Document IIIA/ Section 7.1.1.1.1/01 — Hydrolysis as a function of pH and identification of breakdown products

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Official use only

1 REFERENCE

1.1	**Reference**	Krohn, J. (1997) Hydrolysis of cyfluthrin and beta-cyfluthrin as a function of pH. Bayer AG, Institute for Formulation development and Analysis. D-51368 Leverkusen, Germany. . Bayer Report No.: 14 500 0926 BES Ref.: M-043171-01-1 Report date: 2 October 1997. Unpublished
1.2	**Data protection**	Yes
1.2.1	Data owner	Bayer CropScience AG
1.2.2		
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes, OECD Guidelines No. 111
2.2	**GLP**	Yes
2.3	**Deviations**	No.

3 MATERIALS AND METHODS

3.1	**Test material**	Cyfluthrin and beta-cyfluthrin
3.1.1	Lot/Batch number	MC00123 (cyfluthrin); 920309ELB04 (beta-cyfluthrin)
3.1.2	Specification	Cyfluthrin : As given in Section 2
3.1.3	Purity	99% w/w (cyfluthrin, sum of isomers)
3.1.4	Further relevant properties	-
3.2	**Reference substance**	No
3.2.1	Initial concentration of reference substance	Not applicable

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3.3	**Test solution**	The hydrolysis of cyfluthrin and beta-cyfluthrin was performed according to the OECD Guidelines No. 111 in 0.005 M aqueous buffer solutions adjusted to pH 4 (citrate, 50°C), 7(phosphate, 50/60/70°C) and 9 (borate, 40/50°C). In all tests the initial concentrations of the test substance given as the sum of all diastereoisomers was approx. 4 µg/L which is approx. 50 % of the saturation concentration. See Tables A7.1.1.1.1/01-1 and A7.1.1.1.1/01-2	X
3.4	**Testing procedure**		
3.4.1	Test system	The study was performed according to OECD Guideline No. 111. Although the test substances were demonstrated to be stable in calibration solutions and assumed to be stable at pH 4, the concentrations in sterile solutions, were monitored over a time interval of 20 days instead of 8 days in order to compensate for the variable recovery of the analytical method.	
3.4.2	Temperature	pH 4: 50˚C pH 7: 50˚C, 60˚C, 70˚C pH 9: 40˚C, 50˚C	
3.4.3	pH	As above	
3.4.4	Duration of the test	pH 4: 50˚C 624 hr (26 d) pH 7: 50˚C 192 hr (8 d) pH 7: 60˚C 48 hr (2 d) pH 7: 70˚C 8 hr pH 9: 40˚C 8 hr pH 9: 50˚C 1.5 hr	
3.4.5	Number of replicates	Two replicates for cyfluthrin and beta-cyfluthrin for pH 4: 50˚C, pH 7: 50˚C and pH 7: 60˚C. One replicate for each active substance tested at pH 7: 70˚C, pH 9: 40˚C, pH 9: 50˚C Refer to Table A7.1.1.1.1/01-2	X
3.4.6	Sampling	No information on storage of samples prior to analysis is given. However, at sampling, following the final pH measurement, 1 ml of hydrochloric acid (*ca* 0.1 mol/L) was added to pH 9 and 7 samples in order to stabilize the solutions and to prevent further hydrolysis. Refer to Table A7.1.1.1.1/01-2	
3.4.7	Analytical methods	The diastereomers of Cyfluthrin were enriched and extracted by automated solid phase extraction (SPE) using on-line purging with nitrogen. To prevent adsorption on container walls, acetonitrile (25% by volume) was first added to the incubation vessels. The analytes were determined by adsorption mode HPLC using a silica gel column (Zorbax SIL 5µm) and a mobile phase of heptane: chlorobutane :THF (94:5:1, with UV detection (220 nm). The method was validated by performing recovery measurements at two	

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		concentration levels in solutions buffered at pH 7 corresponding approx. to the initial concentrations in the measurements of the rate of hydrolysis and to 25 % of those. The recoveries at the upper and lower concentration levels were 110 - 150 % and 130 - 180 %, respectively, due to interference of the chromatographic separation with impurities.	
3.5	**Preliminary test**	No No preliminary test with test solutions buffered at pH 4, 7 and 9 stored at 50 °C for 8 days provided by the OECD Guidelines No. 111 was run, as the results from a former study (please refer to Section 7.1.1.1.1/02) gave indications of hydrolysis rates.	X
		4 RESULTS	
4.1	**Concentration and hydrolysis values**	Under all conditions tested, partial conversion of the Beta-cyfluthrin diastereomer II into diastereomer I and of diastereomer IV into III occurs before degradation by hydrolysis becomes significant [cyfluthrin is a mixture containing all of the 4 racemic diastereomers and beta-cyfluthrin consists mainly of 2 racemic diastereomers II and IV]. Therefore, it was only possible to specify half-lives for the degradation of diastereomers I + II and of diastereomers III + IV. The values for 20 and 25 °C were calculated by extrapolation from values measured at higher temperatures for cyfluthrin and beta-cyfluthrin. Results for all observation times are presented in Tables A7.1.1.1.1/01-3 to A7.1.1.1.1/01-8.	
4.2	**Hydrolysis rate constant (k_h)**	The hydrolysis rate constant (k_h) as a function of pH and temperature and the correlation coefficient for each set of experiments are shown in Table A7.1.1.1.1/01-9. The rate constant k and half life $t^1/_2$ were calculated by linear regression using the computer program StatgraphicsPlus 3.0 (Manugistic, USA). The linear relationship between the logarithm of the rate constant k and the reciprocal value of the absolute temperature T was used to calculate half-lives for ambient temperatures by linear regression: $\log k = a - b/T$; $t^1/_2 = 0.693/k$ In the case of pH 4 where only measurements at 50 °C had been performed, a doubling of the half-life for each decrease of the temperature by 10 °C was assumed.	X
4.3	**Dissipation time**	The dissipation times of diastereomers of cyfluthrin, at pHs 4, 7 and 9 and test temperatures 50 – 70°C and at 20°C (extrapolated) are shown in Table A7.1.1.1.1/01-10.	X
4.4	**Concentration – time data**	Concentration-time data are shown in Tables A7.1.1.1.1/01-3 to A7.1.1.1.1/01-8. Degradation curves (log concentration-time plots) of the sum of the diastereomers I + II and of the sum of the diastereomers III + IV from cyfluthrin (FCR1272) and beta-cyfluthrin (FCR4545) are shown in Figure A7.1.1.1.1/01-1.	

