	233 (230)	239 (236)	204 (201)	182 (179)	196 (193)	165 (162)	109 (106)	71.7 (69.0)	64.2 (61.7)	62.6 (59.9)
Max	427 (427)	410 (407)	476 (473)	332 (329)	329 (327)	296 (293)	272 (269)	200 (197)	256 (254)	188 (185)	130 (127)	105 (103)	91.5 (89.0)	82.6 (80.0)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	87 (86)	98 (97)	70 (70)	70 (70)	64 (64)	56 (55)	45 (45)	52 (51)	42 (41)	28 (27)	22 (21)	19 (18)	18 (17)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3
The actual values are given in brackets (expressed as g/ha)

Table 7.1.2.2.1- 64: Residues of fluopicolide in soil from the Vilobi d'Onyar trial after an application of 400 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Vilobi d'Onyar (Spain)	DAT													
	0	**8**	**15**	**22**	**28**	**56**	**128**	**167**	**279**	**370**	**489**	**546**	**628**	**714**
T1	388 (388)	366 (363)	326 (323)	260 (257)	306 (303)	296 (293)	204 (203)	163 (160)	183 (180)	121 (118)	65.1 (62.2)	66.2 (63.1)	42.7 (39.3)	41.9 (38.7)
T2	381 (381)	371 (367)	335 (332)	313 (310)	297 (294)	275 (272)	177 (176)	154 (151)	162 (159)	90.9 (87.9)	70.4 (67.3)	71.4 (68.4)	55.3 (52.0)	35.9 (32.8)
T3	403 (403)	342 (339)	323 (319)	305 (302)	321 (318)	265 (263)	197 (195)	191 (188)	172 (169)	119 (116)	68.8 (65.8)	63.0 (59.8)	54.5 (51.3)	38.6 (35.7)
Mean	**391 (391)**	**360 (356)**	**328 (325)**	**293 (290)**	**308 (305)**	**279 (276)**	**193 (191)**	**169 (166)**	**172 (169)**	**110 (107)**	**68.1 (65.1)**	**66.9 (63.8)**	**50.8 (47.5)**	**38.8 (35.7)**
Min	381 (381)	342 (339)	323 (319)	260 (257)	297 (294)	265 (263)	177 (176)	154 (151)	162 (159)	90.9 (87.9)	65.1 (62.2)	63.0 (59.8)	42.7 (39.3)	35.9 (32.8)
Max	403 (403)	371 (367)	335 (332)	313 (310)	321 (318)	296 (293)	204 (203)	191 (188)	183 (180)	121 (118)	70.4 (67.3)	71.4 (68.4)	55.3 (52.0)	41.9 (38.7)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	92 (91)	84 (83)	75 (74)	79 (78)	71 (71)	49 (49)	43 (42)	44 (43)	28 (27)	17 (17)	17 (16)	13 (12)	10 (9)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3.
The actual values are given in brackets (expressed as g/ha)

The concentration of fluopicolide in the 0.01M calcium chloride extracts are shown in Table 7.1.2.2.1-65 to Table 7.1.2.2.1- 67.

Table 7.1.2.2.1- 65: Residues of fluopicolide in the aqueous $CaCl_2$ extracts from soil from the St. Etienne du Grès trial after an application of 400 g a.s./ha (expressed as g/ha)

St. Etienne du Grès (France)	DAT (Days)				
	0	**58**	**167**	**352**	**714**
T1	149	62.2	[38.7]	[20.1]	[11.4]
T2	130	66.1	[37.6]	[25.7]	[11.3]
T3	166	61.1	[30.4]	[26.6]	[15.8]
Mean	**148.3**	**63.1**	**[35.6]**	**[24.1]**	**[12.8]**
Min	130	61.1	[30.4]	[20.1]	[11.3]
Max	166	66.1	[38.7]	[26.6]	[15.8]
n	3	3	3	3	3

DAT = Days after Treatment, LOQ = 15 µg/kg, LOD = 5 µg/kg, [x] = LOD >< LOQ

Table 7.1.2.2.1- 66: Residues of fluopicolide in the aqueous $CaCl_2$ extracts from soil from the Albaro trial after an application of 400 g a.s./ha (expressed as g/ha)

Albaro (Italy)	DAT (Days)				
	0	**57**	**175**	**364**	**722**
T1	145	78.0	50.4	[29.4]	<LOD
T2	133	61.2	39.8	[31.4]	[10.3]
T3	142	72.7	44.8	[28.3]	[11.4]
Mean	**140**	**70.6**	**45.0**	**[29.6]**	**[7.2]**
Min	133	61.2	39.8	[28.3]	<LOD
Max	145	78.0	50.4	[31.4]	[11.4]
n	3	3	3	3	3

DAT = Days after Treatment, LOQ = 15 µg/kg, LOD = 5 µg/kg, [x] = LOD >< LOQ

Table 7.1.2.2.1- 67: Residues of fluopicolide in the aqueous $CaCl_2$ extracts from soil from the Vilobi d'Onyar trial after an application of 400 g a.s./ha (expressed as g/ha)

Vilobi d'Onyar (Spain)	DAT (Days)				
	0	**56**	**167**	**370**	**714**
T1	147	87.5	[43.9]	[25.2]	<LOD
T2	158	80.7	[38.5]	[18.5]	<LOD
T3	152	80.9	46.5	[25.8]	<LOD
Mean	**152.3**	**83.0**	**[43]**	**[23.2]**	**<LOD**
Min	147	80.7	[38.5]	[18.5]	<LOD
Max	158	87.5	46.5	[25.8]	<LOD
n	3	3	3	3	3

DAT = Days after Treatment, LOQ = 15 µg/kg, LOD = 5 µg/kg, [x] = LOD >< LOQ

III. Conclusion

Under field conditions fluopicolide declined moderately and residues were translocated up to 30 cm (France), 20 cm (Italy), and 10 cm (Spain) depth, respectively. Un-normalised DT_{50} values for the degradation of fluopicolide calculated from the reported data following the recommendations of the FOCUS work group details are provided in Document KCA 7.1.2.2.1/14.

Assessment and conclusion by applicant:

The study is considered valid to assess test substance soil $DegT_{50matrix}$ values for field studies as defined by EFSA (2014). The endpoints may be too conservative to assess persistence as the design minimized soil surface processes as required by EFSA (2014) and such processes may contribute to dissipation.

Data Point:	KCA 7.1.2.2.1/14
Report Author:	[redacted]
Report Year:	2019
Report Title:	Terrestrial field dissipation study with fluopicolide & propamocarb-hydrochloride SC 687.5 in Germany, United Kingdom, France (North), France (South), Italy and Spain
Report No:	EnSa-18-1183
Document No:	M-651636-01-1
Guideline(s) followed in study:	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 including Data Requirements SANCO/11803/2010 Rev. 7 and Test Methods SANCO/11843/2010 Rev. 4 EFSA Guidance Document for Evaluating Laboratory and Field Dissipation Studies to obtain DegT50 Values of Active Substances of Plant Protection Products and Transformation Products of these Active Substances in Soil, EFSA Journal 2014; 12(5):3662, 2014
Deviations from current test guideline:	Yes. Report meets the requirement for assessing test substance soil DegT50matrix values as required by EFSA (2014) for field studies. The endpoints may be too conservative for comparison to field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

A summary report covering two terrestrial field dissipation studies was conducted to provide an overview of the trials and to calculate the kinetic rate of decline for each trial. Soil dissipation of fluopicolide under European field conditions was investigated after application of Fluopicolide + Propamocarb-hydrochloride SC 687.5 on bare soil plots at six sites, three in Northern Europe (see KCA 7.1.2.2.1/12, M-651681-01-1) at Burscheid (Germany), Great Chishill (United Kingdom), Lignieres de Touraine (France North) and three in Southern Europe (see KCA 7.1.2.2.1/13, M-6511790-01-1) at St. Etienne du Grès (France South), Albaro di Ronco all'Adige (Italy) and Vilobi d'Onyar (Spain).

The experiments were carried out in accordance with the EFSA Guidance Document for Evaluating Laboratory and Field Dissipation Studies to obtain $DegT_{50}$ Values of Active Substances of Plant Protection Products and Transformation Products of these Active Substances in Soil (EFSA Journal 2014).

Fluopicolide + Propamocarb-hydrochloride SC 687.5 was sprayed once onto 480 to 600 sqm for each treated plot at a rate of 6.4 L/ha, corresponding to nominal 400 g/ha fluopicolide. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface to meet EFSA requirements. The plots received at least 10 mm water between DAT-0 and DAT-3 by irrigation post application. The control plot was at least 5 m away from the treated plot.

Soil cores were taken 0 day before and at several dates up to 751 days post-application to a maximum depth of 110 cm. The soil cores were cut into 10-cm soil layers, bulked soil layers were homogenized and finally analyzed for fluopicolide.

Sub-samples of homogenized soil (5 g) were extracted in a microwave extractor with a mixture of acetonitrile/water (4/1, v/v). Potential matrix effects were eliminated using an internal standard solution of isotopically labeled reference item added to sample extracts. Following separation of fine particles from soil extracts by centrifugation, identification and quantitation of the analyte was performed by high performance liquid chromatography using MS/MS detection in the multiple reaction monitoring mode. The analytical method was validated using three different soils. The limit of quantitation (LOQ) was 5.0 µg/kg and the limit of detection (LOD) was 1.5 µg/kg.

Dissipation of fluopicolide from soil was moderate to slow with DT_{50} values ranging from 142 and 403 days for all test sites. An overview of the results is given below:

Location	Soil Type (USDA)	pH ($CaCl_2$)[A]	Best Fit Kinetic Model[B]	DT_{50} [d]	DT_{90} [d]
Burscheid (Germany)	Silt Loam (0-50 cm) Loam (50-100 cm)	5.3	FOMC	142	980
Great Chishill (United Kingdom)	Clay (0-100 cm)	7.2	FOMC	403	> 1000
Lignieres de Touraine (France North)	Sandy Loam (0-75 cm) Clay Loam (75-100 cm)	5.9	DFOP	247	> 1000
St.Etienne du Grès (France South)	Clay Loam (0-75 cm) Clay (75-100 cm)	7.3	DFOP	265	> 1000
Albaro di Ronco all'Adige (Italy)	Clay Loam (0-75 cm) Clay (75-100 cm)	7.3	DFOP	187	> 1000
Vilobi d'Onyar (Spain)	Loam (0-30 cm) Sandy Clay Loam (30-100 cm)	6.0	DFOP	152	747

[A] pH in 0-30 cm soil depth
[B] FOMC: first order multi compartment; DFOP: double first order in parallel

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide + propamocarb-hydrochloride formulated as a suspension concentrate (62.5g/L fluopicolide + 625 g/L of propamocarb-hydrochloride)

Certificate of Analysis:	01860-00
Lot No:	2015-000846-01

2. Trial Location & Soil

A terrestrial field dissipation with Fluopicolide & Propamocarb-hydrochloride (SC 687.5) is a suspension concentrate formulation, containing 687.50 g/L fluopicolide & propamocarb-hydrochloride was conducted at three locations in Northern Europe and at three sites in Southern Europe. The six locations were Burscheid (Germany), Great Chishill (United Kingdom) and Lignieres de Touraine (Northern France), St. Etienne du Grès (Southern France), Atbaro (Italy) and Vilobi d'Onyar (Spain). The sites were fully characterised, and the results summarised in Table 7.1.2.2.1- 68 and Table 7.1.2.2.1-69. The plot sizes ranged from 400 sqm to 654 sqm. The control plot was prepared at least 5 m away from the treated plots.

Table 7.1.2.2.1- 68: Location, site description and climatic data of test sites in Northern Europe

Characteristic	Units	Sampling depth [cm]			
		0-30	**30-50**	**50-75**	**75-100**
Soil Designation	-	**Burscheid (Germany)**			
Soil ID	-	VG08			
Geographic Location City Country	-	Burscheid [redacted] Germany			
pH	$CaCl_2$	5.3	5.6	5.6	5.6
Organic carbon	[% Carbon]	1.2	0.4	0.1	0.1
CEC	[meq/100 g]	12.8	11.8	12.4	11.9
Chalk	[% $CaCO_3$]	12.8	11.8	12.4	11.9
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 21 61 18	 23 57 20	 19 43 38	 13 37 50
Textural class	USDA	silt loam	silt loam	loam	loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 49.3 35.2	 50.0 31.9	 45.9 30.3	 39.1 25.9
Soil Designation	-	**Great Chishill (United Kingdom)**			
Soil ID	-	ENG8			
Geographic Location City Country	-	[redacted] Great Chishill, Cambridgeshire United Kingdom			
pH	$CaCl_2$	7.2	7.5	7.7	7.6
Organic carbon	[% Carbon]	2.1	1.1	0.5	0.5
CEC	[meq/100 g]	28.9	26.1	19.9	17.4
Chalk	[% $CaCO_3$]	1.3	5.8	37.9	43.1
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 41 23 36	 45 21 34	 53 23 24	 51 23 26
Textural class	USDA	clay	clay	clay	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 60.8 29.2	 57.2 27.9	 52.6 25.3	 49.5 25.9
Soil Designation	-	**Lignieres de Touraine (France)**			
Soil ID	-	FR09			
Geographic Location City Country	-	[redacted] [redacted] 37130 Lignieres de Touraine, Central Region France			
pH	$CaCl_2$	5.9	6.5	6.8	6.8
Organic carbon	[% Carbon]	0.8	0.4	0.3	0.5
CEC	[meq/100 g]	12.2	13.2	14.7	21.8
Chalk	[% $CaCO_3$]	0.2	0.3	0.3	0.0
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 15 15 70	 15 19 66	 19 25 56	 37 33 30
Textural class	USDA	sandy loam	sandy loam	sandy loam	clay loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 44.4 17.5	 47.7 20.6	 52.3 26.1	 61.5 33.1

Table 7.1.2.2.1- 69: Location, site description and climatic data of test sites in Southern Europe

Characteristic	Units	Sampling depth [cm]			
		0-30	30-50	50-75	75-100
Soil Designation	-	**St. Etienne du Grès (France)**			
Soil ID	-	FR08			
Geographic Location City Country	-	[redacted] 13103 St. Etienne du Grès, Provence-Alpes-Côte d'Azur France			
pH	$CaCl_2$	7.7	7.7	7.8	7.8
Organic carbon	[% Carbon]	0.8	0.6	0.6	0.4
CEC	[meq/100 g]	11.7	11.7	13.2	14.0
Chalk	[% $CaCO_3$]	42.1	42.1	42.5	43.7
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 29 45 26	 31 47 22	 39 41 20	 43 39 18
Textural class	USDA	clay loam	clay loam	clay loam	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 46.2 29.5	 43.4 26.8	 48.3 29.3	 51.4 25.0
Soil Designation	-	**Albaro (Italy)**			
Soil ID	-	IT23			
Geographic Location City Country		[redacted] 37055 Albaro di Ronco, all'Adige, Veneto Italy			
pH	$CaCl_2$	7.3	7.4	7.5	7.4
Organic carbon	[% Carbon]	1.8	1.5	0.7	0.6
CEC	[meq/100 g]	19.7	20.1	17.9	17.1
Chalk	[% $CaCO_3$]	10.6	12.0	14.1	11.8
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 35 43 22	 33 45 22	 35 45 20	 41 39 20
Textural class	USDA	clay loam	clay loam	clay loam	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 61.5 35.5	 62.7 35.9	 67.8 41.1	 65.5 37.6
Soil Designation	-	**Vilobi d'Onyar (Spain)**			
Soil ID	-	SPA1			
Geographic Location City Country		[redacted] 17185 Vilobi d'Onyar, Catalonia Spain			
pH	$CaCl_2$	6.0	6.1	6.6	7.0
Organic carbon	[% Carbon]	0.8	0.3	0.1	0.1
CEC	[meq/100 g]	9.6	11.9	13.5	14.2
Chalk	[% $CaCO_3$]	0.3	0.2	0.1	0.2
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 17 33 50	 27 23 50	 29 15 56	 27 15 58
Textural class	USDA	loam	sandy clay loam	sandy clay loam	sandy clay loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 41.4 21.5	 44.5 20.7	 47.9 23.9	 47.3 22.4

B. Study Design

1. Experimental Conditions

Fluopicolide & Propamocarb-hydrochloride SC 687.5 is a suspension concentrate formulation, containing 62.5 g/L fluopicolide and 625 g/L propamocarb-hydrochloride. The product was sprayed onto bare earth once at each site at an application rate of 6.40 L/ha and 600 L/ha water, corresponding to 400 g/ha fluopicolide during May and June 2015. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface. Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of a nonselective herbicide to control weeds.

Air temperature, precipitation including irrigation and sunshine data were recorded on site during the field tests. Unwanted plant growth was controlled.

Soil dissipation of fluopicolide was studied for up to 751 days.

2. Sampling

The treated plot of each trial was divided into three sub-plots. From each sub-plot 10 soil cores were taken and combined together at each sampling interval (30 cores in total).

Samples were taken on the following occasions: 0 (post-application; 0-10 cm depth), 6-8, 13-15, 21-22, 27-29 (each 0-40 cm depth), 56-68, 118-134 (each 0-60 cm depth) and 166-194, 252-301, 348-398, 435-489, 519-554, 605-667, 700-751 (each 0-85 cm depth) after treatment. From the control plot samples were taken on the following occasions: 0 days before application, 348-398 and 700-751 days after application. Soil cores were deep frozen to -18°C.

3. Analytical Procedures

The analytical method 01445 was used to determine levels of fluopicolide. Soil samples of 5 g were extracted in a microwave extractor with a mixture of acetonitrile/water (4/1, v/v). The extracts were centrifuged to remove fine particles of the soil. Possible matrix effects of fluopicolide were eliminated by using an internal standard solution of isotopic labelled reference items. Quantification was carried out by LC-MS/MS. The limit of quantitation (LOQ) for fluopicolide was 5.0 µg/kg in soil. The limit of determination (LOD) for fluopicolide was 1.5 µg/kg.

During analysis of the dissipation samples, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide. The mean recovery for fluopicolide was 101% (RSD 6.4%).

4. Evaluation of the Data and Kinetic Calculations

For evaluation of degradation kinetics of the test item according to the FOCUS guidance document on degradation kinetics, the total residue of the test item in the soil profile covering all soil horizons was calculated according to the following procedure:

- values between LOD and LOQ were set to the measured values.
- values < LOD were set to 0.5 LOD for samples after, before or deeper as a value > LOD or for samples between (> LOD and <LOQ). The curve was cut off after the first non-detect (< LOD), if no later value > LOQ followed.
- at day 0, values < LOD in deep horizons were set to 0.

The results in [µg/kg] were converted to [g/ha] considering the actual soil density of the corresponding soil layer.

DT_{50} and DT_{90} values for the degradation of fluopicolide have been calculated from the reported data using the software KinGUI (version 2.1). To derive trigger endpoints, a comparison was performed for each site between the SFO, FOMC and DFOP fits. For the derivation of trigger endpoints, FOCUS recommends to use the best-fit model. In an initial step, data for the applied compound fitted using the SFO and FOMC models were compared. If the SFO model provided a better fit overall (both visually and statistically), this fit was selected. If the FOMC model provided a better fit, the FOMC and DFOP fits were compared, and the model that provided the best fit overall was selected. It should be noted that extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using the FOMC model (EFSA, 2009), and this has been considered where relevant in the selection of the most appropriate model.

II. Results and Discussion

The decline of fluopicolide residues with time for the entire soil profile is presented in Table 7.1.2.2.1-70 to Table 7.1.2.2.1- 75.

Table 7.1.2.2.1- 70: Residues of fluopicolide in soil from the Burscheid trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

Burscheid (Germany)	DAT													
	0	**7**	**15**	**21**	**28**	**63**	**121**	**172**	**252**	**385**	**437**	**519**	**605**	**701**
T1	499	435	393	418	390	363	140	211	167	128	97.7	76.5	51.3	60.1
T2	488	418	530	463	467	452	190	241	211	167	148	116	78.3	76.4
T3	366	534	668	502	369	387	252	195	131	140	114	88.6	99.7	70
Mean	**451**	**462**	**530**	**461**	**409**	**401**	**194**	**216**	**170**	**145**	**120**	**93.7**	**76.4**	**68.8**

DAT = Days after treatment

Table 7.1.2.2.1- 71: Residues of fluopicolide in soil from the Great Chishill trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

Great Chishill (UK)	DAT													
	0	**7**	**15**	**22**	**29**	**68**	**134**	**194**	**301**	**398**	**468**	**554**	**667**	**751**
T1	310	319	346	232	269	358	171	163	187	160	177	134	112	94.6
T2	331	333	266	259	293	342	226	224	172	186	157	142	123	106
T3	296	375	471	324	357	403	211	214	189	164	156	191	180	132
Mean	**312**	**342**	**361**	**272**	**304**	**368**	**203**	**200**	**183**	**170**	**163**	**156**	**138**	**111**

DAT = Days after treatment

Table 7.1.2.2.1- 72: Residues of fluopicolide in soil from the Lignieres de Touraine trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

Lignieres de Touraine (France)	DAT													
	0	**7**	**14**	**21**	**28**	**56**	**120**	**166**	**277**	**348**	**435**	**530**	**645**	**700**
T1	399	323	309	311	326	291	198	195	181	154	96.9	82.9	91.4	71.2
T2	327	318	293	306	307	283	205	227	178	156	97.8	89.0	79.8	54.6
T3	342	305	310	280	293	259	174	200	183	150	96.5	69.7	70.1	49.7
Mean	**356**	**315**	**304**	**299**	**309**	**278**	**192**	**207**	**181**	**153**	**97.1**	**80.5**	**80.4**	**58.5**

DAT = Days after treatment

Table 7.1.2.2.1- 73: Residues of fluopicolide in soil from the St. Etienne du Grès trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

St. Etienne du Grès (France)	DAT													
	0	**7**	**14**	**21**	**28**	**58**	**118**	**167**	**280**	**352**	**451**	**519**	**646**	**714**
T1	380	369	333	347	312	272	243	225	180	170	145	134	117	108
T2	332	375	375	354	319	325	245	229	204	188	157	128	116	107
T3	432	401	366	362	325	291	208	213	188	201	157	138	156	140
Mean	**381**	**382**	**358**	**354**	**319**	**296**	**232**	**222**	**191**	**186**	**153**	**137**	**130**	**118**

DAT = Days after treatment

Table 7.1.2.2.1- 74: Residues of fluopicolide in soil from the Albaro trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

Albaro (Italy)	DAT													
	0	**6**	**13**	**21**	**27**	**57**	**121**	**175**	**283**	**364**	**450**	**541**	**618**	**722**
T1	427	322	392	248	230	274	201	187	203	162	106	69.1	61.7	59.9
T2	385	407	360	329	323	236	269	180	254	185	110	103	75.5	73.7
T3	425	362	473	304	327	293	230	197	194	174	127	98.9	89.0	80.0
Mean	**412**	**363**	**408**	**293**	**293**	**267**	**233**	**188**	**216**	**173**	**115**	**90.1**	**75.4**	**71.2**

DAT = Days after treatment

Table 7.1.2.2.1- 75: Residues of fluopicolide in soil from the Vilobi d'Onyar trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

Vilobi d'Onyar (Spain)	DAT													
	0	**8**	**15**	**22**	**28**	**56**	**128**	**167**	**279**	**370**	**489**	**546**	**628**	**714**
T1	388	363	323	257	303	293	203	160	180	118	62.2	63.1	39.3	38.7
T2	381	367	332	310	294	272	176	151	159	87.9	67.3	68.4	52.0	32.8
T3	403	339	319	302	318	263	195	188	169	116	65.8	59.8	51.3	35.7
Mean	**391**	**356**	**325**	**290**	**305**	**276**	**191**	**166**	**169**	**107**	**65.1**	**63.8**	**47.5**	**35.7**

DAT = Days after treatment

In Table 7.1.2.2.1- 76 to Table 7.1.2.2.1- 81 the fluopicolide datasets for the entire soil profile pre-processed according to the procedures described in FOCUS kinetics guidance (as described above) are presented.

Table 7.1.2.2.1- 76: Residues of fluopicolide in soil from the Burscheid trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

Burscheid (Germany)	DAT													
	0	7	15	21	28	63	121	172	252	395	437	519	605	701
T1	499	438	396	421	393	366	143	214	170	130	100	82.4	57.3	63.0
T2	488	421	533	466	470	455	193	244	214	170	151	119	81.2	79.4
T3	366	537	671	505	372	390	254	198	134	143	117	91.6	103.0	72.8
Mean	**451**	**465**	**533**	**464**	**412**	**404**	**197**	**219**	**173**	**148**	**123**	**97.7**	**80.5**	**71.7**

DAT = Days after treatment

Table 7.1.2.2.1- 77: Residues of fluopicolide in soil from the Great Chishill trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

Great Chishill (UK)	DAT													
	0	7	15	22	29	68	134	194	301	398	468	554	667	751
T1	310	322	349	235	272	360	174	169	192	166	180	137	115	97.4
T2	331	336	269	262	296	345	229	227	175	191	160	145	126	111
T3	296	378	414	327	360	406	214	217	192	167	159	193	182	135
Mean	**312**	**345**	**364**	**275**	**309**	**370**	**206**	**204**	**186**	**175**	**166**	**158**	**141**	**114**

DAT = Days after treatment

Table 7.1.2.2.1- 78: Residues of fluopicolide in soil from the Lignieres de Touraine trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

Lignieres de Touraine (France)	DAT													
	0	7	14	21	28	56	120	166	277	348	435	530	645	700
T1	399	326	313	314	329	294	201	198	184	157	99.9	86.0	94.3	74.0
T2	327	321	296	309	310	286	208	230	181	159	101	92.0	82.9	57.6
T3	342	308	313	283	296	262	177	203	186	153	99.4	72.9	73.4	52.5
Mean	**356**	**318**	**307**	**302**	**312**	**281**	**195**	**210**	**184**	**156**	**100**	**83.6**	**83.5**	**61.4**

DAT = Days after treatment

Table 7.1.2.2.1- 79: Residues of fluopicolide in soil from the St. Etienne du Grès trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

St. Etienne du Grès (France)	DAT													
	0	**7**	**14**	**21**	**28**	**58**	**118**	**167**	**280**	**352**	**451**	**519**	**646**	**714**
T1	380	372	336	349	315	274	246	228	183	172	148	137	120	110
T2	332	378	376	356	322	328	248	232	206	190	159	140	119	110
T3	432	403	369	365	325	294	213	215	191	204	159	141	158	142
Mean	**381**	**384**	**360**	**357**	**321**	**299**	**236**	**225**	**193**	**189**	**155**	**139**	**132**	**121**

DAT = Days after treatment

Table 7.1.2.2.1- 80: Residues of fluopicolide in soil from the Albaro trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

Albaro (Italy)	DAT													
	0	**6**	**13**	**21**	**27**	**57**	**121**	**175**	**283**	**364**	**450**	**541**	**618**	**722**
T1	427	325	395	250	233	277	204	190	205	165	109	71.7	64.2	62.6
T2	385	410	363	332	326	239	272	182	256	188	113	105	78.0	76.4
T3	425	365	476	307	329	296	233	200	196	177	130	102	91.5	82.6
Mean	**412**	**367**	**411**	**296**	**296**	**271**	**236**	**191**	**219**	**177**	**117**	**930**	**78.0**	**74.0**

DAT = Days after treatment

Table 7.1.2.2.1- 81: Residues of fluopicolide in soil from the Vilobi d'Onyar trial after an application of 400 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

Vilobi d'Onyar (Spain)	DAT													
	0	**8**	**15**	**22**	**28**	**56**	**128**	**167**	**279**	**370**	**489**	**546**	**628**	**714**
T1	388	366	326	260	306	296	204	163	183	121	65.1	66.2	42.7	41.9
T2	381	371	335	313	297	275	177	154	162	90.9	70.4	71.4	55.3	35.9
T3	403	342	323	305	321	265	197	191	172	119	68.8	63.0	54.5	38.6
Mean	**391**	**360**	**328**	**293**	**308**	**279**	**193**	**169**	**172**	**110**	**68.1**	**66.9**	**50.8**	**38.8**

DAT = Days after treatment

The residual amounts of the test item presented above (Table 7.1.2.2.1- 76 to Table 7.1.2.2.1- 81) were used as input data for determination of degradation kinetics using the software KinGUI 2. The measured initial concentration at day 0 was included in the parameter optimization procedure. Based on criterion for chi2 error to be minimal and visual assessment the best fit kinetic model was chosen for the evaluation of the dissipation time. The calculation considered the quantifiable residues for the whole soil profiles expressed in [g/ha]. The results are summarized in Table 7.1.2.2.1- 82 with best fits highlighted in bold. The dissipation of fluopicolide could be described using a first order multi compartment (FOMC) model for test sites Burscheid (Germany) and Great Chishill (United Kingdom) and double first order in parallel (DFOP) for the other test sites. The best fit half-lives for fluopicolide were between 142 and 403 days for all test sites. Best fit kinetics are highlighted in bold.

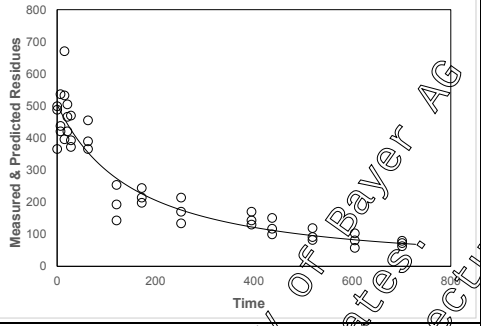
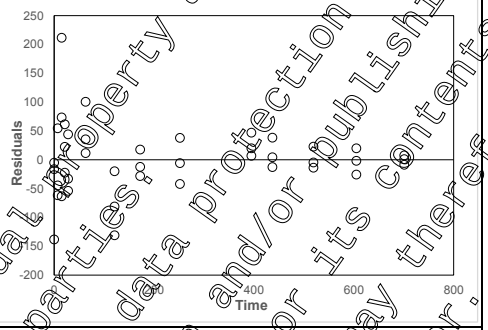
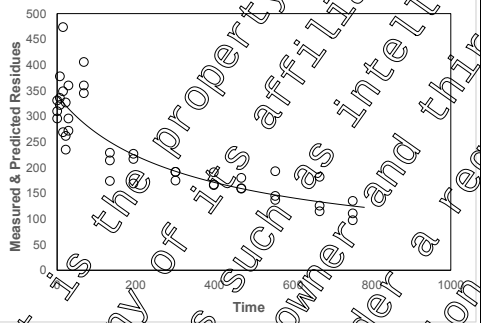
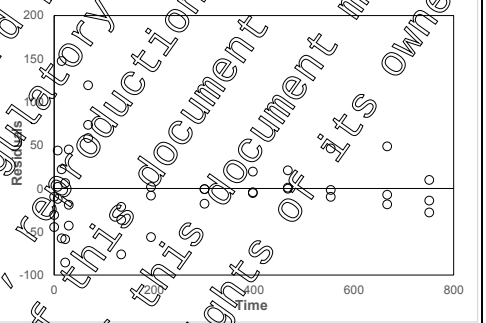
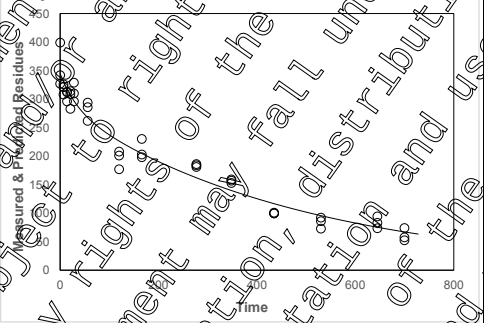
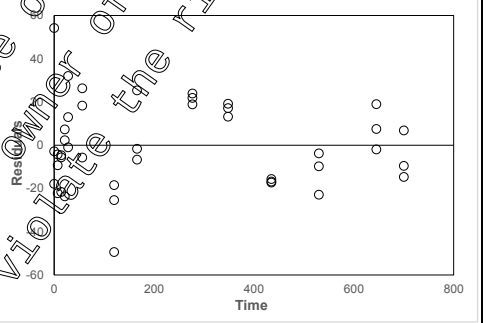
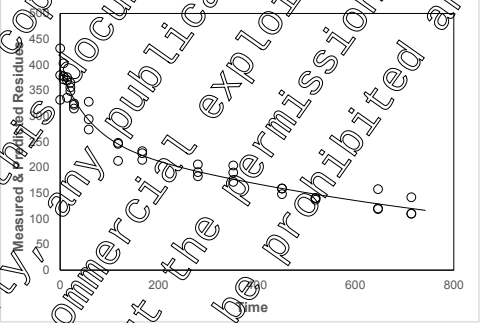
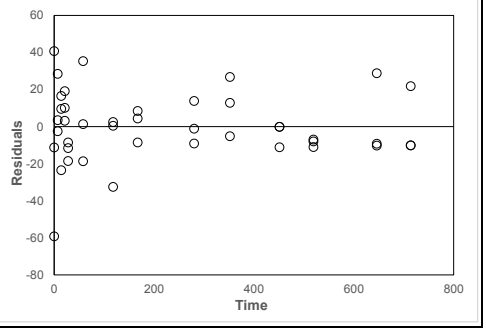
Table 7.1.2.2.1- 82: Degradation rate of fluopicolide under field conditions (DT_{50} values for trigger endpoints)

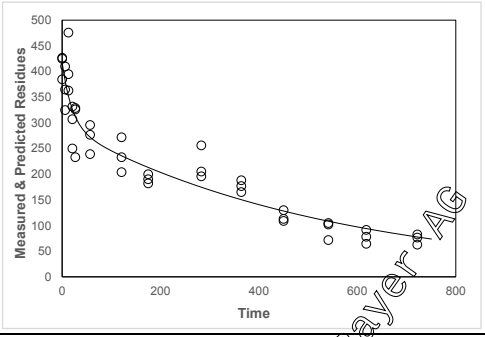
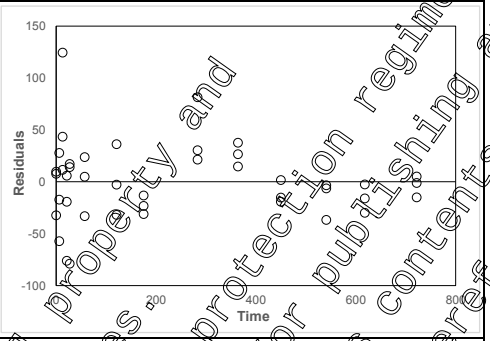
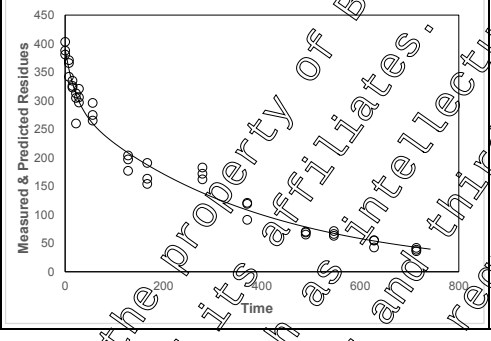
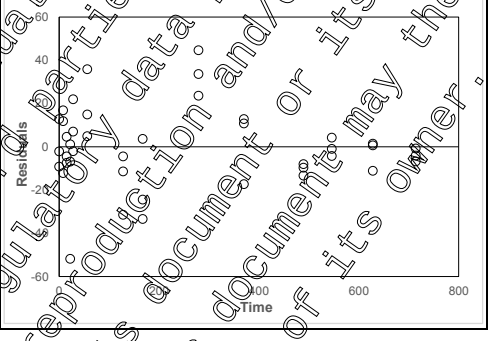
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Burscheid, 15-2701-01	SFO	477.1	k 0.003649	13.1	1.85E-12	0.002917	0.004	189.9	631
	FOMC	**504.1**	**α 1.309 β 204**	**11.2**	**n.r. n.r.**	**0.1224 -86.86**	**2.496 495**	**142.4**	**980.1**
	DFOP	504.2	k1 0.008669 k2 0.001176 g 0.6472	11.3	0.0763 0.2762 n.r.	-0.002971 -0.002669 -0.1398	0.02 0.005 1.434	135.9	1073
Great Chishill, 15-2701-02	SFO	327.1	k 0.001515	11.9	4.86E-11	0.001173	0.002	457.6	1520
	FOMC	**340.7**	**α 0.7268 β 252.9**	**11.4**	**n.r. n.r.**	**-0.2382 -301.6**	**1.692 807.4**	**403.3**	**5755**
	DFOP	341.1	k1 0.005489 k2 0.000533 g 0.4483	10.7	0.258 0.373 n.r.	-0.01088 -0.002667 -0.8855	0.022 0.004 1.782	391.6	3204
Lignieres de Touraine, 15-2701-03	SFO	324.5	k 0.002437	6.93	<2e-16	0.002196	0.003	284.4	944.8
	FOMC	332.8	α 1.895 β 564.9	6.63	n.r. n.r.	0.2044 -97.83	3.586 1228	249.4	1339
	DFOP	**344.8**	**k1 0.02922 k2 0.002081 g 0.1634**	**6.12**	**0.07056 4.48E-13 n.r.**	**-0.008886 0.001692 0.05521**	**0.067 0.002 0.272**	**247.4**	**1021**
St. Etienne du Grès, 15-2702-01	SFO	355.2	k 0.00187	7.98	<2e-16	0.001635	0.002	370.6	1231
	FOMC	389.5	α 0.4704 β 74.06	3.05	n.r. n.r.	0.3392 27.06	0.602 121.1	249.1	9817
	DFOP	**391.2**	**k1 0.001066 k2 0.01672 g 0.6578**	**2.73**	**5.00E-07 0.00116 n.r.**	**0.0007074 0.006681 0.5542**	**0.001 0.027 0.761**	**264.9**	**1766**
Albaro di Ronco all'Adige, 15-2702-02	SFO	357.7	k 0.002436	12.6	7.56E-14	0.001998	0.003	284.6	945.3
	FOMC	395.7	α 0.5744 β 70.92	11.4	n.r. n.r.	0.2616 -14.66	0.887 156.5	166.2	3836
	DFOP	**417.3**	**k1 0.05008 k2 0.001847 g 0.2943**	**9.48**	**0.0255 2.77E-08 n.r.**	**0.001387 0.00131 0.1691**	**0.099 0.002 0.419**	**186.6**	**1058**
Vilobi d'Onyar, 15-2702-03	SFO	347.1	k 0.003328	9.04	<2e-16	0.002965	0.004	208.3	691.9
	FOMC	366.9	α 1.34 β 226.9	7.66	n.r. n.r.	0.6895 55.24	1.991 398.5	153.6	1037
	DFOP	**390.2**	**k1 0.04638 k2 0.002705 g 0.2456**	**6.01**	**0.00246 <2e-16 n.r.**	**0.01594 0.002317 0.165**	**0.077 0.003 0.326**	**152.2**	**747.1**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.2.1- 83: Graphical representations of best fit model

Soil Model Reference	Modelled vs observed	Residuals
Burscheid FOMC 15-2701-01		
Great Chishill FOMC 15-2701-02		
Lignieres de Touraine DFOP 15-2701-03		
St. Etienne du Grès DFOP 15-2702-01		

Soil Model Reference	Modelled vs observed	Residuals
Albaro di Ronco all'Adige DFOP 15-2702-02		
Vilobi d'Onyar DFOP 15-2702-03		

III. Conclusion

Fluopicolide was moderately to slowly degraded in soil at six trial sites in Northern and Southern Europe. Residue levels were in the range of 35.7 to 118 g/ha at the end of the test period. The dissipation of fluopicolide was best described by FOMC or DFOP kinetic models with best fit DT_{50} values ranging from 142 and 403 days.

Assessment and conclusion by applicant:

The study is considered valid to assess test substance soil $DegT_{50matrix}$ values for field studies as defined by EFSA (2014). The endpoints may be too conservative to assess persistence as the design minimized soil surface processes as required by EFSA (2014) and such processes may contribute to dissipation.

Data Point:	KCA 7.1.2.2.1/15
Report Author:	[redacted]
Report Year:	2017
Report Title:	Fluopicolide (FLC): Ecoregion crosswalk - Representativeness of environmental conditions at five North American terrestrial field dissipation trial sites for Europe
Report No:	EnSa-17-0326
Document No:	M-592872-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	none
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The environmental conditions at five North American trial sites were compared to European environmental conditions. It was assessed whether the results of North American fluopicolide terrestrial field dissipation trials are representative for Europe and might be used to derive degradation half-live values ($DegT_{50}$) for European risk assessments.

The concept of ecoregions was the basis of the comparison. Ecoregions are delineated with respect to similar location, climate, soil, vegetation, hydrology, terrain, wildlife, and land use (CEC 2011, PRMA 2015, EEA 2000) and form a classification system that describes environmental conditions which influence degradation of pesticides under field conditions. The ENASGIPS software is recommended by the OECD Guidance Document for Conducting Pesticide Terrestrial Field Dissipation Studies for such an assessment (OECD 2016). It uses the concept of ecoregions under the assumption that degradation should be similar in similar ecoregions.

The similarity model within the ENASGIPS tool merely considers annual mean climate parameters and individual soil parameters. However, climate is often characterised by seasonal variations and pesticide degradation cannot solely be attributed to individual soil properties like topsoil texture, organic carbon content, and pH. Therefore, a refinement of the ecoregion comparison was proposed which takes into account more holistic indicators like climate seasonality and soil types.

Trial sites St. George, Ontario and Madera, California were considered representative for European climate conditions and soil properties. No similar European ecoregions could be identified for trial site Oviedo, Florida, which exhibits a strong subtropical climate. Trial sites at Arkansaw, Wisconsin and Ephrata, Washington were considered not representative for European conditions because of their pronounced continental climate which is atypical for European climate conditions, as well as the limited soil coverage in Europe of their respective soil types.

I. Materials and Methods

When considering terrestrial field dissipation studies from regions outside Europe, EFSA (2014) requires applicants to determine if these studies were conducted on soils from temperate regions; if their pH, organic matter and clay content are representative for soils in the EU; and if temperature and precipitation are comparable to those regions in the EU where the crops relevant to the application are grown.

For the active substance fluopicolide, terrestrial field dissipation studies were conducted at different locations in North America. Five trial sites have been addressed in the following comparison, four of those in the United States and one in Canada. The Canadian site is located near St. George, Ontario (see KCA 7.1.2.2.1/16, M-248833-01-1); the American sites (see KCA 7.1.2.2.1/17, M-251292-01-1) are located in Oviedo (Semnipole, Florida), Arkansaw (Pepin, Wisconsin), Madera (Madera, California) and Ephrata (Grant, Washington). The locations of the five sites are shown in Figure 7.1.2.2.1- 5.

Figure 7.1.2.2.1- 5: Location of the examined fluopicolide field dissipation trial sites

The GIS-based software 'Europe-North America Soil Geographic Information for Pesticide Studies' (ENASGIPS) 3.0 was developed by the Pest Management Regulatory Agency – Health Canada and the US Environmental Protection Agency in collaboration with Agriculture and Agri-Food Canada and the EC Joint Research Centre (PMRA 2015). The development was part of the OECD project 'Harmonized International Guidance for Pesticide Terrestrial Field Dissipation Studies and Crosswalk of North American and European Eco-regions' (OECD 2012).

The tool includes a database with ecoregion boundaries as defined by WWF and a distribution for five environmental parameters and crop information for each ecoregion. The considered environmental parameters are

- mean annual temperature
- mean annual precipitation
- mean soil pH
- mean soil organic carbon
- soil texture

Their distribution was derived by an overlay of the ecoregions with MARS FOODSEC Meteodata (JRC 2011) and the Harmonized World Soil Database (FAO 2012b). It is assumed that these parameters are normally distributed and the distributions are described by mean value and standard deviation. In case of soil texture, the textural classes were mapped onto an integer scale from heavy clay (1) to sand (12).

Ecoregions are compared by calculating a similarity score between a root ecoregion and other ecoregions. The score for a parameter in an ecoregion is 100% if the parameter of ecoregion does not differ more than one standard deviation from the mean of the root ecoregion (PMRA 2015). The ENASGIPS tool can either be used with the so-called 'Holistic Ecoregion Similarity' approach which takes into account all five parameters with equal weights or the 'Weights of Evidence Ecoregion Similarity' which uses only selected parameters for its comparison.

II. Results and Discussion

St. George, Ontario

The climate and soil conditions at the trial site are adequately represented by the root ecoregions' properties. Several European ecoregions were identified as similar to the North American ecoregions by ENASGIPS. Although the trial site has a distinct continental climate, similar environmental conditions are present in parts of Eastern Europe which are relevant to European agriculture. Therefore, the trial site St. George is considered representative for European conditions.

Oviedo, Florida

No European ecoregions similar to the root ecoregion were identified by ENASGIPS and the site's subtropical climate conditions are not present within Europe. Therefore, the trial site Oviedo is considered not representative for European conditions.

Arkansaw, Wisconsin

The climate and soil conditions at the trial site are adequately represented by the root ecoregion's properties. Although some European ecoregions were identified as comparable to the root ecoregion by ENASGIPS, no area with a similar pronounced continental climate could be determined in Europe. In addition, the trial site's soil type has a limited coverage of only 1% in Europe. Therefore, the trial site Arkansaw is considered not representative for European conditions.

Madera, California

The climate conditions at the trial site are adequately represented by the root ecoregions' properties. Several European ecoregions were identified as similar to the North American ecoregions by ENASGIPS. Although the trial site has a subtropical climate with dry and wet seasons, similar environmental conditions are present in parts of Southern Europe which are relevant to European agriculture. Therefore, the trial site Madera is considered representative for European conditions.

Ephrata, Washington

The climate and soil parameters at the trial site are significantly different from conditions in the root ecoregion and relevant ecoregions in Europe. No area with a similar continental dry climate could be identified in Europe. The site's soil type has only a limited coverage of 1% in Europe which represents only 2% of European cropping areas. In addition, the soil at the trial site has a very low organic carbon content of about 0.4%. Therefore, the trial site Ephrata is considered not representative for European conditions.

III. Conclusion

Trial sites St. George, Ontario and Madera, California were considered representative for European climate conditions and soil properties. No similar European ecoregions could be identified for trial site Oviedo, Florida, which exhibits a strong subtropical climate. Trial sites at Arkansaw, Wisconsin and Ephrata, Washington were considered not representative for European conditions because of their pronounced continental climate which is atypical for European climate conditions as well as the limited soil coverage in Europe of their respective soil types.

