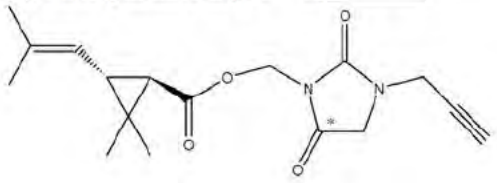
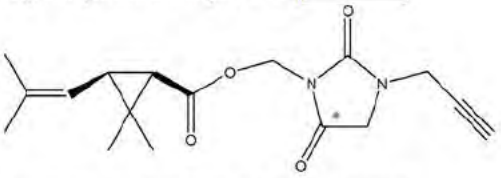
			Official use only
		1. REFERENCE	
1.1	**Reference**	[redacted] Aerobic Soil Metabolism of [^{14}C][redacted]. PTRL West Inc, 625-B Alfred Nobel Drive, Hercules, CA 94547 USA. [redacted], 11 October 2004. Duration of work: 10 April 2003 – 11 October 2004	
1.2	**Data protection**	Yes	
1.2.1	Data owner	Sumitomo Chemical Co. (SCC) Ltd, Japan	
1.2.2	Criteria for data protection	Data submitted to the MS after 13 May 2000 on a new active substance. for inclusion in Annex I of Commission Directive 98/8/EC.	
		2. GUIDELINES AND QUALITY ASSURANCE	
2.1	**Guideline study**	Yes EPA Pesticide Assessment Guidelines Subdivision N, Chemistry: Environmental Fate Series 162-1.	
2.2	**GLP**	Yes	
2.3	**Deviations**	No	
		3. MATERIALS AND METHODS	
3.1	**Test material**	(1*R*)-*trans*-[imidazolinyl-5-^{14}C][redacted]	X
			
		(1*R*)-*cis*-[imidazolinyl-5-^{14}C][redacted]	
			
		* indicates the position of the radiolabel.	
3.1.1	Lot/Batch number	[redacted]	
3.1.2	Specification	Specific activity: 5.04 MBq/mg for both test materials	
3.1.3	Description	Not reported	
3.1.4	Purity	[redacted]	
3.1.5	Stability	Test material stability under conditions of administration was confirmed upon HPLC analysis of time zero sample extracts	
3.2	**Reference substances**	Reference standards [redacted]	

Structures are shown in Table 7.2.1-1

3.3 Testing procedure

3.3.1 Test system

Mutchler sandy clay loam soil from Grand Forks county, North Dakota was used. The soils were passed through a 2 mm sieve and maintained in the dark until use in the study. X

Soil properties

Texture analysis:	
Classification:	Sandy clay loam
Organic matter:	3.2%
pH (in 1:1 soil:H_2O):	7.4
CEC* (meq/100 g):	20.2
Bulk density (g/cc):	1.09
Percent moisture (at 0.33 bar):	24.9%
Max H_2O holding capacity (g/100 g):	40.7

* cation exchange capacity

Soil (equivalent to 50 g dry weight) was weighed into 250 mL amber bottles, moistened with deionised water to 75% of 1/3 bar moisture and incubated at 25 ± 1°C in a Hotpack Walk-in constant temperature chamber in the dark. Approximately 5 days before dosing, bottles were attached to a continuous source of humidified CO_2 free air flow. Each soil sample was connected to individual traps containing ethylene glycol (30 mL) to collect organic volatiles, and two caustic traps (10% aqueous KOH, 30 mL) to collect $^{14}CO_2$. Microbial activity of the soil was determined at the start and end of the study and confirmed soil to be viable throughout.

Dosing solutions were prepared by adding an aliquot of the respective [^{14}C][redacted] stock solution to an amber vial, evaporating the solvent under nitrogen, and reconstituting with acetonitrile to the desired concentration.

Aliquots of dosing solution (100 µL) were directly added to soil samples in a spiral motion, applying as evenly as possible. Application rates were 1.07 and 1.16 µg/g of dry soil for the *trans* and *cis* labelled samples, respectively. Flasks were then capped and shaken to ensure homogeneity of applied soils, connected to the trapping system, and maintained at 25 ± 1°C in the dark. Aliquots of application solutions (10 µL) were taken before, during and after application, and combined with 990 µL acetonitrile in an amber vial. Aliquots (3 x 100 µL) of these dilutions were radioassayed (via liquid scintillation counting (LSC)) to determine application rates and demonstrate test solution homogeneity during application. Soil samples were weighed approximately once every two weeks, and soil moisture adjusted to original levels by addition of sterile deionised water.

3.3.2 Sampling

Soil and volatile trap samples were taken for analysis immediately after application and at 0.25, 1, 2, 4, 7, 14, 30, 61 and 63 days for [*trans*-^{14}C][redacted], and at 0.25, 1, 2, 4, 7, 14, 30, 63 and 64 days for [*cis*-^{14}C].

3.3.3 Extraction and analysis

Duplicate soil samples were extracted with 50 mL of acetonitrile:0.1 N HCl (5:1, v/v). Samples were shaken for 30 minutes, centrifuged at 2500 rpm for 10 minutes, and supernatants decanted. The extraction procedure was repeated twice with two more 50 mL aliquots of fresh extraction solvent. Extracts were combined (3 x 1 mL) and aliquots taken for radioassay (LSC).

Aliquots of soil extracts were analysed via HPLC by direct injection. Soil extracts were also reanalysed by HPLC after concentration/dilution with deionised water, with aliquots of soil extracts diluted to achieve a water:acetonitrile ratio of 7:1 (v/v) prior to reanalysis. If necessary, an aliquot of soil extract was concentrated under nitrogen prior to dilution. The volume of concentrated soil extract used in the concentration step varied depending on the level of radioactivity of each sample.

Extracted soil residues were weighed and aliquots (4 x 0.5 g) combusted (with a Harvey OX-600 Biological Oxidizer) to determine levels of unextracted radiocarbon. Generated $^{14}CO_2$ was trapped with Carbon 14 Cocktail and ^{14}C content determined by LSC. The unextracted residues were further characterised by fractionation into humic acid, fulvic acid and humin following extraction with 25 mL of 0.5 N aqueous NaOH.

The radioactivity in organic and aqueous fractions (extractions of soils, trapping solutions and bound residue extracts) was determined by liquid scintillation counting (LSC) with a Beckman LS 5000 CE or LS 6000 IC liquid scintillation spectrometer equipped with an automatic external standard in standard polyurethane counting vials, using 5 or 15 mL of Safety Solve scintillation cocktail (Research Products International Corp.). All radioassays were conducted in triplicate (1 mL aliquots).

Carbonate ions in the caustic traps, including any due to $^{14}CO_2$ were precipitated by addition of aqueous $BaCl_2$ solution to aliquots (combined in a centrifuge tube) of trap solution in equal volumes, resulting in the quantitative precipitation of the radiocarbon in the KOH traps* as $Ba^{14}CO_3$. The supernatant was separated by centrifugation and aliquots (3 x 1 mL) analysed by LSC to detect the presence of soluble species. The amount of $^{14}CO_2$ was determined by subtraction of the radioactivity in the supernatant from the initial radioactivity in the potassium hydroxide solution.

*It should be noted that there is a typo in the study report (page 33) in which the KOH caustic traps are mistakenly referred to as NaOH traps.

Aliquots from each soil extract were analysed by high-performance liquid chromatography (HPLC) for quantification of [redacted] and its soil metabolites.

Pump:	Thermo separator products SP 8800 ternary pump
UV detector:	Thermo separator products UV detector TSP 100UV/VIS detector at 230 nm
Column:	Waters YMC ODS-A (5 µm, 4.6 mm i.d. x 25 cm)

The following gradient system was employed:

Time(min)	% A	% B	% C	Flow rate
0	100	0	0	1.0 mL/min
5	100	0	0	1.0 mL/min
10	95	5	0	1.0 mL/min
30	60	40	0	1.0 mL/min
45	30	0	70	1.0 mL/min
50	30	0	70	1.0 mL/min
55	0	30	70	1.0 mL/min
58	0	30	70	1.0 mL/min
62	100	0	0	1.0 mL/min
80	100	0	0	1.0 mL/min

Mobile Phase A = 0.2% Trifluoroacetic acid (TFA) in HPLC grade water
Mobile Phase B = 0.2% TFA in HPLC grade acetonitrile
Mobile Phase C = 0.2% TFA in HPLC grade methanol

Two-dimensional thin layer chromatography (TLC) was performed for selected samples to confirm peak assignment for [redacted] and degradates, using pre-coated silica gel $60F_{254}$ TLC plates (20 x 20 cm, 0.25 mm thickness, Merck). Solvent systems used were chloroform:acetonitrile: acetic acid (9:1:1, v/v/v) in the first dimension and butanol:acetic acid:water (6:1:1, v/v/v) in the second dimension. The non-radiolabelled reference standards were detected by exposing TLC plates to UV light (254 nm) or iodine vapour. Plates were scanned with a Storm 820 optical scanner for ^{14}C detection.

Retention times (Rt) and R_f values for each [redacted] isomer and reference standards (which were co-chromatographed with all samples) are shown in Table 7.2.1-1.

3.3.4 Characterisation and identification of transformation products

Quantitation was based upon HPLC analysis. The structural assignments for [^{14}C][redacted] and degradates were based on co-chromatography with reference standard upon HPLC and TLC analysis. Reference standards were co-chromatographed with all samples.

Selected soil extracts for the *cis* and *trans* labelled sets were spotted as a tight band on a silica gel TLC plate and eluted in one dimension using solvent system B [butanol:acetic acid:water (6:1:1, v/v/v)]. A polar band eluting at solvent front (DT1 for the *trans* label, retention time of 3.5

min, and DC1 for the *cis* label, retention time of 3.9 min) was isolated by extraction of the silica gel with methanol, shaking for 30 minutes and decanting the supernatant layer, following centrifugation. An aliquot of the isolated band was treated with a diazomethane solution in ethyl ether. The methylated and untreated band extracts were then analysed by HPLC and 2D TLC.

4 RESULTS

4.1 Dissipation time

The degradation rate constant (k) and half-life ($t½$) of each [^{14}C][redacted] isomer were calculated assuming first-order kinetics. X

Degradation constants were calculated using the percent [redacted] in soil samples, using the following equation:

$$\ln C = kt + \ln C_0 \quad (y = mx + b)\ (m = \text{slope})$$

Where;

k = degradation rate constant

t = time

C = concentration of [redacted] (%)

C_0 = initial concentration of [redacted] (%)

Half-lives were calculated using the following equation:

$$t½ = \frac{In\ 2}{-m} = \frac{0.693}{k}$$

Test substance	Half-life (days) [first order]	Correlation coefficient (r^2) [first-order]
Trans-[redacted]	4.8	0.8728
Cis-[redacted]	14.6	0.9034

Additionally, DT_{50} and DT_{90} values were determined using the Gustafson equation based on an infinite spatial variability model:

$$C = C_0(1 + \beta t)^{-\alpha}$$

Where;

C = concentration of [redacted] (%)

C_0 = initial concentration of [redacted] (%)

t = time of incubation

α, β = constants*

*Constant parameters were estimated by non-linear regression curve fit using NLREG program. α is unitless and β is in days^{-1}.

The DT50 and DT90 are calculated using the following equations:

$$DT_{50} = (0.5^{-1/\alpha} - 1)/\beta$$

$$DT_{90} = (0.1^{-1/\alpha} - 1)/\beta$$

Test substance	DT_{50} (days) [Gustafson]	DT_{90} (days) [Gustafson]	r^2 [Gustafson]
Trans-[redacted]	1.8	7.5	0.9929
Cis-[redacted]	9.6	42.5	0.9814

When the data was fitted to first-order kinetics, correlation coefficients indicated that degradations rates for [redacted] were not linear, especially for the *trans*- labelled set. In contrast, Gustafson's equation afforded better correlation of both isomers (0.9929 and 0.9814).

Since this study was conducted in 2004 and the kinetics evaluation above was not in accordance with any guidance, the applicant has re-assessed the kinetics according to the FOCUS degradation kinetics guidance (2014) using the Cake model (ver 3.2). Summarized parameters of the single first order fitting are presented below and the full report from CAKE is given in Figure 7.2.1-2.

Test substance	DT_{50} (days)	DT_{90} (days)	k	χ^2	r^2
Trans-[redacted]	1.93	6.41	0.3595	5.36	0.9922
Cis-[redacted]	9.83	32.7	0.0705	4.09	0.9831

4.2 Concentration – time data

Distribution of radioactivity in sample matrices is shown in Table 7.2.1-2.

The total ^{14}C recovery was 92.3 to 107.1% and 77.3 to 105.1% of the applied ^{14}C in *trans* and *cis* samples respectively. The amount of extractable ^{14}C gradually decreased to average levels of 4.4 and 11.0% at two months in *trans* and *cis* samples respectively, with formation of $^{14}CO_2$ amounting to average levels of 59.0 and 40.3% in *trans* and *cis* samples respectively. Trapped organic volatiles comprised ≤0.1% of dose throughout the study period in both isomer samples. Unextracted soil residues increased to averages of 41.9 and 32.8% in *trans* and *cis* samples respectively. Further characterisation of unextractable radiocarbon at day 30 and at the end of the two month period by fulvic and humic acid partitioning recovered an average of 9.9 and 3.1% of the dose in fulvic and humic acid fractions respectively, and 19.1% associated with humin in *trans* samples, and 8.7 and 3.0% of the dose in fulvic and humic acid fractions respectively, and 16.3% associated with humin in *cis* samples.

HPLC analysis of soil extracts showed rapid [redacted] degradation, with levels representing <1.0% and 7.1% of the applied ^{14}C dose in *trans*- and *cis*- isomers respectively after 2 months.

X

4.3 Specification of the transformation products

Section 3.3.4 details how each of the degradates were isolated and characterised.

Table 7.2.1-3 shows the amounts of *trans*-, *cis*- [redacted] isomers and their degradation products in Mutchler Sandy Clay Loam Soil.

Several degradates were identified in soil extracts by HPLC analysis. The main degradates observed for both test substances were PGH (39.4 and 14.4% of the applied dose at 4 and 14 days for *trans*- and *cis*- respectively, decreasing below detection limits at two months), CPG-Me (3.7 and 2.8% at 14 and 30 days for *trans*- and *cis*- labels respectively, decreasing to an average of 0.4% at two months in both isomers), and a polar band eluting at the solvent front (DT1 for *trans* label, 12.9% of dose at 14 days and declining to an average of 2.9% at two months, DC1 for *cis* label, 6.4% of dose at 30 days and declining to an average of 4.2% at two months). Further characterization of the polar bands showed that they were comprised of CPG and several other polar degradates, none present as greater than 5.2% of the applied dose.

A proposed degradation pathway for [redacted] is shown in Figure 7.2.1-1.

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1 Materials and methods

An aerobic soil metabolism study was conducted with [^{14}C][redacted], in accordance with the EPA Pesticide Assessment Guidelines Subdivision N, Section 162-1. Mutchler sandy clay loam soils was moistened to 75% of 1/3 bar moisture and fortified with (1R)-*trans*- or (1R)-*cis*-isomer of [imidazolinyl-5-^{14}C][redacted] at concentrations of 1.07 and 1.16 ppm on a dry weight basis, respectively. The test system was incubated in amber glass bottles maintained at 25 ± 1°C in the dark for up to two months.

Humidified CO_2-free air was continuously passed in sequence through one ethylene glycol and two potassium hydroxide traps for collection of organic volatiles and $^{14}CO_2$, respectively.

Each soil sample was analysed immediately after application and at 0.25, 1, 2, 4, 7, 14, 30, 61 and 63 days post-treatment for [*trans*-^{14}C][redacted], and at 0.25, 1, 2, 4, 7, 14, 30, 63 and 64 days post-treatment for [*cis*-^{14}C][redacted].

The soil samples were extracted in duplicate with acetonitrile:0.1 N HCl (5:1, v/v). The soil extracts were analysed by HPLC analysis. *Trans* and *cis* [^{14}C][redacted] and their degradates were identified by 2D-TLC and/or HPLC co-chromatography with reference standards. The radioactivity of unextractable soil residues was quantified as $^{14}CO_2$ by combustion followed by radioassay.

5.2 Results and discussion

The total ^{14}C recovery was 92.3 to 107.1% and 77.3 to 105.1% of the applied ^{14}C in *trans* and *cis* samples respectively. The amount of extractable ^{14}C gradually decreased to average levels of 4.4 and 11.0% at two months in *trans* and *cis* samples respectively, with formation of $^{14}CO_2$ amounting to 59.0 and 40.3% in *trans* and *cis* samples respectively. Unextractable residues accounted for an average of 41.9 and 32.8% of the applied dose at the end of the study in *trans* and *cis* samples respectively. Further characterisation of unextractable radiocarbon at day 30 and at the end of the two-month incubation period by fulvic and humic acid partitioning recovered an average of 9.9 and 3.1% of the dose in fulvic and humic acid fractions respectively, and 19.1% associated with humin in *trans* samples, and 8.7 and 3.0% of the dose in fulvic and humic acid fractions respectively, and 16.3% associated with humin in *cis* samples.

HPLC analysis of soil extracts showed rapid [redacted] degradation, with levels representing <1.0% and 7.1% of the applied ^{14}C dose in *trans*- and *cis*- isomers respectively at two months.

Several degradates were identified in soil extracts by HPLC analysis. The main degradates observed for both test substances were PGH (39.4 and 14.4% of the applied dose at 4 and 14 days for *trans*- and *cis*- respectively, decreasing below detection limits at two months), CPG-Me (3.7 and 2.8% at 14 and 30 days for *trans*- and *cis*- labels respectively, decreasing to an average of 0.4% at two months in both isomers), and a polar band eluting at solvent front (DT1 for *trans* label, 12.9% of dose at 14 days and declining to an average of 2.9% at two months, DC1 for *cis* label, 6.4% of dose at 30 days and declining to an average of 4.2% at two months). Further characterization of the polar bands showed that they were comprised of CPG and several other polar degradates, none present as greater than 5.2% of the applied dose.

The re-assessed half-life of the *trans*-isomer, assuming single first order kinetics, was estimated to be 1.93 days. The re-assessed half-life of the *cis*-isomer, assuming single first order kinetics, was estimated to be 9.83 days.

5.3 Conclusion

[redacted] X

[redacted]

5.3.1	Reliability	[redacted]
5.3.2	Deficiencies	The moisture content of the soil (75% of 1/3 bar) was slightly lower than recommended in the European Guidelines.

Evaluation by Competent Authorities

Use separate "evaluation boxes" to provide transparency as to the comments and views submitted

EVALUATION BY RAPPORTEUR MEMBER STATE

Date

03/02/2017

Materials and Methods

The Applicant's version is considered to be acceptable noting the following:

3.1 The positioning of the radiolabel means that only half of the degradation pathway could be followed.

3.3.1 OECD 307 recommends at least three soil types are used for determination of rates of transformation, and that these soils should be representative of the environmental conditions where use or release will occur. This study used one soil, a sandy clay loam pH 7.4 from North Dakota. The majority of European soils have a pH of < 7. No history of the field site from where the test soil was collected was provided. No information was provided on soil collection, storage, preparation, or any pre-incubation.

Soil moisture content was maintained at 75 % field capacity, which is close to the OECD 307 guideline of a pF between 2 and 2.5.

A test temperature of 25 °C was used, higher than the OECD recommended 20 °C.

As no untreated controls were included, microbial biomass could not be calculated pre and post experiment. However, microbial viability was tested prior and post experiment using plate count methods, and although levels of aerobic bacteria, actinomycetes and total fungi had decreased over the test duration (i.e., 50 % reduction in total aerobic bacteria), the soil remained viable.

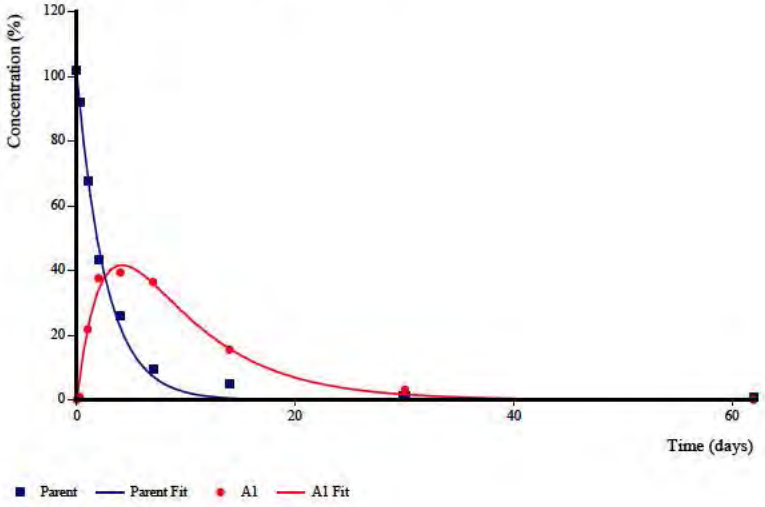
No sterile control was provided, therefore no distinction can be made between abiotic and biotic degradation of the test substance.

Results and discussion

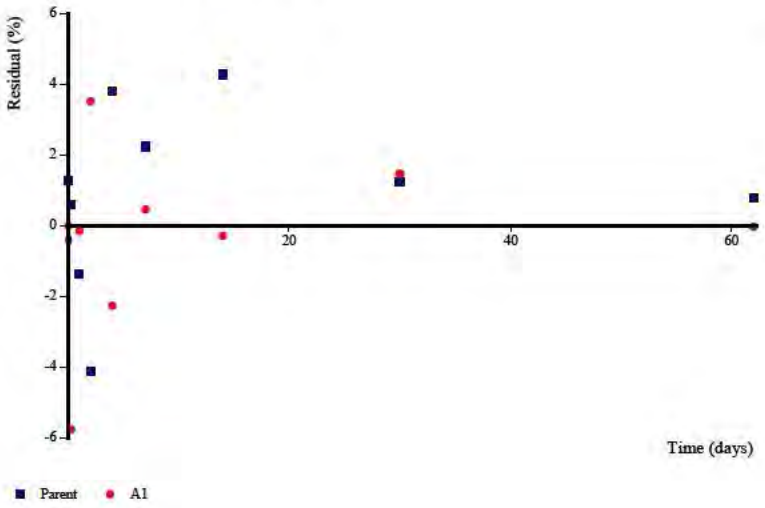
4.1 and **5.3** The eCA has repeated the kinetic analysis using CAKE 3.1 using the sequential fitting of parent with the major metabolite PG. The iteratively reweighted least squares method of minimisation (IRLS) was used and a day 0 total recovery value was used to account for early degradation in line with FOCUS recommendations. All fits are SFO (SingleFirst Order).

Data Set: *Trans*-Imiprothrin and PG

Observations and Fitted Model:



Residuals:



Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	100.6	2.181	N/A	96.71	104.4	95.86	105.3
k_Parent	0.3767	0.02201	1.34E-10	0.3377	0.4157	0.3291	0.424
f_Parent_to_A1	0.7491	0.06549	N/A	0.6331	0.8651	0.6076	0.891
k_A1	0.1427	0.02004	3.90E-06	0.1072	0.1782	0.09944	0.186

χ^2

Parameter	Error %	Degrees of Freedom
All data	7.64	13
Parent	5.39	7
A1	10.6	6

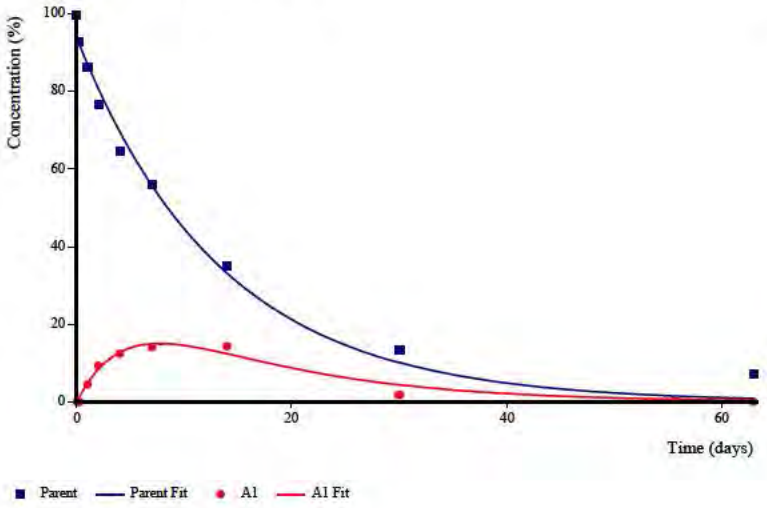
Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	1.84	6.11
A1	4.86	16.1

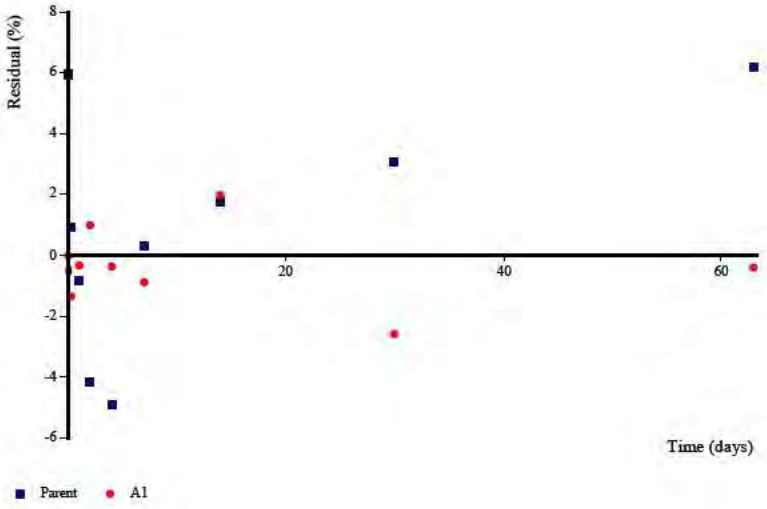
On the basis of the low chi^2 error values and good visual fits based on traditional and residual plots the UK CA considered the SFO fits to be acceptable and no further assessment of non-SFO kinetic models was considered necessary.

