Renewal Assessment Report

beta-cyfluthrin

Volume 3 – B.8 Environmental fate and behaviour

07 March 2017

Rapporteur Member State: Germany

Co-Rapporteur Member State: Hungary

Version history

When	What

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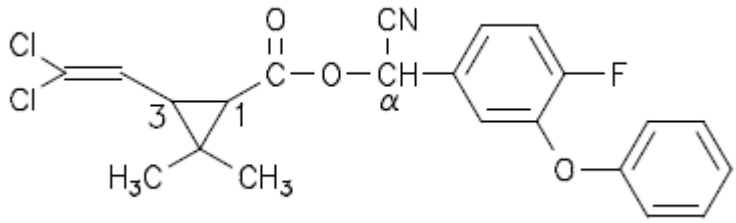
B.8 Environmental fate and behaviour

Remark:
In this chapter B.8 no literature is discussed as the applicant did not deliver the whole set of literature to the RMS.

The chemical composition of beta-cyfluthrin is similar to that of cyfluthrin. The common molecular structure shows three asymmetric carbon atoms (chiral centres) which lead to four diastereomers. The difference between beta-cyfluthrin and cyfluthrin is the ratio between the four diastereomers. While cyfluthrin consists of all four diastereomers (referred to as diastereomer I, II, II and IV), beta-cyfluthrin represents mainly the biologically more active diastereomers II and IV. The diastereomers I and III are present in low portions.

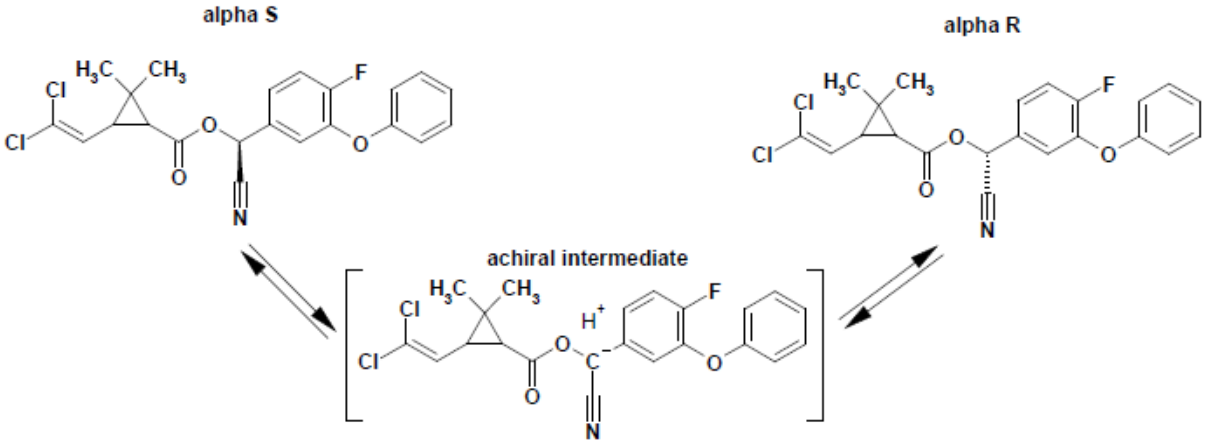
Molecular structure of beta-cyfluthrin:



Diastereomer II	1R, 3R, αS + 1S, 3S, αR (1 : 1),cis	30.0 - 40.0%
Diastereomer IV	1R, 3S, αS + 1S, 3R, αR (1 : 1), trans	57.0 - 76.0%

Two chiral centres originate from the cyclopropyl moiety, the third one from the α-cyano-methyl group. Due to CH-acidic properties of the latter group, isomerisation by means of a deprotonation – protonation sequence via an achiral intermediate can occur in protic matrices.

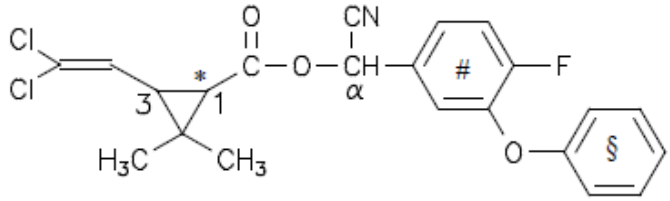
Isomerisation of cyfluthrin in protic solvents:



The fate and behaviour of beta-cyfluthrin and cyfluthrin was investigated using test substances labelled in the fluorophenyl-, phenyl- or cyclopropyl-moieties. The different labelling positions are presented in

the figure below.

Labelling positions of beta-cyfluthrin



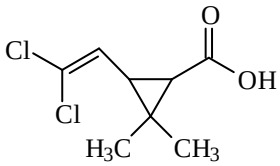
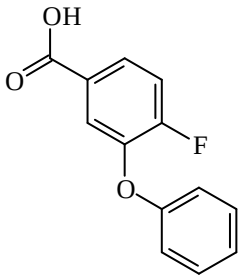
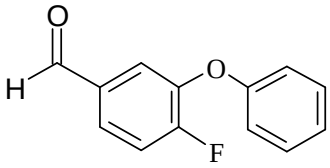
* [cyclopropyl-1-^{14}C] labelling position

[fluorophenyl-UL-^{14}C] labelling position

§ [phenyl-UL-^{14}C] labelling position

In the following table basic information about metabolites most frequently mentioned in this volume are shown for quick orientation.

Metabolites of beta-cyfluthrin

Name and code	Structure and formula	Molar mass (g/mol)
DCVA	 $C_8H_{10}Cl_2O_2$	209.1
FPB-acid COE 538/78 4-Fluoro-3-phenoxybenzoic acid	 $C_{13}H_9FO_3$	232.21
FPB-aldehyde FCR 1260 4-Fluoro-3-phenoxybenzaldehyde	 $C_{13}H_9FO_2$	216.21

B.8.1 Fate and behaviour in soil

B.8.1.1 Route of degradation in soil

B.8.1.1.1 Route of degradation of active substance in soil – aerobic laboratory studies

The studies by **Minor 1986** and **Wagner et al. 1983** on soil metabolism were evaluated in the monograph from 1 October 1996. However the data from these studies are not given in the monograph. Therefore a short summary is given here.

B.8.1.1.1/1 (Minor 1986)

Reference	Minor, R.G.	Incubation time	225 days
Title	Comparison of ^{14}C Baythroid Degradation in Soils from the United States and West Germany	Soils	sandy loam, 1.4 % organic carbon, pH 5.0 (0.01 M $CaCl_2$)
Year of execution	09.02.1986 (rev. 20.06.1988)	Nominal concentration	1 kg/ha Baythroid 200 EC
Study no.	91816		
GLP statement	No	Temperature	unknown (soil was maintained outside)
Guideline	None	Moisture	unknown (the soil was watered periodically)
Test substance	Phenyl-UL-^{14}C Baythroid 200 EC	DT_{50}	not calculated
Purity	Unknown	Metabolites	see results
Test system	aerobic degradation	Acceptability	no valid study

Study design

The study was conducted in Stanley, Kansas between August 1982 and July 1983 as a rotational crop study. The test substance was applied on the soil surface and incorporated into the soil after 36 days. The degradation of Baythroid within the soil was considered secondary.

Soil sampling and extracting

Six 15 × 3 cm soil plugs were taken 0, 70 and 189 days after the test substance had been incorporated into the soil (i.e. 36 days posttreatment). The soil plugs were thoroughly mixed, and aliquots were radioassayed to determine the total radioactive residue. Soil was subjected to a 5-hour Soxhlet extraction with chloroform/methano (7:3). After concentration of the chloroform/methanol mixture to near dryness using a rotary evaporator at 32 °C, the extract was taken up in methanol, radioassayed and subjected to thin-layer chromatography.

Results

Baythroid was the major component identified in the soil. The percent of Baythroid declined from 55 % of the total radioactivity at 36 day posttreatment to 15 % at day 225. Two main degradation products were detected in the soil, FPB acid and FCR2978 (amid-cyfluthrin). At the end of the study (day 225) a 33 % loss of radioactivity occurred; this loss of radioactivity was attributed to $^{14}CO_2$.

Table B.8.1-1: Mass balance and distribution of radioactivity in extracts of Les Evouettes soil under aerobic conditions (values as % of applied)

Fraction	Days after treatment		
	36	**106**	**225**
Baythroid	55	35	15
FPB acid	8	3	2
FCR2978 (amid-cyfluthrin)	4	4	2
other organosolubles	0	4	1
TLC origin	8	6	5
losses including CO_2	0	36	33
bound	25	12	42
TOTAL	100	100	100

Conclusion

As already stated in the monograph dated 01 October 1996 the study cannot be fully accepted as valid especially for the following reasons: formation of CO_2 has not been determined and hence no information on mass balance is available; due to the outside incubation no information on temperature and humidity is given. The study may give some additional information on formation of metabolites and bound residues.

B.8.1.1.1/2 (Wagner et al. 1983)

Reference	Wagner, K., Neitzel, H. and Oehlmann, L.	Incubation time	190 - 365 days
Title	Degradation of Baythroid in soil under aerobic and anaerobic test conditions	2 Soils	loam and sandy loam
Year of execution	19-01-1983	Nominal concentration	1 mg/kg *
Study no.	RA-87/83, MR86052		
GLP statement	no	Temperature	18 – 22 °C
Guideline	none	Moisture	determined moisture content were maintained in all tests
Test substance	(fluorobenzenring-U-^{14}C)cyfluthrin	DT_{50}	not calculated
Purity	> 98 %	Metabolites	see results
Test system	aerobic degradation	Acceptability	no valid study

* equivalent to an application rate of 750 g/ha as, assuming a soil bulk density of 1.5 g/cm^3 and even distribution over 5 cm

Study design

The route and rate of degradation of ^{14}C-cyfluthrin was investigated in two soils from the experimental farm Laacherhof. The characteristics of the soils is given in Table B.8.1-2.

Table B.8.1-2: Summary of characteristics of two soils (Wagner et al. 1983)

Soil :	Laacherhof B	Laacherhof C
particle size %		
sand	40	56
silt	37	25
clay	23	19
class	loam	sandy loam
organic carbon	0.95	0.95
moisture content	17 % (batch 1) 11.2 % (batch 2)	13 % (batch 1) 8.6 % (batch 2)
pH (0.01 M $CaCl_2$)	6.2	5.9
cation exchange capacity meq/100g	14	14
particle density g/mL	2.6	2.6

Samples of 50 g of the moist soils were weighed into 125 mL Erlenmeyer flasks. The test substance was applied at a rate of 1 mg/kg soil, equivalent to an application rate of 750 g/ha as, assuming a soil bulk density of 1.5 g/cm^3 and even destribution over 5 cm. As test substance (fluorobenzenering-^{14}C)cyfluthrin was used. The test was carried out in a greenhous at temperatures of 18 – 22 °C, the Erlenmeyer flasks being wrapped with aluminium foil. The moisture contents of the native soils were maintained during test duration; evaporated water was replaced at 14-day intervals (batch 1) or every 2 – 3 months (batch 2) by weighing back to determine the difference. The $^{14}CO_2$ developing during the test was absorbed in two wash bottles. No absorption vessel for volatile compounds other than CO_2 were used as no volatile compounds were detectable in preliminary tests.

Batch 1 of soil B and soil C respectively was sampled at five sampling dates, beginning 14 days after application to 190 days after application. Batch 2 of soil B and soil C respectively was sampled at three sampling dates, beginning 118 days after application to 365 days after application. For analysis of the test substance and metabolites the soil was extracted twice with 400 mL and 160 mL methanol and twice with 150 mL water; first ultrasonicated and than for 8 hours in a Soxhlet apparatus. Finally the soil was extracted with 5 % formic acid. The extracts were concentrated by evaporation and the residues were dissolved in methanol an analysed by thin-layer chromatography.

Results

The distribution of the ^{14}C radioactivity in two soil is given in Table B.8.1-3 and the distribution of cyfluthrin and its transformation products is given in Table B.8.1-4.

Table B.8.1-3: Distribution of the ^{14}C-radioactivity under aerobic test conditions (% initial activity)

Test duration days	$^{14}CO_2$		^{14}C from methanol extraction		^{14}C from water extraction		Rest activity in soil after combustion		Total ^{14}C activity	
	soil B	soil C	soil B	soil C	soil B	soil C	soil B	soil C	soil B	soil C
14	6	5	88	87	8	7	-	-	102	99
28	17	6	63	76	5	9	17	14	102	105
56	25	21	53	49	3	3	18	21	99	94
84	20	23	33	38	6	7	34	27	93	95
118	3	1	61	71	33	28	3	2	100	102
190	32	36	16	22	2	2	32	31	82	91
265	4	2	30	46	38	43	8	9	80	100
365	18	18	21	27	3	5	29	33	71	83

Table B.8.1-4: Distribution of cyfluthrin and its transformation products (% of the initial activity)

Compound	Soil	Days after treatment							
		batch 1					batch 2		
		14	**28**	**56**	**84**	**190**	**118**	**256**	**365**
cyfluthrin	B	86	60	51	33	15	23	18	16
	C	84	69	47	36	22	30	21	18
$CONH_2$-cyfluthrin	B	1	<1	<1	2	1	15	7	3
	C	1	4	<1	<1	1	22	11	6
COOH-cyfluthrin	B	<1	<1	<1	<1	<1	7	3	<1
	C	<1	<1	<1	<1	<1	7	4	1
$CONH_2$FPB acid	B	<1	<1	<1	<1	<1	14	7	<1
	C	<1	<1	<1	<1	<1	5	9	2
FPB acid	B	7	5	3	2	1	29	18	3
	C	7	10	3	5	<1	31	25	4
unknown	B	0	<1	<2	<2	<2	6	15	<2
	C	<2	0	<2	<4	<1	2	19	1
loss	B	0	0	1	7	18	0	20	29
	C	1	0	6	5	9	0	0	17

Comment

As stated in the monograph 01 October 1996, the results of both studies demonstrate that the major degradative route of cyfluthrin is hydrolysis at the ester linkage, leading to the formation of FPB-acid and DCVA. A minor degradative route is hydroxylation of the cyano group to form amid-cyfluthrin ($CONH_2$-cyfluthrin), which in turn could be hydrolysed at the ester linkage.

It should be mentioned that the two batches of soil with different moisture contents should be treated separately as given in Table B.8.1-4.

Two **new laboratory soil degradation studies** were conducted according OECD guidline 307 using beta-cyfluthrin radio-labelled in the cyclopropyl- and in the fluorophenyl-moiety (Hiler 2013 a, b).

B.8.1.1.1.1/3 (Hiler 2013a)

Reference	Hiler, Tammy	Incubation time	121 days
Title	Aerobic soil metabolism of [cyclopropyl-2-^{14}C]beta-cyfluthrin in three soils	3 Soils	sandy loam, sandy clay loam, silt loam
Year of execution	07.10.2013	Nominal concentration	0.12 mg/kg dry soil *
Study no.	2365W-1		
GLP statement	yes	Temperature	20 ± 2 °C
Guideline	OECD 307 US EPA OPPTS835.4100/835.4200 (October 2008) Canadian Guidance PMRA DACO8.2.3.4.2	Moisture	pF 2 – 2.5
Test substance	(cyclopropyl-1-^{14}C)beta-cyfluthrin	DT_{50}	not calculated
Purity	> 98 %	Metabolites	see results
Test system	aerobic degradation	Acceptability	accepted

* equivalent to an application rate of 900 g/ha as, assuming a soil bulk density of 1.5 g/cm^3 and even distribution over 5 cm

Executive Summary

An aerobic soil metabolism study was conducted using [cyclopropyl-1-^{14}C]beta-cyfluthrin applied to three agricultural soils collected in the USA. Soil samples were incubated over 121 days in the dark at 20 ± 2 °C. Individual samples were treated with the test substance at a target rate of 0.12 µg/g dry soil.

The average mass balance for each of the three field soils across all time points in the study ranged from 96.1 to 98.9 % of applied radioactivity (AR). The average extractable radiocarbon at time 0 ranged from 95.1 to 102.1 % AR in all soils and declined to average 5.4 to 20.3 % AR by the end of the incubation period. Unextracted (bound) soil residues increased to an average of 33.9 - 47.2 % AR at the end of the incubation. Final characterisation of the bound residues in the post-extraction soil was conducted on representative samples by partitioning the organic matter into humic acid, fulvic acid and humin fractions. For all soils most of the unextracted radioactivity partitioned into the insoluble humin fraction, with smaller amounts in the fulvic acid fraction and the least in the humic acid fraction.

Radiocarbon in the NaOH traps was confirmed as $^{14}CO_2$ by precipitation as barium carbonate and increased to maximum averages of 39.0 % - 40.7 % at 121 days.

Parent beta-cyfluthrin and the metabolite DCVA were the major components detected in the soil extracts by HPLC throughout the study. Beta-cyfluthrin declined from a maximum average of 102.1 % AR at time 0 to average 17.5 % - 4.3 % AR at day 121. DCVA increased to maximum averages of 11.2 - 40.5 % AR after 3 - 7 days. DCVA then declined and represented less than 0.5 % AR at the end of the study (day 121). Minor degradate peaks detected in the HPLC radiochromatograms of the soil extracts were less than 5 %, except for the day 14 interval in North Dakota soil (6.2 %).

No isomerisation of beta-cyfluthrin was detected during the study. The only isomers representing greater than 2.2 % of dose were isomers II and IV, which were present in a ratio of approximately 2:3, respectively, at time 0. Isomer IV degraded more quickly in aerobic soil, decreasing from between 56.9- 62.8 % AR at time 0 to between 4.7-11.7 % AR after 30 days of incubation. During this interval, isomer II degraded from between 36.7-39.3 % AR to between 12.0-25.0 % AR. At the end of the incubation period (121° days), isomers II and IV represented between 4.2-15.2 % and 1.1-2.8 % AR, respectively.

Material and Methods

Test material (radiolabelled)
Identification: [cyclopropyl-1-^{14}C]beta-cyfluthrin,
Applicant Remark: In the report title and entire report the name given was [cyclopropyl-2-^{14}C]beteta-cyfluthrin which is not correct. An amendment will be prepared.
Specific Activity: 3.27 MBq/mg
Radiochemical Purity: > 98 % (HPLC and TLC)
Chemical Purity: > 98 % (HPLC)
Stability of test compound: Prior to application, the radiopurity of the test substance was determined by HPLC with radioactive flow-through detection.
The stability of the test substance under conditions of administration was confirmed upon HPLC analysis of the dose solution following application.

Reference material (non-radiolabelled)
Identification: beta-cyfluthrin
Expiry Date: 30 December 2013

Reference material (non-radiolabelled)
Identification: DCVA (permethric acid)

Soils

Three different soils were used for the study (see Table B.8.1-5) The soils were passed through a 2 mm sieve prior to use to ensure uniform particle size. Soils were stored refrigerated at approx. 4 °C for two to four weeks until use. The moisture content of the soils was determined by oven drying soil aliquots and comparing their weight before and after drying.

Table B.8.1-5: Summary of characteristics of three soils (Hiler 2013a)

Soil:	Madera, California, USA	Grand Forks, North Dakota, USA	Carlyle, Illinois, USA
sample depth cm	top 20	top 15	top 15 - 20
collection date	19.09.2012	14.09.2012	25.09.2012
particle size %			
sand	63	58	15
silt	22	14	60
clay	15	28	25
texture (USDA)	sandy loam	sandy clay loam	silt loam
organic matter	0.89	5.9	1.8
organic carbon (org.matter/1.724)	0.52	3.42	1.04
moisture content % w/w 0.1 bar (2.0 pF) 0.33 bar (2.5 pF)	 14.6 7.7	 37.1 24.6	 30.9 21.6
pH (water)	7.6	5.3	6.0
pH (0.01 M $CaCl_2$)	7.1	4.9	5.5
cation exchange capacity meq/100g	9.9	19.0	11.6
bulk density g/mL	1.19	0.94	1.03
microbial biomass (µg C/g soil) 25.09.2012 28.09.2012 15.10.2012 01.04.2013	 202 - 253 149	 353 - 517 251	 - 200 325 141

Study design

Experimental conditions

The study was performed using amber glass bottles. The test systems were maintained in the dark at a temperature of 20 ± 2 °C in a constant temperature room. A continuous flow of humidified air was maintained through the samples during the study. At approximately two-week intervals, the bottles were weighed to assay any water loss from the soil and the water was replaced as needed to maintain the moisture level midway pF values of 2.0 and 2.5 over the course of the study. The target dose rate for the study was 0.12 ppm (µg test substance / g dry soil). Soils were treated with the test item dissolved in methanol. Biomass determination test systems were either performed with untreated soil (at soil collection) or with soil dosed with test item-free application solvent.

After application, each sample bottle was connected to an individual series of traps containing at least one ethylene glycol (EG), one sulfuric acid trap, and two caustic traps (10 % aqueous NaOH), via Teflon® tubing. Air entering the samples was passed through de-ionised water before flushing the headspace of the soil samples and through the traps to collect volatile metabolites. The test systems were incubated under aerobic conditions for 121 days.

Sampling

Duplicate test flasks were collected for analysis immediately after treatment and after 3, 7, 14, 30, 59, 91, and 121 days of incubation, along with their respective traps. Samples were extracted on the day of collection. Extracts were stored frozen (< -10 °C) when not in use. The initial HPLC analysis for each soil sample was conducted within nine days of collection. Solutions of the reference standards prepared for analysis were stored frozen when not in use. No changes in the HPLC/UV analysis of the reference standards were observed during the course of the study.

Description of analytical procedures

After collection of the respective test systems from the incubation chamber, flask and volatile traps were separated. The soil was extracted three times at room temperature with 100 mL aqueous organic solvent (acetone:0.1 M HCl, 9:1, v/v) containing some of the extraction solvent used to aid the transfer of soil and, from day 3 onwards, further by a soxhlet extraction with 125 mL of acetonitrile:1 N HCl (4:1, v/v). The post extraction soil samples were weighed, and the unextracted radioactivity remaining in the soil was determined by combustion analysis (four aliquots of approximately 0.1-0.5 g each, depending on the time point) followed by LSC.

In the 121-day samples, the radioactivity bound to the soil after the initial extraction (acetone/0.1 N HCl, 9/1) was characterised by humic acids/fulvic acids partitioning. [^{14}C]beta-cyfluthrin and its degradates in the combined extracts of the three ambient extraction steps were analysed and quantitated using HPLC with radioactivity and UV-detection. Confirmation of the peak assignment for beta-cyfluthrin and metabolites was performed by two-dimensional TLC analysis of selected samples. The presence of dissolved $^{14}CO_2$ in the caustic traps of selected samples (day 121 replicates A and B) was confirmed by the precipitation with $BaCl_2$.

The LOD was 0.25 % AR. The LOQ, considered was 0.38 % AR. Biomass determinations were performed using the substrate induced respiration method (production rate of CO_2 after glucose addition) as defined by Anderson and Domsch (1978).

Results and discussion

The data of the analytical process are given in Table B.8.1-6 to Table B.8.1-8.

Table B.8.1-6: Metabolism of [cyclopropyl-1-^{14}C]beta-cyfluthrin in the North Dakota soil (% applied radioactivity AR)

Incubation days:		**0**	**3**	**7**	**14**	**30**	**59**	**91**	**121**
beta-cyfluthrin	A	102.8	91.6	70.4	55.0	37.3	28.4	20.6	18.0
	B	101.4	91.1	75.4	53.6	36.8	27.1	18.7	17.9
	mean	**102.1**	**91.4**	**72.9**	**54.3**	**37.1**	**27.8**	**19.7**	**18.0**
DCVA	A	0.0	9.8	15.7	13.6	7.3	3.0	1.4	0.0
	B	0.0	10.3	11.7	12.9	4.2	2.3	0.0	0.0
	mean	**0.0**	**10.1**	**13.7**	**13.3**	**5.8**	**2.7**	**0.7**	**0.0**
others **	A	0.0	0.0	4.1	7.4	5.7	1.3	2.6	2.6
	B	0.0	0.0	3.6	4.9	2.0	3.0	3.4	3.2
	mean	**0.0**	**0.0**	**3.9**	**6.2**	**3.9**	**2.2**	**3.0**	**2.9**
soil extract	A	102.8	101.4	90.1	75.9	50.3	32.7	24.6	20.6
	B	101.4	101.4	90.7	71.5	43.0	32.4	22.1	21.1
	mean	**102.1**	**101.4**	**90.4**	**73.7**	**46.7**	**32.6**	**23.4**	**20.9**
Soxhlet extract	A	NA	1.9	3.5	3.5	4.2	0.0 *	2.3 *	2.3 *
	B	NA	0.0	2.7	3.7	2.9	0.0 *	2.8 *	1.1 *
	mean	**NA**	**1.0**	**3.1**	**3.6**	**3.6**	**0.0**	**2.6**	**1.7**
bound residues	A	1.1	2.4	4.9	9.4	18.0	27.5	26.9	33.9
	B	0.8	1.8	4.7	10.3	20.3	30.1	31.5	33.9
	mean	**1.0**	**2.1**	**4.8**	**9.9**	**19.2**	**28.8**	**29.2**	**33.9**

ethylene glycol trap	A	NA	0.1	0.1	0.1	0.1	0.1	0.0	0.0
	B	NA	0.0	0.1	0.1	0.1	0.0	0.0	0.2
	mean	**NA**	**0.1**	**0.1**	**0.1**	**0.1**	**0.1**	**0.0**	**0.1**
sulfuric acid trap	A	NA	0.0	0.0	0.0	0.1	0.5	0.9	0.0
	B	NA	0.0	0.0	0.0	0.0	0.7	1.1	0.0
	mean	**NA**	**0.0**	**0.0**	**0.0**	**0.1**	**0.6**	**1.0**	**0.0**
NaOH trap (CO2)	A	NA	0.4	3.2	10.3	21.3	25.7	41.1	45.0
	B	NA	0.4	3.0	11.6	24.8	33.6	40.8	45.1
	mean	**NA**	**0.4**	**3.1**	**11.0**	**23.1**	**34.7**	**41.0**	**45.1**
total recovery	A	103.9	106.2	101.8	99.2	94.0	96.5	93.5	99.5
	B	102.2	103.6	101.2	97.2	91.1	96.8	95.5	100.3
	mean	**103.1**	**104.9**	**101.5**	**98.2**	**92.6**	**96.7**	**94.5**	**99.9**

NA: “not applicable”, no data
* Determination of bound residues conducted before Soxhlet extraction, therefore values not used for total recovery
** an unknown metabolite with retentions time 22.8 minutes in reversed phase HPLC comprises 7.4 % AR on day 14 and 5.7 % AR on day 30 only in replicate A; in replicate B the metabolite comprises 4.9 % and 2.0 % AR respectively

Table B.8.1-7: Metabolism of [cyclopropyl-1-^{14}C]beta-cyfluthrin in the California soil (% AR)

Incubation days:		**0**	**3**	**7**	**14**	**30**	**59**	**91**	**121**
beta-cyfluthrin	A	97.2	67.6	41.7	26.9	17.1	11.0	9.6	5.3
	B	97.0	64.8	47.1	30.4	19.3	12.4	9.1	6.6
	mean	**97.1**	**66.2**	**44.4**	**28.7**	**18.2**	**11.7**	**9.4**	**6.0**
DCVA	A	0.0	25.0	42.6	40.4	7.2	0.5	0.5	0.7
	B	0.0	25.6	38.3	36.4	8.7	1.0	0.7	0.0
	mean	**0.0**	**25.3**	**40.5**	**38.4**	**8.0**	**0.8**	**0.6**	**0.4**
others	A	0.0	0.0	0.0	0.0	0.9	1.2	1.7	0.7
	B	0.0	1.3	0.0	0.0	0.0	0.6	0.0	0.8
	mean	**0.0**	**0.7**	**0.0**	**0.0**	**0.5**	**0.9**	**0.9**	**0.8**
soil extract	A	97.2	92.6	84.3	67.3	25.2	12.7	11.8	6.7
	B	97.0	91.6	85.4	66.8	28.0	14.0	9.8	7.4
	mean	**97.1**	**92.1**	**84.9**	**67.1**	**26.6**	**13.4**	**10.8**	**7.1**
Soxhlet extract	A	NA	0.0	2.3	2.6	2.1	1.7 *	1.4 *	2.6 *
	B	NA	0.0	2.3	1.6	5.6	0.0 *	2.5 *	1.4 *
	mean	**NA**	**0.0**	**2.3**	**2.1**	**3.9**	**0.9**	**2.0**	**2.0**
bound residues	A	0.5	2.9	7.2	15.3	25.8	41.4	40.2	41.6
	B	0.6	3.3	7.5	16.0	31.6	43.4	51.2	52.7
	mean	**0.6**	**3.1**	**7.4**	**15.7**	**33.7**	**42.4**	**45.7**	**47.2**
ethylene glycol trap	A	NA	0.3	0.5	0.7	0.5	0.3	0.2	0.0
	B	NA	0.3	0.3	0.7	0.6	0.2	0.2	0.1
	mean	**NA**	**0.3**	**0.4**	**0.7**	**0.6**	**0.3**	**0.2**	**0.1**
sulfuric acid trap	A	NA	0.1	0.0	0.0	0.0	0.0	1.2	0.1
	B	NA	0.0	0.0	0.0	0.5	1.7	0.5	0.0
	mean	**NA**	**0.1**	**0.0**	**0.0**	**0.3**	**0.9**	**0.9**	**0.1**
NaOH trap (CO2)	A	NA	1.3	3.3	10.0	27.0	40.9	39.9	45.5
	B	NA	1.0	3.2	10.0	25.4	37.2	43.2	45.6
	mean	**NA**	**1.2**	**3.3**	**10.0**	**26.2**	**39.1**	**41.4**	**45.6**
total recovery	A	97.7	97.7	97.6	95.5	90.6	95.3	93.3	93.9
	B	97.6	96.2	98.7	95.1	91.7	96.5	104.9	105.8
	mean	**97.7**	**96.7**	**98.2**	**95.5**	**91.2**	**95.9**	**99.1**	**99.9**

NA: “not applicable”, no data
* Determination of bound residues conducted before Soxhlet extraction, therefore values not used for total recovery

Table B.8.1-8: Metabolism of [cyclopropyl-1-^{14}C]beta-cyfluthrin in the Illinois soil (% AR)

Incubation days:		**0**	**3**	**7**	**14**	**30**	**59**	**91**	**121**
beta-cyfluthrin	A	94.7	69.9	48.1	40.5	18.4	10.5	6.6	5.7
	B	95.4	74.4	52.2	35.8	21.6	9.6	7.8	4.7
	mean	**95.1**	**72.2**	**50.2**	**38.2**	**20.0**	**10.1**	**7.2**	**5.2**
DCVA	A	0.0	11.8	9.3	3.9	0.7	0.0	0.0	0.0
	B	0.0	10.6	9.2	4.9	1.8	0.0	0.0	0.0
	mean	**0.0**	**11.2**	**9.3**	**4.4**	**1.3**	**0.0**	**0.0**	**0.0**
others	A	0.0	3.7	3.5	0.0	1.9	1.1	1.2	1.4
	B	0.0	0.0	3.0	2.7	1.8	1.6	1.6	1.7
	mean	**0.0**	**1.9**	**3.3**	**1.4**	**1.9**	**1.4**	**1.4**	**1.6**
soil extract	A	94.7	85.3	61.0	44.4	21.0	11.6	7.8	7.1
	B	95.4	85.0	64.5	43.5	25.3	11.2	9.4	6.4
	mean	**95.1**	**85.2**	**62.8**	**44.0**	**23.2**	**11.4**	**8.6**	**6.8**
Soxhlet extract	A	NA	0.0	1.6	2.2	1.7	0.0 *	1.6 *	1.9 *
	B	NA	1.4	2.7	3.3	1.7	2.5 *	1.7 *	0.9 *
	mean	**NA**	**0.7**	**2.2**	**2.8**	**1.7**	**1.3**	**1.7**	**1.4**
bound residues	A	0.6	9.2	23.0	32.4	37.9	47.9	43.3	44.0
	B	0.6	7.7	22.1	32.1	35.3	53.6	37.6	44.7
	mean	**0.6**	**8.5**	**22.6**	**32.3**	**36.6**	**50.8**	**40.5**	**44.4**
ethylene glycol trap	A	NA	0.1	0.3	0.3	0.2	0.1	0.1	0.1
	B	NA	0.1	0.3	0.2	0.3	0.2	0.0	0.0
	mean	**NA**	**0.1**	**0.3**	**0.3**	**0.3**	**0.2**	**0.1**	**0.1**
sulfuric acid trap	A	NA	0.0	0.0	0.0	0.1	0.0	0.2	0.0
	B	NA	0.0	0.0	0.0	0.5	0.0	0.0	0.0
	mean	**NA**	**0.0**	**0.0**	**0.0**	**0.3**	**0.0**	**0.1**	**0.0**
NaOH trap (CO2)	A	NA	3.0	9.2	15.6	28.5	39.6	43.9	51.1
	B	NA	1.8	8.4	16.3	27.0	39.4	44.3	46.6
	mean	**NA**	**2.4**	**8.8**	**16.0**	**27.8**	**39.5**	**44.1**	**48.9**
total recovery	A	95.3	97.6	95.1	94.9	89.4	99.2	95.3	102.3
	B	96.0	96.0	98.0	95.4	90.1	104.4	91.3	97.7
	mean	**95.7**	**96.8**	**96.6**	**95.2**	**89.8**	**101.8**	**93.3**	**100.0**

NA: “not applicable”, no data
* Determination of bound residues conducted before Soxhlet extraction, therefore values not used for total recovery

Mass balance

Total mass balance in individual replicates was 89.4 – 105.8 % AR in all three soils. The complete material balance found at all sampling intervals demonstrated that no significant portion of radioactivity dissipated from the flasks or was lost during processing.

Bound residues

Soil bound residues increased to a maximum average of 33.9 % - 44.4 % AR at the end of incubation period. Humic/fulvic partition was performed on 121-day soil samples to further characterise the non-extractable residues in soil. The results of the partition showed that the majority of the radiocarbon was present in the insoluble humin fraction, i.e. between 25.1 % and 30.8 % AR was recovered in the fraction. Only 7.0 % AR to 11.7 % AR was found in the fulvic acid fraction and < 5 % AR in the humic acid fraction.

Volatilisation

The aerobic degradation of beta-cyfluthrin produced $^{14}CO_2$, which increased to an average of 45.1 - 48.9 % AR at the end of the incubation period. Negligible radiocarbon (less than ≤ 0.7 % AR) was detected in the ethylene glycol traps and ≤ 0.9 % AR was detected in the sulphuric acid traps.

Transformation of parent compound

During the 121-day incubation period, the amount of beta-cyfluthrin in the entire test systems decreased rapidly from 95.1 % - 102.1 % of the AR to 5.2 % - 18.0 % of the AR. The main metabolite DCVA was detected in all three soils. The maximum was observed in California soil, it increased to 40.5 % AR after 7 days of incubation and decreased then to 0.4 % AR towards the end of the study. Other minor unidentified peaks were detected in the HPLC chromatograms, but no single degradate peak exceeded 5 % AR, except for a single detection on day 14 (6.2 % AR, average of two replicates).

Conclusions

Beta-cyfluthrin degraded rapidly in three soils and represented 5.2 - 18.0 % AR at the end of incubation. The major degradates were $^{14}CO_2$ and the metabolite DCVA (maximum 40.5 % AR).

In the Illinois soil in one of two replicates (on day 30) the recovery was 89.4 % which is slightly below the range of 90 – 110 % that should be covered according to OECD 307. The organic carbon content in the North Dacota soil was 3.4 % which is above the range of 0.5 – 2.5 % recommended for determination of transformation pathways. For determination of transformation rate, however, the three soils represent a good variation in pH, organic carbon content and microbial biomass.

The study is acceptable.

B.8.1.1.1/4 (Hiler 2013b)

Reference	Hiler, Tammy	Incubation time	124 days
Title	Aerobic soil metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin	Soil	sandy loam
Year of execution	02.10.2013	Nominal concentration	0.127 mg/kg dry soil *
Study no.	2326W-1		
GLP statement	yes	Temperature	20 ± 2 °C
Guideline	OECD 307 (April 2002) US EPA, OCSPP835.4100/835.4200 (October 2008) Canadian guidance PmrA DACO8.2.3.4.2	Moisture	pF 2 – 2.5
Test substance	(cyclopropyl-1-^{14}C)beta-cyfluthrin	DT_{50}	see
Purity	> 99 %	Metabolites	see results
Test system	aerobic degradation	Acceptability	accepted

* equivalent to an application rate of 950 g/ha as, assuming a soil bulk density of 1.5 g/cm^3 and even distribution over 5 cm

Executive Summary

The present laboratory study investigated the route and rate of degradation of [fluorophenyl-^{14}C]beta-cyfluthrin in one soil under aerobic conditions. Soil samples were incubated over 124 days in the dark at about 20 ± 2 °C using a sandy loam soil originating from Madera, California.

Following application of the test item to soil, the samples were incubated under aerobic conditions in the dark at about 20 ± 2 °C and a soil moisture approximately midway between pF of 2.0 and 2.5 for 124 days. Radioactivity in the extracts was analysed by liquid scintillation counting (LSC). Beta-cyfluthrin and its degradates were quantified by high performance liquid chromatography (HPLC) with co-injection of appropriate analytical reference standards. The HPLC peak assignments were confirmed by thin layer chromatography (TLC). Additionally, soil extracts were analysed by normal-phase HPLC to determine if any isomerisation occurred during the incubation period. The residual radioactivity in the soil was determined by combustion with subsequent radioassay by LSC.

Total mass balance averaged 96.1 ± 5.0 % applied radioactivity (AR). Soil bound residues increased throughout the aerobic incubation from 0.6 % AR at time 0 to a maximum of 36.9 % AR after 31 days of incubation. Afterwards, the amounts slightly declined. Amounts of $^{14}CO_2$ trapped in sodium hydroxide increased throughout the study and represented 51.6 % AR at the end of the incubation period.

Beta-cyfluthrin in the ambient extracts declined from an average of 100.0 % AR at time 0 to 7.3 % AR at the end of the 124 day incubation period. One major degradate, FPB acid, was detected in the HPLC analysis of the soil extracts(maximum average of 12.7 % AR after 7 days of incubation). Other minor unknown degradates were detected throughout the study, however, all individual peaks were found to be < 2 % AR.

No isomerisation of beta-cyfluthrin was detected during the study. The only isomers representing more than 8.8 % of dose were isomers II and IV, which were present in a ratio of approximately 2:3, respectively, at time 0. Isomer IV degraded more quickly in aerobic soil, decreasing from between 60.2 - 61.0 % AR at time 0 to between 4.9 to 6.8 % AR after 31 days of incubation. During this same interval, isomer II degraded from between 36.2 - 37.4 % AR to between 16.0 - 19.7 % AR. At the end of the incubation (124 days), isomers II and IV represented 5.7 and 1.6 % AR, respectively. Isomer I represented a mean of 4.8 % AR (day 7) or less. Isomer III represented a mean of 2.5 % AR or less in all intervals.

Materials and Methods

Test material (radiolabelled)
Identification: [fluorophenyl-^{14}C]beta-cyfluthrin
Lot/Batch #: KML 9422
Specific Activity: 4.36 MBq/mg
Radiochemical Purity: > 99 % (HPLC and TLC)
Chemical Purity: 99 % (HPLC, UV detector)

Test material (non-radiolabelled)
Identification: beta-cyfluthrin
Sample ID: 2326W-011 Lot #: 1230200301
Expiry Date: 30 December 2013

Reference material (non-radiolabelled)
Identification: 4-fluoro-3-phenoxybenzoic acid
Sample ID: 2326W-003 Lot #: 0125200502
Expiry Date: 23 March 2016

Reference material (non-radiolabelled)
Identification: 4-hydroxy FPB-acid
Sample ID: 2326W-006 Lot #: 0126201107
Expiry Date: 12 November 2015

Soil

A sandy loam soil was used for the study (see Table B.8.1-9) The soil was passed through a 2 mm sieve prior to use to ensure uniform particle size. The soil was stored refrigerated for approximately 3 weeks prior to use. The moisture content of the soils was determined by oven drying soil aliquots and comparing their weight before and after drying.

Table B.8.1-9: Summary of characteristics of test soil used by Hiler 2013b

Soil:	Madera, California, USA
sample depth cm	top 15
collection date	21.08.2012
particle size %	
sand	67
silt	18
clay	15
texture (USDA)	sandy loam
organic matter	1.1
organic carbon (org.matter/1.724)	0.64
moisture content % w/w 0.1 bar (2.0 pF) 0.33 bar (2.5 pF)	 15.7 10.6
pH (water)	7.2
pH (0.01 M $CaCl_2$)	6.9
cation exchange capacity meq/100g	10.2
bulk density g/mL	1.21
microbial biomass (µg C/g soil) begin of study: 24.08.2012 28.09.2012 end of study: 15.10.2012	 271 284 197

Study design

Experimental conditions

The study was performed using amber glass bottles. During incubation, the test systems were maintained in the dark at a temperature of 20 ± 2 °C in a constant temperature room. Soil moisture was maintained at approximately midway between pF 2.0 and 2.5 over the course of the study. A sandy loam soil (pH in 0.01 M $CaCl_2$ 6.9, 0.6 % organic carbon) was chosen for the study. The soil was passed through a 2.0 mm sieve prior to use in the study to ensure uniform particle size. The soil was stored refrigerated for approximately 3 weeks prior to use. The dose rate for the study was 0.127 ppm (µg test substance/g dry soil). Using a glass 100 µL syringe, aliquots (100µL) of the dose solution were delivered to each soil sample. The flasks were then vortexed to ensure thorough mixing of the treated soil. Then, each sample bottle was connected to an individual series of traps containing at least one ethylene glycol, one sulfuric acid (0.01 M), and two caustic traps (10 % aqueous (2.78 M) NaOH), via Teflon tubing (except for time 0 samples which were immediately worked up). Air entering the samples was passed through deionised water before flushing the headspace of the soil samples and through the traps to collect volatile metabolites. The test systems were incubated under aerobic conditions for 129 days. Biomass determination test systems were either left untreated (untreated soil) or dosed with test item-free application solvent (application solvent controls) and incubated for up to 129 days.

Sampling

Duplicate samples were collected for analysis immediately after treatment and after 3, 7, 14, 31, 60, 94 and 124 days of incubation along with their respective traps. Samples were extracted on the day of

collection. Extracts were stored frozen (< -10 °C) when not in use. The initial HPLC analysis for each soil sample was conducted within four days of collection. Solutions of the reference standards prepared for analysis were stored frozen when not in use.

Description of analytical procedures

After collection of the respective test systems from the incubation chamber, the samples were extracted three times with acetone: 0.1 N hydrochloric acid (9:1). The radioactivity in soil extracts was quantified by liquid scintillation counting (LSC). Following extraction, an aliquot of the post-extracted soil underwent Soxhlet extraction with acetonitrile: 1N hydrochloric acid (4:1), except for time 0. The Soxhlet extracts were quantified by LSC. The residual radioactivity in the soil was determined by combustion with subsequent radioassay by LSC (either after or before Soxhlet extraction). Bound radiocarbon was characterised by partitioning the humic and fulvic acid fractions from selected post-extracted soil samples (days 31 and 124).

[^{14}C]beta-cyfluthrin and its degradates were analysed and quantitated using HPLC with UV and radiodetection. Samples were concentrated before HPLC analysis and the concentrates were acidified and extracted with ethyl acetate. Confirmation of the peak assignment for beta-cyfluthrin and metabolites was performed by two-dimensional TLC analysis of selected samples.
The caustic traps of selected samples (day 124, replicates A and B) were treated with a saturated $BaCl_2$ solution to confirm the presence of dissolved $^{14}CO_2$. Aliquots of the resulting composite solutions were combined with aliquots of aqueous saturated $BaCl_2$ solution and vortexed to mix. The samples were then allowed to precipitate at room temperature overnight prior to centrifugation to separate the layers. Aliquots of the supernatants were radioassayed by LSC and compared to the aliquots before treatment.

The LOD was 0.25 % AR. The LOQ was 0.38 % AR. Biomass determinations were performed using the substrate induced respiration method (production rate of CO2 after glucose addition) as defined by Anderson and Domsch (1978).

Results and Discussion

The study was performed under the required temperature and moisture conditions and the soil was microbially active throughout the study. The data of the analysis are given in Table B.8.1-10.

Table B.8.1-10: Metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin in the California soil (Hiler 2013b) (% AR)

Incubation days:		**0**	**3**	**7**	**14**	**31**	**60**	**94**	**124**
beta-cyfluthrin	A	99.8	83.2	64.4	48.3	27.2	16.7	8.5	7.4
	B	100.1	81.9	63.7	47.8	20.9	14.8	9.6	7.1
	mean	**100.0**	**82.6**	**64.1**	**48.1**	**24.1**	**15.8**	**9.1**	**7.3**
FPB-acid	A	0.0	11.3	11.9	11.6	1.9	1.1	0.6	0.5
	B	0.0	10.3	13.4	8.0	1.6	0.8	0.8	0.3
	mean	**0.0**	**10.8**	**12.7**	**9.8**	**1.8**	**1.0**	**0.7**	**0.4**
4-OH-FPB-acid	A	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	B	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	mean	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**
others	A	0.0	0.0	2.8	0.0	0.0	0.7	0.6	0.4
	B	0.0	0.0	1.0	0.0	0.0	1.0	0.6	0.7
	mean	**0.0**	**0.0**	**1.9**	**0.0**	**0.0**	**0.9**	**0.6**	**0.6**
Soxhlet extract	A	NA	2.5	2.6	2.7	2.5	1.3	1.3	1.5
	B	NA	1.6	3.1	2.1	2.3	0.0	1.5	1.3
	mean	**NA**	**2.1**	**2.9**	**2.4**	**2.4**	**0.7**	**1.4**	**1.4**
bound residues	A	0.6	4.0	11.	19.6	29.5	28.8	33.2 *	31.3 *
	B	0.6	5.4	11.1	20.3	44.2	27.8	33.0 *	32.6 *
	mean	**0.6**	**4.7**	**11.1**	**20.0**	**36.9**	**28.3**	**33.4**	**32.0**

ethylene glycol trap	A	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	B	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	mean	**NA**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**
sulfuric acid trap	A	NA	0.0	0.0	0.0	0.0	0.9	0.6	0.0
	B	NA	0.0	0.0	0.0	0.0	0.0	0.6	0.0
	mean	**NA**	**0.0**	**0.0**	**0.0**	**0.0**	**0.5**	**0.6**	**0.0**
NaOH trap (CO2)	A	NA	1.4	6.9	13.3	33.8	41.9	48.0	51.7
	B	NA	1.8	6.6	15.5	36.3	44.2	46.5	51.4
	mean	**NA**	**1.6**	**6.8**	**14.4**	**35.1**	**43.1**	**47.3**	**51.6**
total recovery	A	100.4	102.4	99.6	95.5	94.9	91.3	91.5	91.3
	B	100.7	101.0	98.9	93.7	105.3	88.6	91.1	92.1
	mean	**100.6**	**101.7**	**99.3**	**94.6**	**100.1**	**90.0**	**91.3**	**91.7**

NA: “not applicable”, no data
* bound residue determination following routine extraction, before Soxhlet extraction

Mass balance

Total mass balance was 88.6 – 105.3 % AR for the individual replicates. The complete material balance found at all sampling intervals (except for day 60, replicate 2) demonstrated that no significant portion of radioactivity dissipated from the flasks or was lost during processing.

Bound residues

Soil bound residues increased throughout the aerobic incubation from 0.6 % AR at time 0 to a maximum of 36.9 % AR after 31 days of incubation. At the end of the study they represented 32.0 % AR prior to Soxhlet extraction.

Volatilisation

The amount of $^{14}CO_2$ was 51.6 % of the AR at the end of the incubation period. Negligible radiocarbon (less than < 0.1 % AR) was detected in the ethylene glycol traps and ≤ 0.6 % AR was detected in the sulphuric acid traps.

Transformation of parent compound

Beta-cyfluthrin declined from an average of 100.0 % AR at time 0 to 7.3 % AR at the end of the 124 day incubation period. One major degradate, FPB-acid was detected in the soil extracts, increasing from below detection limit at time 0 to a maximum of 12.7 % AR after 7 days of incubation, then decreasing to 0.4 % AR at the end of the incubation period. Other minor unidentified peaks in the HPLC were detected, but no single degradate exceeded 2 % AR.

Conclusion

In one of two replicates on day 60 the recovery was 88.6 % which is slightly below the range of 90 – 110 % that should be covered according to OECD 307. The study is acceptable.

The **degradation pathway for beta-cyfluthrin** in soil under aerobic conditions is given in Figure B.8.1-1.

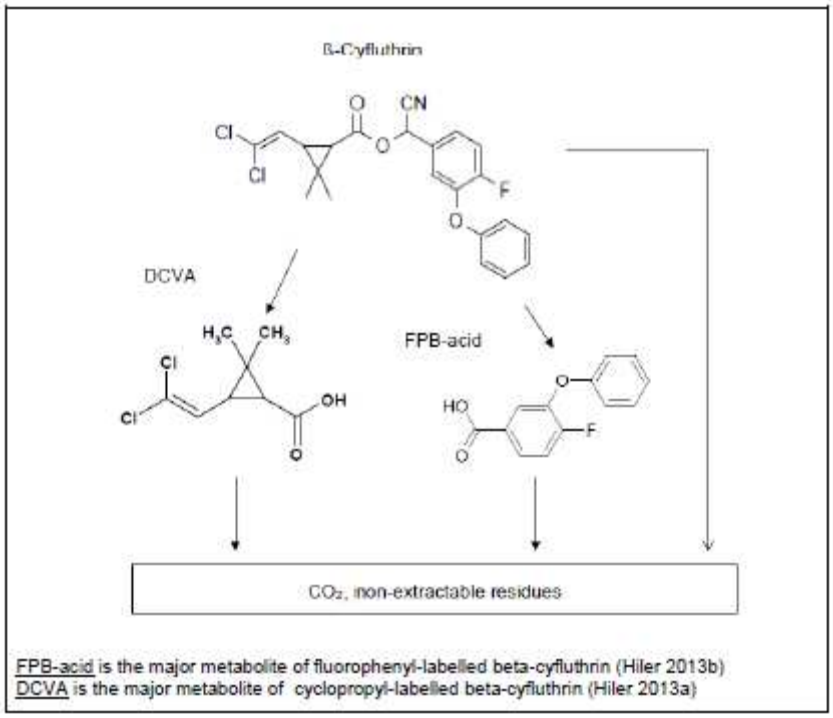


Figure B.8.1-1: Degradation pathway for beta-cyfluthrin in soil under aerobic conditions according the route studies by Hiler 2013 a,b

B.8.1.1.2 Route of degradation in soil - anaerobic laboratory studies

The study by **Wagner et al. 1983** on anaerobic degradation of [fluorobenzenering-U-^{14}C]cyfluthrin was evaluated in the monograph 01 October 1996. FPB-acid was identified as the only metabolite measured in significant amounts (19 % on day 30).

A **new study** was conducted by Hiler 2013c.