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4.5	**Specification of the transformation products**	Identification of transformation products was not carried out in this study. Please refer to Section 7.1.1.1.1/02	
		5 APPLICANT'S SUMMARY AND CONCLUSION	
5.1	**Materials and methods**	The hydrolysis of Cyfluthrin and Betacyfluthrin was performed, according to the OECD Guidelines No.111, in oxygen-free 0.005 mol aqueous buffer solutions adjusted to pH 4, 7 and 9 and at temperatures ranging from 40 to 70 °C under sterile conditions in the dark. Under all conditions tested partial conversion of the cyfluthrin diastereomer II into diastereomer 1 and of diastereomer IV into III occurs before degradation by hydrolysis becomes significant. Therefore, in aqueous solution, betacyfluthrin and cyfluthrin form mixtures of identical composition. Therefore, it was only possible to specify half-lives for the degradation of diastereomers I + II and of diastereomers III + IV. The rate constant k and half life $t^1/_2$ were calculated by linear regression and the values for 20 and 25 °C were calculated by extrapolation from values measured at higher temperatures.	
5.2	**Results and discussion**	Hydrolysis of cyfluthrin is temperature and pH-dependant. DT50s were calculated for diastereomers I+II and III + IV. Cyfluthrin was stable at pH 4 (DT50s >1 year) and relatively stable at pH 7 (DT50s 130 – 280d and 61-120 at 20 and 25°C, respectively). Hydrolysis rates were increased at pH 9 (DT50s 24 – 42 hours and 13 – 23 hours at 20 and 25°C, respectively). Mean hydrolysis half-lives at 20°C and pHs 4, 7 and 9 were >1year, 220 d and 37 hours, respectively.	
5.2.1	k_H	Diastereomers I+II: 5.13E-05 (pH4, 50°C) to 0.7413 (pH 9, 50°C) Diastereomers III+IV: 0.00018 (pH4, 50°C) to 0.9788 (pH 9, 50°C)	X
5.2.2	DT_{50}	Mean DT50s at 20°C were as follows: Diastereomers I-IV: > 1 yr (pH 4); 220 d (pH 7); 37 h (pH 9)	
5.2.3	Correlation constant	-0.9333 to -0.9946 (pHs 7 and 9) -0.3104 to -0.7241 (pH 4)	
5.3	**Conclusion**	Mean hydrolysis half-lives at 20°C and pHs 4, 7 and 9 were >1year, 220 d and 37 hours, respectively. Identification of transformation products was not carried out in this study, however were determined in a separate study conducted previously (refer to Section 7.1.1.1.1/02). Validity criteria are considered as fulfilled.	
5.3.1	Reliability	2	
5.3.2	Deficiencies	Study contains no information on mass balance and degradation products. Recoveries are between 110 and 180 % due to interference of impurities with the chromatographic separation. However, in the 91/414 Monograph, the results were accepted as supplemental information on hydrolysis of the diastereomers which confirm the results reported by Sandie (1983) (see document III-A, section A7.1.1.1.1/02).	

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Evaluation by Competent Authorities

EVALUATION BY RAPPORTEUR MEMBER STATE

Date	2006/09/20
Materials and Methods	Despite minor deficiencies applicant's version is acceptable. Comments: A defined allocation of both Cyfluthrin and beta-Cyfluthrin to applied sample code FCR 1272 and FCR 4545 is missing in the original study. Due to the certified contents of diastereomers I to IV in samples and considering the saturation concentration in water of reliable diastereomer, the initial concentration is approx. 64 % of the saturation concentration, e.g. in case of diastereomer III. The value should increase by immediate conversation processes of different diastereomers described in the study. The preliminary tests (OECD Guidelines No. 111) with test solutions at 50°C refer to a former hydrolysis study (7.1.1.1.1/02), that were conducted at pH 5 instead pH 4.
Results and discussion	Despite minor deficiencies applicant's version is acceptable. Comments: The correct column captions of Table A7.1.1.1.1/01-9 should be hydrolysis parameter and regression coefficients instead of the current caption sampling times (hours) and concentration (µg/L) respectively. In the case of hydrolysis study at pH=7, T=50, 60 and 70°C the differences between values for half-life [h] in table A7.1.1.1.1/01-9 and dissipation time DT_{50} in table A7.1.1.1.1/01-10 (DT_{50} is up to 9.6% higher than half-life time) are not traceable and should be explained. When the hydrolysis follows first-order degradation kinetics, the half-life will be equivalent to the 50% dissipation time. Additional to the extrapolated values of DT_{50} at 20°C the values of DT_{50} at 25°C (equally extrapolated) are given in A7.1.1.1.1/01-10. Assuming correctness of the given coefficients for the linear relationship between rate constant and absolute temperature in the original study A7.1.1.1.1/01 and applying the Arrhenius equation at pH 7, the calculated half-life for extrapolated temperatures 20, 25 and 30°C are not accurate.
Conclusion	Applicant's version is acceptable. The hydrolysis rate constant k_h under item 5.2.1 is listed in the unit [h^{-1}].
Reliability	2
Acceptability	Original study and study summary are acceptable.
Remarks	-

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

Table A7.1.1.1.1/01-1: Type and composition of buffer solutions (specify kind of water if necessary)

pH	Type of buffer (final molarity)	Composition
4	pH 4 citrate buffer solution (0.005 mol/L):	2.30 g $KC_8H_70_7$ dissolved in 2L and adjusted to pH 4.0 with sodium hydroxide solution
7	pH 7 phosphate buffer solution (0.005 mol/L):	1.36 g KH_2P0_4 dissolved in 2L and adjusted to pH 7.0 with sodium hydroxide solution
9	pH 9 borate buffer solution (0.005 mol/L):	3.71 g $Na_2B_40_7$ x10 H_20 dissolved in 2L and adjusted to pH 9.0 with phosphoric acid

Table A7.1.1.1.1/01-2: Test conditions

Criteria	Details
Purity of water	Not stated
Preparation of test medium	Care was taken to ensure that test solutions were sterile, free of oxygen and kept in the dark. All glassware used during the test were sterilized in an incubator at 110 °C. Solutions were prepared in buffers at pH 4, 7 and 9. The dissolved oxygen was displaced by passing nitrogen through the solutions. In order to sterilize them they were then passed through a sterile filter (22 Nm Millex-GS, Fa. Millipore, USA). For each buffer and temperature different sets of portions of the solutions, one portion for each sampling date, consisting of exactly 80 g, were collected under nitrogen in 100 mL brown glass flasks, added with 790 NI of stock solutions of FCR 1272 and FCR 4545, respectively, sealed and placed into a thermostated water bath or into an thermostated incubator.