Assessment and conclusion by applicant:

The study is considered valid to identify non-European terrestrial field dissipation trial sites considered representative for European climate conditions and soil properties

Data Point:	KCA 7.1.2.2.1/16
Report Author:	[redacted]
Report Year:	2004
Report Title:	Field dissipation of AE C638206 in Eastern Canadian soil
Report No:	B004929
Document No:	M-248833-01-1
Guideline(s) followed in study:	PMRA: DACO 8.3.2.1, T-1-255
Deviations from current test guideline:	Yes. The trial site in Ontario was considered representative for European climate conditions and soil properties. The study meets the requirement for field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013. The study does not meet the requirements for assessing parent and metabolite soil DegT50matrix values as required by EFSA (2014) for legacy field studies.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities.
Acceptability/Reliability:	Supportive only

Executive Summary

Soil dissipation of fluopicolide was studied after an application to bare soil plots under field conditions for 453 days at St George, Ontario (Canada).

The three treated plots received three broadcast applications of fluopicolide using small-plot equipment at a nominal application rate of 133 g a.i./ha each applied in 200 L/ha of water. The applications were made at 5 ± 1 day intervals and reflect the maximum annual application rate of 400 a.i./ha.

The predominant analyte found in this study was the parent fluopicolide which was largely confined to the top 0-15 cm soil segment. Fluopicolide was detected in the 0-7.5 cm soil segment after the first application and remained quantifiable throughout the study period to the final sampling date (453 days). Fluopicolide was detected at low levels in only four samples in the 7.5-15 cm segments and in four samples in the 15-30 cm segments. In deeper depth no residues of fluopicolide were found above the LOQ.

The metabolite M-01 was detected in 0-7.5 cm soil segment prior to the second application and remained to the final sampling date (453 days). M-02 was detected in three samples in the 7.5-15 cm segments on day 0 but was not detected in any lower segments or at any later sampling times.

M-02 (AE C657188) and M-03 (AE 0608000) were very rapidly degraded in soil. M-02 was detected in the 0-7.5 cm soil segment prior to the second application until day 55. M-02 was detected in three samples in the 7.5-15 cm segments on day 0 but was not detected in any lower segments or at any later sampling times. No residues of M-03 were found above the LOQ throughout the study.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit SFO not normalised DT_{50} value of 209.8 days and DT_{90} values of 697 days for fluopicolide.

I. Materials and Methods

A.Materials

1. Test Item

Fluopicolide formulated as a spendable concentrate (480 g/L fluopicolide)

Certificate of Analysis: AGF2002-0038-01

Lot No: OP220233

2. Trial Location & Soil

A single site was selected near the town of St George, Ontario (see Table 7.1.2.2.1- 84). The test site had no significant slope and the top soil was a clay loam soil with lower horizons of clay. The total 110 x 34 m trial area consisted of three 30 m x 6 m treated plots each further divided into twenty 1.5 m x 6 m subplots, an untreated plot (60 m²) which served as a control was separated from the treated plots by a 77.2 meter buffer.

Table 7.1.2.2.1- 84: Location, site description and climatic data of test site

Characteristic	Units	St George, Ontario, Canada						
		Horizon 1	Horizon 2	Horizon 3	Horizon 4	Horizon 5	Horizon 6	Horizon 7
Sampling depth	cm	0-7.5	7.5-15	15-30	30-45	45-60	60-75	75-90
pH (1:2)		7.3	7.1	7.5	8.2	8.3	8.5	8.5
Cation exchange capacity	meq/100 g	15.8	16.0	13.8	10.7	9.5	7.5	8.0
Organic matter	%	3.9	3.9	2.0	0.7	0.5	0.4	0.4
Dry (bulk) density	g/cm³	1.1	1.1	1.2	1.1	1.1	1.1	1.1
Field capacity 1/3 bar (pF2.53)	Vol.-%	26.9	31.1	27.3	26.3	27.8	28.7	29.7
Field capacity 15 bar (pF4.2)	Vol.-%	15.6	16.9	17.0	16.5	18.2	19.6	19.9
Particle size distribution								
Clay	%	28	37	32	45	54	50	58
Total silt	%	45	43	41	39	34	28	34
Total sand	%	27	30	27	16	12	21	8
Textural class		clay loam	clay loam	clay loam	clay	clay	clay	clay

B. Study Design

1. Experimental Conditions

The three treated plots received three broadcast applications of fluopicolide using small-plot equipment at a nominal rate of 133 g a.i./ha each applied in 200 L/ha of water. The first application was on 3 July 2002 with two subsequent applications on the 8 July 2002 and 12 July 2002. Nominal application rates were confirmed by measuring the unused formulation remaining in the spray tank to calibrate the amount applied.

All applications were made to bare soil. Throughout the study the plots were maintained as bare soil by the periodic application of the herbicide glyphosate to control weeds.

Temperature and rainfall data were collected from an onsite weather station located approximately 75 meters from the plot area.

2. Sampling

Soil cores (6.25 cm diameter) were taken immediately after treatment 4 hours, 6, 13, 31, 55, 95, 306 and 453 days after the third application. A total of ten, upper, 0-15 cm cores were collected from each of the three treated replicates at each sampling time. Field samples were frozen within approximately 3 hours of sampling and shipped frozen to Enviro-Test Laboratories for analysis. At the analytical laboratory the soil samples from the same horizon of each plot were blended then stored at -10 ºC until analysis.

3. Analytical Procedures

The analytical method AR 265-01 was used to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000, referred to in the report as RPA 427967). Soil samples of 20 g were extracted twice at ambient temperature for 5 minutes by mechanical agitation using acetonitrile/water (70/30, *v/v*) acidified with 0.1% formic acid. After each extraction step, extract and soil were separated by centrifugation and decantation. The soil extracts were combined and diluted with acidified water to result in a final solvent of acetonitrile/water (30/70) with 0.1% formic acid. Quantification was carried out by LC-MS/MS using external standardisation for the parent compound and its metabolites. The limit of quantification (LOQ) was 0.005 mg/kg for each analyte.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide and reference items M-01, M-02 and M-03 at levels of LOQ, 10 x LOQ and 100 x LOQ and processed in parallel to the dissipation samples. The mean recoveries were 96% (SD 9.7%) for fluopicolide, 92% (SD 11%) for M-01, and 97% (SD 12%) for M-02 and 98% (SD 12%) for M-03. No residues of fluopicolide or its metabolites were found above the LOQ in the analysed untreated samples.

The validation of the extraction was carried out during the study with samples taken immediately after the application of the test substance.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were not conducted according to FOCUS guidance document on degradation kinetics and are not acceptable for EU submissions. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.1.24. A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each site between the SFO and FOMC fits for fluopicolide. For the Ontario site, the FOMC fit provided no significant improvement, and the SFO fit was therefore accepted.

II. Results and Discussion

A. Analytical Methodology

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide in soil samples by HPLC-MS/MS.

B. Data:

Residues of fluopicolide and its metabolites M-01 (AE C653711) and M-02 (AE C657188) with time and depth in the Canadian soil are presented in Table 7.1.2.2.1- 85 to Table 7.1.2.2.1- 87. No residues of M-03 (AE 0608000) were detected throughout the trial.

Table 7.1.2.2.1- 85: Residues of fluopicolide in soil after three applications of 133 g a.s./ha values expressed as mg/kg

Depth [cm]	Sub plot	DAA3												
		-9	**-5**	**-4**	**-1**	**0**	**6**	**13**	**31**	**55**	**95**	**306**	**362**	**453**
0-7.5	1	0.11	0.068	0.31	0.23	0.35	0.23	0.21	0.18	0.14	0.091	0.096	0.052	0.029
	1-2	-	-	-	-	0.29	-	-	-	-	-	-	-	-
	2	0.12	0.096	0.25	0.17	0.30	0.24	0.25	0.22	0.23	0.18	0.098	0.053	0.049
	2-2	-	-	-	-	0.3	-	-	-	-	-	-	-	-
	3	0.13	0.14	0.19	0.19	0.30	0.22	0.26	0.16	0.25	0.16	0.086	0.077	0.092
	3-2	-	-	-	-	0.29	-	-	-	-	-	-	-	-
	mean	**0.120**	**0.101**	**0.250**	**0.197**	**0.300**	**0.230**	**0.240**	**0.187**	**0.207**	**0.144**	**0.093**	**0.061**	**0.057**
7.5-15	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	1-2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.02	0.0073	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.0098	0.0087	<LOQ	<LOQ
	2-2	-	-	-	-	<LOQ	-	-	-	-	-	-	-	-
	3	-	-	-	-	<LOQ	-	-	-	-	-	-	-	-
	3-2	-	-	-	-	<LOQ	-	-	-	-	-	-	-	-
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.010**	**0.005**	**<LOQ**	**<LOQ**
15-30	1	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	0.0073	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	0.017	<LOQ	<LOQ	0.0073
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	0.0085	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.011**	**<LOQ**	**<LOQ**	**0.002**
30-45	1	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ
	3	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

LOQ (limit of quantitation) = 0.005 mg/kg, DAA3: days after application three

Table 7.1.2.2.1- 86: Residues of M-01 in soil after in soil after three applications of 133 g a.s./ha values expressed as mg/kg

Depth [cm]	Sub plot	DAA3												
		-9	-5	-4	-1	0	6	13	31	55	95	306	362	453
0-7.5	1	<LOQ	<LOQ	0.0064	0.01	0.011	0.025	0.029	0.027	0.028	0.02	0.026	0.017	0.013
	1-2	-	-	-	-	0.010	-	-	-	-	-	-	-	-
	2	<LOQ	<LOQ	0.0058	0.0099	0.013	0.027	0.031	0.033	0.031	0.03	0.022	0.019	0.026
	2-2	-	-	-	-	0.011	-	-	-	-	-	-	-	-
	3	<LOQ	0.006	0.0058	0.012	0.013	0.024	0.034	0.037	0.029	0.033	0.024	0.022	0.027
	3-2	-	-	-	-	0.013	-	-	-	-	-	-	-	-
	mean	**<LOQ**	**0.002**	**0.006**	**0.011**	**0.012**	**0.025**	**0.031**	**0.032**	**0.029**	**0.028**	**0.024**	**0.019**	**0.022**
7.5-15	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	1-2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2-2	-	-	-	-	0.0058	-	-	-	-	-	-	-	-
	3	-	-	-	-	0.010	-	-	-	-	-	-	-	-
	3-2	-	-	-	-	0.0099	-	-	-	-	-	-	-	-
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.004**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
15-30	1	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.0073
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.002**
30-45	1	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ
	3	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

LOQ (limit of quantitation) = 0.005 mg/kg; DAA3: days after application three

Table 7.1.2.2.1- 87: Residues of M-02 in soil in soil after three applications of 133 g a.s./ha values expressed as mg/kg

Depth [cm]	Sub plot	DAA3												
		-9	-5	-4	-1	0	6	13	31	55	95	306	362	453
0-7.5	1	<LOQ	<LOQ	0.0082	0.013	0.012	0.024	0.020	0.0095	0.011	<LOQ	<LOQ	<LOQ	<LOQ
	1-2	-	-	-	-	0.0086	-	-	-	-	-	-	-	-
	2	<LOQ	<LOQ	0.0064	0.011	0.014	0.026	0.018	0.017	0.014	<LOQ	<LOQ	<LOQ	<LOQ
	2-2	-	-	-	-	0.010	-	-	-	-	-	-	-	-
	3	<LOQ	0.006	0.006	0.013	0.013	0.026	0.016	0.013	0.012	<LOQ	<LOQ	<LOQ	<LOQ
	3-2	-	-	-	-	0.012	-	-	-	-	-	-	-	-
	mean	**<LOQ**	**0.002**	**0.007**	**0.012**	**0.012**	**0.025**	**0.019**	**0.013**	**0.012**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
7.5-15	1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	1-2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.02	0.0073	<LOQ	<LOQ
	2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.0098	0.0087	<LOQ	<LOQ
	2-2	-	-	-	-	0.006	-	-	-	-	-	-	-	-
	3	-	-	-	-	0.013	-	-	-	-	-	-	-	-
	3-2	-	-	-	-	0.011	-	-	-	-	-	-	-	-
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.005**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.010**	**0.005**	**<LOQ**	**<LOQ**
15-30	1	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.0073
	3	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**0.002**
30-45	1	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ
	2	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ
	3	-	-	-	-	-	-	-	-	-	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**

LOQ (limit of quantitation) = 0.005 mg/kg; DAA3: days after application three

C. Residues

Residues of parent fluopicolide and M-01 (AE C653711) were mainly confined to the upper two layers in the soil profile (0-15 cm) with the majority remaining in the 0-7.5 cm segment. Residues of fluopicolide were detected at low levels in only four samples in the 7.5-15 cm segments and in four samples in the 15-30 cm segments. Fluopicolide was not detected below 30 cm in any samples. Residues of M-01 were detected in three samples in the 7.5-15 cm segments on Day 0 but was not detected in any lower segments or at any later sampling times. The metabolite M-02 (AE C657188) was detected in the 0-7.5 cm soil segment prior to the second application (-5 days) and remained quantifiable to the 55 day sampling period only. M-02 was detected in three samples in the 7.5-15 cm segments on Day 0 but was not detected in any lower segments or at any later sampling times. The metabolite M-03 (AE 0608000) was not detected in any of the samples analysed at any depth.

D. Kinetic Analysis

Fluopicolide degraded at a slow rate in a soil residue trial conducted in Canada. The reported DT_{50} and DT_{90} values of 204 and 677 days were derived by exponential equation. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.2.1/24. The resulting best-fit DT_{50} value for trigger endpoints are summarised below in Table 7.1.2.2.1- 88. Best fit kinetics are highlighted in bold.

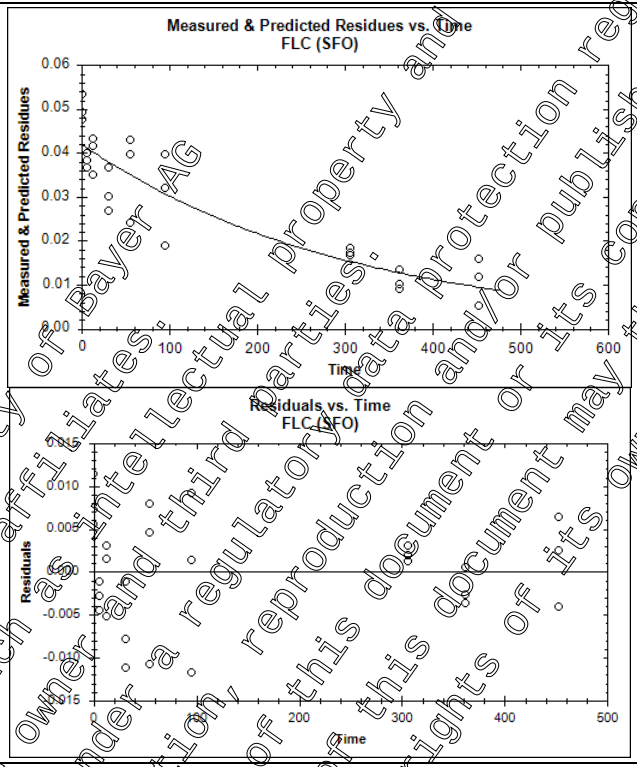
Table 7.1.2.2.1- 88: Degradation rate of fluopicolide under field conditions (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Ontario (Canada), CA 7.1.2.2.1/16, (Cosgrove 2004)	**SFO**	**0.04192**	**k 0.003304**	**10.3**	**1.68E-07**	**0.002361**	**0.004**	**209.8**	**697**
	FOMC	0.04402	α 1.006 β 162	10.2	n.r. n.r.	-0.3943 -199.5	2.407 523.5	160.6	1435

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.2.1- 89: Graphical representations of best fit model

Trial / Best Fit Model	Graphical Representations
Ontario (Canada) / SFO	

III. Conclusion

Following three applications of fluopicolide at a total rate of 400 g/ha to bare soil in summer 2002, the decline of fluopicolide and the formation and decline of its metabolites M-01, M-02 and M-03 was followed for up to 453 days at a site in eastern Canada. The DT_{50} and DT_{90} values of 209.8 and 697.0 days, respectively, were estimated for the decline of fluopicolide assuming simple first order kinetics (SFO).

The metabolite M-01 was detected in the 0-7.5 cm horizon throughout the study, however it was only seen in three samples at a single timepoint in the 7.5-15 cm soil depths. M-02 was only detected in the 0-7.5 cm soil depth for up to 55 days after application. M-02 was also detected in three samples at a single timepoint in the 7.5-15 cm soil depths. No residues of M-03 were found above LOQ (0.005 mg/kg) throughout the study.

Assessment and conclusion by applicant:

The study is considered valid to assess the dissipation of fluopicolide under field conditions in soil. The trial site in Ontario was considered representative for European climate conditions and soil properties.

The study meets the requirements to assess field persistence of fluopicolide and its metabolites under EU 283/2013. It is not suitable for assessing soil $DegT_{50matrix}$ values as the test substance was not applied as a single application as required by EFSA (2014) for legacy field studies and the design did not minimise soil surface processes immediately after application.

Data Point:	KCA 7.1.2.2.1/17
Report Author:	[redacted]
Report Year:	2005
Report Title:	AE C638206: Terrestrial Soil Dissipation Under Agricultural Field Conditions
Report No:	B004962
Document No:	M-251292-01-1
Guideline(s) followed in study:	USEPA (=EPA): 40 CFR 158.240, OPPTS 835.6100
Deviations from current test guideline:	Yes. The trial site in California was considered representative for European climate conditions and soil properties but the other three sites were not. The trial site in California meets the requirement for field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013. The study does not meet the requirements for assessing parent and metabolite soil DegT50matrix values as required by EFSA (2014) for legacy field studies.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities.
Acceptability/Reliability:	Supportive only

Executive Summary

A terrestrial field dissipation with fluopicolide, formulated as a suspendable concentrate containing 400 g/L fluopicolide was conducted at four locations throughout the United States. The four locations were Oviedo, Florida; Arkansaw, Wisconsin; Madera, California and Ephrata, Washington. A nominal application rate of 400 g fluopicolide / ha was applied as a single application between March and June 2001. The trial plots were planted with potatoes 2 to 8 days prior to application. To avoid disturbing the test soils, the crop was not harvested, but was instead allowed to senesce naturally.

Soil samples were collected to a depth of 9 cm in 15 cm increments at predetermined intervals for up to eighteen months after the application of the test substance then analysed for fluopicolide and its major soil metabolites.

The predominate analyte during the dissipation phase in each of the four trials was the parent fungicide, fluopicolide. The major metabolite found at all four sites was M-01 (AE C653711). Trace levels of this metabolite (just above the LOQ of 0.005 mg/kg to 0.016 mg/kg) were generally seen within 0.25 months after application and continued at levels just above the LOQ for the duration of the experiment (except at the California location where residues of M-01 were not seen after the 6 month sampling interval). Trace levels (just above the LOQ of 0.005 mg/kg to 0.010 mg/kg) of the metabolite M-02 were seen in soils from three of the four sites early in the dissipation phase. Residues of M-02 were not seen after two months after application at any of the sites. Residues of the metabolite M-03 were not detected in any soils analysed for this study.

The trial site in California was considered representative for European climate conditions and soil properties but the other three sites were not. A re-evaluation of the degradation kinetics at the Californian site in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit DFOP un-normalised DT_{50} value of 325.8 days and DT_{90} of 28.0 days for fluopicolide.

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide formulated as a suspendable concentrate (480 g/L fluopicolide)

Certificate of Analysis: AGF2001-0032-01

Lot No: OP210058

2. Trial Location & Soil

A terrestrial field dissipation with fluopicolide, formulated as a suspendable concentrate containing 400 g/L fluopicolide, has been conducted at four locations throughout the United States. The four locations were Oviedo, Seminole county, Florida; Arkansaw, Pepin county, Wisconsin; Madera Madera county, California and Ephrata, Grant county, Washington. The site located in Madera is referred to as Fresno, Fresno county, California in the report. This is the location of the PI test site not the trial location. The sites were fully characterised including soil texture, uniformity of soils and historical depth to groundwater. The characteristics of the soil are summarised in Table 7.1.2.2.1- 90. The plot sizes ranged from 0.02 to 0.28 acres and were planted with a variety of potatoes typically grown in the area. For sampling purposes, each test plot was divided into four subplots of comparable dimensions.

The crops were sprinkler irrigated in order to maintain at least the historical average rainfall for each site. Usually sufficient irrigation to maintain a viable crop was applied which exceeded the historical average rainfall during the growing season.

Table 7.1.2.2.1- 90: Location, site description and climatic data of test site

Characteristic	Units	Location			
		Florida	Wisconsin	California	Washington
Sampling depth	cm	0-15	0-15	0-15	0-15
pH	$CaCl_2$	7.4	6.8	7.7	8.2
Organic matter	%	0.9	1.8	1.8	0.9
Soil Density	g/L	1.43	1.44	1.39	1.47
Particle size distribution (USDA)					
Clay < 0.002 mm	%	3	4	3	4
Total silt 0.002 - 0.063 mm	%	1	8	8	16
Total sand 0.063 - 2 mm	%	96	88	89	80
Textural class	USDA	sand	sand	sand	loamy sand
Water Holding Capacity					
WHC at 1/3 bar (pF2.53)	Vol %	2.6	8.6	4.9	10.0
WHC at 15 bar (pF 4.2)	Vol %	2.2	-	2.9	-

n.d. = not determined

B. Study Design

1. Experimental Conditions

Fluopicolide was applied once as a suspendable concentrate at a nominal application rate of 400 g/ha on at each site between March and June 2001. Each application was confirmed by the analysis of filter paper plaques placed in the target area during application then retrieved immediately after application as well as by the analysis of the soil samples taken immediately after each application.

Trial	Florida	Wisconsin	California	Washington
Application date	9 March 2001	25 April 2001	1 June 2001	7 May 2001
Planting date	2 March 2001	18 April 2001	30 May 2001	1 May 2001

Rainfall, air temperature and soil temperature were measured continuously on site. Historical rainfall and pan evaporation which were compiled from the nearest National Oceanic and Atmospheric Administration (NOAA) Climatological Data recording weather station. The crops were sprinkler irrigated in order to maintain at least the historical average rainfall for each site. Usually sufficient irrigation to maintain a viable crop was applied which exceeded the historical average rainfall during the growing season. The potato crop was not harvested, the vines and potatoes were allowed to naturally senesce. Unwanted plant growth was controlled with a nonselective herbicide.

Soil dissipation of fluopicolide was studied for 18 months.

2. Sampling

Immediately after application four soil cores were taken to a depth of 0.00 - 0.15 m. At each additional post-application sampling interval, the soil cores consisted of six increments: 0.00 - 0.15 m, 0.15 - 0.30 m, 0.30 - 0.45 m, 0.45 - 0.60 m, 0.60 - 0.75 m and 0.75 - 0.90 m. At each sampling date 4 samples from each plot were taken (16 cores in total).

Samples were taken, directly after application (day 0) as well as 0.1, 0.25, 0.5, 1, 2, 3, 4, 5, 6, 8, 10, 12, 14, 16 and 18 months after application (MAA).

The soil cores were frozen immediately after sampling, then shipped to the analytical laboratory.

3. Analytical Procedures

The analytical method AR 265-01 was used to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000, referred to as RPA 427967 in the report). Soil samples of 20 g were extracted twice at ambient temperature for 5 minutes by mechanical agitation using acetonitrile/water (70/30, *v/v*) acidified with 0.1% formic acid. After each extraction step, extract and soil were separated by centrifugation and decantation. The soil extracts were combined and diluted with acidified water to result in a final solvent of acetonitrile/water (30/70) with 0.1% formic acid. Quantification was carried out by LC-MS/MS using external standardisation for the parent compound and its metabolites. The limit of quantification (LOQ) was 0.005 mg/kg for each analyte.

During analysis of the dissipation samples, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide and reference items M-01, M-02 and M-03 at levels of LOQ and 100 x LOQ and processed in parallel to the dissipation samples.

Compound		% Recoveries after Fortification							
		Florida		Wisconsin		California		Washington	
		Fortification level (mg/kg)							
		0.005	0.5	0.005	0.5	0.005	0.5	0.005	0.5
Fluopicolide	Mean	91.6	96.2	85.8	101.9	87.4	108.0	84.4	98.8
	SD	12.1	12.8	8.2	3.6	8.6	11.7	18.8	12.0
M-01	Mean	105.1	101.8	98.6	103.2	91.9	109.5	96.0	114.4
	SD	9.1	5.4	10.7	3.5	8.6	9.5	14.2	6.9
M-02	Mean	98.3	101.1	94.9	96.6	94.5	102.0	98.1	102.0
	SD	11.0	5.5	6.5	3.1	8.5	8.9	11.0	4.8
M-03	Mean	91.1	105.2	93.6	101.4	92.0	113.1	86.2	112.0
	SD	12.1	7.6	5.1	8.9	11.3	13.9	15.3	15.3

SD Standard deviation

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

4. Determination of degradation kinetics

The degradation kinetics determined in the report were not conducted according to FOCUS guidance document on degradation kinetics and are not acceptable for EU submissions. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data for the Californian site following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). The trial site in California was considered representative for European climate conditions and soil properties but the other three sites were not. Full details are provided in Documents KCA 7.1.2.2.1/15 and KCA 7.1.2.2.1/24. A brief summary of the approach for trigger endpoints is provided below.

To derive trigger endpoints, an initial comparison was performed for each site between the SFO and FOMC fits for fluopicolide. The comparison of the SFO and FOMC fits suggested bi-phasic decline, and the DFOP model was therefore also fitted. For the California site, DFOP provided the best fit to the residues, with the lowest χ^2 err% value, and was therefore accepted.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) in soil samples by HPLC-MS/MS.

B. Data

The results for fluopicolide and its metabolites M-01 (AE C653711) and M-02 (AE C657188) as soil residue concentrations (on a ppb basis) for each of the treated plots in Table 7.1.2.2.1- 91 to Table 7.1.2.2.1- 101. No residues of M-02 were detected at the Wisconsin site and no residues of M-03 (AE 0608000) were detected at any of the sites.

Table 7.1.2.2.1- 91: Residues of fluopicolide in soil from the Florida trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
0-15	1	72	156	148	241	377	164	107	13	44	46	24	57	34	22	40	13	5
	2	187	101	153	197	195	251	<LOQ	14	72	36	16	67	15	30	16	12	6
	3	83	87	225	557	485	233	8	29	33	40	21	94	68	17	38	17	1
	4	168	66	156	256	216	286	24	10	103	56	7	56	26	55	46	14	8
	mean	**128**	**103**	**171**	**313**	**318**	**234**	**35[1]**	**17**	**63**	**45**	**17**	**69**	**36**	**31**	**35**	**14**	**5**
15-30	1	n.s.	n.d.	<LOQ	n.d.	7	21	<LOQ	38	<LOQ	<LOQ	48	43	31	31	20	33	8
	2	n.s.	<LOQ	<LOQ	<LOQ	12	34	<LOQ	5	n.d.	n.d.	11	30	n.d.	15	<LOQ	7	<LOQ
	3	n.s.	<LOQ	12	<LOQ	5	<LOQ	n.d.	n.d.	n.d.	n.d.	<LOQ	15	6	n.d.	n.d.	8	20
	4	n.s.	n.d.	7	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	25	6	<LOQ	6	52	23
	mean	**n.s.**	<LOQ	**5[1]**	<LOQ	**6[1]**	**14[1]**	<LOQ	**11[1]**	<LOQ	<LOQ	**15[1]**	**28**	**11[1]**	**12[1]**	**7[1]**	**25**	**13[1]**
30-45	1	n.s.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	10	n.d.	25	<LOQ	<LOQ	13	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	33	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	n.d.	<LOQ	<LOQ	<LOQ	**8[1]**	**n.d.**	**n.d.**	**2[1]**	**n.d.**	**6[1]**	<LOQ	<LOQ	**3[1]**	**n.d.**	**n.d.**	**n.d.**
45-60	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ
	2	n.s.	n.d.	n.d.	n.d.	n.d.	28	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ
	mean	**n.s.**	n.d.	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.a.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	<LOQ	<LOQ	<LOQ
60-75	1	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	2	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	3	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	4	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	mean	**n.s.**	n.d.	**n.a.**	**n.a.**	**n.a.**	**2[1]**	**n.a.**	**n.d.**	**n.d.**	**n.a.**	**n.d.**	**n.d.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
75-90	1	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	n.d.	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD), n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value = LOQ

Table 7.1.2.2.1- 92: Residues of M-01 (AE C653711) in soil from the Florida trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
0-15	1	n.d.	<LOQ	<LOQ	6	5	5	8	<LOQ	6	<LOQ	n.d.	n.d.	n.d.	n.d.	8	n.d.	<LOQ
	2	n.d.	<LOQ	<LOQ	6	<LOQ	7	5	<LOQ	6	<LOQ	n.d.	n.d.	n.d.	n.d.	6	n.d.	<LOQ
	3	n.d.	<LOQ	9	8	8	10	<LOQ	<LOQ	6	<LOQ	n.d.	<LOQ	n.d.	n.d.	7	n.d.	<LOQ
	4	n.d.	n.d.	<LOQ	<LOQ	6	9	5	n.d.	11	<LOQ	n.d.	<LOQ	n.d.	<LOQ	7	n.d.	<LOQ
	mean	n.d.	<LOQ	**2[1]**	**5[1]**	**5[1]**	**8**	**3[1]**	**n.d.**	**7**	**<LOQ**	**n.d.**	<LOQ	**n.d.**	<LOQ	**7**	**n.d.**	**<LOQ**
15-30	1	n.s.	<LOQ	n.d.	n.d.	6	<LOQ	<LOQ	7	5	<LOQ	n.d.	<LOQ	n.d.	n.d.	10	n.d.	<LOQ
	2	n.s.	n.d.	n.d.	7	<LOQ	6	n.d.	<LOQ	6	8	n.d.	<LOQ	n.d.	<LOQ	5	n.d.	<LOQ
	3	n.s.	n.d.	n.d.	5	8	<LOQ	n.d.	6	8	6	5	9	n.d.	n.d.	7	<LOQ	<LOQ
	4	n.s.	n.d.	n.d.	<LOQ	5	6	n.d.	<LOQ	16	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	10	<LOQ	<LOQ
	mean	**n.s.**	n.d.	**n.d.**	**3[1]**	**5[1]**	**3[1]**	**<LOQ**	**3[1]**	**9**	**4[1]**	**1**	**2[1]**	**<LOQ**	**<LOQ**	**8**	**<LOQ**	**<LOQ**

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
30-45	1	n.s.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	9	<LOQ	5	7	<LOQ	n.d.	n.d.	n.d.	<LOQ	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**5**	<LOQ	n.d.	**7**	**5**	n.d.	n.d.	n.d.	<LOQ	n.d.
	mean	**n.s.**	n.d.	**n.d.**	**n.d.**	**<LOQ**	**n.d.**	**n.d.**	**4[1]**	**<LOQ**	**1[1]**	**4[1]**	**1[1]**	**n.d.**	**n.d.**	**n.d.**	**<LOQ**	**n.d.**
45-60	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ
	mean	**n.s.**	n.d.	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.a.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**<LOQ**	**<LOQ**	**<LOQ**
60-75	1	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	<LOQ	n.a.	<LOQ	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	2	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	3	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	4	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	<LOQ	<LOQ	n.a.	n.d.	n.a.	n.a.	n.a.
	mean	**n.s.**	n.d.	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.d.**	<LOQ	**n.a.**	<LOQ	<LOQ	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**
75-90	1	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	n.d.	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD), n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value > LOQ

Table 7.1.2.2.1- 93: Residues of M-02 (AE C657188) in soil from the Florida trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
0-15	1	n.d.	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.d.	n.d.	<LOQ	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.d.	<LOQ	5	<LOQ	n.d.	6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	n.d.	<LOQ	**1[1]**	**<LOQ**	**n.d.**	**1[1]**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
15-30	1	n.s.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.
	mean	**n.s.**	n.d.	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
30-45	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	**n.d.**	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	n.d.	**n.d.**	<LOQ	<LOQ	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
45-60	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	n.d.	**n.d.**	**n.d.**	<LOQ	**n.d.**	**n.a.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
60-75	1	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	2	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	3	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	4	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.d.	n.a.	n.d.	n.a.	n.a.	n.a.
	mean	**n.s.**	n.d.	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.d.**	**n.d.**	**n.a.**	**n.d.**	**n.d.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
75-90	1	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.d.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	n.d.	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD), n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value = LOQ

Table 7.1.2.2.1- 94: Residues of fluopicolide in soil from the Wisconsin trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA																
		0	0.1	0.75	1	2	3	4	5	6	8	10	12	14	16	18		
0-15	1	128	123	48	120	46	79	77	26	55	n.s.	n.s.	53	36	37	20		
	2	101	72	72	67	16	38	51	35	41	n.s.	n.s.	42	64	27	30		
	3	148	157	77	45	40	37	53	81	37	n.s.	n.s.	71	74	32	29		
	4	154	139	102	102	30	81	35	42	33	n.s.	n.s.	57	38	27	18		
	mean	**133**	**123**	**75**	**84**	**29**	**59**	**54**	**46**	**42**	**n.s.**	**n.s.**	**76**	**53**	**31**	**24**		
15-30	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7	n.s.	n.s.	n.d.	n.d.	n.d.	n.d.		
	2	n.s.	n.d.	n.d.	n.d.	n.d.	LOQ	<LOQ	n.d.	<LOQ	n.s.	n.s.	n.d.	<LOQ	5	12		
	3	n.s.	5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	6	n.d.	10		
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	n.d.	<LOQ	n.d.		
	mean	**n.s.**	**1[1]**	**n.d.**	**n.d.**	**n.d.**	**<LOQ**	**<LOQ**	**n.d.**	**2[1]**	**n.s.**	**n.s.**	**n.d.**	**2[1]**	**1[1]**	**6[1]**		

Depth [cm]	Sub plot	MAA														
		0	0.1	0.75	1	2	3	4	5	6	8	10	12	14	16	18
30-45	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.s.**	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
45-60	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.s.	n.s.	n.a.	n.d.	n.a.	n.d.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.s.	n.s.	n.a.	n.d.	n.a.	n.d.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.s.	n.s.	n.a.	n.d.	n.a.	n.d.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.s.	n.s.	n.a.	n.d.	n.a.	n.d.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.s.**	**n.s.**	**n.a.**	**n.d.**	**n.a.**	**n.d.**
60-75	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.s.**	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
75-90	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.s.**	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD),
n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value > LOQ

Table 7.1.2.2.1- 95: Residues of M-01 (AE C653711) in soil from the Wisconsin trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA														
		0	0.1	0.75	1	2	3	4	5	6	8	10	12	14	16	18
0-15	1	n.d.	<LOQ	5	10	8	7	6	<LOQ	6	n.s.	n.s.	<LOQ	<LOQ	<LOQ	<LOQ
	2	n.d.	n.d.	7	8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.s.	n.s.	n.d.	5	n.d.	n.d.
	3	n.d.	<LOQ	6	<LOQ	7	6	8	<LOQ	<LOQ	n.s.	n.s.	<LOQ	<LOQ	<LOQ	n.d.
	4	n.d.	n.d.	9	9	8	6	<LOQ	6	<LOQ	n.s.	n.s.	<LOQ	<LOQ	n.d.	n.d.
	mean	**n.d.**	<LOQ	**7**	**7[1]**	**6[1]**	**5[1]**	**4[1]**	**3[1]**	**2[1]**	**n.s.**	**n.s.**	**<LOQ**	**1[1]**	**<LOQ**	**<LOQ**
15-30	1	n.s.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ	<LOQ	n.s.	n.s.	<LOQ	<LOQ	<LOQ	<LOQ
	2	n.s.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ	<LOQ	n.s.	n.s.	<LOQ	6	<LOQ	<LOQ
	3	n.s.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.s.	n.s.	<LOQ	<LOQ	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	n.s.	n.s.	<LOQ	<LOQ	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**n.s.**	**n.s.**	**<LOQ**	**2[1]**	**<LOQ**	**<LOQ**
30-45	1	n.s.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	<LOQ	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	<LOQ	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.s.	n.s.	n.d.	<LOQ	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**<LOQ**	**n.d.**	**n.d.**	**n.d.**	**n.s.**	**n.s.**	**n.d.**	**<LOQ**	**n.d.**	**n.d.**
45-60	1	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.s.	n.s.	n.a.	n.d.	n.a.	n.d.
	2	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.s.	n.s.	n.a.	n.d.	n.a.	n.d.
	3	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.s.	n.s.	n.a.	n.d.	n.a.	n.d.
	4	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.s.	n.s.	n.a.	n.d.	n.a.	n.d.
	mean	**n.s.**	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.s.**	**n.s.**	**n.a.**	**n.d.**	**n.a.**	**n.d.**
60-75	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.s.**	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

Depth [cm]	Sub plot	MAA														
		0	0.1	0.75	1	2	3	4	5	6	8	10	12	14	16	18
75-90	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.s.**	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD),
n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value = LOQ

Table 7.1.2.2.1- 96: Residues of fluopicolide in soil from the California trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA															
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
0-15	1	160	126	168	93	91	83	80	45	24	32	22	27	16	12	8	<LOQ	<LOQ
	2	155	87	105	67	83	103	52	37	28	25	24	18	18	11	6	<LOQ	<LOQ
	3	127	121	113	92	98	66	56	42	45	33	23	20	17	16	10	11	<LOQ
	4	159	175	89	56	72	72	45	42	34	25	24	13	13	13	7	<LOQ	6
	mean	**150**	**127**	**116**	**77**	**65**	**81**	**58**	**42**	**33**	**29**	**23**	**20**	**16**	**13**	**8**	**3**	**2**
15-30	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	6	8	6	5	7	6	<LOQ	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	8	6	6	5	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	9	6	<LOQ	5	<LOQ	<LOQ	6	<LOQ	n.d.	n.d.	n.d.
	4	n.s.	<LOQ	n.d.	n.d.	<LOQ	n.d.	<LOQ	6	6	n.d.	<LOQ	7	<LOQ	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	<LOQ	**n.d.**	**n.d.**	<LOQ	**n.d.**	**2[1]**	**5[1]**	**4**	**5[1]**	**3[1]**	**5[1]**	**4[1]**	<LOQ	**n.d.**	**n.d.**	**n.d.**

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
30-45	1	n.s.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.	<LOQ	n.d.	n.d.	<LOQ	<LOQ	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	<LOQ	**n.d.**	**n.d.**	<LOQ	**n.d.**	**n.d.**	<LOQ	<LOQ	**n.d.**	<LOQ	<LOQ	**n.d.**
45-60	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
60-75	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
75-90	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD), n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value > LOQ

Table 7.1.2.2.1- 97: Residues of M-01 (AE C653711) in soil from the California trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
0-15	1	n.d.	9	11	8	9	10	n.d.	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.d.	7	12	10	13	12	n.d.	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.d.	6	10	10	7	8	n.d.	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.d.	9	10	6	10	6	n.d.	<LOQ	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.d.**	**8**	**11**	**9**	**7**	**9**	**n.d.**	**<LOQ**	**<LOQ**	**<LOQ**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
15-30	1	n.s.	n.d.	n.d.	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	<LOQ	<LOQ	<LOQ	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	n.d.	**n.d.**	**n.d.**	<LOQ	<LOQ	**n.d.**	**n.d.**	**n.d.**	<LOQ	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
30-45	1	n.s.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**<LOQ**	**<LOQ**	**n.d.**	**n.d.**	**<LOQ**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
45-60	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
60-75	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
75-90	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD),
n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value = LOQ

Table 7.1.2.2.1- 98: Residues of M-02 (AE C657188) in soil from the California trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
0-15	1	n.d.	9	8	<LOQ	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.d.	7	13	<LOQ	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.d.	6	7	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.d.	8	8	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.d.**	**8**	**9**	**<LOQ**	**<LOQ**	**<LOQ**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
15-30	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	n.d.	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
30-45	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
45-60	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
60-75	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
75-90	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD), n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value > LOQ

Table 7.1.2.2.1- 99: Residues of fluopicolide in soil from the California trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA																
		0	**0.1**	**0.25**	**0.5**	**0.75**	**1**	**2**	**3**	**4**	**5**	**6**	**8**	**10**	**12**	**14**	**16**	**18**
0-15	1	71	102	54	68	146	120	115	84	43	52	70	36	50	25	60	45	42
	2	95	78	86	64	166	72	90	82	60	61	58	40	63	18	53	31	6
	3	100	157	71	84	139	103	142	94	51	74	60	77	82	32	55	31	21
	4	153	73	91	138	94	56	99	n.d.	55	69	80	60	52	82	70	34	28
	mean	**105**	**103**	**76**	**89**	**95**	**88**	**112**	**65**	**52**	**64**	**67**	**56**	**62**	**39**	**60**	**8**	**7**
15-30	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	n.d.	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
30-45	1	n.s.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.	n.d.	n.d.	n.d.	<LOQ	<LOQ	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**<LOQ**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**<LOQ**	**<LOQ**	**n.d.**
45-60	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
60-75	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
75-90	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD), n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value = LOQ

Table 7.1.2.2.1- 100: Residues of M-01 (AE C653711) in soil from the Washington trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
0-15	1	n.d.	n.d.	<LOQ	6	14	12	15	12	11	9	7	<LOQ	<LOQ	<LOQ	6	n.d.	<LOQ
	2	n.d.	n.d.	<LOQ	6	1	13	<LOQ	9	<LOQ	7	5	<LOQ	8	n.d.	6	n.d.	n.d.
	3	n.d.	<LOQ	<LOQ	10	10	14	9	13	13	18	10	7	15	6	<LOQ	6	5
	4	n.d.	<LOQ	6	11	8	10	5	n.d.	12	13	14	13	10	9	7	<LOQ	<LOQ
	mean	**n.d.**	**<LOQ**	**2[1]**	**8**	**8**	**12**	**9[1]**	**9[1]**	**9[1]**	**12**	**9**	**5[1]**	**8[1]**	**4[1]**	**5[1]**	**2[1]**	**1[1]**
15-30	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5	6	<LOQ	n.d.	n.d.	n.d.	n.d.	**n.d.**
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**1[1]**	**2[1]**	**<LOQ**	**n.d.**	**<LOQ**	**n.d.**	**n.d.**	**n.d.**

Depth [cm]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
30-45	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**2[1]**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
45-60	1	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
60-75	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
75-90	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD), n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value > LOQ

Table 7.1.2.2.1- 101: Residues of M-02 (AE C6557188) in soil from the Washington trial after an application of 400 g a.s./ha (expressed as ppb)

Depth [m]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
0-15	1	n.d.	<LOQ	<LOQ	<LOQ	5	<LOQ	<LOQ	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.d.	<LOQ	<LOQ	<LOQ	<LOQ	5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.d.	<LOQ	<LOQ	7	5	6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOQ	n.d.
	4	n.d.	<LOQ	<LOQ	7	5	<LOQ	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.d.**	**<LOQ**	<LOQ	**4[1]**	**4[1]**	**3[1]**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
15-30	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**n.d.**	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	n.d.	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.a**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
30-45	1	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	2	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	3	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	4	n.s.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	mean	**n.s.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
45-60	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
60-75	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.d.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.d.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

Depth [m]	Sub plot	MAA																
		0	0.1	0.25	0.5	0.75	1	2	3	4	5	6	8	10	12	14	16	18
75-90	1	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	3	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	4	n.s.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean	**n.s.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**

MAA months after application, n.d. was used to distinguish those values less than the limit of detection (LOD),
n.s. not sampled, n.a. not analysed, LOQ (limit of quantitation) = 0.005 mg/kg (5 ppb), [1]Replicate value < LOQ

C. Residues

The predominate analyte during the dissipation phase in each of the four trials was the parent fungicide, fluopicolide. The major metabolite found at all four sites was M-01 (AE C653711). Trace levels of this metabolite (just above the LOQ of 0.005 mg/kg to 0.016 mg/kg) were generally seen within 0.25 months after application and continued at levels just above the LOQ for the duration of the experiment (except at the California location where residues of M-01 were not seen after the 6 month sampling interval). Trace levels (just above the LOQ of 0.005 mg/kg to 0.010 mg/kg) of the metabolite M-02 were seen in soils from three of the four sites early in the dissipation phase. Residues of M-02 were not seen after two months after application at any of the sites. Residues of the metabolite M-03 were not detected in any soils analysed for this study.

Residues of fluopicolide and its metabolites with time and depth in the Florida soil are presented in Table 7.1.2.2.1- 91 to Table 7.1.2.2.1- 93. Residues of parent fluopicolide and M-01 (AE C653711) were mainly confined to the upper two layers in the soil profile (0-30 cm). Residues of fluopicolide were detected in one sub plot down to a depth of 60 to 75 cm one month after application but no residues were detected at the same time in the other three replicate subplots and residues of fluopicolide on the following two time points were only detected in the top 30 cm of the soil profile. Residues of M-01 were detected 8 months after treatment at depths of 30-45 cm in three of the four replicate subplots (maximum 0.005 mg/kg) and one of the four subplots at 45-60 m (maximum 0.007 mg/kg). Residues below this level were not detected or were below the LOQ in all subplots.

Residues of fluopicolide and its metabolites with time and depth in the Wisconsin soil are presented in Table 7.1.2.2.1- 94 to Table 7.1.2.2.1- 95. Residues of parent fluopicolide and M-01 were mainly confined to the upper soil layer in the soil profile (0-15 cm) with only trace amounts, generally less than limit of quantitation, detected in the second and third layers on sporadic occasions. No residues of M-02 or M-03 were detected throughout the trial at Wisconsin.

Residues of fluopicolide and its metabolites with time and depth in the California soil are presented in Table 7.1.2.2.1- 96 to Table 7.1.2.2.1- 98. In this trial trace amounts of M-02 (AE 657188), which is formed at the same time as M-01 through cleavage of the parent, were observed in the first month of the trial. These residues declined to below the limit of detection after 1 month whereas residues of M-01 were detected up to 8 months after application. Residues were mainly confined to the upper soil layer in the soil profile (0-15 cm) with only trace amounts, generally less than limit of quantitation, detected in the second and third layers on sporadic occasions.