B.8.1.1.2/1 (Hiler 2013c)

Reference	Hiler, Tammy	Incubation time	7 days aerobic + 120 days anaerobic
Title	Anaerobic degradation of [fluorophenyl-UL and cyclopropane-1-^{14}C]beta-cyfluthrin	Soil	sandy loam
Year of execution	30.10.2013	Nominal concentration	0.114 – 0.118 mg/kg dry soil *
Study no.	2382W-1		
GLP statement	yes	Temperature	20 ± 2 °C
Guideline	OECD 307 (April 2002) US EPA, OCSPP835.4100/835.4200 (October 2008) Canadian guidance PmrA DACO8.2.3.4.2	Moisture	not relevant

Test substance	[fluorophenyl-UL and cyclopropane-1-^{14}C]beta-cyfluthrin	DT_{50}	route study
Purity	> 99 %	Metabolites	see results
Test system	anerobic degradation in soil	Acceptability	accepted

* equivalent to an application rate of 86 - 88 g/ha as, assuming a soil bulk density of 1.5 g/cm^3 and even distribution over 5 cm

Executive Summary

The present laboratory study investigated the route of degradation of [^{14}C]beta-cyfluthrin in one soil under initially aerobic and then anaerobic flooded conditions. The test was performed in the dark at about 20 °C using static-type incubation test systems. The used soil was a sandy loam. Individual samples were treated with 0.118 and 0.114 ppm of FL or CY labelled [^{14}C]beta-cyfluthrin, respectively. This rate was equivalent to 880 and 860 g/ha as assuming a soil density of 1.5 g cm^{-3} and a depth of 5 cm.

Following application of the test items to soil, the samples were incubated under aerobic conditions in the dark at about 20 °C for seven days (approximately one half-life). Then, the soil samples were flooded with nitrogen-flushed HPLC grade water and then incubated under anaerobic conditions at 20 ± 2 °C for up to 120 days. Soil samples were analysed at time 0 and 7 days for the aerobic phase and soil/water samples were analysed at time 0, 7, 15, 21, 41, 59, 91, and 120 days for the anaerobic phase. Beta-cyfluthrin and its degradates were characterised by reverse phase high performance liquid chromatography (HPLC) and then their identity was confirmed by TLC. Normal phase HPLC was utilised to monitor any parent isomerisation. Liquid scintillation counting (LSC) was used to determine the radioactivity contents in the different compartments.

The average mass balances across all time points in the study were 95.6 ± 3.0 % and 97.0 ± 2.5 % AR (Applied Radiocarbon) for the FL and CY samples, respectively.

Within the aerobic phase of the study [^{14}C]beta-cyfluthrin degraded to ~50 % of dose by day 7 for both radiolabels. Two main metabolites were formed in the aerobic phase of the study (FPB-acid averaged 21.3 % in the FL label and DCVA averaged 29.8 % in the CY label).

[^{14}C]beta-cyfluthrin continued to degrade during the anaerobic (flooded) phase of the study. At the end of the study (day 120), [^{14}C]beta-cyfluthrin represented 8.0 % - 8.8 %. In the FL label, FPB-acid increased from an average of 21.3 % at flooding (T0 of the anaerobic phase) to a maximum average of 63.9 % at day 91 then decreased to average 43.8 % AR by day 120 (end of anaerobic phase). 4-Hydroxy FPB-acid did not exceed 2 % AR at any time during the study. In the CY label, DCVA increased from an average of 33.5 % at flooding (T0 of the anaerobic phase) to a maximum average of 75.7 % at day 120 (end of anaerobic phase). No further metabolites were detected at relevant concentrations (≤ 3.2 % AR).

During the anaerobic phase of the study, the bound residues remained stable for both radiolabels averaging 8.8 - 13.9 % AR by day 120. For both radiolabels, CO_2 recoveries increased to average 6.2 % - 18.5 % AR at day 120. For both radiolabels, the foam plugs and microwave extracts represented negligible amounts of radiocarbon throughout the study.

Material and methods

Test material (radiolabelled)
Identification: [fluorophenyl-UL-^{14}C]beta-cyfluthrin (FL)
Radiochemical Purity: > 99 % (TLC and HPLC)
Chemical Purity: > 99 % (HPLC UV-detector)

Test material (radiolabelled)
Identification: [cyclopropane-1-^{14}C]beta-cyfluthrin (CY)

Specific Activity: 88.44 µCi/mg
Radiochemical Purity: > 98 % (HPLC and TLC)
Chemical Purity: > 98 % (HPLC)

Reference material, (non-radiolabelled)
Description: DCVA (permethric acid)
Expiry Date: 23 October 2018

Reference material, (non-radiolabelled)
Description: 4'-Hydroxy FPB-acid
Expiry Date: 12 November 2015

Reference material, (non-radiolabelled)
Description: FPB-acid
Expiry Date: 23 March 2016

Reference material (non-radiolabelled)
Description: beta-cyfluthrin
Expiry Date: 30 December 2013

Soil

The test soil, a sandy loam soil was collected from Madera, California. Upon receipt, the soil was maintained refrigerated in the dark when not in use. The soil was passed through a 2 mm sieve prior to study set-up. Soil samples were pre-incubated at pF 2-2.5 under aerobic conditions in a controlled temperature chamber maintained at 20 ± 2 °C for 20 days prior to treatment. Soil characteristics see Table B.8.1-11.

Table B.8.1-11: Summary of characteristics of test soil used by Hiler 2013c

soil :	**Madera, California, USA**
sample depth cm	top 15
collection date	27.11.2012
particle size %	
sand	69
silt	17
clay	14
texture (USDA)	sandy loam
organic matter	0.65
organic carbon (org.matter/1.724)	0.38
moisture content % w/w 0.1 bar (2.0 pF) 0.33 bar (2.5 pF)	 19.8 11.2
pH (water)	7.8
cation exchange capacity meq/100g	9.3
bulk density g/mL	1.21
microbial biomass (µg C/g soil) preliminary, untreated: 03.12.2012 end of aerobic phase, untreated13.02.2013	 271 284
anaerobic microbioal analysis colony forming units (per gram) 50 day after flooding, untreated: 26.03.2013 126 days after flooding, untreated: 10.06.2013	 0.108×10^6 0.015×10^6

Study design

Experimental conditions

The target dose rate was 0.12 µg test substance / g dry soil for both labels. This rate is equivalent to 0.95 kg/ha as assuming a soil density of 1.5 g/cm^{-3} and depth of 5 cm.

Samples were immediately capped and vortexed after dosing for thorough exposure of the soil to the test substance. Samples were then stored in the constant temperature room and maintained at 20 ± 2 °C in the dark covered in foil, except for the time 0 samples which were processed immediately after dosing.

Test systems were contained in biometer flasks. A polyurethane foam plug was inserted into the side arm to collect organic volatiles and leading to 40 mL of 10 % aqueous NaOH to trap $^{14}CO_2$. For the aerobic part of the study, a thistle tube was placed in the caustic solution in the sidearm to provide passive air exchange to the system. Following 7 days of aerobic incubation, all remaining samples were flooded with nitrogen-degassed water (70 mL). The water height was 2.75 cm above the soil surface. Before the anaerobic 15 day sampling, an additional NaOH trap was attached with Teflon tubing and stopcocks. The test systems were maintained in the dark at a temperature of 20 ± 2 °C in an incubation chamber.

Sampling

Duplicate test flasks were collected for analysis after aerobic time 0, aerobic day 7, anaerobic days 7, 15, 21, 41, 59, 91 and 120. At each sampling time, duplicate samples for each label were removed from the constant temperature chamber along with their respective traps.

Microbial biomass of the aerobic test system was performed on the soil prior to experimental start and at the end of the aerobic phase (after 7 days). Microbial characterisation of the anaerobic test system was performed 50 days after flooding and at the end of the study (126 days after flooding).

Description of analytical procedures

Pre- and post-flooded soils were extracted three times with 100 mL acetone:0.1 M HCl (9:1, v/v) by shaking for one hour. For samples after aerobic time 0 day, microwave extractions using acetonitrile:water (4:1, v/v) were performed prior to determining residual radioactivity in soils by combustion with subsequent LSC analysis of the evolved $^{14}CO_2$.

A sufficient aliquot of the soil extracts was concentrated via rotary evaporation to just above the aqueous portion and acidified with 6N HCl. The acidified aqueous portion was then extracted with 3 × 15 mL of ethyl acetate. The extracts were combined, rotary evaporated to just dryness, and reconstituted in 2 mL of acetonitrile. The total volumes were measured and aliquots (3 x 1 mL) were taken for radioassay by LSC.

For samples after aerobic time 0 day, microwave extractions using acetonitrile:water (4:1, v/v) were performed prior to determining residual radioactivity in soils by combustions. The water layers were extracted with 2 x 60 mL ethyl acetate. Extracts were combined and concentrated via rotary evaporation to just dryness and reconstituted in 2 mL of acetonitrile. The final volumes were measured and aliquots (3 x 20 µL) were taken for radioassay by LSC. Starting with the anaerobic day 15 sampling, 10 mL of the water layers were transferred to glass vials containing 1 mL of acetonitrile and kept for barium chloride treatment if necessary.

The radioactivity trapped in the PU-foam plugs was extracted with 20 mL of acetonitrile by soaking for at least 4 h and the extracted radioactivity was determined by LSC. Aliquots of the NaOH traps were quenched with 3 mL of methanol and then analysed for radioactivity by LSC. To prepare samples for normal-phase HPLC analysis, the concentrated extracts of soil and water samples were concentrated to

dryness by nitrogen evaporation and reconstituted in HPLC grade hexane. All soil extracts were microfuged prior to analysis. DCVA co-eluted with beta-cyfluthrin in normal phase analysis necessitating the isolation of beta-cyfluthrin (via reverse-phase HPLC) prior to normal-phase HPLC analysis. One rep from the anaerobic day 7 (~50 % degradation of parent) and anaerobic day 91 (~90 % degradation of parent) concentrated soil extracts (CY label only) was chosen for isolation. Water layer extracts were not analysed as beta-cyfluthrin was not present in significant amounts in the corresponding water layers. The time 0 samples were not isolated for normal-phase analysis as no degradation had yet occurred. Confirmation of the peak assignment for beta-cyfluthrin and metabolites was performed by twodimensional TLC analysis of select samples. For further characterisation of bound residues, the post microwave extracted solids of day 120 were selected (both labels). The radioactivity bound to the soil after the microwave extraction was characterised by humic acids/fulvic acids partitioning. The final volumes were recorded and the two fractions were radioassayed by LSC in triplicate.

Limits of detection and quantification for radioassay were determined by the aliquot size and the liquid scintillation counting detection limit. The limits of detection (LOD) and quantification (LOQ) for individual metabolites observed in the HPLC radio-chromatograms were determined by the dpm injected and the liquid scintillation counting detection limit. For the flow-through detector, the typical background was about 50 dpm. The LOD, calculated as 2 x background (100 dpm) was 0.25 % AR. The LOQ, considered to be 3 x typical background (150 dpm), was 0.38 % AR.

The soil was tested for biomass using the substrate induced respiration (SIR) method as defined by Anderson and Domsch (1978). Anaerobic bacteria were determined by culturing under anaerobic conditions.

Results and discussion

The study was performed under the required temperature conditions with a temperature of 20 ± 2 °C (except for five instances where the temperature minimally deviated). The dissolved oxygen content of the systems ranged from 0.19 to 3.62 ppm (FL) and 0.17 to 3.99 ppm (CY) throughout the study. The oxidation/reduction potential (ORP) for the water layer ranged from -45 to +131 mV (FL) and -62 to +124 mV (CY). The pH values for the FL label ranged from 6.34 (anaerobic day 21) to 8.97 (anaerobic day 91) for the water layer and 6.26 (anaerobic day 21) to 8.75 (anaerobic day 91) for the soil layer. The pH values the CY label ranged from 7.08 (anaerobic day 15) to 8.86 (anaerobic day 91) for the water layer and 7.01 (anaerobic day 15) and 8.65 (anaerobic day 120) for the soil layer. The soil was viable throughout the study.

Table B.8.1-12: Metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin in the Madera soil (Hiler 2013c) (% AR)

incubation days:		aerobic		anaerobic							
		0	7	0	7	15	21	41	59	91	120
beta-cyfluthrin	A	98.5	48.8	54.3	40.9	32.33	28.8	19.0	15.7	12.0	8.4
	B	97.1	47.6	53.1	40.9	32.1	29.8	19.0	15.1	10.4	9.2
	mean	**97.8**	**48.2**	**53.7**	**40.9**	**32.2**	**29.3**	**19.0**	**15.4**	**11.2**	**8.8**
FPB-acid	A	0.0	20.4	22.4	33.8	43.8	44.5	49.9	54.7	64.9	29.2
	B	0.0	22.1	20.2	35.0	42.6	44.2	50.8	55.0	62.9	58.3
	mean	**0.0**	**21.3**	**21.3**	**34.4**	**43.2**	**44.4**	**50.4**	**54.9**	**63.9**	**43.8**
4-OH-FPB-acid	A	0.0	0.0	0.0	0.0	0.0	0.0	1.0	1.8	0.0	0.0
	B	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.2	0.0	2.6
	mean	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.5**	**1.5**	**0.0**	**1.3**
others *	A	0.0	0.0	0.0	1.9	0.0	3.1	1.8	2.2	0.0	9.1
	B	0.0	0.0	1.5	1.4	0.0	2.0	1.2	2.0	1.4	0.9
	mean	**0.0**	**0.0**	**0.8**	**1.7**	**0.0**	**2.6**	**1.5**	**2.1**	**0.7**	**5.0**
microwave extract	A	NA	0.6	0.4	0.6	0.5	0.7	0.7	0.8	0.7	0.5
	B	NA	0.4	0.5	0.6	0.6	0.7	0.6	0.7	0.8	0.9
	mean	**NA**	**0.5**	**0.5**	**0.6**	**0.6**	**0.7**	**0.7**	**0.8**	**0.8**	**0.7**

bound residues	A	0.4	13.7	12.9	13.4	15.1	13.5	11.7	10.3	6.3	15.4
	B	0.4	15.5	13.6	14.4	14.2	14.4	13.0	11.4	11.0	12.4
	mean	**0.4**	**14.6**	**13.3**	**13.9**	**14.7**	**14.0**	**12.4**	**10.9**	**8.7**	**13.9**
CO_2	A	NA	6.4	6.3	7.1	7.8	7.6	9.3	8.9	7.9	24.6
	B	NA	6.6	6.8	7.3	8.4	8.9	9.9	9.5	8.3	12.4
	mean	**NA**	**6.5**	**6.6**	**7.2**	**8.1**	**8.3**	**9.6**	**9.2**	**8.1**	**18.5**
foam plug	A	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	B	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.0
	mean	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.1**	**0.0**	**0.0**

NA: “not applicable”, no data

* several peaks ≤ 3.2 % AR present in the HPLC chromatogramm which do not co-eluate with reference standards (e.g. 9.1 % AR in replicate A on day 120 consists of 4 peaks)

Table B.8.1-13: Metabolism of [cyclopropane-1--^{14}C]beta-cyfluthrin in the Madera soil (Hiler 2013c) (% AR)

incubation days:		**aerobic**		**anaerobic**							
		0	**7**	**0**	**7**	**15**	**21**	**41**	**59**	**91**	**120**
beta-cyfluthrin	A	96.0	49.0	50.8	37.6	33.5	27.6	17.8	16.0	9.4	7.3
	B	97.9	50.1	46.3	37.5	37.6	28.5	19.7	12.6	11.9	8.7
	mean	**97.0**	**49.6**	**48.6**	**37.6**	**35.6**	**28.1**	**18.8**	**14.3**	**10.7**	**8.0**
DCVA	A	0.0	30.4	33.5	47.1	56.6	58.8	64.5	67.5	74.7	76.9
	B	0.0	29.2	33.4	45.5	50.5	57.4	66.1	69.2	73.7	74.4
	mean	**0.0**	**29.8**	**33.5**	**46.3**	**53.6**	**58.1**	**65.3**	**68.4**	**74.2**	**75.7**
others *	A	1.9	0.0	0.0	0.6	0.0	0.0	0.6	0.0	0.0	0.5
	B	0.0	0.0	1.0	0.6	0.0	0.0	0.4	2.1	0.0	0.0
	mean	**1.0**	**0.0**	**0.5**	**0.6**	**0.0**	**0.0**	**0.5**	**1.1**	**0.0**	**0.3**
microwave extract	A	NA	0.4	0.0	0.6	0.4	0.4	0.3	0.4	0.3	0.0
	B	NA	0.4	0.3	0.5	0.5	0.4	0.3	0.0	0.2	0.2
	mean	**NA**	**0.4**	**0.2**	**0.6**	**0.5**	**0.4**	**0.3**	**0.2**	**0.3**	**0.1**
bound residues	A	0.5	8.8	8.6	8.0	7.6	8.1	6.7	4.4	2.8	7.2
	B	0.5	9.1	9.2	8.3	7.7	7.4	6.0	6.2	6.0	10.4
	mean	**0.5**	**9.0**	**8.9**	**8.2**	**7.7**	**7.8**	**6.4**	**5.3**	**4.4**	**8.8**
CO_2	A	NA	3.2	3.3	3.4	3.8	3.7	4.7	4.6	4.7	5.6
	B	NA	3.1	3.5	3.7	2.9	4.3	4.3	4.6	4.7	6.7
	mean	**NA**	**3.2**	**3.4**	**3.6**	**3.4**	**4.0**	**4.5**	**4.6**	**4.7**	**6.2**
foam plug	A	NA	1.0	0.2	0.4	0.6	0.3	0.4	0.4	0.2	0.2
	B	NA	1.3	0.7	0.3	0.4	0.4	0.3	0.5	0.2	0.3
	mean	**NA**	**1.2**	**0.5**	**0.4**	**0.5**	**0.4**	**0.4**	**0.5**	**0.2**	**0.3**

NA: “not applicable”, no data

* several peaks ≤ 2.1 % AR present in the HPLC chromatogramm which do not co-eluate with reference standards

Mass balance

Material balance was based on the sum of radioactivity in water layers, soil extracts, microwave extracts, unextracted (bound) soil residues and trapped volatiles (NaOH traps and foam plugs). The average mass balance across all time points in the study was 95.6 ± 3.0 % and 97.0 ± 2.5 % AR for the FL and CY labels, respectively.

Bound and extractable residues

The average extractable radiocarbon at time 0 (based on three extracts) ranged from 97.8 to 97.9 % AR. After the aerobic time 0 sampling, a microwave extraction was added after the routine soil extraction and was performed at each sampling throughout the rest of the study. These microwave extractions resulted in extraction of up to an additional average of 0.6 - 0.8 % AR.

Bound residues increased from averages of 0.4 % - 0.5 % AR at aerobic time 0 to 9.0 - 14.6 % AR during anaerobic incubation. For the FL label, bound residues remained fairly stable, starting and ending at averages of 13.3 % and 13.9 % AR, respectively, and reaching an average low of 8.7 % AR at anaerobic day 91. For the CY label, bound residues also were fairly stable, starting and ending at averages of 8.9 % and 8.8 %, respectively, and reaching an average low of 4.4 % AR at anaerobic day 91.

Volatilisation

During the aerobic phase the maximum amount of $^{14}CO_2$ was 6.5 % of the AR for the FL label and 3.2 % of the AR for the CY label. By the end of the anaerobic incubation, the sodium hydroxide traps contained an average of 18.5 % AR (FL) and 6.2 % AR (CY). The radiocarbon in the sodium hydroxide traps was confirmed as $^{14}CO_2$ by precipitation of barium carbonate.

Transformation of parent compound

Within the aerobic phase of the study (7 days) the amount of beta-cyfluthrin in the entire test systems decreased rapidly from 97.8 / 97.0 % of the AR to 48.2 / 49.6 % of the AR for the FL and CY labels, respectively. During the following anaerobic incubation period (i.e. flooded state) a further decrease was observed to about 8.8 / 8.0 % AR until the end of the study (label FL /CY).

For the FL label, FPB-acid was the only major degradate observed starting at an average of 21.3 % (aerobic day 7 and anaerobic time 0), increasing to 63.9 % (anaerobic day 91) and then declining to 43.8 % AR by the end of the anaerobic incubation period. For the CY label, the only major degradate during the aerobic incubation period was DCVA which represented an average of 29.8 % AR. During the anaerobic phase, the amounts increased from an average of 33.5 % (anaerobic time 0) to 75.7 % AR by anaerobic day 120. For the FL label, 4-Hydroxy FPB-acid was detected in the anaerobic phase of the study (beginning at day 41) and did not exceed <3 % AR at any time during the study. No further metabolites were detected at relevant concentrations (≤ 3.2 % AR).

No isomerisation of beta-cyfluthrin was detected in both labels during the study. Isomers II and IV were consistently the dominant isomers for both labels. Isomer IV degraded most extensively, decreasing from an average of 15.9 % AR at anaerobic day 7 to an average of 2.3 % AR after 91 days of incubation in the FL Label. For the CY label the average was 13.6 % AR at anaerobic day 7 and decreased to an average of 1.4 % AR after 91 days of anaerobic incubation. During this same interval, isomer II degraded from an average of 22.8 % AR to an average of 8.3 % for the FL label. For the CY label, isomer II represented 20.7 % AR at anaerobic day 7 and decreased to 6.6 % AR after 91 days of anaerobic incubation. Isomers 1 and 3 were present in both labels by anaerobic day 7 and persisted to anaerobic day 91. Isomers 1 and 3 individually represented less than 2 % AR.

B.8.1.1.3 Route of degradation in soil - soil photolysis

The study by **Puhl et al. 1983** and **Chopade et al. 1986** on photolytic degradation of cyfluthrin on thin layers of sandy loamy soils were evaluated in the monograph 01 October 1996. FPB-aldehyd was identified as the main metabolite (18 % AR on day 7).

A **new study** was performed by Adam 2013.

B.8.1.1.3/1 (Adam 2013)

Reference	Adam, D.	Incubation time	18 days
Title	[^{14}C] beta-cyfluthrin – photodegradation on soil surface; Identification: 20120108	Soil	silt loam

Date of execution	15.03.2013	Nominal concentration	0.114 – 0.118 mg/kg dry soil *
Study no.	20120108		
GLP statement	yes	Temperature	19 ± 1.4 °C
Guideline	OECD 307 (April 2002) US EPA, OCSPP835.4100/835.4200 (October 2008) Canadian guidance PmrA DACO8.2.3.4.2	Moisture	pF 2
Test substance	[fluorophenyl-UL ^{14}C]beta-cyfluthrin	DT_{50}	see results
Purity	> 99 %	Metabolites	see results
Test system	soil photolysis	Acceptability	accepted

* equivalent to an application rate of 86 - 88 g ai/ha, assuming a soil bulk density of 1.5 g/cm^3 and even distribution over 5 cm

Executive Summary

The effect of light on the route and rate of degradation of beta-cyfluthrin on soil was investigated under artificial sunlight at 19.4 ± 1.4 °C for 18 days. For this purpose, [fluorophenyl-UL-^{14}C]beta-cyfluthrin was applied on moist soil (pF 2). The soil surface (2 mm thickness of soil layer) was uniformly treated at a dose rate of 4.99 µg cm^{-2} of soil corresponding to 0.04 kg/ha.

Besides the test item beta-cyfluthrin one major and up to three minor degradates were observed. The major degradate was identified as 4-fluoro-3-phenoxybenzoic acid (FPB-acid) and occurred with up to 22.3 % of applied radioactivity (% AR) in irradiated samples and with up to 11.2 % AR in in the dark controls. One of the minor degradates was identified as 4-fluoro-3-phenoxybenzaldehyde (FPB aldehyde) by co-chromatography while the remaining two minor degradates were not investigated further nor identified. For FPB-acid a dark soil DT_{50} of 2.2 days was calculated.

The soil photolysis study of beta-cyfluthrin in a silt loam soil resulted in a DT_{50} value of 22.6 days (based on SFO kinetics) under artificial light, corresponding to a DT_{50} of about 41.8 days under natural summer sunlight at 30 to 50°N. The dark soil DT_{50} was calculated to be 18.6 days.

Material and methods

Test material (radiolabelled)
Identification [Fluorophenyl-UL-^{14}C]beta-cyfluthrin
Radiochemical Purity: > 99 % (sum of isomers as stated in Certificate of Analysis of test item); 100 % (sum of isomers as determined by HPLC prior to application).

Reference material (non-radiolabelled)
Identification: Beta-cyfluthrin
Purity: 99.3 %
Expiry date: 26 June 2014

Reference material (non-radiolabelled)
Identification: FPB-ald
Purity: 97 %
Expiry date: 16 October 2013

Reference material (non-radiolabelled)
Identification: FCR1272-1-Diastereomer (Isomer I)
Purity: 99.2 %
Expiry date: 5 December 2014

Reference material (non-radiolabelled)
Identification: FCR1272-1-Diastereomer (Isomer II)
Purity: 99.3 %
Expiry date: 5 December 2014

Reference material (non-radiolabelled)
Identification: FCR1272-1-Diastereomer (Isomer III)
Purity: 99.8 %
Expiry date: 6 December 2014

Reference material (non-radiolabelled)
Identification: FCR1272-1-Diastereomer (Isomer IV)
Purity: 99.2 %
Expiry date: 25 June 2015

Reference material (non-radiolabelled)
Identification: FCR2728
Purity: 73 %
Expiry date: 6 August 2019

Reference material (non-radiolabelled)
Identification: FPB-acid
Purity: 98.8 %
Expiry date: 6 August 2016

Reference material (non-radiolabelled)
Identification: FCR1272-Hydroxyfluorophenoxybenzoic acid
Purity: 96.1 %
Expiry date: 12 November 2015

Soil

A silt loam soil was used in the study, see Table B.8.1-14. Prior to use in the study the soil was stored at 4 °C and passes through a 2 mm sieve.

Table B.8.1-14: Summary of characteristics of test soil used by Adam 2013

soil	Grimmitschau, Germany
sample depth cm	top 20
collection date	29.03.2012
particle size %	
sand (50 µm – 2 mm)	69
silt (50 - 2 µm)	17
clay (< 2 µm)	14
texture (USDA)	silt loam
organic matter (oc ×1.724)	2.67
organic carbon	1.55
moisture at pF 2 (% w/w)	31.7
pH (0.01 M $CaCl_2$)	6.7
cation exchange capacity meq/100g	17.04

Study design

Experimental conditions

Soil thin-layers of 12.5 cm^2 each were prepared by applying aqueous slurry of soil to a glass plate in a way that a layer of 2 mm thickness was formed. Thereafter, the layers were allowed to dry and adjusted to a soil moisture of pF 2. The soil moisture was adjusted daily. An application solution was prepared by dissolving an aliquot of [^{14}C]beta-cyfluthrin in 5 mL of acetonitrile. After ultrasonic treatment, the radioactivity content was determined after preparing two dilutions of 0.01 mL in 10 mL of acetonitrile and measuring triplicate aliquots of 0.1 mL by LSC (liquid scintillation counting). Each soil sample was treated with the test substance by evenly distributing 100 µL of the application solution i.e. 2 µL droplets over the whole soil surface. Prior to, during and after application, the same volume of application solution was applied to three separate control solutions containing 10 mL of acetonitrile/water. The radioactivity present in the control solutions was determined by LSC and was taken as 100 % applied value. Consequently, 5.0 µg of test item was applied to a surface area of 12.5 cm². This amount corresponded to a field rate of 0.04 kg/ha (i.e. 0.399 µg cm^{-2}). The samples were continuously irradiated under artificial sunlight (average light intensity: 46.5 W/m^2 (between 290 and 400 nm) and the temperature was kept at a mean of 19.4 ± 1.4 °C in the exposure tank. Control samples were incubated in the dark at a mean temperature of 21.2 ± 0.1 °C. The temperature was controlled and recorded every 10 minutes during the entire exposure period. Moistened air was drawn through the tanks of irradiated and dark samples at a flow rate of about 5 mL/min. To trap the volatiles, the outlet air was passed through two glass bottles containing 2 N NaOH and ethylene glycol (about 50 mL each). The irradiation time in the laboratory was converted to natural summer sunlight at latitudes 30 to 50°N by comparing the intensity of the artificial light with the reference value of 67 W/m^2 as quoted in the OECD guideline no. 316.

Sampling

Duplicate samples were taken and analysed after 0, 1, 4, 7, 11, and 18 days of continuous irradiation. Dark control samples were taken at the same time intervals. Additionally, two unexposed samples were analysed at the start of the incubation (day 0). At all sampling intervals volatile trapping solutions were removed for analysis and replaced by fresh solutions.

Description of analytical procedures

The soil layer of each sample was quantitatively removed from the glass plate and the soil was extracted up to three times by shaking the sample with 10 mL of acetonitrile/water (4/1; v/v). The extracts were then pooled and the recovery of radioactivity was controlled for every extraction by liquid scintillation counting (LSC). The quantity of radioactivity was determined by Packard liquid scintillation counters. All measurements were performed in duplicate, corrected for background, and counted for a time interval of one minute.

The extracts were submitted to HPLC analysis with radiodetection (primary chromatographic method). The limit of quantification (LOQ) of the primary chromatographic method was set at a level at which a distinct peak could be seen and integrated. For this purpose, different amounts of [^{14}C]beta-cyfluthrin were injected onto the HPLC column. The LOQ was determined to be about 1.4 % of the applied radioactivity, the limit of detection (LOD) was determined to be half these amounts (0.7 % AR). TLC analysis with UV and radiodetection was performed on selected samples to confirm the results. For the investigation of the beta-cyfluthrin isomers by two chiral HPLC methods, the extracts wereconcentrated to dryness and re-dissolved in hexane.

The residual radioactivity remaining in soil after extraction was determined by LSC analysis of the $^{14}CO_2$ evolved after combustion of homogenised soil aliquots (0.6 g in triplicate) in an automated sample oxidizer. The radioactivity in the trapping solutions was determined by LSC. In order to confirm the presence of $^{14}CO_2$, the radioactivity in the sodium hydroxide traps was precipitated with barium hydroxide in samples irradiated for 18 days.

Results and Discussion

The results of analysis of metabolism of irradiated and control soil are given in Table B.8.1-15 and Table B.8.1-16 respectively.

Table B.8.1-15: Metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin in the Grimmitschau irradiated soil (Adam 2013) (% AR)

Incubation days:		0	1	4	7	11	18
beta-cyfluthrin	A	101.8	99.8	86.4	78.4	73.2	64.5
	B	101.3	99.3	91.2	77.8	74.3	53.4
	mean	**101.5**	**99.5**	**88.8**	**78.1**	**73.8**	**59.0**
FPB-acid	A	< LOD	1.9	10.1	13.7	14.5	16.6
	B	< LOD	2.2	9.3	14.4	18.1	27.9
	mean	< LOD	**2.1**	**9.7**	**14.1**	**16.3**	**22.3**
FPB-aldehyd	A	< LOD	1.3	2.7	1.6	< LOD	< LOD
	B	< LOD	1.4	1.1	1.2	< LOD	< LOD
	mean		**1.3**	**1.9**	**1.4**		
others *	A	< LOD	< LOD	1.7	2.5	< LOD	2.2
	B	< LOD	< LOD	< LOD	1.8	< LOD	2.0
	mean			**0.9**	**2.2**		**2.1**
extractables	A	101.8	102.9	100.9	96.2	87.7	83.3
	B	101.3	102.9	101.6	95.3	92.4	83.4
	mean	**101.5**	**102.9**	**101.3**	**95.8**	**90.1**	**83.3**
non-extractables	A	0.4	1.8	4.8	4.9	13.1	16.6
	B	0.3	1.9	3.8	13.5	14.9	15.4
	mean	**0.3**	**1.8**	**4.3**	**9.2**	**14.0**	**16.0**
$^{14}CO_2$	A	NA	< 0.1	0.2	0.3	0.9	1.8
	B	NA	< 0.1	0.2	0.3	0.9	1.8
	mean	**NA**	**< 0.1**	**0.2**	**0.3**	**0.9**	**1.8**
total recovery	A	102.2	104.7	105.9	101.4	101.7	101.7
	B	101.6	104.9	105.6	109.1	108.3	100.5
	mean	**101.9**	**104.8**	**105.75**	**105.25**	**105**	**101.1**

NA: “not applicable”, no data

Table B.8.1-16: Metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin in the Grimmitschau dark control soil (Adam 2013) (% AR)

Incubation days:		0	1	4	7	11	18
beta-cyfluthrin	A	101.8	103.2	85.8	72.9	66.5	56.4
	B	101.3	104.3	88.3	76.5	67.8	60.8
	mean	**101.5**	**103.7**	**87.0**	**74.7**	**67.1**	**58.6**
FPB-acid	A	< LOD	1.8	11.0	10.7	6.5	5.1
	B	< LOD	1.7	11.2	11.7	7.6	4.7
	mean	< LOD	**1.8**	**11.1**	**11.2**	**7.0**	**4.9**
others	A	< LOD	< LOD	< LOD	1.5	2.7	3.7
	B	< LOD	< LOD	< LOD	< LOD	< LOD	2.1
	mean				**0.7**	**1.4**	**2.9**
extractables	A	101.8	105.0	96.9	85.1	75.5	65.1
	B	101.3	106.0	99.4	88.2	75.3	67.5
	mean	**101.5**	**105.5**	**98.2**	**86.6**	**75.4**	**66.3**
non-extractables	A	0.4	0.9	4.7	12.2	22.4	24.1
	B	0.3	0.9	4.7	11.1	24.6	25.4
	mean	**0.3**	**0.9**	**4.7**	**11.6**	**23.5**	**24.8**
$^{14}CO_2$	A	NA	< 0.1	< 0.1	3.1	4.2	5.4
	B	NA	< 0.1	< 0.1	3.1	4.2	5.4
	mean	**NA**	**< 0.1**	**< 0.1**	**3.1**	**4.2**	**5.4**

total recovery	A	102.2	105.9	101.6	100.3	102.2	94.6
	B	101.6	106.9	104.1	102.4	104.1	98.3
	mean	**101.9**	**106.4**	**102.85**	**101.35**	**103.15**	**96.45**

NA: "not applicable", no data

Mass balance

The total mean recovery of the applied radioactivity (AR) was 100.5 – 109.1 % AR in the two replicates of irradiated samples and 94.6 – 106.9 % in the dark controls.

Bound and extractable residues

Non-extractable radioactivity increased from maximum 0.3 % AR at day 0 to 16 % AR in the irradiated samples. The non-extractable radioactivity in the dark control soil after 18 days of incubation was with 24.8 % AR higher when compared to the results for the irradiated samples.

Volatilisation

In irradiated samples, radioactive carbon dioxide amounted to 1.8 % AR at study termination while carbon dioxide formed by mineralisation in dark control samples represented 5.4 % AR at study end. The amount of organic volatiles was < 0.1 % throughout the study in both, irradiated samples and dark controls.

Transformation of parent compound

In irradiated samples, the mean amount of [^{14}C]beta-cyfluthrin decreased from initially 101.5 % to 59.0 % AR on day 18. Besides the test item one major and up to three minor degradates [^{14}C]beta-cyfluthrin were observed. The major degradate was identified as 4-fluoro-3-phenoxybenzoic acid (FPB-acid) and occurred in irradiated samples as well as in the dark controls (up to 11.2 % AR). One of the minor degradates (up to 1.9 % AR in irradiated samples) was identified as 4-fluoro-3-phenoxybenzaldehyde (FPB-aldehyde) by cochromatography while the remaining two minor degradates were not investigated further nor identified.

Beta-cyfluthrin is a mixture of two isomers, isomers II and IV, each consisting of two enantiomers. Initially, the ratio of isomer II (also called diastereomeric pair II, i.e. [1R, 3R, α S (=1,3-cis) + 1S, 3S, α R (=1,3 cis)] and IV (also called diastereomeric pair IV, i.e. [1R, 3S, α S (=1,3-trans) + 1S, 3R, α R (=1,3-trans)] was approximately 37:63. Over 18 days of incubation/irradiation, isomer IV (representing 31.5 % and 28.3 % AR in irradiated and dark control samples, respectively) degraded faster than isomer II (representing 27.4 % and 30.4 % AR, respectively) or partially interconverted to isomer II. The results showed that irradiation had no significant effect on the degradation of both isomers.

Both isomers were analysed further by chiral HPLC for the ratio of the respective enantiomers they represent. This ratio remained at about 1:1 throughout the study. The results indicated that degradation of the test item depended rather on geometric isomerism (cis/trans isomers) than on stereo isomerism.

The DT_{50} and DT_{90} values were calculated assuming single first-order kinetics (SFO). The results obtained for [^{14}C]beta-cyfluthrin and for the major degradate FPB-acid are summarised in Table B.8.1-17)

Table B.8.1-17: Kinetic data for photolytic degradation in soil

Test substance and condition		Kkinetic model	chi²error	k (d^{-1}) *	M0	Experimental DT_{50}	Converted natural sun-light ** DT_{50}
beta-cyfluthrin	irradiated	SFO	1.7	0.0307	101.2	22.6	41.8
	dark	SFO	5.7	0.0372	103.1	18.6	
FPB-acid	dark	SFO	3.6	0.3206		2.2	

* all p-values in t-test < 0.05

** conversion factor 1.85 = 33 natural days / 18 experimental days; natural days = (h × r) / (0.75 × 12) with h = 18 × 24 h experimental irradiation, r = 46.5 / 67 ratio of experimental and natural sunlight (W m-2) 300 – 400 nm, 0.75 = assuming the daily radiation being 75 % of midday maximum, 12 = conversion from hours to days.

Transformation of degradates

The major degradate was identified as 4-fluoro-3-phenoxybenzoic acid (FPB-acid) and occurred in irradiated samples and in the dark controls. In irradiated samples, FPB-acid was formed continuously reaching a mean amount of 22.3 % after 18 days of irradiation and was not degraded further. In dark controls, the mean amount of FPB-acid increased until day 7 reaching 11.2 % AR and decreased to 4.9 % on day 18, indicating better biodegradation in the dark controls when compared to the irradiated samples. This is the consequence of a decreasing microbial activity in the irradiated samples due to UVirradiation, while it remained constant in the dark controls. This could be concluded from the higher amount of carbon dioxide detected in the dark controls when comparing with irradiated samples. The amount of the remaining degradates (including FPB-ald) did not exceed 2.2 % AR in irradiated samples and 1.1 % AR in dark controls (no FPB-ald detected) at any time throughout the study. The DT_{50} value of FPB acid based on SFO kinetics was 2.2 days in the dark control soil see Table B.8.1-17.

Conclusions

The dissipation of beta-cyfluthrin is very similar in both the irradiated and dark control sample. It can therefore be assumed that photo-transformation is no relevant process for the dissipation of beta-cyfluthrin in soil. No additional photo-metabolite is transformed from parent under radiation. The DT_{50} values given here are seen as additional information specific for this test.

B.8.1.1.4 Route of degradation in soil – metabolite FPB-acid

A new study on the aerobic degradation of the metabolite FPB-acid in soil was performed by Hellpointner & Junge 2013.

B.8.1.1.4/1 (Hellpointner & Junge 2013)

Reference	Hellpointner, E. & Junge, T.	Incubation time	30 days
Title	Beta-Cyfluthrin-FPB-acid: aerobic degradation in three soils – final report.	3 Soils	silt loams and clay loam
Date of execution	15-11-2013	Nominal concentration	0.06 mg/kg dry weight
Study no.	EnSa-13-0668, M1252209-1		
GLP statement	yes	Temperature	20 °C

Guideline	OECD Test Guideline no. 307	Moisture	55 % of maximum water holding capacity
Test substance	(phenyl-UL-^{14}C)-AE F105561 (beta-cyfluthrin-FPB-acid)	DT_{50}	0.9 – 1 day
Purity	> 99 %	Metabolites	not relevant
Test system	aerobic degradation	Acceptability	acceptable as route study

Executive Summary

The route and rate of degradation of FPB-acid was investigated in three European soils under aerobic conditions. The test was performed in the dark at about 20 °C. The used soils were two silt loams and one clay loam. A rate of 60 µg/kg dry soil of FPB-acid was applied. Following application, the samples were incubated under aerobic conditions in the dark at about 20 °C and 55 % of the maximum water holding capacity for 30 days. Traps for volatiles included a soda lime trap for absorption of carbon dioxide and a polyurethane foam plug for adsorption of volatile organic compounds. Duplicate samples were taken on days 0, 0.21, 1, 3, 7, 14 and 30 and extracted. The radioactivity in liquid samples was determined by liquid scintillation counting (LSC). Non-extractable residues were determined by LSC after combustion. The test item was identified by TLC and HPLC-MS/MS. Mean material balances were 94.1 – 96.3 % of applied radioactivity.

The maximum amount of carbon dioxide was 44.0 - 56.1 % AR at study end (day 30). Formation of volatile organic compounds (VOC) was ≤ 0.1 % AR at all sampling intervals for all soils. Non-extractable residues (NER) increased from 4.1 – 8.6 % AR at day 0 to 45.4 - 53.7 % at day 3 and declined to 35.6 - 45.4 % AR until the end of the study after 30 days. The amount of FPB-acid in the soil extracts decreased from day 0 to day 30 from 98.5 – 93.4 % AR to 1.1 – 0.3 % AR (0.3 % AR = LOQ). The total unidentified residues amounted to a maximum of 4.5 % AR and no single component exceeded 1.2 % AR at any sampling interval for all soils.

Materials and Methods

Test material (radiolabelled)
Identification: [phenoxy-UL-^{14}C]AE F105561 (FPB-acid)
Specific Activity: 9.02 MBq/mg
Radiochemical Purity: > 99 % (HPLC, radioactivity detector), > 99 % (TLC, scan)
Chemical Purity: > 99 % (HPLC, UV detector)
Stability of test compound: The application solution for the degradation samples was stable during the application procedure and the radiochemical purity was determined as 99.4 % (TLC).

Reference material (non-radiolabelled)
Identification: FPB-acid
Chemical Purity: 98.8 %

Soils

The study was carried out using three different soils (Table B.8.1-18). The soils were taken from agricultural use areas representing different geographical origin and different soil properties as required by the guidelines. The plant protection product use history of the soils for at least 5 years is known. The soils were sampled freshly from the fields (upper horizon of 0 to 20 cm) and sieved to a particle size of ≤ 2 mm.

Table B.8.1-18: Summary of characteristics of three soils (Hellpointner & Junge 2013)

Soil:	Hanscheider Hof, Burscheid, Germany	Hoefchen am Hohenseh, Burscheid, Germany	Dollendorf, Blankenheim, Germany
sample depth cm	top 20	top 20	top 20
collection date	June 2013	June 2013	June 2013
particle size %			
sand (50 µm – 2 mm)	22	18	28
silt (2 µm – 50 µm)	57	63	41
clay (< 2 µm)	21	19	31
texture (USDA)	silt loam	silt loam	clay loam
organic matter (organic carbon × 1.724)	4.8	3.8	9.5
organic carbon	2.8	2.2	5.5
moisture content % w/w maximum 0.1 bar (pF 2)	 64.8 32.8	 56.1 39.7	 85.2 38.9
pH (water)	5.7	6.5	7.4
pH (0.01 M $CaCl_2$)	5.4	6.2	7.3
cation exchange capacity meq/100g	8.7	11.1	21.3
bulk density g/mL	0.98	1.11	0.93
microbial biomass (mg C/kg soil) at test start at test end	 1124 687	 1255 1030	 3018 2909

Study design

Experimental conditions

The study was performed in glass Erlenmeyer flasks of 300 mL volume. The test systems were maintained in the dark at an average temperature of 19.7 °C in an incubation chamber. Two silt loam soils and one clay loam soil were used. Soil characteristics are summarised in Table B.8.1-18. The soil moisture was adjusted to and maintained at 55 % maximum water holding capacity. A rate of 60 µg per kg soil dry weight was applied. This rate was based on the intended single maximum field rate of parent beta-cyfluthrin implying a possible worst-case soil contamination to be in the range of 0.03 mg/kg soil. Since this was regarded to be a rather low concentration, a two-fold was tested, i.e. 6 µg FPB-acid per 100 g dry soil. Small droplets of the application solution (containing the test item in water). were applied directly onto the soil surface using a pipette. Biomass determination test systems were left untreated. After application, the test systems were fitted with trap attachments containing soda lime and a polyurethane (PU) foam plug as trapping media for carbon dioxide and organic volatile compounds, respectively. Aerobic conditions were maintained by passive diffusion of atmospheric oxygen. The test systems were incubated under aerobic conditions for 30 days.

Sampling

Duplicate test flasks were collected for analysis at 0, 0.21, 1, 3, 7, 14 and 30 days after treatment (DAT). Soil samples were immediately extracted and soil extracts were subjected to a first chromatography profiling usually within one day. Therefore no storage stability experiments were necessary. Soil extracts were stored in a freezer (< -18 °C) in the dark after analysis. Test systems used for microbial biomass determinations were sampled 0 DAT and 30 DAT.

Description of analytical procedures

Volatiles possibly still present in the head space of the test system were purged into the trap attachment. Afterwards, the trap attachment was removed and the soil was extracted completely. The soil was extracted twice at ambient temperature with acetonitrile/water (4/1, v/v) and with two microwave accelerated steps at 70 °C (acetonitrile/water (1/1, v/v) and methanol/water (1/1, v/v). Ambient and microwave extracts were analysed separately for radioactivity by liquid scintillation counting (LSC) and acetonitrile/water and methanol/water extracts were analysed separately by TLC with radiodetection. The radioactivity trapped in the PU-foam plugs was extracted with 30 mL ethyl acetate by sonication and the extracted radioactivity was determined by LSC. Radioactivity absorbed by soda lime (i.e. $^{14}CO_2$) was liberated using 18 % aqueous HCl, trapped in scintillation cocktail and measured by LSC as well. The portion of non-extractable radioactivity in soil was determined by combustion of lyophilised and homogenised soil samples. The evolving ^{14}C-carbon dioxide was trapped in scintillation cocktail and analysed by LSC. TLC analysis was performed on silica gel plates (Merck Si60, F254) using an eluent consisting of ethylacetate/isopropyl/water/acetic acid (80/15/5/0.1; v/v). Radioactive zones were determined using a bio-imaging analyser. The limit of detection (LOD) was deduced from profiles of the combined organic extracts and set to 0.3 % AR. For radiodetection the LOQ is in the range of the LOD, thus at approx. 0.3 % of AR.

Test item identity was confirmd by HPLC hyphenated to electrospray ionisation mass spectrometry in single or multistage mode (ESI-MS(/MS)). The microbial biomass was determined based on the method of substrate-induced initial respiratory response.

Results and Discussion

The study was performed under the required temperature conditions with a mean temperature of 19.7 °C and a mean soil moisture of 54.5 % of the maximum water holding capacity. The soil was microbial viable throughout the study.

The data on metabolism are given in Table B.8.1-19 to Table B.8.1-21.

Mass Balance

The mean material balance was 92.6 – 102.7 % AR. The complete material balance found at the sampling intervals for each soil demonstrated that there was no significant loss of radioactivity from test systems or during processing.

Bound and Extractable Residues

Extractable residues decreased from day 0 to day 30 from maximal 94.1 to minimal 2.7 %. Non-extractable residues (NER) increased during the first three days of incubation in all three soils to maximal 53.7 % AR and declined thereafter towards to end of the study after 30 days to maximal 45.4 % AR.

Volatilisation

The maximum amount of carbon dioxide was 56.1 % AR at study end (30 DAT). Formation of volatile organic compounds (VOC) was ≤ 0.1 % AR at all sampling intervals for all soils.

Transformation of test substance FPB-acid

The amount of FPB-acid in the soil extracts decreased from 0 DAT to 30 DAT from maximal 93.4 % AR to 0.7 - 1.1 % AR in the silt loam soils and to less than LOD (0.3 % AR) in the clay loam soil. The total unidentified residues amounted to a maximum of 4.5 % AR and no single component exceeded 1.2 % AR at any sampling interval for all soils.

Table B.8.1-19: Metabolism of metabolite FPB-acid in the Hanscheider Hof soil (% AR) studied by Hellpointner & Junge 2013

Incubation days:		**0**	**0.21**	**1**	**3**	**7**	**14**	**30**
FPB-acid	A	90.7	79.9	46.3	13.6	3.3	2.0	1.1
	B	90.6	79.5	48.2	13.3	3.4	2.0	1.2
	mean	**90.6**	**79.7**	**47.3**	**13.5**	**3.4**	**2.0**	**1.1**
others *	A	0.3	< LOD	0.4	< LOD	< LOD	< LOD	< LOD
	B	< LOD	0.4	< LOD	< LOD	0.5	< LOD	< LOD
	mean	**< LOD**	**< LOD**	**< LOD**	**< LOD**	**< LOD**	**< LOD**	**< LOD**
CO_2	A	2.2	12.3	25.8	35.3	38.9	44.3	2.2
	B	2.2	11.3	26.5	34	39.1	43.6	2.2
	mean	**2.2**	**11.8**	**26.2**	**34.7**	**39.0**	**44.0**	**2.2**
extractable residues **	A	91.1	80.7	47.9	16.6	6.4	4.9	3.7
	B	90.9	80.5	49.5	16.4	6.7	4.7	3.8
	mean	**91.0**	**80.6**	**48.7**	**16.5**	**6.6**	**4.8**	**3.8**
NER	A	2.7	16.3	36.6	53.8	51.2	49.3	45.5
	B	5.5	16.9	35.7	53.5	51.7	49.7	45.3
	mean	**4.1**	**16.6**	**36.2**	**53.7**	**51.5**	**49.5**	**45.4**
total recovery	A	93.8	99.3	96.7	96.3	92.9	93.1	93.5
	B	96.4	99.6	96.5	96.5	92.4	93.5	92.7
	mean	**95.1**	**99.4**	**96.6**	**96.4**	**92.7**	**93.3**	**93.1**

NER: non extractable residues, bound residues
NA: “not applicable”, no data
LOD: limit of detection = 0.3 % AR
* others = inidentified bands and diffuse residues in thin layer chromatogramms
** total extractable residues = sum of FPB-acid, others as well as clean up losses and chromatographic losses

Table B.8.1-20: Metabolism of metabolite FPB-acid in the Hoefchen soil (% AR) studied by Hellpointner & Junge 2013

Incubation days:		**0**	**0.21**	**1**	**3**	**7**	**14**	**30**
FPB-acid	A	92.7	82.2	46.5	8.6	2.6	1.4	0.8
	B	94.1	83.4	46.5	8.9	2.5	1.5	0.7
	mean	**93.4**	**82.8**	**46.5**	**8.7**	**2.5**	**1.4**	**0.7**
others *	A	0.5	0.4	0.7	0.6	0.3	0.3	< LOD
	B	< LOD	0.6	0.4	0.5	0.3	0.3	0.4
	mean	**< LOD**	**< LOD**	**< LOD**	**0.6**	**0.3**	**0.3**	**< LOD**
CO_2	A	NA	2.0	12.3	29.6	37.1	40.7	47.5
	B	NA	1.6	12.1	29.6	36.5	40.4	46.0
	mean	**NA**	**1.8**	**12.2**	**29.6**	**36.8**	**40.6**	**46.8**
extractable residues **	A	93.6	83.4	48.6	12.3	5.5	3.9	3.0
	B	94.6	84.8	48.3	12.6	5.5	4.0	3.0
	mean	**94.1**	**84.1**	**48.5**	**12.4**	**5.5**	**4.0**	**3.0**
NER	A	5.7	14.4	35.9	52.6	50.4	48.9	43.9
	B	11.5	13.4	36.2	53.4	50.4	48.2	44.8
	mean	**8.6**	**13.9**	**36.0**	**53.0**	**50.4**	**48.5**	**44.4**
total recovery	A	99.3	99.8	96.8	94.5	93.1	93.5	94.4
	B	106.2	99.8	96.6	95.6	92.3	92.6	93.9
	mean	**102.7**	**99.8**	**96.7**	**95.0**	**92.7**	**93.0**	**94.2**

NER : non extractable residues, bound residues
NA: “not applicable”, no data
LOD: limit of detection = 0.3 % AR
* others = inidentified bands and diffuse residues in thin layer chromatogramms
** total extractable residures = sum of FPB-acid, others as well as clean up losses and chromatographic losses

Table B.8.1-21: Metabolism of metabolite FPB-acid in the Dollendorf soil (% AR) studied by Hellpointner & Junge 2013

Incubation days:		0	0.21	1	3	7	14	30
FPB-acid	A	89.8	82.0	48.4	7.7	1.3	0.5	< LOD
	B	89.2	81.2	49.0	8.3	1.2	0.5	< LOD
	mean	**89.5**	**81.6**	**48.7**	**8.0**	**1.2**	**0.5**	**< LOD**
others *	A	0.4	1.7	0.9	0.5	< LOD	< LOD	< LOD
	B	0.3	1.6	1.4	0.4	< LOD	0.3	< LOD
	mean	**0.3**	**1.7**	**1.1**	**0.5**	**< LOD**	**< LOD**	**< LOD**
CO_2	A	NA	1.2	12.1	34.1	46.0	50.5	57.0
	B	NA	1.2	12.0	33.0	46.9	50.0	55.2
	mean	**NA**	**1.2**	**12.1**	**33.6**	**46.5**	**50.3**	**56.1**
extractable residues **	A	90.2	84.7	51.1	12.5	4.9	3.4	2.7
	B	89.6	83.9	51.9	12.7	4.8	3.5	2.7
	mean	**89.9**	**84.3**	**51.5**	**12.5**	**4.8**	**3.5**	**2.7**
NER	A	5.4	12.6	28.9	45.6	42.2	39.8	35.6
	B	5.1	12.9	29.0	45.2	41.3	40.1	35.6
	mean	**5.3**	**12.8**	**28.9**	**45.4**	**41.8**	**39.9**	**35.6**
total recovery	A	95.6	98.5	92.1	92.2	93.2	93.7	95.3
	B	94.7	98.1	92.8	90.9	93.0	93.6	93.6
	mean	**95.2**	**98.3**	**92.5**	**91.6**	**93.1**	**93.7**	**94.5**

NER : non extractable residues, bound residues
NA: "not applicable", no data
LOD: limit of detection = 0.3 % AR
* others = inidentified bands and diffuse residues in thin layer chromatogramms
** total extractable residues = sum of FPB-acid, others as well as clean up losses and chromatographic losses

Comment

FPB-acid was rapidly degraded in soil under aerobic conditions in the dark in the laboratory. Hellpointner & Junge provided a kinetic analysis of the data with DT_{50} in all three soils of 0.9 – 1 days. The kinetic data are not given in detail here. The kinetic recalculation of the data from this study is discussed in chapter B.8.1.2.2 ("Rate of degradation in laboratory studies – metabolites") in the context of the general evaluation of beta-cyfluthrin and its metabolites by Hammel & Porschewski 2013.