Data Set: *Cis*-Imiprothrin and PG

Observations and Fitted Model:



Residuals:



Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	93.67	1.835	N/A	90.42	96.92	89.7	97.63
k_Parent	0.07415	0.0052	1.26E-009	0.06496	0.08334	0.06294	0.085
f_Parent_to_A1	0.7981	0.2366	N/A	0.379	1.217	0.2868	1.309
k_A1	0.2083	0.0855	0.01497	0.05694	0.3598	0.02364	0.393

χ^2

Parameter	Error %	Degrees of Freedom
All data	7.32	13
Parent	5.13	7
A1	15.1	6

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	9.35	31.1
A1	3.33	11.1

On the basis of the low chi^2 error values and good visual fits based on traditional and residual plots the UK CA considered the SFO fits to be acceptable and no further assessment of non-SFO kinetic models was considered necessary.

Summary of Calculated DT_{50} and DT_{90} values

Compound	**Measured DT_{50} (d) at 25 °C**	**Measured DT_{90} (d) at 25 °C**	**χ^2**	**Prob. > t**
trans-imiprothrin	1.84	6.11	5.39	1.34E-10
PGH from *trans*-imiprothrin	4.86	16.1	10.6	3.90E-06
cis-imiprothrin	9.35	31.1	5.13	1.26E-09
PGH from *cis*-imiprothrin	3.33	11.1	15.1	1.50E-02

4.2 Recovery of the *cis* sample was as low as 77.3 %, outside of OECD recommended 90 to 110 % range. Recoveries only fell outside the OECD recommended range on days 63 and 64.

Unextracted soil residues increased to 41.9 in the *trans*-labelled samples, and 32.8 % in the *cis*-labelled samples. Analysis of selected soil bound residues revealed that most activity was associated with the fulvic fraction (see table below);

Fraction	% of bound residues		% of initial dose	
	Sample 1	Sample 2	Sample 1	Sample 2
Fulvic Acid	29.8	20.1	11.4	8.4
Humic Acid	7.9	7.3	3.0	3.1
Humin	44.9	49.8	17.2	20.9

Radiocarbon trapped in caustic solutions increased to 59.0 % in the *trans*-labelled samples, and 37.5 % in the *cis*-labelled samples.

The main metabolite of the *trans* isomer were PGH (detected at a maximum of 39.4 % total recovered radioactivity at 4 d). A polar band designated DT1was detected at a maximum of 12.9 % total recovered radioactivity at 14 d. Further characterization of the polar band showed that it was comprised of CPG (5.2 % of dose) and several other polar degradates, with none present as greater than 3.9 % of the applied dose.
The main metabolite of the c*i*s isomer were PGH (detected at a maximum of 14.4 % total recovered radioactivity at 14 d). A polar band designated DC1was detected at a maximum of 6.4 % total recovered radioactivity at 30 d. Further characterization of the polar band showed that it was comprised of CPG (3.1 % of dose) and several other polar degradates, with none present as greater than 2.0 % of the applied dose.

Conclusion The applicant's version is considered to be acceptable.

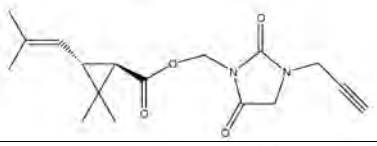
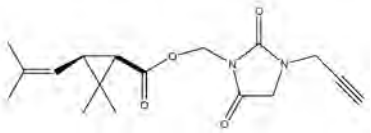
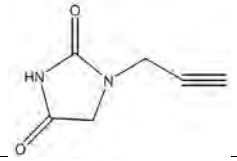
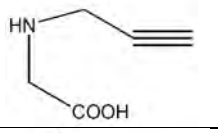
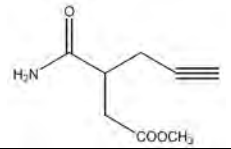
Reliability [redacted]

Acceptability The Applicant's version is considered to be acceptable.

Remarks	This study deviates from the OECD 307 test guidelines in two ways that may affect the reliability of the results. First, the dose rate is higher than that expected to be found in the environment. This may lead to microbial inhibition, and the crude plate count method does seem to show a reduction in microbial life over the duration of the test. Second, recovery rates are low at the final time point. It is the view of the eCA that these deviations are acceptable, as the first will lead to an overestimation of degradation times and the good fits obtained suggest that the second did not have a large effect on the data. (If the kinetic analysis is repeated omitting the final data point, it makes a minimal difference to the fits obtained for the *trans* isomers. For *cis*-imiprothrin, the DT_{50} changes from 9.35 to 9.24 d, and the DT_{90} from 31.1 to 30.7 d., and for PGH, , the DT_{50} changes from 3.33 to 3.37 d, and the DT_{90} from 11.1 to 11.2 d)

	COMMENTS FROM ...
Date	*Give date of comments submitted*
Materials and Methods	*Discuss additional relevant discrepancies referring to the (sub)heading numbers and to Applicant's summary and conclusion.* *Discuss if deviating from view of rapporteur member state*
Results and discussion	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	

Table 7.2.1-1: Structures of non-radiolabelled reference compounds and their chromatographic properties

Designation	Structure	Retention time HPLC Rt (min)	Rf Value TLC	
			Solvent A[1]	Solvent B[2]
trans-[redacted]		48.4	0.61	0.78
cis-[redacted]		47.7	0.61	0.80
[redacted]		14.1	0.31	0.66
[redacted]		3.7	0.00	0.04
[redacted]		17.1	0.30	0.59

[1] Solvent A = chloroform:acetonitrile:acetic acid, 9:1:1 (v/v/v)
[2] Solvent B = butanol:acetic acid:water, 6:1:1 (v/v/v)

Table 7.2.1-2: Distribution of applied radioactivity in Mutchler Sandy Clay Loam Soil under aerobic conditions

Percent of applied radioactivity following application of [*Trans*–^{14}C][redacted]					
Sample time (days)	**Soil**		**Volatile traps**		
	Initial extract	**Bound residues**	**Ethylene glycol [trapped organic volatiles]**	**KOH [trapped $^{14}CO_2$]**	**Total recovery**
0					
Rep A	99.1	3.9	NA	NA	103.0
Rep B	96.8	3.9	NA	NA	100.7
Avg.	98.0	3.9	NA	NA	101.9
0.25					
Rep A	94.8	4.7	0.0	0.2	99.7
Rep B	92.9	4.5	0.0	0.1	97.5
Avg.	93.9	4.6	0.0	0.2	98.7
1					
Rep A	93.1	8.0	0.0	1.0	102.1
Rep B	93.1	7.6	0.0	0.8	101.5
Avg.	93.1	7.8	0.0	0.9	101.8
2					
Rep A	87.3	11.3	0.0	2.9	101.5
Rep B	86.0	12.3	0.0	3.1	101.4
Avg.	86.7	11.8	0.0	3.0	101.5
4					
Rep A	75.1	18.0	0.0	7.6	100.7
Rep B	76.7	17.0	0.0	4.8	98.5
Avg.	75.9	17.5	0.0	6.2	99.6
7					
Rep A	60.4	24.7	0.0	14.4	99.5
Rep B	65.4	22.5	0.0	12.2	100.1
Avg.	62.9	23.6	0.0	13.3	99.8
14					
Rep A	46.8	27.6	0.0	23.4	97.8
Rep B	42.8	31.7	0.1	24.7	99.3
Avg.	44.8	29.7	0.1	24.1	98.7
30					
Rep A	23.5	33.1	0.0	41.8	98.4
Rep B	20.1	38.4	0.0	33.8	92.3
Avg.	21.8	35.8	0.0	37.8	95.4
61					
Rep A[1]	10.7	37.1	0.0	22.7	70.5
Rep B	4.2	42.0	0.0	60.9	107.1
63					
Rep C	4.5	41.7	0.0	57.0	103.2
Avg. (B+C)	4.4	41.9	0.0	59.0	105.3
				Avg	100.2
				Std. dev.	3.0

Percent of applied radioactivity following application of [*Cis*–^{14}C][redacted]					
Sample time (days)	**Soil**		**Volatile traps**		
	Initial extract	**Bound residues**	**Ethylene glycol [trapped organic volatiles]**	**KOH [trapped $^{14}CO_2$]**	**Total recovery**
0					
Rep A	95.9	3.9	NA	NA	99.8
Rep B	95.3	4.1	NA	NA	99.4
Avg.	95.6	4.0	NA	NA	99.6
0.25					
Rep A	92.9	4.5	0.0	0.1	97.5
Rep B	95.1	4.6	0.0	0.1	99.8
Avg.	94.0	4.6	0.0	0.1	98.7
1					
Rep A	89.7	6.1	0.0	0.5	96.3
Rep B	93.8	5.9	0.0	0.5	100.2
Avg.	91.8	6.0	0.0	0.5	98.3
2					
Rep A	88.1	8.4	0.0	1.6	98.1
Rep B	88.4	9.0	0.0	1.6	99.0
Avg.	88.3	8.7	0.0	1.6	98.6
4					
Rep A	80.4	13.9	0.0	5.4	99.7
Rep B	84.8	11.3	0.0	3.2	99.3
Avg.	82.6	12.6	0.0	4.3	99.5
7					
Rep A	78.8	12.5	0.0	6.4	97.7
Rep B	77.2	14.3	0.0	7.6	99.1
Avg.	78.0	13.4	0.0	7.0	98.4
14					
Rep A	58.2	22.6	0.0	19.6	100.4
Rep B	70.7	20.6	0.0	13.8	105.1
Avg.	64.5	21.6	0.0	16.7	102.8
30					
Rep A	29.2	30.8	0.0	33.4	93.4
Rep B	28.2	30.9	0.0	31.8	90.9
Avg.	28.7	30.9	0.0	32.6	92.2
63					
Rep A	18.2	31.1	0.0	34.9	84.2
Rep B	8.2	34.4	0.0	34.7	77.3
Avg.	13.2	32.8	0.0	34.8	80.8
64 [2]					
Rep A	9.4	33.4	0.0	40.3	83.1
Rep B	8.3	32.1	0.0	40.2	80.6
Avg.	8.9	32.8	0.0	40.3	82.0
				Avg	95.0
				Std. dev.	7.7

NA – not analysed

[1] This sample was not used. Due to low mass balance, an additional sample (Rep C) was used.

[2] An additional sampling time was conducted on Day 64 for mass purposes only. Extraction solvent was added to soils while samples were connected to the traps to collect possible CO_2 evolving from soils during extraction.

Table 7.2.1-3: Profile of applied radioactivity in Mutchler Sandy Clay Loam Soil under aerobic conditions

Profile of applied radioactivity following application of [*Trans*-^{14}C][redacted], expressed as percentage of applied dose									
Sample time (days)	***Trans*-[redacted]**	**[redacted]**	**[redacted]**	**DT1**	**DT2**	**DT3**	**DT4**	**Others***	**Total Extract**
0									
Rep A	97.5	ND	ND	ND	ND	ND	ND	1.6	99.1
Rep B	96.2							0.6	96.8
Avg.	96.9							1.1	98.0
0.25									
Rep A	92.7	1.0	ND	ND	ND	ND	ND	1.0	94.8
Rep B	91.6	0.8						0.5	92.9
Avg.	92.2	0.9						0.8	93.9
1									
Rep A	64.1	22.2	0.4	0.8	ND	ND	ND	5.6	93.1
Rep B	71.2	21.4	0.0	0.0				0.5	93.1
Avg.	67.7	21.8	0.2	0.4				3.1	93.1
2									
Rep A	46.9	35.4	0.4	1.8	0.0	ND	1.1	1.7	87.3
Rep B	39.6	39.8	0.8	1.9	0.6		0.0	3.3	86.0
Avg.	43.3	37.6	0.6	1.9	0.3		0.6	2.5	86.7
4									
Rep A	21.9	43.0	1.8	0.5	0.0	0.0	7.2	0.8	75.1
Rep B	30.3	35.8	1.7	3.0	1.2	3.7	0.0	1.0	76.7
Avg.	26.1	39.4	1.8	1.8	0.6	1.9	3.6	0.9	75.9
7									
Rep A	8.5	33.5	3.7	5.8	2.2	3.9	2.9	ND	60.4
Rep B	10.4	39.4	2.4	8.9	0.0	2.9	1.4		65.4
Avg.	9.5	36.5	3.1	7.4	1.1	3.4	2.2		62.9
14									
Rep A	2.7	16.1	3.3	14.1	ND	0.0	5.4	5.2	46.8
Rep B	6.9	15.0	4.1	11.7		5.2	0.0	0.0	42.9
Avg.	4.8	15.6	3.7	12.9		2.6	2.7	2.6	44.8
30									
Rep A	1.1	4.4	2.9	9.6	1.4	0.0	4.2	0.0	23.5
Rep B	1.4	1.9	2.2	9.5	0.0	1.8	1.5	1.8	20.1
Avg.	1.3	3.2	2.6	9.6	0.7	0.9	2.9	0.9	21.8
61-63									
Rep B	0.7	ND	0.7	2.4	ND	ND	ND	0.5	4.2
Rep C	0.9		0.1	3.4				0.2	4.5
Avg.	0.8		0.4	2.9				0.4	4.4

Profile of applied radioactivity following application of [*Cis*-^{14}C][redacted], expressed as percentage of applied dose

Sample time (days)	*Trans-* [redacted]	[redacted]	[redacted]	DC1	DC2		Others*	Total Extract
0								
Rep A	91.6	ND	ND	ND	ND		4.3	95.9
Rep B	94.7						0.6	95.3
Avg.	93.2						2.5	95.6
0.25								
Rep A	91.9	ND	ND	ND	ND		1.0	92.9
Rep B	93.8						1.3	95.1
Avg.	92.9						1.2	94.0
1								
Rep A	83.9	5.2	ND	0.6	ND		0.0	89.7
Rep B	88.4	3.8		0.			1.1	93.8
Avg.	86.2	4.5		0.6			0.6	91.8
2								
Rep A	77.4	8.8	0.0	0.0	ND		1.9	88.1
Rep B	75.8	9.9	0.4	0.4			2.0	88.4
Avg.	76.6	9.4	0.2	0.2			2.0	88.3
4								
Rep A	58.9	13.8	0.9	3.3	0.4		3.1	80.4
Rep B	70.5	11.0	0.0	1.4	0.0		1.9	84.8
Avg.	64.7	12.4	0.5	2.4	0.2		2.5	82.6
7								
Rep A	59.5	11.8	0.8	2.9	2.2		1.6	78.8
Rep B	52.6	16.4	0.7	1.6	0.0		5.9	77.2
Avg.	56.1	14.1	0.8	2.3	1.1		3.8	78.0
14								
Rep A	30.7	12.0	1.9	8.6	0.9		4.1	58.2
Rep B	39.1	16.7	0.0	0.7	0.0		14.2	70.7
Avg.	34.9	14.4	1.0	4.7	0.5		9.2	64.5
30								
Rep A	12.0	2.7	2.9	7.1	2.1		2.4	29.2
Rep B	14.4	0.9	2.7	5.7	2.4		2.1	28.2
Avg.	13.2	1.8	2.8	6.4	2.3		2.3	28.7
63								
Rep A	11.4	ND	0.4	4.1	0.9		1.4	18.2
Rep B	2.7		0.4	4.3	0.5		0.3	8.2
Avg.	7.1		0.4	4.2	0.7		0.9	13.2

ND – not detected

*This column comprises several peaks present in the HPLC analysis which do not co-elute with reference standards.

Figure 7.2.1-1: Proposed Degradation Pathway of [redacted] in Soil under Aerobic Conditions

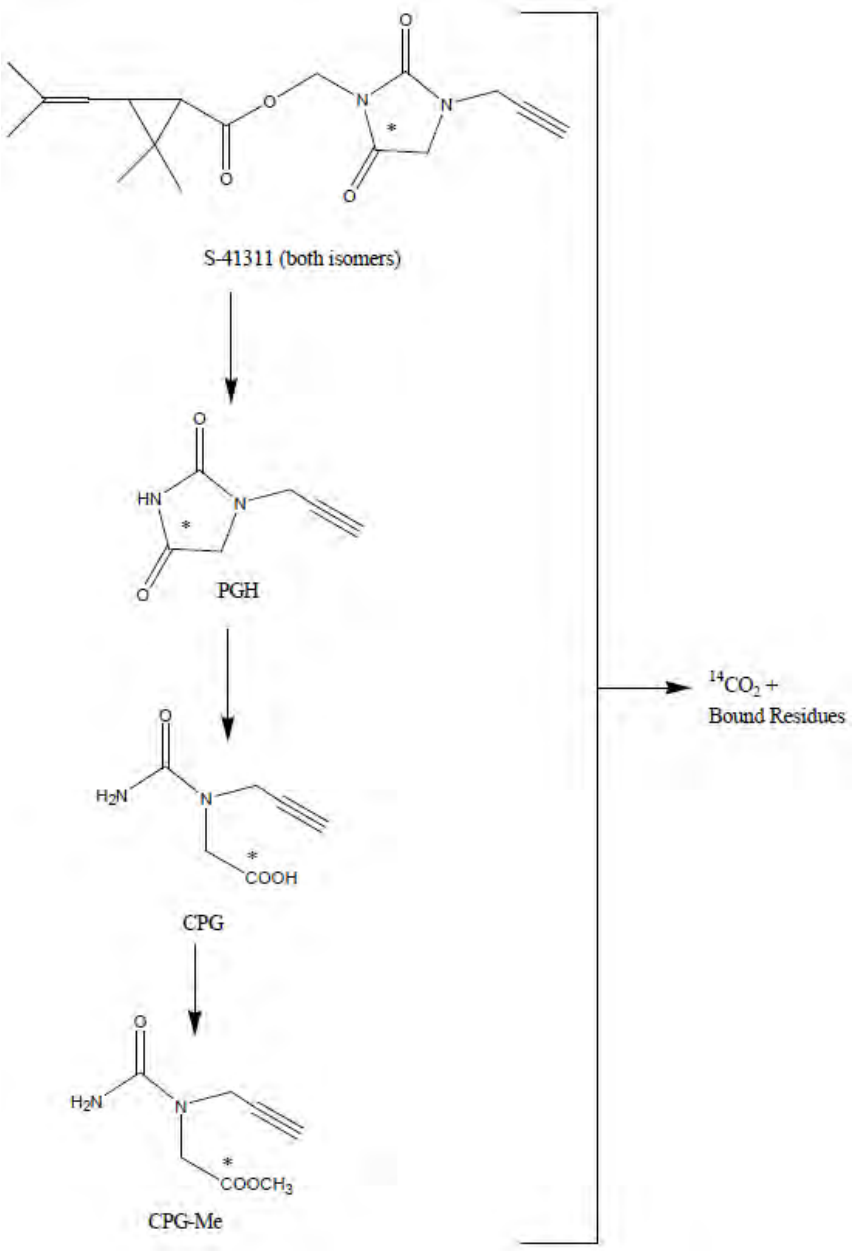


Figure 7.2.1-2: Report from CAKE for the recalculation of the rates of degradation according to FOCUS Kinetics Guidance

CAKE Kinetic Evaluation Report

Study: New Study

Study date: 2016å¹´10æœˆ31æ—¥
Report generated: 2016å¹´12æœˆ8æ—¥

SGM-0013_trans (SFO)

Model Setup:

Topology: Parent only
Optimiser: IRLS (IRLS Its. 10, IRLS Tol. 1E-05, Max. Its. 100, Tol. 1E-05)
SANN Max Iterations: 10000
Extra Solver Option: Use If Required

Initial Values of Sequence Parameters:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0.1	0 to (unbounded)	No

Fit step: Final

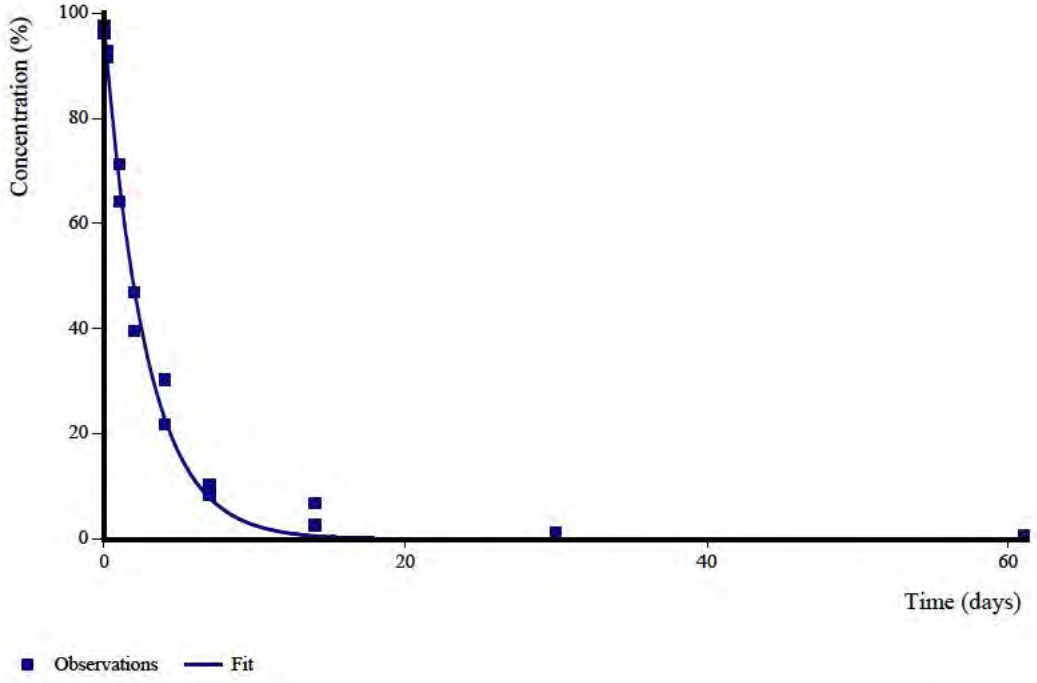
Used Extra Solver: No

Reference Table:

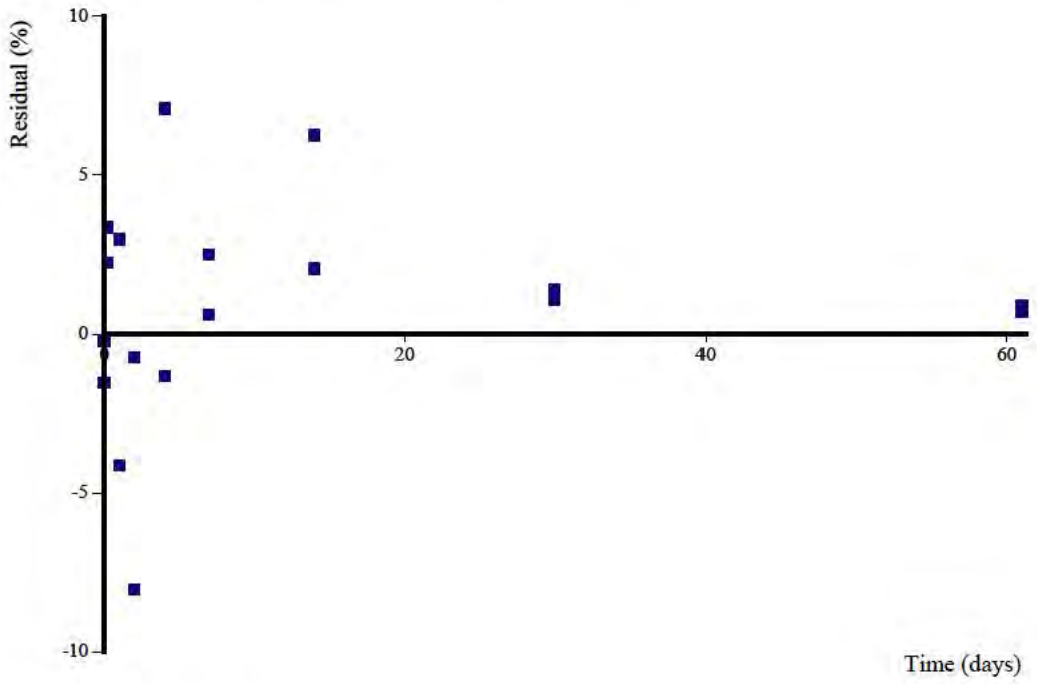
Compartment	Name
Parent	Imiprothrin

Graphical Summary:

Observations and Fitted Model:



Residuals:



Initial Values for this Step:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0.1	0 to (unbounded)	No

Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	97.74	1.907	N/A	94.41	101.1	93.69	101.8
k_Parent	0.3595	0.01917	1.29E-012	0.326	0.393	0.3189	0.4

χ^2

Parameter	Error %	Degrees of Freedom
All data	5.36	7
Parent	5.36	7

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	1.93	6.41

Additional Statistics:

Parameter	r^2 (Obs v Pred)	Efficiency
All data	0.9922	0.9911
Parent	0.9922	0.9911

Parameter Correlation:

	Parent_0	k_Parent
Parent_0	1	0.5402
k_Parent	0.5402	1

Observed v. Predicted:

Compartment Parent

Time (days)	Value (%)	Predicted Value	Residual
0	97.5	97.74	-0.236
0	96.2	97.74	-1.536
0.25	92.7	89.34	3.365
0.25	91.6	89.34	2.265
1	64.1	68.22	-4.122
1	71.2	68.22	2.978
2	46.9	47.62	-0.7198
2	39.6	47.62	-8.02
4	21.9	23.2	-1.302
4	30.3	23.2	7.098
7	8.5	7.891	0.6092
7	10.4	7.891	2.509
14	2.7	0.6371	2.063
14	6.9	0.6371	6.263
30	1.1	0.002022	1.098
30	1.4	0.002022	1.398
61	0.7	0	0.7
61	0.9	0	0.9

Sequence Creation Information:
Fit generated by CAKE version 3.2 (Release)
running on R version 3.0.0 (2013-04-03)

SGM-0013_cis (SFO)

Model Setup:

Topology: Parent only
Optimiser: IRLS (IRLS Its. 10, IRLS Tol. 1E-05, Max. Its. 100, Tol. 1E-05)
SANN Max Iterations: 10000
Extra Solver Option: Use If Required

Initial Values of Sequence Parameters:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0.1	0 to (unbounded)	No

Fit step: Final

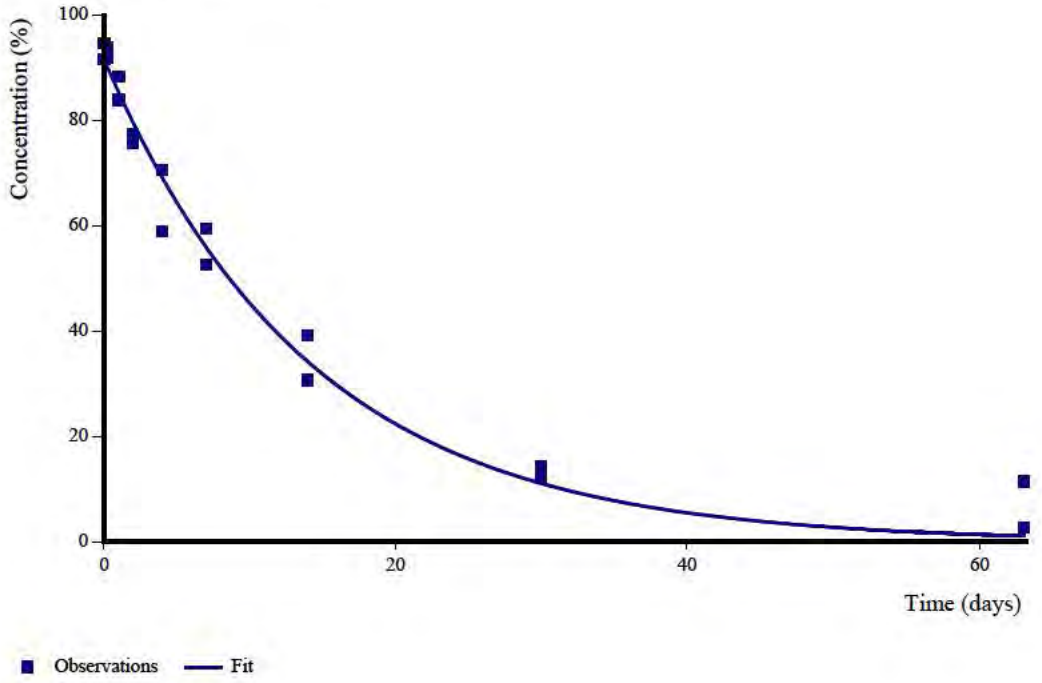
Used Extra Solver: No

Reference Table:

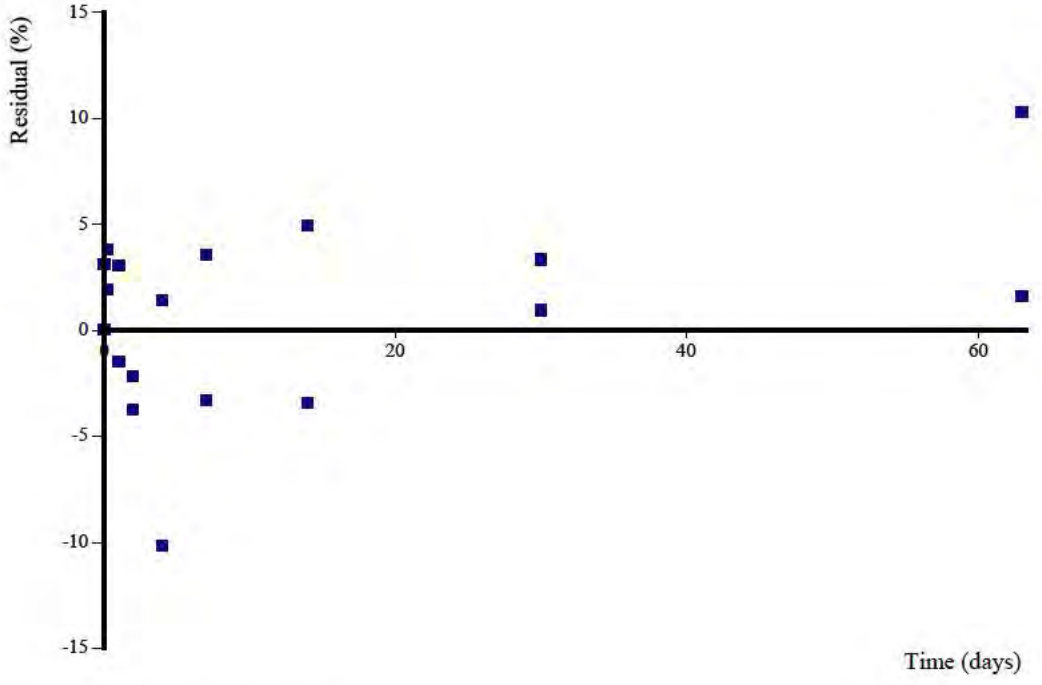
Compartment	Name
Parent	Imiprothrin

Graphical Summary:

Observations and Fitted Model:



Residuals:



Initial Values for this Step:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0.1	0 to (unbounded)	No

Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	91.58	1.8	N/A	88.43	94.72	87.76	95.4
k_Parent	0.0705	0.005008	9.85E-011	0.06176	0.07924	0.05988	0.081

χ^2

Parameter	Error %	Degrees of Freedom
All data	4.09	7
Parent	4.09	7

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	9.83	32.7

Additional Statistics:

Parameter	r^2 (Obs v Pred)	Efficiency
All data	0.9831	0.9805
Parent	0.9831	0.9805

Parameter Correlation:

	Parent_0	k_Parent
Parent_0	1	0.5381
k_Parent	0.5381	1

Observed v. Predicted:

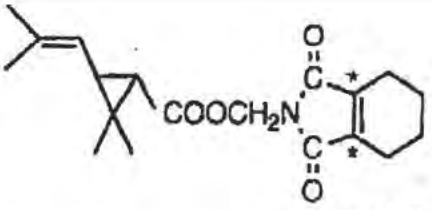
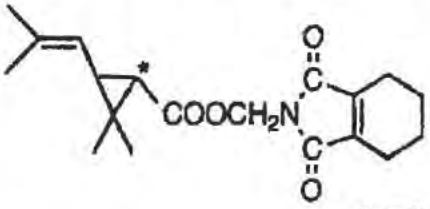
Compartment Parent

Time (days)	Value (%)	Predicted Value	Residual
0	91.6	91.58	0.02216
0	94.7	91.58	3.122
0.25	91.9	89.98	1.922
0.25	93.8	89.98	3.822
1	83.9	85.34	-1.444
1	88.4	85.34	3.056
2	77.4	79.54	-2.135
2	75.8	79.54	-3.735
4	58.9	69.08	-10.18
4	70.5	69.08	1.425
7	59.5	55.91	3.593
7	52.6	55.91	-3.307
14	30.7	34.13	-3.431
14	39.1	34.13	4.969
30	12	11.05	0.9522
30	14.4	11.05	3.352
63	11.4	1.079	10.32
63	2.7	1.079	1.621

Sequence Creation Information:
Fit generated by CAKE version 3.2 (Release)
running on R version 3.0.0 (2013-04-03)

Report Information:
Report generated by CAKE version 3.2 (Release)
CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK, sponsored by Syngenta
Running on .NET version 4.0.30319.34209

Section A7.2.1/03 Annex Point IIA 7.7	Aerobic degradation in soil, initial study	
	4. REFERENCE	**Official use only**
Reference	[redacted]. (1992), Aerobic Soil Metabolism Study of ^{14}C-(1RS)-trans-Tetramethrin, Environmental Health Science Laboratory, Sumitomo Chemical Co. Ltd., [redacted], 1992.	
Data protection	Yes	
4.1.1 Data owner	Sumitomo Chemical Company Limited	
4.1.2 Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I	
	GUIDELINES AND QUALITY ASSURANCE	
Guideline study	Yes Guideline 162-1	X
GLP	Yes	
Deviations	No	
	MATERIALS AND METHODS	
Test material	The study is performed using ^{14}C-(1RS)-trans-Tetramethrin, which are the major isomers in tetramethrin (a 4:1:4:1 mixture of the [1R, trans], [1R, cis], [1S, trans], and [1S, cis] stereoisomers) and hence will give a valid indication of the aerobic degradation of tetramethrin in soil.	
4.1.3 Lot/Batch number	[redacted]	
4.1.4 Specification	[redacted]	
4.1.5 Purity	[redacted]	
4.1.6 Further relevant properties	[redacted]	

Section A7.2.1/03 **Annex Point IIA 7.7**	**Aerobic degradation in soil, initial study**	
4.1.7 Position of radiolabel	 * indicates radiolabeled position [THP-^{14}C]-(1*RS*)-*trans*-Tetramethrin  * indicates radiolabeled position [Cyclopropyl-^{14}C]-(1*RS*)-*trans*-Tetramethrin	
4.1.8 Method of analysis	Radiocarbon: The radiocarbon in aqueous solution was determined as follows: 1 ml of the aqueous solution was mixed well with 10 ml of Emusifier Scintillator 299™ in a glass vial and kept at 4 °C for 8 h prior to liquid scintillator counting (LSC). Soils to be analysed for radiocarbon were dried in a vacuum desiccator and were powdered. Duplicate 300 mg samples were combusted prior to LSC. Each sample was counted for 5 min. Parent and degradates: 2D TLC was used to quantitate parent and degradates as follows: silica gel 60F_{254} thin layer plates were used for preparative and analytical purposes. The radioactive spots were determined by autoradiography. The unlabelled tetramethrin, acid-NPY, THPI, MTI, THPA and d-t-CRA were detected by ultraviolet light at 254 nm and 1-OH-HPA and COOH-CA were detected by exposure to iodine vapour or bromocresol green ethanol solution (slightly basic). The autoradiograms were prepared by exposing the TLC plate to SB-5 X-ray films at 8 °C for a week. The radiocarbon was quantified by scraping the radioactive gel regions into glass vials and counting then by LSC.	
Degradation products	Degradation products tested: Yes	
4.1.9 Method of analysis for degradation products	See section 3.1.6	
Reference substance	Yes Non-radiolabelled standards of (1RS)-trans-tetramethrin and the potential degradation products were synthesised.	
4.1.10 Method of analysis for reference substance	See section 3.1.6	
Soil types	See table A7.2.1-1	
Testing procedure		
4.1.11 Test system	Described in section 3.5.2	

Section A7.2.1/03 Annex Point IIA 7.7		Aerobic degradation in soil, initial study	
4.1.12	Test solution and Test conditions	**Soil preparation and fortification:** A total of 44 beakers, each containing the same amount of soil were prepared and treated as follows. Hanford sandy loam soil equivalent to 10.0 g in a dry weight basis was taken into a 30 ml glass beaker. The soil sample was moistened with distilled water to 75 % of 0.33 bar moisture. The soil was incubated at 25 ± 1 °C in the dark for 26 days to stimulate the microbial activity. A fortification solution was prepared by dissolving a purified ^{14}C-tetramethrin in acetonitrile. Duplicate 10 µl aliquots of the solution were radioassayed by LSC to determine the concentration of ^{14}C-tetramethrin. After pre-incubation, the aliquot of the fortification solution was dropwisely added to each 10 g soil sample using a microsyringe. Duplicate 10 µl aliquots of the solution were radioassayed by LSC before and after soil treatment and the concentration of ^{14}C-tetramethrin in the fortified solution was determined. The treated soil samples were mixed well by spatula to give a concentration of 1.010 ppm (THP-^{14}C) or 0.995 pm (cyclopropyl-^{14}C) relative to dry soil. **Aerobic incubation of treated soil:** The 11 treated soil samples were placed in a 3 l glass jar, covered with aluminium foil and kept at 25 ± 1 °C in the dark. Experiments were conducted in duplicate for each label using 2 glass jars. Each jar was continuously purged with the CO_2 free air at a rate of approximately 50 ml/min. The effluent air was passed in sequence through one gas washing bottle containing 350 ml of ethylene glycol and one containing 450 ml of 0.5 M NaOH solution to trap the volatile compounds. **Sampling schedule:** Each one soil sample was taken from 2 glass jars immediately after dosing and 1, 3, 7, 14 days and 1, 2, 3, 4, 6, 9 and 12 months and trapping media were analysed approximately every 2 weeks. **Extraction of soil samples:** Each soil sample was extracted 3 times with methanol (20 ml, agitation for 10 min, centrifugation at 5000 rpm for 10 min). The supernatants were combined, radioassayed (duplicate 1 ml aliquots) and concentrated at 40 °C by a rotary evaporator. Portions of the soil (approximately 300 mg) that remained were analysed in duplicate for radiocarbon by combustion followed by LSC. The extracted soil samples were further extracted 3 times with methanol: 0.1N HCl (1/1, v/v) (20 ml, agitation for 10 min, centrifugation at 5000 rpm for 10 min). The supernatants were combined, radioassayed (duplicate 1 ml aliquots) and concentrated at 40 °C by a rotary evaporator. Portions of the soil (approximately 300 mg) that remained were analysed in duplicate for radiocarbon by combustion followed by LSC.	X
Test performance			
4.1.13	Preliminary test	n/a	
4.1.14	Dose rates	The concentration of tetramethrin in field soils is calculated to be 0.18 ppm assuming that tetramethrin at the normal field application rate (0.04 g a.i./m^2) is uniformly mixed with soil at 0-6 inches in depth and that the bulk density of soil is 1.50. Since this dose rate would not be sufficient to make clear the degradation rate and degradation pattern of tetramethrin in soil, the dose rate was raised to 1.0 ppm (approximately 5.56 times the normal field application rate) to permit detection of ^{14}C residues down to 1 % of the application dose.	X
4.1.15	Limit of detection	The background level was 50 dpm and more than 150 dpm was quantitatively determined. The minimal quantifiable radiocarbon by LSC is defined as 100 dpm (equivalent to 3.02 x 10^{-4} µg for [THP-^{14}C] -(1RS)-trans-Tetramethrin and 3.41 x 10^{-4} for [Cyclopropyl-^{14}C] -(1RS)-trans-Tetramethrin. For the soil extracts of approximately 60 ml (total combined volume) from an average amount of soil of 10 g, the minimal quantifiable level of radiocarbon was 6000 dpm. This is equivalent to ca. 0.2 % of the applied radiocarbon or ca. 0.002	

Section A7.2.1/03 Annex Point IIA 7.7	Aerobic degradation in soil, initial study	
	ppm for each label.	
	RESULTS	
Preliminary test	n/a	
Extractable residues	Methanol extractable residues decreased from 101.1 – 102.8 % (THP label) and 101.2 – 101.9 % (cyclopropyl label) of the applied radiocarbon at day 0 to 0.7 – 0.9 % and 2.1 – 2.8 %, respectively at 365 days. HCl/methanol extractable residues increased from 8.2 – 15.3 % (THP label) and 6.7 – 6.9 % (cyclopropyl label) of the applied radiocarbon at day 3 to 50.9 – 52.9 % and 35.6 – 36.7 %, respectively at day 58 (maximum) and then decreased to 16.3 – 19.6 % and 19.4 – 22.1 % respectively at 365 days. Volatile residues increased to 67.7 – 74.1 % (THP label) and 68.4 – 78.5 % (cyclopropyl label) of the applied radiocarbon at 365 days. The majority of the activity was observed in the NaOH solution trap and was confirmed to be residues of $^{14}CO_2$.	
Distribution of degradation products in the soil extracts	Residues of the parent compound decreased from 97.0 – 97.6 % (THP label) and 96.9 – 98.0 % (cyclopropyl label) of the applied radiocarbon at day 0 to 0.1 % at 365 days. Of the 7 degradates identified (acid-NPY, THPI, MTI, THPA, 1-OH-HPA, d-t-CRA and COOH-CA) levels of only 2 exceeded 0.01 ppm at the proposed field rate. Levels of 1-OH-HPA reached 0.01 ppm at day 7, increased to a maximum of 0.044 ppm (42.0 – 44.3 % of the applied ^{14}C) at day 58 , then steadily decreased thereafter, reaching 0.015 ppm (13.2 – 16.5 % of the applied ^{14}C) at day 365. Levels of COOH-CA reached 0.01 ppm between days 14 and 30, increased to a maximum of 0.022 ppm between days 58 and 122 (23.7 – 24.3 % of the applied ^{14}C), then steadily decreased, reaching 0.015 ppm (14.2 – 17.8 % of the applied ^{14}C) at day 365.	
Mass balance	Total mass balance accountability through the study was 92.5 – 113.0 % of the applied radiocarbon.	
Expected results under actual field conditions	The study was conducted at a dose rate approximately 5.5 times greater than the proposed field use rate of 0.04 g a.i./m^2. It can be seen that the residues of the parent compound would fall below 0.01 ppm between 30-58 days after application. Residues of 1-OH-HPA would reach levels of > 0.01 ppm 7 days after applications, increase to maximum levels (0.044 ppm) at day 58 and then decrease to 0.015 ppm at day 365. Residues of COOH-CA would reach levels of >0.01 ppm 30 days after applications, increase to maximum levels (0.022 ppm) at day 58 – 122 and then decrease to 0.015 ppm at day 365. Other identified degradation products (acid NPY, THPI, MTI, THPA and d-t-CRA) would be expected to be less than 0.01 ppm at the normal field application rate.	
Degradation rate	[THP-^{14}C] -(1RS)-trans-Tetramethrin: Half lives of 32.7 days (day 0 – 365) and 12.5 days (day 0 – 90) were calculated for degradation.	X

Section A7.2.1/03 Annex Point IIA 7.7	Aerobic degradation in soil, initial study	

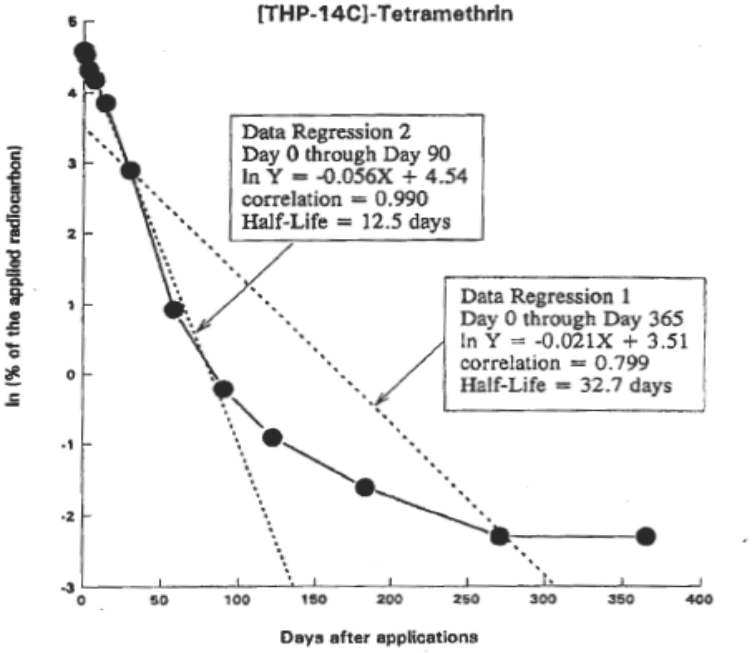


Figure 35 The decline curve of [THP-^{14}C]-(1*RS*)-*trans*-tetramethrin in Hanford soil under aerobic conditions.

[Cyclopropyl-^{14}C] -(1RS)-trans-Tetramethrin:

Half lives of 33.3 days (day 0 – 365) and 14.2 days (day 0 – 90) were calculated for degradation.

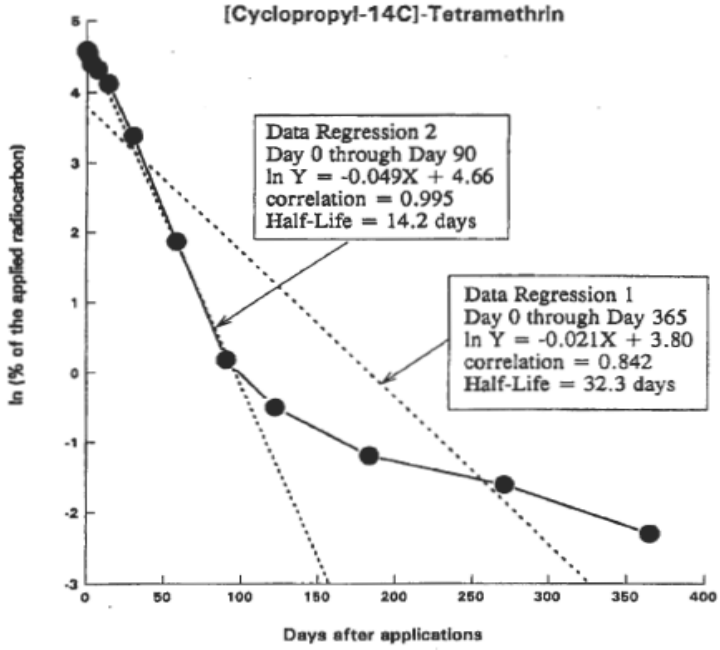
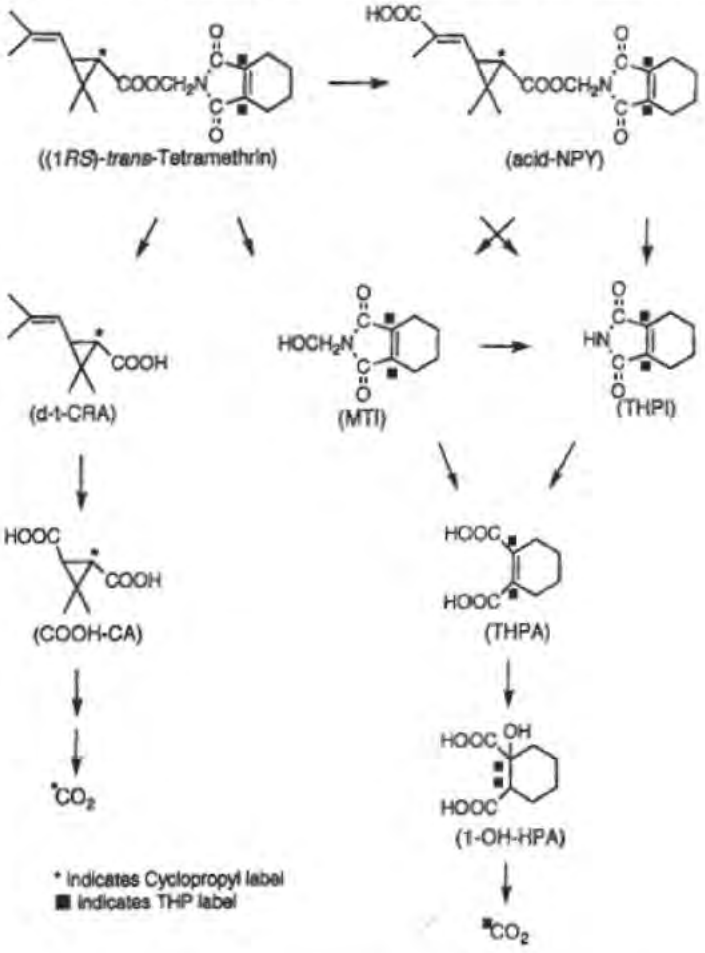


Figure 36 The decline curve of [Cyclopropyl-^{14}C]-(1*RS*)-*trans*-tetramethrin in Hanford soil under aerobic conditions.