Continued

<table>
<tr><td>Test concentrations (µg a.i./L)</td><td>Initial concentration of test substance: 4µg/l. Initial concentrations in diastereomer replicates (test substance cyfluthrin):
pH 4: 50˚C
<table>
<tr><td>I</td><td>II</td><td>III</td><td>IV</td></tr>
<tr><td>1.0058811
0.956732</td><td>0.565534
0.568581</td><td>1.257749
1.173931</td><td>0.801389
0.641119</td></tr>
</table>
pH 7: 50˚C
<table>
<tr><td>I</td><td>II</td><td>III</td><td>IV</td></tr>
<tr><td>0.691301
0.668883</td><td>0.662659
0.593504</td><td>0.733771
0.739945</td><td>0.723411
0.751893</td></tr>
</table>
pH 7: 60˚C
<table>
<tr><td>I</td><td>II</td><td>III</td><td>IV</td></tr>
<tr><td>0.808057
0.855844</td><td>0.712762
0.748700</td><td>0.985227
1.000927</td><td>0.984152
0.954659</td></tr>
</table>
pH 7: 70˚C
<table>
<tr><td>I</td><td>II</td><td>III</td><td>IV</td></tr>
<tr><td>0.752932</td><td>0.745361</td><td>1.047941</td><td>0.991298</td></tr>
</table>
pH 9: 40˚C
<table>
<tr><td>I</td><td>II</td><td>III</td><td>IV</td></tr>
<tr><td>0.772671</td><td>0.632257</td><td>0.947243</td><td>0.845452</td></tr>
</table>
pH 9: 50˚C
<table>
<tr><td>I</td><td>II</td><td>III</td><td>IV</td></tr>
<tr><td>0.67448</td><td>0.63955</td><td>0.84886</td><td>0.81188</td></tr>
</table></td></tr>
<tr><td>Temperature (°C)</td><td>pH 4: 50˚C
pH 7: 50˚C, 60˚C, 70˚C
pH 9: 40˚C, 50˚C</td></tr>
<tr><td>Controls</td><td>-</td></tr>
<tr><td>Identity and concentration of co-solvent</td><td>-</td></tr>
</table>

Continued

Replicates	1 – 2 replicates at each pH/temperature – see section 3.4.5.

	cyfluthrin	beta-cyfluthrin
pH 4: 50˚C	2	2
pH 7: 50˚C	2	2
pH 7: 60˚C.	2	2
pH 7: 70˚C	1	1
pH 9: 40˚C	1	1
pH 9: 50˚C	1	1

Sampling

	pH 4: 50˚C	pH 7: 50˚C	pH 7: 60˚C	pH 7: 70˚C	pH 9: 40˚C	pH 9: 50˚C
Cyfluthrin and beta-cyfluthrin (hours)	0 96 168 194 264 363 624	0 24 33 48 96 120 128 144 168 192	0 1 16 18 20 22 24 40 42 44 46 48	0 0.5 1 2 3 4 5 6 7 8	0 0.5 1 2 3 4 5 6 7 8	0 0.167 0.333 0.5 0.667 0.833 1 1.167 1.333 1.5

Table A7.1.1.1.1/01-3: Hydrolysis of cyfluthrin and beta-cyfluthrin at pH 4 and 50 °C

Sampling times (hours)	Concentration (µg/L) Cyfluthrin		Concentration (µg/L) Beta-cyfluthrin	
	I+II	III+IV	I+II	III+IV
0	1.54836	1.93710	1.32750	2.35561
96	1.48372	1.95686	1.36600	2.38226
168	1.66269	1.84865	1.37958	2.05037
194	1.32307	1.57092	1.41111	2.01890
264	1.27490	1.52700	1.27468	1.85660
363	1.26335	1.48421	1.35056	2.04728
624	1.41958	1.55739	1.31441	2.12028

Table A7.1.1.1.1/01-4: Hydrolysis of cyfluthrin and beta-cyfluthrin at pH 7 and 50 °C

Sampling times (hours)	Concentration (µg/L) Cyfluthrin		Concentration (µg/L) Beta-cyfluthrin	
	I+II	III+IV	I+II	III+IV
0	1.30818	1.47451	1.21369	1.77206
24	0.82385	0.82152	0.97799	1.20885
33	1.06154	1.08868	0.76436	1.01323
48	0.80115	0.74773	0.84868	1.09394
56	0.79523	0.68907	0.65519	0.65016
120	0.60004	0.36363	0.44137	0.36329
128	0.37639	0.28736	0.29711	0.28034
144	0.28348	0.19093	0.25820	0.16247
168	0.29862	0.16440	0.28226	0.15928
192	0.20217	0.11898	0.17768	0.10572

Table A7.1.1.1.1/01- 5: Hydrolysis of cyfluthrin and beta-cyfluthrin at pH 7 and 60 °C

Sampling times (hours)	Concentration (µg/L) Cyfluthrin		Concentration (µg/L) Beta-cyfluthrin	
	I+II	III+IV	I+II	III+IV
0	1.56268	1.96248	1.29814	1.94394
1	1.50281	1.86856	1.34861	2.01064
16	0.96227	0.71101	0.83527	0.94869
18	0.96375	0.89593	0.77823	0.95070
20	0.90088	0.71890	0.75238	0.81798
22	0.97043	0.79582	0.86767	0.84046
24	0.78019	0.65673	0.70971	0.73631
40	0.46611	0.35534	0.36744	0.38219
42	0.42499	0.27614	0.39158	0.32868
44	0.38610	0.25333	0.38015	0.26098
46	0.49256	0.30474	0.30936	0.24100
48	0.31871	0.20109	0.33132	0.23453

Table A7.1.1.1.1/01- 6: Hydrolysis of cyfluthrin and beta-cyfluthrin at pH 7 and 70 °C

Sampling times (hours)	Concentration (µg/L) Cyfluthrin		Concentration (µg/L) Beta-cyfluthrin	
	I+II	III+IV	I+II	III+IV
0	1.49830	2.03924	1.40680	2.28757
0.5	1.54051	1.99862	1.37499	2.28873
1	1.54489	1.92882	1.11798	1.77955
2	1.37664	1.86893	1.18807	1.78109
3	1.21857	1.38251	1.04074	1.45150
4	1.13150	1.29721	0.83994	1.19949
5	1.02946	1.16945	0.97709	1.23157
6	0.94826	0.82212	0.76542	0.90497
7			0.51148	0.70109
8	0.60871	0.61858	0.56146	0.75433

Table A7.1.1.1.1/01– 7: Hydrolysis of cyfluthrin and beta-cyfluthrin at pH 9 and 40 °C

Sampling times (hours)	Concentration (µg/L) Cyfluthrin		Concentration (µg/L) Beta-cyfluthrin	
	I+II	III+IV	I+II	III+IV
0	1.40493	1.79370	1.22073	1.95757
0.5	1.39756	1.68366	1.05762	1.64356
1	1.26577	1.52891	1.00247	1.59672
2	0.97265	1.08995	0.75389	1.17966
3	0.77727	0.79154	0.71952	0.94087
4	0.60745	0.48946	0.51441	0.52156
5	0.53162	0.46437	0.40918	0.46674
6	0.45537	0.31126	0.44619	0.39962
7	0.31323	0.28532	0.20527	0.30552
8	0.30526	0.19879		