The study is accepted as a route study.

B.8.1.2 Rate of degradation

B.8.1.2.1 Rate of degradation in laboratory studies – active substance

In the monograph dated 01 October 1996 three laboratory studies on the rate of aerobic degradation were evaluated (Wagner et al. 1983, Wagner 1983 and Yoshida 1984). All three studies have deficiencies, test substance being cyfluthrin instead of beta-cyfluthrin, restricted information about soil humidity, no GLP statement. They were accepted for calculation of rate of degradation in the monograph from 1996.

In the addendum dated 07 May 2002 to the monograph the study by Riegner 1997 (Aerobic degradation of cyfluthrin in soil at low temperature according to EC requirements) was evaluated as acceptable.

A kinetic re-evaluation according to FOCUS 2006 is conducted by the RMS for three out of these four studies.

In the study by **Wagner et al. 1983** only four data points for batch 1 from day 14 to day 84 are available, day 190 is by far too long for a laboratory study (maximum incubation time according to OECD 307 is

120 days). For batch 2 only three data points from day 118 to day 365 are available. Due to these restrictions a kinetic evaluation seems not appropriate.

In the study by **Wagner 1983** many data points from day = 0 to day = 126 are available for two soils. Therefore the RMS conducted a kinetic evaluation despite of some deficiencies of the study.

B.8.1.2.1/1 (Wagner 1983)

Reference	Wagner, K.	Incubation time	292 - 298 days
Title	Behaviour of pesticides in soil	2 Soils	not characterised
Year of execution	20-05-1983	Nominal concentration	0.5 mg/100g *
Study no.	RR5621/82, M0-01-017034		
GLP statement	no	Temperature	22 °C
Guideline	none	Moisture	not given
Test substance	(cyfluthrin formulated as Baythroid	DT_{50}	calculation by RMS
Purity	not known	Metabolites	rate study
Test system	aerobic degradation in soil	Acceptability	additional information

* equivalent to an application rate of 3.8 kg ai/ha, assuming a soil bulk density of 1.5 g/cm^3 and even distribution over 5 cm

In Table B.8.1-22 the restricted information about the soils and the analysis of the test substance and the data on residues are given. In Table B.8.1-23 the results of kinetic evaluation using KinGUI ver. 2.1 are given.

Table B.8.1-22: Information about soil and analyses from Wagner 1983 and results of kinetic analysis.

	Standard soil 1	**Standard soil 2**
organic carbon	2.64 %	1.06 %
pH	6.0	7.0
particles < 20 µm (medium silt, fine silt and clay)	14.5 %	24.7 %
limit of detection	0.01 mg/kg	0.01 mg/kg
recovery rate	86 %	88 %
residues on day (% applied)		
day 0	86	88
day 7	70	84
day 14	47	59
day 21	56	57
day 28	50	47
day 35	46	46
day 42	45	51
day 49	40	43
day 56	40	35
day 63	39	33
day 70	36	45
day 77	33	33
day 84	41	39
day 91	38	33

day 98	33	36
day 105	28	31
day 126	27	28

Table B.8.1-23: Kinetic evalutation of aerobic degradation in soil (Wagner 1983) by RMS

kinetic model (visual fit)	kinetic parameters	p-value for t-test	Chi^2-error	DT_{50}	DT_{90}
SFO (unacceptable)	M0: 73 k: 0.00943	<0.001	11.8	73	244
FOMC (good)	M0: 88.2 α: 0.406 β: 9.358	no t-test for α and β	6.5	42.3	> 1000
DFOP (good)	**M0: 88.4 k1: 0.0805 k2: 0.0047 g: 0.413**	**0.002 0.001**	**6.4**	**41**	**380**
HS (unacceptable)	M0: 87 k1: 0.084 k2: 0.0078 t_b: 3.6	< 0.001 < 0.001	9.6	54	259

Conclusion

The DFOP kinetic model gives the best fit to the degradation data up to day 126. The overall DT_{50} is 41 days, the DT_{90} 380 days (biphasic DT_{50} fast = 8.6 days, DT_{50} slow = 147 days). The data are not normalised and refer to a temperature of 22 °C and to a unknown soil humidity. The study and the kinetic evaluation are seen as additional information. No further use in modelling is recommended by RMS.

In the study by **Yoshida et al. 1998** eight data points from day = 0 to day = 90 are available for two soils. Despite the deficiencies this study has, the RMS conducted a kinetic evaluation.

B.8.1.2.1/2 (Yoshida et al. 1984)

Reference	Yoshida, H., Yoshimoto, Y. and Takase, I.	Incubation time	90 days
Title	Residual fate of cyfluthrin (fRC1272) in soils under laboratoy and field conditions	2 Soils	loam and silty loam
Year of execution	28-05-1984	Nominal concentration	0.5 mg/kg *
Study no.	1197 (ESR)		
GLP statement	no	Temperature	28 °C
Guideline	none	Moisture	60 % maximum water content
Test substance	(cyfluthrin formulated as Baythroid	DT_{50}	calculated by RMS
Purity	not known	Metabolites	rate study
Test system	aerobic degradation	Acceptability	additional information

* equivalent to an application rate of 0.38 kg ai/ha, assuming a soil bulk density of 1.5 g/cm^3 and even distribution over 5 cm

In Table B.8.1-24 the restricted information about the soils and the analysis of the test substance and the data on residues are given. In Table B.8.1-25 the results of kinetic evaluation using KinGUI ver. 2.1 are given.

Table B.8.1-24: Information about soil and analyses of residues from Yoshida et al. 1984.

	Ibaraki soil	**Nagano soil**
texture	loam	silty loam
organic matter	6.2 %	1.6 %
organic carbon (om / 1.724)	3.6 %	0.9 %
pH	6.3	6.6
clay	10 %	19 %
cation exchange capacity	22.8 meq/100 g	17.0 meq/100 g
temperature	28 °C	28 °C
soil moisture (% maximum water holding capacity)	60 %	60 %
limit of detection	0.02 mg/kg	0.02 mg/kg
recovery rate	92 - 104 %	94 - 104 %
residues on day (% applied)		
day 0	0.48	0.48
day 3	0.27	0.33
day 7	0.16	0.28
day 14	0.12	0.20
day 21	0.09	0.18
day 30	0.06	0.12
day 60	0.04	0.10
day 90	0.04	0.08

Table B.8.1-25: Kinetic evalutation of aerobic degradation in soil (Yoshida et al. 1984) by RMS

kinetic model (visual fit)	**kinetic parameters**	**p-value for t-test**	**Chi²-error**	**DT_{50}**	**DT_{90}**
Ibaraki loam					
SFO (unacceptable)	M0: 0.441 k: 0.112	0.002	20.8	6.2	20.5
FOMC (acceptable)	**M0: 0.481** **α: 0.759** **β: 2.46**	**no t-test for α and β**	**3.9**	**3.7**	**48.7**
DFOP (acceptable)	M0: 0.479 k1: 0.284 k2: 0.0175 g: 0.733	< 0.001 0.013	5.2	3.8	56.3
HS (unacceptable)	M0: 0.48 k1: 0.192 k2: 0.0287 t_b: 6.0	< 0.001 0.008	7.3	3.6	46.2
Nagano silt loam					
SFO (unacceptable)	M0: 0.42 k: 0.041	0.003	16.5	16.9	56
FOMC (good)	**M0: 0.477** **α: 0.559** **β: 3.767**	**no t-test for α and β**	**4.4**	**9.2**	**228**
DFOP (acceptable)	M0: 0.472 k1: 0.480 k2: 0.0116 g: 0.567	0.01 0.03	6.2	9.1	126

HS (unacceptable)	M0: 0.48 k1: 0.125 k2: 0.0194 tb: 5.0	0.02 0.009	9.2	8.4	91

Conclusion

The FOMC kinetic model gives the best fits to the degradation data. The overall DT_{50} under experimental conditions for loam soil is 3.7 day and for silt loam 9.2 days, the DT_{90} is 48.7 day and 228 days, respectively. The data are not normalised and refer to a temperature of 28 °C and to a soil moisture of 60 % MWHC. No further use in modelling is proposed by RMS.

In the study by **Riegner 1997** the aerobic degradation of cyfluthrin n soil at 9 °C was tested. The study was evaluated in the addendum 2002 to be acceptable.

B.8.1.2.1/3 (Riegner 1997)

Reference	Riegner, K.	Incubation time	121 days
Title	Aerobic degradation of cyfluthrin in soil at low termperture according to EC requirements	2 Soils	loam and silty loam
Year of execution	02-06-1997	Nominal concentration	8.9 µg/100g *
Study no.	PF 4241, MR-744/96		
GLP statement	yes	Temperature	9 °C
Guideline	SETAC 1995 Directive 95/36/EC	Moisture	40 % maximum water content corresponds to14 g/100 g
Test substance	[phenyl-UL-^{14}C]cyfluthrin	DT_{50}	calculated by RMS
Purity	> 99 %	Metabolites	< 5 %
Test system	aerobic degradation at low temperature	Acceptability	acceptable

* equivalent to an application rate of 0.68 kg ai/ha, assuming a soil bulk density of 1.5 g/cm^3 and even distribution over 5 cm

The analytical results from Riegner 1997 are summarised in Table B.8.1-26.

Table B.8.1-26: Metabolism of [phenyl-^{14}C]beta-cyfluthrin in the soil (Riegner 1997) (% AR, results of thin layer chromatography)

Incubation days:		**0**	**1**	**3**	**7**	**14**	**30**	**62**	**90**	**121**
cyfluthrin		105.57	97.72	90.42	72.74	63.16	44.26	28.63	25.18	21.81
FPB-acid		0.67	1.18	2.41	3.98	4.93	4.47	2.74	2.32	1.86
unknown		0	0	0.14	0.86	1.43	1.60	0.55	0.36	0.14
others		0.16	0.16	0.27	0.56	1.22	1.50	2.41	2.32	2.29
start		0.02	0.03	0.06	0.16	1.18	1.16	0.93	0.81	0.80
sum of extracted		106.42	99.09	93.29	78.29	71.92	53.00	35.26	30.99	26.90
non extracted residue		2.72	4.27	6.13	11.42	16.53	23.25	29.07	34.54	33.70
CO_2		NA	0.37	1.70	5.33	11.39	22.93	31.10	34.18	39.83
total recovery		**109.1**	**103.7**	**101.1**	**95.0**	**99.8**	**99.2**	**95.4**	**99.7**	**100.4**

NA: “not applicable”, no data

Kinetic evaluation by RMS

The kinetic evaluation of residue data from Riegner 1997 was conducted by RMS. The computer program KinGUI ver. 2.1 was used, non-linear least square method for optimisation. At day = 0 the total recovery (109.1 % AR) was used instead of 105.57 % for cyfluthrin only.

Table B.8.1-27: Kinetic evalutation of aerobic degradation of cyfluthrin in soil at 10 °C (Riegner 1997) by RMS

kinetic model (visual fit)	kinetic parameters	p-value for t-test	Chi^2-error	DT	DT
Laacher Hof					
SFO (unacceptable)	M0: 95.6 k: 0.0198	< 0.001	11	34.	116
FOMC (good)	**M0: 107.2** **α: 0.592** **β: 8.470**	**no t-test for α and β**	**2.**	**18.**	**407**
DFOP (good)	M0: 106 k1: 0.1014 k2: 0.0079 g: 0.51	< 0.001 0.001	3.	18.	20
HS (acceptable)	M0: 106.6 k1: 0.0557 k2: 0.0117 t_b: 9.8	0.002 < 0.001	5.	22.	15

Conclusion

The kinetic model FOMC gives a good fit for a biphasic degradation of cyfluthrin in silt loam under cold conditions (9 °C) and a soil moisture of 40 % MWHC. The DT_{50} is 18.9 days, DT_{90} 407 days. The test substance was cyfluthrin, a mixture of four isomers. Therefore the RMS did not conduct a standardisation to reference values of temperature and soil moisture. No further use in modelling is proposed by RMS.

The kinetic evaluation of the **new studies by Hiler 2013 a+b** on degradation of beta-cyfluthrin in soil are conducted by Hammel & Porschewski 2013.

B.8.1.2.1/4 (Hammel & Porschewski 2013a)

Reference	:	Hammel, Klaus and Porschewski, Ruth
Title	:	Kinetic Evaluation of aerobic metabolsim of beta-cyfluthrin and its metabolites in soil according to FOCUS kinetics
Year of execution	:	26. 11. 2013
GLP statement	:	not relevant
Guideline	:	not relevant; calculation according FOCUS Kinetics (2006)

Test substance	:	beta-cyfluthrin
Test system	:	aerobic degradation in soil under laboratory conditions

Executive Summary

The aerobic degradation of beta-cyfluthrin in soil was kinetically evaluated based on the laboratory studies conducted by Hiler (2013a, 2013b) where beta-cyfluthrin was applied to various agricultural soils.

The kinetic evaluation was conducted following a tiered approach. As basic tier, a conservative simple first order (SFO) DT_{50} values were determined using the models Double-First-Order in Parallel (DFOP) and First-Order Multiple Compartment (FOMC, Gustafson and Holden) following the procedure of kinetic analysis as recommended by FOCUS (2006). SFO DT_{50} values of beta-cyfluthrin at this step ranged from 19.9 to 101.7 days with a geometric mean of 32.2 days.

In the higher tier approach, DFOP was chosen as appropriate kinetic model to describe the kinetic parameters of beta-cyfluthrin. DT_{50} values of beta-cyfluthrin ranged from 3.9 to 8.2 days with a geometric mean of 5.8 days in the fast phase and from 35.2 to 101.7 days with a geometric mean of 57.5 days in the slow phase, respectively.

Material and Methods

The evaluation of the kinetic behaviour of beta-cyfluthrin in soil was based on the study of Hiler (2013a) which was conducted with [cyclopropyl-1-^{14}C]beta-cyfluthrin on three US soils and on the study of Hiler (2013b) conducted with [fluorophenyl-^{14}C]beta-cyfluthrin on the soil Madera (California).

For the kinetic evaluation purpose, the handing of the measured data from the laboratory aerobic soil degradation studies mentioned above, were considered according to the recommendation of FOCUS working group on degradation kinetics (FOCUS, 2006). A LOD of 0.25 % AR was assumed. The experimental residue data were evaluated following the standard procedures recommended by FOCUS (2006) for kinetic modelling. The procedure as recommended by FOCUS (2006) was followed. The DFOP model provides excellent fits not only for the degradation of the parent but considering the whole pathway, including the SFO degradation of the metabolites. Therefore the DFOP model is considered here as appropriate kinetic parameter set. The model fit as well as the statistical evaluation of the results was carried out with the software KinGUI version 2. For the optimisation of the implemented algorithms, Iteratively Reweighted Nonlinear Least Squares (IRLS) was used.

Results and Discussion

Model selection was conducted following the decision scheme defined in FOCUS 2006. The data and criteria to select the appropriate model are given in Table B.8.1-28. All four soils showed clear biphasic behavior. Therefore the visual fit to SFO was poor and this model was not considered acceptable. In the study using the soil North Dakota 10 % of the initially measured concentration was not reached within the experimental period. DFOP was selected because it provided the better fit. For the other three soils 10 % of the initially measured concentration was reached within the experimental period, the FOMC model could be selected. By considering the whole pathway including the SFO degradation of the metabolites, however, the DFOP model for parent degradation was preferred. The kinetic parameters are given in Table B.8.1-29 and Table B.8.1-30

The KinGUI results, whole data sets and graphics, are given in **Appendix 1** to Vol. 3 B.8.

Table B.8.1-28: Criteria to select models for beta-cyfluthrin residues in soil

Soil	Model	chi² (%)	Visual fit	t-test p-value	Selected
North Dakota	SFO	18.0	-	< 0.001	
Illinois		17.8	-	< 0.001	
Madera (Hiler 2013a)		20.7	-	< 0.001	
Madera (Hiler 2013b)		13.2	-	< 0.001	
North Dakota	HS	5.0	+	< 0.001 (k_2)	
Illinois		6.1	o	< 0.001 (k_2)	
Madera (Hiler 2013a)		5.7	+	< 0.001 (k_2)	
Madera (Hiler 2013b)		7.1	o	< 0.001 (k_2)	
North Dakota	FOMC	3.3	+	na	
Illinois		3.1	+	na	trigger + modelling tier 1
Madera (Hiler 2013a)		2.8	+	na	
Madera (Hiler 2013b)		1.6	+	na	
North Dakota	DFOP	2.2	+	< 0.001 (k_2)	trigger + modelling
Illinois		3.9	+	< 0.001 (k_2)	trigger + modelling tier 2
Madera (Hiler 2013a)		1.7	+	< 0.001 (k_2)	
Madera (Hiler 2013b)		1.9	+	< 0.001 (k_2)	

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta

Table B.8.1-29: Kinetic parameters for degradation of beta-cyfluthrin in soil, tier 1 (Hammel & Porschewski 2013a)

Soil	Model	Parameters		DT_{50} fast	DT_{50} slow
North Dakota	DFOP	M0 = 104.3 g = 0.625	k_1 0.0848 k_2 0.00682	8.2	101.7
				DT_{90}	**DT_{90} / 3.32**
Illinois	FOMC	M0 = 97.77	α 1.145 β 10.196	66	19.9
Madera_a (Hiler 2013a)	FOMC	M0 = 98.29	α 0.841 β 4.608	66.7	20.1
Madera_b (Hiler 2013b)	FOMC	M0 = 100.82	α 1.251 β 16.602	87.9	26.5
geomean					**32.2**

Table B.8.1-30: Kinetic parameters for degradation of beta-cyfluthrin in soil, tier 2 (Hammel & Porschewski 2013a)

Soil	Model	Parameters		DT_{50} fast	DT_{50} slow
North Dakota	DFOP	M0 = 104.3 g = 0.625	k_1 0.0848 k_2 0.00682	8.2	101.7
Illinois		M0 = 95.23 g = 0.616	k_1 0.1486 k_2 0.01972	4.7	35.2
Madera_a (Hiler 2013a)		M0 = 97.80 g = 0.740	k_1 0.1786 k_2 0.01176	3.9	59.0
Madera_b (Hiler 2013b)		M0 = 100.2 g = 0.656	k_1 0.0941 k_2 0.0134	7.4	51.6
geomean				**5.8**	**57.5**

Conclusion

The kinetic evaluation in general is acceptable. However, the RMS has concerns at the tiered approach proposed by Hammel & Porschewski. The DFOP and FOMC models give very similar results for kinetic modelling of the parent. The differences between DFOP-SFO and FOMC-SFO for the kinetic modelling of the metabolites are altogether small. The goodness of fit of the whole pathway in the HS-SFO combination is not addressed in the study. In summary, the RMS is of the opinion, that the minor differences on the level of metabolites do not justify two contrasting approaches for further modelling. It is seen problematic and confusing to decide which endpoints, both formation fraction and DT_{50}, in which situation have to be preferred.

The RMS therefore proposes not to follow this tiered approach but to stay on the DFOP-SFO model for North Dakota soil and the FOMC-SFO model for Illinois and Madera soils.

Degradation rate at reference conditions

The studies by Hiler 2013 a+b were conducted at 20 ± 2 °C. Therefore no temperature standardisation is required. The water contents of the soils was maintained in the middle between pF 2.0 and pF 2.5 over the course of the study. A standardisation to reference soil moisture (pF 2.0) is conducted by the RMS. The results are shown in Table B.8.1-31.

Table B.8.1-31: Standardisation of laboratory DT_{50} for beta-cyfluthrin to reference soil moisture by RMS

Soil	Soil type (USDA classification)	Gravimetric water content (experimental) g/100 g	Reference water content at pF 2.0 (g/100 g)	Walker correction factor *	Experimental DT_{50}	Normalised DT_{50}
North Dakota	sandy clay loam	30.9	37.1	0.88	101.7	89.5
Illinois	silt loam	26.3	30.9	0.89	19.9	17.7
Madera_a (Hiler 2013a)	sandy loam	11.2	14.6	0.83	20.1	16.7
Madera_b (Hiler 2013b)	sandy loam	13.2	15.7	0.89	26.5	23.6
geomean					**32.2**	**28.1**

* Walker correction factor f = (experimental moisture / reference moisture)$^{0.7}$

B.8.1.2.2 Rate of degradation in laboratory studies – metabolites

The studies Hellpointner & Junge 2013 on the aerobic degradation of the metabolite FPB-acid and Hiler 2013 a+b on the aerobic degradation of beta-cyfluthrin were kinetically evaluated by Hammel & Porschewski 2013.

B.8.1.2.2/2 (Hammel & Porschewski 2013a)

Reference	:	Hammel, Klaus and Porschewski, Ruth
Title	:	Kinetic Evaluation of aerobic metabolsim of beta-cyfluthrin and its metabolites in soil according to FOCUS kinetics
Year of execution	:	26. 11. 2013
GLP statement	:	not relevant

Guideline	:	not relevant; calculation according FOCUS Kinetics (2006)
Test substance	:	beta-cyfluthrin
Test system	:	aerobic degradation in soil under laboratory conditions

Executive Summary

The kinetic of the aerobic degradation of metabolites DCVA and FPB-acid in soil was evaluated based on the laboratory studies conducted by Hiler (2013a, 2013b) where beta-cyfluthrin was applied to various agricultural soils. Additionally, the kinetic of the degradation of the metabolite FPB-acid in soil was evaluated based on a laboratory study conducted by Hellpointner & Junge (2013) where FPB-acid was applied to three soils.

The kinetic evaluation was conducted following a tiered approach. As basic tier, a conservative simple first order (SFO) DT_{50} values were determined using the models Double-First-Order in Parallel (DFOP) and First-Order Multiple Compartment (FOMC, Gustafson and Holden) following the procedure of kinetic analysis as recommended by FOCUS (2006).

In the higher tier approach, DFOP was chosen as appropriate kinetic model to describe the kinetic parameters of beta-cyfluthrin.

Material and Methods

The evaluation of the kinetic behaviour of the metabolites of beta-cyfluthrin in soil was based on the studies of Hiler (2013a,b). The studies provided data for the metabolite DCVA (Hiler, 2013a) and for the metabolite FPB-acid (Hiler 2013b). Additionally the degradation of FPB-acid was kinetically investigated by using the measured data obtained from the study by Hellpointner & Junge (2013, which was conducted with FPB-acid applied as test substance to the three soils. The model fit as well as the statistical evaluation of the results was carried out with the software KinGUI version 2.

Results and Comment

The data from the study by Hiler 2013 a+b, using beta-cyfluthrin as test substance, double-first-order in parallel (DFOP) and Gustafson and Holden model (FOMC, first-order multi-compartment) were chosen as appropriate kinetic models for the parent beta-cyfluthrin. The metabolites were described by SFO-model. For the North Dakota soil (DFOP-SFO) and the Illinois soil (FOMC-SFO) the SFO-fit for metabolite **DCVA** was good, chi^2-error 6.9 and 2.7, respectively. For the Madera soil the fit was acceptable, chi^2-error 20.6.

The kinetic calculation of the DT_{50} for **FPB-acid** from the study by Hellpointner & Junge 2013 (FPB-acid as applied as test substance) are not discussed in detail. The kinetic model SFO is appropriate for all the soils, chi^2-errors are between 3.5 and 3.7 and the DT_{50} is in all three soils $\leq$ 1 day. The data from the study by Hiler 2013 b, using beta-cyfluthrin as test substance, FOMC was chosen as appropriate kinetic model for the parent beta-cyfluthrin. The metabolite FPB-acid was described by SFO-model, the fit was good, chi^2-error 9.3.

The results (DT_{50} and formation fractions) are given in Table B.8.1-32.

The details of KinGUI calculations for the data from Table B.8.1-32 are given in **Appendix 1** to Vol. 3 B.8.

Table B.8.1-32: Kinetic parameters for degradation of metabolites in soil (tier 1)

Soil	Study	DCVA		FPB-acid	
		DT_{50} /DT_{90} days	ff *	DT_{50} /DT_{90} days	ff *
North Dakota	Hiler 2013a	4.7 / 16	0.766	-	-
Illinois		1.7 / 5.5	0.849	-	-
Madera_a		8.5 / 28	1.000	-	-
Madera_b	Hiler 2013b	-	-	2.9 / 9.8	0.812
Hanscheider Hof	Hellpointner & Junge 2013	-	-	1.0 / 3.4	-
Hoefchen		-	-	0.9 / 2.9	-
Dollendorf		-	-	1.0 / 3.2	-
geometric mean DT_{50}		**4.1**		**1.3**	
arithmetic mean ff			**0.872**		**0.812**

* formation fraction from parent to metabolite

Degradation rate at reference conditions

The studies by **Hiler 2013 a+b** were conducted at 20 ± 2 °C. Therefore no temperature standardisation is required. The water contents of the soils was maintained in the middle between pF 2.0 and pF 2.5 over the course of the study. A standardisation to reference soil moisture (pF 2.0) is conducted by the RMS. The results are shown in Table B.8.1-33.

The study by **Hellpointner & Junge 2013** was conducted at 20 °C. No temperature standardisation is required. The water contents of the soils were maintained at 55 % maximum water holding capacity (MWHC). Both the MWHC and the water contents at pF 2.0 (reference water content, field capacity) are given in the study for the three test soils. A standardisation to reference soil moisture (pF 2.0) is conducted by the RMS. The results are shown in Table B.8.1-33. In two soils (Hanscheider Hof, Dollendorf) the experimental water contents are above reference moisture therefore a value of 1 (i.e. no correction for moisture content) is used in these two cases.

Table B.8.1-33: Standardisation of laboratory DT_{50} for metabolites to reference soil moisture by RMS

Soil	Soil type (USDA classification)	Gravimetric water content (experimental) g/100 g	Reference water content at pF 2.0 (g/100 g)	Walker correction factor *	Experimental DT_{50}	Normalised DT_{50}
DCVA						
North Dakota	sandy clay loam	30.9	37.1	0.88	4.7	4.2
Illinois	silt loam	26.3	30.9	0.89	1.7	1.5
Madera_a (Hiler 2013a)	sandy loam	11.2	14.6	0.83	8.5	7.1
				geomean	4.1	3.5
FPB-acid						
Madera_b (Hiler 2013b)	sandy loam	13.2	15.7	0.89	2.9	2.6
Hanscheider Hof	silt loam	35.6	32.8	1.0	1.0	1.0

Hoefchen	silt loam	30.9	39.7	0.84	0.9	0.8
Dollendorf	clay loam	46.9	38.9	1.0	1.0	1.0
				geomean	**1.3**	**1.2**

* Walker correction factor f = (experimental moisture / reference moisture)$^{0.7}$
Walker factor is set to 1.0 when experimental moisture > reference moisture

B.8.1.2.3 Rate of degradation in field studies

In the monograph dated 01 October 1996 the following field trials were evaluated:

Two field trials in Germany with a single application of cyfluthrin (1 kg/ha ai, formulation type: 10 WP) on bare soil in April.

Five field trials in Germany with a single application of beta-cyfluthrin (0.0075 kg/ha as, formulation type: 25 EC) on bare soil in April or May. The degradation kinetics of two of these **studies were recalculated by Spickermann 2014**, see below.

Eight field trials in Northern America with a single application of cyfluthrin (1 kg/ha as, formulation type 200 EC, 240 EC) in July or August.

Two field trials in Japan with six applications of cyfluthrin on cabbage (0.1 kg/ha as, formulation type 50 EC).

The DT_{50} values were between 0.3 and 49 days (DT_{90} 9 – 163 days). The kinetic data are not in accordance with FOCUS 2006 and no normalisation is available.

Four **new field studies** with beta-cyfluthrin (formulation Bulldock 25 EC) applied at 2 × 0.020 kg/ha as in autumn were conducted by **Robinson 2014 a-d**.

B.8.1.2.3./1 (Robinson 2014a)

Reference	Robinson, N.	Location	Villefranche Du Périgord, S-France
		Incubation time	185 days
Title	Beta-cyfluthrin – field soil dissipation on beta-cyfluthrin from a field trial carried out in Southern France	Soil	loam, 1.1 % organic carbon, pH 7.4 (water)
Year of execution	01.04.2014	Nominal concentration	0.8 L/ha Bulldock 25 EC (0.020 kg/ha beta-cyfluthrin)
Study no.	R-30598		
GLP statement	yes	Temperature	field trial
Guideline	EPA OPPTS850.6100 (terrestrial fied dissipation); SETAC 1995: Marc Lynch, Procedure for assessing the environmental fate and toxicity of pesticides; Sanco/3029/99 rev.4, 2000 Sanco/825/00 rev.8, 2010	Moisture	field trial
Test substance	Bulldock 25 EC	DT_{50}	74.3 days (SFO), not according to FOCUS 2006

Purity	27.5 ± 0.6 g/kg beta-cyfluthrin	Metabolites	not analysed
Test system	field dissipation following 2 applications of 0.8 L/ha Bulldock 25 EC (2 × 0.020 kg/ha ai) on bare soil	Acceptability	acceptable

Executive Summary

The rate of degradation of beta-cyfluthrin following two applications of 0.8 L/ha Bulldock 25 EC (2 × 20 g/ha as) on bare soil was investigated. The field trial was carried out in Villefranche Du Périgord, Southern France. The trial comprised one untreated control plot and one plot treated with Bulldock 25 EC. The treated plot consisted of 4 subplots that were assigned as replicates. Eleven samplings were performed at day -0 DBFA (days before first application), day +0 DALA (days after last application) and at 1, 2, 7, 14, 25, 51, 100, 140 and 185 days after last application (DALA). One last scheduled sampling event (365 DALA) was cancelled because the active ingredient degraded sufficiently before 130 DALA. Dissipation was moderate and 25 days after the second application, residues were below LOQ (< 0.007 mg/kg) in the 0-10 cm top layer. Deeper layers, i.e. 10-20 cm and 20 - 30 cm, were additionally analysed, but no residues were found (< 0.01 mg/kg).

Materials

Test material
Identification: Bulldock 25 EC
Active Ingredient: Beta-Cyfluthrin
Type of Formulation: Emulsifiable concentrate (EC)
Purity: Active substance content: 2.752 ± 0.057 % w/w or 27.52 ± 0.56 g/kg or 24.79 ± 0.51 g/L
Expiry Date: June 20, 2014
Storage Conditions: At a dry, cool, well-ventilated place, at temperatures below 40 °C.

Reference material (non-radiolabelled)
Identification: Beta-cyfluthrin
Purity: 99.8 %
Expiry Date: 12 March 2017
Storage Conditions: Room temperature

Soils

The location in Villefranche Du Périgord, South France, is a representative area in term of crops where Bulldock 25 EC could be used. The soil is a loam (according USDA) containing 2.1 % organic matter (1.2 % oc) and a pH (H2O) of 7.4.

Study design

Experimental conditions

The rate of degradation of beta-cyfluthrin following two applications of 0.8 L/ha Bulldock 25 EC (2 × 20 g ai/ha) to bare soil was investigated in a field trial in Villefranche Du Périgord, Southern France. The trial comprised one untreated control plot and one plot. The untreated plot was 6.4 m x 25 m (160 m²) with 15 subsubplots. Each subsubplot was 3.2 m x 2.5 m (8 m²). The treated plot consisted of 4 replicates of 3.2 m x 42.5 m (4 x 136 m² = 544 m²), with 15 subsubplots. Each subsubplot was 3.2 m x 2.5 m (8 m²). The soil was prepared by ploughing at 25 cm depth on 03 October 2012 and harrowd with a fixed rotary harrow at 15 cm depth.

Test item applications were performed on 5 and 22 October 2012 which is a 17-day application interval. Due to bad weather (heavy rain) the 14-day interval could not be maintained. Calculations of the dose

rates to be applied were made using the nominal concentration of active substance of the test item. The applications were performed with a knapsack sprayer and a side boom powered by compressed air. The boom width was 3.2 m and was equipped with Teejet® XR 110015 VS flat fan nozzles. The percentage of the target rate of as applied was between 98-100 % for both applications. Eleven samplings were performed: day -0 DBFA (days before first application), day +0, 1, 2, 7, 14, 25, 51, 100, 140 and 185 days after last application (DALA). For sampling to 30 cm depth (0-25 DALA), a manual corer with PVC cartridges (length 30 cm, inner diameter 50.4 mm) was used. For sampling to 60 cm depth (51-185 DALA), an electric rotative corer with the same cartridges in PVC (length 30 cm, inner diameter 50.4 mm) was used.

During the trial, monthly mean temperatures ranged between 4.1ºC and 14.5ºC, with monthly rainfall between 64.8 and 123.1 mm (see Table B.8.1-35). During December 2012 and January 2013, more rain (about + 40 %) was noticed compared to the long-term average. Daily rainfall and air temperature during the course of the study are given in Table B.8.1-34. Daily global radiation ranged from a minimum value of 5 J/cm2 (16 January 2013) to 2549 J/cm^2 (24 April 2013).

Table B.8.1-34: Daily weather data at Villefranche Du Périgord, South France in October and November 2012 at a distance of 2 km (rain), 15 km (temperature) and 40 km (global radiation) from the trial site

	T min (°C)	T max (°C)	T mean (°C)	Rain (mm)	Global radiation (J/cm^2)
05.10.2012 (first treatment)	11.9	27.1	19.1	0.0	1663
06.10.	15.8	24.1	19.4	3.1	969
07.10.	16.5	19.0	17.9	7.0	417
08.10.	17.4	23.7	19.3	1.1	850
09.10.	16.7	21.5	18.1	1.0	770
10.10.	13.2	21.8	17.1	3.0	914
11.10.	15.1	21.1	16.9	4.0	781
12.10.	11.1	17.3	13.3	0.0	1061
13.10.	7.7	17.3	12.4	0.4	1270
14.10.	9.1	15.1	11.2	1.1	462
15.10.	4.6	15.0	9.2	0.0	1102
16.10.	7.5	13.4	10.8	3.5	623
17.10.	11.7	22.0	16.7	0.0	1000
18.10.	18.3	23.5	20.4	0.0	880
19.10.	15.0	21.2	17.3	18.0	630
20.10.	13.6	19.0	15.9	2.0	329
21.10.	15.3	20.6	16.5	2.0	940
22.10. (second treatment)	12.5	20.7	15.9	0.0	1056
23.10.	11.6	22.8	16.9	0.0	1099
24.10.	13.3	23.8	17.9	0.0	1184
25	13.9	19.0	15.9	0.0	604
26	14.1	17.0	15.0	4.6	192
27	5.1	9.1	6.8	0.0	753
28	0.0	8.9	4.1	0.0	1244
29	-0.2	11.9	6.1	0.0	1179
30	2.3	13.9	8.0	0.4	1083
31	7.4	12.8	9.8	2.0	558
01	5.7	11.7	8.7	3.5	506
02	8.6	17.5	12.3	4.0	922
03	10.2	17.9	13.2	2.0	870
04	10.4	11.9	10.8	14.0	155
05	7.8	11.0	8.7	5.5	567
06	6.5	8.1	6.8	0.0	185

07	2.7	11.3	6.8	0.0	1003
08	5.1	14.8	9.9	0.0	624
09	9.8	15.6	12.2	8.0	502
10	10.8	16.0	11.4	1.0	576
11	7.6	12.4	8.6	0.0	615
12	0.7	10.2	5.5	0.0	914
13	-0.7	10.3	5.1	0.0	645
14	2.8	20.3	13.8	0.0	852
15	10.4	19.5	14.1	0.0	843

Table B.8.1-35: Monthly weather data from October 2012 to April 2013 at a distance of 2 km (rain) and 15 km (temperature) from the trial site

Month	Rainfall (mm)	T min °C	T max °C	T mean °C
October 2012	53.2	11.2	18.9	14.5
November 2012	64.8	6.7	12.9	9.4
December 2012	123.1	4.5	9.9	6.9
January 2013	112.0	1.9	6.6	4.1
February 2013	68.5	1.0	8.0	4.2
March 2013	76.7	5.0	13.0	8.5
April 2013	80.2	7.2	15.7	11.1

No restricted products were applied on the crops before (within three years) and during the conduct of the trial. However, Round Up (Glyphosate 360 g/L) was used in order to keep the trial area free of weeds during the course of the study. In 2010 the site was used for maize cultivation, in 2011 for winter wheat cultivation and in 2012 the land was left fallow.

Analysis

After sampling, specimens were transported unfrozen to the test site for freezing and were kept frozen at < -18 °C until transportation to the laboratory facility. Core segmentation was carried out at the laboratory facility. Twenty replicate cores from the treated plot (5 cores per sub-plot, 4 sub-plots) at each time point were segmented into 3 horizons: 0-10 cm, 10-20 cm and 20-30 cm and the soil horizons were bulked together. The 30-60 cm cores taken from 50 DAT onwards were not prepared, but stored frozen in case there would be a requirement to analyse the deeper soil horizons. Five cores from the control plot at each time point were bulked together, but the cores were not segmented into depths.

The bulked samples were sieved through a 2 mm mesh in order to provide homogeneous samples for analysis. Sub samples for analysis were then analysed for residues of beta-cyfluthrin. In summary, an aliquot of soil sample (10 g) was extracted by shaking twice with acetone for 30 minutes. The combined soil extract was adjusted to a known volume and an aliquot (5 mL) was evaporated to low volume (0.25 mL) to remove the acetone. 1:1 (v/v) methanol/water solution (5 mL) was added to the sample and the sample partitioned with hexane (2 × 2 mL). The combined hexane extract was evaporated to dryness and re-dissolved in hexane (1 mL) prior to final analysis by Gas Chromatography with Mass Selective Detection (GC-MS) in negative chemical ionisation mode with selected ion monitoring of three ions. The most sensitive ion (*m/z* = 207) was used for residue calculation. The mean recovery values obtained for beta-cyfluthrin during method validation were between 70-110 % in all cases and the relative standard deviation was < 20 %. Furthermore, a storage stability test confirmed the stability of beta-cyfluthrin in soils at < -18 °C for up to 9 month.

The mean procedural recovery generated during the analysis of the samples from the field was 83.8 % ± 6.2 %.

Results

Residues of beta-cyfluthrin measured in the sample from the study are presented in Table B.8.1-36.

Table B.8.1-36: Residues of beta-cyfluthrin at field trial Villefranche Du Périgord, S-France

Date of sampling	Treated plot		Untreated plot	
	depth cm	dry weight residues mg/kg	depth cm	dry weight residues mg/kg
before first application	0 – 30	< 0.01	0 - 30	< 0.01
after second application = day 0	0 – 10	0.015	0 - 30	< 0.01
day 1	0 – 10	0.012	0 - 30	< 0.01
day 2	0 – 10 10 - 20	0.011 < 0.01	0 - 30	< 0.01
day 7	0 – 10 10 - 20	0.010 < 0.01	0 - 30	< 0.01
day 14	0 – 10 10 - 20	0.010 < 0.01	0 - 30	< 0.01
day 25	0 – 10 10 – 20 20 – 30	0.007 < 0.01 < 0.01	0 - 30	< 0.01
day 51	0 – 10 10 – 20 20 – 30	0.012 < 0.01 < 0.01	0 - 30	< 0.01
day 100	0 – 10 10 – 20 20 - 30	0.007 < 0.01 < 0.01	0 - 30	< 0.01
day 140	0 – 10	0.003	0 - 30	< 0.01
day 185	0 – 10	0.001	0 - 30	< 0.01

Limit of quantification = 0.01 mg/kg, Limit of detection = 0.003 mg/kg

No residues of beta-cyfluthrin were detected (< LOD, 0.003 mg/kg) in any of the pre-application or untreated samples in the 0 - 30 cm soil depth. No residues of beta-cyfluthrin were detected (< LOD, 0.003 mg/kg) in any of the 10 - 20 cm or 20 - 30 cm soil depths. Residues in the 0-10 cm horizon declined rapidly and were below the LOQ (< 0.01 mg/kg) at the 25 DALA sampling interval onwards apart from the 51 DALA interval where the residue was 0.012 mg/kg.

The unexpected high value at day 51 of 0.012 mg/kg was confirmed by two soil analysis (0.012 and 0.013 mg/kg, respectively). It does not fit well to the degradation pattern and can be considered to be an outlier.

Kinetic evaluation

In the study a kinetic fitting is provided on the basis of SFO kinetic model. The computer program CAKE version 1.3 was used, IRLS optimiser (iteratively reweighted least squares). The results are given in Table B.8.1-37.

Table B.8.1-37: Kinetic fitting by Robinson 2014a

Parameter	value	prob. > t	Chi^2 error	DT_{50}	DT_{90}
Evaluation including outlier on day 51, SFO-kinetic					
M0	0.0129	1.36E-06	18.34	89.8	298
k	0.0077722	0.0057			
Evaluation outlier on day 51 days excluded, SFO-kinetic					
M0	0.0119	1.26E-06	15.14	74.3	247
k	0.00933	0.0028			

Comment

The residue at day 51 of 0.012 mg/kg can be accepted as outlier. The value is in the range of the residues on day 0 or day 1. However, the author of the study gives no idea about the possible reasons for that outlier.

The kinetic analysis is not in accordance with FOCUS 2006. There is no discussion of kinetic models and therefore no best-fit model is given. The kinetic calculations were therefore conducted by the RMS.

B.8.1.2.3/2 (Robinson 2014b)

Reference	Robinson, N.	Location	Brigné, N-France
		Incubation time	96 days
Title	Beta-cyfluthrin – field soil dissipation on beta-cyfluthrin from a field trial carried out in Northern France	Soil	loam, 1.04 % organic carbon, pH 8.3 (water)
Year of execution	01.04.2014	Nominal concentration	0.8 L/ha Bulldock 25 EC (0.020 kg/ha beta-cyfluthrin)
Study no.	R-33344		
GLP statement	yes	Temperature	field trial
Guideline	EPA OPPTS850.6100 (terrestrial fied dissipation); SETAC 1995: Marc Lynch, Procedure for assessing the environmental fate and toxicity of pesticides; Sanco/3029/99 rev.4, 2000 Sanco/825/00 rev.8, 2010	Moisture	field trial
Test substance	Bulldock 25 EC	DT_{50}	23.7 days (SFO), not according to FOCUS 2006
Purity	27.5 ± 0.6 g/kg beta-cyfluthrin	Metabolites	not analysed
Test system	field dissipation following 2 applications of 0.8 L/ha Bulldock 25 EC (2 × 0.020 kg/ha ai) on bare soil	Acceptability	acceptable

Executive Summary

The rate of degradation of beta-cyfluthrin following two applications of 0.8 L/ha Bulldock 25 EC (2 × 20 g/ha as) to bare soil was investigated. The field trial was carried out in Brigné, Northern France. The trial comprised of one untreated plot and one plot treated with Bulldock 25 EC. The treated plot consisted of 4 subplots that were assigned as replicates. Eleven samplings were performed at day -0 DBFA

(days before first application), day +0, 1, 2, 7, 14, 26, 48, 96 days after last application (DALA). 26 days after the second application, residues were below LOQ (< 0.007 mg/kg) in the 0-10 cm top layer. Deeper layers, i.e. 10-20 cm and 20-30 cm, were additionally analysed, but no residues were found.

Materials and Methods

1. Test material
Identification: Bulldock 25 EC
Active Ingredient: Beta-cyfluthrin
Type of Formulation: Emulsifiable concentrate (EC)
Purity: Active substance content: 2.752 ± 0.057 % w/w or 27.52 ± 0.56 g/kg or 24.79 ± 0.51 g/L
Storage Conditions: At a dry, cool, well-ventilated place, at temperatures below 40 °C.

Reference material (non-radiolabelled)
Identification: Beta-cyfluthrin

Purity: 99.8 %
Storage Conditions: Room temperature

Soil
The location is a representative area in term of crops where Bulldock 25 EC could be used. The soil type is loam (according USDA), organic matter 1.8 % (organic carbon 1.04 %) and pH (water) 8.3.

Study design

Experimental conditions

The rate of degradation of beta-cyfluthrin following two applications of 0.8 L/ha Bulldock 25 EC (2 × 20 g/ha as) to bare soil was investigated. The field trial was carried out in Brigné, Northern France. The trial comprised one untreated plot and one plot treated with Bulldock 25 EC. The untreated plot was 6.4 m × 25 m (160 m²) with 15 subsubplots. Each subsubplot was 3.2 m × 2.5 m (8 m²). The treated plot consisted of 4 replicates of 3.2 m ×42.5 m (4 ×136 m² = 544 m²), with 15 subsubplots. The soil was prepared as follows: stubble ploughing with discs at 8 cm depth (02.August), ploughing at 15 – 20 cm depth (28.September), fixed rotary harrow at 15 cm depth with roller on 01 October.

Test item applications were performed with a knapsack sprayer and a side boom powered by compressed air. The boom width was 3.2 m and was equipped with Teejet® XR 110015 VS flat fan nozzles. Applications were performed on the 10 October 2012 and 24 October 2012. Calculations of the dose rates applied were made using the nominal concentration of the active substance of the test item. The percentage of the target rate of the active substance applied was between 97-103 % for both applications. Eleven samplings were performed at day -0 DBFA (days before first application), day +0 DALA (days after last application) and at 1, 2, 7, 14, 26, 48, 96, 140 and 183 days after last application (DALA). For sampling to 30 cm depth (0 - 25 DALA), a manual corer with PVC cartridges (length 30 cm, inner diameter 50.4 mm) was used. For sampling to 60 cm depth (25 – 180 DALA), an electric rotative corer with the same cartridges in PVC (length 30 cm, inner diameter 50.4 mm) was used.

During the trial, monthly mean temperatures ranged between 3.8ºC and 12.9ºC, with monthly mean rainfall of between 55 mm and 213 mm (see Table B.8.1-39). The period between the first and second application was very wet with 171 mm of rainfall in 14 days. December 2012 was also particularly wet with +60 % of rainfall compared to the long term average. Daily global radiation ranged from a minimum value of 79 J/cm2 (8 January 2013) to 2549 J/cm2 (20 April 2013). Daily weather data after the treatments are given in Table B.8.1-38.

Table B.8.1-38: Daily weather data at Brigné, Northern France in October and November 2012 at a distance of 2 km (rain) and 6 km (temperature and global radiation) from the trial site

	T min (°C)	T max (°C)	T mean (°C)	rain (mm)	global radiation (J/cm²)
10.10.2012 (first treatment)	16.2	21.5	17.7	9.0	735
11.10.	15.1	20.2	16.5	24.0	707
12.10.	9.9	17.5	12.9	0.1	1294
13.10.	9.5	13.3	11.9	34.0	389
14.10.	9.2	12.6	10.3	5.0	461
15.10.	2.8	15.5	9.6	4.1	1171
16.10.	9.9	17.1	12.9	0.6	857
17.10.	11.9	15.1	13.6	21.0	145
18.10.	12.4	15.3	12.9	24.0	265
19.10.	11.6	16.9	13.8	13.0	201
20.10.	10.0	14.1	12.4	34.0	301
21.10.	12.9	16.3	14.7	1.0	286
22.10.	14.3	17.8	15.4	0.6	407
23.10.	12.8	18.3	14.7	0.1	608
24.10. (second treatment)	12.2	17.0	13.3	0.2	749
25.10.	10.2	12.9	11.5	0.1	304
26.10.	10.4	12.0	10.5	1.0	144
27.10.	4.2	9.4	6.0	0.5	803
28.10.	0.7	11.2	5.8	0.1	989
29.10.	2.5	12.8	6.6	0.0	787
30.10.	0.3	14.2	6.2	0.1	1007
31.10.	-0.7	14.3	7.3	6.3	931
01.11.	6.3	12.3	9.0	6.5	637
02.11.	5.7	14.4	9.5	12.0	455
03.11.	8.5	12.8	9.8	5.3	399
04.11.	5.3	13.3	9.8	3.0	614
05.11.	6.6	13.6	8.2	10.2	723
06.11.	4.1	11.9	6.9	0.3	824
07.11.	2.5	14.9	7.0	0.2	710
08.11.	2.7	10.7	5.8	0.2	514
09.11.	1.6	12.3	7.1	0.3	279
10.11.	8.9	15.0	9.6	0.9	438
11.11.	2.0	12.0	6.0	0.1	772
12.11.	1.9	13.0	7.1	0.1	521
13.11.	4.9	13.0	8.0	0.1	685
14.11.	3.2	7.5	4.8	0.1	331
15.11.	3.7	6.3	5.0	0.1	292

Table B.8.1-39: Monthly weather data from October 2012 to April 2013 at a distance of 2 km (rain) and 6 km (temperature) from the trial site

Month	Rainfall (mm)	T min °C	T max °C	T mean °C
October 2012	213.0	10.1	16.5	12.9
November 2012	55.3	5.2	11.8	7.9
December 2012	102.3	4.7	10.7	7.4
January 2013	55.0	2.8	7.6	5.0
February 2013	59.9	1.1	7.3	3.8
March 2013	63.6	3.0	10.3	6.3
April 2013	58.9	5.2	14.7	9.9

No restricted products were applied on the crops before (within three years) and during the conduct of the trial. During 2010 and 2012, the site was used for wheat cultivation and in 2011 for sunflower cultivation.