Residues of fluopicolide and its metabolites with time and depth in the Washington soil are presented in Table 7.1.2.2.1- 99 to Table 7.1.2.2.1- 101. Residues of fluopicolide and its metabolites were mainly confined to the upper soil layer in the soil profile (0-15 cm) with only trace amounts, generally less than limit of quantitation, detected in the second and third layers on sporadic occasions. Residues of M-01 were detected in one subplot at a depth of 45-60 cm after 6 months but residues in the other three subplots were confined to the 0-15 cm layer. Similarly residues of M-01 in cores taken after 5 months and 8 months remained in the upper 30 cm of the soil profile.

D. Kinetic Analysis

The reported dissipation of fluopicolide and M-01, evaluated with the software ModelMaker, are shown below. The degradation kinetics determined in the report were not conducted according to FOCUS guidance document on degradation kinetics and are not acceptable for EU submissions.

Table 7.1.2.2.1- 102: Reported dissipation rates of fluopicolide and M-01 under field conditions

Trial Location	SFO DT_{50} (days)	SFO DT_{50}, normalised to 25 °C (days)	
	Fluopicolide	Fluopicolide	M-01
Florida	107	96	37
Wisconsin	231	105	22
California	72	74	11
Washington	315	151	39

The trial site in California was considered representative for European climate conditions and soil properties but the other three sites were not. DT_{50} and DT_{90} values for the degradation of fluopicolide have been re-calculated from the reported data for the Californian site following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for Document KCA 7.1.2.2.1/24. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.2.1- 103. Best fit kinetics are highlighted in bold.

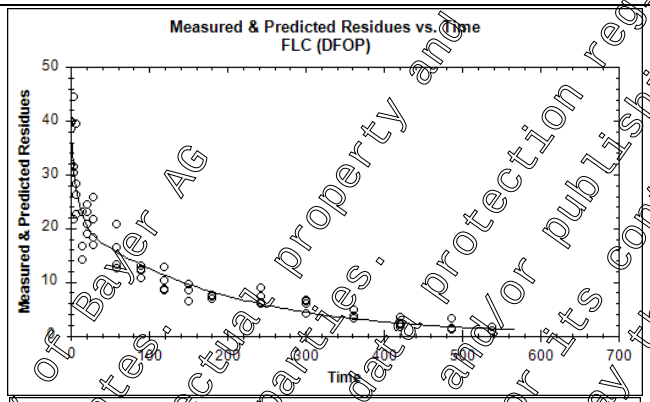
Table 7.1.2.2.1- 103: Re-evaluated degradation rate of fluopicolide under field conditions (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2 - %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
California (USA), CA 7.1.2.2.1/17, ([redacted] 2005)	SFO	29.91	k 0.008943	19.3	9.86E-15	0.007148	0.011	77.5	257.5
	FOMC	36.13	α 0.6687 β 17.43	11	n.r. n.r.	0.4684 4.929	0.869 29.92	31.7	527.7
	DFOP	**37.55**	**k1 0.1191 k2 0.005296 g 0.4383**	**8.23**	**0.00182 7.17E-10 n.r.**	**0.04176 0.003827 0.3343**	**0.196 0.007 0.542**	**28**	**325.8**

Best fit model highlighted in bold

A graphical representation of the final kinetic fit is shown below.

Table 7.1.2.2.1- 104: Graphical representations of best fit model

Trial / Best Fit Model	Graphical Representations
California (USA) / DFOP	

III. Conclusion

Following a single application of fluopicolide at a rate of 400 g/ha to bare soil planted with potatoes in summer 2001, the decline of fluopicolide and the formation and decline of its metabolites M-01, M-02 and M-03 was followed for up to 18 months at four locations throughout the United States.

The metabolites M-01 (AE C653711) and M-02 (AE C657188) were detected in all soils. M-01 was detected at low concentrations in 0-15 cm, 15-30 cm and at one or two occasions in the 30-45 cm soil depths. M-02 was detected only at early timepoints in the 0-15 cm depth. No residues of M-03 were detected throughout the study.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit DFOP un-normalised DT_{50} value of 28 days and DT_{90} value of 325.8 days for fluopicolide at the site in California, which was the only site considered representative for European climate conditions and soil properties.

Assessment and conclusion by applicant:

The study is considered valid in part to assess the dissipation of fluopicolide under field conditions in soil. The trial site in California was considered representative for European climate conditions and soil properties but the other three sites were not.

This trial site meets the requirements to assess field persistence of fluopicolide and its metabolites under EU 283/2013. The study is not suitable for assessing soil $DegT_{50matrix}$ values as defined by EFSA (2014) for legacy field studies as the test plots were cropped and the design did not minimise soil surface processes. Experimental evidence has established plant uptake is significant for fluopicolide and a number of its metabolites including M-01.

Data Point:	KCA 7.1.2.2.1/18
Report Author:	[redacted]
Report Year:	2019
Report Title:	Amendment no. 2 to final report - Terrestrial field dissipation study with BAM SC 125 in Germany and the United Kingdom
Report No:	M-647366-03-1
Document No:	M-647366-03-1
Guideline(s) followed in study:	Regulation (EC) No. 1107/2009 of the European Parliament and of the Council of 21 October 2009 including Data Requirements SANCO/11803/2010 Rev. 7 and Test Methods SANCO/11843/2010 Rev. 4 EFSA Guidance Document for Evaluating Laboratory and Field Dissipation Studies to obtain DegT50 Values of Active Substances of Plant Protection Products and Transformation Products of these Active Substances in Soil, EFSA Journal 2014; 12(5):3662, 2014
Deviations from current test guideline:	Yes. The trial site at Burscheid (Germany) meets the requirement for assessing test substance soil DegT50matrix values as required by EFSA (2014) for field studies. The endpoints may be too conservative for comparison to field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

Soil degradation of M-01 (AE C653711) under Northern European field conditions was investigated after application of M-01 onto bare soil plots in Burscheid (Germany) and Great Chishill (United Kingdom).

BAM SC 125 is a suspension concentrate formulation, containing 125 g/L M-01. The formulation was sprayed once onto 450 sqm to 564 sqm plots at a rate of 0.8 L/ha, corresponding to nominal 100 g/ha M-01. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface.

Soil cores were taken 0 days before up to 744 days post-application to a maximum depth of 110 cm. The soil cores were cut into 10-cm soil layers, bulked soil layers were homogenised and finally analysed for M-01.

Sub-samples of homogenised soil were extracted in a microwave extractor with organic solvent. Potential matrix effects were eliminated by using an internal standard solution of isotopically labeled reference items added to sample extracts. Following separation of fine particles from soil extracts by centrifugation the identification and quantitation of the analytes was performed by high performance liquid chromatography using MS/MS detection in the multiple reaction monitoring mode. The analytical method was validated using three different soils. The limit of quantitation (LOQ) was 1.0 µg/kg and the limit of detection (LOD) was 0.3 µg/kg for M-01.

At Burscheid (Germany), the mean amount of M-01 at day 0 was 108 g/ha, representing 108% of the nominal application rate. M-01 declined from 104 g/ha in soil at day 0 to 11.5 g/ha at day 701.

At Great Chishill (United Kingdom), the mean amount of M-01 at day 0 was 59.8 g/ha, representing 60% of the nominal application rate. M-01 declined from 59.8 g/ha in soil at day 0 to 31.9 g/ha at day 744.

Under field conditions M-01 residues were translocated up to 90 cm (Burscheid, Germany) and 110 cm (Great Chishill, United Kingdom) depth, whilst 67-100% (Burscheid, Germany) and 55-100% (Great Chishill) of residues remained in the top 0-30 cm at all timepoints. It is concluded there was some mobility of M-01 to deeper soil layers at the Burscheid trial but >97% of residues were detected in 0-100 cm soil depth. In contrast there was significant mobility of M-01 to deeper soil layers with only >80% of residues retained in 0-100 cm soil depth at the Great Chishill site. Consequently robust $DegT_{50}$ values can be obtained for the compound from data from the Burscheid trial, but according to FOCUS (2014) the Great Chishill trial site is not acceptable to determine $DegT_{50}$ values due to significant leaching out of the sampled soil depths.

I. Materials and Methods

A. Materials

1. Test Item

AE C653711 (M-01) formulated as a suspension concentrate (125g/L AE C653711)

Certificate of Analysis:	01865-00
Lot No:	2015-000656

2. Trial Location & Soil

A terrestrial field dissipation with M-01 (AE C653711) a suspension concentrate formulation, containing 125 g/L M-01 was conducted at two locations in Northern Europe. The two locations were H[redacted], Burscheid (Germany) and Great Chishill, Cambridgeshire (United Kingdom). The sites were fully characterised, and the results summarised in Table 7.1.2.2.1- 105. The plot sizes ranged from 450 sqm to 564 sqm. The control plot was prepared at least 5 m away from the treated plots.

A terrestrial field dissipation study with fluopicolide was conducted concurrently at the same sites.

Table 7.1.2.2.1- 105: Location, site description and climatic data of test sites

Characteristic	Units	Sampling depth [cm]			
		0-30	30-50	50-75	75-100
Soil Designation	-	**Burscheid (Germany)**			
Soil ID	-	VG08			
Geographic Location City Country	-	Burscheid [redacted] Germany			
pH	$CaCl_2$	5.3	5.6	5.6	5.6
Organic carbon	[% Carbon]	1.2	0.4	0.1	0.1
CEC	[meq/100 g]	12.8	11.8	12.4	11.9
Chalk	[% $CaCO_3$]	12.8	11.8	12.4	11.9
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 21 61 18	 23 57 20	 19 43 38	 13 37 50
Textural class	USDA	silt loam	silt loam	loam	loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 49.3 35.2	 50.0 31.9	 45.9 30.3	 39.1 25.9
Soil Designation	-	**Great Chishill (United Kingdom)**			
Soil ID	-	ENG8			
Geographic Location City Country	-	[redacted] Great Chishill, Cambridgeshire United Kingdom			
pH	$CaCl_2$	7.2	7.5	7.7	7.6
Organic carbon	[% Carbon]	2.1	1.1	0.5	0.5
CEC	[meq/100 g]	28.9	26.1	19.9	17.4
Chalk	[% $CaCO_3$]	1.3	5.8	37.9	43.1
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 41 23 36	 45 21 34	 53 23 24	 51 23 26
Textural class	USDA	clay	clay	clay	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol%	 60.8 29.2	 57.2 27.9	 52.6 25.3	 49.5 25.9

B. Study Design

1. Experimental Conditions

BAM SC 125 is a suspension concentrate formulation, containing 125 g/L M-01 (AE C653711). The product was sprayed onto bare soil once at each site at an application rate of 0.8 L/ha and 600 L/ha water, corresponding to 100 g/ha of M-01 during May 2015. Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of a nonselective herbicide to control weeds.

Air temperature, precipitation including irrigation and sunshine data were recorded on site during the field tests.

Soil dissipation of M-01 was studied for up to 744 days.

2. Sampling

The treated plot of each trial was divided into three sub-plots. From each sub-plot 10 soil cores were taken and combined together at each sampling interval (30 cores in total).

Samples were taken from treated plots on following occasions: 0 (post-application; each 0-10 cm depth), 7, 15 (each 0-60 cm depth), 21-22, 28-29, 63-68 (each 10-85 cm depth), and 121-135, 171-204, 259-307, 395-400, 436-470, 518-558, 605-670, 701-744 (each 0-110 cm depth) after treatment. Samples were taken from the control plot on the following occasions: 0 days before application, 701 and 744 days after application.

Soil cores taken from the sites were deep frozen to -18°C within twenty-four hours after sampling, then shipped frozen to the analytical laboratory in Germany.

3. Analytical Procedures

The analytical method 01445 was used to determine levels of M-01 (AE C653711). Soil samples of 5 g were extracted in a microwave extractor with a mixture of acetonitrile/water (4/1, v/v). The extracts were centrifuged to remove fine particles of the soil. Possible matrix effects of M-01 were eliminated by using an internal standard solution of isotopic labelled reference items. Quantification was carried out by LC-MS/MS. The limit of quantitation (LOQ) for M-01 was 1.0 µg/kg in soil. The limit of determination (LOD) for M-01 was 0.3 µg/kg.

During analysis of samples, concurrent recovery samples were prepared freshly by fortification of control samples with test item M-01 at levels of LOQ, 10 x LOQ, 500 x LOQ and 1000 x LOQ and processed in parallel to the dissipation samples throughout the study. The results are summarised in the table below.

Single Values [%]	No of Recoveries	Fortification Level [µg/kg]	Mean [%]	RSD [%]
70, 76, 77, 77, 77, 78, 78, 78, 79, 79, 80, 81, 81, 82, 82, 83, 84, 85, 85, 85, 85, 86, 86, 87, 87, 87, 87, 88, 88, 88, 88, 88, 88, 88, 89, 89, 89, 89, 89, 89, 89, 89, 90, 90, 90, 91, 91, 91, 91, 91, 91, 91, 91, 91, 92, 92, 92, 92, 92, 93, 93, 93, 93, 94, 94, 94, 94, 94, 94, 94, 95, 95, 95, 95, 95, 95, 95, 95, 95, 96, 96, 96, 96, 96, 97, 97, 97, 97, 97, 97, 97, 97, 98, 98, 98, 98, 98, 98, 98, 98, 99, 99, 99, 99, 99, 99, 100, 100, 100, 100, 100, 100, 100, 100, 100, 101, 101, 101, 101, 102, 102, 102, 102, 102, 102, 102, 103, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 106, 106, 106, 107, 108, 108, 108, 108, 109, 109, 109, 109, 109, 109, 109, 109, 110, 110, 110, 110, 110, 111, 111, 111, 111, 112, 112, 112, 112, 112, 112, 113, 113, 113, 113, 114, 114, 114, 114, 114, 114, 115, 115, 116, 116, 116, 117, 117, 117, 117, 118, 118, 119, 119, 119, 120, 120	206	1.0	99	10.9

Single Values [%]	No of Recoveries	Fortification Level [µg/kg]	Mean [%]	RSD [%]
82, 84, 86, 88, 88, 88, 89, 90, 90, 91, 91, 91, 91, 92, 92, 93, 94, 94, 94, 95, 95, 95, 95, 95, 95, 95, 95, 95, 96, 96, 96, 96, 96, 96, 96, 96, 97, 97, 97, 97, 97, 97, 97, 97, 97, 97, 98, 98, 98, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 100, 100, 100, 100, 100, 100, 100, 100, 100, 100, 101, 101, 101, 101, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 106, 106, 106, 106, 106, 106, 106, 106, 106, 107, 107, 107, 107, 107, 107, 107, 107, 107, 108, 108, 108, 108, 108, 108, 108, 108, 108, 109, 109, 109, 109, 109, 109, 109, 109, 109, 109, 109, 109, 109, 110, 110, 110, 110, 110, 110, 110, 110, 110, 111, 111, 111, 111, 111, 111, 111, 111, 112, 112, 112, 112, 112, 112, 112, 113, 113, 113, 113, 113, 114, 114, 114, 114, 115, 115, 115, 116, 116, 117, 117, 117, 117, 119, 119, 119, 120	206	10	104	7.1
87	1	500		-
80, 85	2	1000	83	-
Overall recovery	415		101	9.5

RSD = Relative standard deviation

The validation of the extraction was carried out during the study with samples taken immediately after the application of the test substance.

4. Evaluation of the Data and Kinetic Calculations

For evaluation of degradation kinetics of the test item according to the FOCUS guidance document on degradation kinetics, the total residues of the test item in the soil profile covering all soil horizons was calculated according to the following procedure:

- values between LOD and LOQ were set to the measured values.
- values < LOD were set to 0.5 LOD for samples after, before or deeper as a value > LOD or for samples between (> LOD and <LOQ). The curve was cut off after the first non-detect (< LOD), if no later value > LOQ followed.
- at day 0, values < LOD in deep horizons were set to 0.

The results in [µg/kg] were converted to [g/ha] considering the actual soil density of the corresponding soil layer.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of M-01 in soil samples by HPLC-MS/MS.

B. Data:

The results for residues of M-01 in different soil depths are presented below (expressed as g/ha) Table 7.1.2.2.1- 106 to Table 7.1.2.2.1- 107.

At the Burscheid trial site, residues of M-01 were detected at concentration > LOQ at soil depths down to 70 cm. The metabolite was detected in the 0-10 cm, 10-20 cm, and 20-30 cm soil horizons throughout the Burscheid trial. The maximum residue level in the 10-20 cm and 20-30 cm horizons were observed at DAT 121 at concentrations of 16.4 g/ha and 11.5 g/ha respectively (both Subplot 3). In samples taken from DAT 63 to 701 days low residues were detected in the underlying 30-40 cm horizon at concentrations ranging from <LOD to 5.78 g/ha. In 40-50 cm, 50-60 cm and 60-70 cm soil layers residues > LOQ were detected at DAT 121 and 171 only. In deeper soil layers (70-80, 80-90, 90-100 and 100-110 cm) no residues above the LOQ were detected.

It is concluded there was some mobility of M-01 to deeper soil layers but >97% of residues were detected in 0-100 cm soil depth (out of a total measured depth of 110 cm) and consequently robust $DegT_{50}$ values can be obtained for the compound from data from the Burscheid trial.

At the Great Chishill trial site, residues of M-01 were detected at concentrations > LOQ at soil depths down to 110 cm. The metabolite was detected in the 0-10 cm, 10-20 cm, 20-30 cm and 30-40 cm soil horizons throughout the Great Chishill trial. The maximum residue level in the 10-20 cm horizon was 19.3 g/ha at DAT 68 (Subplot 2), 5.42 g/ha at DAT 204 in the 20-30 cm horizon (Subplot 2) and 6.86 g/ha at DAT 470 in the 30-40 cm horizon (Subplot 3). In 40-50 cm, 50-60 cm, 60-70 cm and 70-80 cm soil layers residues > LOQ were detected at DAT 135 to DAT 400 only and no residues > LOQ were detected in 80-90 cm soil layer. However in the deepest soil layers 90-100 cm and 100-110 cm, residues at concentrations up to 3.53 g/ha (Subplot 1) and 6.19 g/ha (Subplot 2) were detected from DAT 135 to DAT 744.

The soil at Great Chishill is a heavy clay loam over calcareous clay. Chromatographic mobility is not expected in this soil however high levels of M-01 at soil depths were observed in two out of three subplots (Subplot 1 and 2) due to bypass flow in the heavy clay soil. It is concluded there was significant mobility of M-01 to deeper soil layers with only >79% of residues retained in 0-100 cm soil depth (out of a total measured depth of 110 cm). According to EFSA (2014) the Great Chishill trial site is not acceptable to determine $DegT_{50}$ values due to significant leaching out of the sampled soil depths.

Table 7.1.2.2.1- 106: Residues of M-01 (AE C653711) in different soil depths from the Burscheid trial after an application of 100 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	7	15	21	28	63	121	171	259	395	436	518	605	701
0-10	T1	98.9	86.3	132	114	139	79.3	21.6	18.2	11.8	8.63	7.71	5.6	3.36	3.31
	T2	131	121	129	179	107	84.8	23.9	19.1	12.3	9.17	7.57	8.65	3.75	3.86
	T3	94.7	107	107	143	135	81.2	23.1	17.0	13.4	10.5	9.97	6.37	4.86	3.95
	mean	108.2	104.8	122.7	145.3	127.0	81.8	22.9	18.1	12.5	9.43	8.42	6.87	3.99	3.71
10-20	T1	-	<LOD	<LOD	<LOD	<LOD	3.57	4.73	11.4	10.4	6.96	5.82	3.93	3.53	3.23
	T2	-	<LOD	<LOD	[0.63]	<LOD	4.00	8.44	6.34	6.88	6.82	4.0	4.74	3.08	3.77
	T3	-	<LOD	<LOD	<LOD	<LOD	5.88	16.4	14.7	8.25	10.1	7.72	5.11	4.99	3.96
	mean	-	<LOD	<LOD	0.21	<LOD	4.48	9.86	10.8	8.51	7.96	6.19	4.59	3.87	3.65
20-30	T1	-	<LOD	[0.80]	<LOD	<LOD	[0.84]	3.66	7.83	8.16	4.95	2.71	3.57	3.93	2.28
	T2	-	<LOD	<LOD	<LOD	<LOD	[0.98]	6.06	3.72	3.48	4.04	2.67	3.01	2.02	3.05
	T3	-	<LOD	<LOD	<LOD	<LOD	[1.56]	11.5	8.8	3.39	6.24	4.37	2.86	4.19	3.88
	mean	-	<LOD	[0.27]	<LOD	<LOD	[1.13]	7.07	6.78	4.91	5.08	3.25	3.15	3.38	3.07
30-40	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	[1.08]	3.74	3.23	2.23	[1.92]	2.17	2.20	[1.21]
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	2.73	1.99	[1.15]	[1.35]	[1.20]	[1.54]	<LOD	[0.60]
	T3	-	<LOD	<LOD	<LOD	<LOD	[0.64]	5.78	2.59	[1.75]	2.40	[1.82]	[1.02]	[1.78]	[1.43]
	mean	-	<LOD	<LOD	<LOD	<LOD	[0.21]	3.19	2.77	2.04	1.99	[1.65]	[1.58]	[1.33]	[1.08]
40-50	T1	-	<LOD	-	-	<LOD	<LOD	[0.73]	2.50	[1.61]	[1.20]	[0.87]	[0.86]	[0.78]	<LOD
	T2	-	<LOD	-	-	<LOD	<LOD	2.42	[1.88]	<LOD	[1.29]	[1.00]	[0.91]	<LOD	<LOD
	T3	-	<LOD	-	-	<LOD	<LOD	3.69	2.62	[1.51]	[1.62]	[1.42]	[0.98]	<LOD	<LOD
	mean	-	<LOD	-	-	<LOD	<LOD	2.28	2.33	[1.04]	[1.37]	[1.10]	[0.92]	[0.26]	<LOD
50-60	T1	-	<LOD	-	-	<LOD	<LOD	[0.61]	2.10	[1.56]	[1.39]	[0.78]	[0.83]	[0.72]	<LOD
	T2	-	<LOD	-	-	<LOD	<LOD	2.06	[1.85]	[0.97]	[1.52]	[0.72]	[0.86]	<LOD	<LOD
	T3	-	<LOD	-	-	<LOD	[0.99]	[1.88]	[1.86]	[1.37]	[1.57]	[1.07]	[0.89]	<LOD	<LOD
	mean	-	<LOD	-	-	<LOD	[0.33]	[1.52]	1.94	[1.30]	[1.49]	[0.86]	[0.86]	[0.24]	<LOD

Depth [cm]	Sub plot	DAT													
		0	7	15	21	28	63	121	171	259	395	436	518	605	701
60-70	T1	-	-	-	-	-	-	<LOD	[1.39]	[1.55]	[1.34]	[0.93]	[0.92]	[0.77]	<LOD
	T2	-	-	-	-	-	-	2.32	[0.97]	<LOD	[1.13]	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	<LOD	[1.95]	[1.52]	[1.58]	[1.69]	[1.17]	[1.37]	<LOD	<LOD
	mean	-	-	-	-	-	<LOD	[1.42]	[1.29]	[1.04]	[1.39]	[0.70]	[0.76]	[0.26]	<LOD
70-80	T1	-	-	-	-	-	-	<LOD	[1.31]	[1.20]	[0.99]	<LOD	[0.84]	<LOD	<LOD
	T2	-	-	-	-	-	-	[1.67]	<LOD	<LOD	<LOD	<LOD	[0.60]	<LOD	<LOD
	T3	-	-	-	-	-	-	[0.95]	<LOD	[1.32]	[1.31]	[1.09]	[0.78]	<LOD	<LOD
	mean	-	-	-	-	-	-	[0.87]	[0.44]	[0.84]	[0.77]	[0.36]	[0.74]	<LOD	<LOD
80-90	T1	-	-	-	-	-	-	<LOD	[1.11]	[1.03]	[0.63]	<LOD	[0.75]	[1.04]	<LOD
	T2	-	-	-	-	-	-	[1.18]	<LOD	<LOD	<LOD	<LOD	[0.62]	<LOD	<LOD
	T3	-	-	-	-	-	[1.01]	-	-	-	-	-	-	-	-
	mean	-	-	-	-	-	[0.34]	[0.39]	[0.37]	[0.34]	[0.21]	<LOD	[0.46]	[0.35]	<LOD
90-100	T1	-	-	-	-	-	-	<LOD	[0.70]	[0.72]	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	[0.63]	<LOD	<LOD
	T3	-	-	-	-	-	-	[0.78]	<LOD	[0.74]	[1.21]	[0.76]	[0.61]	<LOD	<LOD
	mean	-	-	-	-	-	-	[0.26]	[0.23]	[0.49]	[0.40]	[0.25]	[0.41]	<LOD	<LOD
100-110	T1	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	[0.57]	<LOD	<LOD
	T3	-	-	-	-	-	-	[0.93]	<LOD	[0.65]	[0.86]	[0.63]	[0.83]	<LOD	<LOD
	mean	-	-	-	-	-	-	[0.31]	<LOD	[0.22]	[0.29]	[0.21]	[0.47]	<LOD	<LOD
Sum	T1	98.9	86.3	133	114	139	83.5	32.4	49.6	41.0	28.3	20.7	[19.5]	[15.6]	[10.0]
	T2	131	121	129	180	107	89.6	50.6	35.9	24.5	25.1	[18.1]	22.1	[8.85]	[11.3]
	T3	94.7	107	107	143	135	90.6	67.6	49.1	34.8	38.1	29.7	[20.1]	[15.8]	[13.2]
	mean	108	105	123	146	127	87.9	50.2	44.9	33.4	30.5	22.8	[20.6]	[13.4]	[11.5]

LOD = 0.3 µg/kg equivalent to *ca.* 0.6 g/ha depending on soil moisture and density
LOQ = 1.0 µg/kg equivalent to *ca.* 1.9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ

Table 7.1.2.2.1- 107: Residues of M-01 (AE C653711) in different soil depths from the Great Chishill trial after an application of 100 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	7	15	22	29	68	135	204	307	400	470	558	670	744
0-10	T1	46.4	74.5	65.6	81.7	83.7	64	31.4	38.7	36.5	30.9	26.5	22.7	23.3	17.2
	T2	62.3	80.5	67.7	75.9	67.3	45.8	16.6	12.1	12.9	11.5	7.25	9.33	7.71	7.72
	T3	70.8	72.2	64.2	68.2	69.1	33.8	33.7	27.9	29.4	23.5	29.1	32.9	25.5	17.2
	mean	59.8	75.7	65.8	75.3	73.4	54.5	27.2	26.2	26.3	22.0	21.0	21.6	18.8	14.0
10-20	T1	-	[0.98]	[0.81]	[1.02]	[1.11]	11.7	8.48	13.1	10.6	11.1	10.8	9.95	11.0	11.1
	T2	-	<LOD	<LOD	<LOD	<LOD	19.3	9.8	10.1	8.23	6.36	6.8	5.22	6.00	5.61
	T3	-	[0.87]	1.89	[1.46]	[1.62]	5.57	7.98	8.55	6.19	6.85	8.12	9.76	10.3	6.98
	mean	-	[0.62]	[0.90]	[0.83]	[0.91]	12.19	8.75	10.58	8.01	8.10	8.57	8.31	9.10	7.90
20-30	T1	-	[1.14]	[0.56]	<LOD	<LOD	2.06	2.67	4.24	4.47	4.5	5.33	3.29	3.96	3.66
	T2	-	<LOD	<LOD	<LOD	<LOD	4.66	2.23	5.43	5.14	3.02	3.07	2.83	3.73	2.91
	T3	-	[0.72]	[0.72]	<LOD	[0.73]	1.87	2.9	2.01	1.99	2.19	3.03	2.59	2.74	2.12
	mean	-	[0.62]	[0.43]	<LOD	[0.24]	2.86	2.60	3.89	3.87	3.24	3.81	2.90	3.48	2.90
30-40	T1	-	<LOD	<LOD	<LOD	<LOD	3.27	1.92	2.65	3.11	2.66	6.86	1.88	1.89	1.97
	T2	-	[0.64]	[0.54]	<LOD	[0.53]	2.41	2.15	2.86	2.24	[1.55]	[1.10]	[1.16]	2.00	[1.19]
	T3	-	<LOD	<LOD	<LOD	<LOD	[1.10]	2.03	[1.39]	[0.99]	[0.85]	[0.56]	[0.67]	[0.99]	<LOD
	mean	-	[0.21]	[0.18]	<LOD	[0.18]	2.26	2.05	2.30	2.11	1.69	2.84	1.24	1.63	1.05
40-50	T1	-	-	-	<LOD	<LOD	[1.16]	[1.69]	[1.33]	2.78	2.01	[1.82]	[1.10]	[1.50]	[1.11]
	T2	-	-	-	<LOD	<LOD	[1.47]	[0.98]	[1.01]	[1.41]	[1.00]	[0.80]	[0.78]	[1.44]	[1.00]
	T3	-	-	-	<LOD	<LOD	<LOD	[0.74]	[1.31]	[0.74]	[0.61]	[0.58]	<LOD	[1.00]	<LOD
	mean	-	-	-	<LOD	<LOD	[0.88]	[1.14]	[1.22]	[1.64]	[1.21]	[1.07]	[0.63]	[1.31]	[0.70]
50-60	T1	-	-	-	<LOD	<LOD	<LOD	[1.77]	[1.32]	[1.76]	2.55	[1.58]	[0.93]	[1.37]	[1.25]
	T2	-	-	-	<LOD	<LOD	[0.70]	<LOD	[0.86]	[1.21]	[0.97]	[0.77]	[0.97]	[1.28]	[0.62]
	T3	-	-	-	<LOD	<LOD	<LOD	<LOD	[1.12]	<LOD	[0.84]	<LOD	<LOD	[0.62]	<LOD
	mean	-	-	-	<LOD	<LOD	[0.23]	[0.59]	[1.10]	[0.99]	[1.45]	[0.78]	[0.63]	[1.09]	[0.62]

Depth [cm]	Sub plot	DAT													
		0	7	15	22	29	68	135	204	307	400	470	558	678	744
60-70	T1	-	-	-	-	-	-	2.32	[1.69]	[1.13]	3.14	[1.87]	[0.89]	[0.82]	[1.45]
	T2	-	-	-	-	-	[1.55]	-	[0.86]	[1.41]	[1.19]	<LOD	[1.19]	[1.38]	<LOD
	T3	-	-	-	-	-	-	-	[0.85]	[0.84]	<LOD	[0.72]	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	[0.52]	[0.77]	[1.13]	[1.13]	[1.44]	[0.86]	[0.69]	[0.73]	[0.48]
70-80	T1	-	-	-	-	-	-	[0.98]	[0.77]	<LOD	3.11	[0.71]	<LOD	[0.58]	[0.79]
	T2	-	-	-	-	-	[2.77]	-	<LOD	[1.18]	<LOD	[0.64]	[0.90]	[1.08]	<LOD
	T3	-	-	-	-	-	-	-	[0.72]	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	[0.92]	[0.33]	[0.50]	[0.39]	[1.04]	[0.45]	[0.30]	[0.55]	[0.26]
80-90	T1	-	-	-	-	-	-	[1.43]	<LOD	<LOD	[1.69]	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	-	[0.73]	[0.97]	<LOD	[0.71]	<LOD	[1.13]	[0.86]
	T3	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	[0.48]	[0.24]	[0.32]	[0.56]	[0.24]	<LOD	[0.38]	[0.29]
90-100	T1	-	-	-	-	-	-	3.52	[1.21]	[0.95]	[1.17]	[1.64]	[0.73]	[1.05]	[1.27]
	T2	-	-	-	-	-	-	-	[1.41]	[1.38]	[1.89]	2.68	[1.27]	2.41	2.41
	T3	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	[1.17]	[0.87]	[0.78]	[1.02]	[1.44]	[0.67]	[1.15]	[1.23]
100-110	T1	-	-	-	-	-	-	5.38	[1.97]	[1.22]	[1.65]	3.02	[1.52]	[1.37]	2.86
	T2	-	-	-	-	-	-	-	2.00	2.42	4.58	6.19	2.60	3.73	4.18
	T3	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[0.89]
	mean	-	-	-	-	-	-	[1.79]	[1.32]	[1.21]	2.08	3.07	[1.37]	[1.70]	2.64
Sum	T1	46.4	76.6	66.4	82.7	84.8	82.2	61.4	67.0	62.5	64.5	60.0	42.8	46.8	42.7
	T2	62.3	81.1	68.2	75.9	67.8	78.7	31.8	37.4	38.2	32.1	30.0	26.3	31.9	25.9
	T3	70.8	73.8	66.8	69.7	71.2	62.3	47.2	43.9	38.2	34.8	42.1	45.9	40.5	27.2
	mean	59.8	77.2	67.1	76.1	74.6	74.4	46.8	49.4	46.3	43.8	44.0	38.3	39.7	31.9

LOD = 0.3 µg/kg equivalent to *ca.* 0.6 g/ha depending on soil moisture and density
LOQ = 1.0 µg/kg equivalent to *ca.* 1.9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ

The dissipation of M-01 with time is presented Table 7.1.2.2.1- 108 and Table 7.1.2.2.1- 109. The values have been pre-processed according to the procedure described in FOCUS kinetics guidance (as described earlier). Actual values are given in brackets.

At Burscheid (Germany), the mean amount of M-01 at day 0 was 108 g/ha, representing 108% of the nominal application rate. M-01 declined from 104 g/ha in soil at day 0 to 11.5 g/ha at day 701. At Great Chishill (United Kingdom), the mean amount of M-01 at day 0 was 59.8 g/ha, representing 60% of the nominal application rate. M-01 declined from 59.8 g/ha in soil at day 0 to 31.9 g/ha at day 744.

The dissipation of M-01 showed a biphasic behaviour in both trials. After treatment, M-01 dissipated in a first step faster followed by a second more slowly step until study termination. Residues of M-01 in control samples were < LOD for all samples taken.

Table 7.1.2.2.1- 108: Residues of M-01 (AE C653711) in soil from the Burscheid trial after an application of 100 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Burscheid (Germany)	DAT													
	0	**7**	**15**	**21**	**28**	**63**	**121**	**171**	**259**	**395**	**436**	**518**	**605**	**701**
T1	98.9 (98.9)	87.5 (86.3)	134 (133)	115 (114)	140 (139)	85.5 (83.5)	35.0 (32.4)	59.9 (49.6)	41.6 (41.0)	28.9 (28.3)	22.0 (20.7)	20.1 (19.5)	17.6 (15.6)	12.5 (10.0)
T2	131 (131)	122 (121)	130 (129)	180 (180)	108 (107)	91.5 (89.5)	51.4 (50.6)	37.1 (32.9)	25.8 (24.5)	26.0 (25.1)	21.4 (18.1)	22.8 (22.1)	13.1 (8.85)	11.9 (11.3)
T3	94.7 (94.7)	108 (107)	108 (107)	144 (143)	137 (135)	92.5 (90.6)	67.6 (67.6)	51.6 (49.1)	34.8 (34.8)	38.3 (38.1)	30.6 (29.7)	21.5 (20.1)	18.8 (15.8)	13.8 (13.2)
Mean	**108 (108)**	**106 (105)**	**124 (123)**	**146 (145)**	**128 (127)**	**89.8 (87.9)**	**51.3 (50.2)**	**46.5 (44.8)**	**34.1 (33.4)**	**31.1 (30.5)**	**24.7 (22.8)**	**21.5 (20.5)**	**16.5 (13.4)**	**12.7 (11.5)**
Min	94.7 (94.7)	87.5 (86.3)	108 (107)	115 (114)	108 (107)	85.5 (83.5)	35.0 (32.4)	37.1 (35.9)	25.8 (24.5)	26.0 (25.1)	21.4 (18.1)	20.1 (19.5)	13.1 (8.85)	11.9 (11.3)
Max	131 (131)	122 (121)	134 (133)	180 (180)	140 (139)	92.5 (90.6)	67.6 (67.6)	51.6 (49.6)	41.6 (41.0)	38.3 (38.1)	30.6 (29.7)	22.8 (22.1)	18.8 (15.8)	13.8 (13.2)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	98 (97)	115 (114)	135 (134)	119 (118)	83 (81)	48 (46)	43 (41)	32 (31)	29 (28)	23 (21)	20 (19)	15 (12)	12 (11)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3.
The actual values are given in brackets (expressed as g/ha)

Table 7.1.2.2.1- 109: Residues of M-01 (AE C653711) in soil from the Great Chishill trial after an application of 100 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Great Chishill (UK)	DAT													
	0	**7**	**15**	**22**	**29**	**68**	**135**	**204**	**307**	**400**	**470**	**558**	**670**	**744**
T1	46.6 (46.4)	77.2 (76.6)	67.0 (66.4)	83.8 (82.7)	86.4 (84.8)	82.7 (82.2)	61.6 (61.4)	67.7 (67.0)	63.8 (62.5)	64.5 (64.5)	60.7 (60.0)	44.2 (42.8)	47.4 (46.8)	43.3 (42.7)
T2	62.3 (62.3)	82.2 (81.1)	69.3 (68.2)	76.4 (75.9)	70.7 (67.8)	78.7 (78.7)	32.3 (31.8)	37.9 (37.4)	38.5 (38.2)	33.3 (32.1)	30.7 (30.0)	26.9 (26.3)	31.9 (31.9)	27.8 (25.9)
T3	70.8 (70.8)	74.3 (73.8)	67.3 (66.8)	70.2 (69.7)	72.0 (71.2)	62.9 (62.3)	47.9 (47.2)	44.4 (43.9)	40.3 (38.2)	35.5 (34.8)	43.3 (42.1)	47.7 (45.9)	42.3 (40.5)	29.6 (27.2)
Mean	**59.8 (59.8)**	**77.9 (77.1)**	**67.9 (67.1)**	**76.6 (74.6)**	**76.1 (74.6)**	**74.8 (74.4)**	**47.3 (46.7)**	**50.0 (49.4)**	**47.5 (46.2)**	**44.4 (43.7)**	**44.9 (44.0)**	**39.6 (38.3)**	**40.5 (39.7)**	**33.6 (31.9)**
Min	46.6 (46.4)	74.3 (73.8)	67.0 (66.4)	70.2 (69.7)	70.7 (67.8)	62.9 (62.3)	32.3 (31.8)	37.9 (37.4)	38.5 (38.2)	33.3 (32.1)	30.7 (30.0)	26.9 (26.3)	31.9 (31.9)	27.8 (25.9)
Max	70.8 (70.8)	82.2 (81.1)	69.3 (68.2)	83.8 (82.7)	86.4 (84.8)	82.7 (82.2)	61.6 (61.4)	67.7 (67.0)	63.8 (62.5)	64.5 (64.5)	60.7 (60.0)	47.7 (45.9)	47.4 (46.8)	43.3 (42.7)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	130 (129)	114 (112)	128 (127)	127 (125)	125 (124)	79 (78)	84 (83)	79 (77)	74 (73)	75 (74)	66 (64)	68 (66)	56 (53)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3
The actual values are given in brackets (expressed as g/ha)

III. Conclusion

Under field conditions M-01 declined moderately and residues were translocated up to 90 cm (Burscheid, Germany) and 110 cm (Great Chishill, United Kingdom) depth, whilst 67-100% (Burscheid, Germany) and 55-100% (Great Chishill) of residues remained in the top 0-30 cm at all timepoints.

Un-normalised DT_{50} for the degradation of M-01 calculated from the reported data for the trial at Burscheid, Germany following the recommendations of the FOCUS work group details are provided in Document KCA 7.1.2.2.1/21. According to EFSA (2014) the Great Chishill trial site is not acceptable to determine $DegT_{50}$ values due to significant leaching out of the sampled soil depths.

Assessment and conclusion by applicant:

The study is considered valid to assess M-01 soil $DegT_{50matrix}$ values for field studies as defined by EFSA (2014) at the trial site in Burscheid (Germany). The trial site at Great Chishill is not suitable to assess soil $DegT_{50matrix}$ values as transport of M-01 residues to deeper soil layers occurred early in the study and residues were not fully contained within the sampled soil layers as required by EFSA (2014). The endpoints may be too conservative to assess persistence as the design minimized soil surface processes as required by EFSA (2014) and such processes may contribute to dissipation.

Data Point:	KCA 7.1.2.2.1/19
Report Author:	[redacted]
Report Year:	2019
Report Title:	Amendment no. 1 to final report - Terrestrial field dissipation study with BAM SC 125 in France (North) and France (South)
Report No:	15-2703
Document No:	M-647370-02-1
Guideline(s) followed in study:	Regulation (EC) No. 1107/2009 of the European Parliament and of the Council of 21 October 2009 including Data Requirements SANCO/11803/2010 Rev. 7 and Test Methods SANCO/11843/2010 Rev. 4 EFSA Guidance Document for Evaluating Laboratory and Field Dissipation Studies to obtain DegT50 Values of Active Substances of Plant Protection Products and Transformation Products of these Active Substances in Soil, EFSA Journal 2014; 12(5):3662, 2014
Deviations from current test guideline:	Yes. Report meets the requirement for assessing test substance soil DegT50matrix values as required by EFSA (2014) for field studies. The endpoints may be too conservative for comparison to field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

Soil degradation of M-01 (AE C653711) under both Northern European and Southern European field conditions was investigated after application of M-01 onto bare soil plots in Lignieres-de-Touraine (France) and St Etienne du Gres (France).

BAM SC 125 is a suspension concentrate formulation, containing 125 g/L M-01. The formulation was sprayed once onto 400 sqm plots at a rate of 0.8 L/ha, corresponding to nominal 100 g/ha M-01. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface.

Soil cores were taken 0 days before up to 714 days post-application to a maximum depth of 110 cm. The soil cores were cut into 10-cm soil layers, bulked soil layers were homogenised and finally analysed for M-01.

Sub-samples of homogenised soil were extracted in a microwave extractor with organic solvent. Potential matrix effects were eliminated by using an internal standard solution of isotopically labeled reference items added to sample extracts. Following separation of fine particles from soil extracts by centrifugation the identification and quantitation of the analytes was performed by high performance liquid chromatography using MS/MS detection in the multiple reaction monitoring mode. The analytical method was validated using three different soils. The limit of quantitation (LOQ) was 1.0 µg/kg and the limit of detection (LOD) was 0.3 µg/kg for M-01.

At Lignieres de Touraine (France North), the mean amount of M-01 at day 0 was 86.2 g/ha, representing 86% of the nominal application rate. M-01 declined from 86.2 g/ha in soil at day 0 to 17.7 g/ha at day 699.

At St Etienne du Gres (France South), the mean amount of M-01 at day 0 was 87.9 g/ha, representing 88% of the nominal application rate. M-01 declined from 87.9 g/ha in soil at day 0 to 8.09 g/ha at day 714.

Under field conditions M-01 residues were translocated up to 90 cm (Lignieres de Touraine, France North) and 50 cm (St. Etienne du Gres, France South) depth, respectively. It is concluded there was some mobility of M-01 to deeper soil layers in the Lignieres de Touraine trial but 100% of residues were detected in 0-100 cm soil depth (out of a total measured depth of 110 cm). In contrast M-01 was fully retained in topsoil layers at the St Etienne du Gres site. Consequently, robust $DegT_{50}$ values can be obtained for the compound from data from both trials.

I. Materials and Methods

A. Materials

1. Test Item

AE C653711 (M-01) formulated as a suspension concentrate (125g/L AE C653711)

Certificate of Analysis:	01865-00
Lot No:	2015-000656

2. Trial Location & Soil

A terrestrial field dissipation with M-00 (AE C653711) a suspension concentrate formulation, containing 125 g/L M-01 was conducted at two locations in Northern and Southern France. The two locations were Lignieres-de-Touraine (France, North) and St Etienne du Gres (France, South). The sites were fully characterised, and the results summarised in Table 7.1.2.2.1-110. The plot sizes were 400 sqm. The control plot was prepared at least 5 m away from the treated plots.

Table 7.1.2.2.1- 110: Location, site description and climatic data of test sites

Characteristic	Units	Sampling depth [cm]			
		0-30	**30-50**	**50-75**	**75-100**
Soil Designation	-	**Lignieres de Touraine (France)**			
Soil ID	-	FR09			
Geographic Location City Country	-	[redacted] 37130 Lignieres de Touraine, Central Region France			
pH	$CaCl_2$	5.9	6.5	6.8	6.8
Organic carbon	[% Carbon]	0.8	0.4	0.3	0.5
CEC	[meq/100 g]	12.2	13.2	14.7	21.8
Chalk	[% $CaCO_3$]	0.2	0.3	0.3	0.0
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 15 15 70	 15 19 66	 19 25 56	 37 33 30
Textural class	USDA	sandy loam	sandy loam	sandy loam	clay loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 44.4 17.5	 47.7 20.6	 52.3 26.1	 61.5 33.1
Soil Designation	-	**St. Etienne du Grès (France)**			
Soil ID	-	FR08			
Geographic Location City Country		[redacted] 13703 St. Etienne du Grès, Provence-Alpes-Côte d'Azur France			
pH	$CaCl_2$	7.7	7.7	7.8	7.8
Organic carbon	[% Carbon]	0.8	0.6	0.6	0.4
CEC	[meq/100 g]	11.7	11.7	13.2	14.0
Chalk	[% $CaCO_3$]	42.1	42.1	42.5	43.7
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 29 45 26	 31 47 22	 39 41 20	 43 39 18
Textural class	USDA	clay loam	clay loam	clay loam	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 46.2 25.5	 43.4 26.8	 48.3 25.3	 51.4 25.0

B. Study Design

1. Experimental Conditions

BAM SC 125 is a suspension concentrate formulation, containing 125 g/L M-01 (AE C653711). The product was sprayed onto bare earth once at each site at an application rate of 0.8 L/ha and 600 L/ha water, corresponding to 100 g/ha of M-01 during May 2015. Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of a nonselective herbicide to control weeds.

Air temperature, precipitation including irrigation and sunshine data were recorded on site during the field tests.

Soil dissipation of M-01 was studied for up to 714 days.

2. Sampling

The treated plot of the trial was divided into three sub-plots. From each sub-plot 10 soil cores were taken and combined together at each sampling interval (30 cores in total).

Samples were taken on the following occasions: 0 (post-application; each 0-10 cm depth), 7, 14 (each 0-60 cm depth), 21, 28, 56-58 (each 0-85 cm depth), and 118-121, 166-167, 277-280, 348-352, 438-450, 519-531, 646-649, 699-714 (each 0-110 cm depth) after treatment. From the control plot samples were taken on the following occasions: 0 days before application, 699 and 714 days after application.