Table A7.1.1.1.1/01– 8: Hydrolysis of cyfluthrin and beta-cyfluthrin at pH 9 and 50 °C

Sampling times (hours)	Concentration (µg/L) Cyfluthrin		Concentration (µg/L) Beta-cyfluthrin	
	I	II	III	IV
0	1.31403	1.66074	1.24667	1.73015
0.167	1.14640	1.32470	1.07791	1.69567
0.333			1.02492	1.37058
0.5	1.18288	1.14572	1.05777	1.18560
0.667	0.88277	0.87745	1.02052	1.02684
0.833		0.83794	0.88209	0.85942
1	0.86525	0.84423	0.72989	0.76905
1.167	0.80171	0.83480	0.55447	0.61461
1.333	0.51959	0.49801	0.45432	0.49654
1.5	0.51255	0.42159	0.41028	0.41601

Table A7.1.1.1.1/01– 9: Hydrolysis rate constants (k_h) as a function of pH and temperature together with correlation coefficients

Sampling times (hours)	Concentration (µg/L) Cyfluthrin		Concentration (µg/L) Beta-cyfluthrin	
	I+II	III+IV	I+II	III+IV
pH 4 50 °C				
slope (-(k / 2.303))	-9.12427E-05	-0.00018633	-2.22672 E-05	-7.81463E-05
intercept (log 10 [ct 0])	0.1741	0.2726	0.1344	0.3437
correlation coefficient	(-0.4123)	(-0.7241)	(-0.3104)	(-0.4142)
rate constant (k) [h^{-1}]	0.00021	0.00043	5.13E-05	0.00018
half-life [h]	*ca* 3 000	*ca* 2 000	*ca* 10 000	*ca* 4 000
pH 7 50 °C				
slope (-(k / 2.303))	-0.00395933	-0.00555678	-0.00418483	-0.00642774
intercept (log 10 [ct 0])	0.1065	0.1497I	0.0754	0.2453
correlation coefficient	-0.9669	-0.9893	-0.9816	-0.9888
rate constant (k) [h^{-1}]	0.009118337	0.012797264	0.009637663	0.014803085
half-life [h]	76.0	54.1	71.9	46.8
pH 7 60 °C				
slope (-(k / 2.303))	-0.0133795	-0.0194559	-0.0133301	-0.0196001
intercept (log 10 [ct 0])	0.2116	0.2887	0.1146	0.3166
correlation coefficient	-0.9768	-0.9877	-0.9836	-0.9917
rate constant (k) [h^{-1}]	0.030822989	0.044806938	0.03069922	0.04513903
half-life [h]	22.5	15.5	22.6	15.4
pH 7 70 °C				
slope (-(k / 2.303))	-0.0469431	-0.0666651	-0.0509485	-0.0651599
intercept (log 10 [ct 0])	0.221	0.3513	0.1543	0.361
correlation coefficient	-0.9676	-0.9807	-0.9454	-0.9806
rate constant (k) [h^{-1}]	0.108109959	0.153529725	0.117334396	0.15006325
half-life [h]	6.41	4.51	5.91	4.62
pH 9 40 °C				
slope (-(k / 2.303))	-0.0894994	-0.123174	-0.0951153	-0.119276
intercept (log 10 [ct 0])	0.1689	0.2699	0.0936	0.2921
correlation coefficient	-0.9946	-0.9936	-0.9654	-0.9898
rate constant (k) [h^{-1}]	0.206117118	0.283669722	0.219050536	0.274692628
half-life [h]	3.36	2.44	3.16	2.52
pH 9 50 °C				
slope (-(k / 2.303))	-0.264277	-0.350667	-0.321889	-0.425016
intercept (log 10 [ct 0])	0.1422	0.2168	0.1409	0.2801
correlation coefficient	-0.9333	-0.9516	-0.9475	-0.9948
rate constant (k) [h^{-1}]	0.608629931	0.807586101	0.741310367	0.978811848
half-life [h]	1.14	0.858	0.935	0.708

Table A7.1.1.1.1/01– 10: Dissipation times of diastereomers of cyfluthrin, at pH 4, pH 7 and pH 9 and test temperatures (50 – 70˚C) and at 20˚C (extrapolated)

Test conditions	DT_{50} cyfluthrin		DT_{50} beta-cyfluthrin		Extrapolated conditions	DT_{50} cyfluthrin		DT_{50} beta-cyfluthrin		Mean DT50
	I+II	III + IV	I+II	III + IV		I+II	III + IV	I+II	III + IV	I-IV
pH 4 50 °C	*ca* 3000h	*ca* 2000h	*ca* 10000h	*ca* 4 000 h	**pH 4 25 °C**	> 1 yr	> 1 yr	> 1 yr	> 1 yr	> 1 yr
					pH 4 20 °C	> 1 yr	> 1 yr	> 1 yr	> 1 yr	> 1 yr
pH 7 50 °C	81.4 h	57.5 h	78.8 h	50.3 h	**pH 7 25 °C**	120 d	89 d	120 d	61 d	97.5 d
pH 7 60 °C	22.5 h	15.7 h	22.5 h	15.1 h	**pH 7 20 °C**	270 d	200 d	280 d	130 d	220 d
pH 7 70 °C	6.68 h	4.65 h	6.32 h	4.84 h						
pH 9 40 °C	3.36 h	2.44 h	3.16 h	2.52 h	**pH 9 25 °C**	20 h	13 h	23 h	20 h	19 h
pH 9 50 °C	1.14 h	0.858 h	0.935 h	0.708 h	**pH 9 20 °C**	37 h	24 h	46 h	42 h	37 h