Section A7.2.1/03 Annex Point IIA 7.7	**Aerobic degradation in soil, initial study**	
Degradation product(s)	Levels of 1-OH-HPA reached 0.01 ppm at day 7, increased to a maximum of 0.044 ppm (42.0 – 44.3 % of the applied ^{14}C) at day 58 , then steadily decreased thereafter, reaching 0.015 ppm (13.2 – 16.5 % of the applied ^{14}C) at day 365. Levels of COOH-CA reached 0.01 ppm between days 14 and 30, increased to a maximum of 0.022 ppm between days 58 and 122 (23.7 – 24.3 % of the applied ^{14}C), then steadily decreased, reaching 0.015 ppm (14.2 – 17.8 % of the applied ^{14}C) at day 365. The proposed degradation pathway for tetramethrin is outlined below:  Figure 37 Proposed degradation pathways of tetramethrin in soil under aerobic conditions.	
	APPLICANT'S SUMMARY AND CONCLUSION	
Materials and methods	EPA Guideline 162-1 A Californian sandy loam soil was moistened to 75 % of the field capacity (0.33 bar), fortified with each labelled test material at a nominal concentration of 1.0 ppm relative to dry soil and then incubated in glass vessels maintained at 25 ± 1 °C in darkness for up to 365 days. The humid CO_2 free air was continuously passed in sequence through one ethylene glycol and one NaOH solution trap for collection of organic volatiles and CO_2. Duplicate soil samples for each label were analysed on days 0, 1, 3, 7, 14, 30, 58, 90, 122, 183, 271 and 365. The soil samples were extracted 3 times with methanol and further extracted 3 times with methanol/ 0.1N HCl (1/1, v/v). The methanol and methanol/ 0.1N HCl soil extracts were separately analysed by 2D TLC. Parent compound and its degradation products were quantitated by TLC methods and identified by TLC and/or HPLC co-chromatography with the authentic standards. The non-extractable ^{14}C soil residues were analysed for radiocarbon by combustion followed by LSC.	

Section A7.2.1/03 **Annex Point IIA 7.7**	**Aerobic degradation in soil, initial study**	
Results and discussion	Methanol extractable residues decreased from 101.1 – 102.8 % (THP label) and 101.2 – 101.9 % (cyclopropyl label) of the applied radiocarbon at day 0 to 0.7 – 0.9 % and 2.1 – 2.8 %, respectively at 365 days. HCl/methanol extractable residues increased from 8.2 – 15.3 % (THP label) and 6.7 – 6.9 % (cyclopropyl label) of the applied radiocarbon at day 3 to 50.9 – 52.9 % and 35.6 – 36.7 %, respectively at day 58 (maximum) and then decreased to 16.3 – 19.6 % and 19.4 – 22.1 % respectively at 365 days. Residues of the parent compound decreased from 97.0 – 97.6 % (THP label) and 96.9 – 98.0 % (cyclopropyl label) of the applied radiocarbon at day 0 to 0.1 % at 365 days. Of the 7 degradates identified (acid-NPY, THPI, MTI, THPA, 1-OH-HPA, d-t-CRA and COOH-CA) levels of only 2 exceeded 0.01 ppm at the proposed field rate Half lives of 12.5 days (day 0 through day 90) and 32.7 days (day 0 through day 365) were calculated for degradation of [THP-^{14}C] -(1RS)-trans-Tetramethrin. Half lives of 14.2 days (day 0 through day 90) and 33.3 days (day 0 through day 365) were calculated for degradation of [Cyclopropyl-^{14}C] -(1RS)-trans-Tetramethrin.	
4.1.16 Degradation products (% of a.s.)	Levels of 1-OH-HPA reached 0.01 ppm at day 7, increased to a maximum of 0.044 ppm (42.0 – 44.3 % of the applied ^{14}C) at day 58 , then steadily decreased thereafter, reaching 0.015 ppm (13.2 – 16.5 % of the applied ^{14}C) at day 365. Levels of COOH-CA reached 0.01 ppm between days 14 and 30, increased to a maximum of 0.022 ppm between days 58 and 122 (23.7 – 24.3 % of the applied ^{14}C), then steadily decreased, reaching 0.015 ppm (14.2 – 17.8 % of the applied ^{14}C) at day 365.	
Conclusion	Tetramethrin undergoes degradation in a sandy loam soil under aerobic dark conditions with the initial half-lives of 12.5 – 14.2 days. Based upon the identified products, the proposed degradation pathways of tetramethrin are shown in section 4.7. The major routes of degradation were ester bond cleavage and oxidation at the acid and alcohol moieties. The resultant degradates were ultimately degraded to $^{14}CO_2$ and/or bound to soil.	
4.1.17 Reliability	1	
4.1.18 Deficiencies	No	
	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
	EVALUATION BY RAPPORTEUR MEMBER STATE	
Date	03/02/2017	
Materials and Methods	The Applicant's version is considered to be acceptable noting the following: **2.1** OECD 307 recommends at least three soil types are used for determination of rates of transformation, and that these soils should be representative of the environmental conditions where use or release will occur. This study used one soil, a sandy loam pH 6.5 from Madera County, California. No history of the field site from where the test soil was collected was provided. No information was provided on soil collection, and storage. A 26 day acclimatisation period was used. **3.5.2** Ten grams of soil were used per test system. This is lower than the OECD 307 recommended minimum of 50 g. The experiment was carried out at a temperature 25 °C rather than 20 °C, was and soil moisture was adjusted to 75 % field capacity (assuming field capacity is -0.33 bar). No information is provided on soil moisture throughout the experiment, therefore it can be assumed that moisture content was not measured during the experiment. As no untreated controls were included, microbial biomass could not be calculated pre and post experiment. However, microbial viability was tested prior and post experiment using	

Section A7.2.1/03 Annex Point IIA 7.7	Aerobic degradation in soil, initial study	

plate count methods, and although levels of aerobic bacteria,and actinomycetes and total fungi had decreased over the test duration (i.e., five fold reduction in bacteria and actinomycetes and a thirty fold reduction in fungi), the soil remained viable.

No sterile control was provided, therefore no distinction can be made between abiotic and biotic degradation of the test substance.

Results and discussion

4.6 This study has been supplied in support of imiprothrin, as the imiprothrin soil study only followed the fate of the ring structure of the molecule due to the positioning of the radioactive label. In this study, (1RS)-*trans*-tetramethrin degrades to form the same chrysanthemic acid moiety as formed during imiprothrin degradation, and here, it can be followed as it has been labelled.

The degradation rate of metabolites is theoretically independent of the degradation rate of the parent, therefore, read across from (1RS)-trans-tetramethrin metabolite degradation rate to imiprothrin metabolite rate can be justified. The following data were used for kinetic analysis:

Concentration data in % AR

	(1RS)-trans-tetramethrin		[redacted]	
Time (days)	Replicate A	Replicate B	Replicate A	Replicate B
0	101.4	102.1	0	0
1	94.2	93.2	0	0
3	80.5	81.4	2.9	2.3
7	81.1	68.6	3.1	6.8
14	76.1	46.8	5.5	9.9
30	37.1	21.2	15.3	16.9
58	9	3.7	23.7	24.3
90	1.6	0.8	22.8	19.1
122	0.6	0.6	21.8	26.1
183	0.3	0.3	20.2	19.9
271	0.2	0.2	15.7	12.9
365	0.1	0.2	17.8	14.2

The initial metabolite, d-t/c-CRA (i.e. chrysanthemic acid) was not formed in sufficient quantity to warrant kinetic analysis (maximum formation 1.3 % AR I experiment A, day 1).

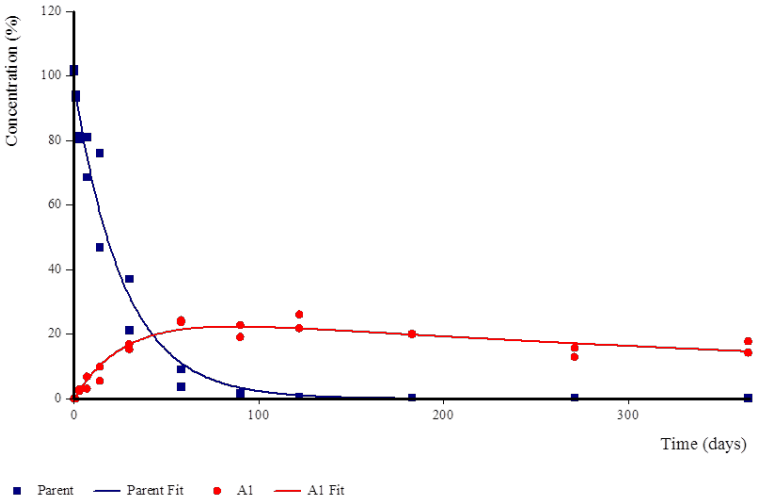
As this stud y has been submitted to several CAs in support of different active substances, the following kinetic analysis has been harmonised between CAs.

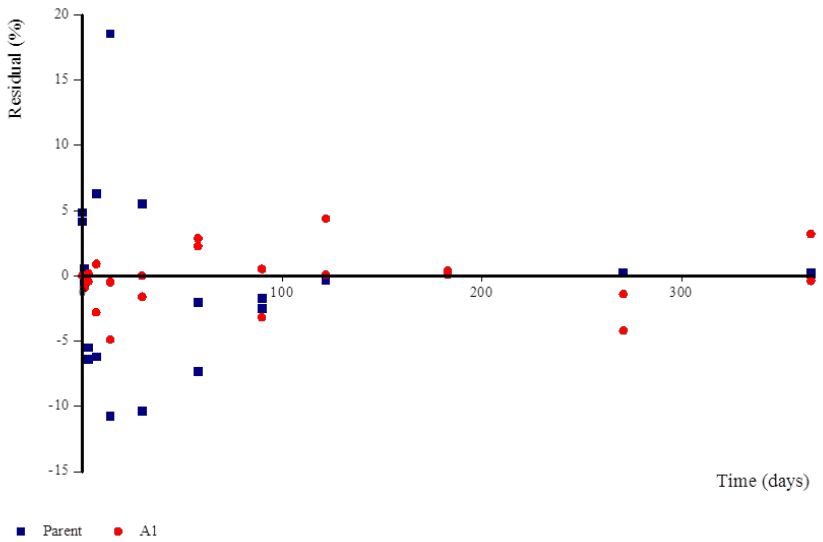
Analysis was performed using CAKE 3.1 using the sequential fitting of parent with the major metabolite PG (IRLS fit) for full details see Figure A7.2.1-1.

Data Set: (1RS)- trans-Tetramethrin and [redacted]

Observations and Fitted Model:

Section A7.2.1/03 Annex Point IIA 7.7	Aerobic degradation in soil, initial study	





Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	97.28	2.652	N/A	92.82	101.7	91.93	102.6
k_Parent	0.03747	0.003044	8.04E-016	0.03235	0.04259	0.03133	0.044
f_Parent_to_A1	0.2654	0.01781	N/A	0.2355	0.2954	0.2295	0.301
k_A1	0.001686	3.57E-004	1.32E-005	0.001085	0.002287	9.65E-004	0.002

Section A7.2.1/03 **Annex Point IIA 7.7**	**Aerobic degradation in soil, initial study**	

χ^2

Parameter	Error %	Degrees of Freedom
All data	8 16	19
Parent	6 35	10
A1	10.2	9

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	18.5	61.5
A1	411	1.37E+03

The DT_{50} of the metabolite, [redacted], was found to be 411 d.

Bound residues were formed at a maximum of 12.8 % AR on day 271.

Conclusion	This study can be used to support [redacted] half –life of 411 d.
Reliability	Accepted as being [redacted] as only one soil type used.
Acceptability	Acceptable
Remarks	
	COMMENTS FROM ...
Date	*Give date of comments submitted*
Materials and Methods	*Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant's summary and conclusion.* *Discuss if deviating from view of rapporteur member state*
Results and discussion	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	

Table A7.2.1-1: Classification and physico-chemical properties of soils used as adsorbents

	Soil
Soil identification	Hanford
Classification	Sandy loam
Location	Hanford, Madera County, California
Sand [%]	54
Silt [%]	36
Clay [%]	10
Organic carbon [%]	0.46
Organic matter [%]	0.8
pH (H_2O)	6.5
Cation exchange capacity (MEQ/100 g)	4.7
Bulk density (g/cm^3)	1.51
1/s bar field capacity (%)	9.49

Figure A7.2.1-1 CAKE Kinetic Evaluation Report of Tetramethrin + [redacted]

Data set: (SFO)

Study date: 03 March 2017
Report generated: 03 March 2017

Model Setup:

Topology: Parent, A1 with link Parent–A1
Optimiser: IRLS (IRLS Its. 10, IRLS Tol. 1E-05, Max. Its. 100, Tol. 1E-05)
SANN Max Iterations: 10000
Extra Solver Option: Do Not Use

Initial Values of Sequence Parameters:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0.1	0 to (unbounded)	No
f_Parent_to_A1	0.5	0 to 1	No
A1_0	0	0 to (unbounded)	Yes
k_A1	0.1	0 to (unbounded)	No

Fit step: Final

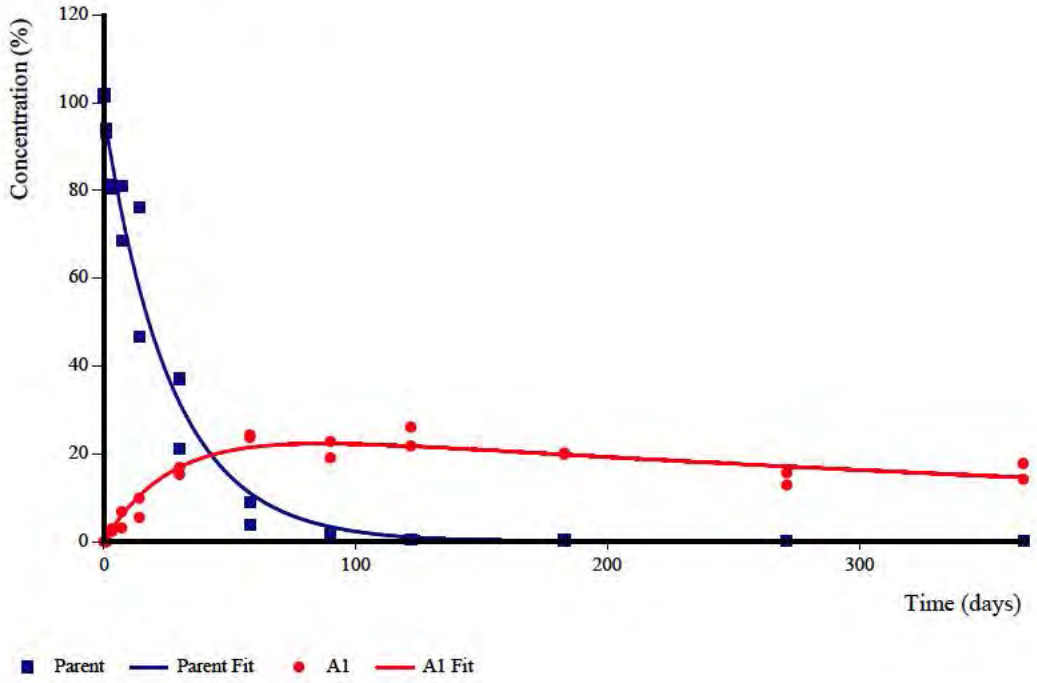
Used Extra Solver: No

Reference Table:

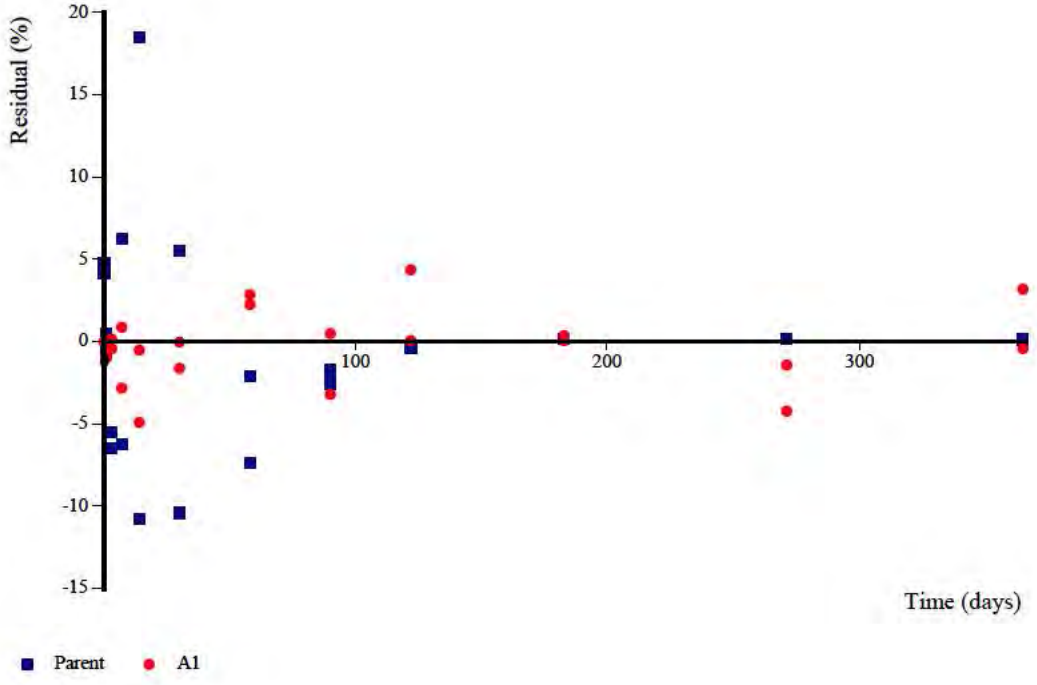
Compartment	Name
Parent	Parent
A1	A1

Graphical Summary:

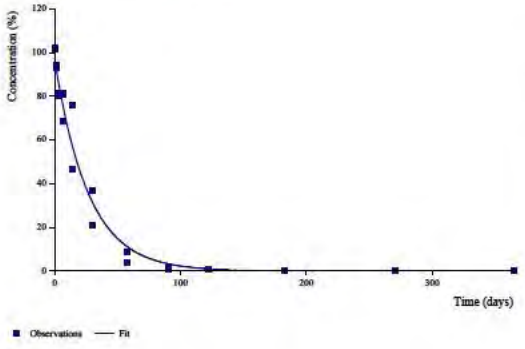
Observations and Fitted Model:

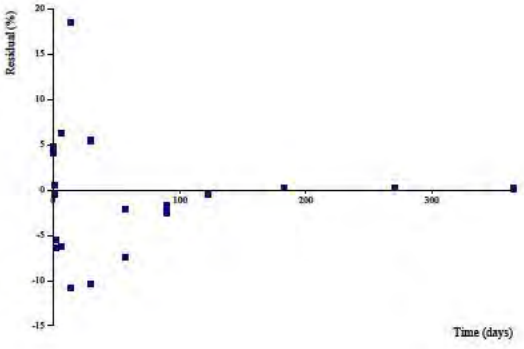


Residuals:

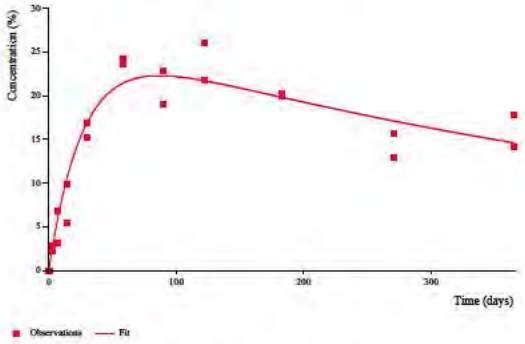


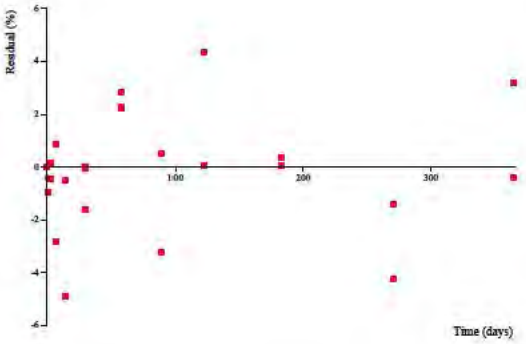
Compartment Parent:





Compartment A1:





Initial Values for this Step:

Parameter	Initial Value	Bounds	Fixed
Parent_0	98.32	0 to (unbounded)	No
k_Parent	0.03996	0 to (unbounded)	No
f_Parent_to_A1	0.2567	0 to 1	No
A1_0	0	0 to (unbounded)	Yes
k_A1	0.001569	0 to (unbounded)	No

Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	97.28	2.652	N/A	92.82	101.7	91.93	102.6
k_Parent	0.03747	0.003044	8.04E-016	0.03235	0.04259	0.03133	0.044
f_Parent_to_A1	0.2654	0.01781	N/A	0.2355	0.2954	0.2295	0.301
k_A1	0.001686	3.57E-004	1.32E-005	0.001085	0.002287	9.65E-004	0.002

χ^2

Parameter	Error %	Degrees of Freedom
All data	8.16	19
Parent	6.35	10
A1	10.2	9

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	18.5	61.5
A1	411	1.37E+03

Additional Statistics:

Parameter	r^2 (Obs v Pred)	Efficiency
All data	0.9802	0.9799
Parent	0.9782	0.9779
A1	0.9442	0.9406

Parameter Correlation:

	Parent_0	k_Parent	f_Parent_to_A1	k_A1
Parent_0	1	0.4836	-0.6053	-0.191
k_Parent	0.4836	1	-0.608	-0.3949
f_Parent_to_A1	-0.6053	-0.608	1	0.7367
k_A1	-0.191	-0.3949	0.7367	1

Observed v. Predicted:

Compartment Parent

Time (days)	Value (%)	Predicted Value	Residual
0	101.4	97.28	4.116
0	102.1	97.28	4.816
1	94.2	93.71	0.4945
1	93.2	93.71	-0.5055
3	80.5	86.94	-6.44
3	81.4	86.94	-5.54
7	81.1	74.84	6.262
7	68.6	74.84	-6.238
14	76.1	57.57	18.53
14	46.8	57.57	-10.77
30	37.1	31.61	5.49
30	21.2	31.61	-10.41
58	9	11.07	-2.07
58	3.7	11.07	-7.37
90	1.6	3.337	-1.737
90	0.8	3.337	-2.537
122	0.6	1.006	-0.406
122	0.6	1.006	-0.406
183	0.3	0.1023	0.1977
183	0.3	0.1023	0.1977
271	0.2	0.003782	0.1962
271	0.2	0.003782	0.1962
365	0.1	0.0001124	0.09989
365	0.2	0.0001124	0.1999

Compartment A1

Time (days)	Value (%)	Predicted Value	Residual
0	0	0	0
0	0	0	0
1	0	0.9489	-0.9489
1	0	0.9489	-0.9489
3	2.9	2.739	0.1615
3	2.3	2.739	-0.4385
7	3.1	5.921	-2.821
7	6.8	5.921	0.8788
14	5.5	10.41	-4.907
14	9.9	10.41	-0.5068
30	15.3	16.92	-1.62
30	16.9	16.92	-0.01956
58	23.7	21.44	2.257
58	24.3	21.44	2.857
90	22.8	22.3	0.4954
90	19.1	22.3	-3.205
122	21.8	21.73	0.06753
122	26.1	21.73	4.368

183	20.2	19.83	0.3674
183	19.9	19.83	0.0674
271	15.7	17.12	-1.422
271	12.9	17.12	-4.222
365	17.8	14.61	3.186
365	14.2	14.61	-0.4136

Sequence Creation Information:
Fit generated by CAKE version 3.2 (Release)
running on R version 3.0.0 (2013-04-03)

Report Information:
Report generated by CAKE version 3.2 (Release)
CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK, sponsored by Syngenta
Running on .NET version 4.0.30319.36373

Section A7.2.1/04 Aerobic degradation in soil

Annex Point IIA 7.7

Official use only

1 REFERENCE

1.1 Reference [redacted] (1992). Aerobic Soil Metabolism Study of [Acid-^{14}C]-d-trans-Allethrin. ABC Laboratories, Inc., Columbia, U.S.A. Final Report number 38484. 18 May 1992

Experimental test dates: 29 June 1990 – 31 January 1992

1.2 Data protection Yes

1.2.1 Data owner Sumitomo Chemical Company Ltd., Japan

1.2.2

1.2.3 Criteria for data protection Data submitted to the MS after 13 May 2000 on existing [a.s. / b.p.] for the purpose of its entry into Annex I.

2 GUIDELINES AND QUALITY ASSURANCE

2.1 Guideline study Yes

EPA Pesticide Assessment Guidelines, Subdivision N, Section 162-1

2.2 GLP Yes

2.3 Deviations Yes

GLP: Soil characterisation data generated by A & L Mid West Laboratories, Inc. for the sandy loam soil used in the study cannot be assured to have been conducted to GLP.