Soil cores taken from the three sites were deep frozen to -18°C within twenty four hours after sampling, then shipped frozen to the analytical laboratory in Germany.

3. Analytical Procedures

The analytical method 01445 was used to determine levels of M-01 (AE C653711). Soil samples of 5 g were extracted in a microwave extractor with a mixture of acetonitrile/water (4/1, v/v). The extracts were centrifuged to remove fine particles of the soil. Possible matrix effects of M-01 were eliminated by using an internal standard solution of isotopic labelled reference items. Quantification was carried out by LC-MS/MS. The limit of quantitation (LOQ) for M-01 was 1.0 µg/kg in soil. The limit of determination (LOD) for M-01 was 0.3 µg/kg.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item M-01 at levels of LOQ, 10 x LOQ and 500 x LOQ and processed in parallel to the dissipation samples throughout the study. The results are summarised in the table below.

Single Values [%]	No of Recoveries	Fortification Level [µg/kg]	Mean [%]	RSD [%]
61, 77, 77, 77, 79, 79, 82, 82, 83, 83, 84, 84, 84, 85, 85, 85, 85, 85, 85, 86, 86, 87, 87, 88, 88, 88, 88, 89, 89, 90, 90, 90, 90, 90, 91, 91, 91, 91, 91, 91, 92, 92, 92, 93, 93, 93, 93, 94, 94, 94, 94, 94, 95, 95, 95, 95, 95, 96, 96, 96, 96, 96, 97, 97, 97, 98, 98, 98, 98, 98, 99, 99, 99, 99, 99, 99, 99, 99, 99, 99, 100, 100, 100, 100, 100, 101, 101, 101, 101, 102, 102, 102, 102, 102, 102, 102, 102, 102, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 105, 105, 105, 106, 106, 106, 107, 107, 107, 108, 108, 108, 109, 109, 109, 110, 110, 110, 110, 110, 111, 111, 111, 112, 112, 112, 112, 113, 113, 113, 113, 113, 113, 114, 114, 115, 115, 115, 115, 115, 116, 116, 116, 117, 118, 119, 119, 119, 119, 120, 120	169	1.0	100	10.7

71, 81, 88, 88, 90, 90, 90, 91, 91, 92, 92, 92, 92, 94, 94, 94, 94, 95, 96, 97, 97, 97, 97, 97, 97, 97, 98, 98, 98, 98, 99, 99, 99, 99, 99, 99, 99, 100, 100, 100, 100, 100, 100, 100, 100, 101, 101, 101, 101, 101, 101, 101, 101, 102, 102, 102, 102, 102, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 105, 105, 106, 106, 106, 106, 106, 106, 106, 106, 106, 106, 106, 106, 107, 107, 107, 107, 107, 107, 108, 108, 108, 108, 108, 108, 108, 109, 109, 109, 109, 109, 109, 109, 109, 109, 109, 110, 110, 110, 110, 110, 110, 111, 111, 111, 111, 111, 111, 112, 112, 112, 112, 112, 112, 112, 112, 112, 112, 112, 113, 113, 113, 113, 113, 114, 114, 114, 114, 114, 114, 114, 115, 115, 115, 115, 115, 115, 115, 115, 116, 117, 118, 118, 118, 118, 118, 118, 119, 119, 119, 120, 120	176	10	105	7.6
101	1	300	-	-
Overall recovery			103	9.5

RSD = Relative standard deviation

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

4. Evaluation of the Data and Kinetic Calculations

For evaluation of degradation kinetics of the test item according to the FOCUS guidance document on degradation kinetics, the total residue of the test item in the soil profile covering all soil horizons was calculated according to the following procedure:

- values between LOD and LOQ were set to the measured values.
- values < LOD were set to 0.5 LOD for samples after, before or deeper as a value > LOD or for samples between (> LOD and <LOQ). The curve was cut off after the first non-detect (< LOD), if no later value >LOQ followed.
- at day 0, values < LOD in deep horizons were set to 0.

The results in [µg/kg] were converted to [g/ha] considering the actual soil density of the corresponding soil layer.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of M-01 in soil samples by HPLC-MS/MS.

B. Data:

The results for residues of M-01 in different soil depths are presented below (expressed as g/ha) in Table 7.1.2.2.-111 to Table 7.1.2.2.1- 112.

At the Lignieres de Touraine (Northern France) trial site, residues of M-01 were detected at concentration > LOQ at soil depths down to 90 cm. The metabolite was detected in the 0-10 cm throughout the trial and from DAT 21 in the 10-20 cm and 20-30 cm soil horizons. The maximum residue level in the 10-20 cm horizon was observed at DAT 120 at a concentration of 21.7 g/ha and in the 20-30 cm horizon at DAT 277 at a concentration of 12.4 g/ha. In samples taken from DAT 120 to DAT 699 low residues were detected in the underlying 30-40 cm, 40-50 cm, 50-60 cm and 60-70 cm horizons at concentrations ranging from <LOD to 9.94 g/ha. In 60-70 cm, 70-80 cm and 80-90 cm soil layers residues > LOQ were detected at DAT 166 to DAT 438 only. In the next layer (90-100 cm) no residues above the LOQ were detected and in the deepest soil layer (100-110 cm) no residues above the LOD were detected

It is concluded there was some mobility of M-01 to deeper soil layers but 100% of residues were detected in 0-100 cm soil depth (out of a total measured depth of 110 cm) and consequently robust $DegT_{50}$ values can be obtained for the compound from data from the Lignieres de Touraine trial.

At the St Etienne du Gres (France, South) trial site, residues of M-01 were detected at concentrations > LOQ at soil depths down to 30 cm. The metabolite was detected in the 0-10 cm and 10-20 cm soil horizons throughout the St Etienne du Gres trial. The maximum residue level in the 10-20 cm horizon was 10.0 g/ha at DAT 352. Low residues were detected in the 20-30 cm soil layer at concentrations from <LOQ to 3.42 g/ha and in the 30-40 cm soil layer at concentrations <LOQ from DAT 58. No residues > LOD were detected in deeper soil layers with the exception of one finding < LOQ in the 30-40 cm soil layer at DAT 352.

It is concluded M-01 was fully retained in topsoil layers and consequently robust $DegT_{50}$ values can be obtained for the compound from data from the St Etienne du Gres trial.

Table 7.1.2.2.1- 111: Residues of M-01 (AE C653711) in different soil depths from the Lignieres de Touraine (Northern France) trial after an application of 100 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	7	14	21	28	56	120	166	277	348	438	531	649	699
0-10	T1	87.6	71.9	74.5	74.3	77.0	63.1	23.2	19.1	5.22	5.62	4.75	4.55	2.14	2.15
	T2	83.8	73.9	66.7	71.7	63.9	60.9	21.8	16.5	6.44	5.39	5.16	3.54	2.84	1.94
	T3	87.2	72.0	64.9	74.6	59.1	55.4	23.9	17.0	6.45	5.58	5.04	4.46	2.15	2.24
	mean	86.2	72.6	68.7	73.5	66.7	61.8	23.0	17.5	6.04	5.53	4.98	4.85	2.38	2.11
10-20	T1	-	<LOD	<LOD	3.56	3.31	[1.72]	21.7	21.0	9.84	10.1	5.57	5.18	3.74	4.01
	T2	-	<LOD	<LOD	4.06	2.73	[1.20]	14.2	18.7	9.95	11.0	5.44	6.42	4.80	4.25
	T3	-	<LOD	<LOD	<LOD	[0.99]	[0.98]	12.3	16.3	9.05	8.93	5.63	4.14	4.14	3.88
	mean	-	<LOD	<LOD	2.54	2.34	[1.30]	16.1	18.7	9.61	10.0	5.55	5.25	4.23	4.05
20-30	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	7.86	9.57	12.4	9.74	4.67	4.24	3.42	3.11
	T2	-	<LOD	<LOD	[1.63]	[0.74]	<LOD	6.66	8.58	7.82	7.48	4.66	4.29	4.09	4.13
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	6.39	7.18	4.93	4.98	3.53	2.26	2.72	3.13
	mean	-	<LOD	<LOD	[0.54]	[0.25]	<LOD	6.77	8.44	8.38	7.40	4.29	3.60	3.41	3.46
30-40	T1	-	-	-	-	-	-	4.16	4.53	9.94	6.98	4.07	2.27	2.24	2.46
	T2	-	-	-	<LOD	<LOD	-	4.13	4.85	5.00	5.04	3.32	2.86	2.63	2.98
	T3	-	-	-	-	-	-	5.03	7.62	3.59	3.31	2.09	[0.95]	[1.43]	[1.58]
	mean	-	-	-	<LOD	<LOD	-	4.44	5.67	6.18	5.11	3.16	2.03	2.10	2.34
40-50	T1	-	-	-	-	-	-	2.80	2.63	7.64	6.42	3.86	2.20	[1.95]	2.26
	T2	-	-	-	<LOD	<LOD	-	2.66	3.20	4.84	5.15	3.43	2.42	2.06	2.57
	T3	-	-	-	-	-	-	3.80	8.66	4.86	4.57	1.95	[0.90]	[1.08]	[1.24]
	mean	-	-	-	<LOD	<LOD	-	3.09	4.83	5.78	5.38	3.08	[1.84]	[1.70]	2.02
50-60	T1	-	-	-	-	-	-	<LOD	[0.96]	4.84	4.25	2.75	[1.40]	2.00	[1.37]
	T2	-	-	-	<LOD	<LOD	-	[1.66]	[1.41]	3.77	3.81	3.28	2.40	[1.84]	1.94
	T3	-	-	-	-	-	-	1.74	6.80	5.41	5.06	2.41	[1.45]	[0.99]	[1.12]
	mean	-	-	-	<LOD	<LOD	-	[1.13]	3.06	4.67	4.37	2.81	[1.75]	[1.61]	[1.48]

Depth [cm]	Sub plot	DAT													
		0	7	14	21	28	56	120	166	277	348	438	531	649	699
60-70	T1	-	-	-	-	-	-	-	<LOD	2.63	2.65	[1.66]	<LOD	[1.24]	[1.00]
	T2	-	-	-	-	-	-	<LOD	[0.93]	2.58	3.20	3.78	2.61	[1.51]	[1.98]
	T3	-	-	-	-	-	-	[0.91]	2.53	3.50	5.19	3.69	2.04	[1.18]	[1.69]
	mean	-	-	-	-	-	-	[0.30]	[1.15]	2.90	3.68	3.03	[1.55]	[1.31]	[1.56]
70-80	T1	-	-	-	-	-	-	-	<LOD	2.20	2.11	[0.89]	[0.55]	<LOD	[0.62]
	T2	-	-	-	-	-	-	<LOD	<LOD	[1.25]	[1.33]	[1.43]	[0.90]	<LOD	[0.71]
	T3	-	-	-	-	-	-	[0.75]	[1.71]	2.08	2.44	2.36	[1.61]	<LOD	[0.80]
	mean	-	-	-	-	-	-	[0.25]	[0.57]	[1.84]	1.96	[1.56]	[1.02]	<LOD	[0.71]
80-90	T1	-	-	-	-	-	-	-	<LOD	[1.05]	[1.02]	<LOD	[0.53]	<LOD	<LOD
	T2	-	-	-	-	-	-	<LOD	<LOD	[0.79]	[0.89]	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	[0.82]	1.79	[1.67]	[1.14]	[0.98]	[0.94]	<LOD	[0.56]
	mean	-	-	-	-	-	-	[0.27]	[0.60]	[1.14]	[1.02]	[0.33]	[0.49]	<LOD	[0.19]
90-100	T1	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	<LOD	[0.77]	[0.75]	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	[0.97]	[1.19]	[0.86]	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	[0.32]	[0.65]	[0.54]	<LOD	<LOD	<LOD	<LOD	<LOD
100-110	T1	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
Sum	T1	87.6	71.9	74.5	77.9	80.3	64.8	59.7	57.8	55.8	48.9	28.2	[20.9]	[16.7]	[17.0]
	T2	83.8	73.9	66.7	77.4	67.2	68.1	50.5	54.9	43.2	43.3	30.5	27.4	[19.8]	[20.5]
	T3	87.2	72	64.9	74.6	60.1	56.4	56.5	70.8	42.1	41.2	27.7	[18.8]	[13.7]	[15.7]
	mean	86.2	72.6	68.7	76.6	69.2	63.1	55.6	61.2	47.0	44.5	28.8	22.4	[16.]7	[17.7]

LOD = 0.3 µg/kg equivalent to *ca.* 0.6 g/ha depending on soil moisture and density
LOQ = 1.0 µg/kg equivalent to ca. 1.9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ

Table 7.1.2.2.1- 112: Residues of M-01 (AE C653711) in different soil depths from the St Etienne du Gres (France, South) trial after an application of 100 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	7	14	21	28	58	118	167	280	352	450	519	646	714
0-10	T1	89.6	78.3	67.8	68.7	69.9	50.9	27.8	29.6	24.2	18.7	9.54	6.19	5.55	4.25
	T2	78.2	77.9	72.7	70.9	79.6	59.5	38.8	26.7	22.0	22.7	14.2	6.77	3.77	4.46
	T3	95.9	74.1	82.8	79.2	73.0	56.7	39.1	35.4	41.3	29.4	12.6	6.39	6.13	4.87
	mean	87.9	76.8	74.4	72.9	74.2	55.7	35.2	30.6	29.2	23.6	12.1	6.45	5.15	4.53
10-20	T1	-	2.45	2.30	[1.06]	2.30	7.70	2.83	3.46	3.17	6.58	2.78	3.31	2.59	[1.30]
	T2	-	2.59	2.36	2.89	3.50	5.62	3.08	5.32	5.09	7.28	5.21	2.45	[1.34]	2.36
	T3	-	[1.68]	<LOD	[1.87]	[0.89]	5.94	2.08	4.10	5.45	10.0	4.35	3.04	[1.34]	[1.58]
	mean	-	2.24	[1.55]	1.94	2.23	6.42	2.66	4.29	4.57	7.95	4.11	2.93	[1.76]	[1.75]
20-30	T1	-	<LOD	<LOD	<LOD	<LOD	2.19	[1.32]	1.81	[1.14]	2.92	[1.25]	[1.67]	[1.37]	[0.78]
	T2	-	<LOD	<LOD	<LOD	<LOD	[1.22]	[1.25]	2.59	2.03	3.42	2.88	[1.66]	[1.20]	[1.54]
	T3	-	<LOD	<LOD	<LOD	<LOD	[1.57]	[0.83]	[1.44]	[1.37]	2.71	1.95	1.85	[0.68]	[1.31]
	mean	-	<LOD	<LOD	<LOD	<LOD	[1.66]	[1.13]	1.95	[1.51]	3.02	2.03	[1.73]	[1.08]	[1.21]
30-40	T1	-	-	-	-	-	[1.25]	<LOD	[0.99]	<LOD	[0.83]	<LOD	[0.73]	<LOD	<LOD
	T2	-	-	-	-	-	[0.74]	<LOD	[1.53]	[1.08]	[1.59]	[0.91]	[1.05]	<LOD	[1.00]
	T3	-	-	-	-	-	<LOD	<LOD	[0.78]	<LOD	[0.73]	<LOD	[0.79]	<LOD	[0.86]
	mean	-	-	-	-	-	[0.66]	<LOD	[1.10]	[0.36]	[1.05]	[0.30]	[0.86]	<LOD	[0.62]
40-50	T1	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	[0.72]	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	[0.24]	<LOD	<LOD	<LOD	<LOD
50-60	T1	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Depth [cm]	Sub plot	DAT													
		0	7	14	21	28	58	118	167	280	352	450	519	646	714
60-70	T1	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T3	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
70-80	T1	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T3	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
80-90	T1	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T3	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
90-100	T1	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T3	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
100-110	T1	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T2	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	T3	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
	mean	-	-	-	-	-	-	-	-	-	<LOD	-	-	-	-
Sum	T1	89.6	80.8	70.1	69.6	72.2	62.0	32.0	35.7	28.5	28.9	13.6	11.9	9.51	[6.33]
	T2	78.2	80.5	75.1	75.8	83.1	67.1	43.1	36.1	30.2	35.7	23.2	11.9	[6.31]	[9.36]
	T3	95.9	75.8	82.8	81.1	73.9	64.2	42.0	41.7	48.1	42.8	18.9	12.1	[8.15]	[8.62]
	mean	87.9	79.0	76.0	74.8	76.4	64.4	39.0	37.8	35.6	35.8	18.6	12.0	[7.99]	[8.10]

LOD = 0.3 µg/kg equivalent to *ca.* 0.6 g/ha depending on soil moisture and density
LOQ = 1.0 µg/kg equivalent to *ca.* 1.9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ

The dissipation of M-01 with time is presented in Table 7.1.2.2.1- 113 and Table 7.1.2.2.1- 114. The values have been pre-processed according to the procedure described in FOCUS kinetics guidance (as described earlier). Actual values are given in brackets.

At Lignieres de Touraine (France North), the mean amount of M-01 at day 0 was 86.2 g/ha, representing 86% of the nominal application rate. M-01 declined from 86.2 g/ha in soil at day 0 to 17.7 g/ha at day 699. At St. Etienne du Gres (France South), the mean amount of M-01 at day 0 was 87.9 g/ha, representing 88% of the nominal application rate. M-01 declined from 87.9 g/ha in soil at day 0 to 8.09 g/ha at day 714. The dissipation of M-01 showed a biphasic behaviour in both trials. After treatment, M-01 dissipated initially very rapidly within a couple of days followed by a second slower rate until study termination.

Table 7.1.2.2.1- 113: Residues of M-01 (AE C653711) in soil from the Lignieres de Touraine (Northern France) trial after an application of 100 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Lignieres de Touraine (N France)	DAT													
	0	7	14	21	28	56	120	166	277	348	438	531	649	699
T1	87.6 (87.6)	72.5 (71.9)	75.1 (74.5)	78.4 (77.9)	80.9 (80.3)	65.4 (64.8)	60.3 (59.7)	59.5 (57.8)	56.3 (55.8)	49.4 (48.9)	28.7 (28.2)	21.5 (20.9)	17.3 (16.7)	17.5 (17.0)
T2	83.8 (83.8)	74.5 (73.9)	67.8 (66.7)	77.9 (77.4)	67.9 (67.2)	68.7 (68.1)	51.6 (50.5)	56.6 (54.9)	43.8 (43.2)	43.8 (43.3)	31.0 (30.5)	28.0 (27.4)	20.3 (19.8)	21.0 (20.5)
T3	87.2 (87.2)	72.6 (72.0)	65.5 (64.9)	75.2 (74.6)	60.7 (60.1)	56.9 (56.4)	57.2 (56.5)	71.3 (70.8)	42.9 (42.4)	41.7 (41.2)	28.2 (27.7)	19.3 (18.8)	14.8 (13.7)	16.8 (15.7)
Mean	86.2 (86.2)	73.2 (72.6)	69.5 (68.7)	77.2 (76.6)	69.8 (69.2)	63.7 (63.1)	56.4 (55.5)	62.5 (61.1)	47.7 (47.0)	45.0 (44.4)	29.3 (28.8)	22.9 (22.3)	17.5 (16.7)	18.4 (17.7)
Min	83.8 (83.8)	72.5 (71.9)	65.5 (64.9)	75.2 (74.6)	60.7 (60.1)	56.9 (56.4)	51.6 (50.4)	56.6 (54.9)	42.9 (42.0)	41.7 (41.2)	28.2 (27.7)	19.3 (18.7)	14.8 (13.7)	16.8 (15.7)
Max	87.6 (87.6)	74.5 (73.9)	75.1 (74.5)	78.4 (77.8)	80.9 (80.3)	68.7 (68.1)	60.3 (59.7)	71.3 (70.7)	56.3 (55.7)	49.4 (48.8)	31.0 (30.4)	28.0 (27.4)	20.3 (19.7)	21.0 (20.5)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	85 (84)	81 (80)	90 (89)	81 (80)	74 (73)	65 (64)	73 (71)	55 (55)	52 (52)	34 (33)	27 (26)	20 (19)	21 (21)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3.
The actual values are given in brackets (expressed as g/ha)

Table 7.1.2.2.1- 114: Residues of M-01 (AE C653711) in soil from the St Etienne du Gres (France, South) trial after an application of 100 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

St Etienne du Gres (S. France)	DAT													
	0	**7**	**14**	**21**	**28**	**58**	**118**	**167**	**280**	**352**	**450**	**519**	**646**	**714**
T1	89.6 (89.6)	81.3 (80.8)	70.6 (70.1)	70.3 (69.6)	72.7 (72.2)	62.6 (62.0)	32.5 (32)	36.4 (35.7)	29.0 (28.5)	29.6 (28.9)	14.1 (13.6)	12.5 (11.9)	10.1 (9.51)	6.85 (6.33)
T2	78.2 (78.2)	81.0 (80.5)	75.6 (75.1)	74.3 (73.8)	83.6 (83.1)	67.6 (67.1)	43.6 (43.1)	36.7 (36.1)	30.8 (30.2)	36.3 (35.7)	23.7 (23.2)	12.5 (11.9)	6.87 (6.31)	9.9 (9.36)
T3	95.9 (95.9)	76.3 (75.8)	83.4 (82.8)	81.6 (81.1)	74.4 (73.9)	64.7 (64.2)	42.5 (42.0)	42.3 (41.7)	48.7 (48.1)	43.4 (42.8)	19.1 (18.9)	12.6 (12.1)	8.71 (8.15)	9.17 (8.62)
Mean	**87.9 (87.9)**	**79.5 (79.0)**	**76.5 (76.0)**	**75.4 (74.8)**	**76.9 (76.4)**	**65.0 (64.4)**	**39.5 (39.0)**	**38.5 (37.8)**	**36.2 (35.6)**	**36.4 (35.8)**	**19.1 (18.6)**	**12.5 (12.0)**	**8.56 (7.98)**	**8.64 (8.09)**
Min	78.2 (78.2)	76.3 (75.7)	70.6 (70.1)	70.3 (69.6)	72.7 (72.2)	62.6 (62.0)	32.5 (31.9)	36.4 (35.9)	29.0 (28.5)	29.6 (29.0)	14.1 (13.6)	12.5 (11.9)	6.87 (6.31)	6.85 (6.33)
Max	95.9 (95.9)	81.3 (80.8)	83.4 (82.8)	81.6 (81.0)	83.6 (83.1)	67.6 (67.1)	43.6 (43.1)	42.3 (41.7)	48.7 (48.1)	43.4 (42.8)	23.7 (23.2)	12.6 (12.1)	10.1 (9.5)	9.9 (9.35)
n	3	3	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	90 (90)	87 (86)	86 (85)	87 (87)	74 (73)	45 (44)	44 (43)	41 (41)	41 (41)	22 (21)	14 (14)	10 (9)	10 (9)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3.
The actual values are given in brackets (expressed as g/ha).

III. Conclusion

Under field conditions M-01 declined moderately with residues translocated up to 50 cm (St. Etienne du Gres, France South) and 90 cm (Lignieres de Touraine, France North) depth, whilst 89-100% (St. Etienne du Gres, France South) and 47-100% (Lignieres de Touraine, France North) of residues remained in the top 0-30 cm at all timepoints. Un-normalised DT_{50} for the degradation of M-01 calculated from the reported data following the recommendations of the FOCUS work group details are provided in Document KCA 7.1.2.2.1/21.

Assessment and conclusion by applicant:

The study is considered valid to assess M-01 soil $DegT_{50matrix}$ values for field studies as defined by EFSA (2014). The endpoints may be too conservative to assess persistence as the design minimized soil surface processes as required by EFSA (2014) and such processes may contribute to dissipation.

Data Point:	KCA 7.1.2.2.1/20
Report Author:	[redacted]
Report Year:	2019
Report Title:	Amendment no. 1 to final report - Terrestrial field dissipation study with BAM SC 125 in Italy and Spain
Report No:	M-647363-02-1
Document No:	M-647363-02-1
Guideline(s) followed in study:	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 including Data Requirements SANCO/11803/2010 Rev. 7 and Test Methods SANCO/11843/2010 Rev. 4 EFSA Guidance Document for Evaluating Laboratory and Field Dissipation Studies to obtain DegT50 Values of Active Substances of Plant Protection Products and Transformation Products of these Active Substances in Soil, EFSA Journal 2014; 12(5):3662, 2014
Deviations from current test guideline:	Yes, Report meets the requirement for assessing test substance soil DegT50matrix values as required by EFSA (2014) for field studies. The endpoints may be too conservative for comparison to field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

Soil degradation of M-01 (AE C653713) under Southern European field conditions was investigated after application of M-01 onto bare soil plots in Albaro (Italy) and Vilobi d'Onyar (Spain).

BAM SC 125 is a suspension concentrate formulation, containing 125 g/L M-01. The formulation was sprayed once onto 400 sqm to 600 sqm plots at a rate of 0.8 L/ha, corresponding to nominal 100 g/ha M-01. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface.

Soil cores were taken 0 days before up to 714 days post-application to a maximum depth of 110 cm. The soil cores were cut into 10-cm soil layers, bulked soil layers were homogenised and finally analysed for M-01.

Sub-samples of homogenised soil were extracted in a microwave extractor with organic solvent. Potential matrix effects were eliminated by using an internal standard solution of isotopically labeled reference items added to sample extracts. Following separation of fine particles from soil extracts by centrifugation the identification and quantitation of the analytes was performed by high performance liquid chromatography using MS/MS detection in the multiple reaction monitoring mode. The analytical method was validated using three different soils. The limit of quantitation (LOQ) was 1.0 µg/kg and the limit of detection (LOD) was 0.3 µg/kg for M-01.

At Albaro (Italy), the mean initial amount of M-01 at day 0 was 100 g/ha, representing 100% of the nominal application rate. M-01 declined from 100 g/ha M-01 in soil at day 0 to 2.03 g/ha at day 541.

At Vilobi d'Onyar (Spain), the mean initial amount of M-01 at day 0 was 100 g/ha, representing 100% of the nominal application rate. M-01 declined from 100 g/ha M-01 in soil at day 0 to 5.28 g/ha at day 714.

Under field conditions M-01 residues were translocated up to 60 cm (Albaro, Italy) and 100 cm (Vilobi d'Onyar, Spain) depth, respectively. It is concluded there was some mobility of M-01 to deeper soil layers in the Vilobi d'Onyar trial but ~99% of residues were detected in 0-100 cm soil depth (out of a total measured depth of 110 cm). In contrast M-01 was fully retained in topsoil layers at the Albaro site. Consequently, robust $DegT_{50}$ values can be obtained for the compound from data from both trials.

I. Materials and Methods

A. Materials

1. Test Item

AE C653711 (M-01) formulated as a suspension concentrate (125 g/L AE C653711)

Certificate of Analysis: 01865-00

Lot No: 2015-000656

2. Trial Location & Soil

A terrestrial field dissipation with M-01 (AE C653711) a suspension concentrate formulation, containing 125 g/L M-01 was conducted at two locations in Southern Europe. The two locations were Albaro Di Ronco All'Adige (Italy) and Vilobi d'Onyar (Spain). The sites were fully characterised, and the results summarised in Table 7.1.2.2.1- 115. The plot sizes ranged from 456 sqm to 600 sqm. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface. The control plot was prepared at least 5 m away from the treated plots.

Table 7.1.2.2.1- 115: Location, site description and climatic data of test sites

Characteristic	Units	Sampling depth [cm]			
		0-30	30-50	50-75	75-100
Soil Designation	-	**Albaro (Italy)**			
Soil ID	-	IT23			
Geographic Location City Country	-	[redacted] 37055 Albaro di Ronco, all' Adige, Veneto Italy			
pH	$CaCl_2$	7.3	7.4	7.5	7.4
Organic carbon	[% Carbon]	1.8	1.5	0.7	0.6
CEC	[meq/100 g]	19.7	20.1	17.9	17.1
Chalk	[% $CaCO_3$]	10.6	12.0	14.1	11.8
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 35 43 22	 33 45 22	 35 45 20	 41 39 20
Textural class	USDA	clay loam	clay loam	clay loam	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 61.5 35.5	 62.7 37.9	 67.8 41.1	 65.8 37.6
Soil Designation	-	**Vilobi d'Onyar (Spain)**			
Soil ID	-	SPA1			
Geographic Location City Country		[redacted] 17185 Vilobi d'Onyar, Catalonia Spain			
pH	$CaCl_2$	6.0	6.1	6.6	7.0
Organic carbon	[% Carbon]	0.8	0.3	0.1	0.1
CEC	[meq/100 g]	9.6	11.9	13.5	14.2
Chalk	[% $CaCO_3$]	0.3	0.2	0.1	0.2
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 17 33 50	 27 23 50	 29 15 56	 27 15 58
Textural class	USDA	loam	sandy clay loam	sandy clay loam	sandy clay loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 41.4 21.5	 44.5 20.7	 47.9 23.9	 47.3 22.4

B. Study Design

1. Experimental Conditions

BAM SC 125 is a suspension concentrate formulation, containing 125 g/L M-01 (AE C653711). The product was sprayed onto bare earth once at each site at an application rate of 0.8 L/ha and 600 L/ha water, corresponding to 100 g/ha of M-01 during May and June 2015. Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of a nonselective herbicide to control weeds.

Air temperature, precipitation including irrigation and sunshine data were recorded on site during the field tests.

Soil dissipation of M-01 was studied for 714 days.

2. Sampling

The treated plot of the trial was divided into three sub-plots. From each sub-plot 10 soil cores were taken and combined together at each sampling interval (30 cores in total).

Samples were taken on the following occasions: 0 (post-application; each 0-10 cm depth), 6-8, 14-15 (each 0-60 cm depth), 21-22, 27-28, 57-56 (each 0-85 cm depth), and 121-128, 167-175, 279-282, 364-369, 450-488, 541-545, 628, 714 (each 0-110 cm depth) after treatment. From the control plot samples were taken on the following occasions: 0 days before application, 364-369 and 714 days after application.

Soil cores taken from the three sites were deep frozen to -18°C within twenty four hours after sampling, then shipped frozen to the analytical laboratory in Germany.

3. Analytical Procedures

The analytical method 01445 was used to determine levels of M-01 (AE C653711). Soil samples of 5 g were extracted in a microwave extractor with a mixture of acetonitrile/water (4/1, v/v). The extracts were centrifuged to remove fine particles of the soil. Possible matrix effects of M-01 were eliminated by using an internal standard solution of isotopic labelled reference items. Quantification was carried out by LC-MS/MS. The limit of quantitation (LOQ) for M-01 was 1.0 µg/kg in soil. The limit of determination (LOD) for M-01 was 0.3 µg/kg.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item M-01 at levels of LOQ, 10 x LOQ and 500 x LOQ and processed in parallel to the dissipation samples throughout the study. The results are summarised in the table below.

Single Values [%]	No of Recoveries	Fortification Level [µg/kg]	Mean [%]	RSD [%]
68, 68, 70, 74, 75, 77, 79, 79, 80, 81, 81, 82, 83, 84, 84, 85, 85, 85, 85, 86, 86, 86, 86, 87, 88, 88, 88, 88, 88, 88, 88, 90, 90, 90, 90, 91, 91, 92, 92, 92, 92, 92, 92, 92, 92, 93, 93, 93, 93, 93, 94, 94, 94, 94, 94, 94, 94, 94, 95, 95, 95, 95, 95, 95, 95, 96, 97, 97, 97, 97, 97, 97, 98, 98, 98, 99, 99, 99, 99, 99, 100, 100, 100, 100, 101, 101, 101, 101, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 106, 106, 106, 107, 107, 107, 107, 107, 108, 108, 108, 108, 109, 109, 109, 109, 110, 110, 110, 110, 111, 111, 112, 112, 112, 112, 113, 113, 114, 114, 114, 114, 115, 116, 116, 116, 117, 117, 118, 119, 119, 119, 120	166	1.0	99	11.0

80, 84, 86, 87, 89, 90, 90, 91, 91, 92, 92, 92, 92, 93, 93, 93, 93, 93, 93, 94, 94, 94, 94, 94, 95, 95, 95, 95, 95, 96, 96, 96, 96, 96, 96, 96, 96, 96, 97, 97, 97, 97, 97, 97, 97, 97, 97, 97, 98, 98, 98, 98, 98, 98, 98, 99, 99, 99, 99, 99, 99, 99, 99, 100, 100, 100, 100, 101, 101, 101, 101, 101, 101, 101, 101, 101, 102, 102, 102, 102, 102, 102, 102, 102, 102, 102, 103, 103, 103, 103, 103, 104, 104, 104, 104, 104, 104, 104, 104, 105, 105, 105, 105, 105, 105, 105, 105, 105, 105, 106, 106, 107, 107, 107, 107, 108, 108, 108, 108, 108, 108, 108, 108, 108, 108, 108, 108, 108, 109, 109, 109, 109, 109, 109, 109, 110, 110, 110, 110, 110, 110, 110, 110, 110, 111, 111, 111, 111, 111, 111, 111, 112, 112, 112, 112, 112, 113, 113, 113, 114, 114, 114, 114, 114, 115, 115, 115, 115, 115, 119, 119	175	10	103	7.2
82, 85	2	500	84	-
Overall recovery	343		101	9.5

RSD = Relative standard deviation

The validation of the extraction was carried out during the study with samples taken immediately after the application of the test substance.

4. Evaluation of the Data and Kinetic Calculations

For evaluation of degradation kinetics of the test item according to the FOCUS guidance document on degradation kinetics, the total residue of the test item in the soil profile covering all soil horizons was calculated according to the following procedure:

- values between LOD and LOQ were set to the measured values.
- values < LOD were set to 0.5 LOD for samples after, before or deeper as a value > LOD or for samples between (> LOD and < LOQ). The curve was cut off after the first non-detect (< LOD), if no later value > LOQ followed.
- at day 0, values < LOD in deep horizons were set to 0.

The results in [µg/kg] were converted to [g/ha] considering the actual soil density of the corresponding soil layer.

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of M-01 in soil samples by HPLC-MS/MS.

B. Data:

The results for residues of M-01 in different soil depths are presented below (expressed as g/ha)in Table 7.1.2.2.1- 116 and Table 7.1.2.2.1- 117.

At the Albaro trial site, residues of M-01 were detected at concentrations > LOQ at soil depths down to 40 cm. The metabolite was detected in the 0-10 cm and 10-20 cm soil horizons throughout Albaro trial. The maximum residue level in the 10-20 cm horizon was 140 g/ha at DAT 282. Low residues were detected in the 20-30 cm soil layer at concentrations from <LOQ to 7.66 g/ha and in the 30-40 cm soil layer at concentrations <LOQ to 3.72 g/ha from DAT 121 in both layers. No residues > LOD were detected in deeper soil layers with the exception of one finding < LOQ in the 50-60 cm soil horizon at DAT 364.

It is concluded M-01 was fully retained in topsoil layers and consequently robust $DegT_{50}$ values can be obtained for the compound from data from the Albaro trial.

At the Vilobi d'Onyar trial site, residues of M-01 were detected at concentration > LOQ at soil depths down to 70 cm. The metabolite was detected in the 0-10 cm soil horizon throughout the trial and from DAT 15 in the 10-20 cm and DAT 56 in the 20-30 cm soil horizons. The maximum residue level in the 10-20 cm horizon was observed at DAT 128 at a concentration of 15.6 g/ha and in the 20-30 cm horizon at DAT 167 at a concentration of 7.82 g/ha. From DAT 545 residues in the 10-20 and 20-30 cm soil horizons were <LOQ. Low residues were detected in the underlying 30-40 cm, 40-50 cm, 50-60 cm and 60-70 cm horizons at concentrations ranging from <LOQ to 6.12 g/ha in samples taken from DAT 128 to DAT 546. In the underlying soil layers, 70-80 cm and 80-90 cm, residues < LOQ were detected from DAT 128 to DAT 399 only. No residues > LOD were detected in deepest soil layers except for three and two single replicates < LOQ in the 90-100 cm and 100-110 cm soil horizons, respectively between DAT 128 and 279. At all other timepoints no residues > LOD were detected below 30 cm.

It is concluded there was some mobility of M-01 to deeper soil layers but >99% of residues were detected in 0-100 cm soil depth (out of a total measured depth of 110 cm) and consequently robust DegT50 values can be obtained for the compound from data from the Vilobi d'Onyar trial.

Table 7.1.2.2.1- 116: Residues of M-01 (AE C653711) in different soil depths from the Albaro trial after an application of 100 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT											
		0	6	14	21	27	57	121	175	282	364	450	541
0-10	T1	94.7	81.5	61.7	70.3	63.8	48.7	44	19.3	10.3	4.76	2.54	[0.96]
	T2	98.3	71.3	61.2	52.3	50.7	61.5	45.1	25.7	9.40	4.25	1.88	[1.01]
	T3	108	56.8	71.0	70.0	112	47.1	48.5	29.8	8.59	4.59	2.15	[1.06]
	mean	100	69.9	64.6	64.2	75.5	52.4	46.0	24.9	9.43	4.53	2.19	1.01
10-20	T1	-	6.84	8.24	1.93	4.24	[1.10]	3.84	9.91	14.0	5.29	[1.13]	<LOD
	T2	-	9.75	8.41	2.96	2.82	3.45	4.18	12.4	10.2	4.52	[0.58]	<LOD
	T3	-	6.85	9.11	4.77	6.85	2.60	6.64	11.4	11.1	4.71	[0.92]	<LOD
	mean	-	7.81	8.59	3.22	4.64	2.35	4.89	11.4	12.1	4.84	[0.88]	<LOD
20-30	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	[0.95]	3.34	7.66	5.17	[1.32]	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	1.59	3.58	6.28	3.84	[0.67]	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	[1.47]	3.31	6.57	4.18	[1.19]	<LOD
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	[1.34]	3.41	6.84	4.40	[1.06]	<LOD
30-40	T1	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	[1.25]	3.02	2.80	<LOD	<LOD
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	[0.76]	[1.12]	2.38	1.80	<LOD	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	[0.64]	[1.62]	3.72	2.15	<LOD	<LOD
	mean	-	<LOD	<LOD	<LOD	<LOD	<LOD	[0.47]	[1.33]	3.04	2.25	<LOD	<LOD
40-50	T1	-	-	-	-	<LOD	<LOD	<LOD	<LOD	[0.99]	[1.04]	<LOD	<LOD
	T2	-	-	-	-	<LOD	<LOD	<LOD	<LOD	[0.79]	[0.70]	<LOD	<LOD
	T3	-	-	-	-	<LOD	<LOD	<LOD	<LOD	[0.90]	[0.74]	<LOD	<LOD
	mean	-	-	-	-	<LOD	<LOD	<LOD	<LOD	[0.89]	0.83	<LOD	<LOD
50-60	T1	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	[0.87]	<LOD	<LOD
	T2	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	[0.29]	<LOD	<LOD

Depth [cm]	Sub plot	DAT											
		0	6	14	21	27	57	121	175	282	364	450	541
60-70	T1	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
70-80	T1	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
80-90	T1	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
90-100	T1	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
100-110	T1	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T2	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD
Sum	T1	94.7	88.3	69.9	72.2	68.0	49.8	48.8	33.8	36.0	19.9	[4.99]	[0.96]
	T2	98.3	81.1	69.6	55.7	56.5	65.0	52.0	42.8	30.1	[15.1]	[3.13]	[1.01]
	T3	108	83.7	80.1	74.8	119	49.7	57.3	46.1	30.9	[16.4]	[4.26]	[1.06]
	mean	100	77.7	73.2	67.1	80.2	54.8	52.7	40.9	32.3	17.1	[4.13]	[1.01]

LOD = 0.3 µg/kg equivalent to *ca.* 0.6 g/ha depending on soil moisture and density
LOQ = 1.0 µg/kg equivalent to *ca.* 1.9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ

Table 7.1.2.2.1- 117: Residues of M-01 (AE C653711) in different soil depths from the Vilobi d'Onyar trial after an application of 100 g a.s./ha (expressed as g/ha)

Depth [cm]	Sub plot	DAT													
		0	8	15	22	28	56	128	167	279	369	488	545 [A]	546 [A]	628	714
0-10	T1	106	98.1	86.6	75.7	89.3	76.5	21.3	21.5	12.2	7.42	3.44	2.83	-	1.79	2.77
	T2	111	98.2	107	81.9	72.2	71.0	19.5	15.2	7.21	5.93	3.94	2.92	3.22	1.88	1.68
	T3	119	90.6	90.9	72.2	80.1	80.7	18.3	15.2	10.0	4.40	2.61	-	2.52	1.82	2.06
	mean	112	95.6	94.8	76.6	80.5	76.1	19.7	17.3	9.8	5.90	3.33	2.87		1.83	2.17
10-20	T1	-	<LOD	[1.02]	[0.80]	<LOD	<LOD	15.6	14.4	7.05	5.02	2.94	[2.03]	-	[1.35]	[2.02]
	T2	-	<LOD	[0.99]	[1.34]	<LOD	[0.98]	11.5	7.99	4.53	3.39	[0.75]	[1.59]	2.32	[1.08]	[1.09]
	T3	-	<LOD	[0.89]	1.55	<LOD	[0.86]	8.69	13.0	7.50	2.47	[1.43]	-	[1.25]	[1.27]	<LOD
	mean	-	<LOD	[0.97]	[1.23]	<LOD	[0.61]	11.9	11.8	6.40	3.63	[1.71]	1.80		[1.23]	[1.04]
20-30	T1	-	<LOD	<LOD	<LOD	<LOD	2.00	5.95	7.82	5.17	3.80	[1.30]	[1.52]	-	[1.31]	1.96
	T2	-	<LOD	<LOD	<LOD	<LOD	<LOD	5.37	3.75	3.63	2.53	[0.60]	[0.99]	[1.58]	[0.83]	<LOD
	T3	-	<LOD	<LOD	<LOD	<LOD	<LOD	5.61	6.98	5.01	2.01	[1.15]	-	<LOD	[0.84]	[0.61]
	mean	-	<LOD	<LOD	<LOD	<LOD	[0.67]	5.64	6.18	4.60	2.78	[1.02]	[1.02]		[0.99]	[0.86]
30-40	T1	-	-	-	-	-	<LOD	5.33	6.12	3.83	2.10	<LOD	<LOD	-	<LOD	<LOD
	T2	-	-	-	-	-	-	4.48	3.00	2.72	[1.53]	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	5.33	5.21	3.60	[1.45]	<LOD	-	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	<LOD	5.05	4.78	3.38	[1.69]	<LOD	<LOD		<LOD	<LOD
40-50	T1	-	-	-	-	-	<LOD	3.65	5.34	3.17	[1.53]	<LOD	<LOD	-	<LOD	<LOD
	T2	-	-	-	-	-	-	3.83	2.88	3.00	1.94	[0.68]	<LOD	2.07	<LOD	<LOD
	T3	-	-	-	-	-	-	2.86	5.35	4.33	[1.57]	[0.68]	-	[0.75]	<LOD	<LOD
	mean	-	-	-	-	-	<LOD	3.45	4.52	3.50	[1.68]	[0.46]	[0.71]		<LOD	<LOD
50-60	T1	-	-	-	-	-	<LOD	1.98	3.87	2.26	[1.18]	[0.82]	<LOD	-	<LOD	<LOD
	T2	-	-	-	-	-	-	2.43	2.62	2.73	[1.54]	[1.16]	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	[1.64]	3.96	3.86	[1.05]	[0.94]	-	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	<LOD	2.02	3.48	2.95	[1.26]	[0.97]	<LOD		<LOD	<LOD

Depth [cm]	Sub plot	DAT														
		0	8	15	22	28	56	128	167	279	369	488	545 [A]	546 [A]	628	714
60-70	T1	-	-	-	-	-	-	[0.63]	2.87	[1.89]	[1.23]	<LOD	<LOD	-	<LOD	<LOD
	T2	-	-	-	-	-	-	[1.68]	2.17	2.23	[1.27]	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	[0.67]	3.44	3.81	[0.86]	<LOD	-	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	[0.99]	2.83	3.64	[1.12]	<LOD	<LOD		<LOD	<LOD
70-80	T1	-	-	-	-	-	-	<LOD	[1.86]	[1.09]	[0.85]	<LOD	<LOD	-	<LOD	<LOD
	T2	-	-	-	-	-	-	[1.42]	[1.16]	[1.34]	[0.78]	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	<LOD	[1.83]	[0.79]	[1.05]	<LOD	-	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	[0.47]	[1.62]	[1.07]	[0.88]	<LOD	<LOD		<LOD	<LOD
80-90	T1	-	-	-	-	-	-	<LOD	[1.32]	<LOD	[0.61]	<LOD	<LOD	-	<LOD	<LOD
	T2	-	-	-	-	-	-	[1.16]	[0.79]	[0.68]	[1.34]	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	<LOD	[1.05]	<LOD	<LOD	<LOD	-	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	[0.39]	[1.04]	[0.23]	[0.65]	<LOD	<LOD		<LOD	<LOD
90-100	T1	-	-	-	-	-	-	<LOD	[1.28]	<LOD	<LOD	<LOD	<LOD	-	<LOD	<LOD
	T2	-	-	-	-	-	-	[0.71]	<LOD	[0.77]	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	-	<LOD	<LOD	<LOD
	mean	-	-	-	-	-	-	[0.24]	[0.43]	[0.26]	<LOD	<LOD	<LOD		<LOD	<LOD
100-110	T1	-	-	-	-	-	-	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	-	<LOD	<LOD
	T2	-	-	-	-	-	-	[0.65]	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	T3	-	-	-	-	-	-	<LOD	<LOD	[0.63]	<LOD	<LOD	-	<LOD	<LOD	[0.72]
	mean	-	-	-	-	-	-	[0.22]	<LOD	[0.21]	<LOD	<LOD	<LOD		<LOD	[0.24]
Sum	T1	106	98.1	87.6	76.5	89.3	78.5	54.4	66.4	36.7	23.7	[8.50]	[6.38]	-	[4.45]	[6.75]
	T2	111	98.2	108	83.2	72.2	72.0	52.7	39.5	28.8	20.2	[7.13]	[5.50]	[9.19]	[3.79]	[2.77]
	T3	119	90.6	91.8	73.8	80.1	81.6	43.1	56.0	39.5	[14.9]	[6.81]	-	[4.52]	[3.93]	[3.39]
	mean	112.0	95.6	95.8	77.8	80.5	77.4	50.1	54.0	35.0	19.6	[7.48]	[6.40]		[4.06]	[4.30]

LOD = 0.3 µg/kg equivalent to *ca.* 0.6 g/ha depending on soil moisture and density
LOQ = 1.0 µg/kg equivalent to *ca.* 1.9 g/ha depending on soil moisture and density
Values in square brackets are values > LOD but < LOQ
[A] Combined mean values are given for DAT 545 and DAT 546

The dissipation of M-01 with time is presented in Table 7.1.2.2.1- 118 and Table 7.1.2.2.1- 119. The values have been pre-processed according to the procedure described in FOCUS kinetics guidance (as described earlier). Actual values are given in brackets.