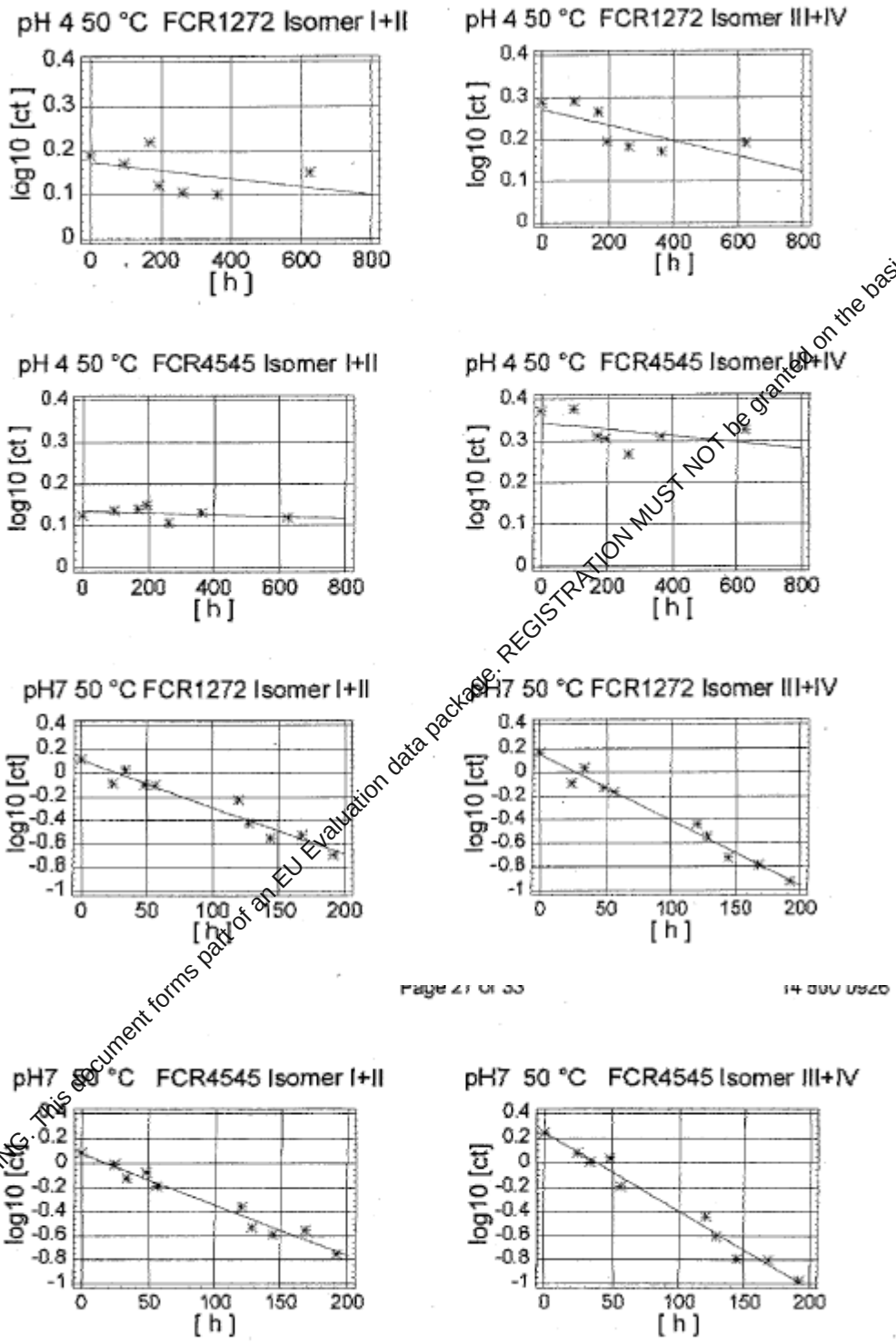
h: hours

d: days

yr year

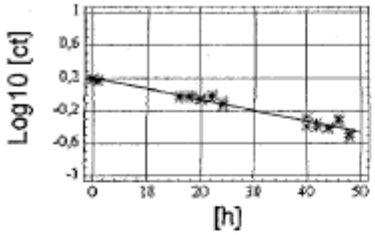
* results from parallel test with beta-cyfluthrin

Figure A7.1.1.1.1/01 – 1: Degradation curves (log concentration-time plots) of the sum of the diastereomers I + II and of the sum of the diastereomers III + IV from cyfluthrin (FCR1272) and beta-cyfluthrin (FCR4545)