3 MATERIALS AND METHODS

3.1 Test material [redacted]

3.1.1 Lot/Batch number [redacted]

3.1.2 Specification [redacted]

3.1.3 Purity Radiochemical purity 98.5% (following repurification)

3.1.4 Further relevant properties Solubility: 5.0±0.11 mg/L at 25°C

Stated specific activity: [redacted]

3.2 Reference substances Reference standards: [redacted]

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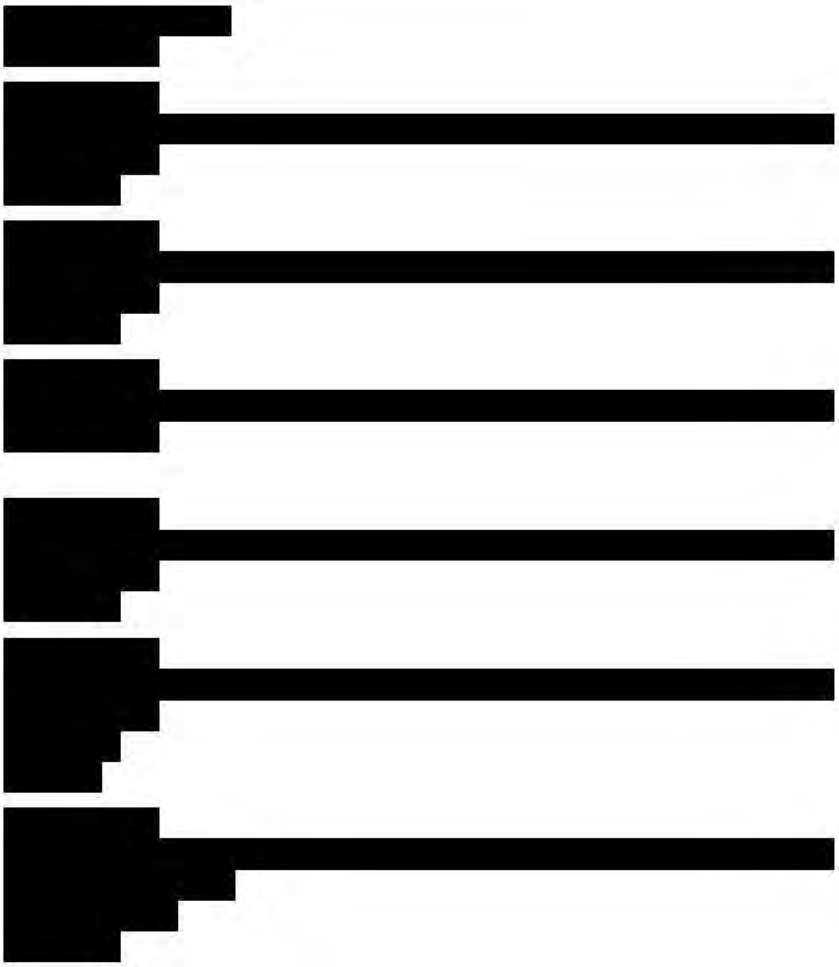

Structures are shown in Table 7.2.1-1.

3.3 Testing procedure

3.3.1 Test system

A California sandy loam soil was used. The soil was passed through a 2 mm sieve.

Soil Properties:

Textural analysis:	sand	54%
silt	36%	
	clay	10%
Classification (USDA):		sandy loam
Organic matter:		0.8%
pH (H_2O):		6.5
CEC* (meq/100g):		4.7
Bulk density (g/cm^3):		1.51
Field capacity (0.33 bar):		9.49%

* cation exchange capacity

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Individual samples of soil (equivalent to 10 g dry weight) were weighed into tared silanised pyrex culture tubes, and moistened to 75% of field capacity and incubated at 25 ± 1°C in the dark for 22 days.

The concentration of allethrin in soils was calculated to be 0.058 ppm, assuming that d-allethrin at the highest proposed field rate (0.058 lb a.i./acre) was uniformly mixed with soil at 0-3 inches in depth. However, as this dose rate would not have been sufficient for monitoring of the degradation rate and pathway, the dose rate was raised to 1.5 ppm (*ca* 26 x the field application rate) to allow detection of ^{14}C residues down to 1% of the applied dose.

Following pre-incubation, an aliquot (70 µL) of acetonitrile solution of the [Acid-^{14}C]-d-trans-Allethrin stock solution (0.180 mg/mL) was added dropwise to each soil and thoroughly mixed. The treated soil samples were placed in a 3 L glass jar and incubated at 25 ± 1°C in the dark. Air was passed over the soil samples into four traps - ethylene glycol, 1N H_2SO_4 and two 1N KOH in sequence to trap volatile ^{14}C. The soil moisture was adjusted to 70-75% by the addition of distilled water once a month.

3.3.2 Sampling — Soil samples were taken for analysis at 0, 1, 2, 3, 4, 7, 14, 30, 61, 92, 122 and 183 days.

Traps were sampled at each sampling interval (0, 1, 2, 3, 4, 7, 14, 30, 61, 92, 122 and 183 days).

3.3.3 Extraction and analysis — Day 0-183 samples were extracted 3x with 100% methanol (10 mL). After each addition of extraction solvent, the tubes were shaken for at least 10 minutes on a mechanical shaker. The samples were then centrifuged at 2-3000 rpm for several minutes and the extract decanted into a graduated cylinder. The extracts were combined and adjusted to a final volume of 30 mL with methanol. Triplicate aliquots were taken for LSC analysis.

Due to the polar nature of the degradation products, the methanol extraction decreased in efficiency over the course of the study. In order to release additional amounts of bound residues, the Day 0 to 3-month soil samples were also extracted using the above-mentioned procedure 3x with methanol : water (1:1 v/v, 10 mL). Due to the decreasing efficiency of the methanol : water (1:1 v/v) extraction over time, the 4 and 6-month samples were not submitted to this extraction.

Again, due to the polar nature of the degradation products, the methanol : water (1:1 v/v) extraction decreased in efficiency over the course of the study. The Day 14 to 6-month samples were therefore also extracted, using the same procedure, 3x with 0.2N HCl in methanol : water (1:1 v/v, 5 mL).

Following the above described extractions, bound residues remained in several samples at >10% of the initial measured dose (IMD). The 3 and 6-month samples were selected for further extraction, as detailed below:

Following drying of the soil, an aliquot (*ca* 3 or 5 g) was added to a 250 mL round-bottomed flask. A volume (25 to 35 mL) of 0.2N HCl in methanol : water (1:1 v/v) was added to the flask and the soil refluxed for 3 hours with constant mechanical stirring. Upon completion of the

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reflux, the soil and extract were transferred to an 8 oz. plastic bottle.
A methanol rinse of the round-bottomed flask was then performed and the rinse solution added to the plastic bottle. Following centrifugation for several minutes, the extract was decanted into a graduated cylinder, the volume was measured, and triplicate aliquots were taken for LSC analysis.

Bound residue fractionation was also performed on aliquots of the 3 and 6-month soil samples. Following drying, an aliquot of soil (2.5 or 4 g) was added to a silanised culture tube. The soil was extracted 3x with 1N NaOH (5 mL). After each addition of 1N NaOH, the tubes were mechanically shaken for 1 hour and centrifuged at 2-3000 rpm. The final volumes were adjusted to 15 or 30 mL, the extracts were transferred to
2 oz. plastic bottles and aliquots were taken for LSC assay.
The pH of the extract was then adjusted to ≤ 2 by addition of concentrated HCl. After a period of at least 1 hour, each bottle was centrifuged at 2-3000 rpm, the extract was transferred to a separate plastic bottle and aliquots removed for quantification. The precipitate was re-dissolved in 1N NaOH and aliquots taken for LSC assay.
The extracted soil was then dried and combusted prior to LSC.

In an effort to identify all residues greater than 0.01 ppm, the first 1N KOH trapping solutions from the day 7 to 4-month timepoints were subjected to characterisation to confirm the residues as $^{14}CO_2$. Enough barium chloride was added to each sample (60 mL) to prepare a 1N solution and shaken mechanically for 4 hours. Following centrifugation, the samples were filtered under vacuum. Triplicate aliquots were removed for LSC before addition of the $BaCl_2$ and after filtration.

The levels of radioactivity in the other traps were negligible (<0.01 ppm) and so no confirmatory analysis was required.

Unextractable soil residues were dried and then powdered. Aliquots (200-300 mg) of each soil were combusted in a Model 306 Packard Sample Oxidiser or a Harvey OX-500 Biological Sample Oxidiser prior to LSC. Recovery of radioactivity was ≥95% in Harvey Carbon 14 Cocktail.

Aliquots from each soil extract were analysed by reverse-phase high- performance liquid chromatography (HPLC) using the isocratic system described below for identification of allethrin and its soil metabolites. Aliquots of non-radiolabelled allethrin were analysed to aid in characterisation of the samples.

Pump:	Varian 5000
UV detector:	Varian 100 Variable Wavelength UV Detector
Mobile phase:	80:20 Methanol:Water
Wavelength:	230 nm
Column:	Alltech Econosil C18, 10 µm, 250 mm x 4.6 mm
Flow rate:	1.0 mL/minute

Fractions collection was performed using a Gilson Model 202 Fraction collector with pre-programmed sub-fraction time windows. The fractions were then quantified by LSC.

Selected soil extracts were characterised by gradient HPLC to aid in the

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identification of the parent compound and suspected degradation products:

Pump:	Shimadzu LC-6A
UV detector:	Shimadzu SPD-6A Variable Wavelength UV Detector
Mobile phase:	A = H_2O with 0.1% H_3PO_4
B = CH_3CN with 0.1% H_3PO_4	
Wavelength:	220 nm
Column:	Sumipax ODS A-212 (5 µm, 150 mm x 6 mm)
Flow rate:	1.0 mL/minute

The following gradient system was employed:

Time(min)	% A	% B
0	85	15
60	0	100
62	0	100
65	85	15

Fractions collection was performed using a Gilson Model 202 Fraction

collector with pre-programmed sub-fraction time windows. The fractions were then quantified by LSC.

Two dimensional thin layer chromatography (TLC) was conducted using pre-coated silica gel thin layer chromatography plates (Merck). Solvent systems used were hexane : acetone (5:2 v/v) in the first dimension and hexane : toluene : acetic acid (3:15:2 v/v/v) in the second dimension.
Autoradiograms were prepared by placing the plates under Kodak x-ray film, type XAR2. After an appropriate time, the film was developed, zones corresponding to radioactivity were scraped from the plate, desorbed in methanol and analysed by LSC.

Retention times (Rt) of each reference standard are shown in Table 7.2.1-1.

4 RESULTS

4.1 Dissipation time The first-order rate constant and half-life (DT-50) for d-trans-Allethrin were calculated according to the following equation:

$Ct = e^{-kt} Co$

where;
Ct = concentration at time "t" Co = concentration at time zero k
= rate constant
t = time

The log transformation of the first-order rate law gives:

$$ln\ Ct = ln\ Co^{-kt}$$

a) setting the initial concentration = 1, at $t_{1/2}$; $C_t = 1 - 0.5 = 0.5$
b) rearranging the above equation gives $\ln C_o/C_t = kt$.
c) therefore, $t_{1/2} = 1/k \ln 1/0.5 = 1/k \ln 2 = 0.693/k$

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d) k is the slope (1st derivative) of the line generated from the linear regression analysis of the plot of ln percent [Alc-^{14}C]-d-trans-Allethrin of the initial dose measured in soil extracts versus days.

Replicate	First order	
System I	$T_{1/2}$ (days)	16.9
	r^2	0.9975
System II	$T_{1/2}$ (days)	22.0
	r^2	0.9832

4.2 Concentration – time data

Distribution of radioactivity in sample matrices is shown in Table 7.2.1-2.

The total ^{14}C recovery was 86.9 to 111% of the applied ^{14}C. The amount of extractable ^{14}C gradually decreased to levels of 38.6 to 45.0% at 6 months (183 days), with similar levels of CO_2 formed, amounting to 37.1 to 41.4%. The amount of soil bound residues also increased with time and peaked at 2 months (day 122) (18.7 to 19.7%), thereafter the levels decreased slightly to around 5%.
Organic matter fractionation of selected samples (3 months and 6 months) confirmed 36 to 49% as humin, 26 to 34% as humic acid and 23 to 35% as fulvic acid.

HPLC analysis of the soil extracts showed rapid degradation of d-trans-Allethrin, with levels accounting for only 1.5 to 2.4% of the applied ^{14}C at 6 months.

4.3 Specification of the transformation products

Table 7.2.1-3 shows the amounts of d-trans-Allethrin and its degradation products in California sandy loam soil.

The only degradation products observed eluted very early under the isocratic HPLC system conditions. The two degradates were labelled as Unknowns I and II. Unknown I appeared at day 1 (1 to 1.13% of the IMD), increased to > 30% at 2 to 3 months, and then decreased to 24.8 to 28.1% at 6 months. It was noted that the methanol extracts contained measurable amounts of the parent compound and both metabolites. However, the supplemental soil extracts contained primarily the degradation products, and the later extracts contained only one metabolite (Unknown I). The unknowns and parent compound were considered to be sufficiently separated so that quantitative data was established through the isocratic system.

Selected soil extracts (3-month samples) were chosen as representative samples to be further analysed by gradient reverse phase HPLC. This analysis confirmed the identity of the parent compound and offered evidence that the degradation products were d-t-CRA and COOH-CA.

To further confirm these identities, the selected soil extracts were also submitted to two-dimensional normal phase TLC analysis. This analysis confirmed the results of the gradient HPLC analysis.

A proposed degradation pathway for d-trans-Allethrin is shown in Figure 7.2.1-1.

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5 APPLICANT'S SUMMARY AND CONCLUSION

5.1 Materials and methods

An aerobic soil metabolism study was conducted with [Acid-^{14}C]-d- trans-Allethrin, in accordance with the EPA Pesticide Assessment Guidelines, Subdivision N, Section 162-1. A Californian sandy loam soil was moistened to 75% of 1/3 bar moisture and fortified with [Acid-^{14}C]- d-trans-Allethrin at a concentration of 1.56 µg/g on a dry weight basis.
The test system was incubated in silanised pyrex culture tubes maintained at 25 ± 1°C in the dark for up to 6 months.

Humidified air was continuously passed in sequence through one ethylene glycol, one 1N H_2SO_4 and two 1N KOH traps for collection of organic volatiles and carbon dioxide.

Each soil sample was analysed immediately after application and at 1, 2, 3, 4, 7, 14, 30, 61, 92, 122 and 183 days post-treatment.

The soil samples were extracted as follows:

Day 0-183 samples were extracted three times with 100% methanol (3 x 10 mL).

Day 0-92 samples were also extracted with methanol : water (1:1 v/v, 3 x 10 mL)

Day 14-183 samples were also extracted with 0.2N HCl in methanol : water (1:1 v/v, 3 x 5 mL)

Day 92 and 183 samples were then refluxed for 4 hours with 0.2N HCl in methanol : water (1:1 v/v, 25-35 mL), prior to organic matter fractionation via IN NaOH (3 x 5-10 mL).

The soil extracts were analysed by HPLC analysis. d-trans-Allethrin and its degradates were identified by HPLC and 2D-TLC co-chromatography with reference standards. The radioactivity of unextractable soil residues was quantified as $^{14}CO_2$ by combustion followed by radioassay.

5.2 Results and discussion

The total ^{14}C recovery was 86.9 to 111% of the applied ^{14}C. The amount of extractable ^{14}C gradually decreased to levels of 38.6 to 45.0% at 6 months (183 days), with similar levels of CO_2 formed, amounting to 37.1 to 41.4%. The amount of soil bound residues also increased with time and peaked at 2 months (day 122) (18.7 to 19.7%), thereafter the levels decreased slightly to around 5%.
Organic matter fractionation of selected samples (3 months and 6 months) confirmed 36 to 49% as humin, 26 to 34% as humic acid and 23 to 35% as fulvic acid.

HPLC analysis of the soil extracts showed rapid degradation of d-trans-Allethrin, with levels accounting for only 1.5 to 2.4% of the applied ^{14}C at 6 months.

The only degradation products observed eluted very early under the isocratic HPLC system conditions. The two degradates were labelled as Unknowns I and II. Unknown I appeared at day 1 (1 to 1.13% of the IMD), increased to > 30% at 2 to 3 months, and then decreased to 24.8 to 28.1% at 6 months. It was noted that the methanol extracts contained measurable amounts of the parent compound and both metabolites.
However, the supplemental soil extracts contained primarily the

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degradation products, and the later extracts contained only one metabolite (Unknown I). The unknowns and parent compound were considered to be sufficiently separated so that quantitative data was established through the isocratic system.

Selected soil extracts (3-month samples) were chosen as representative samples to be further analysed by gradient reverse phase HPLC. This analysis confirmed the identity of the parent compound and offered evidence that the degradation products were d-t-CRA and COOH-CA.

To further confirm these identities, the selected soil extracts were also submitted to two-dimensional normal phase TLC analysis. This analysis confirmed the results of the gradient HPLC analysis.

Half-life of d-trans-Allethrin, assuming first-order kinetics, was estimated to be 16.9 to 22.0 days in the Californian sandy loam soil.

This data generated on [Alc-^{14}C]-d-trans-allethrin (bioallethrin) is being used to read-across for d-allethrin.

Bioallethrin comprises [1R,*trans*: 1S]-isomer:[1R, *trans*;1R]-isomer:at a 1:1 ratio.

d-Allethrin comprises [1R,*trans*: 1S]-isomer:[1R, *trans*;1R]-isomer: [1R, *cis*; 1S]-isomer and [1R *cis*: 1R]-isomer at a ratio of 4:4:1:1.

The data for d-trans-allethrin was fitted using Modelmaker (version 4) with SFO, FOMC and DFOP in accordance with guidance provided by FOCUS (Sanco/10058/2005, version 2.0, June 2006). The goodness of fit was assessed with the χ^2 test and by visual observation.

The three different models were used as a comparison to see which model gave the best fit to the data. The data used was exactly the same as that used in the original report, except the mean was taken of the three 0-h replicates. Once the models had been run, the optimised parameters from Modelmaker (version 4.0) were used to assess the goodness of fit and to determine the DT_{50} values.

The following results were obtained: SFO Kinetics
Soil system I - DT_{50} = 19.4 d, DT_{90} = 64.3 d, χ^2 = 3.4 (pass)
Soil system II - DT_{50} = 31.6 d, DT_{90} = 105.1 d, χ^2 = 9.8 (pass)

FOMC Kinetics
Soil system I - DT_{50} = 20.1 d, DT_{90} = 60.4 d, χ^2 = 3.3 (pass) Soil system II - DT_{50} = 31.7 d, DT_{90} = 105.4 d, χ^2 = 10.2 (pass)

DFOP Kinetics
Soil system I - DT_{50} = 19.5 d, DT_{90} = 64.9 d, χ^2 = 3.7 (pass) Soil system II - DT_{50} = 31.7 d, DT_{90} = 105.3 d, χ^2 = 10.6 (pass)

The half-life in the original report from ABC labs was calculated using linear regression and gave a half-life value in the region of 17 to 22 days.
In order to be consistent with the revised kinetic analysis for the [^{14}C-alc]-d-trans-allethrin study, the decision was taken to re-do the kinetic analysis, in line with recent developments in the EU *(Sanco/10058/2005, version 2.0, June 2006).*

All three models pass the χ^2 test, and visual observation of the data

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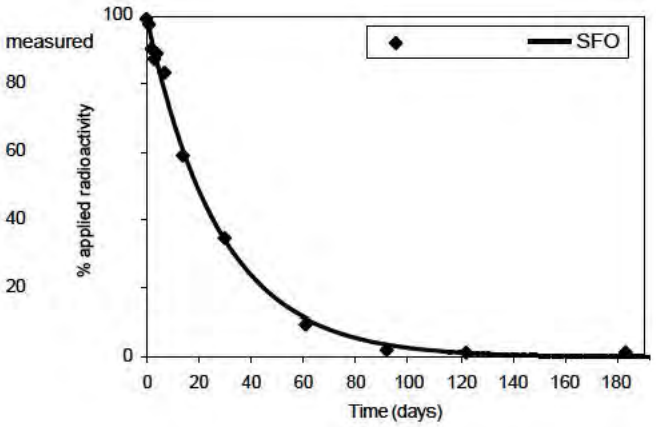
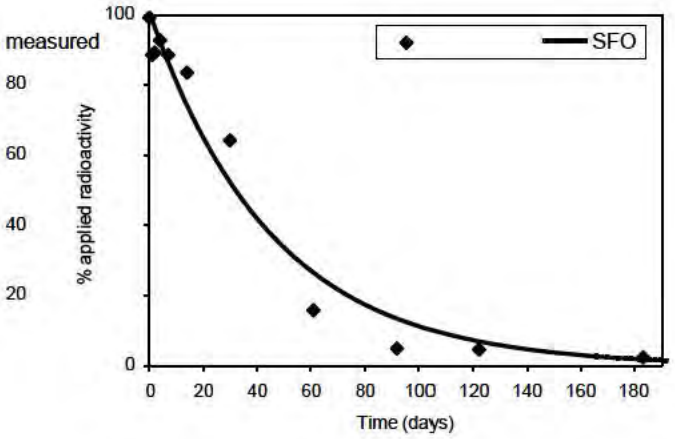
indicates little difference between the models. However, the FOMC model in this case was returning negative values for the optimised parameters, and since there is little difference between the three models the data from the SFO model was considered acceptable.

Updated DT_{50} for d-trans-Allethrin for a Californian Sandy Loam soil was 19 to 32 days, resulting in a mean DT_{50} value 25.5 days.

Soil system I graph- SFO kinetics



Soil system I graph- SFO kinetics



5.3 Conclusion

[Acid-^{14}C]-d-trans-Allethrin undergoes degradation on sandy loam soil to $^{14}CO_2$, bound residues and two identified degradation products (d-t- CRA and COOH-CRA). The half-life of the parent compound was estimated by assuming first order kinetics to be 25.5 days.

Mineralisation of the parent compound to $^{14}CO_2$ was demonstrated, with residues increasing up to 38.7% of the IMD at 6 months. The formation of bound residues also appears to be one mechanism for the degradation

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of the parent compound under aerobic soil conditions. Bound residues increased to > 40% of the IMD at 6 months.

This data generated on [redacted] is being used to 'read-across' for d-allethrin.

5.3.1	Reliability	1
5.3.2	Deficiencies	The moisture content of the soil (75% of 1/3 bar) was slightly higher than recommended in the European Guidelines.

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Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted

EVALUATION BY RAPPORTEUR MEMBER STATE

Date 03/02/2017

Materials and Methods

The Applicant's version is considered to be acceptable noting the following:

2.1 OECD 307 recommends at least three soil types are used for determination of rates of transformation, and that these soils should be representative of the environmental conditions where use or release will occur. This study used one soil, a sandy loam pH 6.5 from Madera County, California. To the soil supplier's knowledge, the ground had been fallow for a number of years with no pesticides having been applied. The soil was air dried, ground, and then sieved.

No information was provided on soil collection, and storage. A 22 day acclimatisation period was used.

3.3.1 Ten grams of soil were used per test system. This is lower than OECD 307's recommended minimum of 50 g. The experiment was carried out at a temperature 25 °C rather than 20 °C, was and soil moisture was adjusted to 75% field capacity and adjusted to 70 – 75 % of the field capacity on a monthly basis throughout the study.

Dose rate of trans-Allethrin was 1.5 µg/g. This is approximately x 15 higher than expected field concentrations when used as a pesticide. It is possible that this high dose rate inhibited soil microbial activity. As no untreated controls were included, microbial biomass could not be calculated pre and post experiment. Microbial viability was tested at study initiation, and at the 3 month and 6 month sample points using plate count methods, but these results were not presented so it is not possible to ascertain the viability of the test system throughout the study.

No sterile control was provided, therefore no distinction can be made between abiotic and biotic degradation of the test substance.

Results and discussion

5.2 This study has been supplied in support of d-trans-allethrin, as the imiprothrin soil study only followed the fate of the ring structure of the molecule due to the positioning of the radioactive label. In this study, d-*trans*-allethrin degrades to form the same chrysanthemic acid moiety as formed during imiprothrin degradation, and here, it can be followed as it has been labelled.