Analysis

After sampling, specimens were transported to the test site for freezing and were kept frozen at <-18 °C until analysis. Twenty replicate cores from the treated plot (5 cores per sub-plot, 4 sub-plots) at each time point were segmented into 3 horizons: 0-10 cm, 10-20 cm and 20-30 cm and the soil horizons bulked together. Five cores from the control plot at each time point were bulked together, but the cores were not segmented into depths. The bulked samples were sieved through a 2 mm mesh in order to achieve homogeneous samples for analysis. Sub samples for analysis were then analysed for residues of beta-cyfluthrin. In summary, aliquots of soil samples (10 g) were extracted by shaking twice with acetone for 30 minutes. The combined soil extracts were adjusted to a known volume and an aliquot (5 mL) was evaporated to low volume (0.25 mL) to remove the acetone. 1:1 (v/v) methanol/water solution (5 mL) was added to each sample and the samples partitioned with hexane (2 × 2 mL). The combined hexane extract was evaporated to dryness and re-dissolved in hexane (1 mL) prior to final analysis by Gas Chromatography with Mass Selective Detection (GC-MS) in negative chemical ionisation mode with selected ion monitoring of three ions. The most sensitive ion (*m/z* = 207) was used for residue calculation. The mean procedural recovery generated during the analysis of the samples from the field was 97.0 % ±11.0 %.

Results

Residues of beta-cyfluthrin measured in the samples from the study Robinson 2014b are presented in Table B.8.1-40.

Table B.8.1-40: Residues of beta-cyfluthrin at field trial Brigné, N-France

Date of sampling	Treated plot		Untreated plot	
	depth cm	dry weight residues mg/kg	depth cm	dry weight residues mg/kg
before first application	0 – 30	< 0.01	0 - 30	< 0.01
after second application = day 0	0 – 10	0.018	0 - 30	< 0.01
day 1	0 – 10	0.020	0 - 30	< 0.01
day 2	0 – 10 10 - 20	0.012 < 0.01	0 - 30	< 0.01
day 7	0 – 10 10 - 20	0.010 < 0.01	0 - 30	< 0.01
day 14	0 – 10 10 - 20	0.015 < 0.01	0 - 30	< 0.01
day 26	0 – 10 10 – 20 20 – 30	0.007 < 0.01 < 0.01	0 - 30	< 0.01
day 48	0 – 10 10 – 20 20 – 30	0.005 < 0.01 < 0.01	0 - 30	< 0.01
day 96	0 – 10 10 – 20 20 - 30	0.003 < 0.01 < 0.01	0 - 30	< 0.01

Limit of quantification = 0.01 mg/kg, Limit of detection = 0.003 mg/kg

Twenty six days after the second application, residues were below LOQ (< 0.007 mg/kg) in the 0-10 cm

top layer. No residues of beta-cyfluthrin were detected in any of the pre-application or untreated samples. No residues of beta-cyfluthrin were detected in any of the 10-20 cm or 20-30 cm soil depths.

Kinetic evaluation

In the study a kinetic fitting is provided on the basis of SFO kinetic model. The computer program CAKE version 1.3 was used, IRLS optimiser (iteratively reweighted least squares). The results are given in Table B.8.1-41.

Table B.8.1-41: Kinetic fitting by Robinson 2014b

Parameter	value	prob. > t	Chi² error	DT_{50}	DT_{90}
SFO-kinetic					
M0	0.0189	2.78E-06	12.35	23.6	78.6
k	0.002928	0.0021			

Comment:

The kinetic analysis is not in accordance with FOCUS 2006. There is no discussion of kinetic models and therefore no best-fit model is given. The kinetic calculations were therefore conducted by the RMS.

B.8.1.2.3/3 (Robinson 2014c)

Reference	Robinson, N.	Location	Cieza, Spain
		Incubation time	49 days
Title	Beta-cyfluthrin – field soil dissipation on beta-cyfluthrin from a field trial carried out in Spain	Soil	silty clay, 0.31 % organic carbon, pH 8.1
Year of execution	02.04.2014	Nominal concentration	0.8 L/ha Bulldock 25 EC (0.020 kg/ha beta-cyfluthrin)
Study no.	R-33345		
GLP statement	yes	Temperature	field trial
Guideline	EPA OPPTS850.6100 (terrestrial fied dissipation); SETAC 1995: Marc Lynch, Procedure for assessing the environmental fate and toxicity of pesticides; Sanco/3029/99 rev.4, 2000 Sanco/825/00 rev.8, 2010	Moisture	field trial
Test substance	Bulldock 25 EC	DT_{50}	23.7 days (SFO), not according to FOCUS 2006
Purity	27.5 ± 0.6 g/kg beta-cyfluthrin	Metabolites	not analysed
Test system	field dissipation following 2 applications of 0.8 L/ha Bulldock 25 EC (2 × 0.020 kg/ha ai) on bare soil	Acceptability	acceptable

Executive Summary

The rate of degradation of beta-cyfluthrin following two applications of 0.8 L/ha Bulldock 25 EC (2 × 20 g/ha as) to bare soil was investigated. The field trial was carried out in Cieza, Spain. The trial comprised of one untreated control plot and one plot treated with Bulldock 25 EC. The treated plot consisted

of 4 subplots that were assigned for replicates. Eight samplings were performed at day -0 DBFA (days before first application), day +0 DALA (days after last application) and at 1, 2, 7, 14, 28 and 49 days after the last application (DALA). In the treated plots, dissipation of beta-cyfluthrin was rapid and residues were below the limit of quantification (< 0.01 mg/kg) from the 7 DALA sampling interval onwards in the 0-10 cm top layer. Deeper layers, i.e. 10-20 cm and 20-30 cm, were additionally analysed, but no residues were found (<0.01 mg/kg).

Materials and Methods

Test material

Identification: Bulldock 25 EC
Active Ingredient: Beta-Cyfluthrin
Type of Formulation: Emulsifiable concentrate (EC)
Purity: Active substance content: 2.752 ± 0.057 % w/w or 27.52 ± 0.56 g/kg or 24.79 ± 0.51 g/L
Storage Conditions: At a dry, cool, well-ventilated place, at temperatures below 40 °C.

Reference material (non-radiolabelled)
Identification: Beta-cyfluthrin
Purity: 99.8 %
Storage Conditions: Room temperature

Soils

The location is a representative area in term of crops where Bulldock 25 EC could be used. The soil is a silty clay (0 – 15 cm depth), organic matter 0.54 (organic carbon 0.31 %) and a pH 8.1.

Study design

Experimental conditions

The rate of degradation of beta-cyfluthrin following two applications of 0.8 L/ha Bulldock 25 EC (2 × 20 g/ha as) to bare soil was investigated. The field trial was carried out in Cieza, Spain. The trial comprised one untreated plot and one plot treated with Bulldock 25 EC. The untreated plot was 3 m × 35 m (105 m²) with 15 subsubplots. Each subsubplot was 3 m × 2 m (6 m²). The treated plot consisted of 4 replicates of 3 m × 35 m (4 × 105 m² = 420 m²), with 15 subsubplots. The soil was prepared on 16 August with a subsoiler and a rotary cultivator.

The applications were performed with a motorised knapsack sprayer and a side boom. The boom width was 3 m and was equipped with ASJ 110 02 flat fan nozzles. Applications were performed on 19 August 2013 and 02 September 2013. Calculations of the dose rates to be applied were made using the nominal concentration of active substance of the test item. The percentage of the target rate of the active substance applied was between 95 and 105 % for both applications. Eight samplings were performed at day -0 DBFA (days before first application), day +0 DALA (days after last application) and at 1, 2, 7, 14, 28 and 49 days after the last application (DALA). For sampling to 30 cm depth (0 - 28 DALA), a manual corer with PVC cartridges (length 30 cm, inner diameter 50.4 mm) was used. For sampling to 60 cm depth (last sampling at 49 DALA), 60 cm long cores with inner diameter of 50.4 mm were used and subsequently cut in two to provide 0-30 cm and 0-60 cm cores.

During the trial, monthly mean minimum temperatures ranged between 13.5ºC and 18ºC, and monthly mean maximum temperatures were between 26.9 to 32.1ºC, with monthly rainfall between 1.3 mm and 44.7mm (see Table B.8.1-43). Daily global radiation ranged from a minimum value of 72.2 W/m^2 (7 September 2013) to 308.5 W/m² (8 August 2013). Daily weather data after application are given in Table B.8.1-42.

Table B.8.1-42: Daily weather data in August and September 2013 at a distance of 6 km from the trial site Cieza, Spain

	T min (°C)	T max (°C)	rain (mm)	global radiation (W/m²)
19.08.2013 (first treatment)	17.7	34.6	0.0	280.0
20.08.	17.0	32.1	0.0	274.8
21.08.	20.3	32.2	0.0	264.1
22.08.	18.0	32.8	0.0	259.1
23.08.	20.0	31.8	0.0	247.0
24.08.	18.8	31.8	0.0	264.5
25.08.	19.4	31.3	0.0	262.5
26.08.	19.9	29.2	0.0	180.0
27.08.	21.3	30.4	0.0	246.8
28.08.	19.0	28.9	5.7	157.0
29.08.	16.6	25.2	38.2	130.1
30.08.	17.4	25.7	0.8	237.9
31.08.	14.2	28.9	0.0	271.0
01.09.	15.7	28.8	0.0	267.5
02.09. (second treatment)	14.7	29.2	0.0	265.8
03.09.	16.1	29.3	0.0	269.3
04.09.	15.7	28.3	0.0	265.2
05.09.	15.3	28.9	0.0	263.0
06.09.	16.8	31.0	0.0	254.6
07.09.	16.2	22.1	2.0	72.2
08.09.	13.8	29.0	0.0	194.4
09.09.	16.4	28.9	2.7	223.2
10.09.	15.4	28.9	0.2	253.4
11.09.	15.0	27.9	0.0	230.4
12.09.	17.6	28.4	0.0	244.8
13.09.	18.5	27.9	0.0	213.1
14.09.	14.6	27.2	0.0	217.8
15.09.	16.5	32.8	0.0	243.0

Table B.8.1-43: Monthly weather data from August to October 2013 at a distance of 6 km from the trial site Cieza, Spain

Month	rainfall (mm)	T min °C	T max °C
August 2013	44.7	18.0	32.1
September 2013	5.5	15.9	28.9
October 2013	1.3	13.5	26.9

No restricted products had been applied within the last three years and during the conduct of the trial.

Analysis

After sampling, specimens were transported from the trial site to the test site for freezing.The maximum interval between sampling and storage in frozen conditions was 8 hours and 15 minutes. During the storage, the specimens were kept frozen at targeted temperatures < -18 °C. Twenty replicate cores from the treated plot (5 cores per sub-plot, 4 sub-plots) at each time point were segmented into 3 horizons: 0-10 cm, 10-20 cm and 20-30 cm and the soil horizons bulked together. Five cores from the control plot

at each time point were bulked together, but the cores were not segmented into depths.

The bulked samples were sieved through a 2 mm mesh in order to achieve homogeneous samples for analysis. Sub samples for analysis were then analysed for residues of beta-cyfluthrin. In summary, aliquots of soil samples (10 g) were extracted by shaking twice with acetone for 30 minutes. The combined soil extracts were adjusted to a known volume and an aliquot (5 mL) was evaporated to low volume (0.25 mL) to remove the acetone. 1:1 (v/v) methanol/water solution (5 mL) was added to each sample and the samples partitioned with hexane (2 × 2 mL). The combined hexane extract was evaporated to dryness and re-dissolved in hexane (1 mL) prior to final analysis by Gas Chromatography with Mass Selective Detection (GC-MS) in negative chemical ionisation mode with selected ion monitoring of three ions. The most sensitive ion (*m/z* = 207) was used for residue calculation.

The mean procedural recovery generated during the analysis of the samples from the field was 88.2 % ±4.1 %.

Results

Residues of beta-cyfluthrin measured in the samples from the study are presented in Table B.8.1-44.

Table B.8.1-44: Residues of beta-cyfluthrin at field trial Cieza, Spain

date of sampling	treated plot		untreated plot	
	depth cm	dry weight residues mg/kg	depth cm	dry weight residues mg/kg
before first application	0 – 30	< 0.01	0 - 30	< 0.01
after second application = day 0	0 – 10	0.020	0 - 30	< 0.01
day 1	0 – 10	0.018	0 - 30	< 0.01
day 2	0 – 10 10 - 20	0.010 < 0.01	0 - 30	< 0.01
day 7	0 – 10 10 - 20	0.006 < 0.01	0 - 30	< 0.01
day 14	0 – 10 10 - 20	0.008 < 0.01	0 - 30	< 0.01
day 28	0 – 10 10 – 20 20 – 30	0.0015 < 0.01 < 0.01	0 - 30	< 0.01
day 49	0 – 10 10 – 20 20 – 30	< 0.01 < 0.01 < 0.01	0 - 30	< 0.01

Limit of quantification = 0.01 mg/kg, Limit of detection = 0.003 mg/kg

Twenty eight days after the second application, residues were below LOD in the 0-10 cm top layer. No residues of beta-cyfluthrin were detected (< LOD, 0.003 mg/kg) in any of the 10-20 cm or 20-30 cm soil depths. No residues of beta-cyfluthrin were detected (< LOD) in any of the pre-application or untreated samples.

Kinetic evaluation

In the study a kinetic fitting is provided on the basis of SFO kinetic model. The computer program CAKE version 1.3 was used, IRLS optimiser (iteratively reweighted least squares). The results are given in Table B.8.1-45.

Table B.8.1-45: Kinetic fitting by Robinson 2014c

Parameter	value	prob. > t	Chi² error	DT_{50}	DT_{90}
SFO-kinetic					
M0	0.01794	0.00104	21.8	6.4	21.3
k	0.1084	0.0397			

Comment

The kinetic analysis is not in accordance with FOCUS 2006. There is no discussion of kinetic models and therefore no best-fit model is given. The kinetic recalculation is conducted by the RMS.

B.8.1.2.3/4 (Robinson 2014d)

Reference	Robinson, N.	Location	Cieza, Spain
		Incubation time	49 days
Title	Beta-cyfluthrin – field soil dissipation on beta-cyfluthrin from a field trial carried out in Germany	Soil	silty clay, 0.31 % organic carbon, pH 8.1
Year of execution	02.04.2014	Nominal concentration	0.8 L/ha Bulldock 25 EC (0.020 kg/ha beta-cyfluthrin)
Study no.	R-33346		
GLP statement	yes	Temperature	field trial
Guideline	EPA OPPTS850.6100 (terrestrial fied dissipation); SETAC 1995: Marc Lynch, Procedure for assessing the environmental fate and toxicity of pesticides; Sanco/3029/99 rev.4, 2000 Sanco/825/00 rev.8, 2010	Moisture	field trial
Test substance	Bulldock 25 EC	DT_{50}	23.7 days (SFO), not according to FOCUS 2006
Purity	27.5 ± 0.6 g/kg beta-cyfluthrin	Metabolites	not analysed
Test system	field dissipation following 2 applications of 0.8 L/ha Bulldock 25 EC (2 × 0.020 kg/ha ai) on bare soil	Acceptability	acceptable

Executive Summary

The rate of degradation of beta-cyfluthrin following two applications of 0.8 L/ha Bulldock 25 EC (2 × 20g/ha as) to bare soil was investigated. The field trial was carried out in Dettelbach-Effeldorf, Southern Germany. The trial comprised of one untreated control plot and one plot treated with Bulldock 25 EC. The treated plot consisted of 4 subplots that were assigned for replicates. Twelve samplings were performed at day -0 DBFA (days before first application), day +0, 1, 2, 7, 13, 28, 48, 89, 146, 180 and 362 days after last application (DALA). In the treated plots, dissipation of beta-cyfluthrin was slow and residues were below the limit of quantification (< 0.01 mg/kg) from the 180 DALA sampling interval onwards. Deeper layers, i.e. 10 - 20 cm and 20 - 30 cm, were additionally analysed, but no residues were found (< 0.01 mg/kg).

Material and Methods

Test material
Identification: Bulldock 25 EC
Active Ingredient: Beta-cyfluthrin
Type of Formulation: Emulsifiable concentrate (EC)
Purity: Active substance content: 2.752 ± 0.057 % w/w or 27.52 ± 0.56 g/kg or 24.79 ± 0.51 g/L
Storage Conditions: At a dry, cool, well-ventilated place, at temperatures below 40 °C.

Reference material (non-radiolabelled)
Identification: Beta-cyfluthrin
Purity: 99.8 %
Storage Conditions: Room temperature

Soils

The location is a representative area in term of crops where Bulldock 25 EC could be used. The soil is a clay loam, 4.1 % organic matter (2.38 % organic carbon), pH 7.2.

Study design

Experimental conditions

The rate of degradation of beta-cyfluthrin following two applications of 0.8 L/ha Bulldock 25 EC (2 × 20 g ai/ha) to bare soil was investigated. The field trial was carried out in Dettelbach-Effeldorf, Southern Germany. The trial comprised one untreated plot and one plot treated with Bulldock 25 EC. The untreated plot was 4 m × 42.5 m (170 m²) with 15 subsubplots. Each subsubplot was 4 m x 2.5 m (10 m²). The treated plot consisted of 4 replicates of 2.5 m × 68 m (4 x 170 m² = 680 m²), with 15 subsubplots. The soil was prepared as follows: 12 September ploughing, 20 September harrowing.

The applications were performed with a knapsack sprayer and a side boom powered by compressed air. The boom width was 2.5 m and was equipped with Agro Top Airmix 110 015 flat fan nozzles. Applications were performed on 05 October 2012 and 19 October 2012. Calculations of the dose rates to be applied were made using the nominal concentration of active substance of the test item. The percentage of the target rate of active substance applied was between 98.5 - 107.4 % for both applications. Twelve samplings were performed at day -0 DBFA (days before first application), day +0 DALA (days after last application) and at 1, 2, 7, 13, 28, 48, 89, 146, 180 and 362 days after last application (DALA). For sampling to 30 cm depth (0 - 28 DALA), a manual corer with PVC cartridges (length 30 cm, inner diameter 50.4 mm) was used. For sampling to 60 cm depth (48 – 362 DALA), the same PVC cartridges (length 30 cm, inner diameter 50.4 mm) were used.

During the trial, monthly mean temperatures ranged between -0.75ºC and 19.68ºC, with monthly mean rainfall of between 27 mm and 143 mm (see Table B.8.1-47). Daily global radiation ranged from a minimum value of 141 J/cm2 (23 December 2012) to 8824.8 J/cm2 (06 June 2013). Daily wether data in October after treatments are shown in Table B.8.1-46.

Table B.8.1-46: Daily weather data in October 2012 at a distance of 7.5 km from the trial site Dettelbach, S-Germany

	T min (°C)	T max (°C)	T mean (°C)	rain (mm)	global radiation (Wh/m²)
05.10.2012 (first treatment)	5.2	19.8	13.1	0.2	2729.5
06.10.	10.6	20.5	15.2	1.0	2098.0
07.10.	4.2	12.9	9.5	14.4	2847.7

08.10.	0.3	13.4	6.6	0.0	3206.5
09.10.	5.9	13.8	9.3	3.2	2590.6
10.10.	2.2	12.1	6.7	0.0	3447.1
11.10.	0.3	14.1	7.5	0.0	3773.6
12.10.	5.8	12.5	9.2	7.1	685.2
13.10.	3.8	14.4	8.6	0.1	3158.8
14.10.	5.8	11.4	9.2	0.3	2002.0
15.10.	5.8	10.4	7.8	0.0	509.7
16.10.	0.8	12.8	7.3	0.0	2973.6
17.10.	1.4	19.1	9.7	0.0	2506.7
18.10.	4.5	17.8	10.9	0.0	2849.5
19.10. (second treatment)	5.8	20.8	11.9	0.0	3051.2
20.10.	5.0	17.8	9.9	0.0	2579.2
21.10.	5.0	11.2	8.0	0.2	1082.6
22.10.	7.6	10.2	8.8	0.0	893.4
23.10.	4.0	11.0	8.9	0.0	801.2
24.10.	5.6	9.2	7.9	0.0	615.2
25.10.	6.4	9.3	7.7	0.0	683.2
26.10.	2.1	7.6	5.1	13.2	995.1
27.10.	0.1	2.1	0.8	6.4	820.6
28.10.	-2.8	5.1	0.9	0.0	2758.2
29.10.	-5.9	3.1	-1.7	0.1	1385.0
30.10.	1.1	5.4	3.7	3.2	831.3
31.10.	1.4	11.3	6.0	0.0	2717.7

Table B.8.1-47: Monthly weather data from August to October 2013 at a distance of 7.5 km from the trial site Dettelbach, S-Germany

Month	Rainfall (mm)	T min °C	T max °C	T mean (°C)
October 2012	52.0	-5.5	20.0	8.3
November 2012	97.7	-1.5	14.2	5.0
December 2012	101.3	-16.0	13.6	1.8
January 2013	24.4	-7.0	12.5	0.6
February 2013	31.5	-9.5	6.5	-0.8
March 2013	25.3	-12.0	16.8	1.1
April 2013	51.1	-4.9	24.1	9.0
May 2013	142.7	3.5	22.1	11.8
June 2013	60.6	6.5	33.8	16.1
July 2013	69.4	6.6	33.7	19.7
August 2013	97.1	6.2	33.0	17.8
September 2013	57.5	4.4	28.1	13.6
October 2013	63.7	-1.2	22.2	10.2

Five maintenance pesticide applications of Roundup Ultramax (450 g glyphosate/L) and U 46 M-fluid (500 g MCPA/L) were performed in accordance with local practices between 25 October 2012 and 29 August 2013 to discard present weeds and prevent new emergences. In 2010, the field site was used for sugar beet; in 2011 for summer barley and in 2012 for rye cultivation. No restricted products were applied on the crops before (within three years) and during the conduct of the trial.

Analysis

After sampling, specimens were transported to the test site for freezing. In all cases, the maximum interval between sampling and storage in frozen conditions was less than8 hours. During the storage, the specimens were kept frozen at targeted temperatures < -18 °C. Twenty replicate cores from the treated plot (5 cores per sub-plot, 4 sub-plots) at each time point were segmented into 3 horizons: 0 - 10 cm, 10 - 20 cm and 20 - 30 cm and the soil horizons bulked together. Five cores from the control plot at each

time point were bulked together, but the cores were not segmented into depths. The bulked samples were sieved through a 2 mm mesh in order to achieve homogeneous samples for analysis. Sub samples for analysis were then analysed for residues of beta-cyfluthrin. In summary, aliquots of soil samples (10 g) were extracted by shaking twice with acetone for 30 minutes. The combined soil extracts were adjusted to a known volume and an aliquot (5 mL) was evaporated to low volume (0.25 mL) to remove the acetone. 1:1 (v/v) methanol/water solution (5 mL) was added to each sample and the samples partitioned with hexane (2 × 2 mL). The combined hexane extract was evaporated to dryness and re-dissolved in hexane (1 mL) prior to final analysis by Gas Chromatography with Mass Selective Detection (GC-MS) in negative chemical ionisation mode with selected ion monitoring of three ions. The most sensitive ion (*m/z* = 207) was used for residue calculation. The mean procedural recovery generated during the analysis of the samples from the field was 88.0 % ± 5.3 %.

Results

Residues of beta-cyfluthrin measured in the samples from the study are given in Table B.8.1-48.

Table B.8.1-48: Residues of beta-cyfluthrin at field trial Dettelbach, S-Germay

Date of sampling	Treated plot		Untreated plot	
	depth cm	dry weight residues mg/kg	depth cm	dry weight residues mg/kg
before first application	0 – 30	< 0.01	0 - 30	< 0.01
after second application = day 0	0 – 10	0.026	0 - 30	< 0.01
day 1	0 – 10	0.027	0 - 30	< 0.01
day 2	0 – 10 10 - 20	0.027 < 0.01	0 - 30	< 0.01
day 7	0 – 10 10 - 20	0.025 < 0.01	0 - 30	< 0.01
day 13	0 – 10 10 - 20	0.021 < 0.01	0 - 30	< 0.01
day 28	0 – 10 10 – 20 20 – 30	0.012 < 0.01 < 0.01	0 - 30	< 0.01
day 48	0 – 10 10 – 20 20 – 30	< 0.011 < 0.01 < 0.01	0 - 30	< 0.01
day 89	0 – 10 10 – 20 20 – 30	0.013 < 0.01 < 0.01	0 - 30	< 0.01
day 146	0 – 10 10 – 20 20 – 30	0.012 < 0.01 < 0.01	0 - 30	< 0.01
day 180	0 – 10 10 – 20 20 – 30	0.0015 < 0.01 < 0.01	0 - 30	< 0.01
day 362	0 – 10 10 – 20 20 – 30	< 0.01 < 0.01 < 0.01	0 - 30	< 0.01

Limit of quantification = 0.01 mg/kg, Limit of detection = 0.003 mg/kg

At the sampling 180 days after the second application, residues were < LOD) in the 0-10 cm top layer. No residues of beta-cyfluthrin were detected (< LOD, 0.003 mg/kg) in any of the 10-20 cm or 20-30 cm soil depths. No residues of beta-cyfluthrin were detected (< LOD) in any of the pre-application or untreated samples.

Kinetic evaluation

In the study a kinetic fitting is provided on the basis of SFO kinetic model. The computer program CAKE version 1.3 was used, IRLS optimiser. The results are given in Table B.8.1-49.

Table B.8.1-49: Kinetic fitting by Robinson 2014d

Parameter	value	prob. > t	Chi^2 error	DT_{50}	DT_{90}
SFO-kinetic					
M0	0.02534	6.342E-07	16.8	64.9	216
k	0.01068	0.00277			

Comment

The kinetic analysis is not in accordance with FOCUS 2006. No discussion of kinetic models is given. The kinetic calculations were therefore conducted by the RMS.

B.8.1.2.3/5 (Spickermann 2014)

Reference	:	Spickermann, G
Title	:	Kinetic evaluation (trigger endpoints) according to FOCUS kinetics 2011, soil field dissipation trials for beta-cyfluthrin (n=5)
Date	:	08 April 2014
GLP statement	:	not relevant
Guideline	:	not relevant; calculation according FOCUS Kinetics (2006)
Test substance	:	beta-cyfluthrin
Test system	:	aerobic degradation - field studies

Summary

Two out of five field studies conducted in Germany in 1989 were kinetically re-calculated according FOCUS 2006. For three out of five field studies too few data points were available and therefore no kinetic calculation was conducted.

A short summary of the field tests is given in Table B.8.1-50 and the soil residues are given in Table B.8.1-51.

Table B.8.1-50: Field tests conducted in 1989 (short summary), kinetic calculation by Spickermann 2014

field trial	**Burscheid, Germany**	**Kleinniedesheim, Germany**
reference	Schmidt & Bachlechner 1991a	Schmidt & Bachlechner 1991b
report no.	report no. 0197-89 (registration no. BOD-94-00-903)	report no. 0204-89 (registration no. BOD-95-50005)
application date	05 May 1989	18 April 1989
test substance	FRC4545, Bulldock 25 EC (25 g/L beta-cyfluthrin)	FRC4545, Bulldock 25 EC (25 g/L beta-cyfluthrin)
rate	0.3 L/ha = 7.5 g/ha as	0.3 L/ha = 7.5 g/ha as
soil	loam soil, pH 5.5-5.6, organic carbon 1.5 – 2.5 %	sandy loam soil, pH 7, organic carbon 2.5 %

Table B.8.1-51: Soil residues of beta-cyfluthrin used for kinetic calculation

Burscheid		Kleinniedesheim	
Time interval	**Residues µg/kg**	**Time interval**	**Residues µg/kg**
day 0	2.6	day 0	5.4
day 14	0.59	day 14	1.7
day 31	0.46	day 30	2.3
day 60	0.2 *	day 60	0.76
days 117 - 242	< LOD	day 90	0.2 *
		days 120 - 240	< LOD

LOD = limit of detection = 0.4 µg/kg
* residue is set to ½ LOD

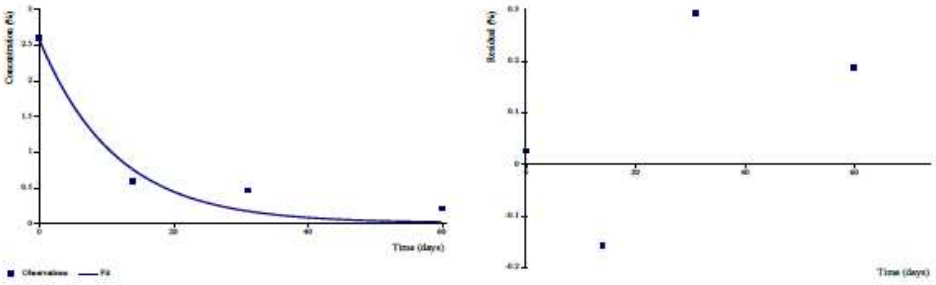
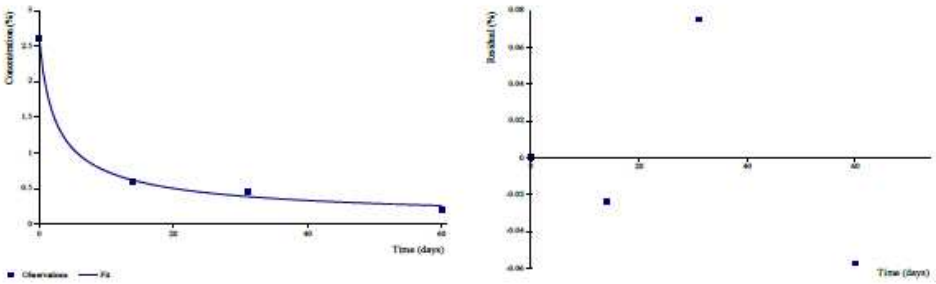
Results

For both field trials FOMC kinetics were found to be the best-fit models (trigger endpoints). The kinetic data are summarised in Table B.8.1-52. The flowchart steps conducted by Spickermann 2014 are given in Figure B.8.1-2.

Table B.8.1-52: Trigger endpoints for beta-cyfluthrin from two field trials conducted in Germany 1989

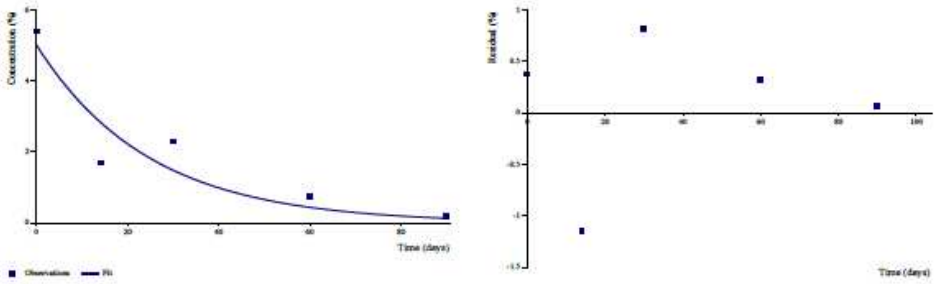
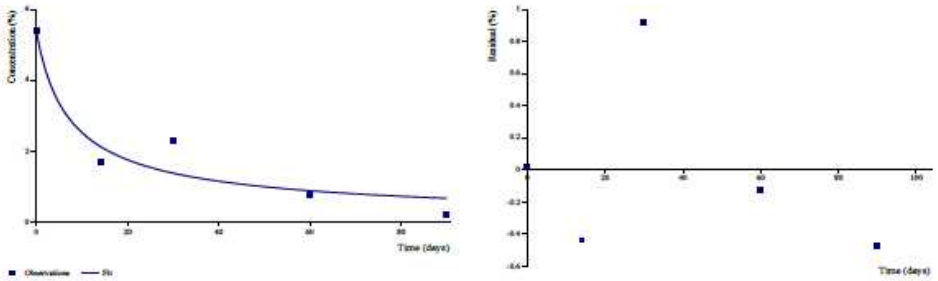
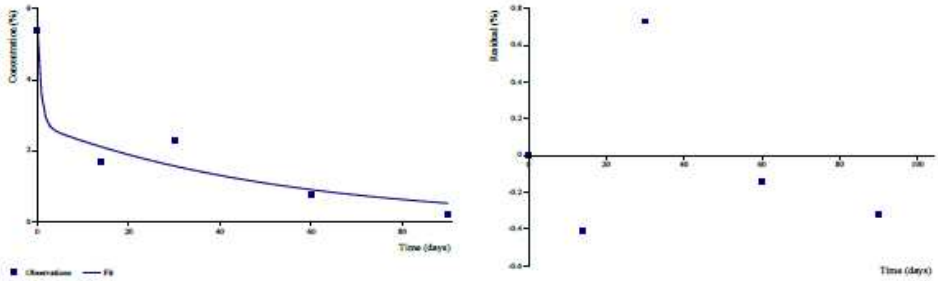
Trial	Best-fit model	Kinetic data	chi^2 error	DT_{50}	DT_{90}
Burscheid	FOMC	M0 = 2.6 α = 0.633 β = 1.594	5.16	3.2	59
Kleinniedesheim	FOMC	M0 = 5.383 α = 0.725 β = 5.431	22.3	8.7	125

Beta-cyfluthrin

Flowchart step	Model	k-rate	Prob > t[a)]	χ^2-error%	Visual	DT_{50}	DT_{90}
Run SFO & FOMC	SFO	0.08821	0.03348	16.3	acceptable	7.86	26.1
							
	FOMC	α = 0.633 β = 1.594	-a)	5.16	good	3.17	59.0
							
Conclusions: • **SFO is visually and statistically acceptable though χ^2-error% > 15 %** • **FOMC is visually and statistically acceptable** **=> FOMC best model** **Comment: The recommendation of FOCUS kinetics (2011) with a minimum of five data points could not be met, due to the rapid degradation of beta-cyfluthrin. Since a visual and statistical reliable fit could be achieved, the endpoint should be considered acceptable (FOCUS kinetics 2011, p. 71).**						**3.2**	**59.0**

a) In order to assess the fitted degradation rates as statistically acceptable, Prob > t (i.e. the p-value) should be < 0.05 (or < 0.1 with a good visual fit). Since both FOMC parameters α and β are shape parameters rather than degradation rates, the t-test is not used for FOMC.

Beta-cyfluthrin

Flowchart step	Model	k-rate	Prob > t[a)]	χ^2-error%	Visual	DT_{50}	DT_{90}
Run SFO & FOMC	SFO	0.04061	0.03579	25.8	acceptable	17.1	56.7
							
	FOMC	α = 0.725 β = 5.431	-[a)]	22.3	acceptable	8.7	125.0
							
FOMC visually and statistically better than SFO, so fit DFOP	DFOP	k_1 = 1.218 k_2 = 0.01846 g = 0.4949	0.497 0.1983 -	22.3	acceptable	2.67	87.7
							
Conclusions: • **SFO is visually and statistically acceptable** • **FOMC is visually and statistically acceptable** • **DFOP is statistically not acceptable, since k_1 and k_2 > 0.05** **=> FOMC best model**						**8.7**	**125.0**

a) In order to assess the fitted degradation rates as statistically acceptable, Prob > t (i.e. the p-value) should be < 0.05 (or < 0.1 with a good visual fit). Since both FOMC parameters α and β are shape parameters rather than degradation rates, the t-test is not used for FOMC.

Figure B.8.1-2: Flowchart steps in kinetic analysis of field trial Burscheid (Spickermann 2014)

Comment

The kinetic calculation by Spickermann 2014 can be accepted. The kinetic data are seen as additional information for degradation of beta-cyfluthrin after application in spring in Germany. The field studies by Schmidt & Bachlechner were non-GLP studies.

Kinetic calculation of field data from the studies Robinson 2014 (a-d) by RMS

The kinetic calculations in the studies by Robinson 2014 a – d were not accepted. Therefore a recalculation of the field trials from S-France, N-France, Spain and Germany was conducted by the RMS.

The model fit and the statistical evaluation of the results were carried out with the software KinGui version 2.2014. For the optimisation of the algorithms, Nonlinear Least Spuares (NLLS) was used.

Table B.8.1-53: Kinetic evalutation of field trials from S-France, N-France, Spain and Germany (Robinson 2014 a-d) by RMS

Kinetic model (visual fit)	Kinetic parameters	p-value t-test	Chi^2 error	DT_{50}	DT_{90}
Robinson 2014a : S-France					
SFO (unacceptable)	M0: 0.012 k: 0.00932	0.004	15.2	74.4	247
FOMC (acceptable)	M0: 0.0134 α: 0.466 β: 9.843	(no t-test for α or β)	13.9	33.8	1370
DFOP (acceptable)	M0: 0.015 k1: 0.9525 k2: 0.00758 g: 0.3074	0.14 0.004	10.6	43	255
HS (acceptable)	**M0: 0.015 k1: 0.1621 k2: 0.00756 t_b: 2.3**	**0.046 0.006**	**10.9**	**45**	**258**
Robinson 2014b: N-France					
SFO (acceptable)	**M0: 0.0165 k: 0.0234**	**0.013**	**17.4**	**29.7**	**99**
FOMC (acceptable)	M0: 0.0167 α: 3.786 β: 141	(no t-test for α or β)	18.4	28.3	118
DFOP	no further testing as FOMC gives no better fit than SFO				
HS	no further testing as FOMC gives no better fit than SFO				
Robinson 2014c: Spain					
SFO (unacceptable)	M0: 0.018 k: 0.109	0.038	21.7	6.4	21.1
FOMC (acceptable)	**M0: 0.021 α: 0.687 β: 1.893**	**(no t-test for α or β)**	**18.0**	**3.3**	**52**
DFOP (acceptable)	M0: 0.021 k1: 0.565 k2: 0.0398 g: 0.567	0.18 0.26	19.0	2.9	37
HS (acceptable)	M0: 0.021 k1: 0.293 k2: 0.0437 t_b: 3.0	0.09 0.26	17.6	2.4	35.5
Robinson 2014d: Germany					
SFO (acceptable)	M0: 0.025 k: 0.0107	0.003	16.8	64.9	216
FOMC (acceptable)	M0: 0.0278 α: 0.605 β: 18.21	(no t-test for α or β)	14.1	39.1	802

DFOP (acceptable)	M0: 0.0279 k1: 0.0626 k2: 0.0045 g: 0.473	 0.21 0.21	14.2	35.6	370
HS (acceptable)	**M0: 0.028** **k1: 0.0249** **k2: 0.00485** **t_b: 28**	 **0.04** **0.1**	**12.9**	**27.9**	**359**

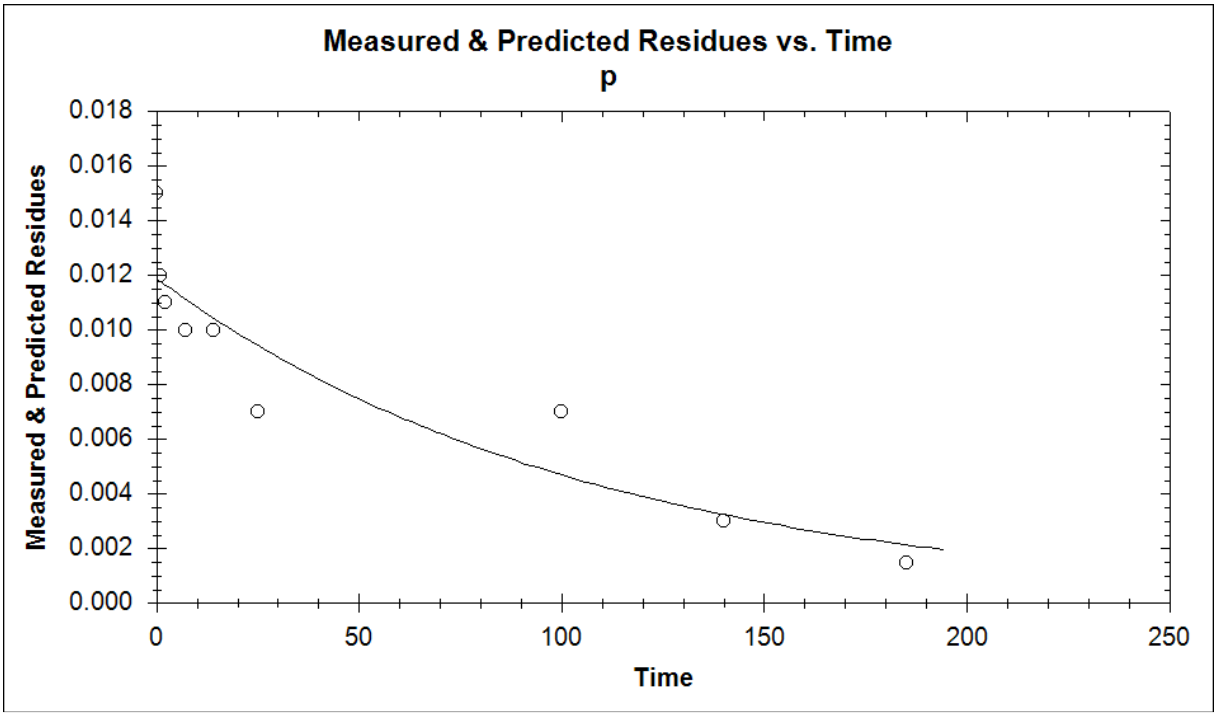
Results
The results are summarised in Table B.8.1-53.

There is only little difference in visual fits between the kinetic models tested. Only the visual fits for SFO-kinetics for trials in S-France and Spain are seen not acceptable due to a big error in prediction of M0 value, a non-random pattern in residual-time-plot or an underestimation of final sampling points. All the other kinetic models are seen acceptable. The main reasons to identify a best-fit model are therefore the chi^2 errors and the p-values for the t-tests of k-rates.

The kinetic models printed in bold letters are seen as best-fit models to derive trigger endpoints for the particular trial: HS-kinetics for trials in S-France and Germany, SFO in N-France, FOMC in Spain.
A quite fast degradation can be seen in Spain (DT_{50} = 3.3 days, DT_{90} = 52 days) and N-France (DT_{50} = 29.7 days, DT_{90} = 99 days). The overall-DT_{50} in S-France and Germany is 45 days and 27.9 days, respectively, the DT_{90} 258 days and 359 days. Due to the very early breakpoint (t_b = 2.3 days) in S-France, the trial in Germany represents the worst case for degradation of beta-cyfluthrin in the field.

No temperature or temperature-moisture normalisation is provided for the field trials. Therefore only trigger endpoints can be derived.

The modelled and measured data of beta-cyfluthrin residues and the residuals are given as graphic output of KinGUI in Figure B.8.1-3 to Figure B.8.1-16.



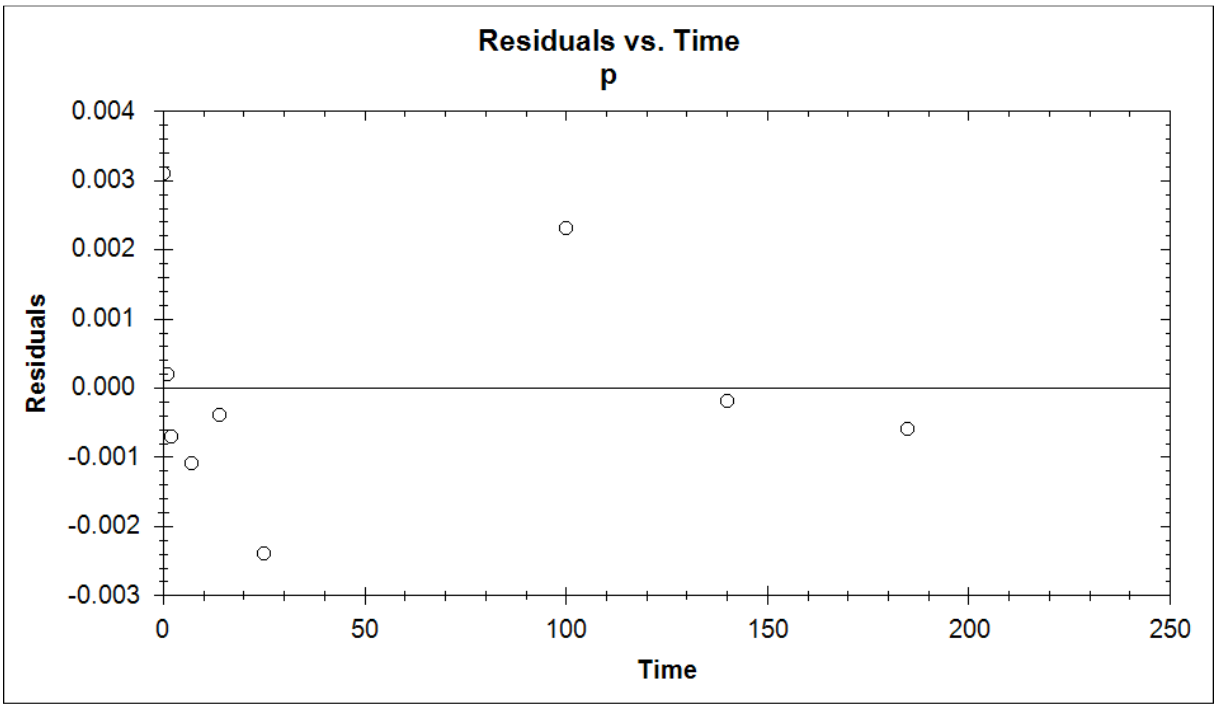
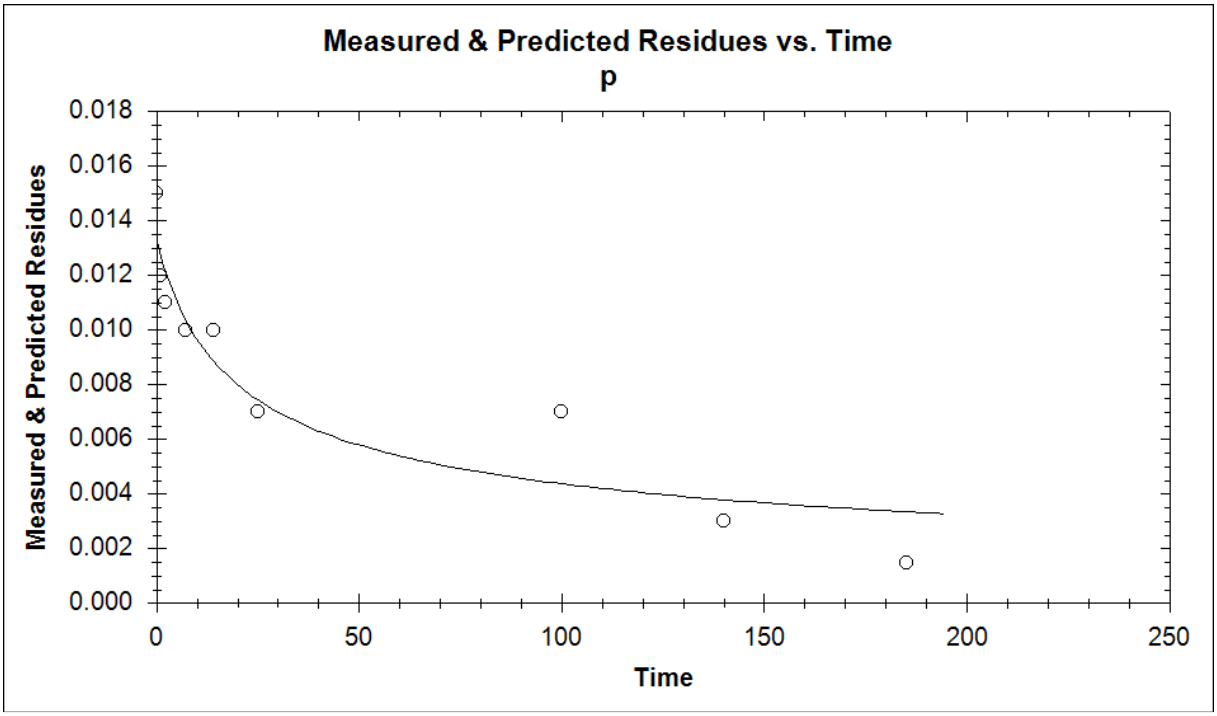


Figure B.8.1-3: SFO-kinetic for degradation of beta-cyfluthrin in field trial in S-France



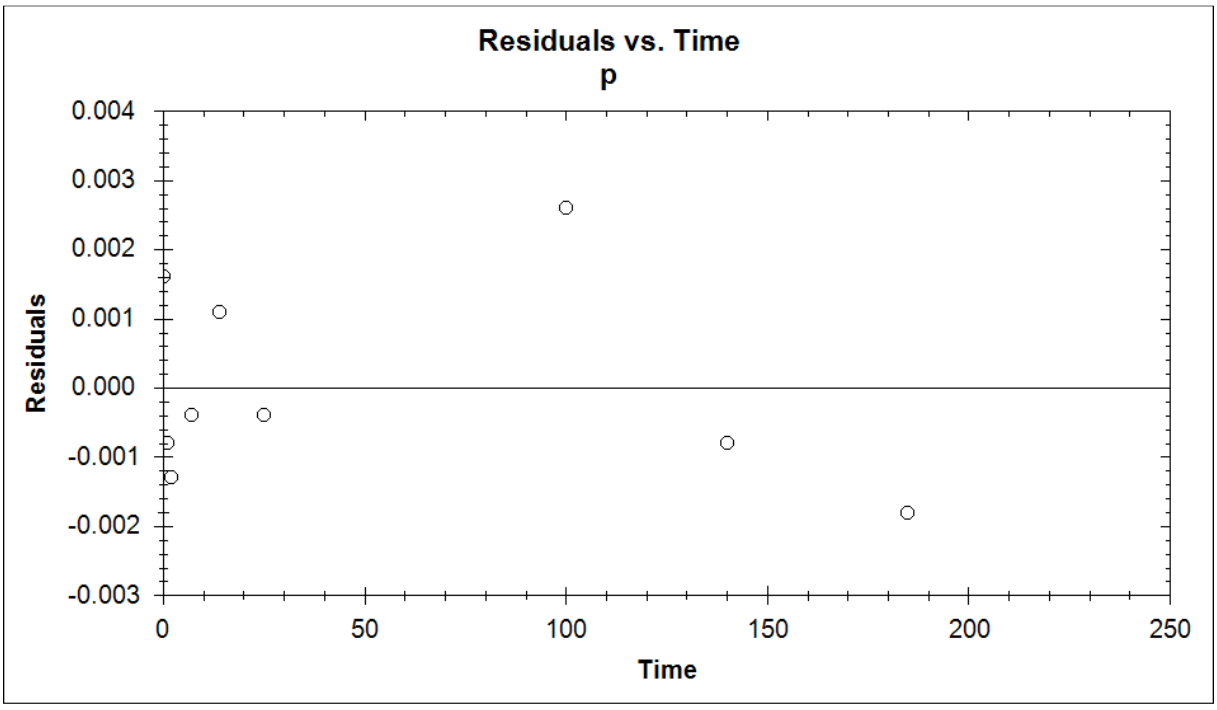
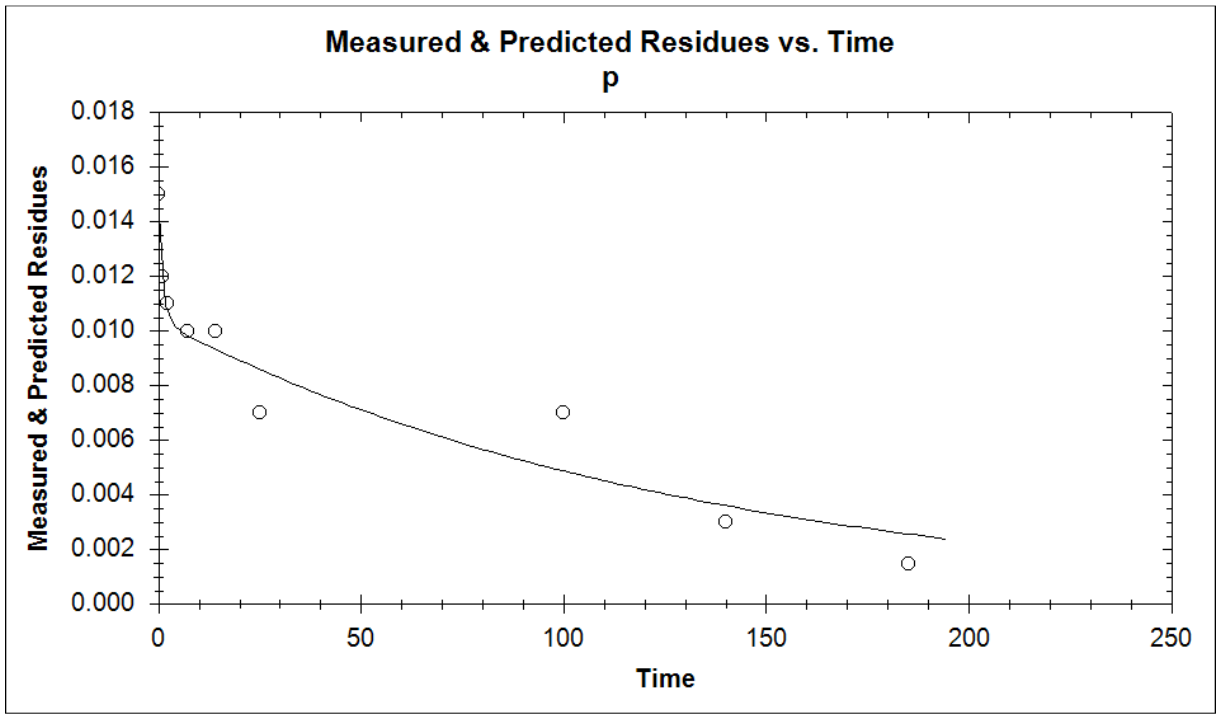