At Albaro (Italy), the mean amount of M-01 at day 0 was 100 g/ha, representing 100% of the nominal application rate. M-01 declined from 100 g/ha in soil at day 0 to 1.01 g/ha at day 541. At Vilobí d'Onyar (Spain), the mean amount of M-01 at day 0 was 112 g/ha, representing 112% of the nominal application rate. M-01 declined from 112 g/ha in soil at day 0 to 4.30 g/ha at day 714.

The dissipation of M-01 showed a biphasic behaviour. After treatment, M-01 dissipated in a first step very fast within a couple of days followed by a second more slowly step until study termination. Residues of M-01 in control samples were < LOD for all samples taken.

Table 7.1.2.2.1- 118: Residues of M-01 (AE C653711) in soil from the Albaro trial after an application of 100 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Albaro (Italy)	DAT											
	0	**6**	**14**	**21**	**27**	**57**	**121**	**175**	**282**	**364**	**450**	**541**
T1	94.7 (94.7)	88.9 (88.4)	70.5 (69.9)	72.7 (72.0)	68.5 (68.2)	50.3 (49.7)	49.3 (49.1)	34.3 (33.8)	36.5 (35.8)	20.5 (19.9)	6.49 (4.85)	2.00 (0.95)
T2	98.3 (98.0)	81.6 (80.7)	70.1 (70.3)	55.8 (52.4)	54.0 (53.5)	65.9 (65.1)	52.6 (52.3)	43.3 (42.7)	30.6 (30.0)	15.6 (15.1)	4.15 (3.11)	2.04 (1.01)
T3	108 (108)	64.2 (63.6)	80.6 (79.7)	75.3 (79.9)	119 (119)	50.7 (49.6)	57.8 (57.2)	46.7 (46.0)	31.4 (31.4)	16.9 (16.4)	5.26 (4.25)	2.06 (1.06)
Mean	**100 (100)**	**78.2 (77.6)**	**73.7 (73.3)**	**67.9 (68.1)**	**80.5 (80.2)**	**55.6 (54.8)**	**53.2 (52.9)**	**41.4 (40.8)**	**32.8 (32.4)**	**17.7 (17.1)**	**5.30 (4.07)**	**2.03 (1.01)**
Min	94.7 (94.7)	64.2 (63.6)	70.1 (69.9)	55.5 (52.4)	54.0 (53.5)	50.3 (49.6)	49.3 (49.1)	34.3 (33.8)	30.6 (30.0)	15.6 (15.1)	4.15 (3.11)	2.00 (0.95)
Max	108 (108)	88.9 (88.4)	80.6 (79.7)	75.3 (79.9)	119 (119)	65.9 (65.1)	57.8 (57.2)	46.7 (46.0)	36.5 (35.8)	20.5 (19.9)	6.49 (4.85)	2.06 (1.06)
n	3	3	3	3	3	3	3	3	3	3	3	3
% of day 0	100 (100)	78 (78)	74 (73)	68 (68)	81 (80)	56 (55)	53 (53)	41 (41)	33 (32)	18 (17)	5 (4)	2 (1)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3.
The actual values are given in brackets (expressed as g/ha)

Table 7.1.2.2.1- 119: Residues of M-01 (AE C653711) in soil from the Vilobi d'Onyar trial after an application of 100 g a.s./ha (pre-processed values according to FOCUS, expressed as g/ha)

Vilobi d'Onyar (Spain)	DAT														
	0	**8**	**15**	**22**	**28**	**56**	**128**	**167**	**279**	**369**	**488**	**545**	**546**	**628**	**714**
T1	106 (106)	98.8 (98.1)	88.2 (87.6)	77.1 (76.5)	90.4 (89.3)	80.5 (78.5)	56.3 (54.4)	67.0 (66.4)	37.9 (36.7)	23.7 (23.7)	10.9 (8.5)	7.55 (6.38)	- (-)	5.04 (4.45)	7.32 (6.75)
T2	111 (111)	98.8 (98.2)	109 (108)	83.8 (83.2)	72.8 (72.2)	72.5 (72.0)	52.7 (52.7)	40.1 (39.5)	29.4 (28.8)	20.8 (20.2)	9.53 (7.13)	7.21 (5.50)	10.3 (9.19)	5.00 (3.79)	3.38 (2.77)
T3	119 (119)	91.3 (90.6)	92.4 (91.8)	74.3 (73.8)	80.6 (80.1)	82.1 (81.6)	44.3 (43.1)	57.3 (56.0)	40.7 (39.5)	16.1 (14.9)	8.46 (6.81)	- (-)	6.20 (4.52)	5.75 (3.92)	5.15 (3.39)
Mean	**112 (112)**	**96.3 (95.6)**	**96.5 (95.8)**	**78.4 (77.8)**	**81.3 (80.5)**	**78.4 (77.4)**	**51.1 (50.1)**	**54.8 (54.0)**	**36.0 (35.0)**	**20.2 (19.6)**	**9.63 (7.59)**	**7.82 (6.4)[A]**		**5.26 (4.05)**	**5.28 (4.30)**
Min	106 (106)	91.3 (90.6)	88.2 (87.6)	74.3 (73.8)	72.8 (72.2)	72.5 (72.0)	44.3 (43.1)	40.1 (39.5)	29.4 (28.8)	16.1 (14.9)	8.46 (6.81)	6.20 (4.52)[A]		5.00 (3.79)	3.38 (2.77)
Max	119 (119)	98.8 (98.2)	109 (108)	83.8 (83.2)	90.4 (89.3)	82.1 (81.6)	56.3 (54.4)	67.0 (66.4)	40.7 (39.5)	23.7 (23.7)	10.9 (8.5)	10.3 (9.19)[A]		5.75 (4.45)	7.32 (6.75)
n	3	3	3	3	3	3	3	3	3	3	3	4[A]		3	3
% of day 0	100 (100)	86 (85)	86 (86)	70 (69)	73 (72)	70 (69)	46 (45)	49 (48)	32 (31)	18 (18)	9 (7)	(6)		5 (4)	5 (4)

DAT = Days after treatment; TX = Treated Subplot X with X = 1 to 3.
The actual values are given in brackets (expressed as g/ha)
[A] Combined values are given for DAT 545 and DAT 546

III. Conclusion

Under field conditions M-01 declined moderately and residues were translocated up to 60 cm (Albaro, Italy) and 100 cm (Vilobi d'Onyar, Spain) depth, whilst 76-100% (Albaro, Italy) and 53-100% (Vilobi d'Onyar, Spain) of residues remained in the top 0-30 cm at all timepoints. Un-normalised DT_{50} for the degradation of M-01 calculated from the reported data following the recommendations of the FOCUS work group details are provided in Document KCA 7.1.2.2.1/21.

Assessment and conclusion by applicant:

The study is considered valid to assess M-01 soil $Deg T_{50matrix}$ values for field studies as defined by EFSA (2014). The endpoints may be too conservative to assess persistence as the design minimized soil surface processes as required by EFSA (2014) and such processes may contribute to dissipation.

Data Point:	KCA 7.1.2.2.1/21
Report Author:	[redacted]
Report Year:	2020
Report Title:	Amendment no. 01: Terrestrial field dissipation study with BAM SC 125 in Germany, United Kingdom, France (North), France (South), Italy and Spain
Report No:	EnSa-18-1177
Document No:	M-650733-02-1
Guideline(s) followed in study:	Regulation (EC) No. 1107/2009 of the European Parliament and of the Council of 21 October 2009 including Data Requirements SANCO/11803/2010 Rev. 7 and Test Methods SANCO/11843/2010 Rev. 4 EFSA Guidance Document for Evaluating Laboratory and Field Dissipation Studies to obtain DegT50 Values of Active Substances of Plant Protection Products and Transformation Products of these Active Substances in Soil, EFSA Journal 2014; 12(5):3662, 2014
Deviations from current test guideline:	Yes. Report meets the requirement for assessing test substance soil DegT50matrix values as required by EFSA (2014) for field studies. The endpoints may be too conservative for comparison to field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

A summary report covering three terrestrial field dissipation studies was conducted to provide an overview of the trials and to calculate the kinetic rate of decline for each trial. Soil dissipation of M-01 (AE C653711) under European field conditions was investigated after application of BAM SC 125 on bare soil plots at six sites, three in Northern Europe; Burscheid (Germany), Great Chishill (United Kingdom) and Lignieres de Touraine (France North) and three in Southern Europe; St. Etienne du Grès (France South), Albaro di Ronco all'Adige (Italy) and Vilobi d'Onyar (Spain). Full summaries of the studies are provided in KCA 7.1.2.2.1/18, M-647366-03-1; KCA 7.1.2.2.1/19, M-M-647370-02-1 and KCA 7.1.2.21/20, M-647363-02-1.

The experiments were carried out in accordance with the EFSA Guidance Document for Evaluating Laboratory and Field Dissipation Studies to obtain $DegT_{50}$ Values of Active Substances of Plant Protection Products and Transformation Products of these Active Substances in Soil (EFSA Journal 2014).

BAM SC 125 was sprayed once onto 400 to 600 sqm for each treated plot at a rate of 0.8 L/ha, corresponding to nominal 100 g/ha M-01. Subsequently the test item was incorporated by a rotary harrow to a target depth of 7 cm followed by rolling the soil surface to meet EFSA requirements. The plots received at least 10 mm water between DAT-0 and DAT-3 by irrigation post application. The control plot was at least 5 m away from the treated plot.

Soil cores were taken 0 day before and at several dates up to 744 days post-application to a maximum depth of 110 cm. The soil cores were cut into 10-cm soil layers, bulked soil layers were homogenized and finally analyzed for M-01.

Sub-samples of homogenized soil (5 g) were extracted in a microwave extractor with a mixture of acetonitrile/water (4/1, v/v). Potential matrix effects were eliminated using an internal standard solution of isotopically labeled reference item added to sample extracts. Following separation of fine particles from soil extracts by centrifugation, identification and quantitation of the analyte was performed by high performance liquid chromatography using MS/MS detection in the multiple reaction monitoring mode. The analytical method was validated using three different soils. The limit of quantitation (LOQ) was 1.0 µg/kg and the limit of detection (LOD) was 0.3 µg/kg.

The amount of M-01 decreased from DAT-0 to study end (DAT-701) from 108 to 11.5 g/ha at Burscheid (Germany), from DAT-0 to DAT-744 from 59.8 g/ha to 31.9 g/ha at Great Chishill (United Kingdom), from DAT-0 to DAT-699 from 86.2 to 17.7 g/ha at Lignieres-deTouraine (France North), from DAT-0 to DAT-714 from 87.9 g/ha to 8.09 g/ha at St. Etienne du Gres (France South), from DAT-0 to DAT-541 from 100 g/ha to 1.01 g/ha at Albaro di Ronco all'Adige (Italy) and from DAT-0 to DAT-714 from 112 to 4.30 g/ha at Vilobi d'Onyar (Spain)

Under field conditions BAM residues were translocated up to 110 cm depth in the trial at Great Chishill, United Kingdom with 55-100% of measured residues remained in the top 0-30 cm depending on the timepoint. However, there was significant mobility of BAM to deeper soil layers with levels as low as 79% of residues retained in 0-100 cm soil depth (out of a total measured depth of 110 cm). Compatable calculations at the other five sites show >97 to 100% of measured residues are contained within 0-100 cm depth. Consequently, the trials at Burscheid (Germany), Lignieres de Touraine (France North), St. Etienne du Grès (France South), Albaro di Ronco all'Adige (Italy) and Vilobi d'Onyar (Spain) are acceptable to determine reliable degradation rates but the trial at Great Chishill (United Kingdom) is not.

Dissipation of M-01 from soil was moderate to slow with DT_{50} values ranging from 133 and 344 days for all test sites. An overview of the results is given below:

Location	Soil Type (USDA)	pH ($CaCl_2$)[A]	Best Fit Kinetic Model[B]	DT_{50} [d]	DT_{90} [d]
Burscheid (Germany)	Silt Loam (0-50 cm) Loam (50-100 cm)	5.3	FOMC	133	651
Lignieres de Touraine (France North)	Sandy Loam (0-75 cm) Clay Loam (75-100 cm)	5.9	SFO	344	> 1000
St.Etienne du Grès (France South)	Clay Loam (0-75 cm) Clay (75-100 cm)	7.7	DFOP	152	788
Albaro di Ronco all'Adige (Italy)	Clay Loam (0-75 cm) Clay (75-100 cm)	7.3	SFO	156	519
Vilobi d'Onyar (Spain)	Loam (0-30 cm) Sandy Clay Loam (30-100 cm)	6.0	SFO	160	532

[A] pH in 0-30 cm soil depth
[B] FOMC: first order multi compartment; DFOP: double first order in parallel; SFO: Simple first order

I. Materials and Methods

A. Materials

1. Test Item

AE C653711 (M-01) formulated as a suspension concentrate (125g/L AE C653711)

Certificate of Analysis: 01863-00

Lot No: 2015-000656

2. Trial Location & Soil

A terrestrial field dissipation with M-01 (AE C653711, called BAM in the report) prepared as a suspension concentrate formulation, containing 125 g/L M-01 was conducted at three locations in Northern Europe and at three sites in Southern Europe. The six locations were Burscheid (Germany), Great Chishill (United Kingdom) and Lignieres de Touraine (Northern France), St. Etienne du Grès (Southern France), Albaro (Italy) and Vilobi d'Onyar (Spain). The sites were fully characterised, and the results summarized Table 7.1.2.2.1- 120 and Table 7.1.2.2.1- 121. The plot sizes ranged from 400 sqm to 600 sqm. The control plot was prepared at least 5 m away from the treated plots.

Table 7.1.2.2.1- 120: Location, site description and climatic data of test sites in Northern Europe

Characteristic	Units	Sampling depth [cm]			
		0-30	30-50	50-75	75-100
Soil Designation	-	**Burscheid (Germany)**			
Soil ID	-	VG08			
Geographic Location City Country	-	Burscheid [redacted] Germany			
pH	$CaCl_2$	5.3	5.6	5.6	5.6
Organic carbon	[% Carbon]	1.2	0.4	0.1	0.1
CEC	[meq/100 g]	12.8	11.8	12.4	11.9
Chalk	[% $CaCO_3$]	12.8	11.8	12.4	11.9
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 21 61 18	 23 57 20	 19 43 38	 13 37 50
Textural class	USDA	silt loam	silt loam	loam	loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 49.3 35.2	 50.0 31.9	 45.9 30.3	 39.4 25.9
Soil Designation	-	**Great Chishill (United Kingdom)**			
Soil ID	-	ENG8			
Geographic Location City Country	-	[redacted] Great Chishill, Cambridgeshire United Kingdom			
pH	$CaCl_2$	7.2	7.5	7.7	7.6
Organic carbon	[% Carbon]	2.1	1.1	0.5	0.5
CEC	[meq/100 g]	28.9	26.1	19.9	17.4
Chalk	[% $CaCO_3$]	1.3	5.8	37.9	43.1
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 41 23 36	 45 21 34	 53 23 24	 51 23 26
Textural class	USDA	clay	clay	clay	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 60.8 29.2	 57.2 27.9	 52.6 25.3	 49.5 25.9
Soil Designation	-	**Lignieres de Touraine (France)**			
Soil ID	-	FR09			
Geographic Location City Country	-	[redacted] 37130 Lignieres de Touraine, Central Region France			
pH	$CaCl_2$	5.9	6.5	6.8	6.8
Organic carbon	[% Carbon]	0.8	0.4	0.3	0.5
CEC	[meq/100 g]	12.2	13.2	14.7	21.8
Chalk	[% $CaCO_3$]	0.2	0.3	0.3	0.0
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 15 15 70	 15 19 66	 19 25 56	 37 33 30
Textural class	USDA	sandy loam	sandy loam	sandy loam	clay loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 44.4 17.5	 47.7 20.6	 52.3 26.1	 61.5 33.1

Table 7.1.2.2.1- 121: Location, site description and climatic data of test sites in Southern Europe

Characteristic	Units	Sampling depth [cm]			
		0-30	30-50	50-75	75-100
Soil Designation	-	St. Etienne du Grès (France)			
Soil ID	-	FR08			
Geographic Location City Country	-	[redacted] 13103 St. Etienne du Grès, Provence-Alpes-Côte d'Azur France			
pH	$CaCl_2$	7.7	7.7	7.8	7.8
Organic carbon	[% Carbon]	0.8	0.6	0.6	0.4
CEC	[meq/100 g]	11.7	11.7	13.2	14.0
Chalk	[% $CaCO_3$]	42.1	42.1	42.5	43.7
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 29 45 26	 31 47 22	 39 41 20	 43 39 18
Textural class	USDA	clay loam	clay loam	clay loam	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 46.2 25.5	 43.4 26.8	 48.3 25.3	 51.4 25.0
Soil Designation	-	Albaro (Italy)			
Soil ID	-	IT23			
Geographic Location City Country	-	[redacted] 37055 Albaro di Ronco, all'Adige, Veneto Italy			
pH	$CaCl_2$	7.3	7.4	7.5	7.4
Organic carbon	[% Carbon]	1.8	1.5	0.7	0.6
CEC	[meq/100 g]	19.7	20.1	17.9	17.1
Chalk	[% $CaCO_3$]	10.6	12.0	14.1	11.8
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 35 43 22	 33 45 22	 35 45 20	 41 39 20
Textural class	USDA	clay loam	clay loam	clay loam	clay
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 61.5 35.5	 62.7 35.9	 67.8 41.1	 65.5 37.6
Soil Designation	-	Vilobi d'Onyar (Spain)			
Soil ID	-	SPA1			
Geographic Location City Country	-	[redacted] 17185 Vilobi d'Onyar, Catalonia Spain			
pH	$CaCl_2$	6.0	6.1	6.6	7.0
Organic carbon	[% Carbon]	0.8	0.3	0.1	0.1
CEC	[meq/100 g]	9.6	11.9	13.5	14.2
Chalk	[% $CaCO_3$]	0.3	0.2	0.1	0.2
Particle size distribution (USDA) Clay < 0.002 mm Total silt 0.002 - 0.050 mm Total sand 0.050 - 2 mm	 % % %	 17 33 50	 27 23 50	 29 15 56	 27 15 58
Textural class	USDA	loam	sandy clay loam	sandy clay loam	sandy clay loam
Water Holding Capacity MWHC (pF 0.05) WHC at 0.1 bar (pF2)	 Vol % Vol %	 41.4 21.5	 44.5 20.7	 47.9 23.9	 47.3 22.4

B. Study Design

1. Experimental Conditions

BAM SC 125 is a suspension concentrate formulation, containing 125 g/L M-01 (AE C653711). The product was sprayed onto bare earth once at each site at an application rate of 0.8 L/ha and 600 L/ha water, corresponding to 100 g/ha of M-01 during May and June 2015. Throughout the study no cultivation was carried out and the plots were maintained as bare plots by periodic application of a nonselective herbicide to control weeds.

Air temperature, precipitation including irrigation and sunshine data were recorded on site during the field tests.

Soil dissipation of M-01 was studied for up to 744 days.

2. Sampling

The treated plot of the trial was divided into three sub-plots. From each sub-plot 10 soil cores were taken and combined together at each sampling interval (30 cores in total).

Samples were taken on the following occasions: 0 (post-application, each 0-10 cm depth), 6-8, 14-15 (each 0-60 cm depth), 21-22, 27-29, 56-68 (each 0-85 cm depth), and 118-135, 166-204, 259-307, 348-400, 436-488, 518-558, 605-670, 699-744 (each 0-110 cm depth) days after treatment (DAT). From the control plot samples were taken on the following occasions: 0 days before application, 364-369 or 699-701 and 714 days after application. Soil cores were deep frozen to -18°C.

3. Analytical Procedures

The analytical method 01445 was used to determine levels of M-01. Soil samples of 5 g were extracted in a microwave extractor with a mixture of acetonitrile/water (4/1, v/v). The extracts were centrifuged to remove fine particles of the soil. Possible matrix effects of M-01 were eliminated by using an internal standard solution of isotopic labelled reference items. Quantification was carried out by LC-MS/MS. The limit of quantitation (LOQ) for M-01 was 1.0 µg/kg in soil. The limit of determination (LOD) for M-01 was 0.3 µg/kg.

During analysis of the dissipation samples, concurrent recovery samples were prepared freshly by fortification of control samples with test item M-01. The mean recovery for M-01 was 101 and 103% (RSD 9.5.%).

4. Evaluation of the Data and Kinetic Calculations

For evaluation of degradation kinetics of the test item according to the FOCUS guidance document on degradation kinetics, the total residue of the test item in the soil profile covering all soil horizons was calculated according to the following procedure:

- values between LOD and LOQ were set to the measured values.
- values < LOD were set to 0.5 LOD for samples after, before or deeper as a value > LOD or for samples between (> LOD and <LOQ). The curve was cut off after the first non-detect (< LOD), if no later value > LOQ followed.
- at day 0, values < LOD in deep horizons were set to 0.

The results in [µg/kg] were converted to [g/ha] considering the actual soil density of the corresponding soil layer.

DT_{50} and DT_{90} values for the degradation of M-01 (AE C653711) have been calculated from the reported data using the software KinGUI (version 2.1). To derive trigger endpoints, a comparison was performed for each site between the SFO, FOMC and DFOP fits. For the derivation of trigger endpoints, FOCUS

recommends to use the best-fit model. In an initial step, data for the applied compound fitted using the SFO and FOMC models were compared. If the SFO model provided a better fit overall (both visually and statistically), this fit was selected. If the FOMC model provided a better fit, the FOMC and DFOP fits were compared, and the model that provided the best fit overall was selected. It should be noted that extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using the FOMC model (EFSA, 2009), and this has been considered where relevant in the selection of the most appropriate model.

II. Results and Discussion

Under field conditions BAM residues were translocated up to 110 cm depth in the trial at Great Chishill, United Kingdom with 55-100% of measured residues remained in the top 0-30 cm depending on the timepoint. However, there was significant mobility of BAM to deeper soil layers (see Table 7.1.2.2.1-122) with levels as low as 79% of residues retained in 0-100 cm soil depth (out of a total measured depth of 110 cm). Comparable calculations at the other five sites show >97 to 100% of measured residues are contained within 0-100 cm depth. Consequently, the trials at Burscheid (Germany), Lignieres de Touraine (France North), St. Etienne du Grès (France South), Albaro di Ronco all'Adige (Italy) and Vilobi d'Onyar (Spain) are acceptable to determine reliable degradation rates but the trial at Great Chishill (United Kingdom) is not due to significant leaching of residues out of the sampled soil depth.

Table 7.1.2.2.1- 122: Retention of M-01 residues with soil depth

Trial site	Residues of M-01 with soil depth	Percent of M-01 residues in 0-100 cm depth [A]	Trial acceptable for $DegT_{50}$ evaluation
St Etienne du Gres (France South)	M-01 retained in topsoil layers	100%	Yes
Albaro di Ronco all'Adige (Italy)	M-01 retained in topsoil layers	100%	Yes
Burscheid (Germany)	Some mobility of M-01 to deeper layers	>97%	Yes
Vilobi d'Onyar (Spain)	Some mobility of M-01 to deeper layers	>99%	Yes
Lignieres de Touraine (France North)	Some mobility of M-01 to deeper layers	100%	Yes
Great Chishill (United Kingdom)	Heavy clay loam over calcareous clay. Chromatographic mobility of M-01 not expected. High bypass flow in 2 out of 3 subplots.	Only >80%	Unacceptable

[A] Out of a total measured depth of 110 cm

The decline of M-01 residues with time for the entire soil profile of the five acceptable trial sites is presented in Table 7.1.2.2.1- 123 to Table 7.1.2.2.1- 127.

Table 7.1.2.2.1- 123: Residues of M-01 in soil from the Burscheid trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

Burscheid (Germany)	DAT (days)													
	0	**7**	**15**	**21**	**28**	**63**	**121**	**172**	**259**	**395**	**436**	**518**	**603**	**701**
T1	98.9	86.3	1333	114	139	83.5	32.4	49.6	41.0	28.3	20.7	19.5	15.6	10.0
T2	131	121	129	180	107	89.6	50.6	35.9	24.5	25.1	18.1	22.1	8.85	11.3
T3	94.7	107	107	143	135	90.6	67.6	49.1	34.8	38.1	29.7	20.1	15.8	13.2
Mean	**108**	**105**	**123**	**145**	**127**	**87.9**	**50.2**	**44.8**	**33.4**	**30.5**	**22.8**	**20.5**	**13.4**	**11.5**

DAT = Days after treatment

Table 7.1.2.2.1- 124: Residues of M-01 in soil from the Lignieres de Touraine (France North) trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

Lignieres de Touraine (N France)	DAT (days)													
	0	**7**	**14**	**21**	**28**	**56**	**120**	**166**	**277**	**348**	**438**	**531**	**649**	**699**
T1	87.6	71.9	74.5	72.9	80.3	64.8	59.7	57.8	55.8	48.9	28.2	20.9	16.7	17.0
T2	83.8	73.9	66.7	77.4	67.2	68.1	50.5	54.9	43.2	43.3	30.5	27.4	19.8	20.5
T3	87.2	72.0	64.9	74.6	60.4	56.4	56.5	70.8	42.1	41.2	27.7	18.8	13.7	15.7
Mean	**86.2**	**72.6**	**68.7**	**76.6**	**69.2**	**63.1**	**55.5**	**61.1**	**47.0**	**44.4**	**28.8**	**22.3**	**16.7**	**17.7**

DAT = Days after treatment

Table 7.1.2.2.1- 125: Residues of M-01 in soil from the St. Etienne du Grès (France South) trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

St. Etienne du Grès (S France)	DAT (days)													
	0	**7**	**14**	**21**	**28**	**58**	**118**	**167**	**280**	**352**	**450**	**519**	**646**	**714**
T1	89.6	80.8	70.1	69.6	72.2	62.0	32.0	35.7	28.5	28.9	13.6	11.9	9.51	6.33
T2	78.2	80.3	75.1	73.8	83.1	67.1	43.1	36.1	30.2	35.7	23.2	11.9	6.31	9.36
T3	95.9	75.8	82.8	81.1	73.9	64.2	42.0	41.7	48.1	42.8	18.9	12.1	8.15	8.62
Mean	**87.9**	**79.0**	**76.0**	**74.8**	**76.4**	**64.4**	**39.0**	**37.8**	**35.6**	**35.8**	**18.6**	**12.0**	**7.98**	**8.09**

DAT = Days after treatment

Table 7.1.2.2.1- 126: Residues of M-01 in soil from the Albaro trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

Albaro (Italy)	DAT (days)											
	0	**6**	**14**	**21**	**27**	**57**	**121**	**175**	**282**	**364**	**450**	**541**
T1	94.7	88.4	69.6	72.0	68.2	49.7	49.1	33.8	35.8	19.9	4.85	0.95
T2	98.0	80.7	70.3	52.4	53.5	65.1	52.3	42.7	30.0	15.1	3.11	1.01
T3	108	63.6	79.7	79.9	119	49.6	57.2	46.0	31.4	16.4	4.25	1.06
Mean	**100**	**77.6**	**73.3**	**68.1**	**80.2**	**54.8**	**52.9**	**40.8**	**32.4**	**17.1**	**4.07**	**1.01**

DAT = Days after treatment

Table 7.1.2.2.1- 127: Residues of M-01 in soil from the Vilobi d'Onyar trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha)

Vilobi d'Onyar (Spain)	DAT (days)														
	0	**8**	**15**	**21**	**27**	**56**	**128**	**167**	**279**	**369**	**488**	**545**	**546**	**628**	**714**
T1	106	98.1	87.6	76.8	89.3	78.5	54.4	66.4	36.7	33.7	8.50	6.38	-	4.45	6.75
T2	111	98.2	108	83.2	72.2	72.0	52.7	39.5	28.8	20.2	7.13	8.50	9.19	3.79	2.77
T3	119	90.6	91.8	73.8	80.1	81.6	43.1	56.0	39.5	14.9	6.81	-	4.52	3.92	3.39
Mean	**112**	**95.6**	**95.8**	**77.8**	**80.5**	**77.4**	**50.1**	**54.0**	**35.0**	**19.6**	**7.50**	**6.40***		**4.05**	**4.30**

DAT = Days after treatment, *combined values of day 545 & 546 (mean of 4 values)

In Table 7.1.2.2.1- 128 to Table 7.1.2.2.1- 132. the M-01 datasets for the entire soil profile pre-processed according to the procedures described in FOCUS kinetics guidance (as described above) are presented.

Table 7.1.2.2.1- 128: Residues of M-01 in soil from the Burscheid trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

Burscheid (Germany)	DAT (days)													
	0	**7**	**15**	**21**	**28**	**63**	**121**	**171**	**259**	**395**	**436**	**518**	**605**	**701**
T1	98.9	87.5	134	115	140	85.5	35.0	50.9	41.6	28.9	22.0	20.1	17.6	12.5
T2	131	122	130	180	108	91.5	51.4	37.1	25.8	26.0	21.4	22.8	13.1	11.9
T3	94.7	108	108	144	137	92.5	67.6	51.6	34.8	38.3	30.6	21.5	18.8	13.8
Mean	**108**	**106**	**124**	**146**	**128**	**89.8**	**51.3**	**46.5**	**34.1**	**31.1**	**24.7**	**21.5**	**16.5**	**12.7**

DAT = Days after treatment

Table 7.1.2.2.1- 129: Residues of M-01 in soil from the Lignieres de Touraine (France North) trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

Lignieres de Touraine (N France)	DAT (days)													
	0	**7**	**14**	**21**	**28**	**56**	**120**	**166**	**277**	**348**	**438**	**531**	**649**	**699**
T1	87.6	72.5	75.1	78.4	80.9	65.4	60.3	59.5	56.3	49.4	28.7	21.5	17.3	17.5
T2	83.8	74.5	67.8	77.9	67.9	68.7	51.6	56.6	43.8	43.8	31.0	28.0	20.3	21.0
T3	87.2	72.6	65.5	75.2	60.7	56.9	57.2	71.3	42.9	41.7	28.2	19.3	14.8	16.8
Mean	**86.2**	**73.2**	**69.5**	**77.2**	**69.8**	**63.7**	**56.4**	**62.5**	**47.7**	**45.0**	**29.3**	**22.9**	**17.5**	**18.4**

DAT = Days after treatment

Table 7.1.2.2.1- 130: Residues of M-01 in soil from the St. Etienne du Grès (France South) trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

St. Etienne du Grès (S France)	DAT (days)													
	0	**7**	**14**	**21**	**28**	**58**	**118**	**167**	**280**	**352**	**450**	**519**	**646**	**714**
T1	89.6	81.3	70.6	78.3	72.7	62.6	32.5	36.4	29.0	29.6	14.1	12.5	10.1	6.85
T2	78.2	81.0	75.6	74.3	83.6	67.6	43.6	36.7	30.8	36.3	23.7	12.5	6.87	9.90
T3	95.9	76.3	83.4	81.6	74.4	64.7	42.5	42.3	48.7	43.4	19.4	12.6	8.71	9.17
Mean	**87.9**	**79.5**	**76.5**	**78.4**	**76.9**	**65.0**	**39.5**	**38.5**	**36.2**	**36.4**	**19.1**	**12.5**	**8.56**	**8.64**

DAT = Days after treatment

Table 7.1.2.2.1- 131: Residues of M-01 in soil from the Albaro trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

Albaro (Italy)	DAT (days)											
	0	**6**	**14**	**21**	**27**	**57**	**121**	**175**	**282**	**364**	**450**	**541**
T1	94.7	88.9	80.5	72.7	68.5	50.3	49.3	34.3	36.5	20.5	6.49	2.00
T2	98.3	81.6	70.1	55.8	54.9	65.9	52.6	43.3	30.6	15.6	4.15	2.04
T3	108	64.2	80.6	75.3	100	50.7	57.8	46.7	31.4	16.9	5.26	2.06
Mean	**100**	**78.2**	**73.7**	**67.9**	**80.5**	**55.6**	**53.2**	**41.4**	**32.8**	**17.7**	**5.30**	**2.03**

DAT = Days after treatment

Table 7.1.2.2.1- 132: Residues of M-01 in soil from the Vilobi d'Onyar trial after an application of 100 g a.s./ha, pre-processed values in entire soil profiles (0-110 cm) (expressed as g/ha) according to the FOCUS guidance document

Vilobi d'Onyar (Spain)	DAT (days)														
	0	**8**	**15**	**21**	**27**	**56**	**128**	**167**	**279**	**369**	**488**	**545**	**546**	**628**	**714**
T1	106	98.8	88.2	77.1	90.4	80.5	56.3	67.0	37.9	23.7	10.9	7.55	-	5.04	7.32
T2	111	98.8	109.0	83.8	72.8	72.5	52.7	40.1	29.4	20.8	9.33	7.21	10.3	5.00	3.38
T3	119	91.3	92.4	74.3	80.6	82.1	44.3	57.3	40.7	16.7	8.46	-	6.20	5.75	5.13
Mean	**112**	**96.3**	**96.5**	**78.4**	**81.3**	**78.4**	**51.1**	**54.8**	**36.0**	**20.2**	**9.63**	**7.82***		**5.26**	**5.28**

DAT = Days after treatment, *combined values of day 545 & 546 (mean of 4 values)

The residual amounts of the test item presented above (Table 7.1.2.2.1- 128 to Table 7.1.2.2.1- 132) were used as input data for determination of degradation kinetics using the software KinGUI 2. The measured initial concentration at day 0 was included in the parameter optimization procedure. Based on criterion for chi^2 error to be minimal and visual assessment the best fit kinetic model was chosen for the evaluation of the dissipation time. The calculation considered the quantifiable residues for the whole soil profiles expressed in [g/ha]. The results are summarized in Table 7.1.2.2.1- 133 with best fits highlighted in bold letters. The dissipation of M-01 could be described using a first order multi compartment kinetic model for test sites Burscheid (Germany), a double first order in parallel kinetic model for test site St. Etienne du Gres (France South) and a single first order kinetic model for test sites Lignieres-de-Touraine (France North), Albaro di Ronco all'Adige (Italy) and Vilobi d'Onyar (Spain). The best fit half-lives for M-01 were between 133 and 344 days for the test sites. Best fit kinetics are highlighted in bold.

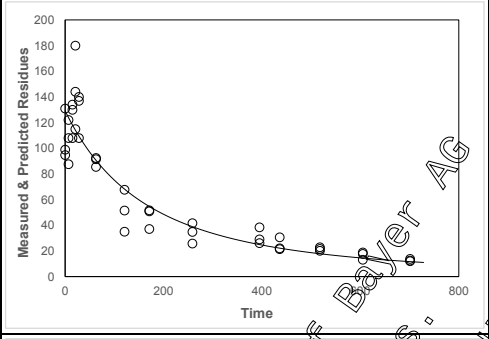
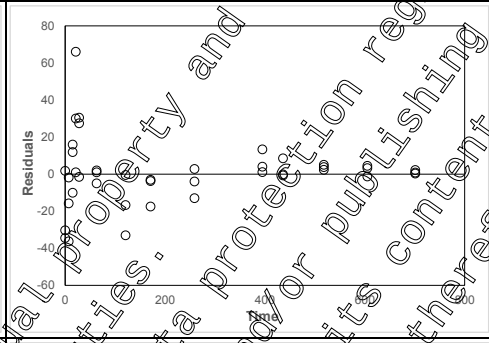
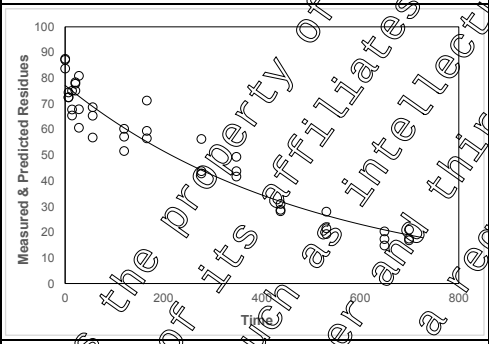
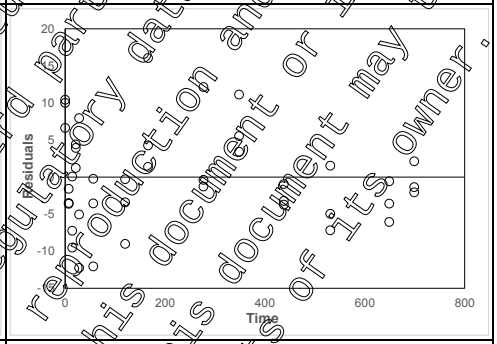
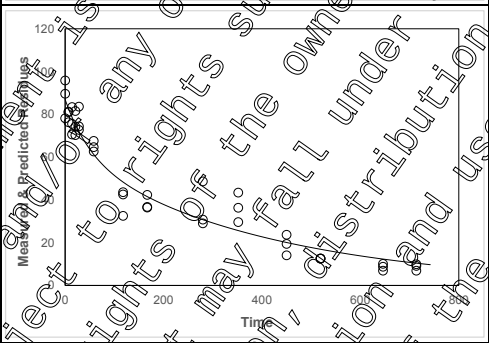
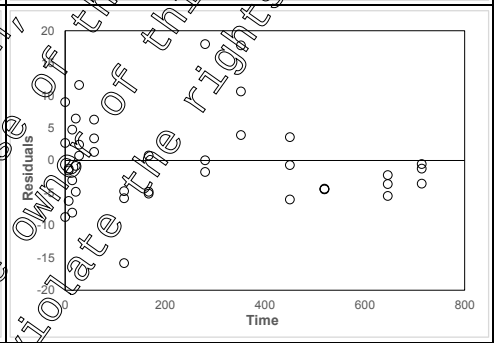
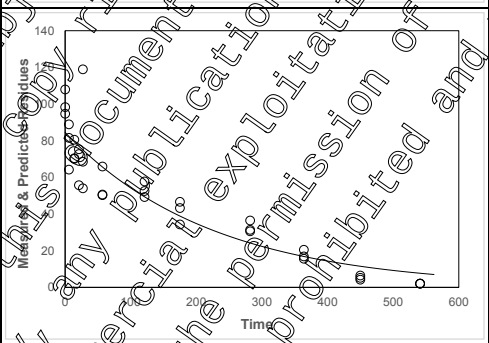
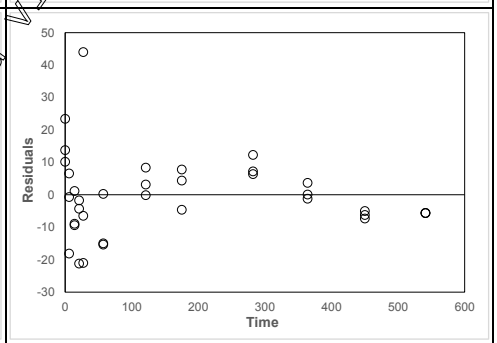
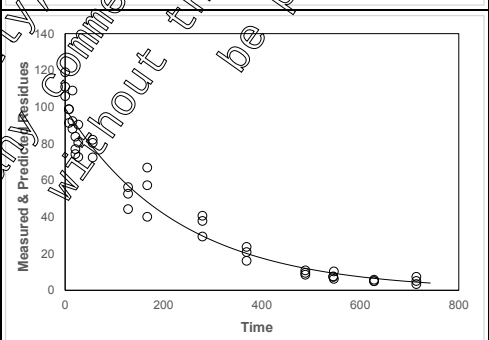
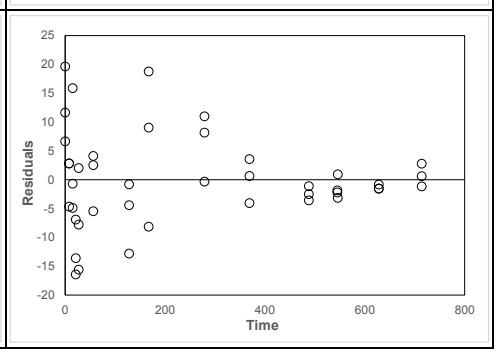
Table 7.1.2.2.1- 133: Degradation rate of fluopicolide under field conditions (DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Burscheid 15-2705-01	SFO	125.3	k 0.004468	17.6	1.77E-10	0.003407	0.006	155.1	515.4
	FOMC	**129.2**	**α 2.277 β 372.3**	**17.2**	**n.r. n.r.**	**-1.309 -413.9**	**5.863 1158**	**132.5**	**651.1**
	DFOP	129.6	k1 0.006776 k2 0.0001793 g 0.8658	17.4	0.0466 0.4846 n.r.	-0.0009369 -0.008861 0.08081	0.014 0.009 1.651	125.9	1639
Lignieres-de-Touraine 15-2703-03	**SFO**	**77.18**	**k 0.002015**	**7.47**	**<2e-16**	**0.001767**	**0.002**	**344**	**1143**
	FOMC	77.18	α 5547 β 2.75E+06	7.72	n.r. n.r	4855 2.75E+06	6239 2.75E+06	344	1143
	DFOP	86.2	k1 2.909 k2 0.001935 g 0.1276	6.68	<2e-16 2e-16 n.r.	2.909 0.001698 0.09003	2.909 0.002 0.165	287.7	1120
St. Etienne du Gres 15-2703-04	SFO	80.96	k 0.002391	10.4	<2e-16	0.002913	0.004	204.4	679
	FOMC	85.05	α 1.482 β 261.5	9.45	n.r. n.r.	0.3867 -27.37	2.578 550.3	155.9	974.6
	DFOP	**86.91**	**k1 0.01978 k2 0.002484 g 0.2927**	**8.89**	**0.0697 5.01E-06 n.r.**	**-0.005905 0.001527 0.05384**	**0.045 0.003 0.532**	**151.6**	**787.7**
Albaro di Ronco all'Adige 15-2706-02	**SFO**	**84.54**	**k 0.004439**	**12**	**1.23E-09**	**0.003353**	**0.006**	**156.2**	**518.8**
	FOMC	84.54	α 6785 β 1.53E+06	12.5	n.r. n.r.	5100 1.53E+06	8470 1.53E+06	156.2	518.8
	DFOP	100.3	k1 0.6752 k2 0.00408 g 0.2048	9.42	0.4272 6.30E-09 n.r.	-6.524 0.003024 0.07007	7.874 0.005 0.339	113.7	508.1
Vilobi d'Onyar 15-2706-03	**SFO**	**99.34**	**k 0.004325**	**9.64**	**<2e-16**	**0.003773**	**0.005**	**160.2**	**532.3**
	FOMC	99.68	α 237 β 5298	9.94	n.r. n.r.	-160.9 -37290	208.3 47890	157.2	540.5
	DFOP	112.2	k1 0.1313 k2 0.003954 g 0.1742	7.5	0.07851 <2e-16 n.r.	-0.04706 0.003382 0.0856	0.31 0.005 0.263	126.9	533.9

Best fit model highlighted in bold

A graphical representation of the final kinetic fits is shown below.

Table 7.1.2.2.1- 134: Graphical representations of best fit model

Soil Model Reference	Modelled vs observed	Residuals
Burscheid FOMC 15-2705-01		
Lignieres-de-Touraine SFO 15-2703-03		
St. Etienne du Gres DFOP 15-2703-04		
Albaro di Ronco all'Adige SFO 15-2706-02		
Vilobí d'Onyar SFO 15-2706-03		

III. Conclusion

M-01 was moderately to slowly degraded in soil at five trial sites in Northern and Southern Europe. Residue levels were in the range of 1.01 to 17.7 g/ha at study end. The dissipation of M-01 was best described by FOMC or DFOP kinetic models at two sites and SFO at three sites with best fit DT_{50} values ranging from 133 and 344 days. A six site was not considered acceptable to determine half-lives due to significant leaching out of the sampled soil depth.

Assessment and conclusion by applicant: The study is considered valid to assess test substance soil $DegT_{50matrix}$ values for field studies as defined by EFSA (2014). The endpoints may be too conservative to assess persistence as the design minimized soil surface processes as required by EFSA (2014) and such processes may contribute to dissipation.

Data Point:	KCA 7.1.2.2.1/22
Report Author:	[redacted]
Report Year:	2020
Report Title:	Fluopicolide - Kinetic evaluation of degradation in soil under field conditions to derive modelling endpoints - Six sites in three European countries (legacy studies)
Report No:	VC/19041B
Document No:	M-685676-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	None
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

$DegT50_{matrix}$ values for fluopicolide normalised to 20°C and pF2, were derived for use as modelling endpoints according to FOCUS Kinetics guidance (FOCUS, 2006 and 2014a) and the EFSA Guidance Document for deriving $DegT_{50}$ values (EFSA, 2014). The influence of surface processes, e.g. photodegradation and volatilisation, was minimised following EFSA recommendations (EFSA, 2014). The $DegT50_{matrix}$ values derived in this study represent degradation in the bulk soil matrix.

Fluopicolide was applied to bare soil in six terrestrial field dissipation trials in Germany, France, and Spain, at an application rate of 400 g/ha (Philippsburg, Rödelsee, Huntlosen, Valencia and Appilly sites) or 500 g/ha (Senas site). No cultivation, tillage or irrigation was carried out at these sites after application.

Kinetic analysis was performed for the transformed data sets using KinGUI v. 2.1. To minimise the influence of surface processes, in the first instance data points before 10 mm rainfall and irrigation were eliminated (EFSA, 2014). The selection of the most appropriate kinetic model was based on a detailed analysis, including visual assessment of the fit and residuals, χ^2error% and t-test significance, following EFSA recommendations (EFSA, 2014) and FOCUS guidance (2006 and 2014a).