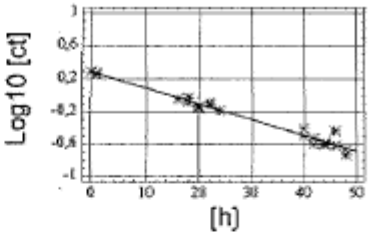


continued

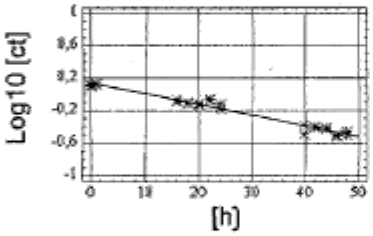
pH7 60 °C FCR 1272 Isomer I+II



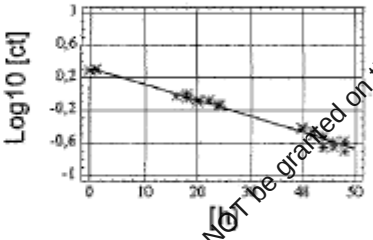
pH7 60 °C FCR 1272 Isomer III+IV



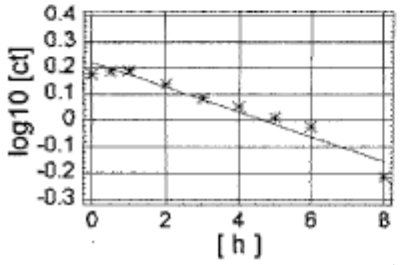
pH7 60 °C FCR 4545 Isomer I+II



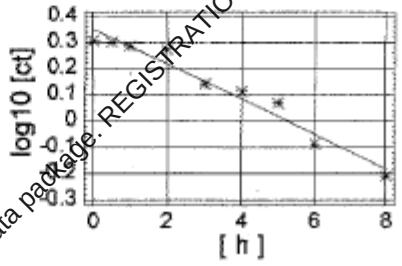
pH7 60 °C FCR 4545 Isomer III+IV



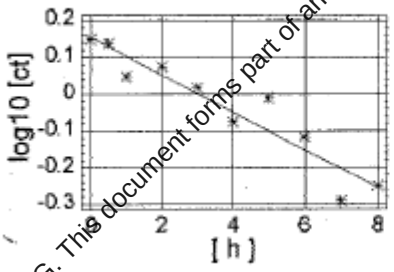
pH7 70 °C FCR1272 Isomer I+II



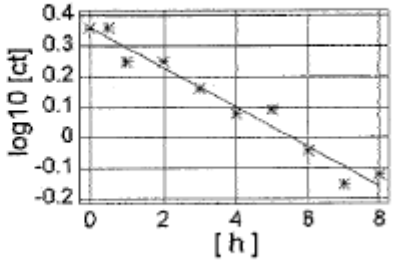
pH7 70 °C FCR1272 Isomer III+IV



pH7 70 °C FCR4545 Isomer I+II

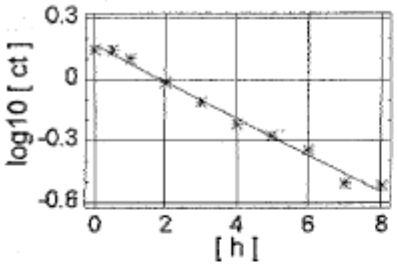


pH7 70 °C FCR4545 Isomer III+IV

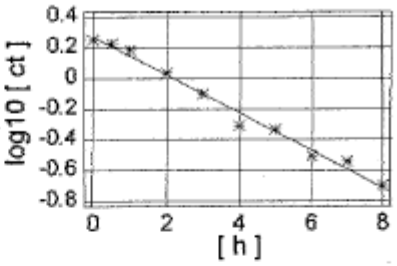


continued

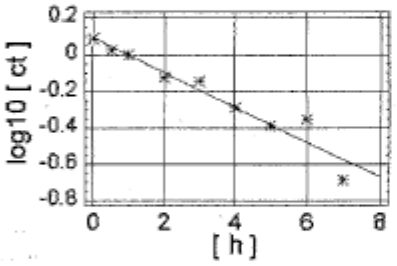
pH9 40 °C FCR1272 Isomer I+II



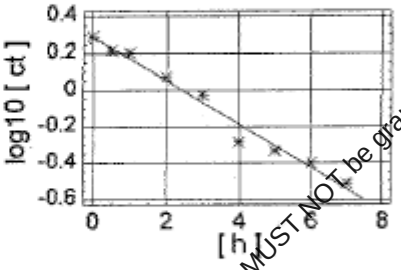
pH9 40 °C FCR1272 Isomer III+IV



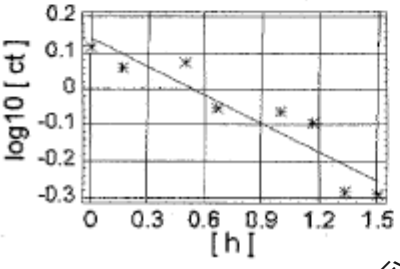
pH9 40 °C FCR4545 Isomer I+II



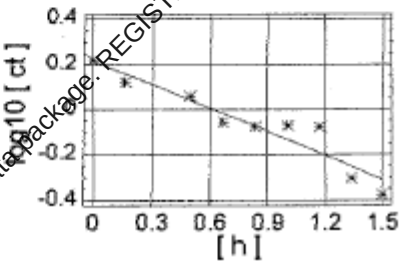
pH9 40 °C FCR4545 Isomer III+IV



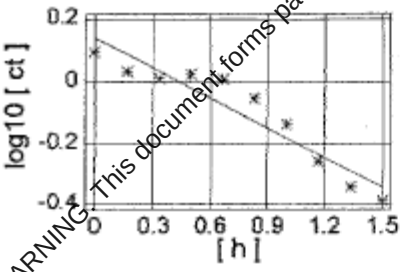
pH9 50 °C FCR1272 Isomer I+II



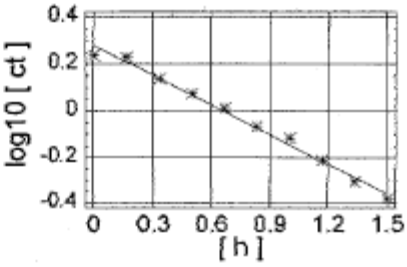
pH9 50 °C FCR1272 Isomer III+IV



pH9 50 °C FCR4545 Isomer I+II



pH9 50 °C FCR4545 Isomer III+IV



Document IIIA/ Section 7.1.1.1.1/02 Hydrolysis as a function of pH and identification of breakdown products

BPD Data set IIA/ Annex Point VII 7.6.2.1

Official use only

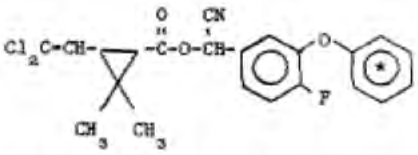
1 REFERENCE

1.1	**Reference**	Sandie, F.E. (1983) Hydrolysis of Baythroid in sterile aqueous buffered solutions. Mobay Chemical Corporation, Agricultural Chemicals Division. Bayer Report No.: 86051. BES Ref.: M-073571-01-1 Report date: 7 October 1983. Unpublished
1.2	**Data protection**	Yes
1.2.1	Data owner	Bayer CropScience AG
1.2.2		
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	No When the study was conducted no specific guidelines were available. However, the study design was comparable to OECD Guideline No. 111, as stated in the in the monograph from the PPP dossier.
2.2	**GLP**	No (not required, as study started before June 30 1988).
2.3	**Deviations**	No.

3 MATERIALS AND METHODS

3.1	**Test material**	[Phenyl-UL-^{14}C] Baythroid (cyfluthrin)
3.1.1	Lot/Batch number	-
3.1.2	Specification	 (*) indicates position of 14C label
3.1.3	Purity	Radiochemical purity 97.9% w/w
3.1.4	Further relevant properties	Specific activity 21.74 mCi/mmole
3.2	**Reference substance**	No
3.2.1	Initial concentration of reference substance	Not applicable

Document IIIA/ Section 7.1.1.1.1/02 Hydrolysis as a function of pH and identification of breakdown products

BPD Data set IIA/ Annex Point VII 7.6.2.1

3.3	**Test solution**	The hydrolysis of cyfluthrin was studied using [phenyl-UL-^{14}C] cyfluthrin at a concentration of 0.02 mg/l in sterile phosphate buffers of pH 5, 7 and 9 containing 1% acetonitrile as an organic solvent (25 °C) because of the very low water solubility. See Tables A7.1.1.1.1/02-1 and A7.1.1.1.1/02-2	
3.4	**Testing procedure**		
3.4.1	Test system	The study design was comparable to OECD Guideline No. 111 Eighteen bottles of each buffer were fortified at 0.02 mg/l by adding 5 ml of the acetonitrile solution of [^{14}C]Baythroid into each sample bottle under a flame blanket to maintain sterility. Each solution was mixed thoroughly and bottles were then filled to the rim with sterile buffer (total volume 550 ml). The samples were maintained at 25 ± 1°C in the dark. See Tables A7.1.1.1.1/02-3	
3.4.2	Temperature	25 + 1°C	X
3.4.3	pH	pH 5, 7 and 9	X
3.4.4	Duration of the test	Up to 35 days.	
3.4.5	Number of replicates	Duplicate samples were analyzed at each time point.	
3.4.6	Sampling	Samples were analyzed at 0, 1, 3, 7, 14, 21, 28 and 35 days No information on storage of samples prior to analysis is given.	
3.4.7	Analytical methods	Samples, including acetone washes of the sample bottles, were extracted twice with dichloromethane. The organic extract was concentrated to *ca* 0.5 ml and the aqueous and organic phases (before and after concentration) were radioassayed by LSC in triplicate. The samples were analysed by thin-layer chromatography (TLC) using three different solvent systems: System I - toluene/ethyl ether/acetic acid (100:5:1); System II- hexane/1,4-dioxane/acetone/acetic acid (80:30:2:1); System III - methanol/acetonitrile/0.5 M NaCl (40:40:20). Compound identity was established by co-chromatography with the following non-radioactive reference standards in addition to parent: 4-fluoro-3-phenoxybenzaldehyde (FPBald); 4-fluoro-3-(4-hydroxyphenoxy)- benzoic acid (4'-OH-FPBacid); 4-fluoro-3-phenoxybenzoic acid (FPBacid); Extracts were analysed using UV detection, autoradiography, LSC and reverse phase HPLC (C18 column) with variable wavelength and radiochemical detection.	
3.5	**Preliminary test**	Cyfluthrin has very low water solubility (1-2 µg/l) which potentially could be reduced by the buffer salts used in the buffer hydrolysis study. Therefore a preliminary solubility study was conducted in 0.1 M pH 7 buffer solution containing 1% acetonitrile as an organic solubilizer.	