Figure B.8.1-4: FOMC-kinetic for degradation of beta-cyfluthrin in field trial in S-France



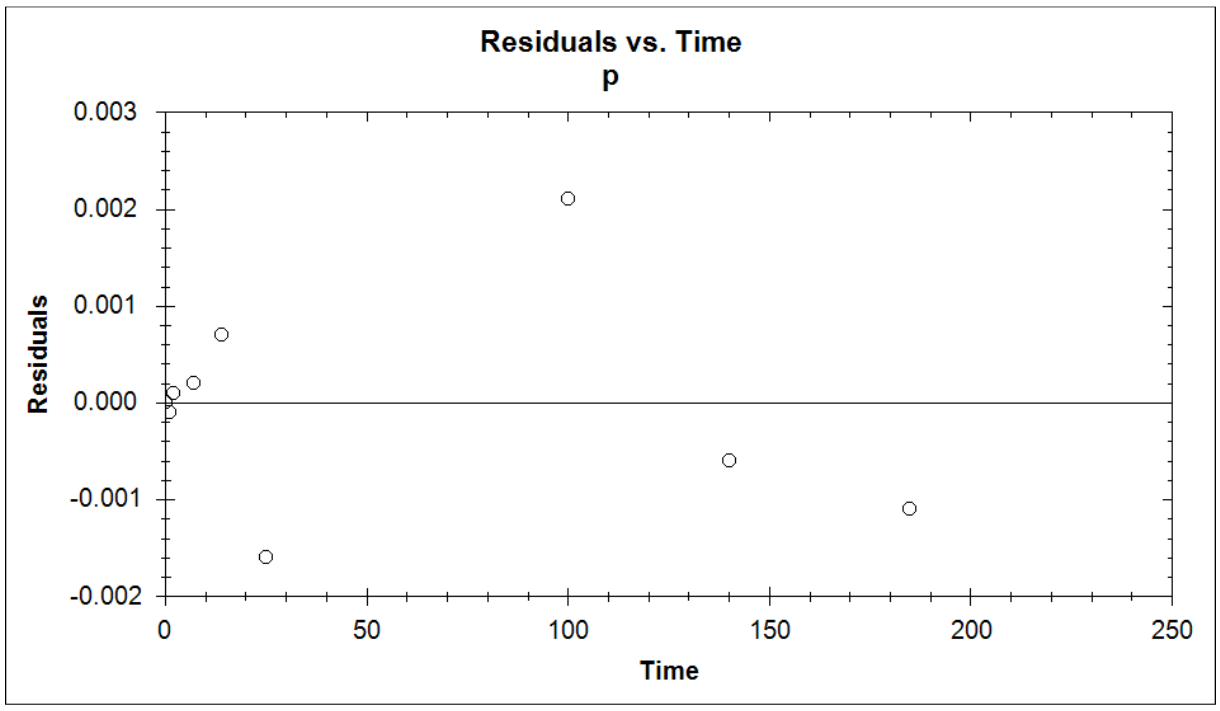
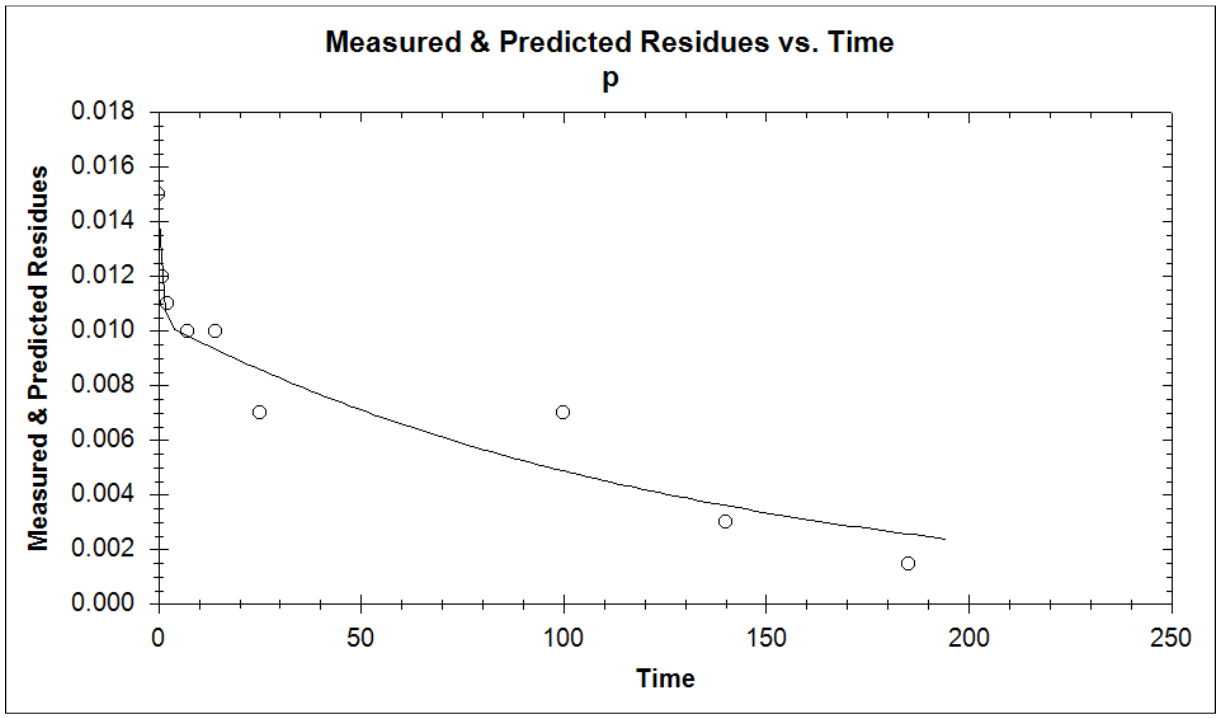


Figure B.8.1-5: DFOP-kinetic for degradation of beta-cyfluthrin in field trial in S-France



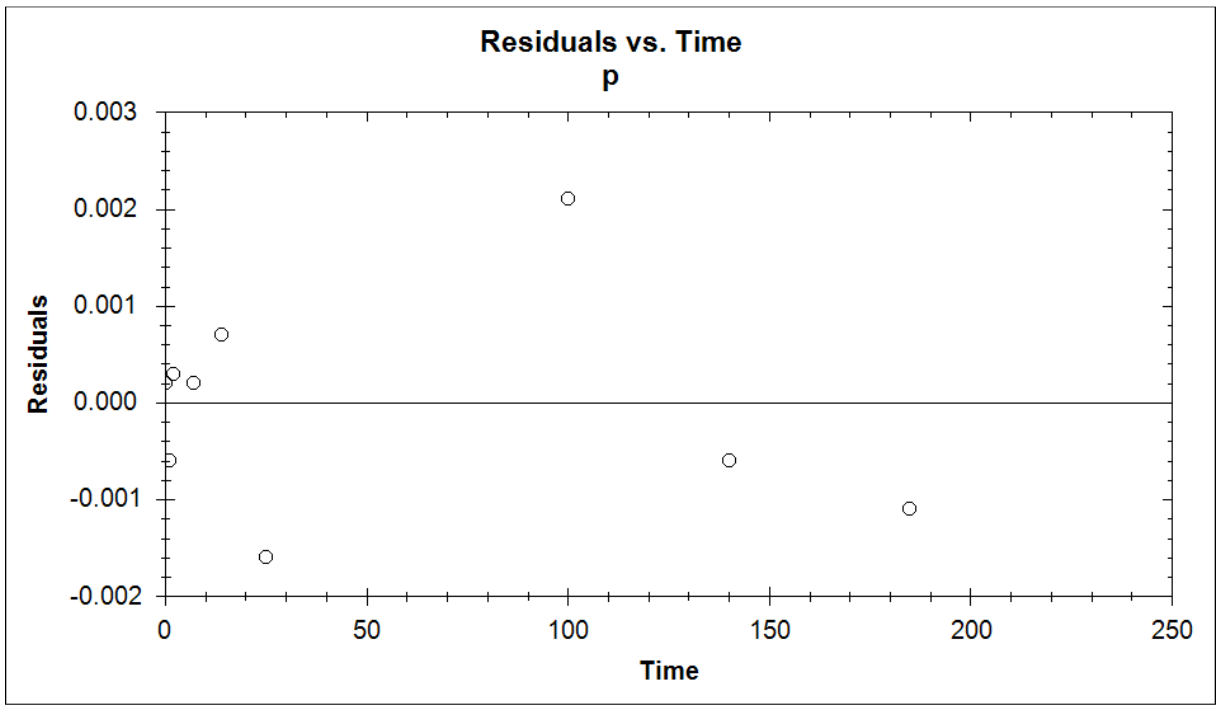
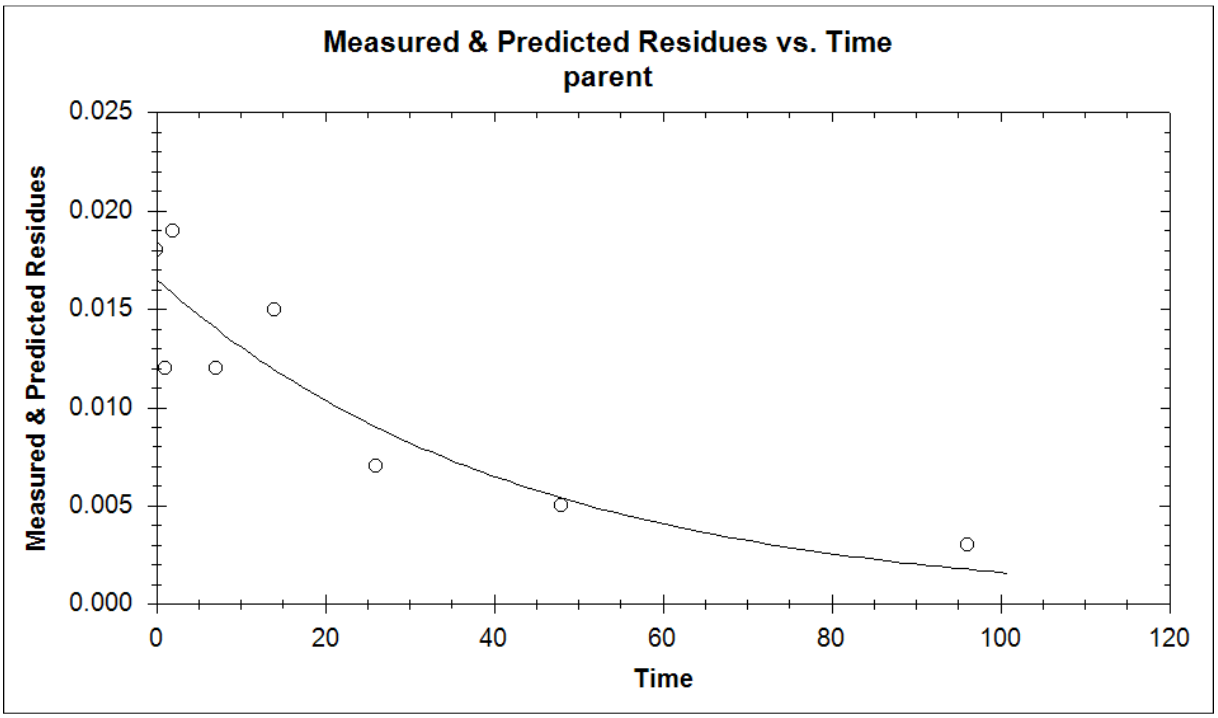


Figure B.8.1-6: HS-kinetic for degradation of beta-cyfluthrin infield trial in S-France



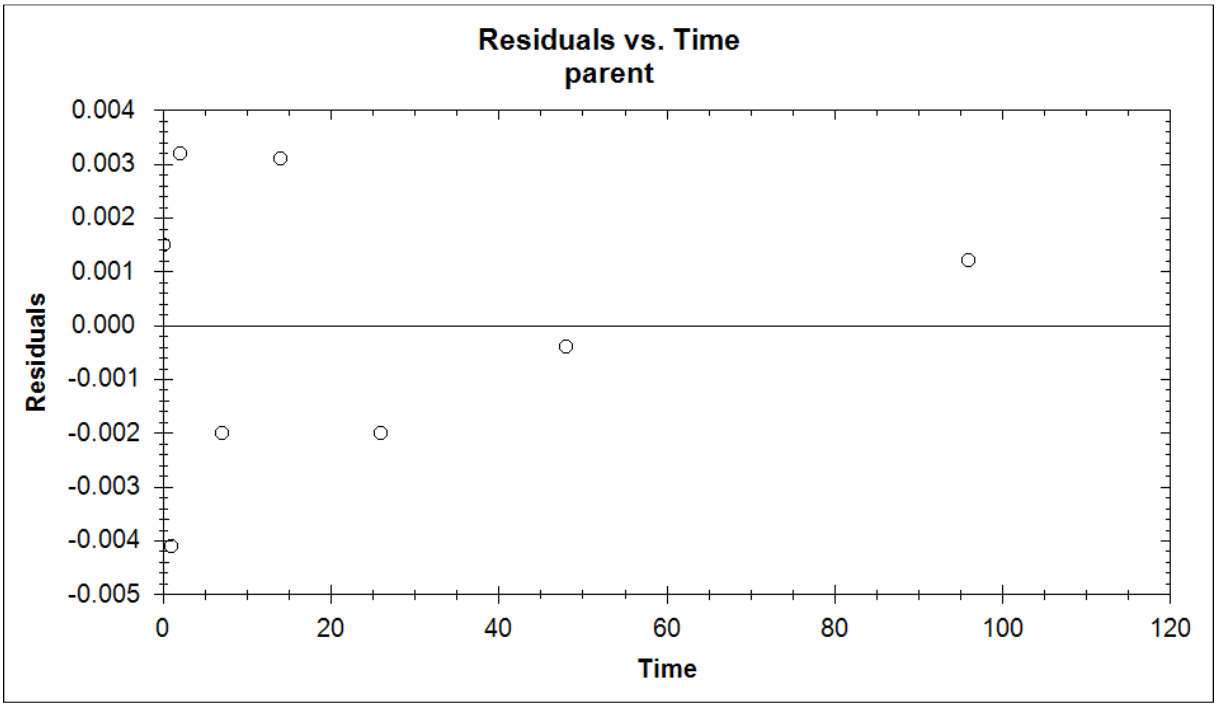
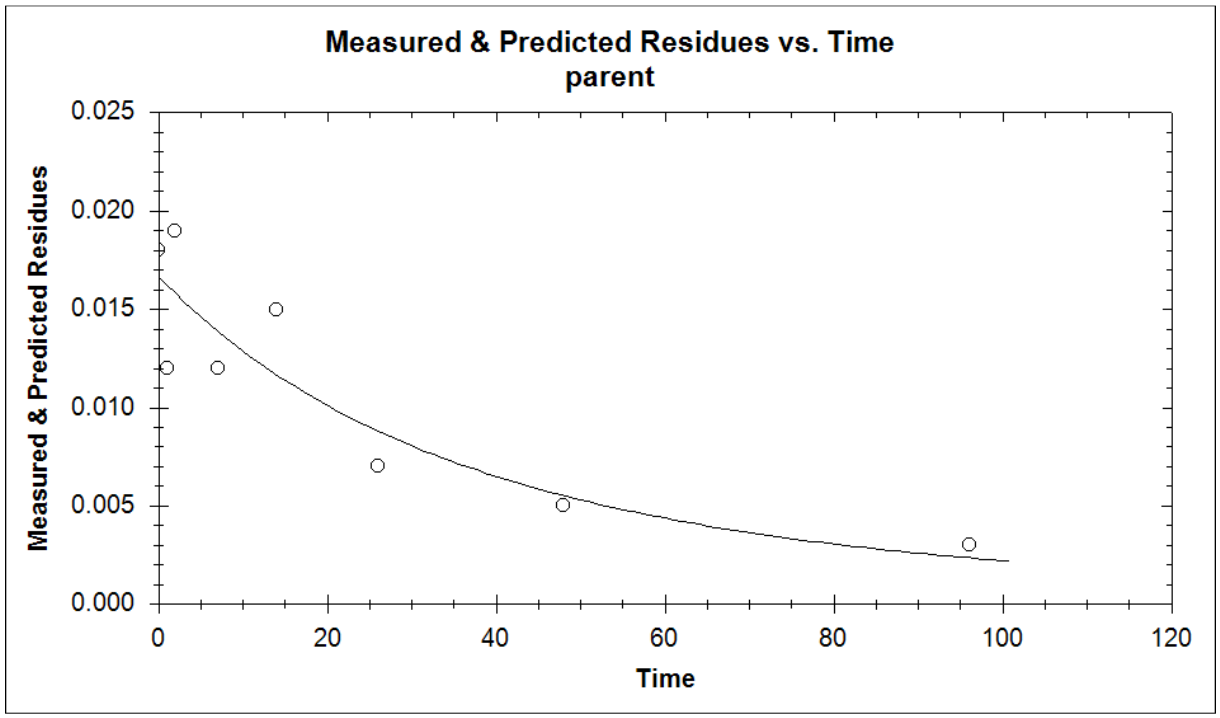


Figure B.8.1-7: SFO-kinetic for degradation of beta-cyfluthrin in field trial in N-France



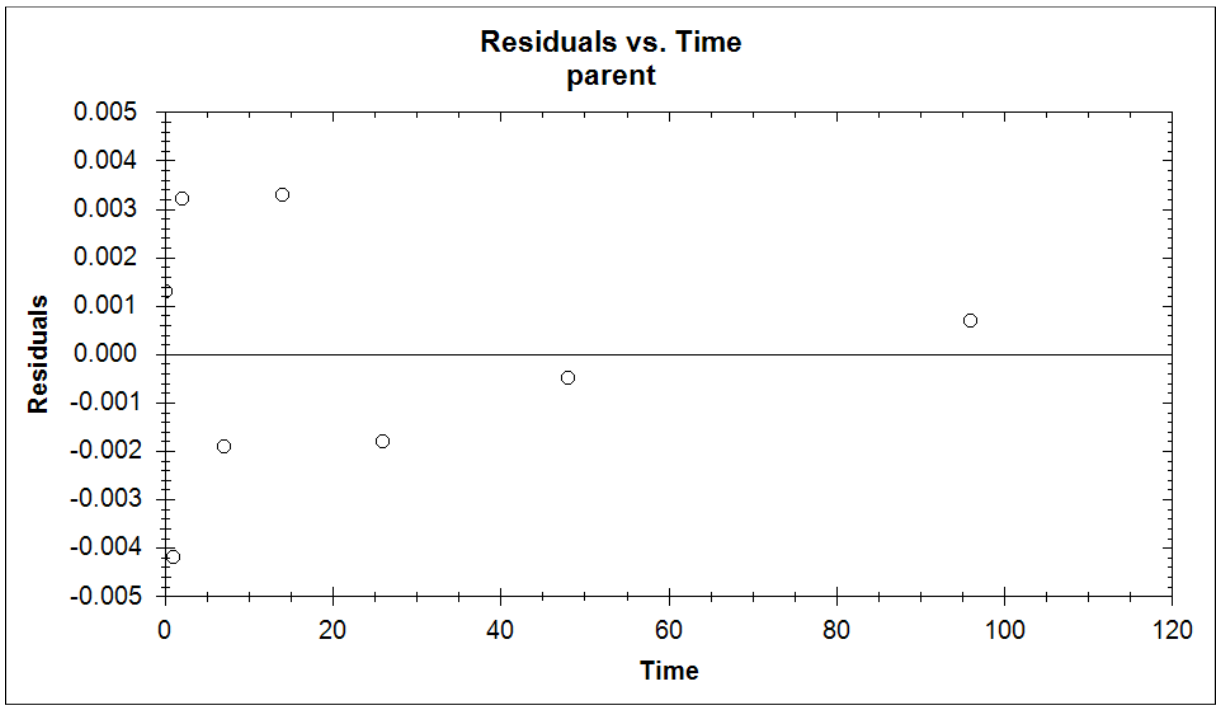


Figure B.8.1-8: FOMC-kinetic for degradation of beta-cyfluthrin in field trial in N-France

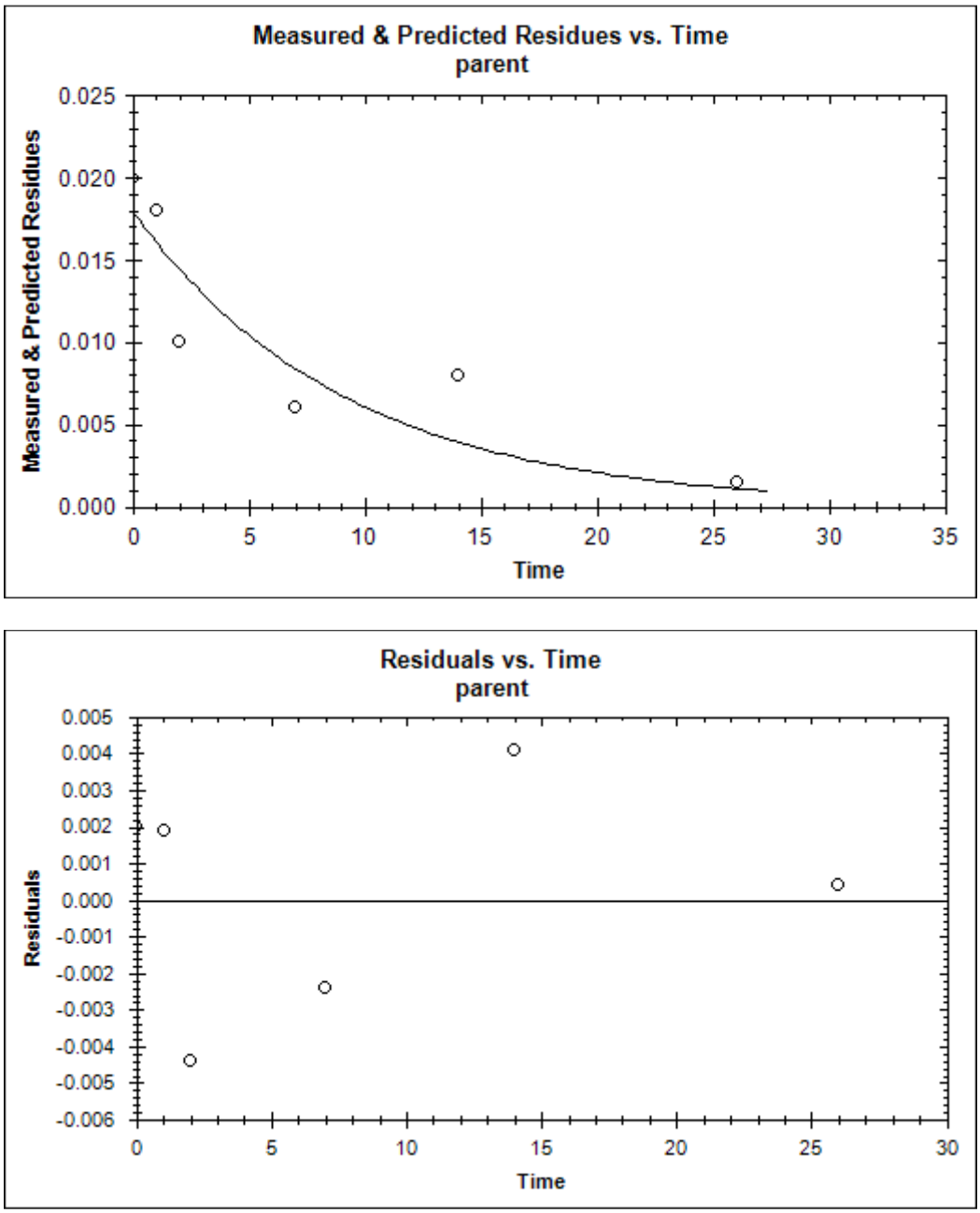


Figure B.8.1-9: SFO-kinetic for degradation of beta-cyfluthrin in field trial in Spain

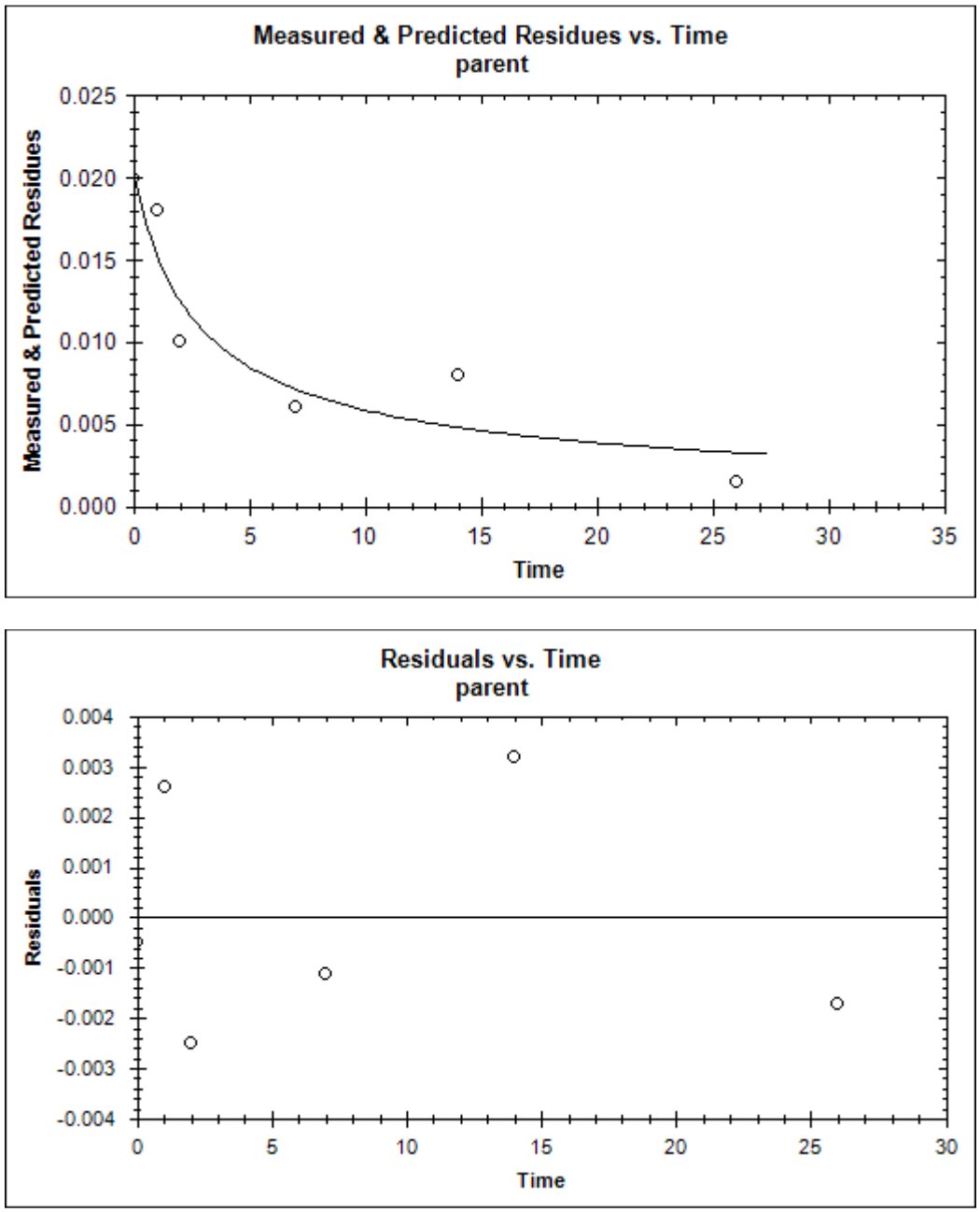


Figure B.8.1-10: FOMC-kinetic for degradation of beta-cyfluthrin in field trial in Spain

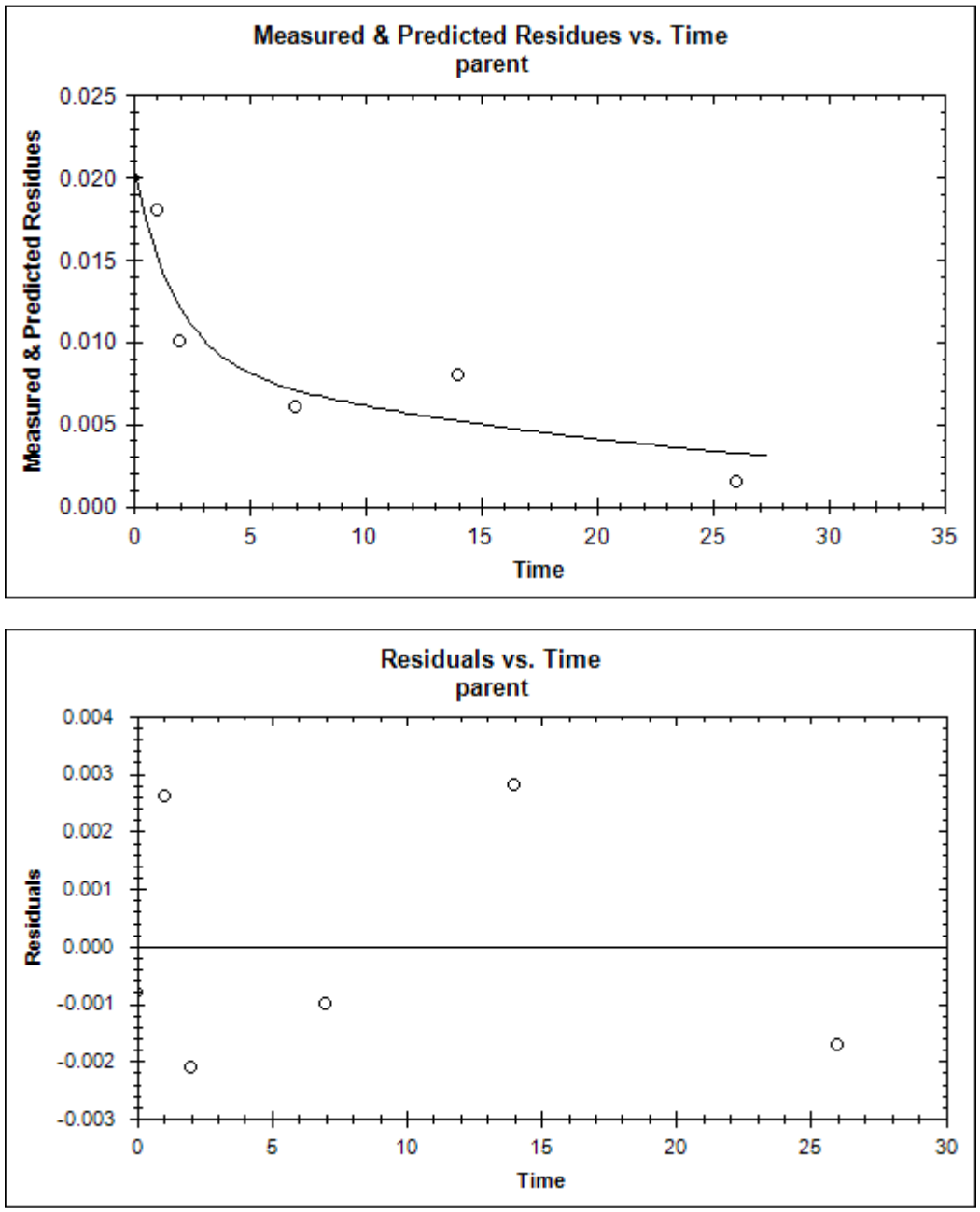
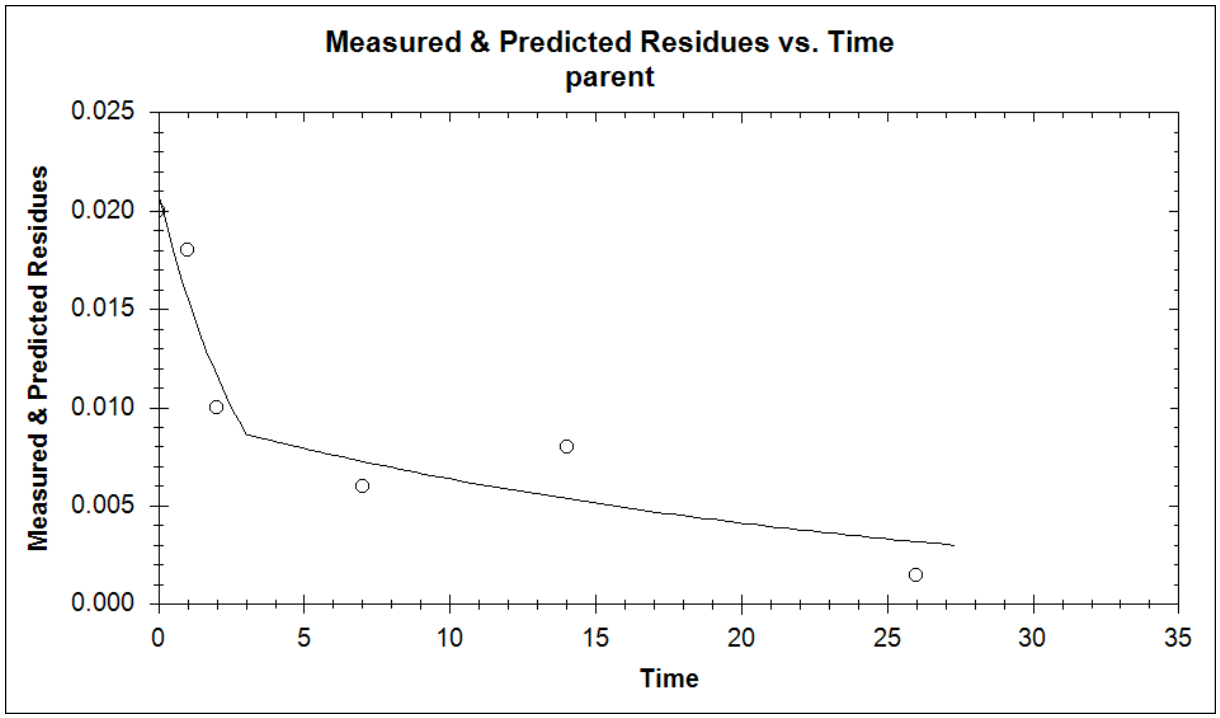


Figure B.8.1-11: DFOP-kinetic for degradation of beta-cyfluthrin in field tial in Spain



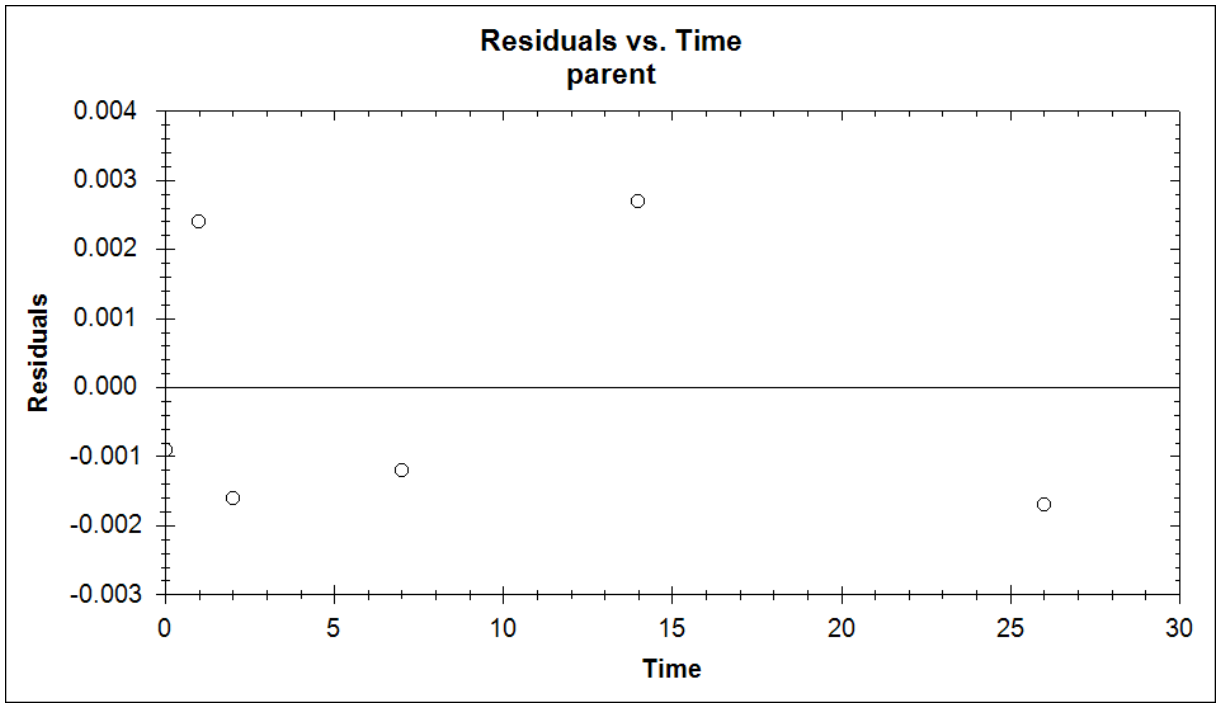
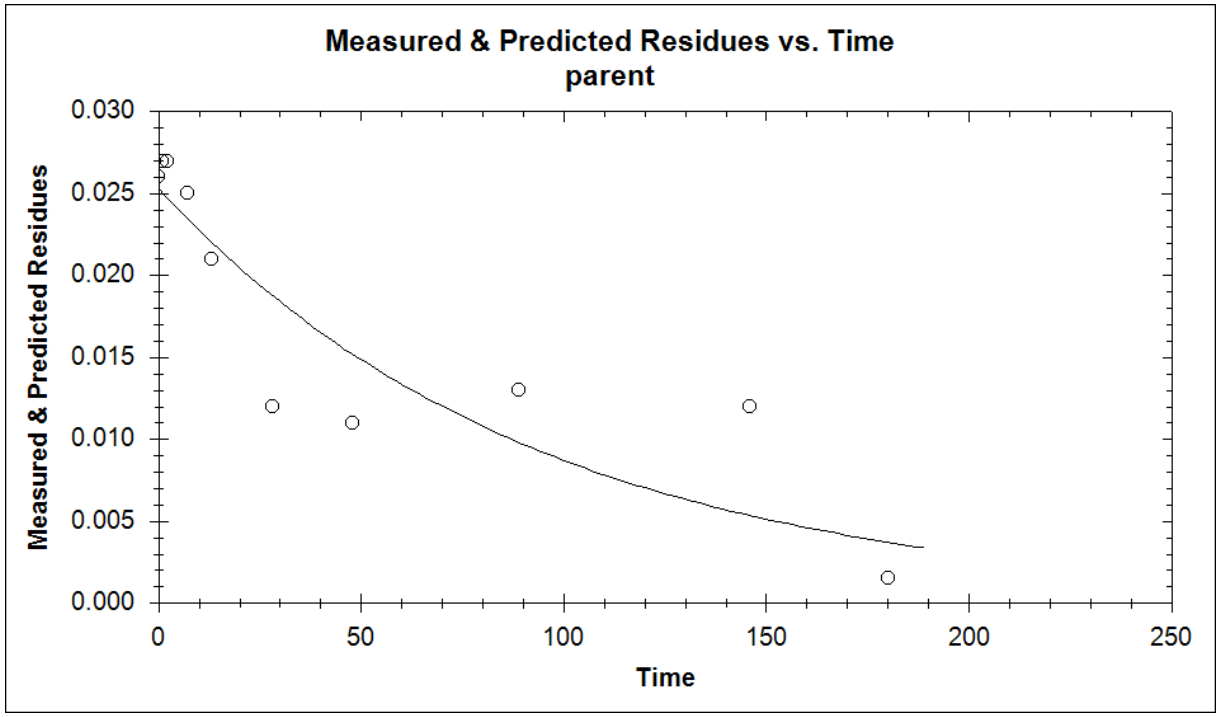


Figure B.8.1-12: HS-kinetic for degradation of beta-cyfluthrin in field trial in Spain



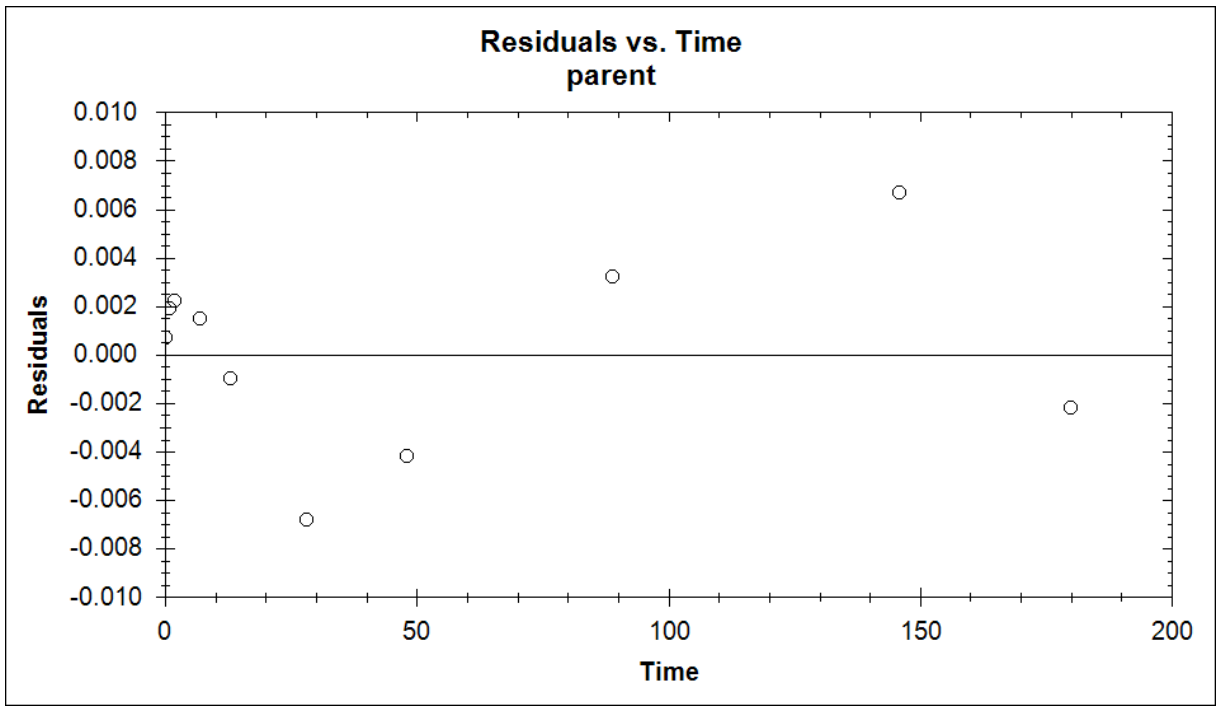
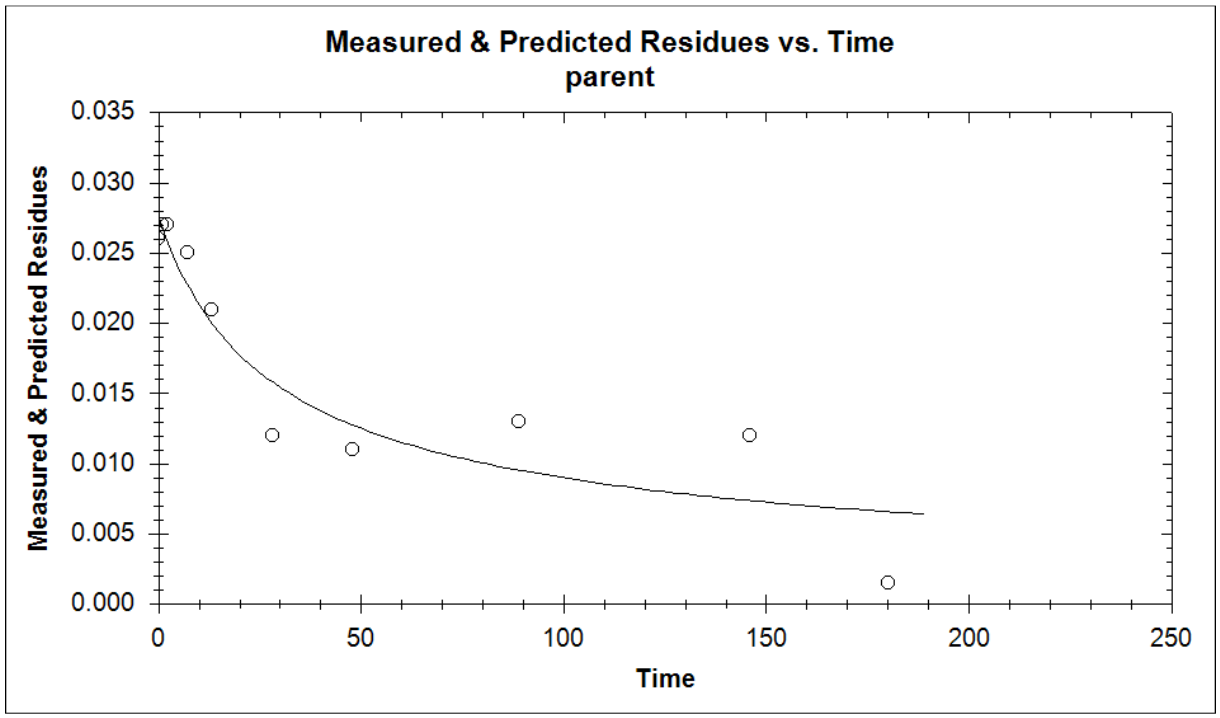


Figure B.8.1-13: SFO-kinetic for degradation of beta-cyfluthrin in field trial in Germany



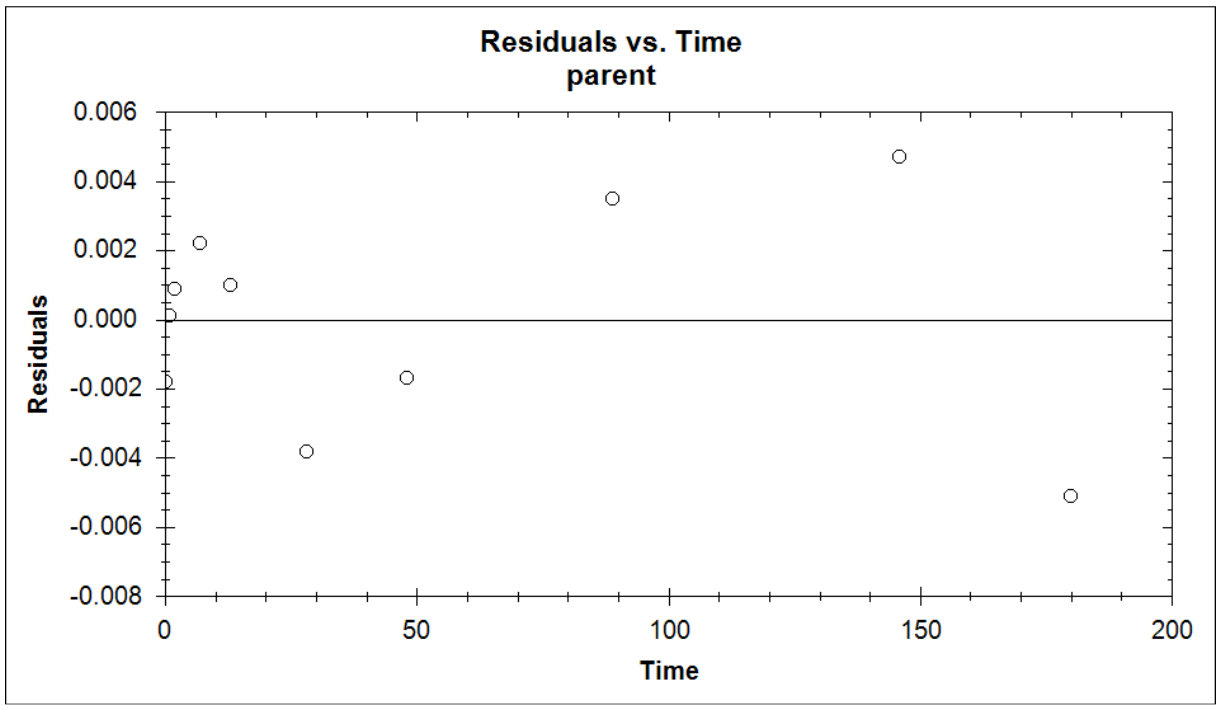
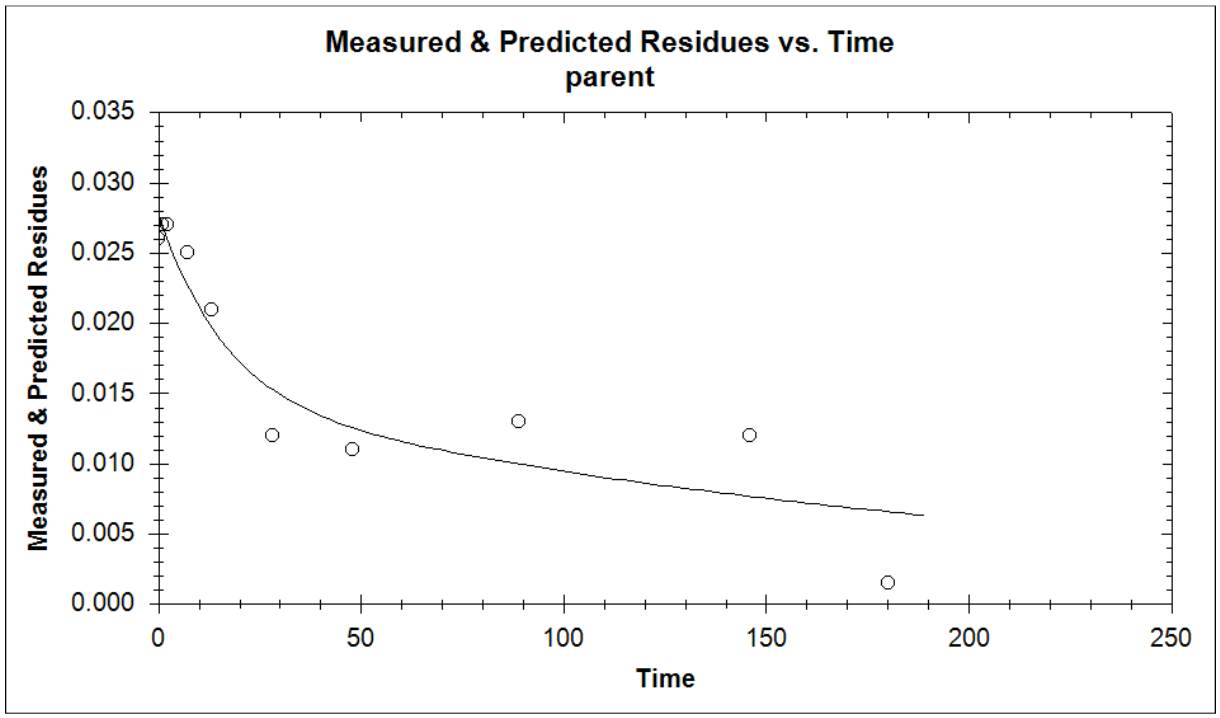