Calculated $DegT50_{matrix}$ values for fluopicolide using the Single First Order (SFO) model are summarised in the table below:

Fluopicolide	Aerobic field conditions							
Soil type	**Location (country or USA state)**	**pH ($CaCl_2$)**	**Depth (cm)**	**DT_{50} (d) actual**	**DT_{90} (d) actual**	**St. (χ^2err) (%)**	**$DegT50_{matrix}$ (d; 20°C, pF2)**	**Method of calculation**
Loamy sand	Philippsburg (Germany)	6.4	0-50	-	-	9.477	199.6	SFO
Sandy clay loam	Rödelsee (Germany)	7.4	0-30	-	-	21.59	146.4	SFO
Sand	Huntlosen (Germany)	4.9	0-50	-	-	15.46	168.4	SFO
Loamy sand	Valencia (Spain)	7.3	0-30	-	-	13.95	317.4	SFO
Sandy silt	Appilly (France)	7.1	0-30	-	-	11.16	144.2	SFO
Sandy silt loam	Senas (France)	7.6	0-45	-	-	9.864	136.5	SFO
Geometric mean (if not pH dependent)							177	

In accordance with EFSA guidance (EFSA, 2014), no $DegT50_{matrix}$ values were derived for the metabolites of fluopicolide observed in these trials, as kinetic parameters derived for metabolites from legacy studies may not be wholly reflective of bulk soil matrix degradation.

I. Materials and Methods

The purpose of this study is to evaluate six legacy European field trials conducted at the field sites of Philippsburg, Rödelsee, Huntlosen, located in Germany, Valencia in Spain and Appilly and Senas in France to derive $DegT50_{matrix}$ values (normalised to 20°C and pF2) in the bulk soil matrix.

The datasets collected were evaluated following the EFSA Guidance Document for deriving $DegT_{50}$ values (EFSA, 2014), and the recommendations of the FOCUS Kinetics group (FOCUS, 2006 and 2014a). In accordance with EFSA guidance (EFSA, 2014), no $DegT50_{matrix}$ values were derived for the metabolites of fluopicolide observed in these trials, as kinetic parameters derived for metabolites from legacy studies may not be wholly reflective of bulk soil matrix degradation.

Details of the terrestrial field dissipation studies used in the kinetic evaluation are summarised in CA 7.1.2.2.1/01, CA 7.1.2.2.1/02, CA 7.1.2.2.1/03, CA 7.1.2.2.1/04, CA 7.1.2.2.1/08 and CA 7.1.2.2.1/09. At all sites, fluopicolide was applied onto bare soil in late spring/early summer. No cultivation or irrigation activities occurred during the trial periods, and no fertilisers were applied during the field phase of the studies. A summary of the trials is given in Table 7.1.2.2.1- 135.

Table 7.1.2.2.1- 135: Summary of terrestrial field dissipation studies

Document	Location	Day of application	Rate (g a.s./ha)	Soil Texture	pH ($CaCl_2$)	Duration (days)	Last sampling date
KCA 7.1.2.2.1/09, ([redacted] 2005a)	Philippsburg (Germany)	20/06/2000	400	Loamy sand	6.4	735	25/06/2002
KCA 7.1.2.2.1/01, ([redacted] 2003)	Rödelsee (Germany)	07/06/2000	400	Sandy clay loam	7.4	721	29/05/2002
KCA 7.1.2.2.1/02, ([redacted] 2003)	Huntlosen (Germany)	31/05/2000	400	Sand	4.9	722	23/05/2002
KCA 7.1.2.2.1/03, [redacted] (2004)	Valencia (Spain)	04/07/2001	400	Loamy sand	7.3	708	12/06/2003
KCA 7.1.2.2.1/08, ([redacted] 2005b)	Appilly (France)	16/06/2000	400	Sandy silt	7.1	735	21/06/2002
KCA 7.1.2.2.1/04, ([redacted] 2003)	Senas (France)	24/06/1999	500	Sandy silt loam	7.6	716	09/06/2001

In accordance with EFSA guidance (EFSA, 2014), data points prior to 10 mm rainfall were discarded from each data set to minimise the influence of soil surface processes such as photo-degradation and volatilisation. A summary of the data points removed from each data set is given in Table 7.1.2.2.1- 136.

Table 7.1.2.2.1- 136: Elimination of data points before 10 mm rainfall

Site	Weather station for precipitation	Actual day at which 10 mm rainfall reached	Normalised day at which 10 mm rain reached	Rain before this day (mm)	First experimental sample after 10 mm (normalised day)	Data points removed
Philippsburg	Site	5	3.0	3.4	12.8	d0,d1
Rödelsee	Site	18	18.1	3.8	27	d0,d1,d3,d4
Huntlosen	Site	4	3.0	0	3.9	d0,d1
Valencia	Alboraya	86	145.3		183.4	d0,d1,d3,d4,d5
Appilly	Site	16	11.1	3.4	20.6[a]	d0,d1,d3,d4 (d5)[a]
Senas	Site	36	56.5	5.8	90.9	d0,d1,d3,d4,d5

[a] Additional analysis was performed for the Appilly data set with the data point at 20.6 days also removed.

Soil samples were collected to a maximum depth of 45-70 cm, depending upon the test site. After sampling, the cores were cut into increments according to depth and analysed. These core samples were analysed for fluopicolide, with a limit of quantification LOQ of 0.005 mg/kg (dry weight) for all studies.

Where applicable, residue data from replicate subplots were processed separately. Residue data were checked for consistency and obvious outliers. Experimental data sets and data points were weighted equally in the kinetic analysis.

The Limit of Detection (LOD) was not reported for any of the data sets analysed; instead only the Limit of Quantification (LOQ) was given, with any residues below this level reported as "<LOQ". It has been assumed that residues reported as <LOQ were also <LOD, and these residues have been adjusted as outlined below, in keeping with the FOCUS Kinetics guidance (FOCUS, 2006 and 2014a) and also accounting for the movement of the test compound down the soil profile:

In the top horizon:

- Samples <LOQ just after a quantifiable amount were set to ½ LOQ.
- All subsequent samples <LOQ were omitted, unless later samples >LOQ were observed.

In lower horizons:

- No adjustments were made in lower horizons until the first sample >LOQ was observed below the top horizon.
- Thereafter:
  - Samples <LOQ just before or just after a quantifiable amount in the same layer were set to ½ LOQ.
  - All subsequent samples <LOQ were omitted, unless later samples >LOQ were observed.
  - Where a sample >LOQ in a deeper layer was reported, samples <LOQ in higher layers at the same time point were set to ½ LOQ.
  - An additional adjustment was made to the shallowest residue-free layer at every time point, which was set to ½ LOQ.

In all cases, adjustments were made only to layers where samples had been analysed.

Residue values were given in the original study reports in units of mg/kg (dry weight). Insufficient data were available to convert these residues to units of g/ha, therefore the kinetic analysis was performed using residues averaged across all relevant soil layers, with the contributions from each layer weighted to account for the depth of the layer, expressed in units of mg/kg (dry weight).

Kinetic calculations for the degradation of fluopicolide in field soils were performed using KinGUI 2.1 with three kinetic models – SFO, FOMC and DFOP. The goodness of fit with each model was evaluated based on visual assessment and chi-square test, and the degradation rate was then also evaluated via the t-test. The degradation rates for persistence trigger and exposure modelling were then determined from an acceptable kinetic fit following FOCUS and EFSA guidance.

II. Results and Discussion

Following the EFSA decision tree an initial SFO fit was performed for each data set after eliminating all data points before 10 mm rain. For five of the six trial sites, an acceptable fit was obtained at this step using the SFO model.

For the Appilly site, a further fit was performed using the DFOP model with the complete data set (i.e. no data points eliminated). The parameter g_a which defines the fraction of mass applied to the fast-degrading compartment in the DFOP model, exceeded the threshold of 0.75 recommended in the EFSA guidance, and the DFOP fit was therefore not accepted.

Subsequently, the complete data set for the Appilly site was fitted using the HS model. The optimised breakpoint, tb, occurred at 0.7 days, i.e. before 10 mm rain had occurred, and the HS fit was therefore not accepted. The breakpoint parameter, tb, was subsequently fixed to the normalised time at which rainfall exceeded 10 mm (11.1 days), however this resulted in a fit that was visually unacceptable.

Finally, an acceptable fit was obtained for the Appilly site using the SFO model, excluding all data up to and including 20.6 days (i.e. removing one additional time point). It is proposed that the $DegT_{50}$ value derived from this fit provides a robust description of degradation in the bulk soil matrix at this test site, i.e. the pattern of decline in later residues is appropriately described. The results are summarised in Table 7.1.2.2.1-137.

Table 7.1.2.2.1- 137: Results for fluopicolide: summary of kinetic analysis

Kinetic model	DT_{50}	DT_{90}	VA	χ^2err	k_1 / α	k_2 / β	t_b / g	t-test k_1 / k_2	MS
	(d)			(%)	(1/d / -)	(1/d / -)	(d / -)	(-)	
Philippsburg									
SFO[a]	199.6	663	+	9.477	0.0034732	-	-	3.00E-08	M
Roedelsee									
SFO[A]	146.4	486.2	o	21.59	0.0047355	-	-	7.57E-07	M
Huntlosen									
SFO[A]	168.4	559.4	+	15.46	0.0041161	-	-	1.64E-06	M
Valencia									
SFO[A]	317.4	1055	+	13.95	0.0021835	-	-	4.83E-07	M
Appilly									
SFO[A]	83.04	275.8	-	18.59	0.0083474	-	-	3.41E-11	
DFOP[B]	87.4	1.66E+13	+	13.84	0.01005	2.22E-14	0.8554	0.187 / 0.5	
HS[B]	92.05	329	o	14.6	0.1036037	0.0067937	0.6999999	0.388 / 1.41E-08	
HS[B,C]	103.5	318.1	-	13.94	2.22E-14	0.007501	11.1	0.5 / 3.31E-08	
SFO[D]	144.2	479	o	11.16	0.0048073	-	-	2.54E-09	M
Senas									
SFO[A]	136.5	453.6	+	9.864	0.0050766	-	-	2.09E-11	M

[A] – Data points before 10 mm rainfall eliminated
[B] – All data points included
[C] – Breakpoint (t_b) fixed to 11.1 days
[D] – Data up to and including 20.6 days (normalised time) excluded

A summary is given in Table 7.1.2.2.1-138 of the $DegT50_{matrix}$ values derived for fluopicolide for use as modelling endpoints. These values are normalised to 20°C and pF2.

Table 7.1.2.2.1- 138: $DegT50_{matrix}$ values for fluopicolide, normalised to 20°C and pF2, for use as modelling endpoints

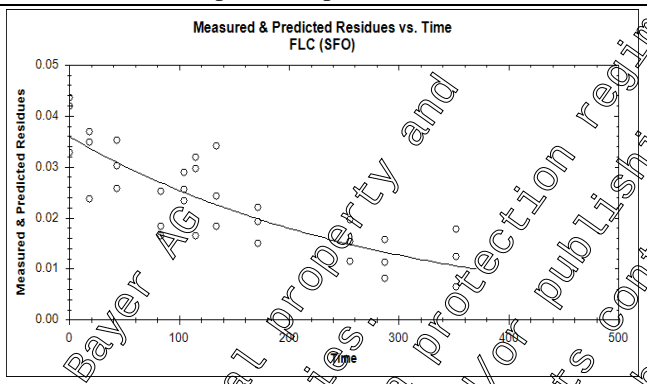
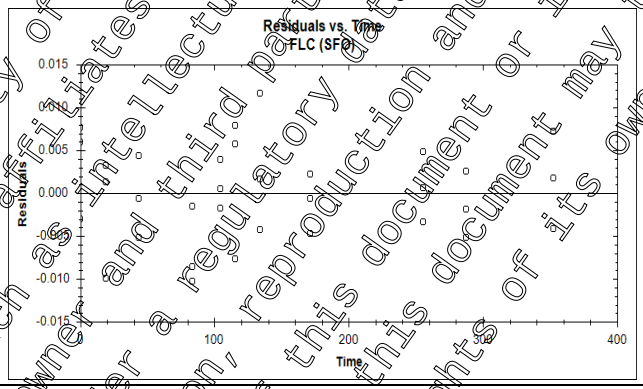
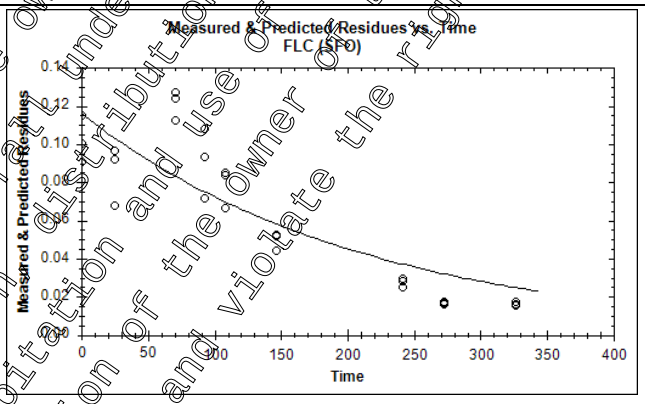
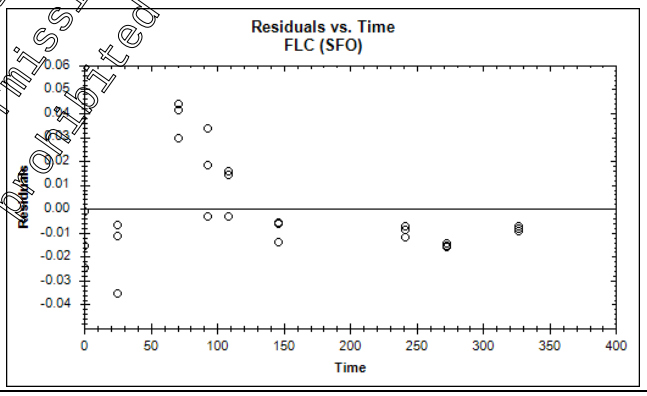
Soil	Kinetic model	$DegT50_{matrix}$ (days)
Philippsburg (Germany)	SFO	199.6
Rödelsee (Germany)	SFO	146.4
Huntlosen (Germany)	SFO	168.4
Valencia (Spain)	SFO	317.4
Appilly (France)	SFO	144.2
Senas (France)	SFO	136.5
Geometric mean		**177**

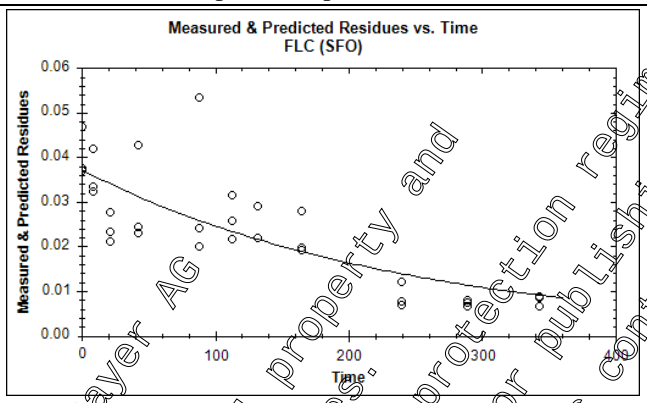
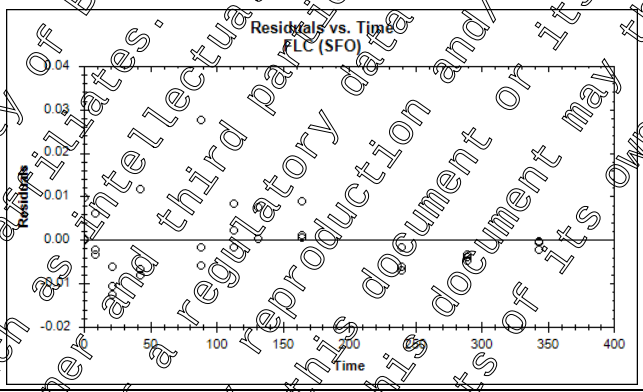
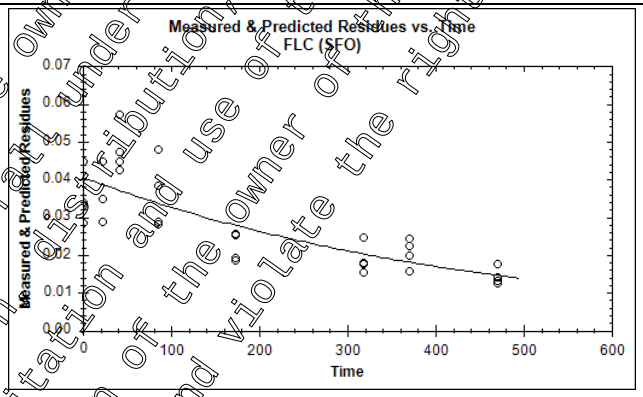
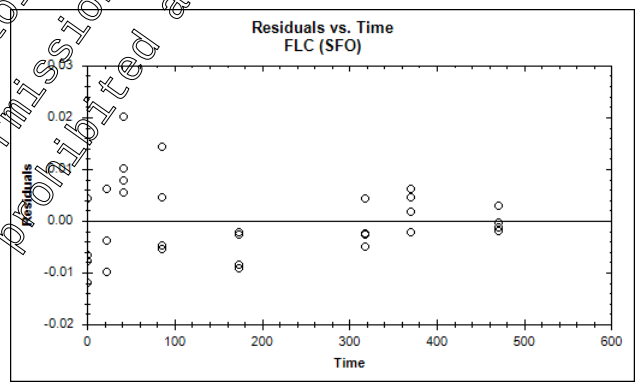
The standard EFSA template can be seen in Table 7.1.2.2.1- 139 and graphical representations in Table 7.1.2.2.1- 140

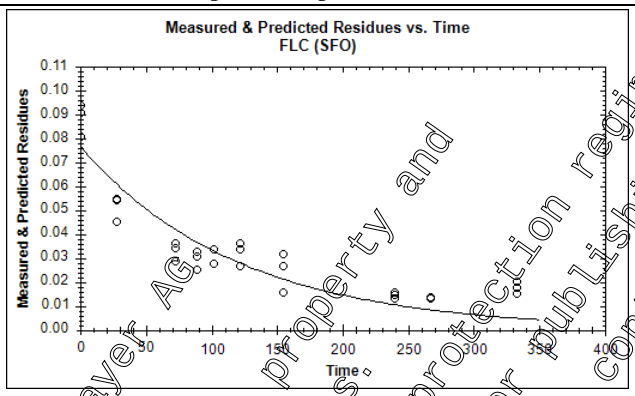
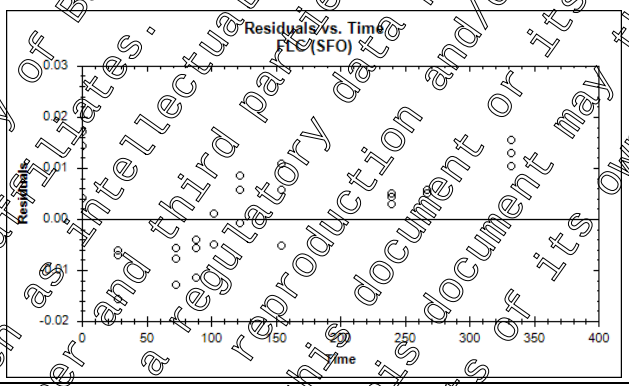
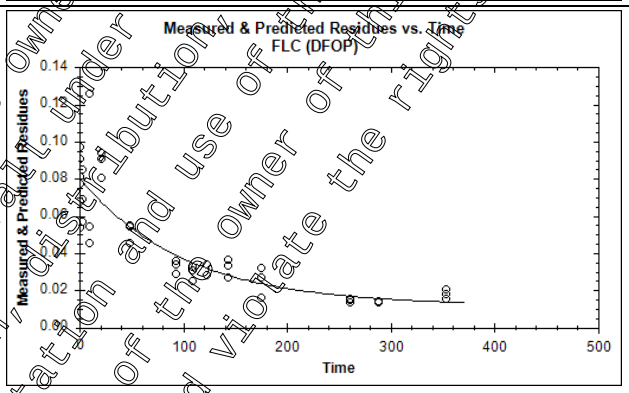
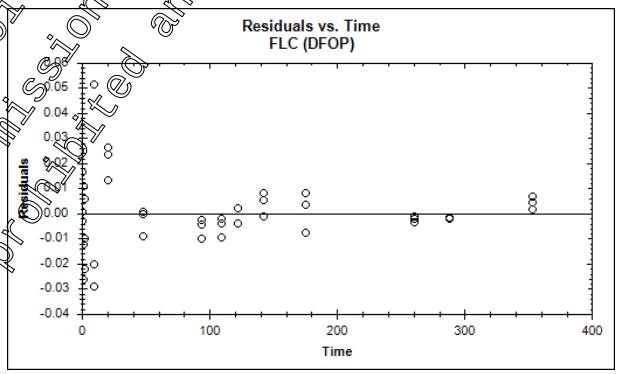
Table 7.1.2.2.1- 139: Standard EFSA template for kinetic fitting

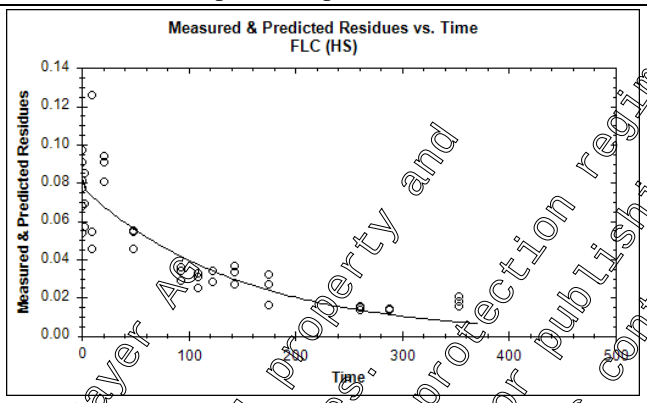
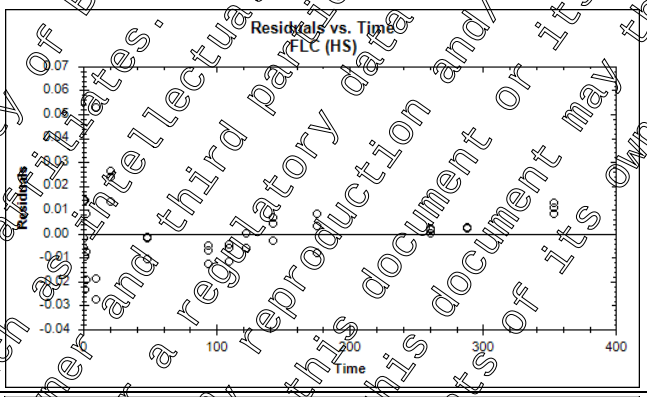
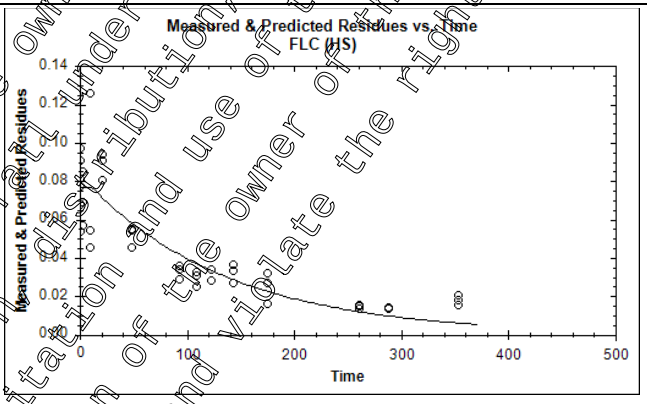
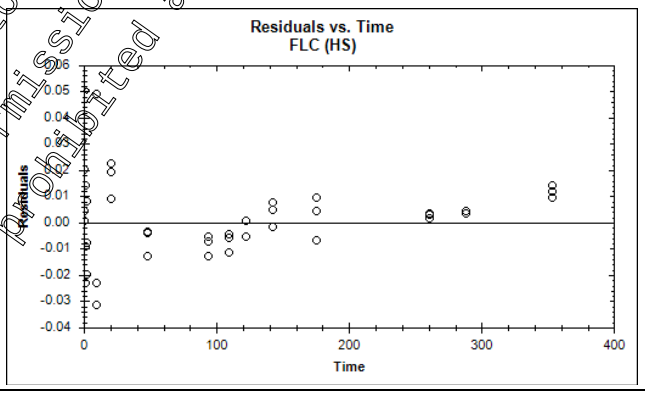
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Philippsburg	SFO	0.1	k = 0.0034732	9.477	3e-08	0.0319968	0.040	199.6	663
Rödelsee	SFO	0.1	k = 0.0047355	21.59	7.57e-07	0.0994397	0.132	146.4	486.2
Huntlosen	SFO	0.1	k = 0.0041161	15.46	1.64e-6	0.0320969	0.042	168.4	559.4
Valencia	SFO	0.1	k = 0.0021835	13.95	4.83E-07	0.0362463	0.045	317.4	1000
Appilly	*SFO*	*0.1*	*k = 0.0083474*	*18.59*	*3.41e-11*	*0.0683718*	*0.086*	*83.04*	*275.8*
	DFOP (all data)	*0.1*	*k 1.005e-02 k2 2.220e-14 g 8.554e-01*	*13.84*	*0.187 0.500*	*7.188e-02 - -8.464e-01*	*0.089 - 2.55*	*87.4*	*>1000*
	HS (all data)	*0.1*	*k 0.1036037 k2 0.067937 tb 0.6999999*	*14.6*	*0.388 1.41e-08 -*	*0.0666507 -4.5861556*	*0.100 - -5.986*	*92.05*	*329*
	HS (all data; tb fixed)	*0.1*	*k 2.221e-14 k2 7.501e-03*	*13.94*	*0.5 3.31e-08*	*6.646e-02*	*0.087*	*103.5*	*318.1*
	SFO (data up to and including 20.6 days excluded)	0.1	k = 0048073	11.16	2.54e-09	0.0415751	0.051	144.2	479
Senas	SFO	0.1	k = 0.0050766	9.864	2.09e-11	0.0238824	0.029	136.5	453.6

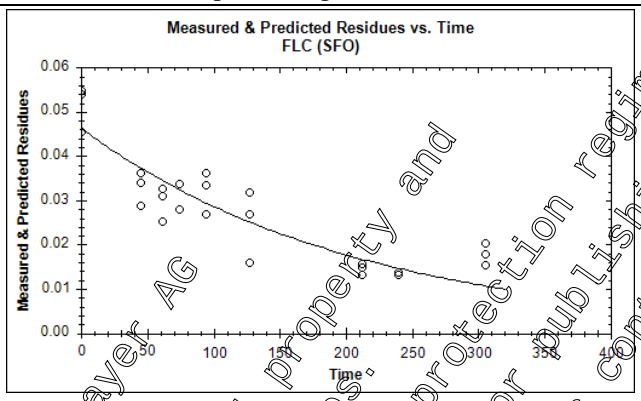
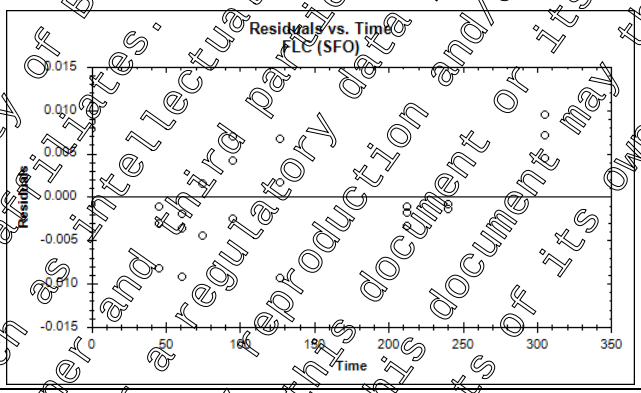
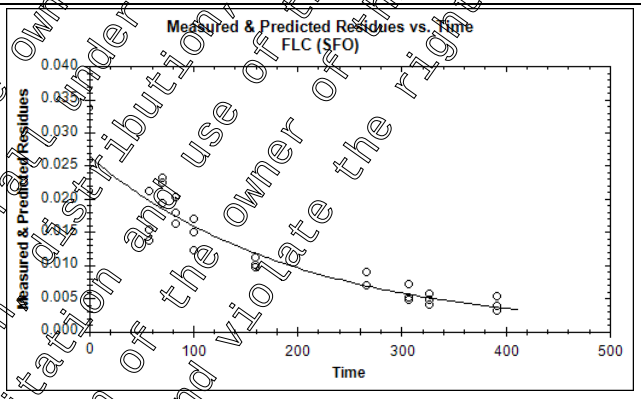
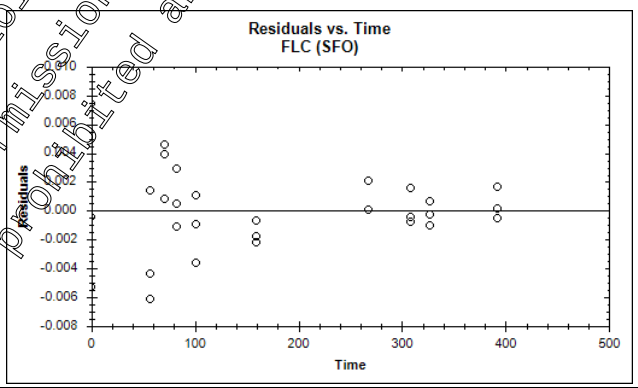
Table 7.1.2.2.1- 140: Graphical representations of best fit models

Trial / Best Fit Model	Graphical Representations
Philippsburg (Germany) / SFO	 
Rödelsee (Germany) / SFO	 

Trial / Best Fit Model	Graphical Representations
Huntlosen (Germany) / SFO	 
Valencia (Spain) / SFO	 

Trial / Best Fit Model	Graphical Representations
Appilly (France) / SFO	 
Appilly (France) / DFOP (all data)	 

Trial / Best Fit Model	Graphical Representations
Appilly (France) / HS (all data)	 
Appilly (France) / HS (all data, tb fixed)	 

Trial / Best Fit Model	Graphical Representations
Appilly (France) / SFO (data up to and including 20.6 days excluded)	 
Senas (France) / SFO	 

III. Conclusion

The data from the six 'legacy' field dissipation trials was evaluated to derived modelling endpoints for fluopicolide. Data points before 10 mm rainfall and irrigation were eliminated to minimise any influence from surface processes as advised by EFSA, 2014.

$DegT50_{matrix}$ values for fluopicolide, normalised to 20°C and pF2, were derived for use as modelling endpoints according to FOCUS Kinetics guidance (FOCUS, 2006 and 2014a) and the EFSA Guidance Document for deriving $DegT_{50}$ values (EFSA, 2014).

A geometric mean value $DegT50_{matrix}$ of 177 days was calculated for use in FOCUS calculations.

Assessment and conclusion by applicant:

The modelling report was conducted according to FOCUS Degradation Kinetics (2006, 2014) and is considered valid to assess best fit and modelling DT_{50} values for fluopicolide in field dissipation studies.

Data Point:	KCA 7.1.2.2.1/2300
Report Author:	[redacted]
Report Year:	2020
Report Title:	Fluopicolide and its metabolite M-01 - Kinetic evaluation of the degradation in soil under field conditions for modelling purposes - Six trials in Five European countries
Report No:	VC/19/041A
Document No:	M-685675-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	None
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

Normalised (20°C and pF2) degradation DT_{50} matrix values of fluopicolide and its metabolite M-01 (AE C653711) in the soil under European field conditions were derived for modelling purpose according to FOCUS kinetics (FOCUS, 2006; 2014a) and EFSA guidance on field dissipation studies (EFSA, 2014). Processes potentially occurring at the soil surface, e.g. photo-degradation and volatilisation, during the field study were eliminated to resulting in a DT_{50} matrix representing the degradation in the soil.

Fluopicolide and M-01 (AE C653711) were applied to bare soil at six trial sites, followed by incorporation to 7 cm, at nominal rates of 400 and 100 g/ha, respectively. The field trials were carried out in 2015 - 2017 in Germany (Burscheid); UK (Great Chishill); France (Lignieres de Touraine and St. Etienne du Grès); Spain (Vilobi); and Italy (Albaro).

Daily soil temperatures and moisture contents, estimated with PEARL, were used to normalise the evaluated parameters to reference conditions according to FOCUS groundwater assumptions (20°C and pF2; Arrhenius activation energy, Ea 65.4 KJ Mol^{-1}; Walker equation, B=0.7) (FOCUS, 2014a, c). The residue data together with the transformed incubation times (transformed time approach, time step

normalisation) were kinetically and statistically evaluated, based on the procedure explained by FOCUS kinetics, using the software tool KinGUI 2.1.

The model fit as well as the statistical evaluation of the results were carried out with the software KinGUI, version 2.1. The selection of the most appropriate kinetic model was based on a detailed statistical analysis including visual assessment, χ2err statistics, randomness of residuals, and t-test significance following the FOCUS guidance (2006; 2014a).

Calculated DT_{50} and DT_{90} values for fluopicolide and M-01 using the Single First Order (SFO) model under normalised conditions are summarised in the table below:

Fluopicolide and M-01 Field matrix degradation endpoints for modelling purpose; normalised to 20°C and pF2

	Aerobic field conditions							
Soil type	**Location (country)**	**pH ($CaCl_2$)**	**Depth (cm)**	**DT_{50} (d) actual**	**DT_{90} (d) actual**	**St. (χ²err) (%)**	**DT_{50} matrix (d) norm**	**Method of calculation**
Silt loam	Burscheid (Germany)	5.9	0-120	111.9	371.8	9.80	111.9	SFO
Clay	Great Chishill (UK)	7.8	0-120	216.9	720.4	11.64	216.9	SFO
Sandy loam	Lignieres de Touraine (France North)	6.9	0-120	158.6	526.8	4.82	158.6	SFO
Clay loam	St.Etienne du Grès (France South)	8.1	0-120	303.2	>1000	4.90	303.2	SFO
Clay loam	Albaro di Ronco all'Adige (Italy)	7.7	0-120	237.3	788.5	9.99	237.3	SFO
Sandy clay loam	Vilobi d'Onyar (Spain)	6.9	0-120	166.8	554.0	6.20	166.8	SFO
Fluopicolide Geometric mean							**189**	
Silt loam	Burscheid (Germany)	5.9	0-120	94.0	312.3	14.68	94.0	SFO
Sandy loam	Lignieres de Touraine (France North)	6.9	0-120	191.1	643.9	7.82	191.1	SFO
Clay loam	St.Etienne du Grès (France South)	8.1	0-120	179.9	597.5	5.87	179.9	SFO
Clay loam	Albaro di Ronco all'Adige (Italy)	7.7	0-120	151.8	504.3	13.93	151.8	SFO
Sandy clay loam	Vilobi d'Onyar (Spain)	6.9	0-120	136.2	452.7	10.94	136.3	SFO
M-01 Geometric mean							**146**	

I. Materials and Methods

The behaviour of fluopicolide and M-01 were investigated in six terrestrial field soil dissipation studies designed to determine the dissipation under representative European field conditions. The modelling analysis was based on residue data from studies conducted at the field sites of Burscheid, located in Germany, Great Chishill in UK, Lignieres de Touraine and St. Etienne du Grès, both located in France, Albaro di Ronco all'Adige in Italy, Vilobi d'Onyar in Spain (Table 7.1.2.2.1- 141).

Table 7.1.2.2.1- 141: Geographical locations of field trials

Site	Latitude	Longitude
Burscheid, Germany [A]	[redacted]	[redacted]
Great Chishill, United Kingdom	[redacted]	[redacted]
Lignieres de Touraine, France North	[redacted]	[redacted]
St. Etienne du Grès, France South	[redacted]	[redacted]
Albaro, Italy	[redacted]	[redacted]
Vilobi, Spain	[redacted]	[redacted]

[A] Coordinates taken from Appendix 25 of the field dissipation report

The evaluation was conducted to derive kinetic parameters suitable for modelling purpose according to FOCUS kinetics (FOCUS, 2006, 2014a) and EFSA guidance on field dissipation studies (EFSA, 2014). It includes a time-step normalisation to standard reference conditions for soil temperature (20°C and pF2), as well as a quality check of the results. Processes potentially occurring at the soil surface, e.g. photodegradation and volatilisation, during the field study should be eliminated to result finally in a $DegT_{50}$ representing the degradation in the soil.

In all sites fluopicolide and M-01 were applied onto bare soil in late spring (May-June) followed by incorporation to 7 cm in order to minimise surface processes. The plots were not cropped but they received irrigation during the trial periods. No fertilisers were applied to the trials during the field phase of the study. A summary of the studies can be seen in Table 7.1.2.2.1- 142.

Details of the terrestrial field dissipation studies used in the kinetic evaluation are summarised in KCA 7.1.2.2.1/12 and KCA 7.1.2.2.1/13 for fluopicolide and KCA 7.1.2.2.1/18 to KCA 7.1.2.2.1/20 for M-01. In all treated plots of the trials the samples were taken to a maximum depth of 110 cm for up to 2 years and split into 10 cm layers for analysis. For the M-01 trial at Great Chishill, UK, significant movement of residues to deeper soil layers occurred, resulting in no residue-free layer down to 110 cm. Thus, this trial is not considered suitable for soil matrix DT_{50} determination and has been excluded from further evaluation.

Table 7.1.2.2.1- 142: Summary of terrestrial field dissipation studies

Document	Location	Day of application	Rate (g a.s./ha)	Soil Texture	pH ($CaCl_2$)	Duration (days)	Last sampling date
Fluopicolide							
CA 7.1.2.2.1/12 & CA 7.1.2.2.1/14, [redacted] 2019)	Burscheid (Germany)	11/05/2015	451	Silt loam	5.9	701	11/04/2017
CA 7.1.2.2.1/12 & CA 7.1.2.2.1/14, [redacted] 2019)	Great Chishill, (United Kingdom)	27/05/2015	312	Clay	7.8	751	16/06/2017
CA 7.1.2.2.1/12 & CA 7.1.2.2.1/14, [redacted] 2019)	Lignieres de Touraine (France North)	28/05/2015	356	Sandy loam	6.9	700	27/04/2017
CA 7.1.2.2.1/13 & CA 7.1.2.2.1/14, [redacted] 2019)	St. Etienne du Grès, (France South)	12/05/2015	381	Clay loam	8.1	714	25/04/2017
CA 7.1.2.2.1/13 & CA 7.1.2.2.1/14, [redacted] 2019)	Albaro (Italy)	20/05/2015	412	Clay loam	7.7	722	11/05/2017
CA 7.1.2.2.1/13 & CA 7.1.2.2.1/14, [redacted] 019)	Vilobi, (Spain)	03/06/2015	391	Sandy clay loam	6.9	714	17/05/2017
M-01							
CA 7.1.2.2.1/18 & CA 7.1.2.2.1/21, [redacted] 2019)	Burscheid (Germany)	11/05/2015	108	Silt loam	5.9	701	11/04/2017
CA 7.1.2.2.1/18 & CA 7.1.2.2.1/21, [redacted] 2019)	Great Chishill, (United Kingdom)	27/05/2015	59.8	Clay	7.8	744	09/06/2017
CA 7.1.2.2.1/19 & CA 7.1.2.2.1/21, [redacted] 2019)	Lignieres de Touraine (France North)	28/05/2015	86.2	Sandy loam	6.9	699	26/04/2017
CA 7.1.2.2.1/19 & CA 7.1.2.2.1/21, [redacted] 2019)	St. Etienne du Grès, (France South)	12/05/2015	87.9	Clay loam	8.1	714	25/04/2017
CA 7.1.2.2.1/20 & CA 7.1.2.2.1/24, [redacted] 019)	Albaro (Italy)	19/05/2015	100	Clay loam	7.7	541	11/11/2016
CA 7.1.2.2.1/20 & CA 7.1.2.2.1/21, [redacted] 2019)	Vilobi, (Spain)	03/06/2015	112	Sandy clay loam	6.9	714	17/05/2017

The reported residue data (KCA 7.1.2.2.1/12, KCA 7.1.2.2.1/13, KCA 7.1.2.2.1/18, KCA 7.1.2.2.1/19 and KCA 7.1.2.2.1/20) had previously been processed according to FOCUS (2006; 2014a) and used to derive un-normalised endpoints (KCA 7.1.2.2.1/14 and KCA 7.1.2.2.1/21).

Current kinetic calculations for the degradation of fluopicolide in field soils were performed using KinGUI 2.1 with three kinetic models – SFO, FOMC and DFOP. The goodness of fit with each model was evaluated based on visual assessment and chi-square test, and the degradation rate was then also evaluated via the t-test. The degradation rates for persistence trigger and exposure modelling were then determined from an acceptable kinetic fit following FOCUS and EFSA guidance.

II. Results and Discussion

Normalised (20°C and pF2) degradation $DT_{50\ matrix}$ values of fluopicolide and M-01 in the soil matrix under European field conditions were derived for modelling purpose according to FOCUS kinetics (FOCUS 2006; 2014a) and the EFSA guidance on field dissipation studies (EFSA 2014). Processes potentially occurring at the soil surface, e.g. photo-degradation, volatilisation, during the field study were eliminated to result finally in a $DT_{50\ matrix}$ representing the degradation in the soil matrix or bulk.

Simulated (with PEARL) daily soil temperatures and moisture were used to normalise the evaluated parameters to reference conditions according to FOCUS groundwater assumptions (Arrhenius activation energy, Ea = 65.4 KJ/Mol; Walker equation, B = 0.7) (European Commission 2014; FOCUS 2014b). The residue data together with the transformed times (transformed time approach, time step normalisation) were kinetically and statistically evaluated, based on the procedure explained by FOCUS kinetics, using KinGUI 2.1.

According to the FOCUS decision tree (FOCUS, 2006, 2014a), the kinetic evaluation was started by assuming a simple first-order (SFO) degradation for the parent compound in soil (Table 7.1.2.2.1- 143).

Table 7.1.2.2.1- 143: Data for fluopicolide and M-01: kinetic and statistical results of the SFO

Kinetic model	DT_{50}	DT_{90}	VA	χ^2err	k_1 / α	k_2 / β	t_b / g	t-test k_1 / k_2
	(d)			(%)	(1/d / -)	(1/d / -)	(d / -)	(-)
Burscheid (Germany)								
FLC SFO	111.9	371.8	+	9.80	6.194e-03	-	-	3.64e-15
M-01 SFO	94.0	312.3	+	14.68	7.373e-03	-	-	6.27e-13
Great Chishill (UK)								
FLC SFO	216.9	720.4	+	11.64	3.196e-03	-	-	2.34e-11
Lignieres de Touraine (France North)								
FLC SFO	158.8	526.8	+	4.82	4.371e-03	-	-	<2e-16
M-01 SFO	191.1	643.9	+	7.82	3.627e-03	-	-	<2e-16
St.Etienne du Grès (France South)								
FLC SFO	303.2	> 1000	+	4.90	2.286e-03	-	-	<2e-16
M-01 SFO	179.9	597.5	+	5.87	3.854e-03	-	-	<2e-16
Albaro di Ronco all'Adige (Italy)								
FLC SFO	257.3	788.3	+	9.99	2.921e-03	-	-	<2e-16
M-01 SFO	151.8	504.3	o	13.93	4.566e-03	-	-	4.67e-10
Vilobi d'Onyar (Spain)								
FLC SFO	166.8	554.0	+	6.20	4.156e-03	-	-	<2e-16
M-01 SFO	136.2	452.7	+	10.94	5.087e-03	-	-	<2e-16

VA = visual assessment, + = good fit, o = acceptable fit, - = non accepytable fit

In all the sites matrix degradation of fluopicolide and M-01 is sufficiently well described assuming a simple first-order decay.

A summary is given in Table 7.1.2.2.1- 144 of the geometric mean $DT_{50matrix}$ values derived for fluopicolide and M-01 for use as modelling endpoints. These values are normalised to 20°C and pF2. The overall geometric mean $DT_{50\ matrix}$ of fluopicolide for modelling purposes according to FOCUS kinetics and EFSA (2014) based on these six values, can be given as 189 days. The overall geometric mean $DT_{50\ matrix}$ of M-01 for modelling purposes according to FOCUS kinetics and EFSA (2014) based on these five values, can be given as 146 days.

Table 7.1.2.2.1- 144: Estimated field matrix degradation of fluopicolide and M-01 for modelling purposes, normalised to 20°C and pF2

Soil	Kinetic model	$DT_{50\ matrix}$ (d)
Burscheid (Germany)	SFO	111.9
Great Chishill (UK)	SFO	216.9
Lignieres de Touraine (France North)	SFO	158.8
St.Etienne du Grès (France South)	SFO	303.2
Albarodi Ronco all'Adige (Italy)	SFO	237.3
Vilobi d'Onyar (Spain)	SFO	166.8
Geometric mean		**189**
Soil	**Kinetic model**	**$DT_{50\ matrix}$ (d)**
Burscheid (Germany)	SFO	94.0
Lignieres de Touraine (France North)	SFO	191.1
St.Etienne du Grès (France South)	SFO	179.9
Albarodi Ronco all'Adige (Italy)	SFO	151.8
Vilobi d'Onyar (Spain)	SFO	136.3
Geometric mean		**146**

The standard EFSA template can be seen in Table 7.1.2.2.1- 145 for fluopicolide and Table 7.1.2.2.1-146 for M-01.