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BPD Data set IIA/ Annex Point VII 7.6.2.1

Hydrolysis as a function of pH and identification of breakdown products

4 RESULTS

4.1 Concentration and hydrolysis values

Material balance ranged from 97-102% with the exception of the pH 7, 35-day samples which showed recovery of 95%.

Results for all observation times are presented in Tables A7.1.1.1.1/02-4 to A7.1.1.1.1/02-6.

4.2 Hydrolysis rate constant (k_h)

The hydrolysis rate constant (k_h) as a function of pH and the correlation coefficients are shown in Table A7.1.1.1.1/02-7.

The rate constant k was calculated by linear regression.

4.3 Dissipation time

The dissipation times of cyfluthrin, at pHs 5, 7 and 9 and 25˚C are shown in A7.1.1.1.1/02-7.

Cyfluthrin was hydrolytically stable at pH 5 and half-lives at pHs 7 and 9 were 193 d and <2 d, respectively.

FPB-ald was stable to hydrolysis under the test conditions. Further hydrolytic degradation of FPB-ald is not expected under sterile conditions due to it's molecular structure.

4.4 Concentration – time data

Concentration-time data are shown in Tables A7.1.1.1.1/02-4 to A7.1.1.1.1/02-6. X

4.5 Specification of the transformation products

One major component (FPB-ald) was identified, accounting for up to 89 % of the radioactivity at pH 9 (day 21) and for up to 11 % at pH 7 (day 35). X

Two minor unidentified compounds were detected at ≤ 3 %, which are stated to be impurities from test material that remained unchanged.

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1 Materials and methods

The behaviour of [phenyl-U-^{14}C] cyfluthrin was examined at a concentration of 0.02 mg/l in sterile, aqueous buffers (pH 5, 7 and 9) containing 1% acetonitrile at 25°C for 35 days in the dark. The study design was comparable to OECD Guideline No. 111.
Samples were extracted with dichloromethane and the aqueous and organic phases were radioassayed and further analysed by TLC and reverse phase HPLC with UV and radiochemical detection. Compound identity was established by co-chromatography with reference standards

5.2 Results and discussion

Hydrolysis of cyfluthrin is pH-dependant. Rapid hydrolysis occurred in pH 9 buffer with cyfluthrin having a half-life of <2 days. The extrapolated half-life in pH 7 buffer was 193 days. Cyfluthrin was stable in pH 5 buffer.

The only significant hydrolysis product was 4-fluoro-3-phenoxy benzaldehyde, (FPB-ald), accounting for up to 89 % of the radioactivity at pH 9 (day 21) and for up to 11 % at pH 7 (day 35).

5.2.1 k_H

pH 5	Stable
pH 7	3.6×10^{-3} days

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BPD Data set IIA/ Annex Point VII 7.6.2.1

Hydrolysis as a function of pH and identification of breakdown products

		pH 9	3.7 x 10^{-1} days
5.2.2	DT_{50}	pH 5	Stable
		pH 7	193 days
		pH 9	<2 days
5.2.3	correlation coefficient	0.97 – 0.99	
5.3	**Conclusion**	Cyfluthrin was stable at pH 5. Hydrolysis half-lives at 25°C and pHs 7 and 9 were 193 days and <2 days, respectively. The major transformation product was 4-fluoro-3-phenoxy benzaldehyde, (FPB-ald), which was stable to hydrolysis. Validity criteria are considered as fulfilled.	
5.3.1	Reliability	1	
5.3.2	Deficiencies	None	

Evaluation by Competent Authorities

EVALUATION BY RAPPORTEUR MEMBER STATE

Date
2006/09/22

Materials and Methods
The applicant's version is acceptable.

Comments:

An assignment of ^{14}C BAYTHROID to one of the diastereomers would be preferable.

The hydrolysis test in accordance to OECD Guidelines No. 111 should be performed at least at 3 pH values, in general at pH values of 4, 7 and 9 and at various temperatures.

The temperature should be given by 25 ± 1°C.

Results and discussion
Despite minor deficiencies applicant's version is acceptable.

Comments:

The non-linear log concentration-time plot is shown in Figure A7.1.1.1.1/01 -1.

Neither the transformation pathways of BAYTHROID to FPB-ald as hydrolysis product nor potential decline of FPB-ald have been indicated or discussed.

Conclusion
Applicant's version is acceptable.

The hydrolysis rate constant k_h under item 5.2.1 is listed in the unit [day^{-1}].

Reliability
2

Acceptability
Original study and study summary are acceptable.

Remarks
-

Document IIIA/ Section 7.1.1.1.1/02 — Hydrolysis as a function of pH and identification of breakdown products

BPD Data set IIA/ Annex Point VII 7.6.2.1

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

Table A7.1.1.1.1/02-1: Type and composition of buffer solutions (specify kind of water if necessary)

pH	Type of buffer (final molarity)	Composition
5	pH 5.03 buffer solution (0.1 M):	0.1 M solutions of potassium bi-phosphate (KH_2PO_4) and disodium phosphate ($Na_2HPO_4 \cdot 7H_20$)
7	pH 7.02 buffer solution (0.1 M):	0.1 M solutions of disodium phosphate and potassium biphosphate (as above)
9	pH 8.99 buffer solution (0.1 M):	0.1 M solutions of potassium biphosphate and trisodium phosphate ($Na_3PO_4 \cdot 12H_20$)

Table A7.1.1.1.1/02-2: Description of test solution

Criteria	Details
Purity of water	Not stated
Preparation of test medium	Buffer solutions were sterilized in an autoclave at 121°C and 15 psi for two hours; after cooling, the caps were tightened.
Test concentrations (µg a.i./L)	Initial concentration of test substance: 0.02 mg/l.
Temperature (°C)	25 $\pm$1˚C
Controls	Four control bottles
Identity and concentration of co-solvent	1% acetonitrile
Replicates	3 replicates at each pH