Figure B.8.1-14: FOMC-kinetic for degradation of beta-cyfluthrin in field trial in Germany



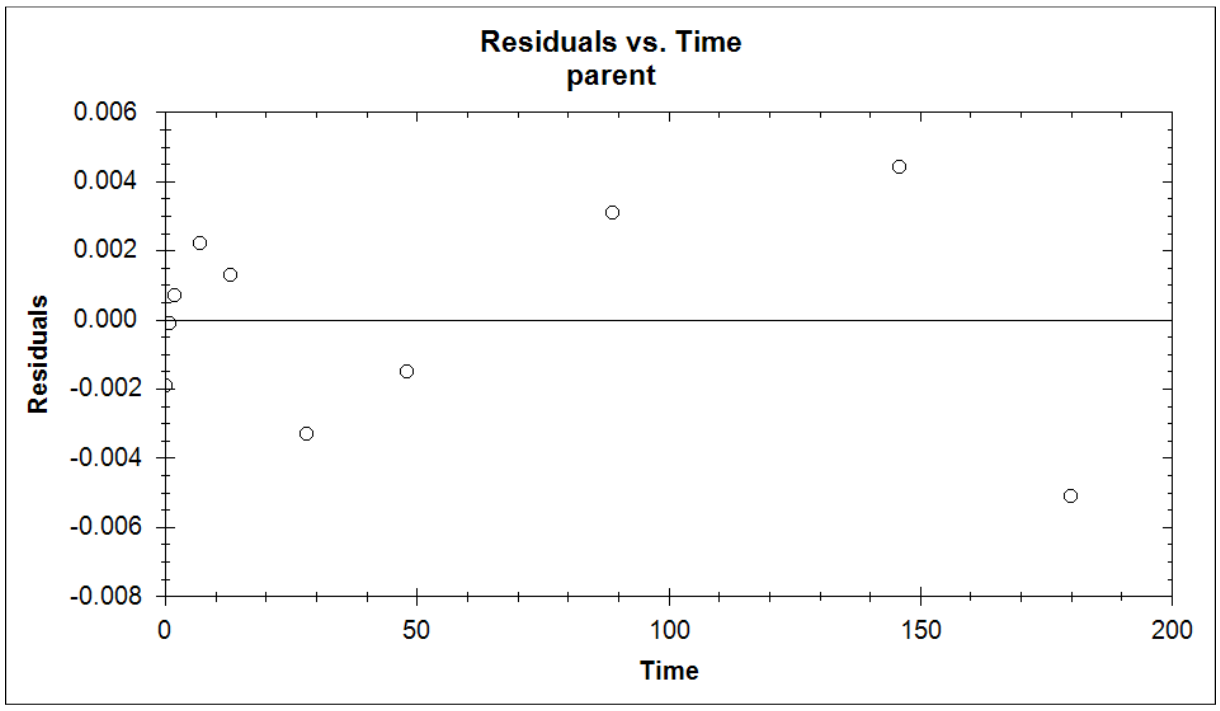
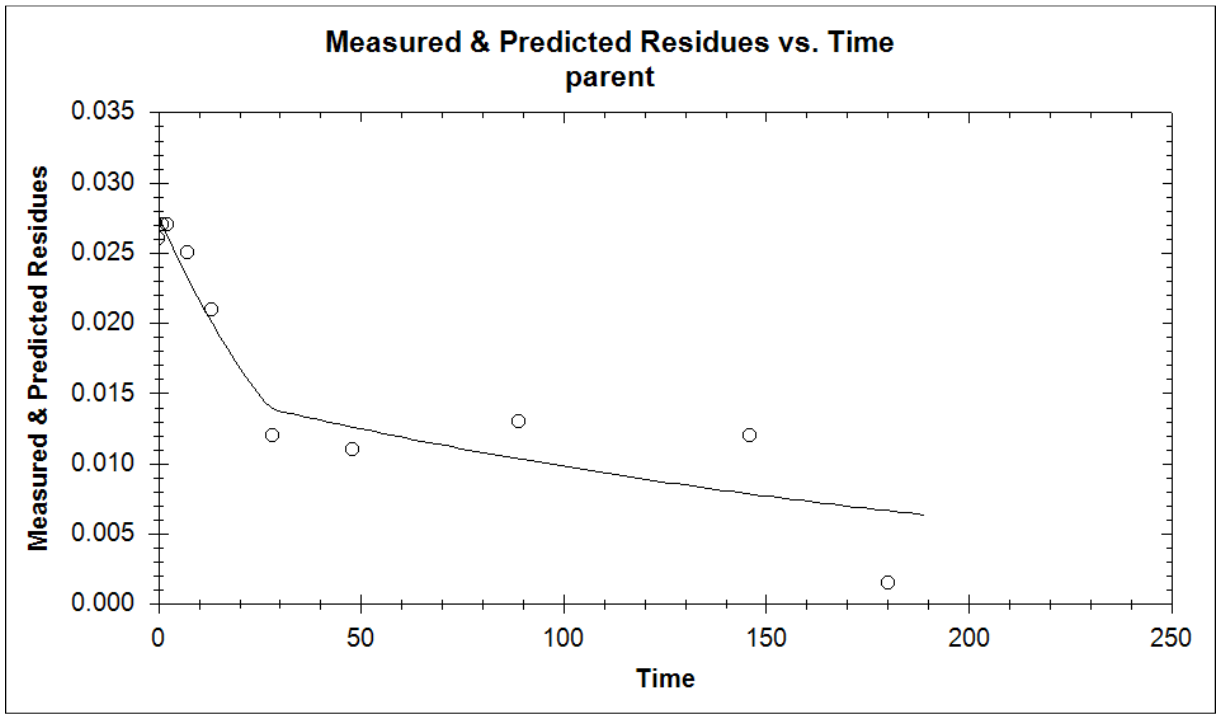


Figure B.8.1-15: DFOP-kinetic for degradation of beta-cyfluthrin in field trial in Germany



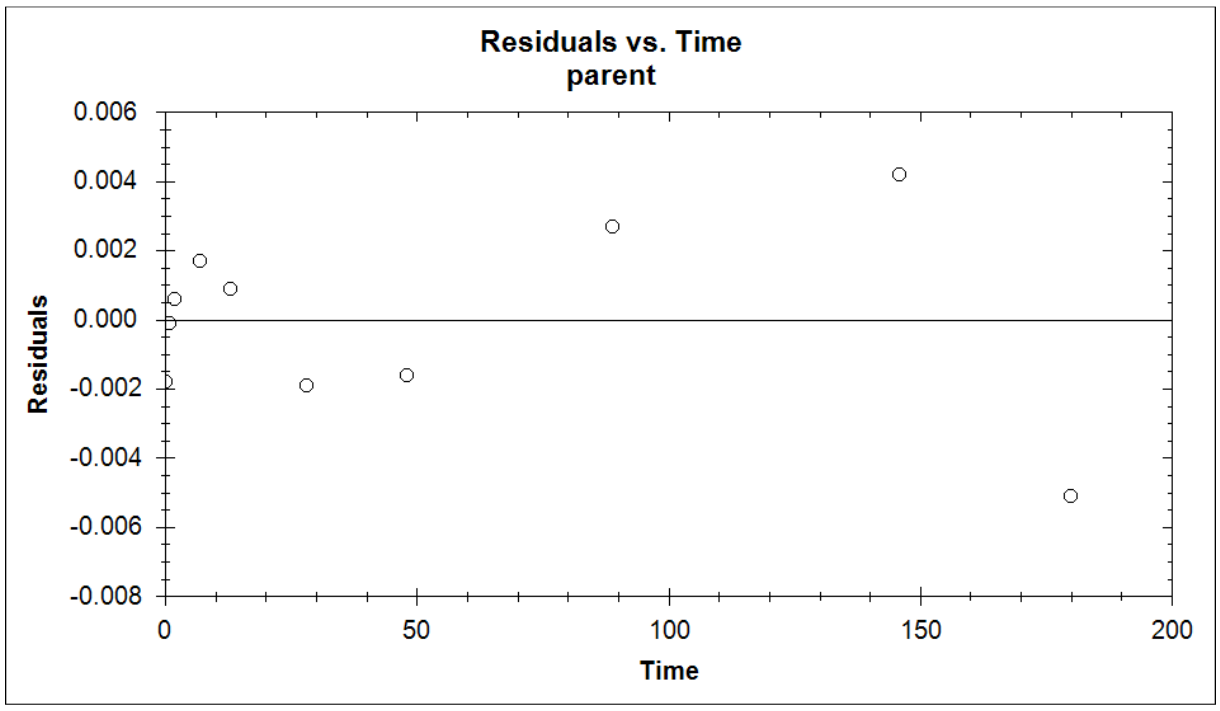


Figure B.8.1-16: HS-kinetic for degradation of beta-cyfluthrin in field trial in Germany

B.8.1.3 Adsorption to and desorption from soil

B.8.1.3.1 Adsorption and desorption of active substance

Adsorption of cyfluthrin was characterised in one soil by Gronberg 1987. In the study by Burhenne 1996, adsorption and desorption of cyfluthrin was characterised in four soils. The studies were evaluated in the monograph from 01 October 1996 and the addendum 1 from 07 May 2002.

As a summary of these two studies the soil characteristics are given in Table B.8.1-54 and results are given in Table B.8.1-55 The means of K_d and K_{oc} given in Table B.8.1-55 are the arithmetic means. The geometric mean is 1216 for K_d and 104491 for K_{oc} .

Table B.8.1-54: Characteristics of test soils used for study of adsorption

Study:	**Gronberg 1987**	**Burhenne 1996**			
Soil:	**greenhouse soil**	**Laacher Hof (Monheim, Germany)**	**Borstel (Germany)**	**Howe (Indiana, USA)**	**Sable-91 (Illinois, USA)**
particle size %					
sand	66	36.9	77.9	65.7	35
silt	32	51.1	18.5	26.4	36
clay	2	12.0	3.6	7.9	29
texture	sandy loam	silt loam	loamy sand	loamy sand	clay loam
organic carbon	1.26	0.9	0.69	1.12	2.44
max. water holding capacity g/100 g	-	14.0	-	34.3	-
pH (0.01 M $CaCl_2$)	5.1	7.3	6.0	6.7	-
pH (H_2O)	-	8.1	5.9	6.7	6.5
cation exchange capacity meq/100g	17	-	5.0	10	27.7
bulk density g/cm^3	2.6	-	2.58	2.53	1.08

Table B.8.1-55: Adsorptions- and desorptions data for cyfluthrin

study	Gronberg 1987	Burhenne 1996				
soil	greenhouse soil sandy loam	Laacher Hof silt loam	Borstel loamy sand	Howe loamy sand	Sable-91 clay loam	
organic carbon %	1.26	0.9	0.69	1.12	2.44	
Test substance and nominal concentration	[phenyl-^{14}C]cyfluthrin, 1 µg/L	[phenyl-^{14}C]cyfluthrin, 5.9 µg/L				
soil-solution ratio	1 : 5	1 : 20				
						mean
K_d adsorption mL/g	sample A: 1063 sample B: 735 sample C: 633 **mean: 810**	1116	1244	1321	1793	**1257**

K_{oc} adsorption mL/g	64286	124000	180290	117946	73484	**112000**
k_d desorption mL/g	-	1448	974	1307	1705	**1359**
K_{oc} desorption mL/g	-	160889	141159	116696	69877	**122155**

The water solubility of cyfluthrin is low (about 6 µg/L) and the sorption to soil is high, therefore no Freundlich isotherms was determined and the distribution coefficients K_d at one concentration were determined. For adsorption to five soils an arithmetic mean $K_{d\ oc}$ of 112000 mL/g was calculated, and for desorption from four soils an arithmetic mean $K_{d\ oc}$ of 122155 mL/g.

Comment

Both studies were conducted with cyfluthrin as test substance in one concentration. The study by Burhenne 1996 was conducted according to GLP standards. The study by Gronberg 1987 is a non-GLP study. It is obvious that the test substance absorbs very strong to all soils tested. The RMS cannot see that a new study using a batch equilibrium method according to new guideline or including a certification of GLP will provide any relevant information. Therefore no new studies are required.

B.8.1.3.2 Adsorption and desorption of of metabolite FPB-acid (= AEF105561)

Two new studies (Oddy&Brett 2005 and Hiler 2013d) are available.

B.8.1.3.2/1 (Oddy&Brett 2005)

Reference	:	Oddy, A. and Brett, R.	Incubation time		2 hours
Title	:	[^{14}C]-AE F105561: adsorption to and desorption from five soils	Soils		5 soils from USA and Germany
Report no.		CX/05/054			
Date of execution	:	5 December 2005	Nominal concentration		0.005, 0.01, 0.05, 0.1 and 0.5 mg/L
GLP statement	:	Yes	Temperature	:	20 °C
Guideline	:	OECD 106 (2000) US EPA 163-1 PMRA Environm.Chem. Fate Guidelines for registration of pesticides in Canada 1987	Moisture		not relevant
Test substance	:	[^{14}C]-AE F105561	Kf/Kfoc, 1/n	:	see results
Purity	:	Radiochemical purity 99 %.		:	
Test system	:	adsorption/desorption study (standard batch equilibrium method)	Acceptability	:	acceptable

Executive Summary

The adsorption/desorption of [^{14}C]FPB-acid (AE F105561 = COE 538/78) was investigated in a batch equilibrium experiment using 2 US soils and 3 Germany soils (pH range of 5.3 to 6.5). The adsorption of [^{14}C]FPB-acid was tested at concentrations of approximately 0.5, 0.1, 0.05, 0.01 and 0.005 mg/L in 0.01 M $CaCl_2$ in the dark and at 20 ± 2 °C. Different soil/solution ratios were used. The amount of the applied test material adsorbed in soils ranged from 21.9 % to 82.0 %. The calculated Freundlich adsorption constants K_F ranged from 0.65 to 1.80 mL/g, with corresponding adsorption constants ($K_{F,oc\ ads}$ values) ranging from 39 to 123 mL/g for the five test soils (arithmetic mean 73 mL/g). The Freundlich exponents, 1/n, displayed a degree of nonlinearity in all five soils tested with values ranging from 0.60 to 0.75. The Freundlich desorption coefficient $K_{F\ des}$ values ranged from 0.89 to 2.32 mL/g with corresponding desorption KF,oc des values ranging from 54 to 232 mL/g for the five test soils (arithmetic

mean 106 mL/g). The overall material balances from the supernatants after adsorption, desorption, solvent extraction and the combustion of the remaining soils were determined and ranged from 91 to 99 % of applied radioactivity (AR).

Materials and methods

Test material (radiolabelled)
Identification: [^{14}C]-AE F105561 (FPB-acid)
Description: Solid
Specific Radioactivity: 9.02 MBq/mg
Radiochemical Purity: 99 %
Storage: < -15 °C in the dark

Test material (non-radiolabelled)
Identification: AE F105561
Description: White needles
Purity: > 94 %
Expiry date: 19 April 2007

Soils

Five agricultural soils (top 20 cm for all soils) were used for the study. On reception at the laboratory, the soils were stored in the dark at a temperature of about 4 °C. The moisture content of each soil was determined prior to use in the study by comparing weights of soil before and after oven-drying. The characteristics of the test soils is given in Table B.8.1-56.

Table B.8.1-56: Characteristics of test soils used for study of adsorption of metabolite FPB-acid

Soil	**05/012 Pikeville North Carolina USA**	**05/013 Stanley USA**	**05/017 Höfchen, Germany**	**05/018 Laacher Hof, Germany**	**05/019 Wurmwiese, Germany**
particle size %					
sand (50 – 2000 µm)	68	36	11.6	72.6	47.3
silt (2 – 50 µm)	20	36	70.5	17.5	36.9
clay (< 2 µm)	12	28	17.9	9.9	15.8
texture (USDA)	sandy loam	clay loam	silt loam	sandy loam	loam
organic carbon	1.0	2.1	2.07	1.64	2.08
organic matter	1.7	3.6	3.56	2.82	3.58
water holding capacity g/100 g pF 0.05 (max.) pF 2.0 (0.1 bar)	 - 17.7	 - 52.8	 57.1 -	 43.6 -	 54.1 -
pH (0.01 M $CaCl_2$)	5.3	5.7	6.5	6.1	5.6
pH (H_2O)	6.1	6.4	7.24	6.8	6.4
cation exchange capacity meq/100g	5.4	23.6	12.8	7.3	9.6
bulk density g/cm^3	1.45	1.13	1.09	1.15	1.1

Description of analytical procedures

The concentration of [^{14}C]FPB-acid in the adsorption and desorption solutions was determined by LSC. Following desorption, the soils were extracted 2 times with acetonitrile/water solution (4:1 v/v). The concentrations in the extracts were measured by LSC. Radioactivity in residual soil samples was determined by combustion followed by LSC. The limit of quantification (LOQ) for LSC was 0.00002 µg/g.

Selected supernatants and extracts from the preliminary experiments were chromatographic analysed using HPLC coupled with an UV and ^{14}C-detector in order to confirm the stability of the test substance in the test system. The limit of quantification was equal to 0.003 % of the applied amount at the nominal 0.5 mg/L level.

Results and discussion

The adsorbed and desorbed amount of [^{14}C]FPB-acid at each concentration is provided in percentage of applied radioactivity (% AR, mean of two replicates) in Table B.8.1-57 and Table B.8.1-58.

Table B.8.1-57: Adsorption data of metabolite FPB-acid (mean of 2 replicates)

Concentration	Soil (mg/kg)	Solution (mg/L)	K_d*	% Adsorbed
	Pikeville (soil : solution ratio 1 : 5)			
0.5 mg/L	0.627	0.404	1.55	23.5
0.1 mg/L	0.170	0.073	2.33	31.5
0.05 mg/L	0.101	0.033	3.06	37.9
0.01 mg/L	0.023	0.006	3.83	45.4
0.005 mg/L	0.014	0.002	7.00	53.6
	Stanley (soil : solution ratio 1 : 10)			
0.5 mg/L	1.213	0.413	2.94	22.7
0.1 mg/L	0.331	0.075	4.41	30.7
0.05 mg/L	0.212	0.032	6.63	40.1
0.01 mg/L	0.065	0.004	16.25	63.4
0.005 mg/L	0.037	0.001	37.00	72.4
	Höfchen (soil : solution ratio 1 : 5)			
0.5 mg/L	0.737	0.384	1.92	27.5
0.1 mg/L	0.188	0.070	2.69	34.8
0.05 mg/L	0.104	0.032	3.25	39.0
0.01 mg/L	0.034	0.003	11.33	68.7
0.005 mg/L	0.020	0.001	20.00	78.2
	Laacher Hof (soil : solution ratio 1 : 3)			
0.5 mg/L	0.352	0.419	0.84	21.9
0.1 mg/L	0.096	0.076	1.26	29.7
0.05 mg/L	0.055	0.036	1.53	34.4
0.01 mg/L	0.013	0.006	2.17	42.9
0.005 mg/L	0.008	0.002	4.00	54.5
	Wurmwiese (soil : solution ratio 1 : 5)			
0.5 mg/L	0.839	0.362	2.32	31.4
0.1 mg/L	0.232	0.061	3.80	42.9
0.05 mg/L	0.137	0.025	5.48	51.8
0.01 mg/L	0.038	0.003	12.67	73.3
0.005 mg/L	0.021	0.001	21.00	82.0

* K_d calculated by RMS from mean values: K_d = [soil (mg/kg)] / [solution (mg/L)]

Table B.8.1-58: Desorption data of metabolite FPB-acid

Soil	% AR desorbed				
	0.5 mg/L	**0.1 mg/L**	**0.05 mg/L**	**0.01 mg/L**	**0.005 mg/L**
Pikeville	67.8	41.8	37.3	31.7	27.5
Stanley	74.3	42.2	33.5	18.1	15.6
Höfchen	72.5	38.2	35.5	16.9	13.6
Laacher Hof	75.7	45.4	40.1	34.3	24.7
Wurmwiese	69.0	36.6	39.1	17.2	13.5

Adsorption coefficients (K_F) ranged from 0.65 to 1.80 with corresponding adsorption constants (K_{OC}

values) ranging from 39 to 123 for the five test soils. Desorption coefficient $K_{f\,des}$ ranged from 0.89 to 2.32 with corresponding desorption constants ($K_{OC\,des}$ values) ranging from 54 to 232 for the five test soils. Individual results are given in Table B.8.1-59.

Table B.8.1-59: Freundlich adsorption/desorption data of metabolite FPB-acid

Soil	% oc	Adsorption			Desorption		
		K_F	1/n	$K_{F\,oc}$	$K_{F\,des}$	1/n	$K_{F\,oc\,des}$
Pikeville	1.0	1.23	0.749	123	2.32	0.77	232
Stanley	2.1	1.80	0.60	86	2.13	0.571	101
Höfchen	2.07	1.03	0.595	50	1.22	0.584	59
Laacher Hof	1.64	0.65	0.733	39	0.89	0.710	54
Wurmwiese	2.08	1.39	0.609	67	1.76	0.609	85

oc : organic carbon

Mass balance

The overall recovery expressed as percentage of applied radioactivity after adsorption and desorption in the definitive study was in the range of 91.0 to 99.4 %.

Transformation of parent compound

For periods longer than 6 hours it was not possible to demonstrate a satisfactory parental mass balance due to lower extractability and a degree of instability of FPB-acid. In order to ensure the stability of the test item an adsorption equilibrium time of 2 hours was used in the main study. Therefore the determined KF,OC ads represent a lower limit, due to the necessary restriction on the adsorption period used in the study.

Conclusions

The K_d values in Table B.8.1-57 clearly reflect an increase of adsorption at lower dose rates in all three soils. The Freundlich exponent is 0.657 (mean of n=5) and therefore at the lower end of the general range of 0.7 – 1.0. The study is acceptable.

B.8.1.3.2/2 (Hiler 2013d)

Reference	:	Hiler, Tammy	Incubation time		48 hours
Title	:	Soil adsorption/desorption of [^{14}C]-FPB acid by the batch equilibrium methods	Soils		3 soils from USA
Report no.		2381W-1			
Date of execution	:	1 October 2013	Nominal concentration		0.01, 0.05, 0.11 and 0.54 and 1.05 mg/L
GLP statement	:	Yes	Temperature	:	20 °C
Guideline	:	OECD 106 (2000) US EPA OCSPPP 835.1230	Moisture		not relevant
Test substance	:	[^{14}C]FPB-acid	Kf/Kfoc, 1/n	:	see results
Purity	:	Radiochemical purity > 99 %.		:	
Test system	:	adsorption/desorption study (standard batch equilibrium method)	Acceptability	:	acceptable

Executive Summary

The adsorption/desorption of FPB-acid was investigated in a batch equilibrium experiment using 3 US

soils. The adsorption phase of the study was carried out with radiolabelled FPB-acid at concentrations of approximately 1.0, 0.5, 0.1, 0.05 and 0.01 mg/L at 20 °C for 48 hours for all soils. Different soil/solution ratios were used. The desorption phase of the study was carried out for 48 hours with fresh 0.01 M $CaC1_2$ applied to pre-adsorbed soil. Adsorption coefficients ($K_{F\,ads}$ values) ranged from 0.62 to 14.42 mL/g with corresponding adsorption constants ($K_{F,OC\,ads}$ values) ranging from 124 to 424 mL/g for the three test soils. The arithmetic mean of the Koc values was 241 mL/g. The Freundlich exponents, 1/n, ranged from 0.56 to 0.80. The Freundlich desorption coefficient $K_{F\,des}$ value for soil North Dakota was 22.6 mL/g with a corresponding desorption $K_{F,oc\,des}$ value of 665 mL/g. No desorption values were calculated for the California or Illinois soils due to the fact that no desorption occurred (adsorption was considered irreversible in these soils).
For the definitive study, the overall mass balance total recovery ranged from 89.9 % to 106.5 %, except for the soil Illinois at 0.01 mg/L, which averaged 83.3 %.

Materials and methods

Test material (radiolabelled)
Identification: [Phenoxy-UL-^{14}C]FPB-acid
Specific Radioactivity: 9.02 MBq/mg
Radiochemical purity: > 99

Reference material (non-radiolabelled)
Identification: FPB acid
Purity: 99.6 %
Expiry date: 23 March 2016

Soils

Three agricultural soils were used for the study (see Table B.8.1-60).The soils were stored either refrigerated at ~ 4 °C or at room temperature upon receipt at the laboratory in plastic bags open to air. Prior to use in the study, the soils were passed through a 2-mm sieve and the sieved soils were stored in plastic bags.

Table B.8.1-60: Characteristics of test soils used for study of adsorption of metabolite FPB-acid

Soil:	North Dakota Grand Forks County, USA	California, Madera, USA	Illinois, Clinton County
particle size %			
sand (50 – 2000 µm)	58	63	15
silt (2 – 50 µm)	14	22	60
clay (< 2 µm)	28	15	25
texture (USDA)	sandy clay loam	sandy loam	silt loam
organic carbon	3.4	0.52	1.04
organic matter	5.9	0.89	1.8
water holding capacity g/100 g 0.1 bar	37.1	14.6	30.9
pH (0.01 M $CaCl_2$)	4.9	7.1	5.5
cation exchange capacity meq/100g	19.0	9.9	11.6
bulk density g/cm^3	0.94	1.19	1.03

Study design

Experimental conditions

The test systems were maintained at a temperature of 20 °C throughout the study. Dose solutions were prepared using aliquots of a [^{14}C]FPB-acid stock solution in acetonitrile. The respective aliquots were

evaporated to dryness and the remainder was diluted in 0.01 M $CaCl_2$ to obtain dose solutions at the required concentrations. The following concentrations were tested in the definitive test: 0.01, 0.05, 0.11, 0.54 and 1.05 mg/L.

Data from a preliminary study indicated an adsorption range of 20 to 80 % using a 1:10 soil to solution ratio (2 g soil : 20 mL aqueous solution) for soil North Dakota, a 1:1 soil to solution ratio (5 g soil : 5 mL aqueous solution) for soil California and a 1:2 soil to solution ratio (5 g soil : 10 mL aqueous solution) for soil Illinois. Based on the results from the preliminary study, the equilibration time for the adsorption and desorption cycles was 48 hours, respectively.

To determine the adsorption and desorption isotherms, the soil aliquots were pre-equilibrated overnight by shaking with the necessary volume of 0.01 M $CaCl_2$ solution (in duplicate). Thereafter, appropriate volumes of the dose solutions were added and all samples were shaken for 48 hours on a rocker basket for the adsorption phase. Then, the test samples were centrifuged. Supernatants were aliquoted for radioassay by LSC. The volume of the adsorption solution was determined gravimetrically by weighing the containers immediately after dosing and after decantation.

The desorption phase was performed on the remaining soil following removal of the adsorption supernatant, with the addition of 0.01 M $CaCl_2$ solution equivalent to the volume removed during the adsorption phase. Following desorption equilibrium (after 48 hours of shaking), the supernatants were decanted following centrifugation and aliquoted for radioassay. The amount of desorption solution remaining in the soil pellet was determined by weighing each tube after decanting the desorption solutions.

Following desorption, both replicates of the 0.01 to 0.10 mg/L samples (ND and IL soils), and the 0.01 mg/L samples (CA soil) were extracted with acetone : 0.01 M HCl (9:1, v/v) followed by shaking for 60 minutes. Following centrifugation the supernatants were decanted and the extraction procedure was repeated an additional two times. Volumes of combined extracts were measured and triplicate aliquots (1 mL) were taken for radioassay by LSC. At least one replicate of the adsorption and desorption solutions from the 1 mg/L samples as well as one soil extract replicate were analysed by HPLC with radiodetection.

Description of analytical procedures

The concentration of [^{14}C]FPB-acid in adsorption and desorption solutions as well as in soil extracts was determined by LSC. Radioactivity in residual soil samples was determined by combustion followed by LSC. The limit of detection for LSC was 0.0001 mg/L in adsorption and desorption solutions and 0.001 ppm for the combusted soil samples. Supernatants and extracts were chromatographically analysed using HPLC coupled with an UV and ^{14}Cdetector in order to confirm the stability of FPB-acid in the test systems. For radiodetection, the limit of quantification for a single peak was about 1 % of AR.

Results and discussion

The adsorbed amount of FPB-acid at each concentration is provided in percentage of applied radiocarbon (% AR) in Table B.8.1-61.

The results from Freundlich isotherm experiments are given in Table B.8.1-62. Adsorption coefficients (K_F) ranged from 0.62 to 14.42 with corresponding adsorption constants ($K_{F,OC}$) ranging from 124 to 424 for the three test soils. The Freundlich desorption coefficient $K_{F\,des}$ for soil North Dakota was 22.6 with a corresponding desorption $K_{F,OC\,des}$ value of 665. Desorption values were not calculated for the California or Illinois soils due to the fact that no desorption occurred (adsorption was considered irreversible in these soils).

Mass balance

The overall mass balance total recovery ranged from 89.9 % to 106.5 %, except for the soil Illinois at a

dose rate of 0.01 mg/L, which averaged 83.3 %.

Transformation of parent compound

[^{14}C]FPB-acid was stable in the control solution (no soil present) for the duration of the equilibration period of 48 hours. [^{14}C]FPB-acid was also stable in all adsorption solutions, desorption solutions and soil extracts.

Table B.8.1-61: Mass balance and adsorption of metabolite FPB-acid on soil

Dose rate (µg/mL)	Replicate	Mass balance (% applied radioactivity)			
		% AR in solution (ads test)	% AR in solution (desorption test)	% AR in soil	Total recovery
North Dekota soil (soil-solution ratio 1 : 10)					
0.01	A	6.6	2.7	87.4	96.7
	B	6.7	2.5	96.4	105.6
0.05	A	7.9	3.5	95.1	106.5
	B	8.2	3.7	89.3	101.2
0.11	A	10.5	4.3	87.4	102.2
	B	8.8	3.3	88.7	100.8
0.54	A	26.9	11	53.4	91.3
	B	27.9	10.9	52.8	91.6
1.05	A	33	14.9	47.5	95.4
	B	34.3	14.8	46.4	95.5
California soil (soil-solution ratio 1 : 1)					
0.01	A	25.5	8.9	55.5	89.9
	B	24.1	8.4	56.5	89.0
0.05	A	35.8	14.6	39.9	90.3
	B	37.0	15.4	37.8	90.2
0.11	A	37.5	16.3	38.3	92.1
	B	39.1	16.6	41.1	96.8
0.54	A	45.9	20.6	32.4	98.9
	B	46.0	20.7	32.8	99.5
1.05	A	50.5	21.2	29.6	101.3
	B	49.7	21.4	31.1	102.2
Ilinois soil (soil-solution ratio 1 : 2)					
0.01	A	3.6	1.2	78.5	83.3
	B	3.6	1.3	78.4	83.3
0.05	A	6.3	1.6	82.3	90.2
	B	7.0	1.6	83.4	92.0
0.11	A	11.7	1.8	77.6	91.1
	B	11.8	1.8	81.5	95.1
0.54	A	34.9	10.0	47.8	92.7
	B	35.2	9.2	48	92.4
1.05	A	41.3	12.9	10.8	65.0
	B	41.4	12.9	41.2	95.5

Table B.8.1-62: Equilibrium concentrations of metabolite FPB-acid in solution and on soil (mean of 2 replicates)

Dose rate (µg/mL)	Adsorption						Desorption		
	solution µg/mL	soil µg/g	K_d	K_F	$K_{F\,oc}$	1/n	K_F	$K_{F\,oc}$	1/n
North Dekota soil (organic carbon 3.4 %)									
0.01	0.0007	0.0932	133.14	14.42	424	0.6636	22.61	665	0.6212
0.05	0.0045	0.4599	102.2						
0.11	0.0103	0.8730	84.76						

0.54	0.1569	3.9120	24.93						
1.05	0.3723	6.9365	18.63						
California soil (organic carbon 0.5 %)									
0.01	0.0038	0.0076	2.00	0.62	124	0.7991	no desorption occured from these soils		
0.05	0.0301	0.0348	1.16						
0.11	0.0629	0.0667	1.06						
0.54	0.3783	0.2915	0.77						
1.05	0.7885	0.5220	0.66						
Illinois soil (organic carbon 1.0 %)									
0.01	0.0004	0.0194	48.5	1.76	176	0.5605	no desorption occured from these soils		
0.05	0.0041	0.0944	23.02						
0.11	0.0140	0.1714	12.24						
0.54	0.2279	0.7014	3.08						
1.05	0.5202	1.2241	2.35						

Comment

The K_d values in Table B.8.1-62 clearly reflect an increase of adsorption at lower dose rates in all three soils. The Freundlich exponent is 0.674 (mean of n=3) and therefore at the lower end of the general range of 0.7 – 1.0. The study is acceptable.

Comment on pH-dependency of adsorption of metabolite FPB-acid

Six out of eight soils from both studies (n = 8) cover a pH($CaCl_2$)-range between 5.3 and 6.5. The adsorption in the pH-ranges above or below that range cannot be well understood because there is only one slightly alkaline soil (pH 7.1) and one very acid soil (pH 4.9) from the study by Hiler 2013d. Despite these restrictions a pH-dependency can be assumed both for K_f and K_{foc} : There is a slight increase of adsorption with decreasing pH between pH 6.5 and pH 5.3. A strong decrease below pH 5.3 can be assumed but is very uncertain due to only one very acid soil.

As a consequence, the RMS proposes to calculate the mean of soils pH 6.1 – 7.1 (geomean K_{foc} = 106, n = 3) which represents adsorption in slightly acid and neutral soils and the mean of soils pH 5.3 – 5.7 (geomean K_{foc} = 62, n = 4) which represents all acid and very acid soils pH < 6.1. The adsorption data of the very acid soil North Dakota (pH 4.9) are proposed not to use in modelling or for trigger endpoint due to high uncertainty.

B.8.1.3.3 Adsorption of metabolite DCVA (= permethrin acid)

The study by **Slangen 1999** was evaluated in the addendum dated 07 May 2002 to the monograph. A short summary of characteristics of soils and of the results is given in Table B.8.1-63. The study was evaluated in the addendum to be acceptable.

Table B.8.1-63: Characteristics of soils and results on adsorption of metabolite DCVA on soils (short summary)

Soil	Speyer 2.1	Cranfield 115	Cranfield 230
texture (USDA)	sand	clay loam	sandy loam
particle size %			
sand (53 – 2000 µm)	89.5	45	71.1
silt (2 – 53 µm)	8.0	23	18.9
clay (< 2 µm)	2.5	32	10
organic carbon	0.59	1.6	0.8
organic matter	1.02	2.76	1.4
pH (water)	6.9	8.1	5.1
pH ($CaCl_2$)	6.0	7.5	4.3

cation exchange capacity meq/100g	4	25.9	10.6
soil-solution ratio	1 : 1		
dose rate (µg/mL)	0.04, 0.1, 0.5, 1.0		
$K_{F\ ads}$	0.184	0.224	2.893
$K_{F\ oc\ ads}$	31.05	13.95	356.15
1/n	0.884	0.871	0.957
$K_{F\ des}$	0.676	0.498	5.678
$K_{F\ oc\ des}$	114.19	31.11	699.17

A **new study** on adsorption of metabolite DCVA (= BCS-AA53389) by Hein & D'Ambrosio 2013 is available.

B.8.1.3.3/1 (Hein & Dambrosio 2013)

Reference	:	Hein, Werner and D'Ambrosio, Anja	Incubation time		24 hours
Title	:	[cyclopropane-1-^{14}C]-BCS-AA53389: adsorption/desorption in five different soils	Soils		5 soils from Germany
Report no.		AS251			
Date of execution	:	4 April 2013	Nominal concentration		0.01, 0.03, 0.1 and 0.3 and 1.0 mg/L
GLP statement	:	Yes	Temperature	:	20 °C
Guideline	:	OECD 106 (2000) US EPA OPPTS 835.1230	Moisture		not relevant
Test substance	:	[^{14}C] BCS-AA53389 (= DCVA, permetric acid)	Kf/Kfoc, 1/n	:	see results
Purity	:	Radiochemical purity > 99 %.		:	
Test system	:	adsorption/desorption study (standard batch equilibrium method)	Acceptability	:	acceptable

Executive Summary

The adsorption/desorption characteristics of [cyclopropane-1-^{14}C]DCVA (permethric acid) were studied in five soils with a pH range of 5.1 to 7.3 using batch equilibrium experiments. The adsorption phase of the study was carried out over 24 hours in the dark at 20 °C ± 2 °C. Air-dried soil was pre-equilibrated and thereafter [cyclopropane-1-^{14}C]DCVA was applied at nominal concentrations of 1.00, 0.30, 0.10, 0.03, and 0.01 mg/L. The equilibration solution used was 0.01 M aqueous $CaCl_2$ (with 50 mg/L $HgCl_2$ in soil Dollendorf). The soil-solution ratio was 1:2 (1:1 for Höfchen and Laacher Hof). For the desorption phase soils were supplied with fresh 0.01 M aqueous $CaCl_2$ solution (soil Dollendorf addition of 50 mg/L $HgCl_2$) and shaken for 24 hours. In the definitive adsorption test 32.2 - 65.4 % of DCVA was adsorbed to soils. The calculated adsorption constants $K_{F\ ads}$ of the Freundlich isotherms for the five test soils ranged from 0.4 to 1.9. The Freundlich exponents 1/n were in the range of 0.7429 to 0.8878, indicating that the concentration of the test item did affect the adsorption behaviour. The normalised $K_{F,OC}$ ads values ranged from 9.0 to 74.3 (mean $K_{F,OC}$ ads: 37.9 mL/g). At the end of one adsorption and one desorption phase 1.7 - 39.5 % of the initially adsorbed amount were desorbed from soils. The mean desorption K_F des ranged from 0.5 – 2.1 mL/g and the normalised $K_{F,OC}$ des ranged from 12.0 – 82.4 mL/g and were 1.10 – 1.33 times higher than those obtained for adsorption phase. The overall material balance for all concentrations for individual specimens was in the range of 85.2 - 105.0 % of the applied radioactivity.

Material and methods

Test material (radiolabelled)
Identification: [cyclopropane-1-^{14}C]BCS-AA53389 (DCVA or permethric acid)
Description: Solid, dried in vacuo
Specific Radioactivity: 3.22 MBq/mg (87.0 µCi/mg)
Radiochemical Purity: > 99 % (sum of isomers) by TLC
Chemical Purity: > 98 % (sum of isomers) by HPLC
Storage conditions: ≤ -18 °C

Reference material (non-radiolabelled)
Identification: FCR1272-trans-permethric acid
Content: 98.1 %
Expiry date: 02 June 2017

Reference material (non-radiolabelled)
Identification: FCR1272-cis-permethric acid
Content: 99.8 %
Expiry date: 27 May 2017

Soils

Five test soils from Germany were used within this study, chosen to cover a representative range in soil physico-chemical properties. After collection (0-20 cm) all soils were air-dried and sieved (2 mm). At the test facility, the soils were stored in plastic containers at 1 – 10 °C for up to 637 days. The moisture content of each soil was determined prior to use in the study by comparing weights of soil before and after airdrying. The characteristics of the soils is given in Table B.8.1-64.

Table B.8.1-64: Characteristics of test soils used for study of adsorption of metabolite DCVA

Soil	Wurmwiese, Germany	Höfchen, Germany	Dollendorf, Germany	Laacher Hof, Germany	Hanscheider Hof, Germay
texture (USDA)	sandy loam	silt loam	loam	loamy sand	silt loam
particle size %					
sand (50 – 2000 µm)	57	27	37	77	31
silt (2 – 50 µm)	30	60	40	16	54
clay (< 2 µm)	13	13	23	7	15
pH (water)	5.4	6.5	7.5	6.2	5.5
pH ($CaCl_2$)	5.1	6.3	7.3	5.9	5.2
organic carbon %	2.0	2.9	4.4	2.0	2.9
organic matter 5	3.44	4.99	7.57	3.44	4.99
cation exchange capacity meq/100g	10.3	12.9	19.2	9.3	10.0

Study design

Experimental conditions

The test systems were maintained in the dark and at a temperature of 20 ± 2 °C. The pH of the equilibrium solutions (0.01 M $CaCl_2$ with or without 50 ppm $HgCl_2$) during this study was found to be in the range of 6.2 to 6.9. A stock solution of [cyclopropane-1-^{14}C]DCVA was prepared in acetonitrile. For preparation of the respective application solutions, aliquots were pipetted in an appropriate flask. The solvent was evaporated and the flasks were filled up with equilibration solution (0.01 M $CaCl_2$, addition

of 50 ppm $HgCl_2$ for soil Dollendorf). The concentrations of the test substance were 1, 0.3, 0.1, 0.03 and 0.01 mg/L. Based on the results of the preliminary testing, the selected soil:solution ratio for the definitive test was 1:2 for soils Wurmwiese, Dollendorf and Hanscheider Hof and 1:1 for the soils Höfchen and Laacher Hof. Duplicate tubes with soil and solution were mixed for 24 hours on a mechanical overhead shaker. Following equilibration for adsorption, the test samples were centrifuged and the supernatant was completely decanted, and the remaining soil was weighed. The desorption phase was performed on the remaining soil following removal of the adsorption supernatant, with the addition of 0.01 M $CaCl_2$ (with or without $HgCl_2$) equivalent to the volume removed after the adsorption phase. The test vessels were shaken for the same period as for adsorption (24 hours) and handled analogously. At the end of the desorption cycle all soil residues were mixed with approximately 0.4 g cellulose/g soil, air-dried, homogenised and aliquots were combusted with subsequent LSC analysis of the evolved $^{14}CO_2$. Mass balance was established on all specimens from the definitive tests. For the determination of the parental mass balance at various sampling times, the soil was exhaustively extracted with 40 mL acetonitrile/0.1 M hydrochloric acid 9:1 (v:v) for 30 min and additionally (if needed) with 40 mL acetonitrile/0.1 M sodium hydroxide 9:1 (v:v).

Description of analytical procedures

Liquid scintillation counting (LSC) was used to determine the radioactivity in the adsorption and desorption solutions and soil extracts. Radioactivity in residual soil samples mixed with cellulose was determined by LSC after combustion. Stock and application solutions as well as selected supernatants and extracts from the preliminary experiments were chromatographically analysed using HPLC coupled with an UV and ^{14}C-detector in order to confirm the stability of [cyclopropane-1-^{14}C]DCVA in the test system.

Results and discussion

The adsorbed and desorbed amount of [^{14}C]DCVA at each concentration is provided in percentage of applied radioactivity (% AR) in Table B.8.1-65.

Table B.8.1-65: Data for adsorption of metabolite DCVA on soil and in solution (mean of 2 replicates)

Nominal dose rate (µg/mL)	Solution µg/mL	Soil µg/g	% Adsorbed	K_d*	K_F	$K_{F\,oc}$	1/n
Wurmwiese soil (soil-solution 1:2, organic carbon 2.0 %)							
0.01	0.004	0.011	58.2	2.75	1.4857	74.3	0.8845
0.03	0.013	0.031	55.0	2.38			
0.1	0.046	0.097	51.2	2.11			
0.3	0.148	0.279	48.4	1.89			
1	0.531	0.836	43.9	1.57			
Höfchen soil (soil-solution 1:1, oc 2.9 %)							
0.01	0.003	0.006	64.9	2.00	0.4331	14.9	0.7429
0.03	0.012	0.016	57.1	1.33			
0.1	0.048	0.045	47.5	0.94			
0.3	0.169	0.115	39.7	0.68			
1	0.626	0.310	32.6	0.50			
Dollendorf soil (soil-solution 1:2, oc 4.4 %)							
0.01	0.007	0.005	25.2	0.71	0.3946	9.0	0.8878
0.03	0.022	0.013	23.0	0.59			
0.1	0.073	0.039	20.8	0.53			
0.3	0.227	0.103	18.3	0.45			
1	0.782	0.320	16.7	0.41			

Laacher Hof soil (soil-solution 1:1, oc 2.0 %)							
0.01	0.003	0.006	64.4	2.00	0.5381	26.9	0.7895
0.03	0.012	0.016	57.1	1.33			
0.1	0.048	0.046	49.0	0.96			
0.3	0.158	0.129	44.6	0.82			
1	0.589	0.357	37.5	0.61			
Hanscheider Hof soil (soil-solution 1:2, oc 2.9 %)							
0.01	0.003	0.012	64.4	4.00	1.8673	64.4	0.8844
0.03	0.011	0.035	60.7	3.18			
0.1	0.040	0.108	55.5	2.70			
0.3	0.133	0.308	53.4	2.32			
1	0.465	0.967	50.7	2.08			
mean (n = 5)					**0.9438**	**37.9**	**0.8378**

* K_d calculated by RMS from mean values: K_d = [soil (mg/kg)] / [solution (mg/L)]

Table B.8.1-66: Data for desorption of metabolite DCVA from soil (mean of 2 replicates)

Nominal dose rate (µg/mL)	% Desorbed	K_F	$K_{F\,oc}$	1/n
Wurmwiese soil (soil-solution 1:2, organic carbon 2.0 %)				
0.01	22.6	1.6472	82.4	0.8639
0.03	25.0			
0.1	28.5			
0.3	31.1			
1	34.4			
Höfchen soil (soil-solution 1:1, oc 2.9 %)				
0.01	1.7	0.4786	16.5	0.6817
0.03	4.3			
0.1	10.5			
0.3	14.5			
1	21.0			
Dollendorf soil (soil-solution 1:2, oc 4.4 %)				
0.01	23.3	0.5292	12.0	0.8473
0.03	28.4			
0.1	28.1			
0.3	38.1			
1	37.2			
Laacher Hof soil (soil-solution 1:1, oc 2.0 %)				
0.01	8.3	0.5896	29.5	0.7421
0.03	10.6			
0.1	17.0			
0.3	20.9			
1	26.4			
Hanscheider Hof soil (soil-solution 1:2, oc 2.9 %)				
0.01	15.0	2.1268	73.3	0.8529
0.03	17.9			
0.1	22.2			
0.3	24.2			
1	26.6			

Adsorption coefficients $K_{F\,ads}$ ranged from 0.39 to 1.87 with corresponding carbon normalised adsorption coefficients ($K_{F\,oc\,ads}$) ranging from 9.0 to 74.3 for the five test soils (mean: 37.9). The Freundlich exponents 1/n were in the range of 0.7429 to 0.8878 indicating that the concentration of the test item affected the adsorption behaviour. Desorption coefficient $K_{F\,des}$ values ranged from 0.48 to 2.13 with corresponding desorption constants ($K_{F\,oc\,des}$ values) ranging from 12.0 to 82.4 for the five test soils.

The regression coefficients for the Freundlich isotherms were > 0.99 for all soils testet. The mean desorption $K_{F\,des}$ and the normalised $K_{F\,oc\,des}$ values were 1.10 – 1.33 times higher than those obtained for adsorption phase.

Mass balance

The mass balance in the definitive test was determined by LSC of the supernatants after adsorption /desorption and by combustion of the remaining soils. The overall material balance for all concentrations was 85.2 – 95.2 % of the applied radioactivity for the soil Laacher Hof; for the other four soils it was in the range of 91.0 - 105.0 % of the applied radioactivity.

Transformation of parent compound

Test systems without soil did not show adsorption to the vessels or degradation. For the soils Wurmwiese, Höfchen, Laacher Hof and Hanscheider Hof the parental mass balance after 72 hours showed that > 90 % of applied [cyclopropane-1-^{14}C]BCS-AA53389 could be recovered. This demonstrates that the test item was sufficiently stable for the test in these soils. The mass balance of soil Dollendorf was up to 24 h > 90 %. With biocide (50 ppm $HgCl_2$ in the 0.01 M aqueous $CaCl_2$ solution) the test item was sufficiently stable for 72 hours (mass balance >90 %).

Comment

The K_d values in Table B.8.1-65 reflect a light increase of adsorption at lower dose rates in all five soils. The Freundlich exponent is 0.838 (mean of n=5) and therefore well within the general range of 0.7 – 1.0. The study is acceptable.

Comment on pH-dependency of adsorption of metabolite DCVA

The soils from both studies (n = 8) cover the pH($CaCl_2$)-range between 4.3 and 7.5 There is an increase of adsorption with decreasing pH between pH($CaCl_2$) 7.5 and pH 4.3 both for K_f and K_{foc}.

As a consequence the RMS proposes to calculate the mean of soils pH 6.0 – 7.5 (geomean K_{foc} = 16, n = 4) which represents adsorption in slightly acid, neutral and slightly alkaline soils and the mean of soils pH 5.1 – 5.9 (geomean K_{foc} = 50, n = 3) which represents acid and very acid soils pH < 6.0. The adsorption data of the very acid soil Cranfield 230 (pH 4.3) are proposed not to use in modelling or for trigger endpoint due to high uncertainty.

B.8.1.4 Mobility in soil

In the monograph from 1 October 1996 the studies on mobility of cyfluthrin (Obrist & Thornton 1982, Whitfield & Obrist 1983, Scholz & Umgelder 1985) were evaluated to be acceptable.

The results obtained by thin-layer chromatography and by a column leaching study (without and with aging of the treated soil) demonstrated the immobility of cyfluthrin in soil (≤1 % of the recovered radioactivity). Moreover, negligible amounts of metabolites containing the fluorophenyl moiety were found in the leachates (3.5 % of the recovered radioactivity without aging, <1 % with aging).

Kaufman et al. 1981 studied the leaching behaviour of the relevant soil metabolites FPB-acid and DCVA with the conclusion, that both may be fairly mobile in soils. The study was evaluated to be acceptable.

B.8.2 Fate and behaviour in water and sediment

B.8.2.1 Route and rate of degradation in aquatic systems (chemical and photochemical degradation)

B.8.2.1.1 Aqueous hydrolysis

The hydrolysis of cyfluthrin was investigated by Krohn (1983) and Sandie (1983). Both studies were evaluated in the monograph of 01 October 1996. A study of Krohn (1997a) using cyfluthrin and beta-cyfluthrin was evaluated in the addendum 1 from 07 May 2002 to the monograph of 01 October 1996.