Table 7.1.2.2.1- 145: Standard EFSA template for kinetic fitting fluopicolide

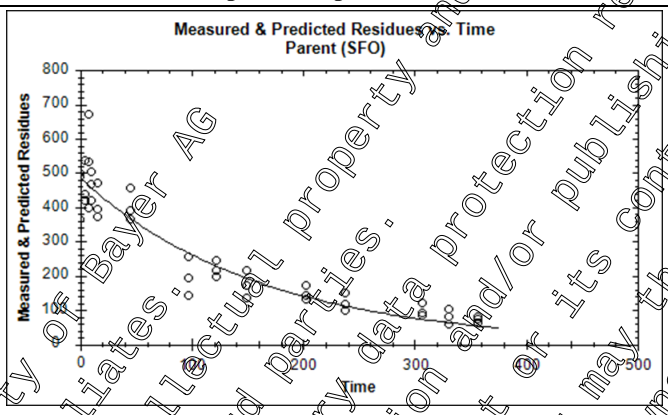
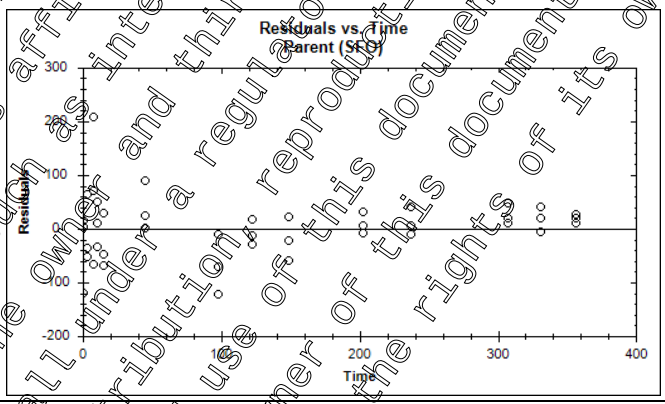
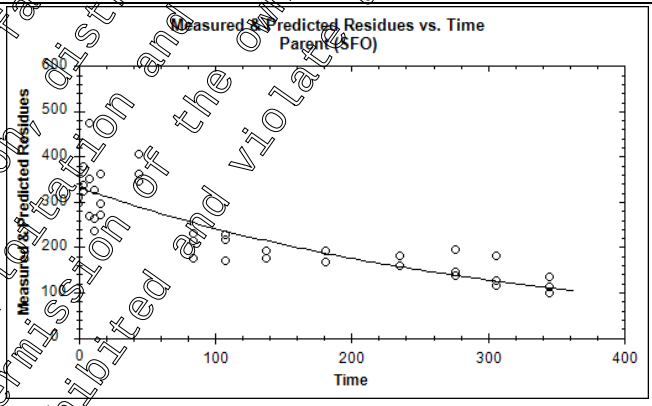
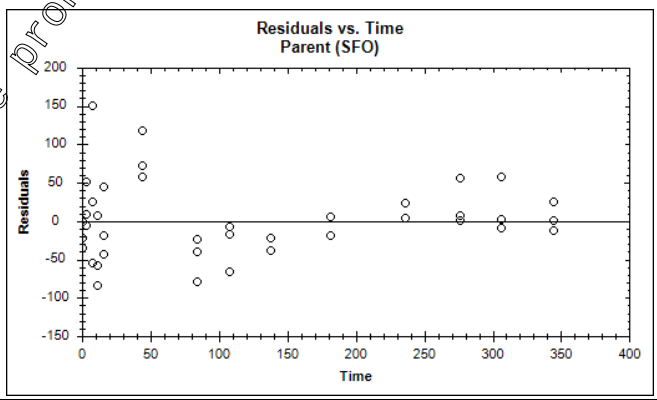
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Burscheid (Germany), KCA 7.1.2.2.1/12, ([redacted] 2019)	SFO	484.7	k 0.006194	9.8	3.64E-15	0.005184	0.007	111.9	371.8
Great Chishill (UK), KCA 7.1.2.2.1/12, ([redacted] 2019)	SFO	331.5	k 0.003196	11.6	2.34E-11	0.002494	0.004	216.9	720.4
Lignieres de Touraine (France North), KCA 7.1.2.2.1/12, ([redacted] 2019)	SFO	335	k 0.004371	4.82	<2e-16	0.004039	0.005	158.6	526.8
St.Etienne du Grès (France South), KCA 7.1.2.2.1/13, ([redacted] 2019)	SFO	365.1	k 0.002286	4.9	<2e-16	0.00208	0.002	303.2	1007
Albaro di Ronco all'Adige (Italy), KCA 7.1.2.2.1/13, ([redacted] 2019)	SFO	371.3	k 0.002921	9.99	<2e-16	0.002496	0.003	237.3	788.3
Vilobi d'Onyar (Spain), KCA 7.1.2.2.1/13 ([redacted] 2019)	SFO	361.5	k 0.004156	6.2	<2e-16	0.003841	0.004	166.8	554

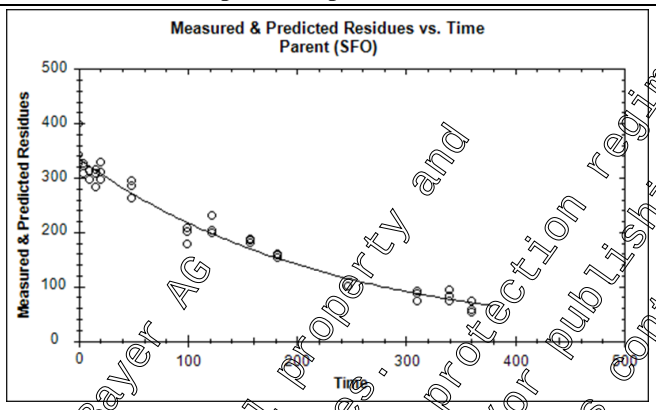
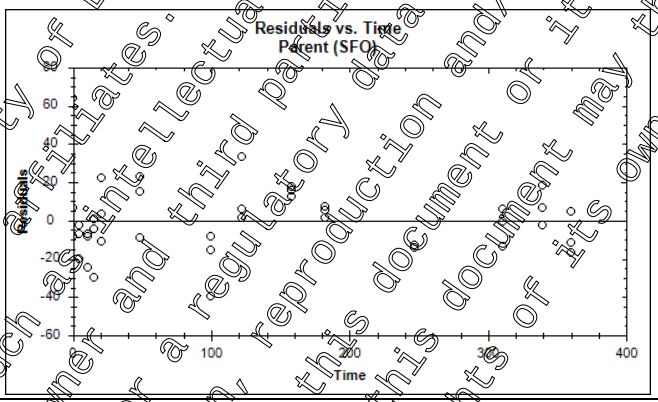
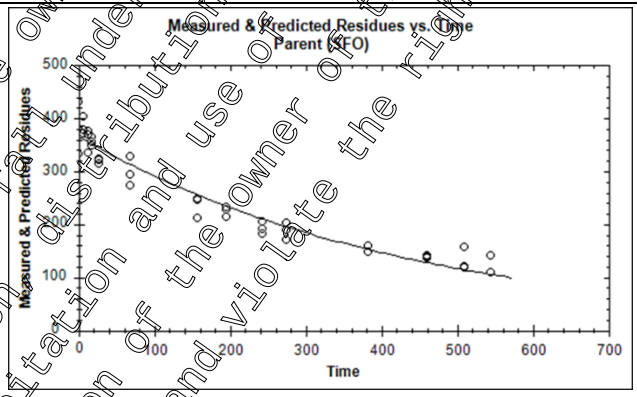
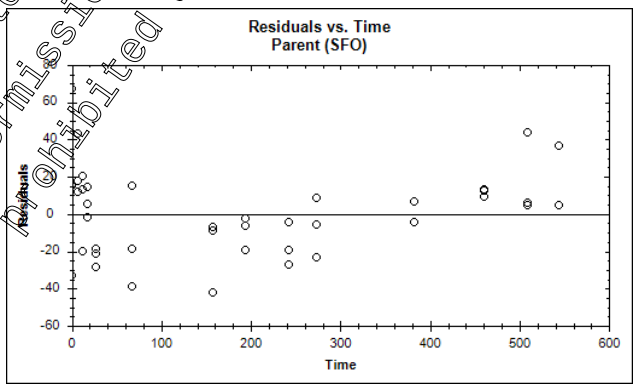
Table 7.1.2.2.1- 146: Standard EFSA template for kinetic fitting M-01

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Burscheid (Germany), KCA 7.1.2.2.1/18, ([redacted] 2019)	SFO	126.8	k 0.007373	14.7	6.27E-13	0.005949	0.009	94	312.3
Lignieres de Touraine (France North), KCA 7.1.2.2.1/19, ([redacted] 2019)	SFO	79.17	k 0.003627	7.82	<2e-16	0.003184	0.004	191.1	634.9
St.Etienne du Grès (France South), KCA 7.1.2.2.1/19, ([redacted] 2019)	SFO	83.12	k 0.003854	5.87	<2e-16	0.00348	0.004	179.9	597.5
Albaro di Ronco all'Adige (Italy), KCA 7.1.2.2.1/20, ([redacted] 2019)	SFO	86.06	k 0.004566	13.9	4.67E-10	0.003495	0.006	151.8	504.3
Vilobi d'Onyar (Spain), KCA 7.1.2.2.1/20, ([redacted] 2019)	SFO	102.4	k 0.005087	10.9	<2e-16	0.004467	0.006	136.3	452.7

Graphical representations of the best fit models are shown in Table 7.1.2.2.1- 147 for fluopicolide and Table 7.1.2.2.1- 148 for M-01.

Table 7.1.2.2.1- 147: Graphical representations of best fit models for fluopicolide

Trial / Best Fit Model	Graphical Representations
Burscheid (Germany) / fluopicolide / SFO	 
Great Chishill (United Kingdom) / fluopicolide / SFO	 

Trial / Best Fit Model	Graphical Representations
Lignieres de Touraine (France North)/ fluopicolide / SFO	 
St.Etienne du Grès (France South) / fluopicolide / SFO	 

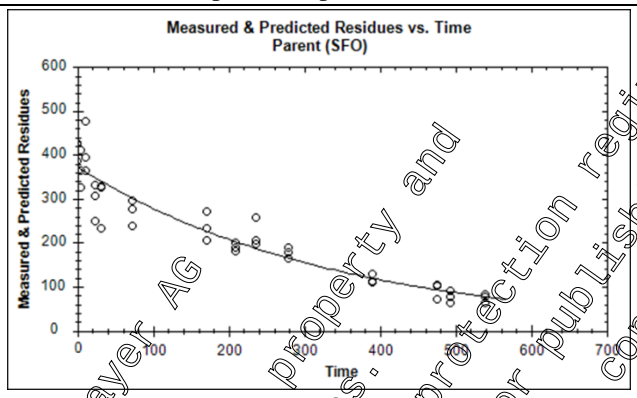
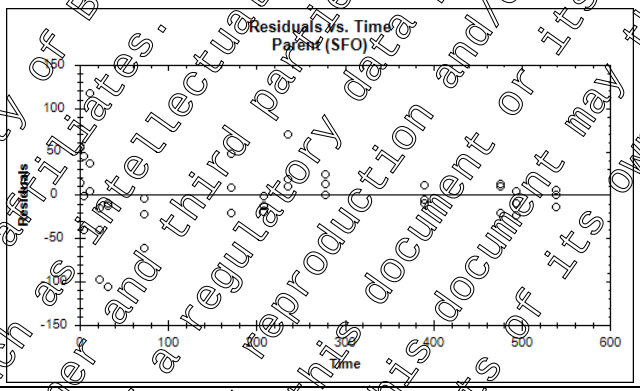
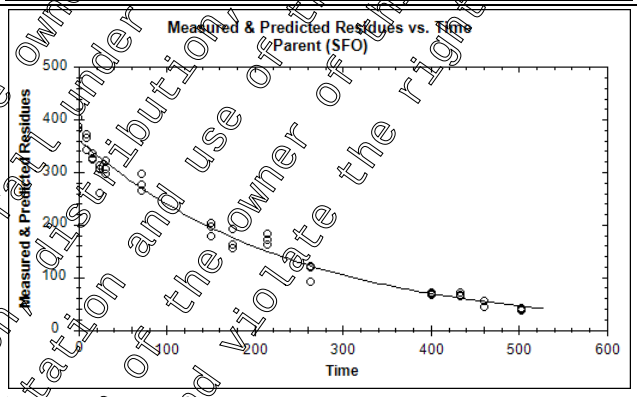
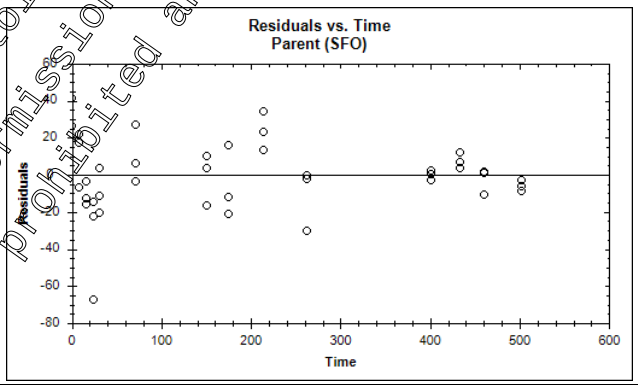
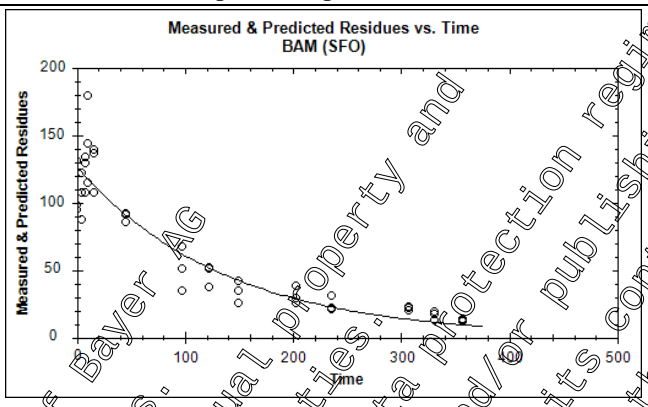
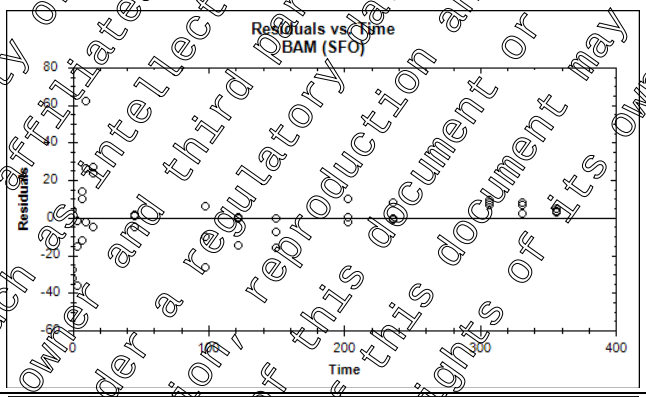
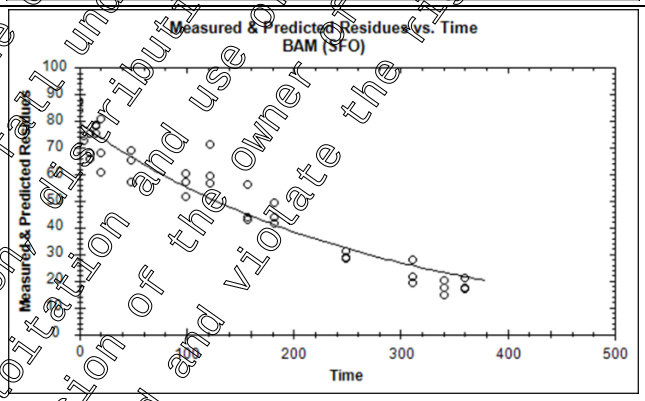
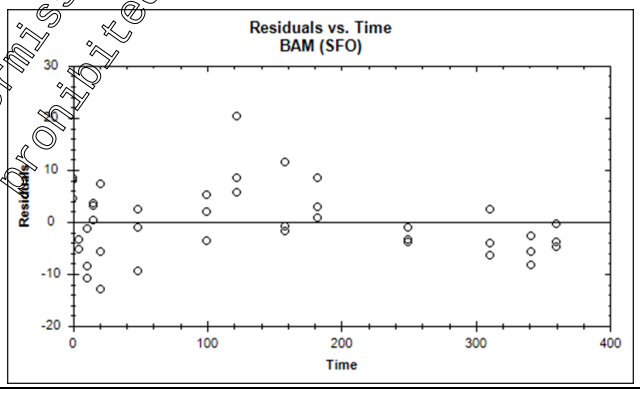
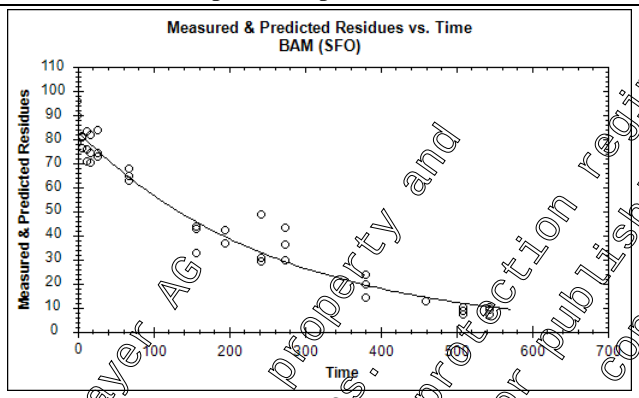
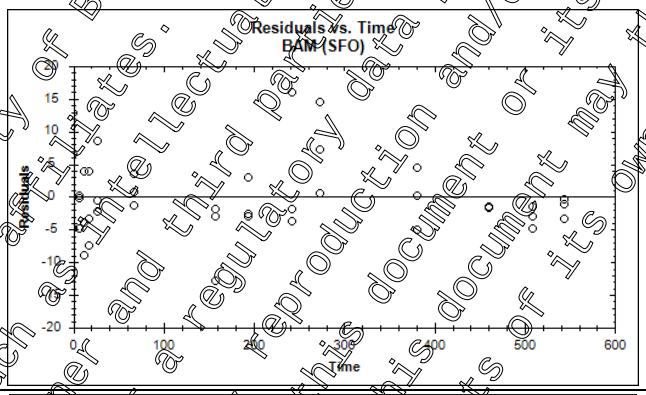
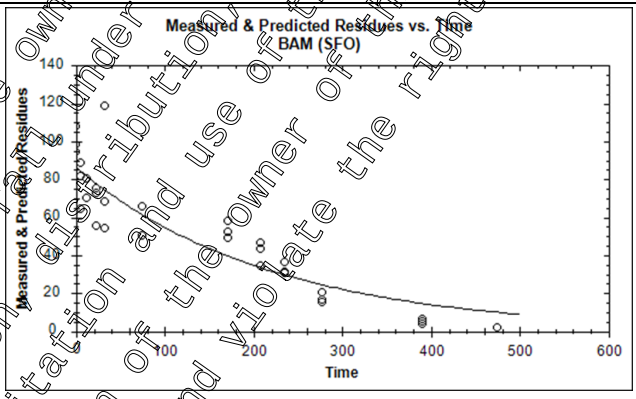
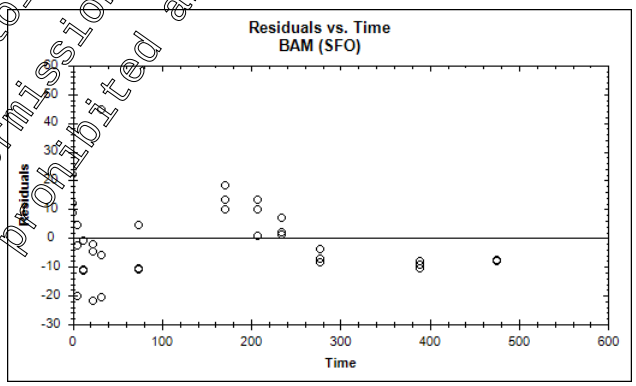
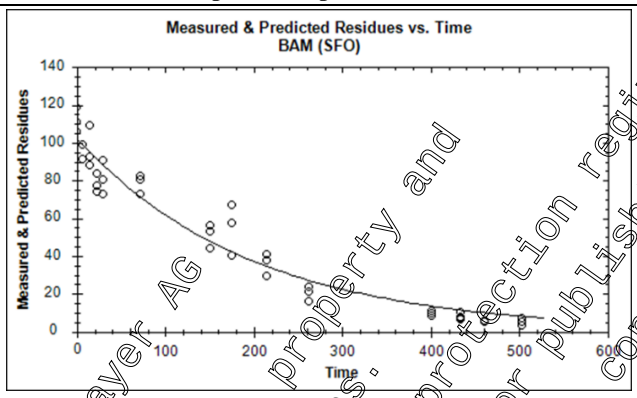
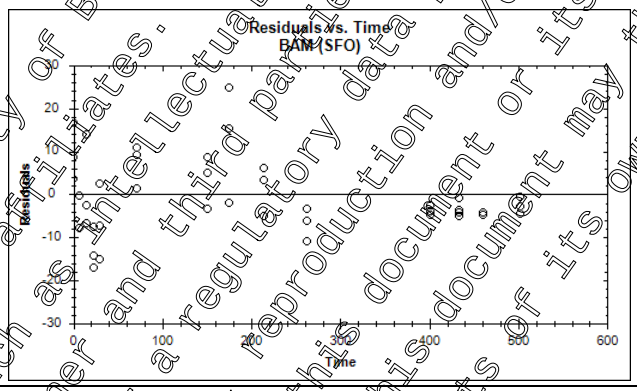
Trial / Best Fit Model	Graphical Representations
Albaro di Ronco all'Adige (Italy) / fluopicolide / SFO	 
Vilobi d'Onyar (Spain) fluopicolide / SFO	 

Table 7.1.2.2.1- 148: Graphical representations of best fit models for M-01

Trial / Best Fit Model	Graphical Representations
Burscheid (Germany) / M-01 / SFO	 
Lignieres de Touraine (France North) / M-01 / SFO	 

Trial / Best Fit Model	Graphical Representations
St.Etienne du Grès (France South) / M-01 / SFO	 
Albaro di Ronco all'Adige (Italy) / M-01 / SFO	 

Trial / Best Fit Model	Graphical Representations
Vilobi d'Onyar (Spain) M-01 / SFO	 

III. Conclusion

The data from field dissipation trials run concurrently with fluopicolide and M-01 were evaluated to derived modelling endpoints. In the trial the test item had been incorporated into the soil immediately after application to eliminate processes potentially occurring at the soil surface such as photodegradation or volatilisation.

DegT50$_{matrix}$ values for fluopicolide and M-01, normalised to 20°C and pF2, were derived for use as modelling endpoints according to FOCUS Kinetics guidance (FOCUS, 2006 and 2014a).

The overall geometric mean DT$_{50\ matrix}$ of fluopicolide for modelling purposes according to FOCUS kinetics and EFSA (2014) is 189 days. The overall geometric mean DT$_{50\ matrix}$ of M-01 for modelling purposes according to FOCUS kinetics and EFSA (2014) is 146 days.

Assessment and conclusion by applicant:

The modelling report was conducted according to FOCUS Degradation Kinetics (2006, 2014) and is considered valid to assess best fit modelling DegT50$_{matrix}$ values for fluopicolide and M-01 in field dissipation studies.

Data Point:	KCA 7.1.2.2.1/24
Report Author:	[redacted]
Report Year:	2020
Report Title:	Fluopicolide (FLC) - Kinetic evaluation of dissipation in soil under field conditions to derive trigger endpoints - Eight sites in Europe and North America (legacy studies)
Report No:	VC/19/041F
Document No:	M-685682-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	None
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

DT_{50} and DT_{90} values were derived for fluopicolide from eight terrestrial field dissipation trials, according to FOCUS Kinetics guidance (FOCUS, 2006 and 2014a). The values derived in this study are suitable for use as trigger endpoints.

A single application of fluopicolide was made to bare soil in six trials in Germany, France, and Spain, at an application rate of 400 g/ha was applied at five of the sites Philippsburg, Rodelsee, Huntlosen, Valencia and Appilly; 500 g/ha was applied at the remaining Senas site. No cultivation, tillage or irrigation was carried out at these sites after application.

Two additional trials were also considered: a trial in the USA (California) where a single application of fluopicolide was made at a rate of 400 g/ha to bare soil prior to the emergence of a potato crop; and a trial in Canada (Ontario) where three applications of fluopicolide, each at a rate of 133 g/ha, were made 5 ± 1 days apart to bare soil. For the Ontario data set, the kinetic evaluation was performed considering the third application as the beginning of the modelled decline period.

Residue data were processed prior to analysis according to FOCUS Kinetics recommendations (FOCUS, 2006 and 2014a). Kinetic analysis was performed using KinGUI v. 2.1. The selection of the most appropriate kinetic model was based on a detailed analysis, including visual assessment of the fit and residuals, χ^2 error% and t-test significance, following FOCUS guidance (FOCUS, 2006 and 2014a).

The calculated DT_{50} and DT_{90} values for fluopicolide are shown in the table below. These values are appropriate for use as trigger endpoints.

DT_{50} and DT_{90} values for fluopicolide (un-normalised), for use as trigger endpoints

Fluopicolide	Aerobic field conditions						
Soil type	**Location (country or USA state)**	**pH**	**Depth (cm)**	**DT_{50} (d)**	**DT_{90} (d)**	**χ^2 err%**	**Method of calculation**
Loamy sand	Philippsburg (Germany)	6.4	0-50	133.0	1417.0	12.7	DFOP
Sandy clay loam	Rödelsee (Germany)	7.4	0-30	256.9	853.6	18.5	SFO
Sand	Huntlosen (Germany)	4.9	0-50	290.2	963.9	16.5	SFO
Sandy loam	Valencia (Spain)	7.3	0-30	53.9	987.5	12.0	DFOP
Silt loam	Appilly (France)	7.1	0-30	143.4	1695.0	15.0	DFOP
Silt loam	Senas (France)	7.3	0-45	109.8	627.2	7.9	DFOP
Sand	California (USA)	7.7	0-60	28.0	325.8	8.2	DFOP
Clay loam	Ontario (Canada)	7.3	0-45	209.8	697.0	10.3	SFO

I. Materials and Methods

The purpose of this study was to derive DT_{50} and DT_{90} values for fluopicolide from eight terrestrial field dissipation trials in Europe and North America, according to FOCUS Kinetics guidance (FOCUS, 2006 and 2014a) to be used as trigger endpoints. All sites are considered representative of European field conditions. The graphical location are shown in Table 7.1.2.2.1- 149.

Table 7.1.2.2.1- 149: Geographical locations of field trials

Site	Latitude	Longitude
Philippsburg, Germany		
Rödelsee, Germany		
Huntlosen, Germany		
Valencia, Spain		
Appilly, France		
Senas, France		
California, USA		
Ontario, Canada		

Details of the terrestrial field dissipation studies used in this kinetic evaluation are summarised in KCA 7.1.2.2.1/01, KCA 7.1.2.2.1/02, KCA 7.1.2.2.1/03, KCA 7.1.2.2.1/04, KCA 7.1.2.2.1/08, KCA 7.1.2.2.1/09, KCA 7.1.2.2.1/16 and KCA 7.1.2.2.1/17. At all sites, fluopicolide was applied onto bare soil. For the trial in California, a potato crop was sown prior to application. To avoid disturbing the test soil, this crop was not harvested, but was instead allowed to senesce naturally. At all other sites, no cultivation or irrigation activities occurred during the trial periods, and no fertilisers were applied during the field phase of the studies.

A single application of fluopicolide was made to bare soil in six trials in Germany, France and Spain in either spring or summer, at an application rate of 400 g/ha to the Philippsburg, Rödelsee, Huntlosen, Valencia and Appilly sites and an application of 500 g/ha to the Senas site.

Two additional trials were also considered: a trial in the USA (California) where a single application of fluopicolide was made at a rate of 400 g/ha to bare soil prior to the emergence of a potato crop (KCA 7.1.2.2.1/17); and a trial in Canada (Ontario) where three applications of fluopicolide, each at a rate of 133 g/ha were made 5 ± 1 days apart to bare soil (KCA 7.1.2.2.1/16). For the Ontario data set, the kinetic evaluation was performed considering the third application as the beginning of the modelled decline period.

The datasets collected were evaluated following the EFSA Guidance Document for deriving DT_{50} and DT_{90} values (EFSA, 2014), and the recommendations of the FOCUS Kinetics group (FOCUS, 2006 and 2014a).

A summary of the trials is given in Table 7.1.2.2.1- 150.

Table 7.1.2.2.1- 150: Summary of terrestrial field dissipation studies

Document	Location	Day of application	Rate (g a.s./ha)	Soil Texture	pH ($CaCl_2$)	Duration (days)	Last sampling date
KCA 7.1.2.2.1/09, ([redacted] 2005a)	Philippsburg (Germany)	20/06/2000	400	Loamy sand	6.4	735	25/06/2002
KCA 7.1.2.2.1/01, ([redacted] 2003)	Rödelsee (Germany)	07/06/2000	400	Sandy clay loam	7.4	721	29/05/2002
KCA 7.1.2.2.1/02, ([redacted] 2003)	Huntlosen (Germany)	31/05/2000	400	Sand	4.9	722	23/05/2002
KCA 7.1.2.2.1/03, [redacted] (2004)	Valencia (Spain)	04/07/2001	400	Sandy loam	7.3	708	12/06/2003
KCA 7.1.2.2.1/08, ([redacted] 2005b)	Appilly (France)	16/06/2000	400	Silt loam	7.1	735	21/06/2002
KCA 7.1.2.2.1/04, ([redacted] 2003)	Senas (France)	24/06/1999	500	Silt loam	7.3	716	09/06/2001
KCA 7.1.2.2.1/17, ([redacted] 2005)	California, (USA)	01/06/2001	400	Sand	7.7	539	22/11/2002
KCA 7.1.2.2.1/16, ([redacted] 2004)	Ontario (Canada)	03/07/2002 08/07/2002 12/07/2002	3 x 133	Clay loam	7.3	453	08/10/2003

Soil samples were collected to a maximum depth of 45 - 90 cm, depending upon the test site. After sampling, the cores were cut into increments according to depth and analysed.

Experimental data sets and data points were weighted equally in the kinetic analysis. Where applicable, residue data from replicate subplots were processed separately. For the Philippsburg site (Pollman, 2005a), an outlier was removed on day 3 for one of the plots, as the residues for this sample corresponded to approximately 4 times the initial measured residues after application.

For all data sets except for the California trial, the Limit of Detection (LOD) was not reported; instead only the Limit of Quantification (LOQ = 0.005 mg/kg dry weight) was given. For these data sets it has been assumed that residues reported as <LOQ were also <LOD, and these residues have been adjusted as outlined below, in keeping with the FOCUS Kinetics guidance (FOCUS, 2006 and 2014a) and also accounting for the movement of the test compound down the soil profile:

In the top horizon:

- Samples <LOQ just after a quantifiable amount were set to ½ LOQ.
- All subsequent samples <LOQ were omitted, unless later samples >LOQ were observed.

In lower horizons:

- No adjustments were made in lower horizons until the first sample >LOQ was observed below the top horizon.
- Thereafter:
  - Samples <LOQ just before or just after a quantifiable amount in the same layer were set to ½ LOQ.
  - All subsequent samples <LOQ were omitted, unless later samples >LOQ were observed.
  - Where a sample >LOQ in a deeper layer was reported, samples <LOQ in higher layers at the same time point were set to ½ LOQ.
  - An additional adjustment was made to the shallowest residue-free layer at every time point, which was set to ½ LOQ.

For the California trial, the LOD was reported as 2 ppb (dry weight), with the LOQ reported as 5 ppb (dry weight). The adjustments made to the California data set were analogous to those described above, with residues <LOD adjusted to ½ LOD where required. Residues between LOD and LOQ were set to their measured values.

In all cases, adjustments were made only to layers where samples had been analysed.

Residue values were given in the original study reports in units of mg/kg or ppb (dry weight). Insufficient data were available to convert these residues to units of g/ha, therefore the kinetic analysis was performed using residues averaged across all relevant soil layers, expressed in units of mg/kg or ppb (dry weight), with the contributions from each layer weighted to account for the depth of the layer.

H. Results and Discussion

To derive trigger endpoints, an initial comparison was performed for each site between the SFO and FOMC fits for fluopicolide. For the Rödelsee, Huntlosen and Ontario sites, the FOMC fits provided no significant improvement, and the SFO fits were therefore accepted.

For the remaining sites, an initial comparison of the SFO and FOMC fits suggested bi-phasic decline, and the DFOP model was therefore also fitted. For the Valencia, California and Senas sites, DFOP provided the best fit to the residues, with the lowest χ^2 err% value, and was therefore accepted.

For the Philippsburg site, confidence in the k1 DFOP rate constant was slightly low (p=0.064), however the DFOP fit provided the best visual description of the residues and was accepted.

The DFOP model was also accepted for the Appilly test site, despite a lack of confidence in the optimised rate constants, as DFOP kinetics provided the best visual description of the decline. The FOMC fit was not accepted, as extrapolation beyond the experimental period is not recommended for deriving robust DT_{90} values using this model (ETSA, 2009). It is noted that the estimated DT_{90} exceeded the relevant regulatory triggers for all models.

A summary of the fitted parameters for all test sites is given in Table 7.1.2.2.1- 151.

Table 7.1.2.2.1- 151: Results for fluopicolide: summary of kinetic analysis

Kinetic model	DT_{50}	DT_{90}	VA	χ^2err	k_1 / α	k_2 / β	t_b / g	t-test k_1 / k_2	MS
	(d)			(%)	(1/d / -)	(1/d / -)	(d / -)	(-)	
Philippsburg									
SFO[a]	288.3	957.8	o	18.81	0.0024039	-	-	6.53E-08	
FOMC[a]	117.3	>10000	+	13.45	0.30881	13.90617	-	-	
DFOP[a]	133	1417	+	12.68	0.0464214	0.0012519	0.4164171	0.06404 / 0.00473	T
Rödelsee									
SFO	256.9	853.5	o	18.5	0.0026978	-	-	1.10E-09	T
FOMC	256.9	853.6	o	19.18	7858	2.91E+06	-	-	
Huntlosen									
SFO	290.2	963.9	+	16.53	0.0023887	-	-	1.13E-07	T
FOMC	290.2	964.2	+	17.13	2442	1.02E+06	-	-	
Valencia									
SFO	177.4	589.3	o	21.52	0.003907	-	-	1.92E-10	
FOMC	59.81	3490	+	12.69	0.41692	13.98216	-	-	
DFOP	53.86	987.5	+	11.98	0.0387182	0.0015785	0.5270615	0.01124 / 0.00433	T
Appilly									
SFO	194.4	6457	o	16.32	0.0035658	-	-	7.97E-09	
FOMC	150.7	1202	+	14.79	1416	175.1	-	-	
DFOP	143.4	1695	+	15.04	0.0079308	6.56E-04	0.6959214	0.1236 / 0.3988	T
Senas									
SFO	158.6	593.3	o	14.31	0.003881	-	-	1.18E-14	
FOMC	104.4	1264	o	11.84	0.802507	76.62662	-	-	
DFOP	109.8	627.2	+	7.902	0.3278513	0.0031104	0.2965286	0.0207 / 4.90E-14	T
California									
SFO	77.51	257.5	o	19.32	0.008943	-	-	9.86E-15	
FOMC	31.7	527.9	o	11	0.6687	17.4256	-	-	
DFOP	27.98	325.8	+	8.234	0.1191	0.005296	0.4383	0.00182 / 7.17E-10	T
Ontario									
SFO	209.8	697	+	10.31	0.0033037	-	-	1.68E-07	T
FOMC	160.6	1435	+	10.21	1006	162	-	-	

MS: Model selected (T: for trigger evaluation)
[a] – Outlier removed on day 3 for plot T1
VA: Visual assessment (+ = good fit, o = acceptable fit, - = non acceptable fit)

A summary is given in Table 7.1.2.2.1- 152 of the DT_{50} and DT_{90} values derived for fluopicolide for use as trigger endpoints.

Table 7.1.2.2.1- 152: DT_{50} and DT_{90} values for fluopicolide (un-normalised) for use as trigger endpoints

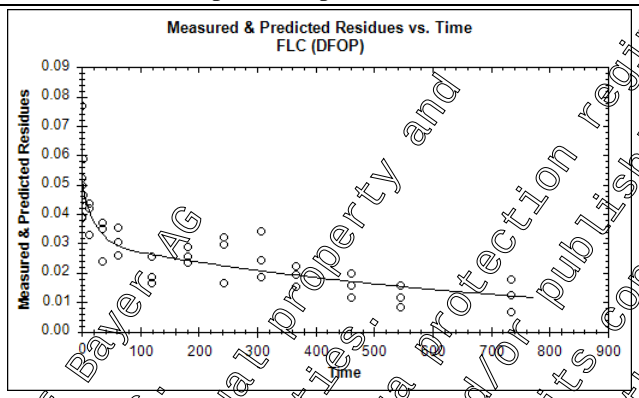
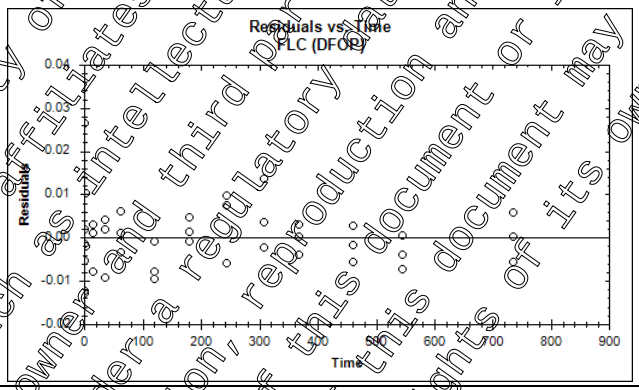
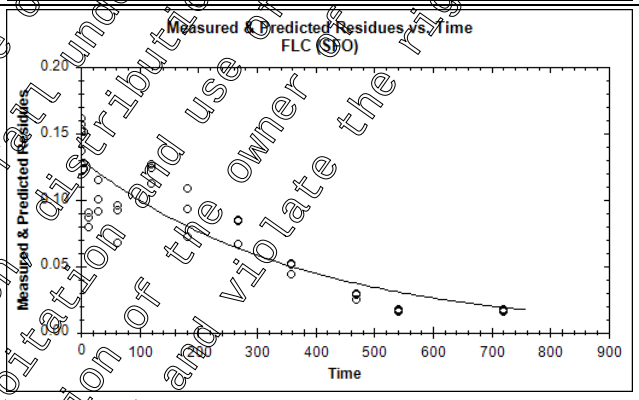
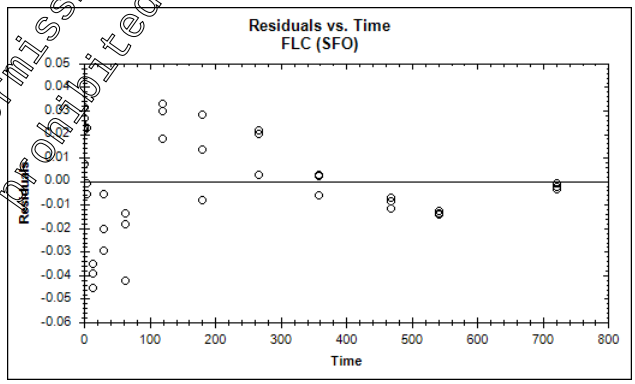
Soil	Kinetic model	DT_{50} (days)	DT_{90} (days)
Philippsburg (Germany)	DFOP	133.0	1417.0
Rödelsee (Germany)	SFO	256.9	853.5
Huntlosen (Germany)	SFO	290.2	963.9
Valencia (Spain)	DFOP	53.9	987.5
Appilly (France)	DFOP	143.4	1695.0
Senas (France)	DFOP	109.8	627.2
California (USA)	DFOP	28.0	325.8
Ontario (Canada)	SFO	209.8	697.0

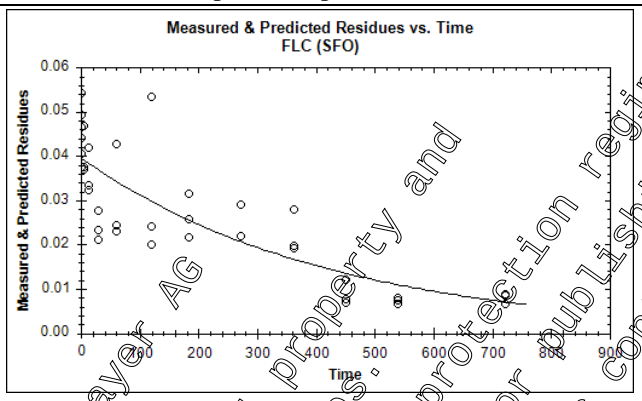
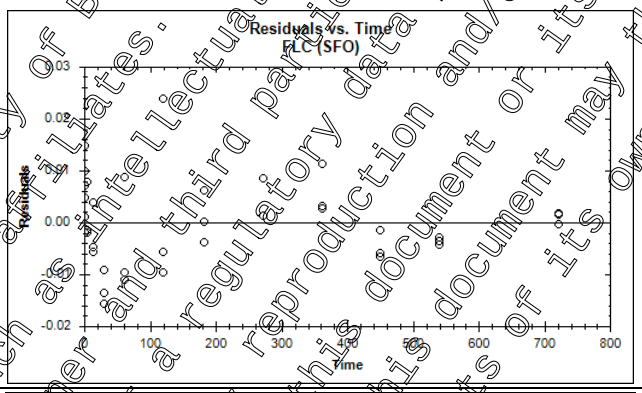
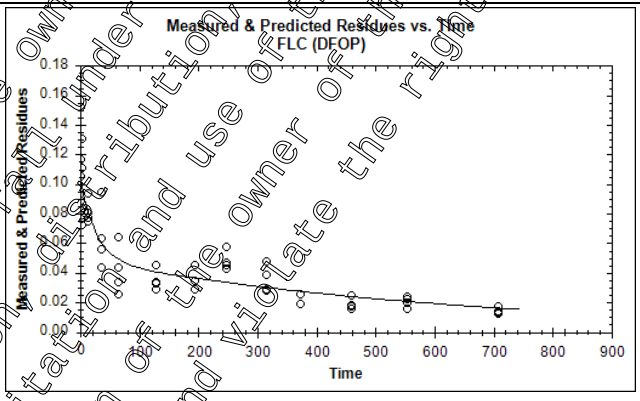
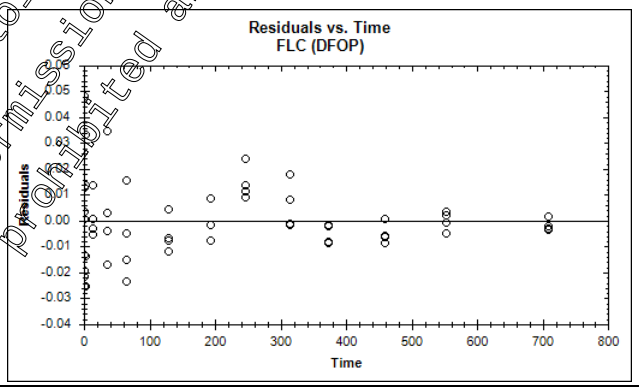
The standard EFSA template can be seen in Table 7.1.2.2.1- 153 and graphical representations in Table 7.1.2.2.1- 154.