The degradation rate of metabolites is theoretically independent of the degradation rate of the parent therefore, read across can be justified. The following data were used for kinetic analysis:

Concentration data in % AR

	d-trans-allethrin		[redacted]	
Time (days)	Replicate A	Replicate B	Replicate A	Replicate B
0	100	100	0	0
1	97.6	88.6	0.931	1.13
2	90.4	89.3	2.09	2.26

3	87.4	103	3.15	3.55
4	89	92.8	4.19	4.17
7	83.4	88.6	10.6	7.82
14	59	83.6	13.7	12.7
30	34.7	64.3	26.5	15.6
61	9.55	15.8	30	32
92	2.06	4.97	21.4	35.1
122	1.41	4.66	22.1	24.1
183	1.48	2.42	24.8	28.1

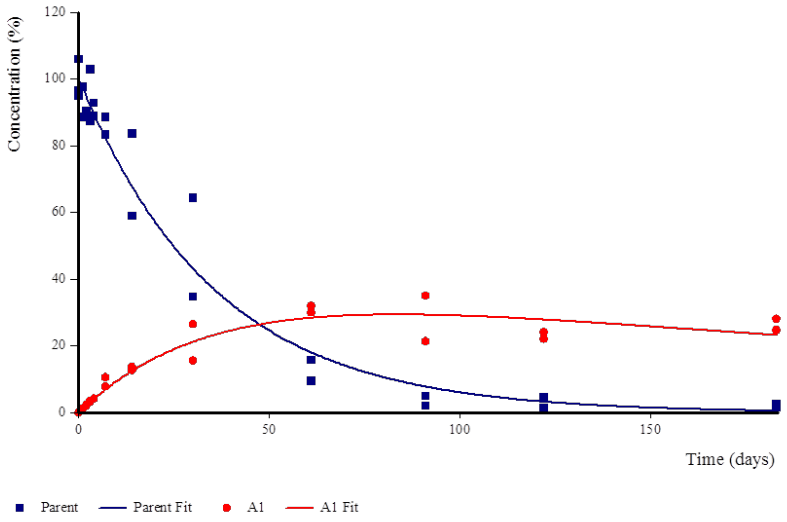
The initial metabolite, d-c/t-CRA (i.e. chrysanthemic acid) was not formed in sufficient quantity to warrant kinetic analysis (maximum formation, 5.1 % IMD, day 92).

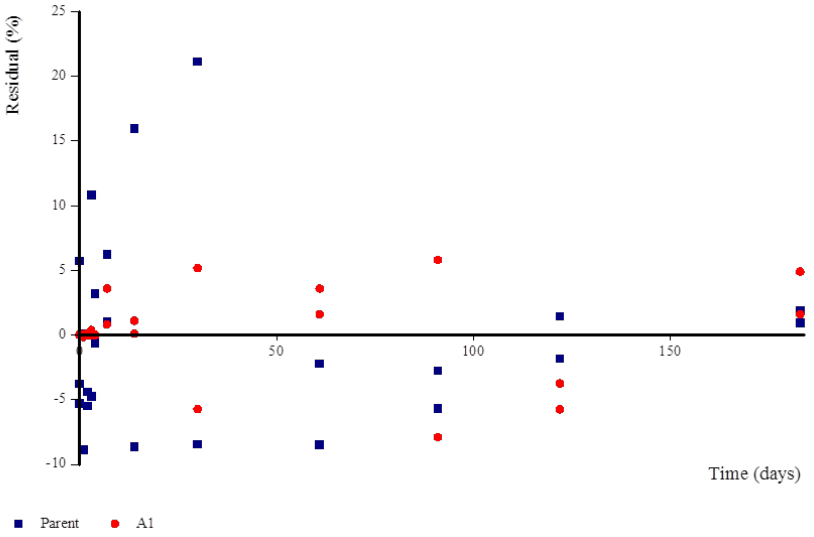
As this stud y has been submitted to several CAs in support of different active substances, the following kinetic analysis has been harmonised between CAs.

Analysis was performed using CAKE 3.1 using the sequential fitting of parent with the major metabolite PG (IRLS fit).

Data Set: d-trans-Allethrin and [redacted]

Observations and Fitted Model:





Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	100.3	2.552	N/A	96.02	104.6	95.16	105 5
k_Parent	0.0281	0.002775	2.92E-013	0.02344	0.03277	0.02251	0.034
f_Parent_to_A1	0.3976	0.04278	N/A	0.3257	0.4695	0.3113	0.484
k_A1	0.003653	0.001139	0.001268	0.001738	0.005568	0.001356	0.006

χ^2

Parameter	Error %	Degrees of Freedom
All data	7 19	19
Parent	5 28	10
A1	11.1	9

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	24.7	81.9
A1	190	630

The re-assessed half-life of d-trans-allethrin is 24.7 days and the DT_{50} of the metabolite t- COOH-CA was found to be 190 d.

Levels of the major degradate [redacted] were > 10 % from day 14 onwards and reached a peak on day 61 (31.0 % average value). Levels of primary metabolite chrysanthemic acid were > 5 % only at one timepoint, day 122 so could not be included in the kinetic analysis.

CO_2 synthesis reached an average peak of 39.3 % IMD, and bound residues were a maximum of 41.7 % AR. Analysis of selected samples showed bound residues to be found mainly in the humin fraction.

Conclusion	This study can be used to support a [redacted] half –life of 190 d.
Reliability	[redacted]
Acceptability	Acceptable
Remarks	

	COMMENTS FROM ...
Date	*Give date of comments submitted*
Materials and Methods	*Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant's summary and conclusion.* *Discuss if deviating from view of rapporteur member state*
Results and discussion	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	

Table A7.2.1/02-1: Structures of non-radiolabelled reference compounds and their chromatographic properties

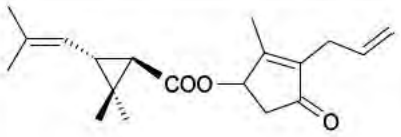
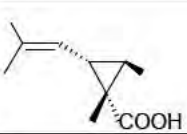
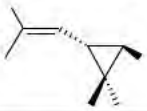
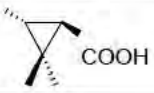
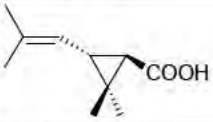
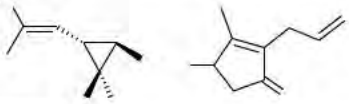
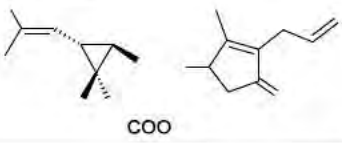
Designation	Structure	Retention time HPLC Rt (min)
Allethrin (d-t-ALL)		52.27
COOH-CRA		15.84
CH_2OH-CRA		17.02
COOH-CA		4.62
d-t-CRA		32.96
wt-acid-t-ALL		34.17/35.15
wt-alc-t-ALL		39.55/40.14

Table 7.2.1/02-2: Distribution of applied radioactivity in California Sandy Loam Soil under Aerobic Conditions

Percent of applied radioactivity following application of [Acid-^{14}C]-d-trans-Allethrin									
Sample time (days)	**Replicate**	**Soil Extract I**	**Soil Extract II**	**Soil Extract III**	**Soil Extract IV**	**Soil Extract V**	**Unextracted Residues**	**Cumulative Volatiles**	**TOTAL**
0	1+2	97.8	1.73	-	-	-	0.072	-	100
1	1	96.4	3.09	-	-	-	0.432	0.166	100
	2	87.8	2.88	-	-	-	0.504	0.244	91.4
2	1	89.2	4.03	-	-	-	0.719	0.255	94.2
	2	89.9	4.39	-	-	-	1.08	0.313	95.7
3	1	86.3	5.97	-	-	-	1 94	0.415	94.7
	2	102	5.68	-	-	-	2 23	0.470	111
4	1	89.2	6.98	-	-	-	1 58	0.566	98.3
	2	92.1	7.05	-	-	-	1.80	0.654	102
7	1	87.8	10.4	-	-	-	3 24	2.01	103
	2	87.1	10.4	-	-	-	4 96	1.61	104
14	1	65.1	13.5	2.37	-	-	7.05	7.00	95.0
	2	82.0	16.9	-	-	-	7.77	2 35	109
30	1	43.2	20.6	3.88	-	-	14.2	19.5	101
	2	70.3	12.1	-	-	-	6 33	5 57	94.3
61	1	19.0	18.1	5.25	-	-	18.7	31.7	92.8
	2	26.9	19.4	7.27	-	-	19.7	20.9	94.1
92	1	8.85	7.63	5.04	6.04	15.1	9 21	40.4	92.3
	2	15.5	17.6	7.19	6.40	10.1	7.48	34.4	98.7
122	1	7 12	-	19.2	-	-	24.9	41.4	92.8
	2	12.5	-	26.5	-	-	23.1	36.7	98.8
183	1	7.63	-	15.9	5.68	9.42	5.40	41.4	85.4*
	2	10.1	-	19.6	5.68	9.57	4.82	37.1	86.9*

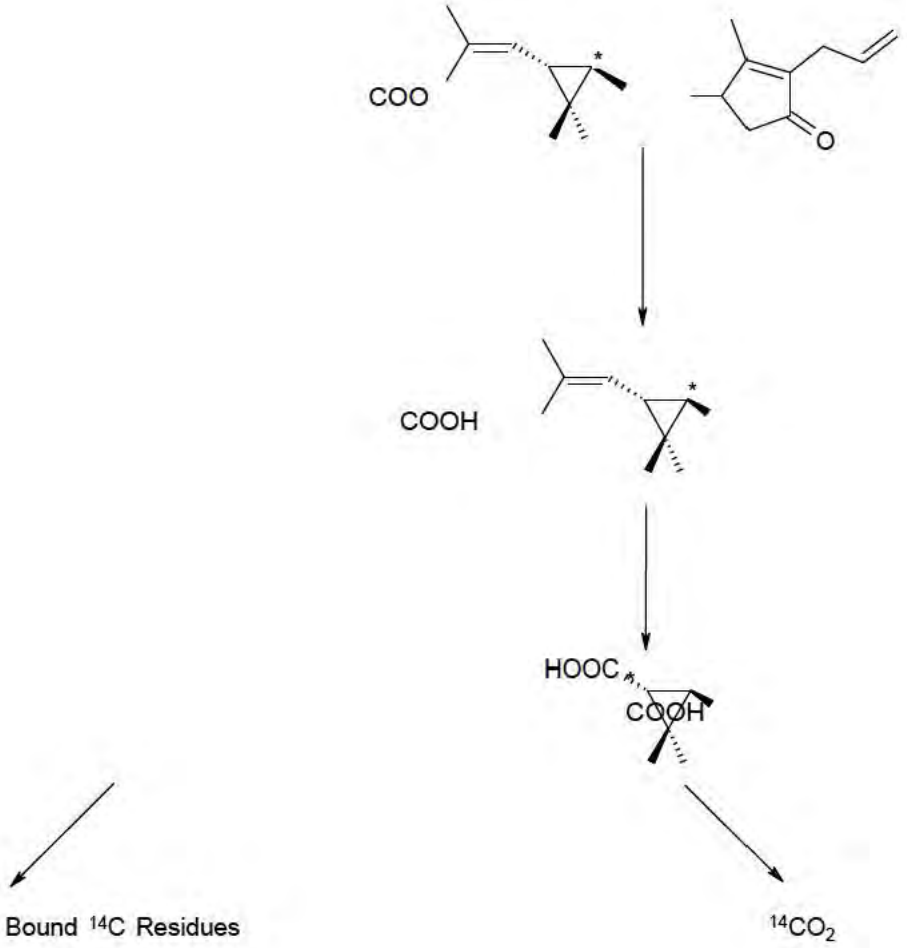
Soil Extracts: (I) Methanol, (II) methanol : water (1:1 v/v); (III) 0.2N HCl in methanol : water (1:1 v/v); (IV) reflux with 0.2N HCl in methanol : water (1:1 v/v); 1N NaOH.
- Extraction not performed
All results provided as percent of the initial measured dose (IMD), IMD = 1.39 µg/g.
*Lower mass balances can be attributed to losses inherent to multiple extractions.

4. Table 7.2.1/02-3: Profile of applied radioactivity in California Sandy Loam Soil under Aerobic Conditions

Sample time (days)	Replicate	[redacted]	[redacted]	[redacted]
0-(1)	1+2	95.0	NO	NO
0-(2)	1+2	96.5	0.079	NO
0-(3)	1+2	106	NO	NO
1	1	97.6	0.931	0.411
	2	88.6	1.13	0.873
2	1	90.4	2.09	0.721
	2	89.3	2.26	2.62
3	1	87.4	3.15	1.46
	2	103	3.55	1.59
4	1	89.0	4.19	2.86
	2	92.8	4.17	2.19
7	1	83.4	10.6	2.96
	2	88.6	7.82	0.935
14	1	59.0	13.7	5.58
	2	83.6	12.7	2.59
30	1	34.7	26.5	6.16
	2	64.3	15.6	1.94
61	1	9.55	30.0	2.72
	2	15.8	32.0	5.76
91	1	2.06	21.4	3.96
	2	4.97	35.1	6.18
122	1	1.41	22.1	2.72
	2	4.66	24.1	9.28
183	1	1.48	24.8	2.70
	2	2.42	28.1	3.32

NO – Not observed
Expressed as percent of initial measured dose (IMD), IMD = 1.39 μg/g.

5. Figure 7.2.1/02-1: Proposed Degradation Pathway of [Acid-^{14}C]-d-trans-Allethrin in Soil under Aerobic Conditions



*indicates radiolabel position

6. Annex 1 Evaluation by Rapporteur Member State, CA-Tables and CA-Figures

1. CA-Tables

1.1. CA-Table 1: Organic Matter Fractionation of Selected Test Samples

Organic Matter Fractionation of Selected Test Samples

Sample	% IMD in Organic Matter*	% of Total** in Humin	% of Total in Humic Acid	% of Total in Fulvic Acid
3MO-T(I)	21.4	43.1	33.7	23.2
3MO-T(II)	15.2	49.3	27.5	23.2
6MO-T(I)	13.8	39.1	25.5	35.4
6MO-T(II)	13.3	36.2	31.9	31.9

* Total = Humic Acid + Fulvic Acid + Humin

** Percent of total activity in organic matter as that fraction.

1.2. CA-Table 2: Degradation rate of [redacted] in two soil systems under aerobic conditions

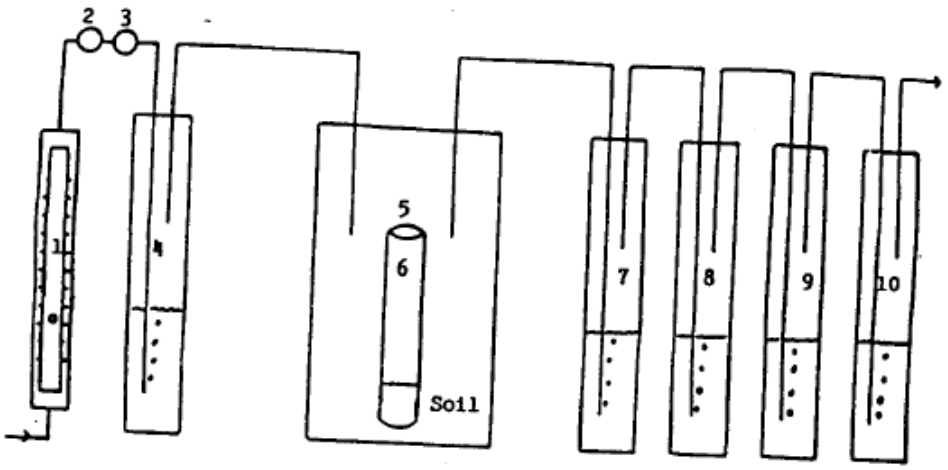
Test system	Kinetic model	DT50 (days) 25°C	DT50 (days) 12°C	DT90 (days)	χ^2 error %	Correlation coefficient (r^2)	$DT50_{mod}$** (days) 25°C	$DT50_{mod}$** (days) 12°C
System I	SFO*	19.4	54.9	64.4	3.4	0.997	19.4	54.9
	FOMC	20.1	-	60.5	3.2	0.997	-	-
	DFOP	19.4	-	64.4	3.6	0.997	-	-
System II	SFO*	31.7	89.7	105.4	9.8	0.965	31.7	89.7
	FOMC	34.4	-	90.3	n.a.	0.975	-	-
	DFOP	31.7	-	105.4	10.6	0.965	-	-

* best fit model selected according to FOCUS degradation kinetics report (2006), p. 108

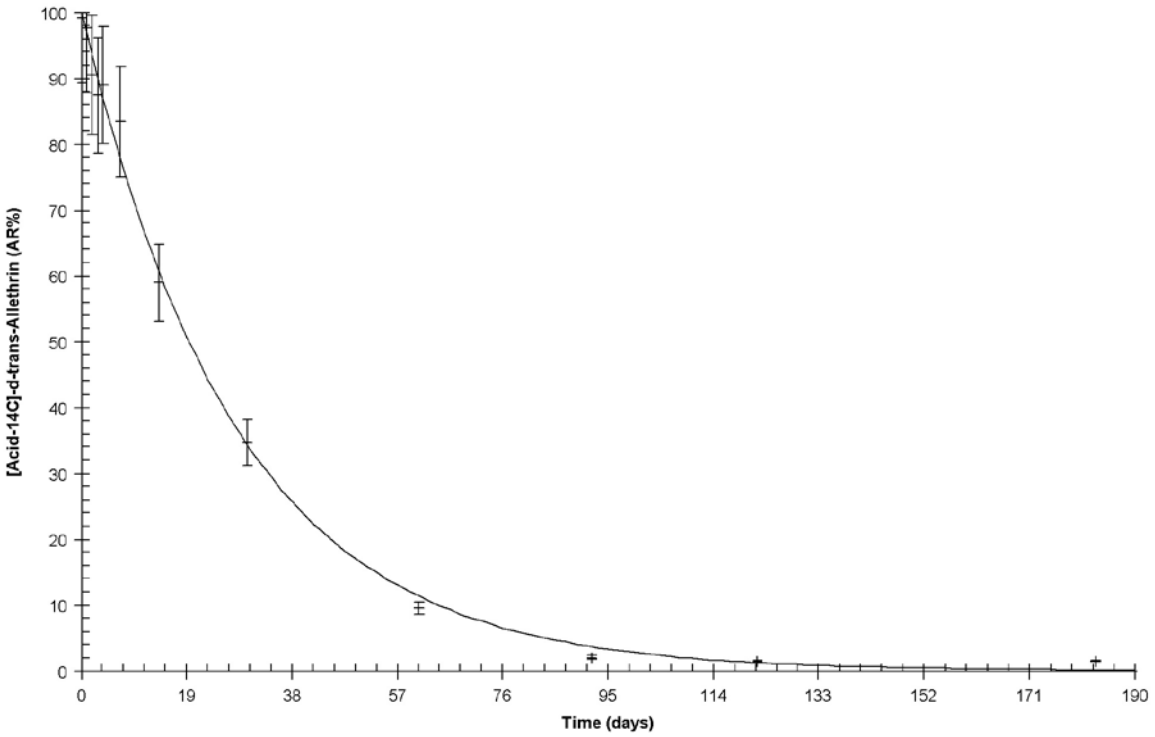
**DT50 modelling determined according to FOCUS degradation kinetics report (2006), p. 108

2. CA-Figures

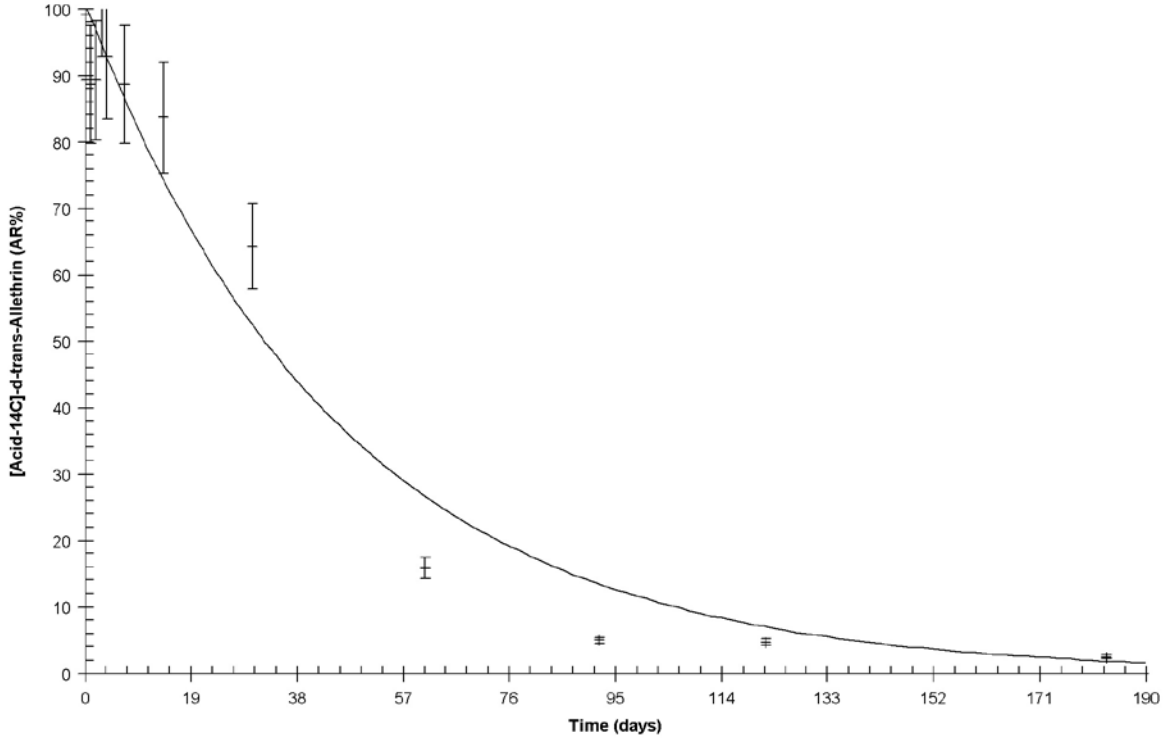
2.1. CA-Figure 1: Schematic Representation of the Experimental Setup Used for the Study.



1. Flow Meter
2. Drierite
3. Ascarite
4. Saturation Bottle
5. Metabolism Vessel
6. Sample Tube
7. Ethylene Glycol Trap
8. 1 N. H_2SO_4
9. 1 N. KOH Trap 1
10. 1 N. KOH Trap 2



2.2. CA-Figure 2: [Acid-^{14}C]d-trans-Allethrin, Schmidt, J.M. (1992), aerobic soil metabolism study (US-EPA 162-1, similar to OECD 307). System I. Calculation of DT50 by RMS with ModelMaker 4.0, 0-190 days, model: SFO. DT50: 19.4 days; k_deg: 0.0357368; M0: 100.165; R^2: 0.997; Error level Chi² test: 3.4%.



2.3. CA-Figure 3: [Acid-^{14}C]d-trans-Allethrin, Schmidt, J.M. (1992), aerobic soil metabolism study (US- EPA 162-1, similar to OECD 307). System II. Calculation of DT50 by RMS with ModelMaker 4.0, 0- 190 days, model: SFO. DT50: 31.7 days; k_deg: 0.0218544; M0: 100.971; R^2: 0.965; Error level Chi^2 test: 9.8%.