The hydrolysis of the metabolite DCVA was studied by Krohn (1997b), the study was evaluated in the addendum 1 from 07 May 2002. No study on the hydrolytic degradation of the metabolite FPB-aldehyde is provided. Hydrolysis is not expected under sterile conditions because of the molecular structure (Review report 6841/VI/97-final, 02 December 2002).

A short summary of the results is given in Table B.8.2-1. The half-lives for hydrolysis of cyfluthrin and DCVA from the studies of Krohn (1997 a+b) are given in Table B.8.2-1.

Table B.8.2-1: Hydrolysis of cyfluthrin and its metabolite DCVA (short summary)

Study	Test conditions	Test substance	Results
cyfluthrin			
Krohn 1983	sterile buffer solutions: pH 4 (citrate) 60, 70, 80 °C pH 7 (phosphate) 60, 70, 80 °C pH 9 (borate), 30, 40, 50, 60 °C	4 diastereomeres of cyfluthrin, not radio-labeled, test concentration 2 µg L^{-1}	hydrolysis products identified by HPLC were DCVA and FPB-aldehyde, half-lives extrapolated to 22 °C are seen erroneous
Sandie 1983	sterile, aqueous buffers (pH 5, 7, 9) containing 1 % acetonitrile at 25 °C in the dark, test duration 35 days (21 days at pH 9)	[phenyl-UL-^{14}C]-cyfluthrin, test concentration 20 µg L^{-1}	half-live at pH 9 < 2 days, hydrolysis product identified by TLC was FPB-aldehyde (11 % AR at pH 7 on day 35, 89 % AR at pH 9 on days 14 and 21)
Krohn 1997a	sterile buffer solutions in the dark: pH 4 (citrate) 50 °C for 26 days, pH 7 (phosphate) 50, 60, 70 °C for 8 days to 8 hours, pH 9 (borate), 40 and 50 °C for 8 and 1.5 hours	cyfluthrin and beta-cyfluthrin, 4 µg L^{-1} (sum of all diastereomers)	Due to epimerisation both test substances formed mixtures of identical composition under the conditions of this test. half-lives for 20 °C were calculated by extrapolation.
metabolite DCVA			
Krohn 1997b	pH 4, 7, 9 at 50 °C for 1 week	cis-DCVA and trans-DCVA, 100 mg L^{-1}	decrease of concentration < 2 %

Table B.8.2-2: Half-lives for hydrolytic degradation of cyfluthrin at 20 °C and metabilite DCVA

test substance	pH	half-lives
cyfluthrin *	4	> 1 year (all isomers)
	7	270 days (isomers I + II) 160 days (isomers III + IV)
	9	42 hours (isomers I + II) 33 hours (isomers III + IV)
metabolite DCVA	4, 7, 9	stable at 50 °C for 1 week (both isomers), therefore hydrolytically stable, half-live > 1 year at 25 °C

* mean values from measurements with cyfluthrin and beta-cyfluthrin

A re-evaluation of the half-lives was conducted by Xu & Ripperger 2013.

B.8.2.1.1/1 (Xu & Ripperger 2013)

Reference	:	Xu, Tianbo and Ripperger, Randy
Title	:	Cyfluthrin and beta-cyfluthrin – hydrolysis half-live evaluation
Year of execution	:	14 June 2013
GLP statement	:	not relevant
Guideline	:	not relevant; calculation according OECD 111
Test substance	:	Cyfluthrin and beta-cyfluthrin
Test system	:	hydrolysis under sterile conditions

Executive Summary

The original study (Krohn 1997a) was re-evaluated and the detailed half-lives were calculated according OECD guideline 111. The re-evaluation showed that the half-lives reported in the original study report (Krohn, 1997a) were calculated according the OECD 111.

Study design

The half-lives from the original study of Krohn (1997a) were re-evaluated. No experiment was performed for the re-evaluation.

Description

According to OECD guideline no. 111 (Annex 2), the Arrhenius equation can be used to calculate the rate constant k for other temperatures, when the rate constants are known for 2 temperatures. The linear relationship between rate constants at higher temperatures and reciprocals of temperature in Kelvin were calculated. Afterwards, the rates at lower temperatures were extrapolated by using the linear regression equations. These rates were used to calculate the half-lives.

Results

The half-lives recalculated by Xu & Ripperger in comparison to those by Kohn 1997a are summarised in Table B.8.2-3. Only data for 20 °C are given here.

As can be seen in Table B.8.2-3., the half-lives in the original study report were the same as the half-lives calculated according to OECD guidelines with three exceptions as highlighted. However, the differences were very small (4750 *versus* 4740 hours and 41.5 *versus* 41.6 hours) and assumed to be caused by the rounding during the calculations.

Conclusions

The half-life values in the original study report were the same as the half-life values calculated according to OECD guidelines.

Table B.8.2-3: Comparison of half-lives recalculated by Xu & Ripperger 2013 with data from Kohn 1997a

pH	Test substance	Isomers	Temperature °C	half-lives re-calculated (hours)	half-lives in original study (hours)
7.0	cyfluthrin	isomers I + II	20	6560	6560
		isomers III + IV	20	**4750**	**4740**
	beta-cyfluthrin	isomers I + II	20	6610	6610
		isomers III + IV	20	3060	3060
9.0	cyfluthrin	isomers I + II	20	36.6	36.6
		isomers III + IV	20	24.5	24.5
	beta-cyfluthrin	isomers I + II	20	46.5	46.5
		isomers III + IV	20	**41.5**	**41.6**

B.8.2.1.2 Aqueous phototransformation

Four studies on direct and indirect photolysis in aqueous systems (Wilmes 1980, Puhl et al. 1983, Gronberg 1984, Westphal 1984) were evaluated in the monograph 01 October 1996. A short summary of the results is given in Table B.8.2-4.

Table B.8.2-4: Photolysis of cyfluthrin in aquous systems (short summary)

Study	Test conditions	Test substance	Results
Willmes 1980	high-pressure Hg-lamp with glass filter tube to block wavelength < 300 nm	cyfluthrin 100 mg L^{-1} in acetonitrile/water (1+1) with and without sensitiser 2 % acetone	photolysis products FPB-aldehyde, FPB-acid, formyl-dibenzofuran detected but not quantified, half-lives 7 hours (without acetone) and < 1 hour (with acetone)
Puhl et al. 1983	test duration 6 days, medium pressure Hg-lamp suspended in a glass immersion well circulated by cool water, intensity 67 W m^{-2}	[phenyl-UL-^{14}C]-cyfluthrin, 5 µg L^{-1} in sterile, aqueous phosphate buffer (pH 5) containing 1 % acetonitrile	experimental half-life 12.2 days, photolysis products identified by TLC were FPB-aldehyde (3.0 % on day 6), FPB-acid (8.5 % on day 6, increasing to the end of the study)
Gronberg 1984	test duration 14 days, natural sunlight in Kansas (38°49' N, 320 m above sea level), intensity end of August 20 – 102.5 W m^{-2}	[phenyl-UL-^{14}C]-cyfluthrin, 5 µg L^{-1} in sterile, aqueous phosphate buffer (pH 5) containing 1 % acetonitrile	experimental half-life < 1 day, photolysis products identified by TLC were FPB-aldehyde (18.0 % on day 7), FPB-acid (37 % on day 14)

Westphal 1984	high-pressure Hg-lamp with glass filter tube to block wavelength < 300 nm	aqueous solution of 4 µg L^{-1} cyfluthrin with and without humic acid	half-life in water 13 – 18 hours, half-lives in water with humic acids reduced to 7 hours (3 mg L^{-1}) and 4 hours (10 mg L^{-1})

The quantum yield of direct photodegradation of cyfluthrin was determined by Hellpointner 1991, which was evaluated in the monograph from 01 October 1996. From the UV absorption data and the kinetic results of two photodegradation experiments the quantum yield was calculated to be 0.0052. These data were further used to estimate the environmental half-lives of cyfluthrin due to direct photolysis by two simulation programs. The results are summarised in Table B.8.2-5.

Table B.8.2-5: Environmental half-lives for cyfluthrin calculated by Hellpointner 1991 (short summary)

GC-solar program				
Season	**Degree of latitude**			
	30°	**40°**	**50°**	**60°**
spring	3.1	3.4	3.9	4.8
summer	2.8	2.8	3.0	3.2
fall	4.5	5.9	8.9	17
winter	6.2	9.8	20	58
Frank-Klöpfer program				
Month	**Minimum**	**Mean**	**Maximum**	
April	3.4	6.1	25	
May	2.9	4.7	19	
June	2.8	4.2	17	
July	3.1	4.7	16	
August	3.3	4.9	16	
September	5.1	8.7	32	

A new study to determine the quantum yield and the environmental half-live of the direct photo-degradation in water was conducted by Hellpointner & Mahlburg 2013.

B.8.2.1.2/1 (Hellpointner & Mahlburg 2013)

Reference	:	Hellpointner, E. and Halburg, G.
Title	:	Beta-cyfluthrin: determination of the quantum yield and assessment of the environmental half-live of the direct photo-degradation in water
Year of execution	:	02 September 2013
GLP statement	:	yes
Guideline	:	OECD test guidelines 101, 1981 and 316, 2008
Test substance	:	beta-cyfluthrin
Test system	:	photolysis in water

Executive Summary

The UV-VIS absorption spectrum of beta-cyfluthrin was determined in water/acetonitrile (50/50, v/v). It showed one weak maximum at 268 nm (ε = 2082 L × mol-1 cm-1, bandwidth up to 281 nm). In buffered aqueous solutions of pH 4 and pH 7 the UV-VIS absorption properties were similar. The molar extinction coefficients ε at 290 and 295 nm were determined to be 158 and 84 L × mol^{-1} cm^{-1}, respectively. The absorption of beta-cyfluthrin extends only weak but very far into the environmentally relevant range of wavelengths. This indicates some potential for direct photolytic interactions of beta-cyfluthrin with sunlight.

The quantum yield of direct photo-transformation of the beta-cyfluthrin was determined in above mentioned aqueous solution using polychromatic light according to the ECETOC method. By HPLC analysis a beta-cyfluthrin degradation of 18.5 % - 20.9 % was measured after a maximum irradiation period of 500 minutes. This indicates a moderate degradability of beta-cyfluthrin via direct photo-transformation in aqueous solutions. A low mean quantum yield (Φ) of 0.001149 was calculated on the basis of UV absorption data and the kinetics determined from two degradation experiments.

A comparison of the estimates derived from models of Zepp & Cline and Frank & Kloepffer shows that both approaches are well comparable. The two approaches consider the quantum yield and the absorption in a range of wavelengths relevant for the environment. Environmental half-lives in top surface water layer exposed to sunlight were estimated to range between one week and one month, for direct phototransformation of beta-cyfluthrin during periods of main use in May and June. Direct photo-transformation in water may contribute to the dissipation of beta-cyfluthrin from the environment.

Material and methods

Test material

Non-radiolabelled beta-cyfluthrin, purity 98.8 %, minimum concentration of diastereomers II + IV is 95 % in the pure active substance, 30 – 42 % cis / 58 – 70 % trans.

Study design

UV-VIS absorption spectra
For the determination of UV-VIS spectra of beta-cyfluthrin, a stock solution of beta-cyfluthrin was prepared in acetonitrile (ACN). Buffered aqueous solution of pH 4 and pH 7 were prepared by diluting the required aliquots of stock solution with the respective 0.01 M aqueous acetate (pH 4) or 0.01 M phosphate (pH 7) buffer solutions. In addition, a solution of beta-cyfluthrin in water/ACN (1:1, v:v) was prepared (15.41 mg beta-cyfluthrin L-1) and used for calculating the quantum yield and for the corresponding environmental modelling.
The UV-VIS absorption properties of the test item were characterised by the number and position of the absorption maxima as well as for each maximum by the molar extinction coefficient ε [L × mol-1 cm-1]. Moreover, the bandwidth was determined for each resolved maximum. In case of the respective UV-VIS spectrum, the extinction values were considered until 490 nm.

Quantum yield
The experiment was based on the method by ECETOC. It is based on the partition of the polychromatic light into sectors of wavelengths. In this study, sectors of 5 nm were defined 295 – 400 nm and sectors fo 10 nm from 401 nm on.

Photo-transformation
For the solutions for the photo-transformation experiments, 0.195 mL of the beta-cyfluthrin stock solution were pipetted into a 50-mL volumetric flask and made up to volume with water (c = 5.01 mg/L) corresponding to 1.15 × 10-5 mol L-1. The degradation experiment was conducted in a merry-go-round irradiation apparatus which was fitted with a mercury immersion lamp TQ 150. The light intensity acting on the test solution was measured by means of uranyl oxalate as chemical actinometer and the total amount of radiation entering the measuring cell was calculated from the number of photons being absorbed by the actinometer. From the titration difference derived from actinometry, the average intensity of radiation acting upon the test solution during the exposure was calculated.

The results of beta-cyfluthrin analysis from the photo-transformation test (usually means of duplicates) were evaluated on the basis of linear regression and represented as a degradation line in a concentration versus time diagram (log % beta-cyfluthrin = - kt + b). The time (in min) after which 10 % of the molecules of the test item have been degraded (necessary to calculate the quantum yield) was calculated

according to the decay law from the determined rate constant k [1/min] of photo-transformation (single first order degradation).

Description of analytical procedures

The beta-cyfluthrin concentration in the liquid samples was determined by reversed phase HPLC and evaluation of the respective UV signals (as sum of the main two diastereomeric enantiomer pairs, II + IV) by means of external reference standard). A significant change of ratio of diastereomer pairs (II : IV) was not measured during the irradiation.

Results

UV-VIS absorption spectra:
The UV-VIS absorption spectrum of a solution of 15.41 mg beta-cyfluthrin/L water/ACN (1/1, v/v) showed one weak maximum at 268 nm (abs 0.0739, ε 2082 L × mol^{-1} cm^{-1}). The respective UV-VIS absorption spectra of 15.41 mg beta-cyfluthrin per litre pH 4 and pH 7 buffered aqueous solutions showed similar absorption properties. The molar extinction coefficient ε at 290 and 295 nm was determined to be 158 and 84 L × mol^{-1} cm^{-1}, respectively. In general, the absorption properties indicate a weak potential for direct photolytic interactions of beta-cyfluthrin with sunlight in the environment.

Photo-transformation – Intensity of irradiation
The intensity of irradiation was calculated to 7.8482 – 7.8025 x 10^{16} photons absorbed per second for the 3 mL actinometry solution in the range of wavelength from 295 to 490 nm. By HPLC- analysis a beta-cyfluthrin degradation of 18.5 % - 20.9 % was measured after a maximum irradiation period of 500 minutes. This indicates a moderate degradability of beta-cyfluthrin via direct photo-transformation in aqueous solutions.

Results of photo-transformation experiments #1 and #2 in aqueous solution are shown in Table B.8.2-6.

Table B.8.2-6: Results of photo-transformation experiments in aqueous solution

Parameter	Exp. #1	Exp. #2
no. of data pairs	11	11
rate constant (k) min^{-1}	0.0004	0.0005
half-life (min)	1575.7	1446.8
correlation coefficient	-0.9915	-0.9869

Photo-transformation – Quantum yield
Based on both degradation experiments performed, quantum yields Φ of 1.0980 – 1.2005 × 10^{-3} were calculated. Thus, a mean quantum yield Φ of 0.001149 was obtained for the direct photo-transformation in aqueous solution.

Photo-transformation – Environmental half-lives according to Zepp & Cline
Environmental half-lives were calculated according to Zepp & Cline (GC Solar) by using an arithmetic model which allows for a transfer of laboratory data concerning the direct photo-transformation in water to field conditions. Based on a mean quantum yield Φ of 0.001149 and the molar extinction coefficients ε from 297.5 to 490 nm, environmental half-lives were calculated and summarised in Table B.8.2-7.

Table B.8.2-7: Environmental half-lives (days) calculated according to Zepp&Cline

Season	Degree of latitude			
	30°	40°	50°	60°
spring	5.51	5.88	6.57	7.76
summer	4.97	4.99	5.14	5.44

fall	7.65	9.71	14.1	25.9
winter	10.2	15.4	30.2	93.5

Marginal conditions: pure surface water at 0-5 cm depth, 10th degree longitude, clear sky, typical ozone concentrations in the atmosphere, half-lives integrated over the entire day. The results for the 50th degree of latitude are regarded to be relevant to the conditions of Central Europe.

Photo-transformation – Environmental half-lives according to Frank & Kloepffer
Environmental half-lives were also calculated according to Frank & Kloepffer, using an arithmetic model which considers the influence of clouded sky for the region Central Europe, i.e. Germany. Using the mean quantum yield Φ of 0.001149 and the molar extinction coefficients ε from 292.5 to 490 nm, environmental half-lives were calculated as summarised in Table B.8.2-8.

Table B.8.2-8: Environmental half-lives (days) calculated according to Frank&Klöpfer

Month	Minimum	Mean	Maximum
April	6.1	11	44
May	5.4	8.7	35
June	5.2	7.7	31
July	5.8	8.7	29
August	5.9	8.9	30
September	9.0	15	56

Marginal conditions: pure stagnant surface water at 0-5 cm depth, geographic and climatic conditions of Germany (50° lat.), no contribution of another mono- or bimolecular elimination process.

Conclusion

In general, the absorption properties indicate a weak potential for direct photolytic interactions of beta-cyfluthrin with sunlight in the environment. A moderate photo-degradation of beta-cyfluthrin in aqueous solution in a range of 19 to 21 % was measured by HPLC after a maximum irradiation period of 500 minutes. A low mean quantum yield of Φ = 0.001149 was calculated on the basis of UV absorption data and the kinetics determined from two degradation experiments.

A comparison of the estimates derived from models of Zepp & Cline and Frank & Kloepffer shows that both approaches are well comparable. The two approaches consider the quantum yield and the absorption in a range of wavelengths relevant for the environment. Environmental half-lives in top surface water layer exposed to sunlight were estimated to range between one week and one month, for direct phototransformation of beta-cyfluthrin during periods of main use in May and June.
Direct photo-transformation in water may contribute to the dissipation of beta-cyfluthrin from the environment. This assessment does not consider other potential mechanisms which may enhance the photo-degradation in natural water, e.g. by indirect photolytic processes.

B.8.2.2 Route and rate of biological degradation in aquatic systems

B.8.2.2.1 Water-sediment studies

Two water-sediment studies were carried out to investigate the route and rate of degradation or dissipation of beta-cyfluthrin with different radioactive labelling (Anderson 1987, Sneikus 2000). The main metabolites were FPB-aldehyd (16 % in sediment) and FPB-acid (29 % in water).

The studies were evaluated in the monograph dated 01 October 1996 and the addendum 1 dated 07 May 2002. No new water-sediment studies were provided by the applicant. However, the degradation rates were **recalculated by Hammel & Porschewski 2013b** following the latest FOCUS kinetic guidelines.

B.8.2.2.1/1 Hammel & Porschewski 2013b)

Reference	:	Hamel, Klaus and Porschewski, Ruth
Title	:	Kinetic evaluation of the aerobic aquatic metabolism of cyfluthrin and beta-cyfluthrin and their metabolites in water/sediment systems according to FOCUS kinetics
Year of execution	:	26 November 2013
GLP statement	:	not relevant
Guideline	:	not relevant; calculation according FOCUS Kinetics (2006)
Test substance	:	Cyfluthrin and beta-Cyfluthrin
Test system	:	aerobic degradation in water/sediment systems under laboratory conditions

Study data

The water/sediment study of **Anderson 1987** was evaluated in the monograph from 01 October 1996. A description of the test system is missing there and therefore is given in Table B.8.2-9.

Table B.8.2-9: Data of water phases and sediments in the study of Anderson 1987 (short summary)

	Ijzendoorn (NL)	**Lienden (NL)**
	orchard drainage ditch	fishpond
water phase during experiment		
O_2 content (% saturation)	82 - 87	75 – 86
pH	7.0 – 8.3	7.0 – 8.3
sediment		
sand (2000 – 63 µm) %	68.5	76.6
silt (63 – 2 µm) %	16.4	15.1
clay (< 2 µm) %	15.1	8.3
texture	loamy sand	loamy sand
organic C (%)	0.51	1.05
total N (%)	0.09	0.08
total P (mg/kg)	390	290
$CaCO_3$ (g/kg)	2.97	16.9
pH (KCl)	6.81	7.76

The test substance was ^{14}C-labelled (fluorobenzene-U-^{14}-C)-cyfluthrin at a concentration of 12 µg/L.The study was conducted at a temperature of 22 °C in the dark over an experimental period of 70 days with samplings at day 1, 11, 22 (Lienden) / 28 (Ijzendoorn), 40 and 70. No day zero measurements were available. The whole amount of radioactivity (metabolites, non-extractable residues and CO_2) was attributed to the parent on the day of first measurement. The cyfluthrin residues for total system and sediment extracts used for kinetic analysis are given in Table B.8.2-10. The residues for the metabolites FPB-aldehyde and FPB-acid in water, sediment and total system are given in Table B.8.2.-11. The data for total system are used for kinetic analysis (decline fit from maximum).

Table B.8.2-10: Cyfluthrin residues in total system used for kinetic analysis (% applied radioactivity)

Day	Total system		Sediment	
	Ijzendoorn	Lienden	Ijzendoorn	Lienden
1	88.9 **	101.1 **	8.6	21.3
1	103.2 **	100.8 **	31.5	3.8
11	4.2	3.5	4.1	3.5
11	10.7	3.4	8.5	3.4
28/22 *	1.6	6.5	1.5	6.5
28/22 *	0.9	0.6	0.7	0.6
40	5.2	0.9	5.2	0.9
40	4	2.2	4	2.2
70	0.9	0.7	0.9	0.7
70	1.3	1	1.3	1

* day 28 for system Ijzendoorn, day 22 for system Lienden
** total recovery on day 1
AR : applied radioactivity, nd : not detected

Table B.8.2-11: FPB-aldehyde and FPB-acid residues in water/sediment systems (% applied radioactivity)

Day	Water phase		Sediment extract		Total system	
	FPB-aldehyde	FPB-acid	FPB-aldehyde	FPB-acid	FPB-aldehyde	FPB-acid
Ijzendoorn						
1	nd	9.8	15.7	16.9	15.7	26.7
1	nd	13.4	15.6	16.0	15.6	29.4
11	nd	30.2	<0.1	17.8	0.1	48.0
11	nd	28.0	2.4	13.0	2.4 *	41.0
28	0.1	0.4	1.4	1.6	1.5	2.0
28	0.1	0.6	1.3	1.9	1.4	2.5
40	nd	nd	0.8	0.7	0.8	0.7
40	nd	nd	1.0	0.8	1.0	0.8
70	nd	nd	0.2	0.3	0.2	0.3
70	nd	nd	0.5	1.0	0.5	1.0
Lienden						
1	1.7	12.3	11.3	18.2	13.0	30.5
1	0.5	10.4	5.5	30.4	6.0	40.8
11	nd	12.8	7.8	4.3	7.8	17.1
11	nd	10.6	4.4	3.2	4.4	13.8
22	nd	2.1	0.7	1.4	0.7	3.5
22	nd	1.8	0.4	4.3	0.4	6.1
40	nd	nd	2.5	0.4	2.5	0.4
40	nd	nd	0.6	0.5	0.6	0.5
70	nd	nd	0.3	0.7	0.3	0.7
70	nd	nd	0.2	0.3	0.2	0.3

* in the study 3.5 % was used instead of 2.4 %
nd : not detected

The water/sediment study of **Sneikus 2000** was evaluated in the addendum 1to the monograph of October 1996, dated 07 May 2002. For overview of the test systems the data are given in Table B.8.2-12.

Table B.8.2-12: Analytical data of water phases and sediments in the study of Sneikus 2000 (short summary)

	Barmener See (Jülich, DE)	**Genkel creek (Meinerzhagen, DE)**
	small disused gravel-pit	catchment basin
water phase during experiment		
O_2 content (% saturation)	57 - 124	49 - 114
pH	5.1 – 8.1	4.6 – 8.0
total org C (TOC, mg C L^{-1})	start: 2.7 / end: 15	start: 1.5 / end: 12
dissolved org C (DOC mg C L^{-1})	start: 2.4 / end: 4	start: 1.5 / end: 2
hardness (° dGH)	start: 8	start: 4
total N (mg L^{-1})	start: 1.95 / end: 10.0	start: 2.12 / end: <1
total P (mg L^{-1})	start: 0.1 / end: 0.36	start: 0.01 / end: 0.27
sediment at start of experiment		
sand (2000 – 63 µm) %	97.7	8.2
silt (63 – 2 µm) %	5.3	73.4
clay (< 2 µm) %	<0.1	18.4
texture	sand	silt loam
organic C (%)	0.48	4.91
organic matter (OC × 1.72)		
total N (%)	0.18	0.43
total P (mg/kg)	119	835
$CaCO_3$ (g/kg)	0.5	<0.1
pH (H_2O / $CaCl_2$)	7.5 / 6.9	5.0 / 4.6

The test substance was ^{14}C-labelled (cyclopropane-1-^{14}-C)-cyfluthrin at a concentration of 8.14 µg/L. The study was conducted at a temperature of 20 °C in the dark over a period of 100 days with samplings at 0.5, 3 and 6 hours and at day 1, 2, 3, 7, 10, 14, 28, 56 and 100. In the study of Sneikus 2000 the diastereomers were analysed separately and the ratio of the diastereomers was measured at the different sampling dates. To account for beta-cyfluthrin the relative amounts of the two active diastereomers II and IV were summed up and combined with the measured concentration (% AR) of cyfluthrin. The cyfluthrin residues in total systems are given in Table B.8.2-13, together with relative amounts of isomers II + IV and beta-cyfluthrin residues. The residues for the metabolite DCVA in the water/sediment system are given in Table B.8.2-14. The data for total system are used for kinetic analysis (decline fit from maximum).

Table B.8.2-13: Cyfluthrin in water, sedimentextract and total system and beta-cyfluthrin in total system from Sneikus 2000

Time	Cyfluthrin (% AR)						% Isomers II + IV		beta-Cyfluthrin (% AR)	
	Barmener			Genkel			Barmener	Genkel	Barmener	Genkel
	Water	Sediment	Total	Water	Sediment	Total			Total system	
0.5 h	46.94 +	41.91	92.43 *	29.71 +	63.80	98.14 *	47 #	43 #	39.01	41.42
0.5 h	46.29 +	41.95	91.45 *	28.15 +	58.31	90.58 *			38.59	38.22
3 h	17.74	64.58	82.32	37.12	42.18	79.30	47	41	38.36	37.35
3 h	31.91	56.01	87.92	25.98	55.69	81.67			40.97	38.47
6 h	22.54	68.36	90.90	17.53	66.37	83.90	42	44	40.91	37.76
6 h	NaN	NaN	NaN	18.35	59.65	78.00			NaN	35.10
1 d	15.88	48.18	64.06	3.34	56.05	59.39	48.7	38	31.20	22.57
1 d	18.69	48.25	66.94	4.64	57.22	61.86			32.60	23.51
2 d	3.38	35.15	38.53	1.14	52.49	53.63	48.8	38	18.80	20.38
2 d	2.65	46.77	49.42	1.00	62.82	63.82			24.12	24.25
3 d	0.32	39.98	40.30	0.61	49.09	49.70	40.8	37.1	16.44	18.44
3 d	0.35	41.86	42.21	0.68	44.03	44.71			17.22	16.59
7 d	LOD	25.07	25.11	0.05	42.74	42.79	40.4	38.9	10.14	16.65
7 d	LOD	25.69	25.69	0.16	38.45	38.61			10.38	15.02
10 d		21.75	22.41	0.31	31.06	31.37	31.6	36.1	7.08	11.32
10 d		22.12	22.82	2.15	28.93	31.08			7.21	11.22
14 d		15.15	15.15		41.80	41.80	46.8	34.4	7.09	14.38
14 d		10.98	10.98		32.61	32.61			5.14	11.22
28 d		9.33	9.33		19.25	19.25	49.5	33.6	4.62	6.47
28 d		13.32	13.32		19.02	19.02			6.59	6.39
56 d		7.30	7.30		16.49	16.49	36.2	41.4	2.64	6.83
56 d		7.87	7.87		18.21	18.21			2.85	7.54
100 d		7.01	7.01		13.30	13.30	38.8	38.1	2.72	5.07
100 d		7.17	7.17		18.45	18.45			2.78	7.03

* total recocery of radioactivity in whole system
+ including dissolved ^{14}C-carbonates
NaN : not a number, only single measurement at 6 h
LOD : limit of detection, 0.05 %
relative amounts before application (day 0) is 42.2 % for both systems Barmener and Genkel

Table B.8.2-14: Metabolite DCVA in water, sediment and total system from study by Sneikus 2000

Time	Barmener			Genkel		
	Water	Sediment	Total	Water	Sediment	Total
0.5 h			0.00			0.00
0.5 h			0.00			0.00
3 h	4.01		4.01	3.44		3.44
3 h	3.83		3.83	3.49		3.49
6 h	5.45	0.28	5.73	7.37	0.45	7.82
6 h	NaN	NaN	0.00	8.30	0.25	8.55
1 d	19.63	0.76	20.39	17.13	3.18	20.31
1 d	16.03	0.50	16.53	18.47	2.46	20.93
2 d	39.81	4.45	44.26	17.88	4.41	22.29
2 d	32.15	4.44	36.59	11.97	2.56	14.53
3 d	21.00	1.73	22.73	23.34	5.27	28.61
3 d	21.90	1.41	23.31	21.88	6.39	28.27
7 d	32.83	4.69	37.52	24.53	9.14	33.67
7 d	23.61	1.21	24.82	23.69	9.56	33.25
10 d	37.33	5.53	42.86	21.51	10.80	32.31
10 d	26.67	4.00	30.67	26.77	11.11	37.88
14 d	20.55	4.19	24.74	21.04	8.04	29.08
14 d	11.61	1.57	13.18	24.50	10.36	34.86
28 d	20.46	2.29	22.75	31.76	16.35	48.11
28 d	32.29	3.43	35.72	32.60	14.55	47.15
56 d	34.03	4.73	38.76	28.51	17.37	45.88
56 d	30.85	5.08	35.93	28.97	18.64	47.61
100 d	19.52	7.71	27.23	7.83	20.37	28.20
100 d	31.76	8.28	40.04	14.46	26.93	41.39

Modelling

The water/sediment studies were evaluated by Hammel & Porschewski 2013b according to FOCUS 2006. The dissipation or degradation kinetics are evaluated only in single compartments (level P-I and M-1). The model fit and the statistical evaluation of the results was carried out with the software KinGui version 2, developed by one of the notifiers, Bayer Crop Science. For the optimisation of the algorithms, Iteratively Reweighted Nonlinear Least Spuares (IRLS) was used.

Results

The relevant criteria to select the appropriate models which describe the residues of cyfluthrin, beta-cyfluthrin and its metabolites in the **total systems** are given in Table B.8.2-15 to Table B.8.2-19.

Table B.8.2-15: Criteria to select models for cyfluthrin residues in total systems from studies Anderson 1987 and Sneikus 2000

	Model	chi^2 (%)	Visual fit	t-test p-value	Selected
Barmer	SFO	15.92	-	< 0.001	
Genkel		19.22	-	< 0.001	
Ijzendoorn		7.88	o	< 0.001	modelling
Lienden		6.33	o	< 0.001	modelling
Barmer	HS	17.29	o	0.37 (k_2)	
Genkel		9.80	o	< 0.001 (k_2)	modelling
Ijzendoorn		6.28	o	0.43 (k_2)	
Lienden		1.86	o	0.111 (k_2)	

Barmer	FOMC	6.74	+	na	mod.+trigger
Genkel		6.28	+	na	trigger
Ijzendoorn		5.55	+	na	trigger
Lienden		2.23	+	na	
Barmer	DFOP	7.29	+	0.006 (k_2)	
Genkel		9.84	o	0.001 (k_2)	
Ijzendoorn		6.31	+	0.43 (k_2)	
Lienden		1.86	+	0.101 (k_2)	trigger

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta

Table B.8.2-16: Criteria to select models for DCVA in total systems (SFO for DCVA)

	Model	chi² (%)	Visual fit	t-test p-value	Selected
Barmer	FOMC-SFO	25.42	-	na	
Genkel		11.51	o	na	
Barmer	DFOP-SFO	25.16	-	0.155 (k_2)	
Genkel		parent: 10.54 metabolite: 10.5	o	< 0.001 (k_2)	mod.+trigger

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta

Table B.8.2-17: Criteria to select models for FPB-aldehyde in total systems (decline fit)

	Model	chi² (%)	Visual fit	t-test p-value	Selected
Ijzendoorn	SFO	15.08	-	< 0.001	
Lienden		23.09	o	0.011	mod.+trigger
Ijzendoorn	HS	3.07	+	0.121 (k_2)	
Lienden		22.12	-	0.095 (k_2)	
Ijzendoorn	FOMC	4.80	o	na	modelling
Lienden		26.77	o	na	
Ijzendoorn	DFOP	3.08	+	0.116 (k_2)	trigger
Lienden		32.74	o	0.500 (k_2)	

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta

Table B.8.2-18: Criteria to select models for FPB-acid in total systems (decline fit)

	Model	chi² (%)	Visual fit	t-test p-value	Selected
Ijzendoorn	SFO	2.71	+	0.001	mod.+trigger
Lienden		4.13	+	< 0.001	mod.+trigger
Ijzendoorn	HS	**	-	0.5 (k_2)	
Lienden		2.38		0.5 (k_2)	
Ijzendoorn	FOMC	1.74	+	na	
Lienden		5.99	+	na	
Ijzendoorn	DFOP	**	o	0.5 (k_2)	
Lienden		5.89		0.47 (k_2)	

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta
** degree of freedom too low, no chi² calculation applicable

Table B.8.2-19: Criteria to select models for beta-cyfluthrin in total systems

	Model	chi² (%)	Visual fit	t-test p-value	Selected
Barmer	SFO	13.06	-	< 0.001	
Genkel		21.08	-	0.493	
Barmer	HS	5.45	+	0.004 (k_2)	
Genkel		11.15	o	0.001 (k_2)	

Barmer	FOMC	7.03	o	na	
Genkel		7.45	+	na	trigger
Barmer	DFOP	5.03	+	0.009	mod.+trigger
Genkel		9.83	+	0.010	modelling

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta

The relevant criteria to select the appropriate model which describes the residues of cyfluthrin in the **water phase** are given in Table B.8.2-20.

Table B.8.2-20: Criteria to select models for cyfluthrin in water

	Model	chi^2 (%)	Visual fit	t-test p-value	Selected
Barmer	SFO	26.27	o	0.004	
Genkel		19.56	+	<0.001	mod.+trigger
Barmer	HS	31.26	-	0.5 (k_2)	
Genkel		22.53	-	0.5 (k_2)	
Barmer	FOMC	20.49	-	na	
Genkel		22.62	+	na	
Barmer	DFOP	12.77	o	<0.001 (k_2)	mod.+trigger
Genkel		22.45	+	0.5 (k_2)	

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta

The relevant criteria to select the appropriate model which describes the residues of cyfluthrin in the **sediment** are given in Table B.8.2-21 to Table B.8.2-24.

Table B.8.2-21: Criteria to select models for cyfluthrin residues in sediment

	Model	chi^2 (%)	Visual fit	t-test p-value	Selected
Barmer	SFO	17.75	-	< 0.001	
Genkel		14.82	-	< 0.001	
Ijzendoorn		24.04	o	0.086	
Lienden		22.12	o	0.120	
Barmer	HS	13.22	-	0.021 (k_2)	
Genkel		9.97	o	< 0.001 (k_2)	modelling
Ijzendoorn		25.05	+	0.306 (k_2)	
Lienden		9.33	o	0.405 (k_2)	
Barmer	FOMC	7.78	+	na	mod.+trigger
Genkel		7.62	+	na	trigger
Ijzendoorn		18.72	+	na	mod.+trigger
Lienden		10.90	+	na	mod.+trigger
Barmer	DFOP	11.6	o	0.127 (k_2)	
Genkel		8.12	+	0.306 (k_2)	
Ijzendoorn		22.21	+	0.452 (k_2)	
Lienden		9.33	+	0.334 (k_2)	

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta

Table B.8.2-22: Criteria to select models for beta-cyfluthrin in sediment

	Model	chi^2 (%)	Visual fit	t-test p-value	Selected
Barmer	SFO	16.50	-	<0.001	
Genkel		18.62	-	0.003	
Barmer	HS	8.02	-	0.006 (k_2)	
Genkel		11.84	o	0.022 (k_2)	modelling

Barmer	FOMC	5.43	+	na	mod.+trigger
Genkel		8.44	+	na	trigger
Barmer	DFOP	5.63	+	0.015 (k_2)	
Genkel		10.46	o	0.161 (k_2)	

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta

Table B.8.2-23: Criteria to select models for FPB-aldehyd in sediment (decline fit)

	Model	chi^2 (%)	Visual fit	t-test p-value	Selected
Ijzendoorn	SFO	15.09	-	0.001	
Lienden		26.05	+	0.012	mod.+trigger
Ijzendoorn	HS	4.91	+	0.132 (k_2)	
Lienden		23.57	-	0.223 (k_2)	
Ijzendoorn	FOMC	5.57	o	na	modelling
Lienden		30.29	o	na	
Ijzendoorn	DFOP	4.91	+	0.106 (k_2)	trigger
Lienden		37.08	+	0.500 (k_2)	

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta

Table B.8.2-24: Criteria to select models for FPB-acid in sediment (decline fit)

	Model	chi^2 (%)	Visual fit	t-test p-value	Selected
Ijzendoorn	SFO	6.42	-	0.006	
Lienden		13.27	o	0.007	
Ijzendoorn	HS	**	+	0.348 (k_2)	
Lienden		6.52	+	0.256 (k_2)	
Ijzendoorn	FOMC	3.37	+	na	mod.+trigger
Lienden		6.97	+	na	mod.+trigger
Ijzendoorn	DFOP	**	+	0.500 (k_2)	
Lienden		6.52	+	0.227 (k_2)	

visual fit: + good, o acceptable, - not acceptable
na: t-test not applicable for FOMC alpha or beta
** degree of freedom too low, no chi^2 calculation applicable

The resulting data for DT_{50} and DT_{90} of cyfluthrin, beta-cyfluthrin and the metabolites are given in Table B.8.2-25 to Table B.8.2-29 both for triggers and for modelling endpoints. For the metabolite DCVA only total system was calculated, for FPB-aldehyde and FPB-acid total system and sediment. For water (all metabolites) and both water and sediment (DCVA) nor reliable values were determinable.

Table B.8.2-25: Kinetic parameters for degradation of cyfluthrin and beta-cyfluthrin in total system

System	Trigger endpoints			Modelling endpoints				
	Model	DT_{50} days	DT_{90} days				DT_{50}	DT_{90}
cyfluthrin								
Barmener	FOMC	2.1	35.1	FOMC			10.6 *	35.1
Genkel	FOMC	3.9	527.9	HS	k_1 0.2128 k_2 0.0194		35.7 (k_2)	84.8
Ijzendoorn	FOMC	0.6	7.0	SFO			2.7	9.0
Lienden	DFOP	0.5	1.8	SFO			2.1	6.9
geometric mean		**1.3**					**6.8**	

beta-cyfluthrin								
Barmener	DFOP	2.4	47.9	DFOP	k_1 0.3826 k_2 0.0128 g 0.815		14.4 *	47.9
Genkel	FOMC	2.4	295.4	DFOP	k_1 0.6929 k_2 0.0131 g 0.613		53.0 (k_2)	103.7
geometric mean		**2.4**					**27.6**	

* DT90 / 3.32, backcalculation as final residues < 10 % of applied

Table B.8.2-26: Kinetic parameters for degradation of metabolites in total system

System	Trigger endpoints			Modelling endpoints				
	Model	**DT_{50} days**	**DT_{90} days**	**Model**			**DT_{50} days**	**DT_{90} days**
DCVA								
Genkel	DFOP-SFO	113.8	377.9	DFOP-SFO			113.8	377.9
FPB-aldehyde (decline fit)								
Ijzendoorn	DFOP	0.6	17.7	FOMC			4.3 *	14.3
Lienden	SFO	10.0	33.3	SFO	k 0.0692		10.0	30.7
geometric mean		2.4					**6.6**	
FPB-acid (decline fit)								
Ijzendoorn	SFO	4.0	13.3	SFO			4.0	13.3
Lienden	SFO	7.8	25.9	SFO			7.8	25.9
geometric mean		5.6					**5.6**	

* DT_{90} / 3.32

Table B.8.2-27: Kinetic parameters for dissipation of cyfluthrin in water

System	Trigger endpoints			Modelling endpoints				
	Model	**DT_{50} days**	**DT_{90} days**	**Model**			**DT_{50} days**	**DT_{90} days**
Barmener	DFOP	0.25	2.3	DFOP	k_1 107.4 k_2 0.7925 g 0.389		0.7 *	2.3
Genkel	SFO	0.4	1.3	SFO	k 1.791		0.4	1.3
geometric mean		**0.3**					**0.5**	

* DT_{90} / 3.32

Table B.8.2-28: Kinetic parameters for dissipation of cyfluthrin and beta-cyfluthrin in sediment

System	Trigger endpoints			Modelling endpoints				
	Model	**DT_{50} days**	**DT_{90} days**	**Model**			**DT_{50} days**	**DT_{90} days**
cyfluthrin								
Barmener	FOMC	3.5	78.7	FOMC			23.0 *	78.7
Genkel	FOMC	13.0	529.8	HS	k_1 0.0924 k_2 0.0130		53.3 (k_2)	139.7
Ijzendoorn	FOMC	3.9	49.0	FOMC			14.7 *	49.0
Lienden	FOMC	3.6	70.8	FOMC			21.3 *	70.8
geometric mean		**5.0**					**24.9**	

beta-cyfluthrin								
Barmener	FOMC	3.1	47.6	FOMC			14.3 *	47.6
Genkel	FOMC	6.9	650.8	HS	k_1 0.0890 k_2 0.0085		81.5 (k_2)	180.8
geometric mean		**4.6**					**34.1**	

* DT_{90} / 3.32

Table B.8.2-29: Kinetic parameters for dissipation of metabolites in sediment

System	Trigger endpoints			Modelling endpoints				
	Model	**DT_{50} days**	**DT_{90} days**	**Model**			**DT_{50} days**	**DT_{90} days**
FPB-aldehyde (decline fit)								
Ijzendoorn	DFOP	0.5	4.3	FOMC			2.2 *	7.3
Lienden	SFO	11.2	37.1	SFO	k 0.0621		11.2	37.1
geometric mean		**2.4**					**5.0**	
FPB-acid (decline fit)								
Ijzendoorn	FOMC	2.3	18.4	FOMC			5.5 *	18.4
Lienden	FOMC	2.1	17.3	FOMC			5.2 *	17.3
geometric mean		**2.2**					**5.3**	

* DT_{90} / 3.32

The KinGUI results, whole data sets and graphics, are given in **Appendix 2** to Vol. 3, B.8.

Comment

The kinetic evaluation can be accepted. Only data referring to beta-cyfluthrin are used for further modelling or as trigger endpoints.

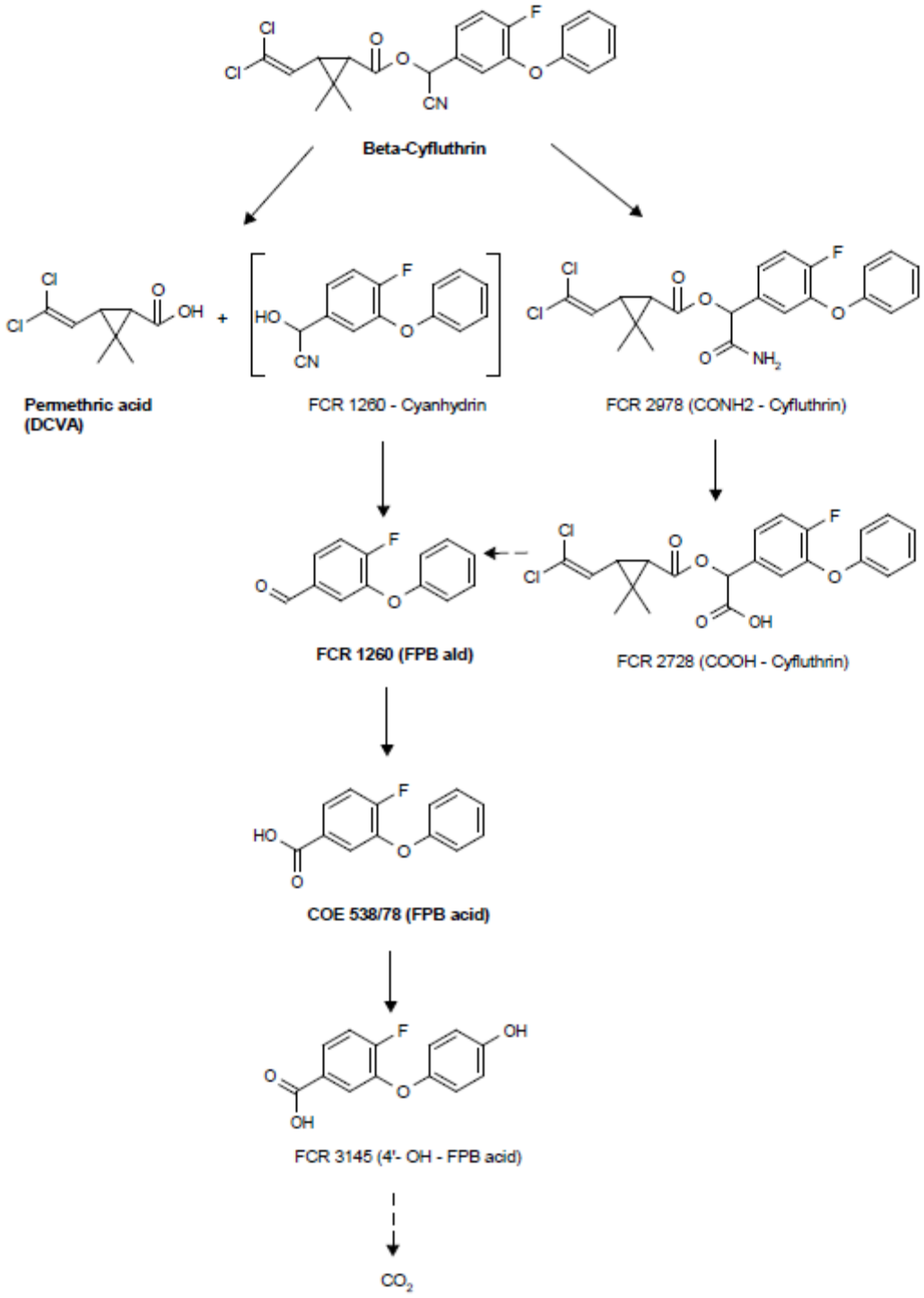


Figure B.8.2-1: Proposed pathway of cyfluthrin in sediment-water system under aerobic conditions. The metabolites DCVA, FPB-ald and FPB-acid are calculated by Hammel & Porschewski 2013b.

B.8.2.2.2 Aerobic mineralisation in surface water

The degradation of cyfluthrin in natural water was studied by Anderson 1986. The study was evaluated in the monograph dated 01 October 1996. The study was accepted.

The main degradation product was FPB-acid. It was assumed that the degradation occurred due to abiotic factors like alkaline pH and inorganic and organic compounds (e.g. copper and humic substances). The degradation decreased after day 7, an adsorption of cyfluthrin onto fine particles was assumed reducing the availability for degradation process.

B.8.3 Fate and behaviour in air

In the monograph dated 01 October 1996 the vapour pressure of cyfluthrin was based on a study by Sewekow 1981. For the renewal of the Annex I approval the vapour pressure was reassessed for the active isomers II and IV (pure active ingredient) and with technical grade of current production. The new studies are summarised in Volume 3 B.2 (Physical and chemical properties). For isomer II, the vapour pressure is 4.5×10^{-7} Pa (20 °C), for isomer IV a vapour pressure of 2.2×10^{-6} (at 20 °C) was determined.

The tropospherical half-life of beta-cyfluthrin was calculated to be below one day (17.8 h) using the Atkinson approach (Hellpointner, 1992, Monograph of 01 October 1996).

A study on the volatilisation was performed with beta-cyfluthrin (Fritz, 1993, monograph dated 01 October 1996). On average 18 % of the applied radioactivity was volatilised from the plant surfaces within 24 hours and 9 % from the soil surface. If the whole system (plants + soil) is considered, losses ranged from 0 – 32 % of the applied radioactivity.