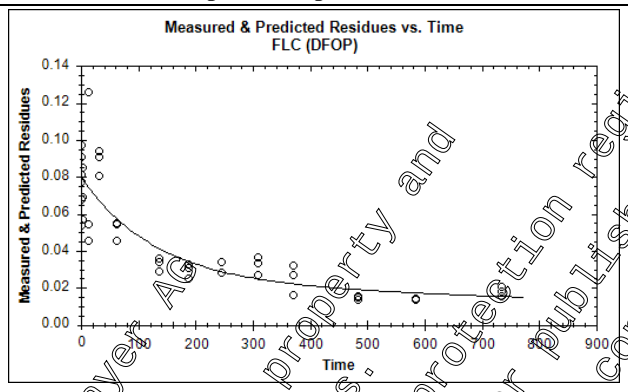
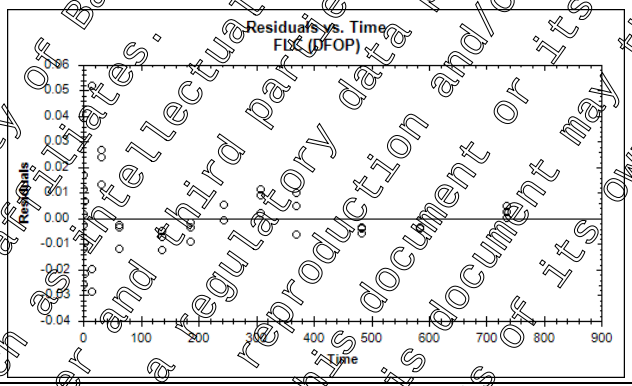
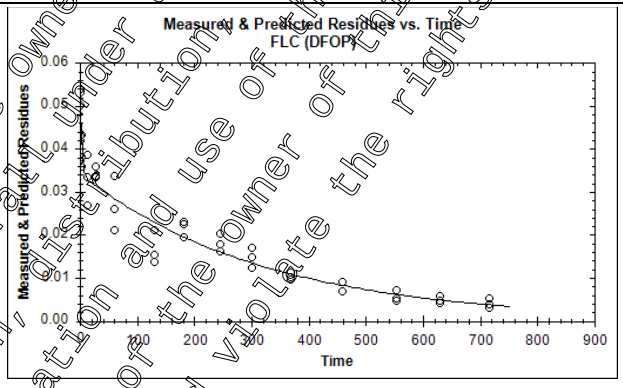
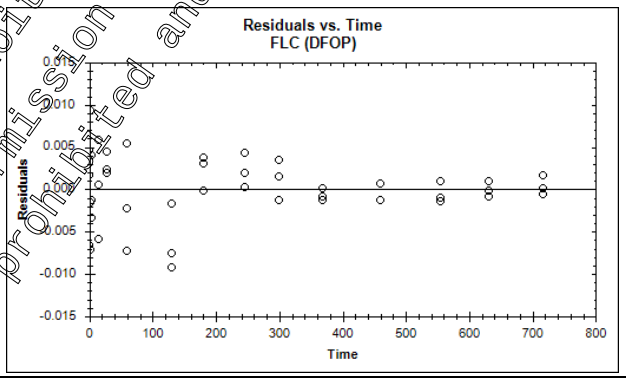
Table 7.1.2.2.1- 153: Standard EFSA template for kinetic fitting

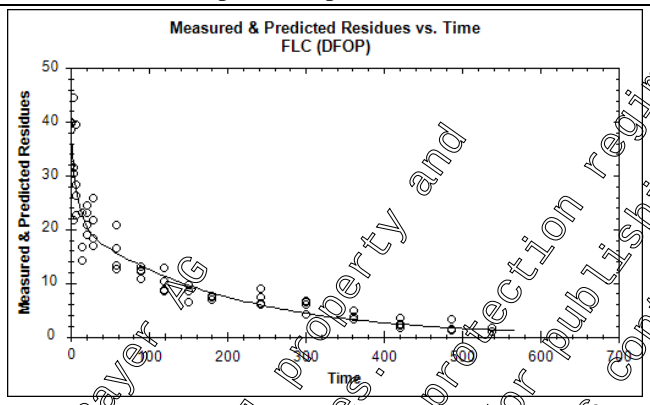
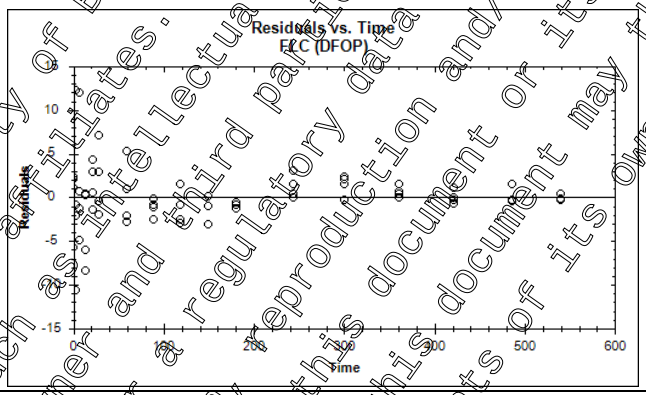
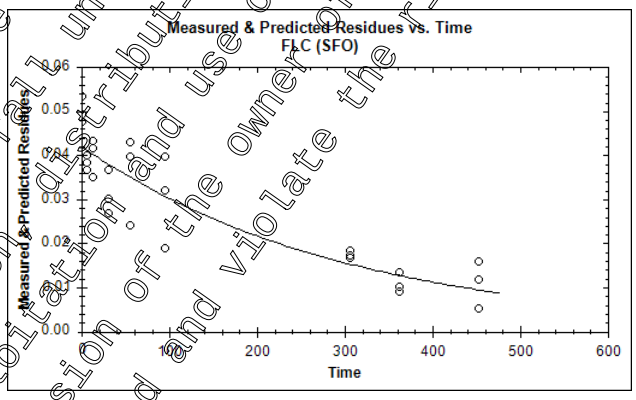
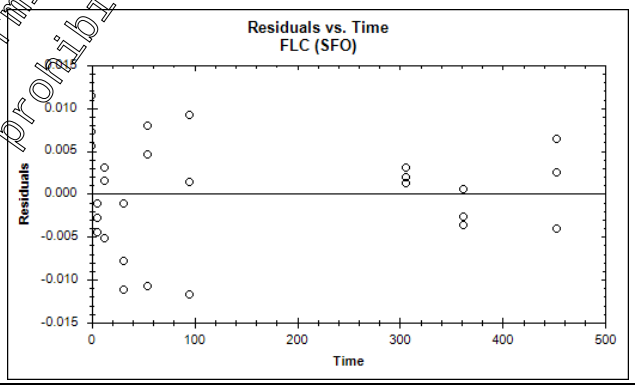
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Philippsburg (Germany), KCA 7.1.2.2.1/09, ([redacted] 2005a)	SFO	0.04317	k 0.002404	18.8	6.53E-08	0.001671	0.003	288.3	957.8
	FOMC	0.05099	α 0.3088 β 13.91	13.5	n.r. n.r.	0.1372 -10.57	0.48 38.38	117.3	>10000
	DFOP	0.05131	k1 0.04642 k2 0.001252 g 0.4104	12.7	0.06404 0.00473 n.r.	-0.01203 0.0003557 0.2359	0.105 0.002 0.582	135	1417
Rödelsee (Germany), KCA 7.1.2.2.1/01, ([redacted] 2003)	SFO	0.1302	k 0.002698	18.5	1.10E-09	0.002024	0.003	256.9	853.5
	FOMC	0.1302	α 7858 β 2.91E+06	19.2	n.r. n.r.	7858 2.91E+06	7858 2.91E+06	256.9	853.6
Huntlosen (Germany), KCA 7.1.2.2.1/02, ([redacted] 2003)	SFO	0.03948	k 0.002389	16.5	1.13E-07	0.001649	0.003	290.2	963.9
	FOMC	0.03948	α 2442 β 1.02E+06	17.1	n.r. n.r.	2442 1.02E+06	2442 1.02E+06	290.2	964.2
Valencia (Spain), KCA 7.1.2.2.1/03, [redacted] (2004)	SFO	0.08862	k 0.003908	21.5	1.92E-10	0.002905	0.005	177.4	589.3
	FOMC	0.1048	α 0.4169 β 14	13.7	n.r. n.r.	0.2407 3.434	0.593 31.43	59.8	3490
	DFOP	0.104	k1 0.03872 k2 0.001574 g 0.5271	12	0.01124 0.00433 n.r.	0.006458<0.000443 0.3584	0.071 0.003 0.696	53.9	987.5
Appilly (France), KCA 7.1.2.2.1/08, ([redacted] 2005b)	SFO	0.07668	k 0.003566	16.3	7.97E-09	0.002575	0.005	194.4	645.7
	FOMC	0.07992	α 1.116 β 175.1	14.8	n.r. n.r.	-0.3699 -197.3	2.603 547.4	150.7	1202
	DFOP	0.0799	k1 0.007931 k2 0.0006561 g 0.6959	15	0.1236 0.3988 n.r.	-0.005294 -0.004323 -0.2273	0.021 0.006 1.619	143.4	1695
Senas (France), KCA 7.1.2.2.1/04, ([redacted] 2003)	SFO	0.04026	k 0.003881	14.3	1.18E-14	0.003203	0.005	178.6	593.3
	FOMC	0.0436	α 0.8025 β 76.06	11.8	n.r. n.r.	0.3857 -1.463	1.219 153.6	104.4	1264
	DFOP	0.04855	k1 0.3279 k2 0.00311 g 0.2965	7.9	0.0207 4.90E-14 n.r.	0.02257 0.002553 0.2113	0.633 0.004 0.382	109.8	627.2
California (USA), KCA 7.1.2.2.1/17, ([redacted] 2005)	SFO	29.91	k 0.008943	19.3	9.86E-15	0.007148	0.011	77.5	257.5
	FOMC	36.13	α 0.6687 β 17.43	11	n.r. n.r.	0.4684 4.929	0.869 29.92	31.7	527.7
	DFOP	37.35	k1 0.1191 k2 0.005296 g 0.4383	8.23	0.00182 7.17E-10 n.r.	0.04176 0.003827 0.3343	0.196 0.007 0.542	28	325.8
Ontario (Canada), KCA 7.1.2.2.1/16, ([redacted] 2004)	SFO	0.04192	k 0.003304	10.3	1.68E-07	0.002361	0.004	209.8	697
	FOMC	0.04402	α 1.006 β 162	10.2	n.r. n.r.	-0.3943 -199.5	2.407 523.5	160.6	1435

Table 7.1.2.2.1- 154: Graphical representations of best fit models for modelling endpoints

Trial / Best Fit Model	Graphical Representations
Philippsburg (Germany) / DFOP	 
Rödelsee (Germany) / SFO	 

Trial / Best Fit Model	Graphical Representations
Huntlosen (Germany) / SFO	 
Valencia (Spain) / DFOP	 

Trial / Best Fit Model	Graphical Representations
Appilly (France) / DFOP	 
Senas (France) / DFOP	 

Trial / Best Fit Model	Graphical Representations
California (USA) / DFOP	 
Ontario (Canada) / SFO	 

III. Conclusion

The data from the eight 'legacy' field dissipation trials was evaluated to derived trigger endpoints for fluopicolide according to FOCUS Kinetics guidance (FOCUS, 2006 and 2014a). Best-fit un-normalised DT_{50} values from eight legacy terrestrial field dissipation trials ranged from 53.9 to 290.2 days. Corresponding DT_{90} values ranged from 325.8 to 1995 days.

Assessment and conclusion by applicant:
The modelling report was conducted according to FOCUS Degradation Kinetics (2006, 2014) and is considered valid to assess best fit and modelling DT_{50} values for fluopicolide in field dissipation studies.

CA 7.1.2.2.2 Soil accumulation studies

Soil accumulation studies with fluopicolide were evaluated during the previous EU review. No new soil accumulation studies are submitted.

KCA 7.1.2.2.2/01 was a continuation of a field dissipation trial (KCA 7.1.2.2.1/04) conducted at the same site in previous years. Two additional soil accumulation studies (KCA 7.1.2.2.1/08 and KCA 7.1.2.2.1/09) were evaluated during the previous EU review. As these studies combined separate dissipation and accumulation trials in the same report both are summarised in full under Point KCA 7.1.2.2.1.

In the Addendum to the DAR (2007) it was concluded fluopicolide residues had reached a plateau concentration in two of the studies (KCA 7.1.2.2.1/09 and KCA 7.1.2.2.2/01) but was inconclusive in the third study (KCA 7.1.2.2.1/08), while M-01 (AE C653711) residues had reached a plateau only in KCA 7.1.2.2.2/01. The metabolites M-02 (AE C657188) and M-03 (AE 0608000) were rapidly degraded in soil and were either not detected or disappeared completely within one month. The studies are considered as acceptable supportive information to assess the possibility of accumulation of fluopicolide and M-01 in soil. Definitive assessment of the accumulation of fluopicolide and its metabolites in soil is addressed in Document MCP 9 by calculation.

Report reference	Author, Year	Test item	Comment
KCA 7.1.2.2.2/01 M-223186-01-1	[redacted], 2004	Fluopicolide	Submitted and reviewed for first approval of fluopicolide, 2005. Considered valid and acceptable.

Data Point:	KCA 7.1.2.2.2/01
Report Author:	[redacted]
Report Year:	2004
Report Title:	Field soil accumulation of AE C638206 following a single application to bare soil plots at 1 location in France, 2000
Report No:	C037581
Document No:	M-223186-01-1
Guideline(s) followed in study:	BBA: Part IV, 4-1; IVA: (1993); SETAC: (1995)
Deviations from current test guideline:	Yes. Report meets the requirement for field persistence criteria and ecotoxicological risk assessment as required by EU 283/2013. Report does not meet the requirement for assessing soil Deg150matrix values as required by EFSA (2014) for field studies.
Previous evaluation:	yes, evaluated and accepted Accumulation phase not originally accepted in the DAR (2005) but subsequently accepted in Addendum 1 to DAR (2007).
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

A three year terrestrial field dissipation and accumulation study with fluopicolide, formulated as a suspo-emulsion containing 97.9 g/L fluopicolide (AE C638206 00 SE10 A3), has been conducted at a site at Senas in Southern France. The top soil at the test site was a loamy silt soil. The formulated material was applied once a year, at the rate required to achieve an annual application of 500 g/ha of fluopicolide using a calibrated boom sprayer. Nominal application rates were confirmed by measuring the unused formulation remaining in the spray tank to calibrate the amount applied. The initial application in the current study was on 21 June 2000 (to Plots T1 and T3) and on 4 August 2000 (to Plot T2n). Subsequent applications were made to all three plots on 19 June 2001 and 27 June 2002. Samples of soil have been taken at intervals over a three year period and analysed by an LC/MS/MS method to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) present in the samples.

The initial dissipation of fluopicolide was relatively rapid followed by a slower dissipation phase during the winter months due to the cold climate and possibly reduced availability of fluopicolide due to increased adsorption to soil with ageing. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. Low residues were detected in the 10-20 cm horizon at concentrations ranging from 0.005 to 0.017 mg/kg (mean values). Residue levels of parent declined to 129 g/ha one year after the first treatment which represented approximately 20 % of the initial concentration. Throughout the dissipation phase no residues of fluopicolide were detected above the LOQ below 20 cm depth.

The concentration of M-01 (AE C653711) in the soil profile varied with the degradation rate of fluopicolide. During the summer months when the degradation rate of the parent compound is relatively rapid, M-01 concentrations were relatively high and declined during the winter as the degradation rate of fluopicolide slowed. Residues levels of M-01 reached a maximum one to two months after first application at a mean concentration equivalent to 109.5 g/ha in summer 2000 (mean of Plots T1 and T3, 126 g/ha). After one year residue levels of M-01 had declined to 81.0 g/ha (June 2001). The maximum residue level in the 0-10 cm horizon was observed 59 days after application at 0.084 mg/kg (mean of Plots T1 and T3) and 31 days after application at 0.059 mg/kg (Plot T2n). The peak concentration in the underlying 10-20 cm was slightly later, after 121 days at 0.019 mg/kg and 136 days at 0.021 mg/kg in Plots T1/T3 and T2n, respectively. In the lower soil horizons residue levels were generally at or below

the LOQ until the winter months where the maximum concentration observed was 0.016 mg/kg and 0.010 mg/kg at 20-30 cm and 30-50 cm soil depths, respectively.

M-02 (AE C657188) and M-03 (AE 0608000) were rapidly degraded in the trial. Residues of M-02 was only detected at early time-points in the 0-10 cm soil depth up to 59 days after application in Plots T1 and T3 (31 days in Plot T2n) at a maximum concentration of 0.046 mg/kg, equivalent to 69 g/h. No residues of M-02 were detected above the LOQ below 10 cm depth. No residues of M-03 were found above the LOQ throughout the study. The degradation of M-03 is known to be pH dependent and is very rapidly degraded in neutral to alkaline soils such as the soil at the Senas trial site.

The reported DT_{50} and DT_{90} values of 58 and 679 days were evaluated using a biphasic hockey stick model (HS). The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. The data is no longer considered suitable to derive degradation rates as the experimental plots had previously been treated with fluopicolide (in 1999).

The plateau concentrations of fluopicolide and M-01 after four years are summarised below.

Plateau concentration	Time-point	Fluopicolide (mg/kg)		Time-point	M-01 (mg/kg)	
		0-10 cm	0-20 cm		0-10 cm	0-20 cm
High[1]	Day 0 3rd Application	0.354	0.186	Day 119 after 2nd Application	0.047	0.030
Low[2]	Day 355 after 3rd Application	0.061	0.046	Day 355 after 3rd Application	0.015	0.014

[1] maximum of the high values of the "saw teeth" curve
[2] maximum of the low values of the "saw teeth" curve

In the Addendum to the DAR (2007) it was concluded fluopicolide and M-01 residues had reached a plateau concentration in the Senas trial.

Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the accumulation phase. Low residues were detected in the 10-20 cm soil depth at concentrations ranging from 0.006 to 0.033 mg/kg (mean values) throughout the accumulation phase. In the 20-30 cm soil depth fluopicolide was detected in October 2001 at a mean concentration of 0.003 mg/kg. Residues of the test item detected in 20-30 cm and 30-50 cm soil depths immediately after second and third applications were concluded to be a result of sample contamination during sampling.

The metabolite M-01 was detected in 0-10 cm soil depth at a maximum concentration of 0.047 mg/kg (mean value). Levels in the 10-20 cm soil were lower with a maximum of 0.013 mg/kg (mean value). In the 20-30 cm layer residues reached a maximum of 0.009 mg/kg (mean value) and did not exceed 0.007 mg/kg (mean value) in 30-50 cm depth.

M-02 was detected only at early time-points after the initial application during the dissipation phase of the trial. Residues of M-02 were only detected once throughout the accumulation phase at a concentration of 0.007 mg/kg. No residues of M-03 were detected throughout the dissipation or accumulation phases of the trial.

I. Materials and Methods

A. Materials

1. Test Item

Fluopicolide formulated as liquid suspo-emulsion

Year of application:	2000 and 2001	2002
Content of a.s.:	97.9 g/L	97.5 g/L
Certificate of Analysis:	AGF2000-0106-01	AGF2001-0227-01
Lot No:	OP200271	OP210914

2. Trial Location & Soil

A three year terrestrial field accumulation study with fluopicolide, formulated as a suspo-emulsion containing 97.9 g/L fluopicolide (AE C638206-00 SE10 A3), has been conducted at a site at Sénas, Bouche du Rhône, France. The top soil was a low organic carbon silty sand (DIN classification) overlying sandy silt subsoil. The characteristics of the soil are summarised in Table 7.1.2.2.2- 1. Three experimental plots, each measuring 2.5 meters by 14 meters (105 m² in total), were treated with the test substance. A fourth plot measuring 2 meters by 12 meters was left untreated to provide control samples.

Table 7.1.2.2.2- 1: Location, site description and climatic data of test site

Characteristic	Units	Sénas, Bouche du Rhône, France		
		Horizon 1	Horizon 2	Horizon 3
Sampling depth	cm	0 - 20	20 - 50	50 - 90
pH	$CaCl_2$	7.3	7.5	7.6
Cation exchange capacity	mval/100 g	17.5	14.1	23.4
Total organic carbon (TOC)	%	1.62	0.88	0.90
Soil Density	g/L	1280	1300	1250
Particle size distribution (DIN)				
Clay < 0.002 mm	%	10.8	3.2	3.6
Total silt 0.002 - 0.063 mm	%	57.6	61.1	61.4
Total sand 0.063 - 2 mm	%	31.6	35.7	35.0
Textural class	DIN	loamy silt	sandy silt	sandy silt
Water Holding Capacity	Vol % at 1/10 bar (pF2)	30.4	30.0	27.3
	Vol % at 15 bar (pF4.2)	8.7	11.5	13.5

Biomass		Control	T1	T2n	T3
2000	mg C/100 g	70.3	22.1	32.5	26.9
2001	mg C/100 g	n.a.	n.a.	n.a.	n.a.
2002	mg C/100 g	32.8	27.5	26.7	27.5
2003	mg C/100 g	31.8	31.4	24.8	35.8

n.a. = not applicable

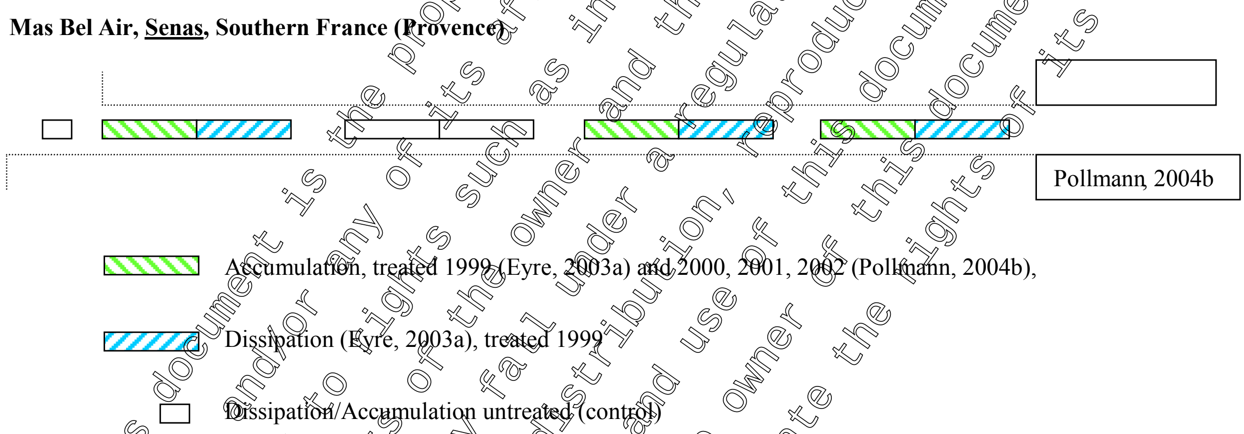
B. Study Design

1. Experimental Conditions

The formulated material was applied once a year, at the rate required to achieve an annual application of 500 g/ha of fluopicolide using a calibrated boom sprayer. Nominal application rates were confirmed by measuring the unused formulation remaining in the spray tank to determine the actual amount applied. This study was a continuation of a field dissipation trial conducted in 1999 to 2001 (KCA 7.1.2.2.1/04, M-220477-02-1). The accumulation plots T1, T2n and T3 had already been treated in 1999 at a nominal application rate of 500 g/ha. The original control plot, Plot C was treated in error instead of Plot T2 on the first application on 20 June 2000. Consequently Plot T2n was treated on 4 August 2000 and a new control plot, Plot Cn set up.

The initial application in the current study was on 21 June 2000 to Plots T1 and T3 and on 4 August 2000 to Plot T2n. Subsequent applications were made to all three plots on 19 June 2001 and 27 June 2002. Further details of the relationship between the experimental plots in the dissipation phase at Senas (KCA 7.1.2.2.1/04, M-220477-02-1) and the accumulation phase are provided in the figure below.

Figure 7.1.2.2.2- 1: Overview of sample plots in dissipation ([redacted] 2003; M-220477-02-1) and accumulation ([redacted] 2004; M-223186-01-1) studies conducted at Senas



All applications were made to bare soil. Throughout the study the plots were maintained as bare soil by the periodic application of the herbicide glyphosate to control weeds.

Wind speed, global radiation, rainfall, air temperature, relative air humidity was taken from the nearest relevant weather station of the regional official weather service (Eyguieres, 6 km away or Avignon, 24 km away).

2. Sampling

Samples were taken immediately after treatment and at selected timepoints up to 12 months after the first application. Additional samples were taken immediately after each subsequent application and approximately 4 (or 3 months after the third application) and 12 months after each application. Soil cores were taken to a depth of 30 cm until 59 days after first application for Plots T1 and T3 and 76 days for Plot T2n. Soil cores were taken to a depth of 50 cm at time-points up to one year. Soil cores for the accumulation phase were taken to a depth of 50 cm following the second and up to 83 days after the third application. At the final timepoint soil cores were taken to a depth of 90 cm. At each sampling date 7 samples from each plot were taken (21 cores in total). Field samples were frozen immediately after sampling and shipped frozen to GAB Biotechnologie GmbH, Germany. The soil samples from the same horizon of each plot were blended in Germany and a subsample dispatched frozen to the Bayer CropScience analytical laboratory in France. The samples were then stored at -18 °C until required for analysis.

3. Analytical Procedures

The analytical method AR 265-01 was used to determine levels of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 060800). Soil samples of 20 g were extracted twice at ambient temperature for 5 minutes by mechanical agitation using acetonitrile/water (70/30, *v/v*) acidified with 0.1% formic acid. After each extraction step, extract and soil were separated by centrifugation and decantation. The soil extracts were combined and diluted with acidified water to result in a final solvent of acetonitrile/water (30/70) with 0.1% formic acid. Quantification was carried out by LC-MS/MS using external standardisation for the parent compound and its metabolites. The limit of quantification (LOQ) was 0.005 mg/kg for each analyte.

During analysis of the dissipation samples of the current study, concurrent recovery samples were prepared freshly by fortification of control samples with test item fluopicolide and reference items M-01, M-02 and M-03 at 1 x LOQ and processed in parallel to the dissipation samples. The mean recoveries of were 104% (RSD 8.7 %) for fluopicolide, 100% (RSD 6.9%) for M-01, 90% (RSD 10.1%) for M-02 and 95% (RSD 13.9%) for M-03. No residues of fluopicolide or its metabolites were found above the LOQ in the analysed untreated samples.

The validation of the extraction was carried out during the study, with samples taken immediately after the application of the test substance.

4. Determination of degradation kinetics

The reported DT_{50} and DT_{90} values were evaluated using a biphasic hockey stick model (HS) using the programme ModelMaker. The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. The data is no longer considered suitable to derive degradation rates as the experimental plots had previously been treated with fluopicolide (in 1999).

II. Results and Discussion

A. Analytical Methodology:

Full details and acceptable validation data to support this method are presented in Document M-CA 4, Section 4.1.2. The method complies with the EU regulatory requirements outlined within SANCO/3029/99 rev. 4 and is suitable for the determination of fluopicolide and its metabolites M-01 (AE C653711), M-02 (AE C657188) and M-03 (AE 0608000) in soil samples by HPLC-MS/MS.

B. Data

The results for fluopicolide and its metabolites M-01 (AE C653711) and M-02 (AE C657188) are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots in Table 7.1.2.2.2- 2 to Table 7.1.2.2.2- 7. No residues of M-03 (AE 0608000) were detected throughout the trial. The dissipation and accumulation of fluopicolide (mean values and individual plots) and M-01 (mean values) at Senas are presented in Figure 7.1.2.2.2- 2 and Figure 7.1.2.2.2- 3.

In order to calculate mean values for this summary, concentrations <LOQ (0.005 mg/kg) were assumed to be 0 mg/kg. Where individual replicate values exceeded the LOQ the calculated mean concentration has been reported, including mean values that are below the LOQ. For the conversion of mg/kg into g/ha a soil density of 1.5 g/cm^3 was used.

Table 7.1.2.2.2- 2: Residues of fluopicolide in soil after annual applications of 500 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Plots T1 and T3

Depth [cm]	Sub plot	DAA1										
		0	**1**	**3**	**14**	**28**	**59**	**121**	**181**	**244**	**302**	**363**
0-10	T1	0.389	0.583	0.604	0.386	0.404	0.311	0.149	0.164	0.145	0.097	0.067
	T3	0.344	0.317	0.544	0.291	0.272	0.292	0.143	0.169	0.176	0.093	0.070
	mean	**0.367**	**0.450**	**0.574**	**0.339**	**0.338**	**0.302**	**0.146**	**0.167**	**0.161**	**0.095**	**0.069**
10-20	T1	ns	<LOQ	<LOQ	<LOQ	0.009	<LOQ	0.013	0.012	0.008	0.012	0.010
	T3	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.013	0.015	<LOQ	<LOQ
	mean	ns	<LOQ	<LOQ	<LOQ	**0.005**	<LOQ	**0.008**	**0.013**	**0.017**	**0.006**	**0.005**
20-30	T1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	T1	ns	ns	ns	ns	ns	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T3	ns	ns	ns	ns	ns	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	ns	ns	ns	ns	ns	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
50-90	T1	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
	T3	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
	mean	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**

Plots T2n

Depth [cm]	Sub plot	DAA1									
		0	**1**	**3**	**14**	**31**	**76**	**136**	**199**	**257**	**318**
0-10	T2n	0.417	0.440	0.362	0.277	0.232	0.102	0.143	0.123	0.100	0.065
10-20	T2n	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.013	0.015	<LOQ
20-30	T2n	ns	<LOQ	<LOQ	<LOQ	0.005	<LOQ	0.008	0.013	0.017	0.005
30-50	T2n	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
50-90	T2n	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

Table 7.1.2.2.2- 3: Residues of fluopicolide in soil after annual applications of 500 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3		
		0	119	372	0	83	355
0-10	T1	0.295	0.224	0.095	0.369	0.248	0.039
	T2n	0.366	0.197	0.066	0.354	0.224	0.084
	T3	0.344	0.281	0.090	0.355	0.209	0.050
	mean	**0.351**	**0.253**	**0.082**	**0.354**	**0.214**	**0.061**
10-20	T1	0.069	0.014	0.007	0.007	0.009	0.063
	T2n	0.031	0.032	0.005	0.040	0.023	0.019
	T3	<LOQ	0.015	0.005	0.005	<LOQ	0.010
	mean	**0.033**	**0.020**	**0.006**	**0.017**	**0.011**	**0.031**
20-30	T1	0.061	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T2n	<LOQ	0.008	<LOQ	<LOQ	<LOQ	<LOQ
	T3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**0.020**	**0.030**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	T1	0.008	<LOQ	<LOQ	0.042	<LOQ	<LOQ
	T2n	0.036	<LOQ	<LOQ	0.029	0.012	<LOQ
	T3	0.018	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**0.021**	**<LOQ**	**<LOQ**	**0.024**	**0.004**	**<LOQ**
50-90	T1	ns	ns	ns	ns	ns	ns
	T2n	ns	ns	ns	ns	ns	ns
	T3	ns	ns	ns	ns	ns	ns
	mean	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**

Table 7.1.2.2.2- 4: Residues of M-01 in soil after annual applications of 500 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Plots T1 and T3

Depth [cm]	Sub plot	DAA1										
		0	**1**	**3**	**14**	**28**	**59**	**121**	**181**	**244**	**302**	**363**
0-10	T1	0.023	0.024	0.041	0.067	0.082	0.089	0.022	0.017	0.013	0.016	0.026
	T3	0.016	0.021	0.034	0.046	0.063	0.079	0.035	0.018	0.016	0.017	0.021
	mean	**0.020**	**0.023**	**0.038**	**0.057**	**0.073**	**0.084**	**0.029**	**0.018**	**0.015**	**0.017**	**0.024**
10-20	T1	ns	0.007	0.009	0.007	0.006	<LOQ	0.017	0.013	0.008	0.009	0.010
	T3	ns	0.009	0.006	0.007	0.006	<LOQ	0.021	0.019	0.013	0.010	0.009
	mean	**ns**	**0.08**	**0.008**	**0.007**	**0.006**	**<LOQ**	**0.019**	**0.016**	**0.011**	**0.010**	**0.010**
20-30	T1	ns	0.006	0.006	0.006	<LOQ	<LOQ	0.010	0.011	0.008	0.009	0.007
	T3	ns	0.006	<LOQ	0.006	<LOQ	<LOQ	0.007	0.016	0.011	0.009	0.009
	mean	**ns**	**0.006**	**0.003**	**0.006**	**<LOQ**	**<LOQ**	**0.009**	**0.014**	**0.010**	**0.009**	**0.008**
30-50	T1	ns	ns	ns	ns	ns	ns	0.006	0.009	0.009	0.007	0.007
	T3	ns	ns	ns	ns	ns	ns	<LOQ	0.011	0.01	0.007	0.006
	mean	ns	ns	ns	ns	ns	ns	**0.003**	**0.010**	**0.010**	**0.007**	**0.007**
50-90	T1	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
	T3	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
	mean	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**

Plots T2n

Depth [cm]	Sub plot	DAA1									
		0	**1**	**3**	**14**	**31**	**76**	**136**	**199**	**257**	**318**
0-10	T2n	0.025	0.027	0.048	0.056	0.059	0.029	0.019	0.017	0.015	0.017
10-20	T2n	ns	0.006	<LOQ	<LOQ	<LOQ	0.014	0.021	0.015	0.011	0.009
20-30	T2n	ns	<LOQ	<LOQ	<LOQ	<LOQ	0.008	0.016	0.015	0.011	0.007
30-50	T2n	ns	ns	ns	ns	ns	ns	0.007	0.007	<LOQ	<LOQ
50-90	T2n	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns

Table 7.1.2.2.2- 5: Residues of M-01 in soil after annual applications of 500 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3		
		0	119	372	0	83	355
0-10	T1	0.018	0.041	0.030	0.020	0.042	0.014
	T2n	0.020	0.045	0.036	0.019	0.033	0.017
	T3	0.020	0.055	0.029	0.018	0.034	0.015
	mean	**0.019**	**0.047**	**0.032**	**0.019**	**0.036**	**0.015**
10-20	T1	0.011	0.013	0.015	0.015	0.015	0.018
	T2n	0.008	0.016	0.011	0.012	0.017	0.009
	T3	0.009	0.011	0.013	0.012	0.006	0.010
	mean	**0.009**	**0.013**	**0.013**	**0.013**	**0.013**	**0.012**
20-30	T1	0.012	0.008	0.011	0.010	0.009	0.006
	T2n	0.007	0.008	0.007	0.009	0.009	0.006
	T3	0.009	<LOQ	0.009	0.008	<LOQ	0.006
	mean	**0.009**	**0.005**	**0.009**	**0.009**	**0.006**	**0.006**
30-50	T1	0.007	<LOQ	0.007	0.008	<LOQ	0.008
	T2n	0.007	<LOQ	<LOQ	0.006	<LOQ	<LOQ
	T3	0.008	<LOQ	0.006	0.007	<LOQ	0.007
	mean	**0.007**	**<LOQ**	**0.004**	**0.007**	**<LOQ**	**0.005**
50-90	T1	ns	ns	ns	ns	ns	ns
	T2n	ns	ns	ns	ns	ns	ns
	T3	ns	ns	ns	ns	ns	ns
	mean	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**

Table 7.1.2.2.2- 6: Residues of M-02 in soil after annual applications of 500 g a.s./ha DISSIPATION phase 2000-2002 values expressed as mg/kg

Plots T1 and T3

Depth [cm]	Sub plot	DAA1										
		0	**1**	**3**	**14**	**28**	**59**	**121**	**181**	**244**	**302**	**363**
0-10	T1	<LOQ	<LOQ	0.010	0.031	0.048	0.045	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T3	<LOQ	<LOQ	0.011	0.025	0.038	0.047	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**0.011**	**0.028**	**0.043**	**0.046**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	T1	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T3	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**ns**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	T1	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T3	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**ns**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	T1	ns	ns	ns	ns	ns	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T3	ns	ns	ns	ns	ns	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
50-90	T1	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
	T3	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
	mean	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**

Plots T2n

Depth [cm]	Sub plot	DAA1									
		0	**1**	**3**	**14**	**31**	**76**	**136**	**199**	**257**	**318**
0-10	T2n	<LOQ	0.009	0.016	0.034	0.026	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
10-20	T2n	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
20-30	T2n	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
30-50	T2n	ns	ns	ns	ns	ns	ns	<LOQ	<LOQ	<LOQ	<LOQ
50-90	T2n	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns

Table 7.1.2.2.2- 7: Residues of M-02 in soil after annual applications of 500 g a.s./ha ACCUMULATION phase values expressed as mg/kg

Depth [cm]	Sub plot	DAA2			DAA3		
		0	119	372	0	83	355
0-10	T1	<LOQ	0.007	<LOQ	<LOQ	<LOQ	<LOQ
	T2n	<LOQ	0.006	<LOQ	<LOQ	<LOQ	<LOQ
	T3	<LOQ	0.008	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**0.007**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
10-20	T1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T2n	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
20-30	T1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T2n	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
30-50	T1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T2n	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	T3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	mean	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**	**<LOQ**
50-90	T1	ns	ns	ns	ns	ns	ns
	T2n	ns	ns	ns	ns	ns	ns
	T3	ns	ns	ns	ns	ns	ns
	mean	**ns**	**ns**	**ns**	**ns**	**ns**	**ns**

C. Residues

Dissipation phase (2000-2001)

The measured initial concentration of fluopicolide was 0.383 mg/kg (mean value) immediately after application and 0.503 mg/kg (mean value) three days later, equivalent to 574.5 g/ha and 758.5 g/ha, respectively assuming a soil density of 1.5 g/cm^3. These deviations to the nominal application rate of 500 g/ha appear to be acceptable taking into account the uncertainties (e.g. soil density, homogenisation, application) related to the samples collected soon after treatment and considering the residues remaining in soil from the previous application in 1999 conducted as part of a separate study (KCA 7.1.2.2.1/04, M-220472-02-1).

The initial dissipation of fluopicolide was relatively rapid followed by a slower dissipation phase during the winter months due to the cold climate. Dissipation continued the following summer at a slower rate than the initial rapid phase possibly due to the reduced availability of fluopicolide due to increased adsorption to soil with ageing. Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the trial. Low residues were detected in the 10-20 cm horizon in the dissipation phase at concentrations ranging from 0.005 to 0.017 mg/kg (mean values). Residue levels of parent declined to 129 g/ha one year after the first treatment which represented approximately 20 % of the measured (758.5 g/ha) initial concentration. Throughout the dissipation phase no residues of fluopicolide were detected above the LOQ below 20 cm depth.

The concentration of M-01 (AE C653711) in the soil profile varied with the degradation rate of fluopicolide. During the summer months when the degradation rate of the parent compound is relatively rapid, M-01 concentrations were relatively high and declined during the winter as the degradation rate of fluopicolide slowed. Residues levels of M-01 reached a maximum one to two months after first

application at a mean concentration equivalent to 109.5 g/ha in summer 2000 (mean of Plots T1 and T3, 126 g/ha). After one year residue levels of M-01 had declined to 81.0 g/ha (June 2001). The maximum residue level in the 0-10 cm horizon was observed 59 days after application at 0.084 mg/kg (mean of Plots T1 and T3) and 31 days after application at 0.059 mg/kg (Plot T2n). The peak concentration in the underlying 10-20 cm was slightly later, after 121 days at 0.019 mg/kg and 136 days at 0.021 mg/kg in Plots T1/T3 and T2n, respectively. In the lower soil horizons residue levels were generally at or below the LOQ until the winter months where the maximum concentration observed was 0.016 mg/kg and 0.010 mg/kg at 20-30 cm and 30-50 cm soil depths, respectively.

M-02 and M-03 were rapidly degraded in the trial. No residues of M-03 (AE 0608000) were found above the LOQ throughout the study. M-02 was only detected at early time-points in the 0-10 cm soil depth up to 59 days after application in Plots T1 and T3 (31 days in Plot T2n) at a maximum concentration of 0.046 mg/kg, equivalent to 69 g/ha. No residues of M-02 were detected above the LOQ below 10 cm depth. The degradation of M-03 is known to be pH dependant and is very rapidly degraded in neutral to alkaline soils such as the soil at the Senas trial site.

Accumulation:

For the assessment of the plateau concentrations, the residue levels after the first application were not considered appropriate as the soil concentrations of fluopicolide measured after the first application indicated the rate applied had significantly exceeded the intended application rate of 500 g/ha. Therefore only the second and third year were considered for the evaluation of the plateau concentrations.

The average concentration of fluopicolide in soil immediately after the second application in 2001 was 0.354 mg/kg in 0 to 10 cm soil depth which declined to 0.082 mg/kg by 372 days after the second treatment (DAA2). A mean concentration of 0.354 mg/kg was detected immediately after the third application in 2002 which declined to 0.061 mg/kg in upper 10 cm soil depth by 355 days after the third application (DAA3).

Residues of fluopicolide were detected mainly in the 0-10 cm soil horizon throughout the accumulation phase. Low residues were detected in the 10-20 cm soil depth at concentrations ranging from 0.006 to 0.033 mg/kg (mean values) throughout the accumulation phase. In the 20-30 cm soil depth fluopicolide was detected in October 2001 at a mean concentration of 0.003 mg/kg. Residues of the test item detected in 20-30 cm and 30-50 cm soil depths immediately after second and third applications were concluded to be a result of sample contamination during sampling.

The metabolite M-01 was detected in 0-10 cm soil depth at a maximum concentration of 0.047 mg/kg (mean value). Levels in the 10-20 cm soil were lower with a maximum of 0.013 mg/kg (mean value). In the 20-30 cm layer residues reached a maximum of 0.009 mg/kg (mean value) and did not exceed 0.007 mg/kg (mean value) in 30-50 cm depth.

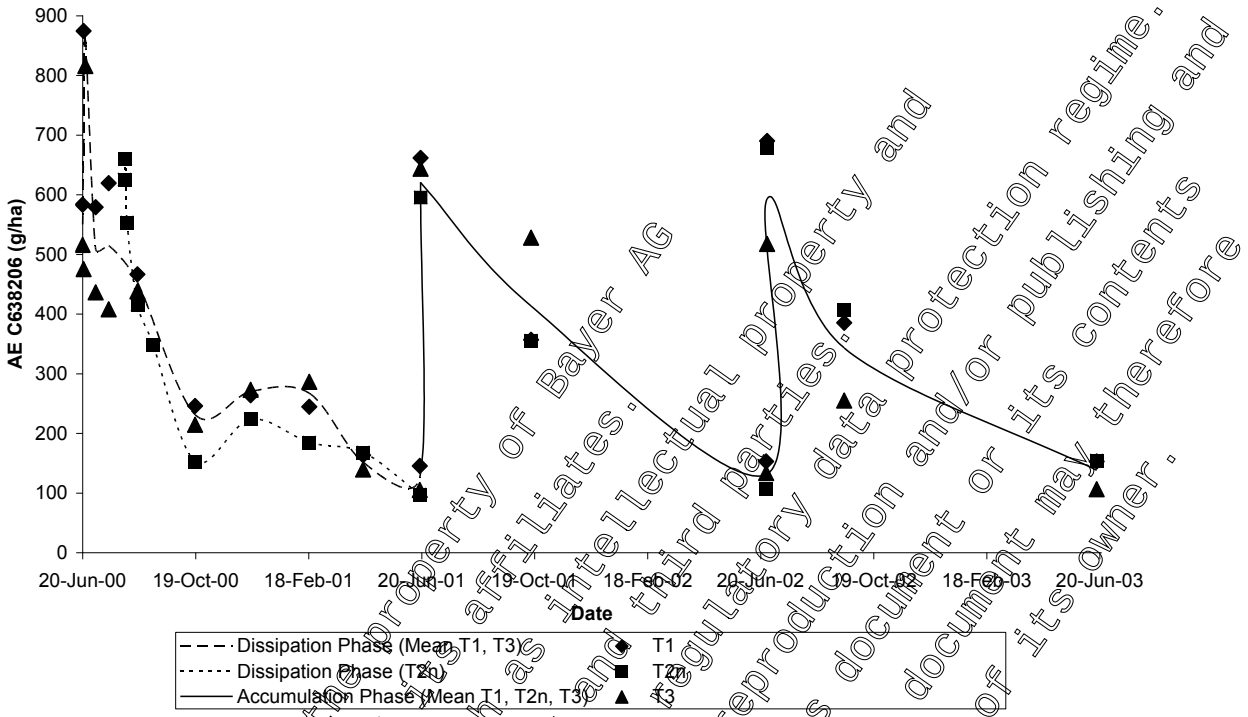
Residues of M-02 were detected only once throughout the accumulation phase at a concentration of 0.007 mg/kg. No residues of M-03 were detected throughout the trial.

The plateau concentrations after three years are summarised below. As the accumulation trial was conducted with bare soil plots and the fungicide fluopicolide would only be applied to growing crops, soil plateau concentrations following normal agricultural use would be significantly lower.

Plateau concentration	Time-point	Fluopicolide (mg/kg)		Time-point	AE C653711 (mg/kg)	
		0-10 cm	0-20 cm		0-10 cm	0-20 cm
High[1]	Day 0 3rd Application	0.354	0.186	Day 119 after 2nd Application	0.047	0.030
Low[2]	Day 355 after 3rd Application	0.061	0.046	Day 355 after 3rd Application	0.015	0.014

[1] maximum of the high values of the "saw teeth" curve
[2] maximum of the low values of the "saw teeth" curve

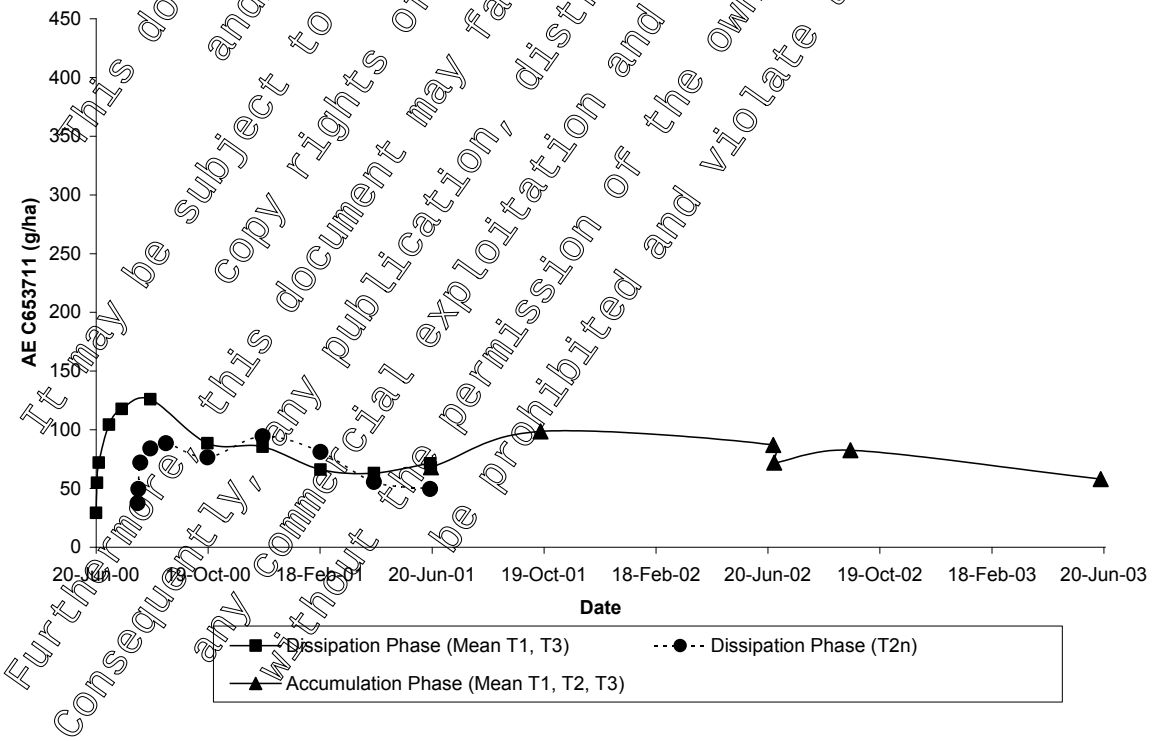
Figure 7.1.2.2.2- 2: Dissipation and Accumulation of Fluopicolide at Senas



Mean values are given as solid and dashed lines for the accumulation phase and dissipation phase as indicated in the key. The results for individual plots are also given for fluopicolide as solid symbols.

Figure 7.1.2.2.2- 3: Dissipation and Accumulation of M-01 at Senas

NB Scale different



D. Kinetic Analysis

The half-life of fluopicolide included in the report was calculated using a biphasic hockey stick kinetic model (HS) as 58 days. The DT_{90} was 679 days (tb 69.8 days) and the r^2 was 0.928. The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. Moreover, the data is no longer considered suitable to derive degradation rates as the experimental plots had previously been treated with fluopicolide (in 1999, see KCA 7.1.2.2.1/04).

III. Conclusion

The accumulation potential of fluopicolide and its metabolites M-01, M-02 and M-03 was assessed for up to three years after application to bare soil at a site in Senas, France. Fluopicolide and M-01 degraded at a moderately fast rate in soil. The reported DT_{50} and DT_{90} values were 58 and 679 days, respectively, assuming biphasic hockey stick kinetics. The metabolites M-01 and M-02 were rapidly degraded in soil and were either not detected or disappeared completely within one month.

It was concluded during the previous evaluation that fluopicolide and M-01 residues had reached plateau concentrations in the Senas trial. Accumulation measured in the field did not indicate a significantly higher plateau than that observed following a single annual application.

In this submission the definitive assessment of the accumulation of fluopicolide and its metabolites in soil is addressed in Document MCP-9 by calculation.

Assessment and conclusion by applicant
The study is considered valid to assess the accumulation of fluopicolide and M-01 in soil under field conditions. Definitive assessment of the accumulation of fluopicolide and its metabolites in soil is addressed in Document MCP-9 by calculation.

Data Point:	KCA 7.1.2.2.2/02
Report Author:	[redacted]
Report Year:	2006
Report Title:	An evaluation of the potential accumulation of fluopicolide in the field
Report No:	CX/06/005A
Document No:	M-267721-01-1
Guideline(s) followed in study:	91/414/EEC
Deviations from current test guideline:	not specified
Previous evaluation:	yes, evaluated and accepted Addendum 1 to the DAR (2007)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

For procedural reasons the previously submitted statement is included under Point KCA 7.1.2.2.2 in the current dossier (KCA 7.1.2.2.2/02). However the report has been fully superseded by PEC_{soil} accumulation calculations provided in Document MCP-9. Consequently no summary of the statement has been included in this dossier.