CAKE Kinetic Evaluation Report

Study: New Study

Data set: Experiment 1 (SFO)

Study date: 03 March 2017
Report generated: 03 March 2017

Model Setup:

Topology: Parent, A1 with link Parent–A1
Optimiser: IRLS (IRLS Its. 10, IRLS Tol. 1E-05, Max. Its. 100, Tol. 1E-05)
SANN Max Iterations: 10000
Extra Solver Option: Do Not Use

Initial Values of Sequence Parameters:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0 1	0 to (unbounded)	No
f_Parent_to_A1	0 5	0 to 1	No
A1_0	0	0 to (unbounded)	Yes
k_A1	0 1	0 to (unbounded)	No

Fit step: Final

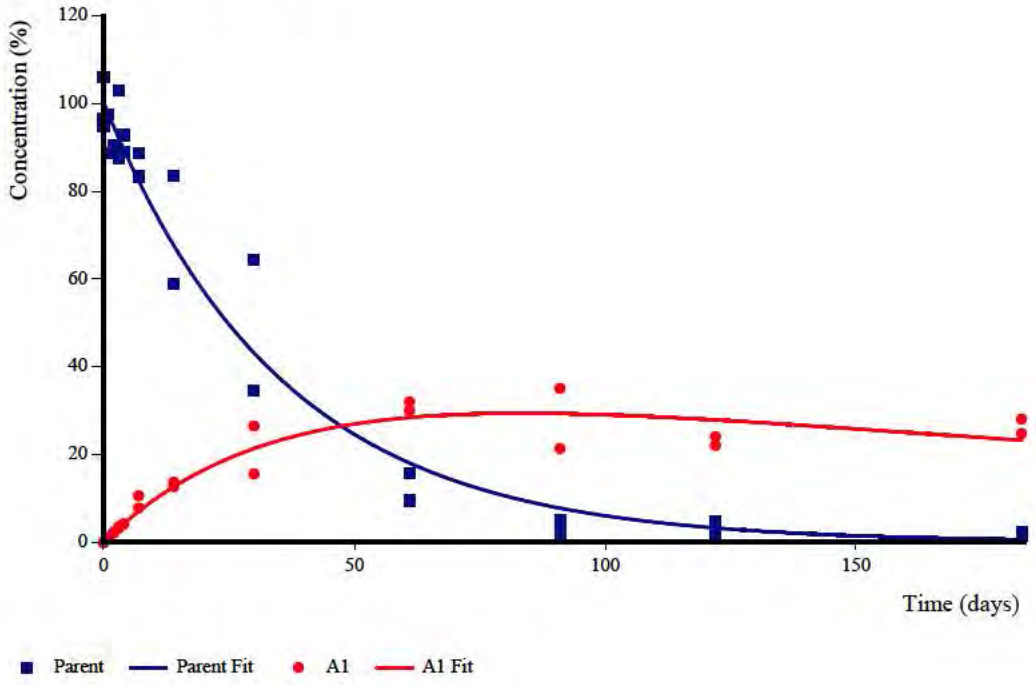
Used Extra Solver: No

Reference Table:

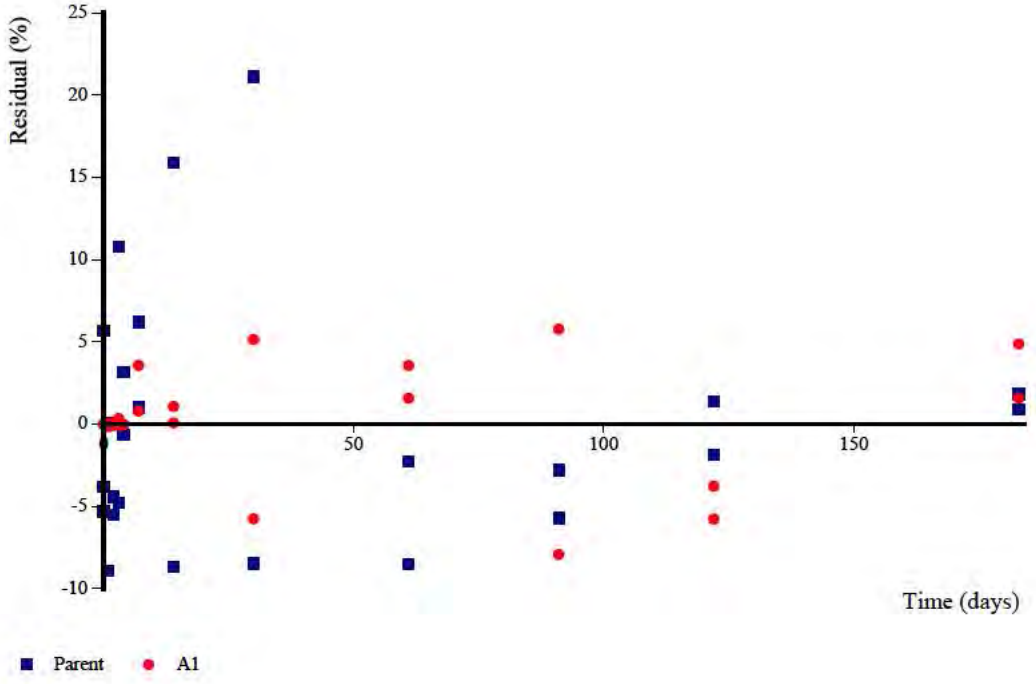
Compartment	Name
Parent	Parent
A1	A1

Graphical Summary:

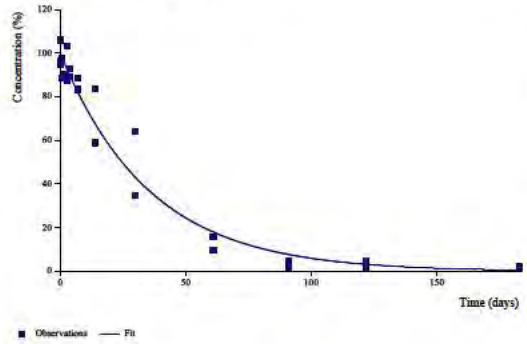
Observations and Fitted Model:

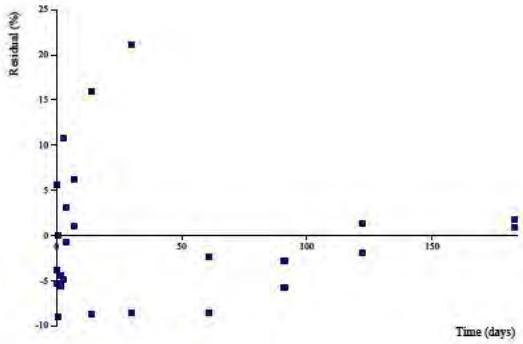


Residuals:

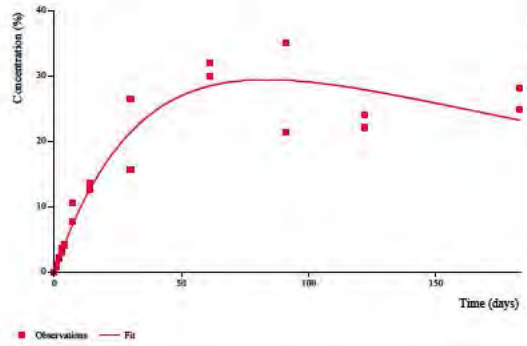


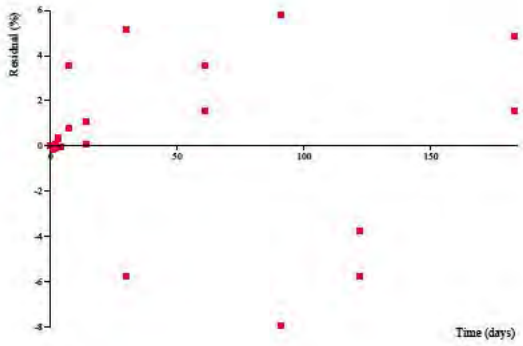
Compartment Parent:





Compartment A1:





Initial Values for this Step:

Parameter	Initial Value	Bounds	Fixed
Parent_0	99.84	0 to (unbounded)	No
k_Parent	0.02704	0 to (unbounded)	No
f_Parent_to_A1	0.4093	0 to 1	No
A1_0	0	0 to (unbounded)	Yes
k_A1	0.003887	0 to (unbounded)	No

Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	100.3	2.552	N/A	96.02	104.6	95.16	105.5
k_Parent	0.0281	0.002775	2.92E-013	0.02344	0.03277	0.02251	0.034
f_Parent_to_A1	0.3976	0.04278	N/A	0.3257	0.4695	0.3113	0.484
k_A1	0.003653	0.001139	0.001268	0.001738	0.005568	0.001356	0.006

χ^2

Parameter	Error %	Degrees of Freedom
All data	7.19	19
Parent	5.28	10
A1	11.1	9

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	24.7	81.9

A1	190	630

Additional Statistics:

Parameter	r² (Obs v Pred)	Efficiency
All data	0 9769	0.9769
Parent	0 9657	0.9657
A1	0 9238	0.9231

Parameter Correlation:

	Parent_0	k_Parent	f_Parent_to_A1	k_A1
Parent_0	1	0.478	-0.51	-0.2447
k_Parent	0.478	1	-0.6854	-0.512
f_Parent_to_A1	-0.51	-0.6854	1	0.8559
k_A1	-0.2447	-0.512	0.8559	1

Observed v. Predicted:

Compartment Parent

Time (days)	Value (%)	Predicted Value	Residual
0	95	100.3	-5.307
0	96.5	100.3	-3.807
0	106	100.3	5.693
1	97.6	97.53	0.0731
1	88.6	97.53	-8.927
2	90.4	94.82	-4.424
2	89.3	94.82	-5.524
3	87.4	92.2	-4.796
3	103	92.2	10.8
4	89	89.64	-0.6414
4	92.8	89.64	3.159
7	83.4	82.39	1.007
7	88.6	82.39	6.207
14	59	67.68	-8.679
14	83.6	67.68	15.92
30	34.7	43.17	-8.469
30	64.3	43.17	21.13
61	9.55	18.06	-8.514
61	15.8	18.06	-2.264
91	2.06	7.774	-5.714
91	4.97	7.774	-2.804
122	1.41	3.253	-1.843
122	4.66	3.253	1.407
183	1.48	0.5858	0.8942
183	2.42	0.5858	1.834

Compartment A1

Time (days)	Value (%)	Predicted Value	Residual

0	0	0	0
0	0	0	0
0	0	0	0
1	0.931	1.103	-0.1722
1	1.13	1.103	0.02679
2	2.09	2.172	-0.08179
2	2.26	2.172	0.08821
3	3.15	3.207	-0.05677
3	3.55	3.207	0.3432
4	4.19	4.209	-0.01907
4	4.17	4.209	-0.03907
7	10.6	7.029	3.571
7	7.82	7.029	0.791
14	13.7	12.63	1.075
14	12.7	12.63	0.0747
30	26.5	21.35	5.146
30	15.6	21.35	-5.754
61	30	28.43	1.572
61	32	28.43	3.572
91	21.4	29.32	-7.923
91	35.1	29.32	5.777
122	22.1	27.87	-5.769
122	24.1	27.87	-3.769
183	24.8	23.22	1.576
183	28.1	23.22	4.876

Sequence Creation Information:
Fit generated by CAKE version 3.2 (Release)
running on R version 3.0.0 (2013-04-03)

Report Information:
Report generated by CAKE version 3.2 (Release)
CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK, sponsored by Syngenta
Running on .NET version 4.0.30319.36373

Section A7.2.1/05 Aerobic Soil Metabolism of the metabolite [redacted]

Annex Point IIA 7.7

Official use only

1. REFERENCE

Reference

[redacted] Aerobic Degradation of [[redacted] in Three Soils. PTRL West, Inc. Study Number KM0026

Data protection Yes

1.1.1 Data owner: Sumitomo Chemical Company, Ltd. 27-1,Shinkawa 2-chome, Chuo-ku, Tokyo 104-8260

1.1.2

1.1.3 Criteria for data protection: Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I/IA

2. GUIDELINES AND QUALITY ASSURANCE

Guideline study OECD 307

GLP Yes

Deviations No

3. MATERIALS AND METHODS

Test material [redacted]

3.1.1 Lot/Batch number: [redacted]

3.1.2 Specification: As given in section 2

3.1.3 Purity: [redacted]

3.1.4 Further relevant properties: None reported.

3.1.5 Method of analysis

High Performance Liquid Chromatography

The method used for HPLC analysis of samples was as follows:

HPLC Method:

Column: Capcell-Pak 5u C18 MG II 120A (250 x 4.6 mm)

Solvents: Reservoir A: 0.05% FA in Water (HPLC Grade)

Reservoir B: 0.05% FA in ACN

Gradient:linear

Flow rate 1 mL/minute

UV at 230 nm

Analysis of Radiochromatographic Data

Samples with adequate radioactivity were analyzed using the Beta ram flow through detector and chromatograms were created using Laura® software. HPLC radiochromatograms were generated for samples with lower concentrations of radioactivity by collecting fractions (0.5 min. fractions) of the eluant. From these chromatograms, retention times and areas under peaks were obtained. These conditions were performed using computer-assisted techniques developed in the laboratory (Appendix F). Peak assignments were based on co-elution with reference standards injected with each sample.

Section A7.2.1/05 Aerobic Soil Metabolism of the metabolite [redacted]

Annex Point IIA 7.7

		Confirmatory Thin Layer Chromatography (TLC) Confirmation of the peak assignment for [redacted] and its degradates *c*-COOH-CA was performed by two-dimensional TLC analysis of selected samples. TLC analysis utilized pre-coated TLC plates (EMD Silica Gel 60 F254 pre-coated 20 cm × 20 cm × 250 µm thickness). For two-dimensional TLC, selected soil extracts and the corresponding reference standards were spotted at the origin and the plates were developed in two dimensions using the following solvent systems: Solvent System A: Hexane/ethyl acetate/acetic acid, 3/10/1, v/v/v Solvent System B: Toluene/acetic acid, 15/2, v/v Reference standards, also spotted in the lanes of each plate, were visualized after spraying with 0.3% bromocresol green solution and heating the plates in an oven for 5-10 minutes.
	Degradation products	Degradation products tested: Yes
3.1.6	Method of analysis for degradation products	HPLC
	Reference substance	Yes
3.1.7	Method of analysis for reference substance	HPLC
	Soil types	Refer to Table A7_2_1-1
	Testing procedure	*Non-entry field*
3.1.8	Test system	Soils were collected from three agricultural sites in France and received at PTRL West on 18 November, 2010. On arrival at PTRL West, Inc., the soil was stored refrigerated in the dark until use in the study. The moisture content of the soil was determined by oven drying soil aliquots (5 x 5 g) and comparing their weight before and after drying. Physicochemical characterization of the soil was determined at Agvise Laboratories, Northwood, ND..
3.1.9	Test solution and Test conditions	Approximately 50 g dry weight equivalent of each soil were weighed into 250 mL amber bottles. The samples were placed in soil chambers in the Hotpack constant temperature room and pre-incubated at 20 ± 2°C for 7 days in the dark with a humidified air flow. Empty bottle weights prior to addition of soil as well as total sample weights were recorded for all samples for moisture monitoring during the study.
	Test performance	*Non-entry field*
3.1.10	Preliminary test	According to (a)"OECD 106": No. Not applicable.
3.1.11	Screening test: Adsorption	According to (a)"OECD 106": No. Not applicable.
3.1.12	Screening test: Desorption	According to (a)"OECD 106": No. Not applicable
3.1.13	HPLC-method	According to (a)" OECD-HPLC-method":Not applicable
3.1.14	Other test	None

Section A7.2.1/05 Aerobic Soil Metabolism of the metabolite [redacted]

Annex Point IIA 7.7

RESULTS

Preliminary test

Summary of Results for the Preliminary Study.

2111W-012 Aschard loam soil

% of applied dose		
Sample	[redacted]	[redacted]
Time 0 rep A	97.7	0.0
1 DAT rep A	86.3	0.0
7 DAT rep A	11.9	0.0

2111W-013 France site 2 loam soil

% of applied dose		
Sample	[redacted]	[redacted]
Time 0 rep A	95.3	0.4
1 DAT rep A	89.6	0.0
7 DAT rep A	31.2	0.0

2111W-014 Norbert sandy clay loam soil

% of applied dose		
Sample	[redacted]	[redacted]
Time 0 rep A	98.4	0.0
1 DAT rep A	90.8	0.0
7 DAT rep A	59.2	0.0

Screening test: Adsorption

Not applicable

Screening test: Desorption

Not applicable

Calculations

Non-entry field

3.1.15 Ka , Kd

Not applicable

3.1.16 Ka_{oc} , Kd_{oc}

Not applicable

Degradation product(s)

n/a

APPLICANT'S SUMMARY AND CONCLUSION

Materials and methods

An aerobic soil metabolism study was conducted using [acid-14C]trans-COOH-CA on three soils. Soils were freshly collected from three sites in France designated Aschard (loam soil), France site #2 (loam soil) and Norbert (sandy clay loam soil). Soil samples were treated with [acid-14C][redacted] at approximately 0.39 ppm and incubated in the dark at 20°C for periods of 14 to 30 days. The soil samples were continuously aerated throughout the incubation period. Traps for volatiles included an ethylene glycol trap for organic volatiles and 10% aqueous NaOH traps for CO2. [acid-14C][redacted] was quantified by high performance liquid chromatography (HPLC) of soil extracts with co-injection of the analytical reference standard. A reference standard for cis-COOH-CA was included in the analysis to assess isomerization during the study. A preliminary experiment was conducted to determine

appropriate extraction methods and sampling intervals.

Section A7.2.1/05 Aerobic Soil Metabolism of the metabolite [redacted]

Annex Point IIA 7.7

Results and discussion

Average radiocarbon recoveries, based on the applied radiocarbon (AR), were 94.0 ± 4.0% for the Aschard soil. Extractable radiocarbon declined from an average of 98.7% AR at time 0 to 2.7% at 14 days. Soil bound residues increased from 1.7% AR at time 0 to a maximum of 49.3% at day 10 then declined to 46.9% at day 14. Radiocarbon recovered in the NaOH traps increased rapidly to 41.6% AR by day 10.

For the France site #2 soil, average radiocarbon recoveries were 95.1 ± 2.9% AR. Extractable radiocarbon declined from an average of 97.2% AR at time 0 to 4.1% at 30 days. Soil bound residues increased from 1.3% AR at time 0 to a maximum of 43.6% at day 14 then declined to 39.6% at day 30. Radiocarbon recovered in the NaOH traps increased to 50.3% AR by day 30.

For the Norbert soil set, average radiocarbon recoveries were 94.5 ± 4.1% AR. Extractable radiocarbon declined from an average of 97.5% AR at time 0 to 10.7% at 30 days. Soil bound residues increased from 2.2% AR at time 0 to a maximum average of 33.0% at day 30. Radiocarbon recovered in the NaOH traps increased to 45.4% AR by day 30.

Harsh extraction using overnight Soxhlet was conducted on representative samples of the Aschard and France site #2 soils at day 8 and 10 as well as all Norbert soils after time 0.

This additional extraction released a maximum of 1.1, 1.6 and 5.1% AR for the Aschard, France site #2 and Norbert soils, respectively. Further characterization was done by partitioning the samples with the highest bound residues into the humic acid (HA) and fulvic acid (FA) fractions and the insoluble humin.

The DT50 and DT90 values for [acid-14C][redacted] degradation in the three soils were calculated using single first-order kinetics (SFO) with KinGUI version 1.1 software. The DT50 and DT90 in each test system were determined using the FOCUS Approach.

Soil	DT_{50} (days) SFO	DT_{90} (days) SFO	Correlation Coefficient (r^2)	Chi^2 Error %	M0 std dev	Prob > t
Aschard	1.9	6.3	0.98	13.3	3.76	2.7x10-8
France site #2	3.1	10.3	0.98	9.8	3.32	4x10-10
Norbert	5.3	17.5	0.95	9.2	3.65	3.8x10-9

Section A7.2.1/05 Aerobic Soil Metabolism of the metabolite [redacted]

Annex Point IIA 7.7

3.1.17	Adsorbed a.s. [%]	Not applicable
3.1.18	K_a	Not applicable
3.1.19	K_d	Not applicable
3.1.20	Ka_{oc}	Not applicable
3.1.21	Ka/Kd	Not applicable
3.1.22	Degradation products (% of a.s.)	Not applicable
	Conclusion	Degradation of [acid-14C][redacted] was rapid in Aschard loam, France site #2 loam and Norbert sandy clay loam soils. The half lives were calculated to be 1.9, 3.1, and 5.3 days, respectively, using single first order kinetics. No isomerization from *trans*-COOH-CA to *cis*-COOH-CA was detected during the course of the study.
3.1.23	Reliability	[redacted]
3.1.24	Deficiencies	None.

Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	03/02/2017
Materials and Methods	Three French soils were used (Aschard, loam, pH 7.7; Site #2, loam, pH 6.9; Norbert, sandy clay loam, pH 6.5). All soils had a fairly low organic matter content (1.6 – 1.8 %). Soil was stored refrigerated in the dark until use in the study. Microbial viability was tested pre-study, at study commencement and at the end of the study using substrate induced respiration (SIR). Test was done on untreated soil. Soil moisture was adjusted to75 % field capacity and checked and topped up every two weeks. There was a 7 day pre-incubation period. *Extraction methods*

Section A7.2.1/05 Aerobic Soil Metabolism of the metabolite [redacted]

Annex Point IIA 7.7

Results and discussion

Mass balance fell below the OECD recommended minimum of 90 % (Ranged from 88.6 to 100 % for the Aschard soil test, 89.0 to 99.0 % for France #2 soil, and from 86.5 to 101.6 % for the Norbert soil).

The proportion of AR in bound residues was high (maximum of 51 % in Aschard, 43.6 % in France #2, and 33 % in Norbert). Analysis of selected soil bound residues revealed that most activity was associated with the humic fraction (see table below). This may indicate poor initial extraction.

	Fulvic Acid Fraction		Humic Acid Fraction	
Sample	% of bound residues	% of initial dose	% of bound residues	% of initial dose
Aschard	14.2	7.6	29.6	15.8
France #2	19.5	8.7	37.7	16.8
Norbert	21.5	7.6	19.7	7.0

The applicant's kinetic analysis was checked by the UK CA using CAKE version 3.1 (Figure A7_2_1-2, 3 and 4) and the UK is agreement with the results presented by the applicant.

Conclusion The applicant's version is considered to be acceptable.

Reliability 1

Acceptability Acceptable

Remarks

COMMENTS FROM ...

Date *Give date of comments submitted*

Materials and Methods *Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant's summary and conclusion.*
Discuss if deviating from view of rapporteur member state

Results and discussion *Discuss if deviating from view of rapporteur member state*

Conclusion *Discuss if deviating from view of rapporteur member state*

Reliability *Discuss if deviating from view of rapporteur member state*

Acceptability *Discuss if deviating from view of rapporteur member state*

Remarks

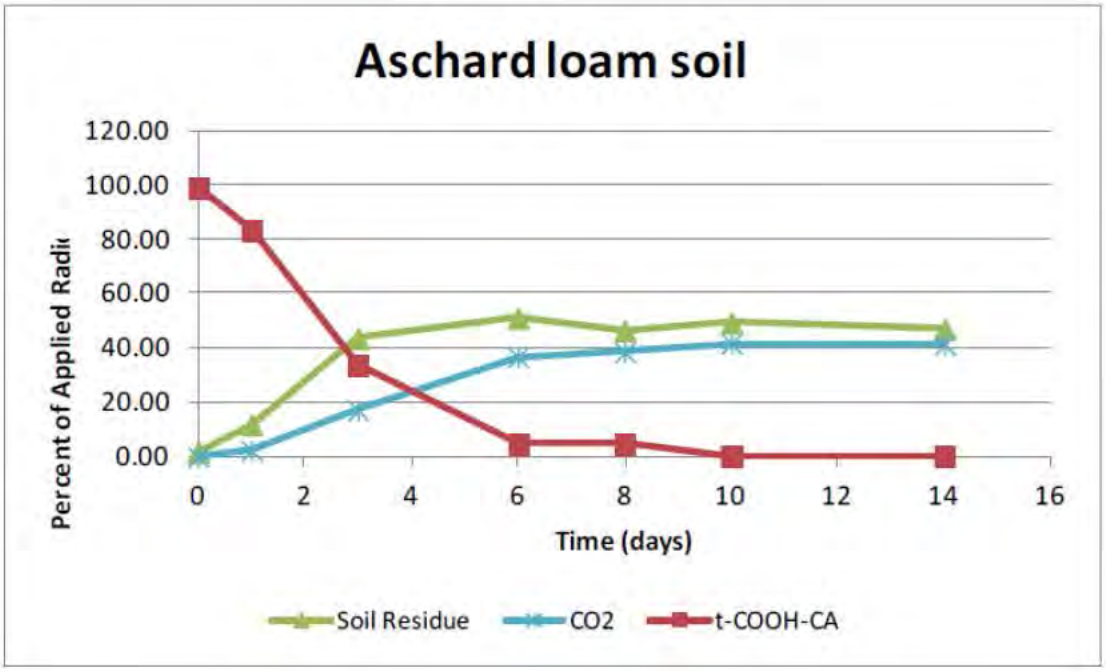
Table A7_2_1-1: Classification and physico-chemical properties of soils used as adsorbents

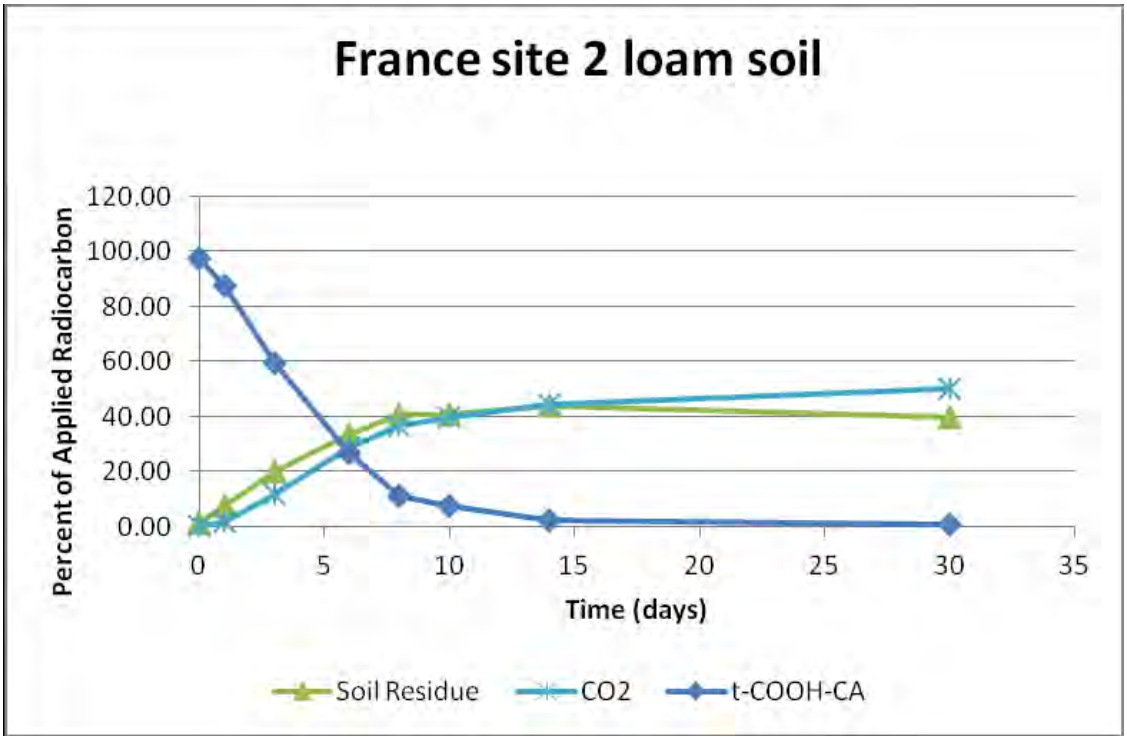
	Soil 1 Aschard	**Soil 2 Guthmann**	**Soil 3 Norbert**
Soil order	-		
Soil series			
Classification	Loam	Loam	Sandy Clay Loam
Location	France	France	France
Horizon	-	-	-
Sand [%]	41	49	49
Silt [%]	42	38	24
Clay [%]	17	13	27
Organic matter [%]	1.8	1.7	1.6
Organic carbon [%]	-	-	-
Carbonate as $CaCO_3$	-	-	-
insoluble carbonates [%]	-	-	-
pH (1:1 H_2O)	7.7	6.9	6.5
Cation exchange capacity (MEQ/100 g)	11.2	7.7	16.5
Base saturation (%)	-	-	-
Ca	79.3	61.5	56.2
Mg	3.3	9.8	22.2
Na	0.4	0.7	0.9
K	3.5	4.1	1.7
H	13.5	24.0	18.9
Special chemical/mineralogical features	-	-	-
Clay fraction mineralogy	-	-	-
Bulk Density	1.22	1.21	1.14
Maximum water capacity (%)	32.0	21.8	24.1