B.8.4 Monitoring data concerning fate and behaviour of the active substance, metabolites, degradation and reaction products

No studies

B.8.5 References relied on

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA Section 7 /01	Krohn, J.	1997	Hydrolysis of Cyfluthrin and Betacyfluthrin as a function of pH Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 145000926, Edition Number: M-043171-01-1 Date: 1997-10-02 GLP/GEP: yes, unpublished **...also filed: KCA 2.8 /05** **...also filed: KCA 7.2.1 /02** **...also filed: KCA 7.2.1.1 /05**	N	N		Bayer CropScience
KCA Section 7 /02	Heimbach, F.	1989	Biological effects and fate of FCR 4545 EC 025 (R Bulldock) in experimental ponds Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: M-022657-01-2, Edition Number: M-022657-01-2 Date: 1989-12-21 GLP/GEP: no, unpublished **...also filed: KCA 8.2.8 /01**	N	N		Bayer CropScience

Annex point / reference number	**Author(s)**	**Year**	**Title** **Source** ***(where different from company)*** **Company name, Report No., Date, GLP status** ***(where relevant)*****, published or not**	**Vertebrate study Y/N**	**Data protection claimed Y/N**	**Justification if data protection is claimed**	**Owner**
KCA Section 7 /03	Leicht, W.; Fuchs, R.; Londershausen, M.	1996	Stability and biological activity of cyfluthrin isomers Publisher:SCI, Location:Great Britain, Journal:Pesticide Science, Volume:48, Pages:325-332, Year:1996, Report No.: MO-01-000681, Edition Number: M-031723-01-1 GLP/GEP: n.a., published	N	N		
KCA 7.1 /01	Krauskopf, B.; Grau, R.	1995	Cyfluthrin - Statement on behaviour and fate of the major metabolites especially permetric acid (DCVA) in soil and in water Bayer AG, Leverkusen, Germany Report No.: M9663, Edition Number: M-073368-01-2 Date: 1995-06-14 GLP/GEP: n.a., unpublished **...also filed: KCA 7.2 /01**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1 /02	Meylan, W.; Howard, P. H.; Boethling, R. S.	1992	Molecular topology/fragment contribution method for predicting soil sorption coefficients Publisher:American Chemical Society, Journal:Environ. Sci. Technol., Volume:26, Issue:8, Pages:1560-1567, Year:1992, Report No.: M-460516-01-1, Edition Number: M-460516-01-1 GLP/GEP: n.a., published	N	N		
KCA 7.1.1.1 /01	Minor, R. G.; Ernst, V. J.	1983	Radioactive residues of Baythroid TM in rotational crops Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: MR86050, Edition Number: M-067406-01-1 Method Report No.: MR86050 EPA MRID No.: 00131496, 00137541 Date: 1983-09-15 GLP/GEP: no, unpublished **...also filed: KCA 6.6.2 /01**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.1 /02	Leslie, W. L.	1988	Baythroid R - residues in field rotational crops: field Cambridge Analytical Associates, Inc., Boston, MA, USA Bayer CropScience, Report No.: MR98429, Edition Number: M-067638-01-1 Method Report No.: MR98429 Date: 1988-11-28 GLP/GEP: yes, unpublished **...also filed: KCA 6.2.1 /08** **...also filed: KCA 6.6.2 /02**	N	N		Bayer CropScience
KCA 7.1.1.1 /03	Leslie, W. L.	1989	Baythroid R - residues in field rotational Cereal crops - addendum no. 1 Mobay Chemical Corporation, Stilwell, KS, USA Bayer CropScience, Report No.: MR98429-1, Edition Number: M-067604-01-1 Method Report No.: MR98429-1 EPA MRID No.: 40942701, 41190202 Date: 1989-07-12 GLP/GEP: yes, unpublished **...also filed: KCA 6.2.1 /09** **...also filed: KCA 6.6.2 /03**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.1 /04	Wagner, K.; Neitzel, H.; Oehlmann, L.	1983	Degradation of Baythroid R in soil under aerobic and anaerobic test conditions Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: RA-87/83, Edition Number: M-072890-01-2 EPA MRID No.: 00131494 Date: 1983-01-19 GLP/GEP: no, unpublished **...also filed: KCA 7.1.1.2 /01**	N	N		Bayer CropScience
KCA 7.1.1.1 /05	Minor, R. G.	1986	Comparison of [^{14}C] R Baythroid degradation in soils from the United states and West Germany Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 91816, Edition Number: M-072885-01-1 Date: 1986-02-19 GLP/GEP: no, unpublished	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.1 /06	Roberts, T. R.; Standen, M. E.	1977	Degradation of the pyrethroid cypermethrin NRDC 149 (±)-a-cyano-3-phenoxybenzyl (±)-cis,trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate and the respective cis-(NRDC 160) and trans- (NRDC 159) isomers in soils Publisher:Shell Research Limited, Journal:Pesticide Science, Volume:8, Pages:305-319, Year:1977, Report No.: 90262, Edition Number: M-073407-02-1 GLP/GEP: n.a., published	N	N		
KCA 7.1.1.1 /07	Roberts, T. R.; Standen, M. E.	1980	Further studies of the degradation of the pyrethroid insecticide cypermethrin in soils Location:USA, Journal:Pesticide Science, Volume:12, Pages:285-296, Year:1981, Report No.: M2330, Edition Number: M-073380-01-1 GLP/GEP: n.a., published	N	N		

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.1 /08	Sakata, S.; Mikami, N.; Matsuda, T.; Miyamoto, J.	1986	Degradation and leaching behavior of the pyrethroid insecticide cypermethrin in soils Journal:Journal of Pesticide Science, Volume:11, Pages:71 - 79, Year:1986, Report No.: MO-02-010564, Edition Number: M-074042-01-1 GLP/GEP: n.a., published	N	N		
KCA 7.1.1.1 /09	Kaufman, D. D.; Haynes, S. C.; Jordan, E. G.; Kayser, A. J.	1977	Permethrin degradation in soil and microbial cultures Journal:American Chemical Society, Volume:42, Pages:147-161, Year:1977, Report No.: M9, Edition Number: M-073411-01-1 GLP/GEP: n.a., published	N	N		
KCA 7.1.1.1 /10	Kaneko, H.; Ohkawa, H.; Miyamoto, J.	1977	Degradation and movement of permethrin isomers in soil Location:Japan, Journal:Journal of Pesticide Science, Volume:3, Pages:43-51, Year:1978, Report No.: 86095, Edition Number: M-073393-01-1 GLP/GEP: n.a., published	N	N		

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.1 /11	Jordan, E. G.; Kaufman, D. D.; Kayser, A. J.	1981	The effect of soil temperature on the degradation of cis, trans - permethrin Location:Maryland, Journal:Journal of the Environmental Science and Health, Volume:B17, Issue:1, Pages:1-17, Year:1982, Report No.: M768, Edition Number: M-073370-01-1 GLP/GEP: n.a., published	N	N		
KCA 7.1.1.1 /12	Sakata, S., Mikami, N., Yamada, H.	1992	Degradation of pyrethroid optical isomers in soils Journal:Journal of Pesticide Science, Volume:17, Pages:169-180, Year:1992, Report No.: MO-04-003278, Edition Number: M-001653-01-1 GLP/GEP: n.a., published	N	N		

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.1 /13	Jordan, E. G.; Kaufman, D. D.	1986	Degradation of cis- and trans-permathrin in flooded soil Publisher:Journal of Agricultural and Food Chemistry, Prinecton, NJ, U, Location:USA, Journal:Journal of Agricultural and Food Chemistry, Volume:34, Issue:5, Pages:880-884, Year:1986, Report No.: M4986, Edition Number: M-073356-01-1 GLP/GEP: n.a., published	N	N		
KCA 7.1.1.1 /14	Anon.	1983	FCR 1272; AI; soil; degradation; BBA Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: RR5621/82, Edition Number: M-073011-01-2 Date: 1983-05-20 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.1.1 /15	Yoshida, H.; Yoshimoto, Y.; Takase, I.	1984	Residual fate of cyfluthrin (FCR 1272) in soils under laboratory and field conditions Nihon Tokushu Noyaku Seizo K. K., Japan Bayer CropScience, Report No.: NR1197, 1197 (ESR) Edition Number: M-072767-01-1 Date: 1984-05-28 GLP/GEP: no, unpublished **...also filed: KCA 4.2 /02** **...also filed: KCA 7.1.2.1 /01** **...also filed: KCA 7.1.2.2.1 /12**	N	N		Bayer CropScience

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Black writing indicates a Supplementary Dossier Study

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.1 /16	Riegner, K.	1997	Aerobic degradation of cyfluthrin in soil at low temperature according to EC requirements Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF4241, Edition Number: M-022206-01-1 Date: 1997-06-02 GLP/GEP: yes, unpublished	N	N		Bayer CropScience
KCA 7.1.1.1 /17	Jersch-Schmitz, S.	1997	Calculation of DT_{50}- and DT_{90}-values of cyfluthrin isomers in soil Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: M9677, Edition Number: M-022236-01-1 Date: 1997-06-03 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.1.1 /18	Hiler, T.	2013	Aerobic soil metabolism of [cyclopropyl-2-^{14}C]ß-cyfluthrin in three soils PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2365W, Edition Number: M-466494-01-1 Date: 2013-10-07 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.2.1 /02** **...also filed: KCA 7.1.2.1.1 /02** **...also filed: KCA 7.2.1 /17** **...also filed: KCA 7.2.1.1 /03**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.1 /19	Hiler, T.	2013	Aerobic soil metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin in one soil - (FCR4545) PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2326W, Edition Number: M-466491-01-1 Date: 2013-10-02 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.2.1 /03** **...also filed: KCA 7.1.2.1.1 /01** **...also filed: KCA 7.2.1 /16** **...also filed: KCA 7.2.1.1 /02**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita
KCA 7.1.1.2 /01	Wagner, K.; Neitzel, H.; Oehlmann, L.	1983	Degradation of Baythroid R in soil under aerobic and anaerobic test conditions Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: RA-87/83, Edition Number: M-072890-01-2 EPA MRID No.: 00131494 Date: 1983-01-19 GLP/GEP: no, unpublished **...also filed: KCA 7.1.1.1 /04**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.2 /02	Hiler, T.	2013	Anaerobic degradation of [fluorophenyl-UL and cyclopropane-1-^{14}C]ß-cyfluthrin in one soil PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2382W, Edition Number: M-468475-01-1 Date: 2013-10-30 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.2.1.3 /01**	N	Y	to fulfil data requirement for beta-cyfluthrin (degradation in soil)	BCS-Irvita
KCA 7.1.1.3 /01	Puhl, R. J.; Hurley, J. B.; Dime, R. A.	1983	Photodecomposition of Baythroid-^{14}C in aqueous solution and on soil Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 86182, Edition Number: M-072776-01-1 EPA MRID No.: 00137543 Date: 1983-12-02 GLP/GEP: no, unpublished **...also filed: KCA 2.8 /08** **...also filed: KCA 7.2.1 /05** **...also filed: KCA 7.2.1.2 /02**	N	N		Bayer CropScience
KCA 7.1.1.3 /02	Chopade, H. M.	1986	Photodecomposition of (^{14}C) R Baythroid on soil Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 88981, Edition Number: M-072660-01-1 EPA MRID No.: 00157043 Date: 1986-01-09 GLP/GEP: no, unpublished	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.1.3 /03	Roy L. Holmstead,* John E. Casida, Luis 0. Ruzo, and Donald G. Fullmer	1977	Pyrethroid photodecomposition: Permethrin Journal:Journal of Agricultural and Food Chemistry, Volume:26, Issue:3, Pages:590-595, Report No.: IM713, Edition Number: M-073341-01-1 GLP/GEP: n.a., published **...also filed: KCA 7.2.1 /15**	N	N		
KCA 7.1.1.3 /04	Takahashi, N.; Mikami, N.; Matsuda, T.; Miyamoto, J.	1985	Photodegradation of the pyrethroid insecticide cypermethrin in water and on soil surface Journal:Journal of Pesticide Science, Volume:10, Issue:4, Pages:629-642, Year:1985, Edition Number: M-072742-01-1 GLP/GEP: n.a., published **...also filed: KCA 7.2.1 /14** **...also filed: KCA 7.2.1.2 /05**	N	N		e
KCA 7.1.1.3 /05	Adam, D.	2013	[^{14}C] beta-cyfluthrin - Photodegradation on soil surface Innovative Environmental Services (IES) Ltd, Witterswil, Switzerland BCS-Irvita, Report No.: 20120108, Edition Number: M-481075-01-1 Date: 2013-03-15 GLP/GEP: n.a., unpublished	N	Y		BCS-Irvita

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.2 /01	Anon.	1983	Effect of frozen storage at 0 to -10 degrees Fahrenheit on residues Mobay Chemical Corporation, Kansas City, MO, USA Report No.: MO-04-009081, Edition Number: M-088204-01-1 Date: 1983-09-23 GLP/GEP: no, unpublished **...also filed: KCA 6.1 /13**	N	N		Bayer CropScience
KCA 7.1.2 /02	Grace, T. J.	1990	Freezer storage stability of FPBacid and DCVA in soil Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 100153, Edition Number: M-073783-01-1 Date: 1990-09-07 GLP/GEP: yes, unpublished	N	N		Bayer CropScience
KCA 7.1.2.1 /01	Yoshida, H.; Yoshimoto, Y.; Takase, I.	1984	Residual fate of cyfluthrin (FCR 1272) in soils under laboratory and field conditions Nihon Tokushu Noyaku Seizo K. K., Japan Bayer CropScience, Report No.: NR1197, 1197 (ESR) Edition Number: M-072767-01-1 Date: 1984-05-28 GLP/GEP: no, unpublished **...also filed: KCA 4.2 /02** **...also filed: KCA 7.1.1.1 /15** **...also filed: KCA 7.1.2.2.1 /12**	N	N		Bayer CropScience

Annex point / reference number	**Author(s)**	**Year**	**Title** **Source** ***(where different from company)*** **Company name, Report No., Date, GLP status** ***(where relevant)*****, published or not**	**Vertebrate study Y/N**	**Data protection claimed Y/N**	**Justification if data protection is claimed**	**Owner**
KCA 7.1.2.1 /02	Hiler, T.	2013	Aerobic soil metabolism of [cyclopropyl-2-^{14}C]ß-cyfluthrin in three soils PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2365W, Edition Number: M-466494-01-1 Date: 2013-10-07 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.1.1 /18** **...also filed: KCA 7.1.2.1.1 /02** **...also filed: KCA 7.2.1 /17** **...also filed: KCA 7.2.1.1 /03**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita
KCA 7.1.2.1 /03	Hiler, T.	2013	Aerobic soil metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin in one soil - (FCR4545) PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2326W, Edition Number: M-466491-01-1 Date: 2013-10-02 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.1.1 /19** **...also filed: KCA 7.1.2.1.1 /01** **...also filed: KCA 7.2.1 /16** **...also filed: KCA 7.2.1.1 /02**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.2.1.1 /01	Hiler, T.	2013	Aerobic soil metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin in one soil - (FCR4545) PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2326W, Edition Number: M-466491-01-1 Date: 2013-10-02 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.1.1 /19** **...also filed: KCA 7.1.2.1 /03** **...also filed: KCA 7.2.1 /16** **...also filed: KCA 7.2.1.1 /02**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita
KCA 7.1.2.1.1 /02	Hiler, T.	2013	Aerobic soil metabolism of [cyclopropyl-2-^{14}C]ß-cyfluthrin in three soils PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2365W, Edition Number: M-466494-01-1 Date: 2013-10-07 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.1.1 /18** **...also filed: KCA 7.1.2.1 /02** **...also filed: KCA 7.2.1 /17** **...also filed: KCA 7.2.1.1 /03**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita

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KCA 7.1.2.1.2 /01	Calovich, C. A.	1987	Dissipation of Baythroid and its major metabolites in field soil Chemonics Laboratories, Phoenix, AZ, USA Bayer CropScience, Report No.: MR95071, Edition Number: M-070039-01-1 Date: 1987-11-13 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.2.2.1 /11**	N	N		Bayer CropScience
KCA 7.1.2.1.2 /02	Hellpointer, E.; Junge, T.	2013	Beta-cyfluthrin-FPB-acid: Aerobic degradation in three soils BCS-Irvita, Report No.: EnSa-13-0668, Edition Number: M-470667-01-1 Date: 2013-11-15 GLP/GEP: yes, unpublished	N	Y	to complete data requirement for soils (3 soils required)	BCS-Irvita
KCA 7.1.2.1.2 /03	Hammel, K.; Porschewski, R.	2013	Kinetic evaluation of aerobic metabolism of beta-cyfluthrin and its metabolites in soil according to FOCUS Kinetics BCS-Irvita, Report No.: EnSa-13-0876, Edition Number: M-471163-01-1 Date: 2013-11-26 GLP/GEP: no, unpublished	N	Y	to fulfil data requirement for beta-cyfluthrin (degradation in soil)	BCS-Irvita

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KCA 7.1.2.1.3 /01	Hiler, T.	2013	Anaerobic degradation of [fluorophenyl-UL and cyclopropane-1-^{14}C]ß-cyfluthrin in one soil PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2382W, Edition Number: M-468475-01-1 Date: 2013-10-30 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.1.2 /02**	N	Y	to fulfil data requirement for beta-cyfluthrin (degradation in soil)	BCS-Irvita
KCA 7.1.2.2.1 /01	Anon.	1991	FCR 4545; 25 EC; Boden; Deutschland; BBA Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 0197-89, Edition Number: M-033990-01-1 Date: 1991-06-10 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.2.2.1 /02	Anon.	1991	FCR 4545; 25 EC; Boden; Deutschland; BBA Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 0201-89, Edition Number: M-033985-01-1 Date: 1991-06-10 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.2.2.1 /03	Anon.	1991	FCR 4545; 25 EC; Boden; Deutschland; BBA Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 0202-89, Edition Number: M-033980-01-1 Date: 1991-06-10 GLP/GEP: no, unpublished	N	N		Bayer CropScience

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Black writing indicates a Supplementary Dossier Study

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.2.2.1 /04	Anon.	1991	FCR 4545; 25 EC; Boden; Deutschland; BBA Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 0204-89, Edition Number: M-033962-01-1 Date: 1991-06-10 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.2.2.1 /05	Anon.	1991	FCR 4545; 25 EC; Boden; Deutschland, BBA Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 0205-89, Edition Number: M-033950-01-1 Date: 1991-06-10 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.2.2.1 /06	Anon.	1982	Soil residue trial: Baythroid, Versuchsgut Höfchen, Germany FCR 1272; 10 WP;; Germany; BBA Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 5643-81, Edition Number: M-072886-01-2 Date: 1982-04-01 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.2.2.1 /07	Anon.	1982	Soil residue trial: Baythroid, Versuchsgut Höfchen, Germany FCR 1272; 10 WP; soil; Germany; BBA Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 5644-81, Edition Number: M-072884-01-2 Date: 1982-04-01 GLP/GEP: no, unpublished	N	N		Bayer CropScience

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Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.2.2.1 /08	Anon.	1983	Baythroid, Loam, Ontario/Canada. FCR 1272; 240 EC; soil; USA Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 84344, Edition Number: M-072862-01-1 Date: 1983-09-19 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.2.2.1 /09	Anon.	1983	Baythroid, Silty Loam, Mississippi/USA. FCR 1272; 240 EC; soil; USA Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 84346, Edition Number: M-072840-01-1 Date: 1983-09-19 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.2.2.1 /10	Anon.	1983	Baythroid, Loamy Sand, Georgia/USA. FCR 1272; 240 EC; soil; USA Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 84348, Edition Number: M-072826-01-1 Date: 1983-09-19 GLP/GEP: no, unpublished	N	N		Bayer CropScience

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KCA 7.1.2.2.1 /11	Calovich, C. A.	1987	Dissipation of Baythroid and its major metabolites in field soil Chemonics Laboratories, Phoenix, AZ, USA Bayer CropScience, Report No.: MR95071, Edition Number: M-070039-01-1 Date: 1987-11-13 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.2.1.2 /01**	N	N		Bayer CropScience
KCA 7.1.2.2.1 /12	Yoshida, H.; Yoshimoto, Y.; Takase, I.	1984	Residual fate of cyfluthrin (FCR 1272) in soils under laboratory and field conditions Nihon Tokushu Noyaku Seizo K. K., Japan Bayer CropScience, Report No.: NR1197, Edition Number: M-072767-01-1 Date: 1984-05-28 GLP/GEP: no, unpublished **...also filed: KCA 4.2 /02** **...also filed: KCA 7.1.1.1 /15** **...also filed: KCA 7.1.2.1 /01**	N	N		Bayer CropScience
KCA 7.1.2.2.1 /13	Anon.	1983	Baythroid, Sandy Clay Loam, Arizona/USA FCR 1272; 240 EC; soil; USA Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 84343, Edition Number: M-072869-01-1 Date: 1983-09-19 GLP/GEP: no, unpublished	N	N		Bayer CropScience

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KCA 7.1.2.2.1 /14	Anon.	1983	Baythroid, Sand, Florida/USA. FCR 1272; 240 EC; soil; USA Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 84349, Edition Number: M-072818-01-1 Date: 1983-09-19 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.2.2.1 /15	Anon.	1983	Baythroid, Sand, Florida/USA Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 84350, Edition Number: M-072812-01-1 Date: 1983-09-19 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.2.2.1 /16	Robinson, N.	2014	Beta-cyfluthrin - Field soil dissipation of beta-cyfluthrin from a field trial carried out in southern France Innovative Enviromental Service Ltd, Witterswil, Switzerland Irvita Plant Protection, Report No.: 20120152, Edition Number: M-482355-01-1 Date: 2014-04-01 GLP/GEP: yes, unpublished **...also filed: KCA 4.1.2 /74**	N	Y	data not submitted on EU Level	Irvita Plant Protection

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KCA 7.1.2.2.1 /17	Robinson, N.	2014	Beta-cyfluthrin - Field soil dissipation of beta-cyfluthrin from a field trial carried out in northern France Innovative Enviromental Services Ltd., Witterswil, Switzerland Irvita Plant Protection, Report No.: 20120153, Edition Number: M-482356-01-1 Date: 2014-04-01 GLP/GEP: yes, unpublished	N	Y	data not submitted on EU Level	Irvita Plant Protection
KCA 7.1.2.2.1 /18	Robinson, N.	2014	Beta-cyfluthrin - Field soil dissipation of beta-cyfluthrin from a field trial carried out in Spain Innovative Enviromental Services Ltd., Witterswil, Switzerland Irvita Plant Protection, Report No.: 20120154, Edition Number: M-482359-01-1 Date: 2014-04-02 GLP/GEP: yes, unpublished	N	Y	data not submitted on EU Level	Irvita Plant Protection
KCA 7.1.2.2.1 /19	Robinson, N.	2014	Beta-cyfluthrin - Field soil dissipation of beta-cyfluthrin from a field trial carried out in Germany Innovative enviromental Science Ltd., Witterswil, Switzerland Irvita Plant Protection, Report No.: 20120155, Edition Number: M-482358-01-1 Date: 2014-04-02 GLP/GEP: yes, unpublished	N	Y	data not submitted on EU Level	Irvita Plant Protection

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KCA 7.1.3.1 /01	Gronberg, R. R.	1987	Adsorption of Baythroid TM to sandy loam Mobay Chemical Corporation, Stilwell, KS, USA Bayer CropScience, Report No.: M5184, 94541, Edition Number: M-073507-01-1 EPA MRID No.: 40223402 Date: 1987-05-12 GLP/GEP: no, unpublished **...also filed: KCA 7.1.3.1.1 /01**	N	N		Bayer CropScience
KCA 7.1.3.1 /02	Burhenne, J.	1996	Adsorption/desorption of cyfluthrin on soils Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 1310003-8, Edition Number: M-022224-01-1 Date: 1996-04-29 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.3.1.1 /02**	N	N		Bayer CropScience
KCA 7.1.3.1 /03	Rice, P.; Seebinger, J.; Koskinen, W.	2006	Experimental Variability in Characterisation of Cyfluthrin Sorption to Soil. Journal:J. Environ. Sci. Health, Part B, Volume:41, Issue:4, Pages:323-331, Year:2006, Edition Number: M-459459-01-1 Date: 2006-12-31 GLP/GEP: no, published **...also filed: KCA 7.1.3.1.1 /03**	N	N		

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KCA 7.1.3.1.1 /01	Gronberg, R. R.	1987	Adsorption of Baythroid TM to sandy loam Mobay Chemical Corporation, Stilwell, KS, USA Bayer CropScience, Report No.: M5184, Edition Number: M-073507-01-1 EPA MRID No.: 40223402 Date: 1987-05-12 GLP/GEP: no, unpublished ...also filed: KCA 7.1.3.1 /01	N	N		Bayer CropScience
KCA 7.1.3.1.1 /02	Burhenne, J.	1996	Adsorption/desorption of cyfluthrin on soils Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 1310003-8, Edition Number: M-022224-01-1 Date: 1996-04-29 GLP/GEP: yes, unpublished ...also filed: KCA 7.1.3.1 /02	N	N		Bayer CropScience
KCA 7.1.3.1.1 /03	Rice, P.; Seebinger, J.; Koskinen, W.	2006	Experimental Variability in Characterisation of Cyfluthrin Sorption to Soil. Journal:J. Environ. Sci. Health, Part B, Volume:41, Issue:4, Pages:323-331, Year:2006, Edition Number: M-459459-01-1 Date: 2006-12-31 GLP/GEP: no, published **...also filed: KCA 7.1.3.1 /03**	N	N		

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KCA 7.1.3.1.1 /04	Hiler, T.	2013	Soil adsorption/desorption of [^{14}C]FPB acid by the batch Equilibrium method PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2381W-2, Edition Number: M-466496-02-1 Date: 2013-10-01 **...Amended: 2014-04-16** GLP/GEP: yes, unpublished **...also filed: KCA 7.1.3.1.2 /04**	N	Y	to provide sorption data for major metabolite	BCS-Irvita
KCA 7.1.3.1.2 /01	Hellpointner, E.	1995	SAR-Assessment of Koc of permethrinic acid by the program PCKOCWIN Year:1995, Report No.: M9195, Edition Number: M-069631-01-1 GLP/GEP: n.a., published	N	N		
KCA 7.1.3.1.2 /02	Slangen, P. J.	1999	Adsorption/desorption of FCR 1272-permethric acid on soil Notox B.V., 's-Hertogenbosch, Netherlands Bayer CropScience, Report No.: IM1983, Edition Number: M-015423-01-1 Date: 1999-08-30 GLP/GEP: yes, unpublished	N	N		Bayer CropScience

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KCA 7.1.3.1.2 /03	Oddy, A. M.; Brett, R.	2005	[^{14}C]-AE F105561: Adsorption to and desorption from five soils Battelle AgriFood Ltd., Ongar, Essex, United Kingdom Bayer CropScience, Report No.: CX/05/054, Edition Number: M-263792-01-1 Date: 2005-12-05 GLP/GEP: yes, unpublished	N	Y	data not submitted on EU level	BCS-Irvita
KCA 7.1.3.1.2 /04	Hiler, T.	2013	Soil adsorption/desorption of [^{14}C]FPB acid by the batch Equilibrium method PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2381W-2, Edition Number: M-466496-02-1 Date: 2013-10-01 **...Amended: 2014-04-16** GLP/GEP: yes, unpublished **...also filed: KCA 7.1.3.1.1 /04**	N	Y	to provide sorption data for major metabolite	BCS-Irvita
KCA 7.1.3.1.2 /05	Hein, W.; D'Ambrosio, A.	2013	[cyclopropane-1-^{14}C] BCS-AA53389: Adsorption/desorption in five different soils RLP AgroScience GmbH, Neustadt a. d. Weinstraße, Germany BCS-Irvita, Report No.: AS251, Edition Number: M-455223-01-1 Date: 2013-04-04 GLP/GEP: yes, unpublished	N	Y	to provide sorption data for major metabolite	BCS-Irvita

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KCA 7.1.4.1 /01	Whitfield, S. L.; Obrist, J. J.	1983	Soil thin-layer mobility of aged extracts of soil treated with amethydione, Baythroid, Alsystin, and BAJ SLJ 0312 Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: MR86158, Edition Number: M-041538-03-1 EPA MRID No.: 00137544 Date: 1983-10-12 **...Amended: 1986-04-22** GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.1.4.1 /02	Kaufman, D. D.; Russell, A. B.; Helling, C. S.; Kayser, A. J.	1981	Movement of cypermethrin, decamethrin, permethrin, and their degradation products in soil Publisher:American Chemical Society, Location:USA, Journal:Journal of Agricultural and Food Chemistry, Volume:29, Pages:239 - 245, Year:1981, Report No.: M950, Edition Number: M-069699-01-1 GLP/GEP: n.a., published	N	N		

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KCA 7.1.4.1.1 /01	Obrist, J. J.; Thornton, J. S.	1982	Soil thin-layer mobility of BAY FCR 1272, BAY SIR 9514, and BAY SSH 0860 Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: MR82232, Edition Number: M-041545-01-1 EPA MRID No.: 00131495, 00137540 Date: 1982-07-15 GLP/GEP: no, unpublished **...also filed: KCA 7.1.4.1.2 /02**	N	N		Bayer CropScience
KCA 7.1.4.1.1 /02	Fenoll, J.; Ruiz, E.; Navarro, S.; Flores, P.; Hellin, P.	2011	Reduction of the movement and persistence of pesticides in soil through common agronomic practices. Journal:Chemosphere, Volume:85, Issue:8, Pages:1375-1382, Year:2011, Report No.: M-459846-01-1, Edition Number: M-459846-01-1 Date: 2011-12-31 GLP/GEP: no, published	N	N		
KCA 7.1.4.1.2 /01	Schaefer, H.; Krauskopf, B.	1995	Predicted environmental concentration of the cyfluthrin metabolite DCVA (permethric acid) in ground water recharge based on the calculation of PELMO Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: M9196, Edition Number: M-069309-01-1 Date: 1995-10-15 GLP/GEP: no, unpublished	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.1.4.1.2 /02	Obrist, J. J.; Thornton, J. S.	1982	Soil thin-layer mobility of BAY FCR 1272, BAY SIR 9514, and BAY SSH 0860 Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: MR82232, Edition Number: M-041545-01-1 EPA MRID No.: 00131495, 00137540 Date: 1982-07-15 GLP/GEP: no, unpublished **...also filed: KCA 7.1.4.1.1 /01**	N	N		Bayer CropScience
KCA 7.1.4.2 /01	Scholz, K.; Umgelder, U.	1985	Leaching characteristics of cyfluthrin (FCR 1272; Baytthroid) aged in soil Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF2474, Edition Number: M-073678-01-2 Date: 1985-09-27 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.2 /01	Krauskopf, B.; Grau, R.	1995	Cyfluthrin - Statement on behaviour and fate of the major metabolites especially permetric acid (DCVA) in soil and in water Bayer AG, Leverkusen, Germany Report No.: M9663, Edition Number: M-073368-01-2 Date: 1995-06-14 GLP/GEP: n.a., unpublished **...also filed: KCA 7.1 /01**	N	N		Bayer CropScience

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KCA 7.2 /02	Ganzelmeier, H.; Rautmann, D.; Spangenberg, R.; Streloke. M.; Herrmann, M.; Wenzelburger, H. J.; Walter, H. F.	1995	Untersuchungen zur Abdrift von Pflanzenschutzmitteln Publisher:Blackwell Wissenschafts-Verlag, Location:Berlin/Wien, Germany, Journal:Mitteilungen aus der Biologischen Bundesanstalt fuer Land- und Forstwirtschaft, Volume:304, Pages:110, Year:1995, Report No.: HEFT 304, Edition Number: M-307374-01-1 GLP/GEP: n.a., published	N	N		
KCA 7.2.1 /01	Krohn, J.	1983	FCR 1272 - Fate/behaviour of crop protection products in water Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: M1590, Edition Number: M-137221-01-2 Date: 1983-04-22 GLP/GEP: no, unpublished **...also filed: KCA 2.8 /01** **...also filed: KCA 7.2.1.1 /06**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1 /02	Krohn, J.	1997	Hydrolysis of Cyfluthrin and Betacyfluthrin as a function of pH Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 145000926, Edition Number: M-043171-01-1 Date: 1997-10-02 GLP/GEP: yes, unpublished **...also filed: KCA 2.8 /05** **...also filed: KCA 7.2.1.1 /05** **...also filed: KCA Section 7 /01**	N	N	global usable	Bayer CropScience
KCA 7.2.1 /03	Wilmes, R.	1980	Preliminary studies on stability to light Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: WFL-OL-008, Edition Number: M-072806-02-1 Date: 1980-02-19 **...Amended: 1980-03-18** GLP/GEP: no, unpublished **...also filed: KCA 2.8 /06** **...also filed: KCA 7.2.1.2 /01**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1 /04	Westphal, C.	1984	Report on studies to investigate photodegradation of cyfluthrin Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: M2034, Edition Number: M-073670-01-2 Date: 1984-10-16 GLP/GEP: no, unpublished **...also filed: KCA 2.8 /07** **...also filed: KCA 7.2.1.2 /03** **...also filed: KCA 7.2.1.3 /01**	N	N		Bayer CropScience
KCA 7.2.1 /05	Puhl, R. J.; Hurley, J. B.; Dime, R. A.	1983	Photodecomposition of Baythroid-^{14}C in aqueous solution and on soil Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 86182, Edition Number: M-072776-01-1 EPA MRID No.: 00137543 Date: 1983-12-02 GLP/GEP: no, unpublished **...also filed: KCA 2.8 /08** **...also filed: KCA 7.1.1.3 /01** **...also filed: KCA 7.2.1.2 /02**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1 /06	Gronberg, R. R.	1984	Photodecomposition of [Phenyl-UL-^{14}C] Baythroid in aqueous solution by sunlight Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 88598, Edition Number: M-040090-01-1 EPA MRID No.: 45022102 Date: 1984-10-18 GLP/GEP: no, unpublished **...also filed: KCA 2.8 /09** **...also filed: KCA 7.2.1.2 /04**	N	N		Bayer CropScience
KCA 7.2.1 /07	Hellpointner, E.	1991	Determination of the quantum yield and assessment of the environmental half-life of the direct photodegradation of cyfluthrin in water Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF3555, Edition Number: M-073620-01-2 Date: 1991-09-04 GLP/GEP: yes, unpublished **...also filed: KCA 2.8 /10**	N	N		Bayer CropScience
KCA 7.2.1 /08	Krohn, J.	1987	Calculation of the Henry Law Constant of Cyfluthrin Bayer AG, Leverkusen, Germany Report No.: PC 182, Edition Number: M-043077-01-1 Date: 1987-05-27 GLP/GEP: no, unpublished	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1 /09	Sandie, F. E.	1983	Hydrolysis of Baythroid TM in sterile, aqueous buffered solutions Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: MR86051, Edition Number: M-073571-01-1 EPA MRID No.: 00131493, 00137539 Date: 1983-10-07 GLP/GEP: no, unpublished **...also filed: KCA 2.8 /03** **...also filed: KCA 7.2.1.1 /01** **...also filed: KCA 7.2.2 /06** **...also filed: KCA 7.2.2.3 /01**	N	N		Bayer CropScience
KCA 7.2.1 /10	Anderson, C. A.	1986	Degradation of ^{14}C-Cyfluthrin in natural water Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF2542, Edition Number: M-073248-01-2 EPA MRID No.: 45022101 Date: 1986-02-26 GLP/GEP: no, unpublished **...also filed: KCA 7.2.2 /05** **...also filed: KCA 7.2.2.2 /01**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1 /11	Anderson, C.	1987	Degradation characteristics of cyfluthrin (Baythroid) in water/sediment systems Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF2875, Edition Number: M-071937-01-2 Date: 1987-10-01 GLP/GEP: no, unpublished **...also filed: KCA 7.2.2 /01** **...also filed: KCA 7.2.2.3 /02**	N	N		Bayer CropScience
KCA 7.2.1 /12	Krohn, J.	1997	Hydrolysis of Permethric acid as a function of pH Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 145000921, Edition Number: M-043185-01-1 Date: 1997-06-16 GLP/GEP: yes, unpublished **...also filed: KCA 7.2.1.1 /04**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1 /13	Sharom, M. S.; Solomon, K. R.	1989	The influence of adsorption on glass, pH and temperature on the disappearance of permethrin in aqueous systems Publisher:Applied Science Publishers Ltd,, Location:Great Britain, Journal:Environmental Pollution (Series B), Volume:4, Pages:269 - 279, Year:1982, Report No.: M4377, Edition Number: M-073354-01-1 GLP/GEP: n.a., published **...also filed: KCA 7.2.2 /04**	N	N		
KCA 7.2.1 /14	Takahashi, N.; Mikami, N.; Matsuda, T.; Miyamoto, J.	1985	Photodegradation of the pyrethroid insecticide cypermethrin in water and on soil surface Journal:Journal of Pesticide Science, Volume:10, Issue:4, Pages:629-642, Year:1985, Report No.: M7834, Edition Number: M-072742-01-1 GLP/GEP: n.a., published **...also filed: KCA 7.1.1.3 /04** **...also filed: KCA 7.2.1.2 /05**	N	N		

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1 /15	Roy, L.; Holmstead, L.; E.; C.; O.; R.; Fullmer, G.	1977	Pyrethroid photodecomposition: Permethrin Journal:Journal of Agricultural and Food Chemistry, Volume:26, Issue:3, Pages:590-595, Report No.: IM713, Edition Number: M-073341-01-1 GLP/GEP: n.a., published **...also filed: KCA 7.1.1.3 /03**	N	N		
KCA 7.2.1 /16	Hiler, T.	2013	Aerobic soil metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin in one soil - (FCR4545) PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2326W, Edition Number: M-466491-01-1 Date: 2013-10-02 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.1.1 /19** **...also filed: KCA 7.1.2.1 /03** **...also filed: KCA 7.1.2.1.1 /01** **...also filed: KCA 7.2.1.1 /02**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1 /17	Hiler, T.	2013	Aerobic soil metabolism of [cyclopropyl-2-^{14}C]ß-cyfluthrin in three soils PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2365W, Edition Number: M-466494-01-1 Date: 2013-10-07 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.1.1 /18** **...also filed: KCA 7.1.2.1 /02** **...also filed: KCA 7.1.2.1.1 /02** **...also filed: KCA 7.2.1.1 /03**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita
KCA 7.2.1.1 /01	Sandie, F. E.	1983	Hydrolysis of Baythroid TM in sterile, aqueous buffered solutions Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: MR86051, Edition Number: M-073571-01-1 EPA MRID No.: 00131493, 00137539 Date: 1983-10-07 GLP/GEP: no, unpublished **...also filed: KCA 2.8 /03** **...also filed: KCA 7.2.1 /09** **...also filed: KCA 7.2.2 /06** **...also filed: KCA 7.2.2.3 /01**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1.1 /02	Hiler, T.	2013	Aerobic soil metabolism of [fluorophenyl-^{14}C]beta-cyfluthrin in one soil - (FCR4545) PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2326W, Edition Number: M-466491-01-1 Date: 2013-10-02 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.1.1 /19** **...also filed: KCA 7.1.2.1 /03** **...also filed: KCA 7.1.2.1.1 /01** **...also filed: KCA 7.2.1 /16**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita
KCA 7.2.1.1 /03	Hiler, T.	2013	Aerobic soil metabolism of [cyclopropyl-2-^{14}C]ß-cyfluthrin in three soils PTRL West, Hercules, CA, USA BCS-Irvita, Report No.: 2365W, Edition Number: M-466494-01-1 Date: 2013-10-07 GLP/GEP: yes, unpublished **...also filed: KCA 7.1.1.1 /18** **...also filed: KCA 7.1.2.1 /02** **...also filed: KCA 7.1.2.1.1 /02** **...also filed: KCA 7.2.1 /17**	N	Y	to fulfill data requirement for beta-cyfluthrin degradation in soil	BCS-Irvita

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1.1 /04	Krohn, J.	1997	Hydrolysis of Permethric acid as a function of pH Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 145000921, Edition Number: M-043185-01-1 Date: 1997-06-16 GLP/GEP: yes, unpublished ...also filed: KCA 7.2.1 /12	N	N		Bayer CropScience
KCA 7.2.1.1 /05	Krohn, J.	1997	Hydrolysis of Cyfluthrin and Betacyfluthrin as a function of pH Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: 145000926, Edition Number: M-043171-01-1 Date: 1997-10-02 GLP/GEP: yes, unpublished ...also filed: KCA 2.8 /05 ...also filed: KCA 7.2.1 /02 ...also filed: KCA Section 7 /01	N	N		Bayer CropScience
KCA 7.2.1.1 /06	Krohn, J.	1983	FCR 1272 - Fate/behaviour of crop protection products in water Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: M1590, Edition Number: M-137221-01-2 Date: 1983-04-22 GLP/GEP: no, unpublished ...also filed: KCA 2.8 /01 ...also filed: KCA 7.2.1 /01	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1.1 /07	Xu, T.; Ripperger, R.	2013	Cyfluthrin and beta-cyfluthrin - Hydrolysis half-life evaluation (Supplemental information for Bayer report number 145000926, M-043171-01-1) Bayer CropScience LP, RTP, NC, USA Bayer CropScience, Report No.: US0360, Edition Number: M-456473-01-1 Date: 2013-06-14 GLP/GEP: no, unpublished	N	Y	Re-evaluation acc. to OECD 111	Bayer CropScience
KCA 7.2.1.2 /01	Wilmes, R.	1980	Preliminary studies on stability to light Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: WFL-OL-008, Edition Number: M-072806-02-1 Date: 1980-02-19 ...Amended: 1980-03-18 GLP/GEP: no, unpublished ...also filed: KCA 2.8 /06 ...also filed: KCA 7.2.1 /03	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1.2 /02	Puhl, R. J.; Hurley, J. B.; Dime, R. A.	1983	Photodecomposition of Baythroid-^{14}C in aqueous solution and on soil Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 86182, Edition Number: M-072776-01-1 EPA MRID No.: 00137543 Date: 1983-12-02 GLP/GEP: no, unpublished ...also filed: KCA 2.8 /08 ...also filed: KCA 7.1.1.3 /01 ...also filed: KCA 7.2.1 /05	N	N		Bayer CropScience
KCA 7.2.1.2 /03	Westphal, C.	1984	Report on studies to investigate photodegradation of cyfluthrin Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: M2034, Edition Number: M-073670-01-2 Date: 1984-10-16 GLP/GEP: no, unpublished ...also filed: KCA 2.8 /07 ...also filed: KCA 7.2.1 /04 ...also filed: KCA 7.2.1.3 /01	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1.2 /04	Gronberg, R. R.	1984	Photodecomposition of [Phenyl-UL-^{14}C] Baythroid in aqueous solution by sunlight Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: 88598, Edition Number: M-040090-01-1 EPA MRID No.: 45022102 Date: 1984-10-18 GLP/GEP: no, unpublished ...also filed: KCA 2.8 /09 ...also filed: KCA 7.2.1 /06	N	N		Bayer CropScience
KCA 7.2.1.2 /05	Takahashi, N.; Mikami, N.; Matsuda, T.; Miyamoto, J.	1985	Photodegradation of the pyrethroid insecticide cypermethrin in water and on soil surface Journal:Journal of Pesticide Science, Volume:10, Issue:4, Pages:629-642, Year:1985, Report No.: M7834, Edition Number: M-072742-01-1 GLP/GEP: n.a., published ...also filed: KCA 7.1.1.3 /04 ...also filed: KCA 7.2.1 /14	N	N		

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.1.2 /06	Hellpointner, E.; Malburg, G.	2013	Beta-Cyfluthrin: Determination of the Quantum Yield and Assessment of the Environmental Half-life of the Direct Photo-Degradation in Water BCS-Irvita, Report No.: EnSa-13-0519, Edition Number: M-463475-01-1 Date: 2013-09-02 GLP/GEP: yes, unpublished	N	Y	to complete data for soil (3 soils required)	BCS-Irvita
KCA 7.2.1.3 /01	Westphal, C.	1984	Report on studies to investigate photodegradation of cyfluthrin Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: M2034, Edition Number: M-073670-01-2 Date: 1984-10-16 GLP/GEP: no, unpublished ...also filed: KCA 2.8 /07 ...also filed: KCA 7.2.1 /04 ...also filed: KCA 7.2.1.2 /03	N	N		Bayer CropScience
KCA 7.2.2 /01	Anderson, C.	1987	Degradation characteristics of cyfluthrin (Baythroid) in water/sediment systems Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF2875, Edition Number: M-071937-01-2 Date: 1987-10-01 GLP/GEP: no, unpublished **...also filed: KCA 7.2.1 /11** **...also filed: KCA 7.2.2.3 /02**	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.2 /02	Sneikus, J.	2000	Aerobic aquatic degradation and metabolism of cyfluthrin in the water-sediment system Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: MR-268/00, Edition Number: M-022319-02-1 Date: 2000-09-15 **...Amended: 2000-10-24** GLP/GEP: yes, unpublished **...also filed: KCA 7.2.2.3 /03**	N	N		Bayer CropScience
KCA 7.2.2 /03	Sharom, M. S.; Solomon, K. R.	1981	Adsorption-desorption, degradation, and distribution of permethrin in aqueous systems Publisher:National Science and Engineering Research Council, Location:Canada, Journal:Journal of Agricultural and Food Chemistry, Volume:29, Pages:1122-1125, Year:1981, Report No.: M4869, Edition Number: M-073817-01-1 GLP/GEP: n.a., published	N	N		

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.2 /04	Sharom, M. S.; Solomon, K. R.	1989	The influence of adsorption on glass, pH and temperature on the disappearance of permethrin in aqueous systems Publisher:Applied Science Publishers Ltd,, Location:Great Britain, Journal:Environmental Pollution (Series B), Volume:4, Pages:269 - 279, Year:1982, Report No.: M4377, Edition Number: M-073354-01-1 GLP/GEP: n.a., published **...also filed: KCA 7.2.1 /13**	N	N		
KCA 7.2.2 /05	Anderson, C. A.	1986	Degradation of ^{14}C-Cyfluthrin in natural water Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF2542, Edition Number: M-073248-01-2 EPA MRID No.: 45022101 Date: 1986-02-26 GLP/GEP: no, unpublished **...also filed: KCA 7.2.1 /10** **...also filed: KCA 7.2.2.2 /01**	N	N		Bayer CropScience

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KCA 7.2.2 /06	Sandie, F. E.	1983	Hydrolysis of Baythroid TM in sterile, aqueous buffered solutions Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: MR86051, Edition Number: M-073571-01-1 EPA MRID No.: 00131493, 00137539 Date: 1983-10-07 GLP/GEP: no, unpublished **...also filed: KCA 2.8 /03** **...also filed: KCA 7.2.1 /09** **...also filed: KCA 7.2.1.1 /01** **...also filed: KCA 7.2.2.3 /01**	N	N		Bayer CropScience
KCA 7.2.2 /07	Rawn, G. P.; Webster, G. R. B.; Muir, D. C. G.	1982	Fate of permethrin in model outdoor ponds Publisher:Marcel Dekker, Inc., Journal:Journal of Environmental Science and Health, Volume:B17, Issue:5, Pages:463 - 486, Year:1982, Report No.: M7696, Edition Number: M-090183-01-1 GLP/GEP: n.a., published	N	N		

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.2 /08	Qin, S.; Budd, R.; Bondarenko, S.; Liu, W.; Gan, J.	2012	Enantioselective Degradation and Chiral Stability of Pyrethroids in Soil and Sediment Journal:J. Agric. Food Chem., Volume:54, Issue:14, Pages:5040-5045, Year:2006, Edition Number: M-459462-01-1 GLP/GEP: n.a., published **...also filed: KCA 7.1.2.1.1 /03** **...also filed: KCA 7.1.2.1.3 /02** **...also filed: KCA 7.5 /02**	N	N		
KCA 7.2.2.2 /01	Anderson, C. A.	1986	Degradation of ^{14}C-Cyfluthrin in natural water Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF2542, Edition Number: M-073248-01-2 EPA MRID No.: 45022101 Date: 1986-02-26 GLP/GEP: no, unpublished ...also filed: KCA 7.2.1 /10 ...also filed: KCA 7.2.2 /05	N	N		Bayer CropScience
KCA 7.2.2.2 /02	Anon.	2013	Beta-cyfluthrin - Waiving of the aerobic mineralisation study in surface water Irvita Plant Protection, Curacao, BCS-Irvita, Report No.: M-482798-01-1, Edition Number: M-482798-01-1 Date: 2013-05-08 GLP/GEP: n.a., unpublished	N	Y	New data requirment	BCS-Irvita

Grey shaded writing indicates a Baseline Dossier Study; owner Bayer CropScience, license Irvita Plant Protection B.V.
Black writing indicates a Supplementary Dossier Study

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.2.3 /01	Sandie, F. E.	1983	Hydrolysis of Baythroid TM in sterile, aqueous buffered solutions Mobay Chemical Corporation, Kansas City, MO, USA Bayer CropScience, Report No.: MR86051, Edition Number: M-073571-01-1 EPA MRID No.: 00131493, 00137539 Date: 1983-10-07 GLP/GEP: no, unpublished **...also filed: KCA 2.8 /03** **...also filed: KCA 7.2.1 /09** **...also filed: KCA 7.2.1.1 /01** **...also filed: KCA 7.2.2 /06**	N	N		Bayer CropScience
KCA 7.2.2.3 /02	Anderson, C.	1987	Degradation characteristics of cyfluthrin (Baythroid) in water/sediment systems Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF2875, Edition Number: M-071937-01-2 Date: 1987-10-01 GLP/GEP: no, unpublished ...also filed: KCA 7.2.1 /11 ...also filed: KCA 7.2.2 /01	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.2.2.3 /03	Sneikus, J.	2000	Aerobic aquatic degradation and metabolism of cyfluthrin in the water-sediment system Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: MR-268/00, Edition Number: M-022319-02-1 Date: 2000-09-15 ...Amended: 2000-10-24 GLP/GEP: yes, unpublished ...also filed: KCA 7.2.2 /02	N	N		Bayer CropScience
KCA 7.2.2.3 /04	Hammel, K.; Porschewski, R.	2013	Kinetic evaluation of the aerobic aquatic metabolism of cyfluthrin and beta-cyfluthrin and their metabolites in water / sediment systems according to FOCUS Kinetics BCS-Irvita, Report No.: EnSa-13-0711, Edition Number: M-471180-01-1 Date: 2013-11-26 GLP/GEP: no, unpublished	N	Y	New kinetic evaluation	BCS-Irvita
KCA 7.2.2.3 /05	Gajbhiye, V.; Gupta, S.	2012	Dissipation of -cyfluthrin in water as affected by sediment, pH, and temperature. Journal:Bull. Environ. Contam. Toxicol., Volume:74, Issue:1, Pages:40-47, Year:2005, Report No.: M-459452-01-1, Edition Number: M-459452-01-1 GLP/GEP: no, published	N	N		

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.3 /01	Atkinson, R.	1987	Estimation of gas-phase hydroxyl radical rate constants for organic chemicals Journal:Environmental Toxicology, Volume:7, Pages:435-442, Year:1988, Report No.: A47123, Edition Number: M-135564-01-1 Date: 1987-11-05 GLP/GEP: no, published	N	N		
KCA 7.3 /02	Atkinson, R.	1989	Kinetics and mechanisms of the gas-phase reactions of the hydroxyl radial with organic compounds Journal:Journal of Physical and Chemical Reference Data; Monograph No.1, Volume:1, Pages:1;246, Year:1989, Report No.: C045278, Edition Number: M-236940-01-1 Date: 1989-04-13 GLP/GEP: no, published	N	N		
KCA 7.3.1 /01	Hellpointner, E.	1992	Calculation of the chemical lifetime of betacyfluthrin in the troposphere Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF3766, Edition Number: M-033634-01-1 Date: 1992-09-25 GLP/GEP: no, unpublished	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.3.2 /01	Sewekow, B.	1981	Vapour pressure of cyfluthrin pure active ingredient Mobay Chemical Corporation, Kansas City, MO, USA Report No.: 94670, Edition Number: M-077323-01-1 Date: 1981-02-04 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.3.2 /02	Krohn, J.	1987	Watersolubility of Cyfluthrin (FCR 1272, Baythroid) at 20 °C and pH 3 and pH 7 Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PC 109, Edition Number: M-043101-01-2 Date: 1987-01-21 GLP/GEP: no, unpublished	N	N		Bayer CropScience
KCA 7.3.2 /03	Fritz, R.	1993	Determination of the volatilisation of betacyfluthrin (Bulldock) in a Field Experiment Bayer AG, Leverkusen, Germany Bayer CropScience, Report No.: PF-3824, Edition Number: M-033579-01-2 Date: 1993-01-12 GLP/GEP: yes, unpublished	N	N		Bayer CropScience

Annex point / reference number	Author(s)	Year	Title Source *(where different from company)* Company name, Report No., Date, GLP status *(where relevant)*, published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner
KCA 7.5 /01	Suess, A.; Bischoff, G.; Mueller, A.; Buhr, L.	2012	Chemical and biological monitoring of the load of plant protection products and of zoocoenoses in ditches of the orchard region /Altes Land/. Journal:Nachrichtenbl. Dtsch. Pflanzenschutzdienstes (Braunschweig, Ger.), Volume:58, Issue:2, Pages:28-42, Year:2006, Edition Number: M-459458-01-2 GLP/GEP: no, published	N	N		
KCA 7.5 /02	Qin, S.; Budd, R.; Bondarenko, S.; Liu, W.; Gan, J.	2012	Enantioselective Degradation and Chiral Stability of Pyrethroids in Soil and Sediment Journal:J. Agric. Food Chem., Volume:54, Issue:14, Pages:5040-5045, Year:2006, Edition Number: M-459462-01-1 GLP/GEP: n.a., published **...also filed: KCA 7.1.2.1.1 /03** **...also filed: KCA 7.1.2.1.3 /02** **...also filed: KCA 7.2.2 /08**	N	N		

Annex point / reference number	**Author(s)**	**Year**	**Title** **Source** ***(where different from company)*** **Company name, Report No., Date, GLP status** ***(where relevant)*****, published or not**	**Vertebrate study Y/N**	**Data protection claimed Y/N**	**Justification if data protection is claimed**	**Owner**
KCA 7.5 /03	Hladik, M.; Kuivila, K.	2009	Assessing the occurrence and distribution of pyrethroids in water and suspended sediments. Journal:J. Agric. Food Chem., Volume:57, Issue:19, Pages:9079-9085, Year:2009, Edition Number: M-459493-01-1 Date: 2009-12-31 GLP/GEP: no, published	N	N		