Table A7_2_1-2: Degradation of [redacted] in soil

Soil	DT_{50} (days) SFO	DT_{90} (days) SFO	Correlation Coefficient (r^2)	Chi^2 Error %	M0 std dev	Prob > t
Aschard	1.9	6.3	0.98	13.3	3.76	2.7x10-8
France site #2	3.1	10.3	0.98	9.8	3.32	4x10-10
Norbert	5.3	17.5	0.95	9.2	3.65	3.8x10-9

Figure A7_2_1-1 Graphical Representation of for the Test Substance and Degradates for the Aerobic Degradation of [^{14}C][redacted]




France site 2 loam soil
Percent of Applied Radiocarbon
120.00
100.00
80.00
60.00
40.00
20.00
0.00
0
5
10
15
20
25
30
35
Time (days)
Soil Residue
CO2
t-COOH-CA

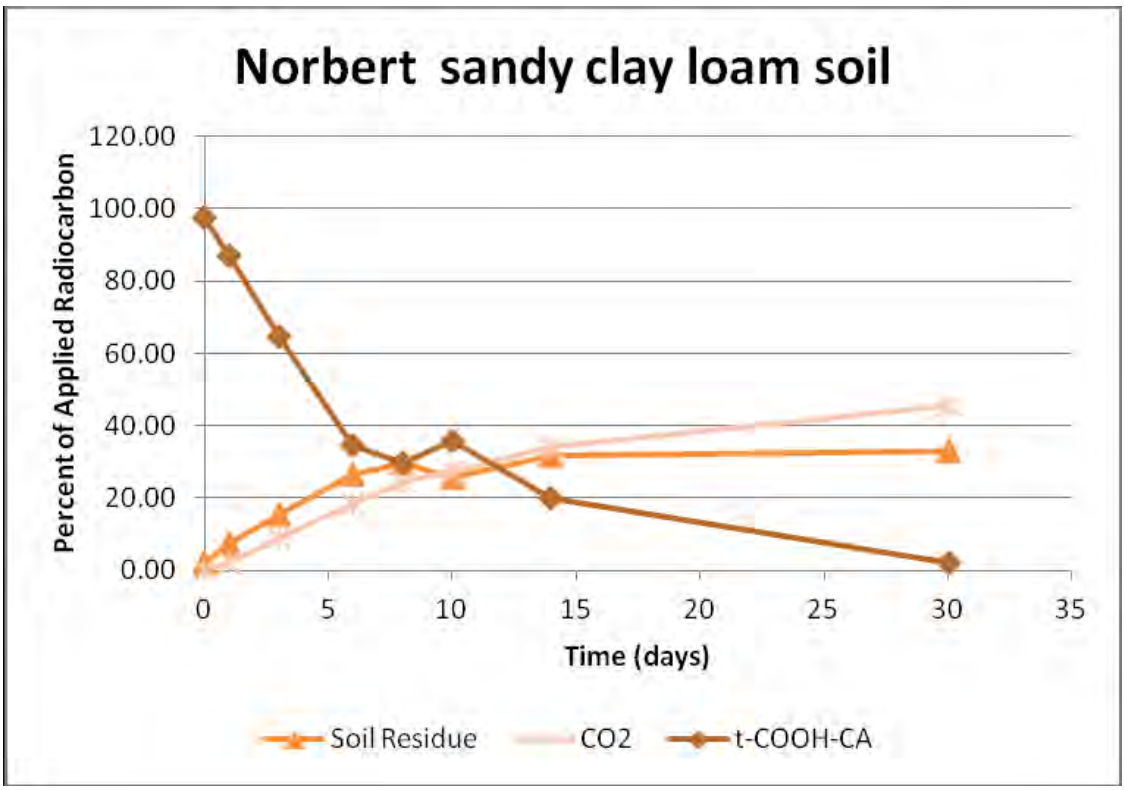

Norbert sandy clay loam soil
Percent of Applied Radiocarbon
120.00
100.00
80.00
60.00
40.00
20.00
0.00
0
5
10
15
20
25
30
35
Time (days)
Soil Residue
CO2
t-COOH-CA

Figure A7_2_1-2 CAKE Kinetic Evaluation Report of [redacted] in Aschard Soil

Study: [redacted] Aschard soil

Data set: Experiment 1 (SFO) with OLS

Study date: 17 January 2017
Report generated: 17 January 2017

Model Setup:

Topology: Parent only
Optimiser: OLS (Max. Its. 100, Tol. 1E-05)
SANN Max Iterations: 10000
Extra Solver Option: Do Not Use

Initial Values of Sequence Parameters:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0 1	0 to (unbounded)	No

Fit step: Final

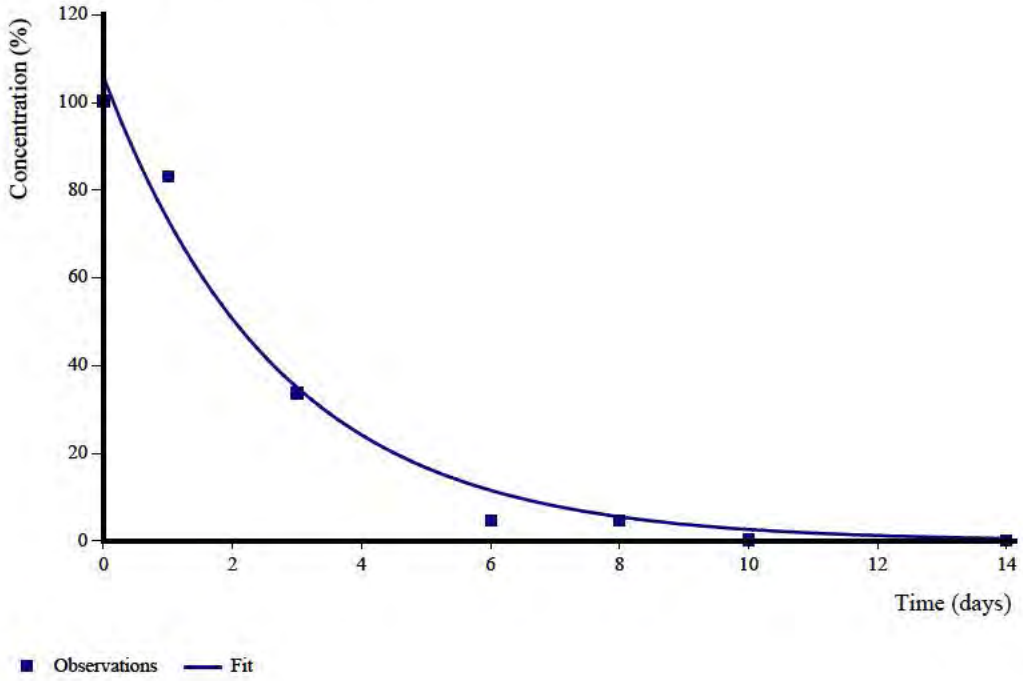
Used Extra Solver: No

Reference Table:

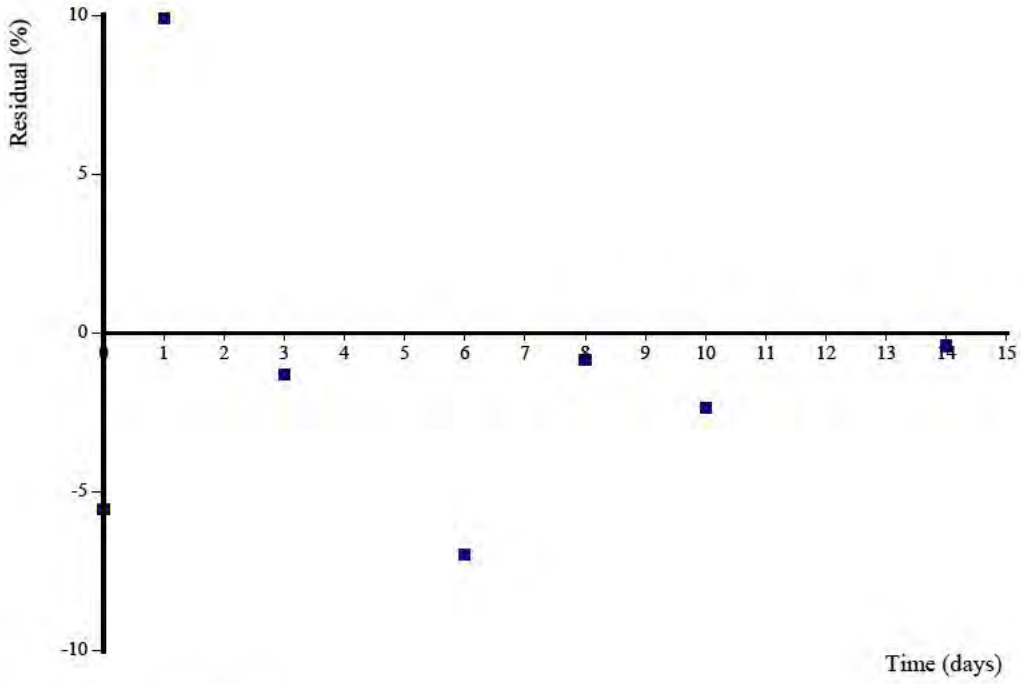
Compartment	Name
Parent	Parent

Graphical Summary:

Observations and Fitted Model:



Residuals:



Initial Values for This Step:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0 1	0 to (unbounded)	No

Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	105.8	5.52	N/A	94.72	117	91.66	120
k_Parent	0 3688	0.04516	2.24E-004	0.2778	0.4598	0.2527	0.485

χ^2

Parameter	Error %	Degrees of Freedom
All data	12.6	5
Parent	12.6	5

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	1.88	6.24

Additional Statistics:

Parameter	r^2 (Obs v Pred)	Efficiency
All data	0 9841	0.9828
Parent	0 9841	0.9828

Parameter Correlation:

	Parent_0	k_Parent
Parent_0	1	0.4899

k_Parent	0.4899	1

Observed v. Predicted:

Compartment Parent

Time (days)	Value (%)	Predicted Value	Residual
0	100 3	105.8	-5.547
1	83.1	73.2	9.901
3	33.7	35.01	-1.307
6	4.6	11.58	-6.978
8	4.7	5.537	-0.8372
10	0 3	2.648	-2.348
14	0 2	0.6057	-0.4057

Sequence Creation Information:
Fit generated by CAKE version 3.1 (Release)
running on R version 3.0.0 (2013-04-03)

Report Information:
Report generated by CAKE version 3.1 (Release)
CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK for Syngenta
Running on .Net version 4.0.30319.34209

Figure A7_2_1-3 CAKE Kinetic Evaluation Report of [redacted] in France #2 Soil

Study: [redacted] France #2 soil

Data set: Experiment 1 (SFO) with OLS

Study date: 17 January 2017
Report generated: 17 January 2017

Model Setup:

Topology: Parent only
Optimiser: OLS (Max. Its. 100, Tol. 1E-05)
SANN Max Iterations: 10000
Extra Solver Option: Do Not Use

Initial Values of Sequence Parameters:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0 1	0 to (unbounded)	No

Fit step: Final

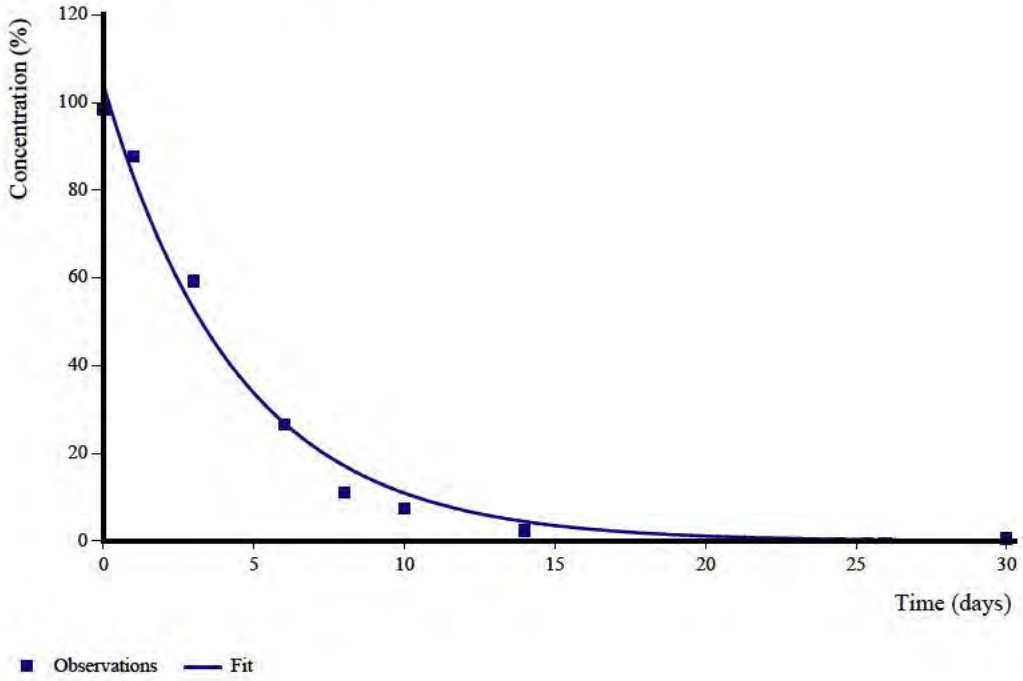
Used Extra Solver: No

Reference Table:

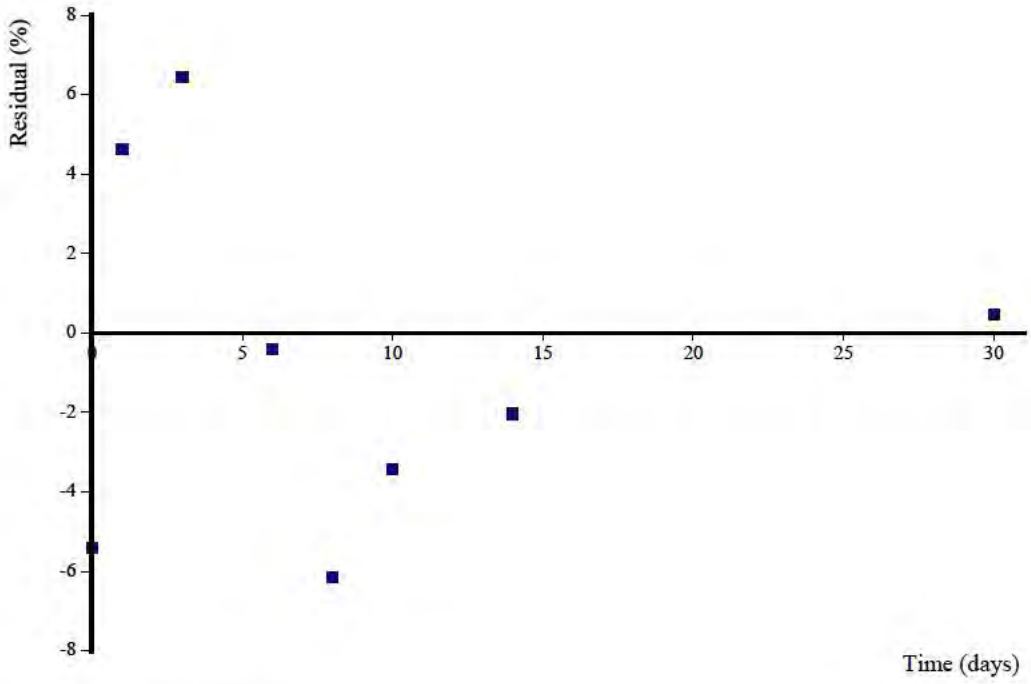
Compartment	Name
Parent	Parent

Graphical Summary:

Observations and Fitted Model:



Residuals:



Initial Values for This Step:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0 1	0 to (unbounded)	No

Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	103 9	4.093	N/A	95.96	111.9	93.9	113.9
k_Parent	0 2253	0.01903	1.10E-005	0.1883	0.2622	0.1787	0.272

χ^2

Parameter	Error %	Degrees of Freedom
All data	9.29	6
Parent	9.29	6

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	3.08	10.2

Additional Statistics:

Parameter	r^2 (Obs v Pred)	Efficiency
All data	0 9875	0.9867
Parent	0 9875	0.9867

Parameter Correlation:

	Parent_0	k_Parent
Parent_0	1	0.5232

k_Parent	0 5232	1

Observed v. Predicted:

Compartment Parent

Time (days)	Value (%)	Predicted Value	Residual
0	98.5	103.9	-5.416
1	87.6	82.96	4.643
3	59.3	52.87	6.432
6	26.5	26.9	-0.3973
8	11	17.14	-6.142
10	7 5	10.92	-3.424
14	2.4	4.437	-2.037
30	0.6	0.1207	0.4793

Sequence Creation Information:
Fit generated by CAKE version 3.1 (Release)
running on R version 3.0.0 (2013-04-03)

Report Information:
Report generated by CAKE version 3.1 (Release)
CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK for Syngenta
Running on .Net version 4.0.30319.34209

Figure A7_2_1-4 CAKE Kinetic Evaluation Report of [redacted] in Norbert Soil

Study: [redacted] France Norbert soil

Data set: Experiment 1 (SFO) with OLS

Study date: 17 January 2017
Report generated: 17 January 2017

Model Setup:

Topology: Parent only
Optimiser: OLS (Max. Its. 100, Tol. 1E-05)
SANN Max Iterations: 10000
Extra Solver Option: Do Not Use

Initial Values of Sequence Parameters:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0 1	0 to (unbounded)	No

Fit step: Final

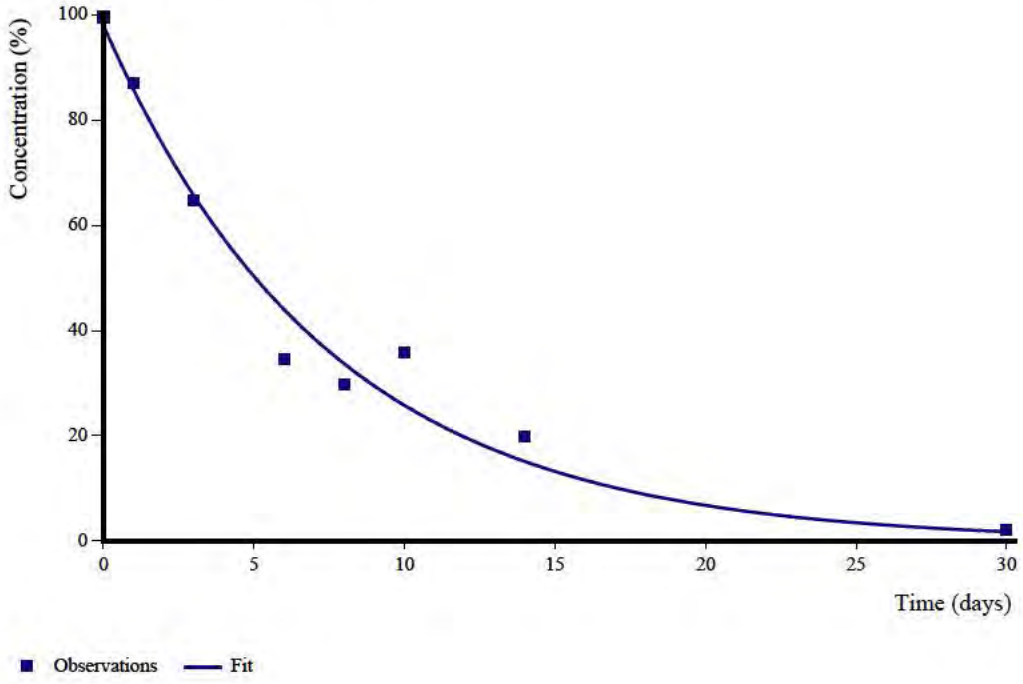
Used Extra Solver: No

Reference Table:

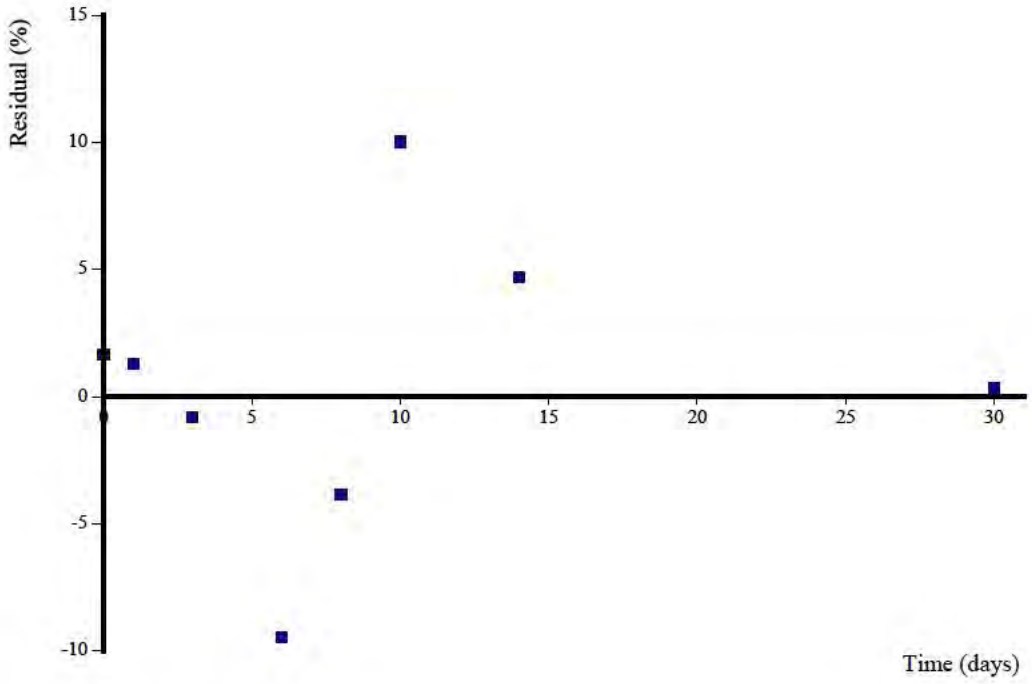
Compartment	Name
Parent	Parent

Graphical Summary:

Observations and Fitted Model:



Residuals:



Initial Values for This Step:

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0 1	0 to (unbounded)	No

Estimated Values:

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	97.96	4.722	N/A	88.78	107.1	86.4	109.5
k_Parent	0.1335	0.01392	3.68E-005	0.1065	0.1606	0.09944	0.168

χ^2

Parameter	Error %	Degrees of Freedom
All data	9 2	6
Parent	9 2	6

Decay Times:

Compartment	DT50 (days)	DT90 (days)
Parent	5.19	17.3

Additional Statistics:

Parameter	r^2 (Obs v Pred)	Efficiency
All data	0 9717	0.971
Parent	0 9717	0.971

Parameter Correlation:

	Parent_0	k_Parent
Parent_0	1	0.5827

k_Parent	0 5827	1

Observed v. Predicted:

Compartment Parent

Time (days)	Value (%)	Predicted Value	Residual
0	99.6	97.96	1.643
1	87	85.72	1.286
3	64.8	65.63	-0.8287
6	34.5	43.97	-9.47
8	29.8	33.67	-3.866
10	35.8	25.78	10.02
14	19.8	15.11	4.688
30	2 1	1.785	0.3151

Sequence Creation Information:
Fit generated by CAKE version 3.1 (Release)
running on R version 3.0.0 (2013-04-03)

Report Information:
Report generated by CAKE version 3.1 (Release)
CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK for Syngenta
Running on .Net version 4.0.30319.34209