Table A7.1.1.1.1/02-3: Description of test system

Glassware	Wheaton "400" glass sample bottles, lightly capped with Teflon lined caps.
Other equipment	Temperature-controlled growth chamber
Method of sterilization	Autoclave

Table A7.1.1.1.1/02 - 4: Hydrolysis of cyfluthrin at pH 5 and 25 °C

Compound	% applied radioactivity							
	0	**1**	**3**	**7**	**14**	**21**	**28**	**35**
Cyfluthrin	96	94	97	96	95	96	96	96
Impurities[1]	2	3	1	1	2	1	1	1
Diffuse	2	3	2	3	2	3	3	3
Total	100	100	100	100	100	100	100	100

1 Impurities were present in the [^{14}C]Cyfluthrin at the beginning of the study.

Table A7.1.1.1.1/02 - 5: Hydrolysis of cyfluthrin at pH 7 and 25 °C

Compound	% applied radioactivity							
	0	**1**	**3**	**7**	**14**	**21**	**28**	**35**
Cyfluthrin	96	NA[2]	95	93	89	87	87	84
FPBald	<1	NA[2]	1	3	6	8	8	11
Impurities[1]	2	NA[2]	2	1	2	2	2	2
Diffuse	2	NA[2]	2	3	2	2	2	2
Aqueous Unknown	ND[3]	NA[2]	ND[3]	ND[3]	1	1	1	1
Total	100	NA[2]	100	100	100	100	100	100

1 Impurities were present in the [^{14}C]Cyfluthrin at the beginning of the study.

2 NA = not analysed.

3 ND a not detected.

Table A7.1.1.1.1/02- 6: Hydrolysis of cyfluthrin at pH 9 and 25 °C

Compound	% applied radioactivity							
	0	**1**	**3**	**7**	**14**	**21**	**28**	**35**
Cyfluthrin	91	65	28	7	< 1	< 1	-	-
FPBald	5	28	64	86	89	89	-	-
Impurities[1]	2	2	2	<1	3	3	-	-
Organic Unknown-I	<1	<1	1	2	3	4	-	-
Organic Unknown-II	<1	2	3	3	2	2	-	-
Diffuse	2	3	2	2	2	1	-	-
Aqueous Unknown	ND[3]	ND[3]	ND[3]	ND[3]	1	1	-	-
Total	100	100	100	100	100	100	-	-

1 Impurities were present in the [^{14}C]cyfluthrin at the beginning of the study.

2 NA not analysed.

3 ND not detected.

Table A7.1.1.1.1/02 - 7: Hydrolysis rate constants (k_h) and dissipation times as a function of pH and temperature together with correlation coefficients

pH	Half-life [days][1]	Rate constant (k) [d^{-1}] [2]	Correlation coefficient
5	Stable	NA[3]	NA[3]
7	193	3.6 x 10^{-3}	0.97
9	< 1	3.7 x 10^{-1}	0.99

1 Half-life = 0.693/rate constant.

2 Determined by linear regression analysis.

3 Not applicable.

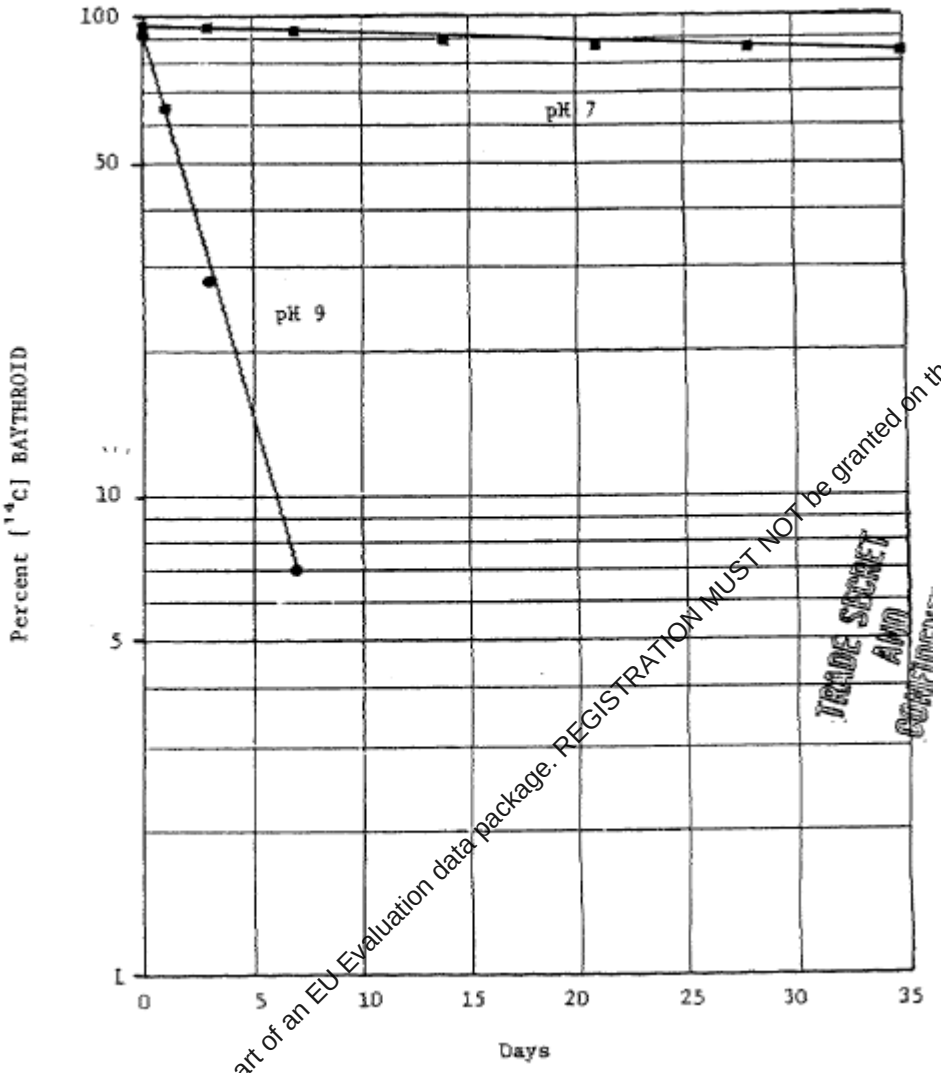


Figure A7.1.1.1.1/01 – 1: Degradation curves (log concentration-time plots) of cyfluthrin at pHs 7 and 9

Document IIIA/ Section 7.1.1.1.1/03 — Hydrolysis as a function of pH and identification of breakdown products

BPD Data set IIA/

Annex Point VII 7.6.2.1

Official use only

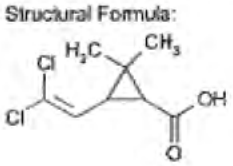
1 REFERENCE

1.1	**Reference**	*From addendum in the monograph p117* Krohn, J. (1997) Hydrolysis of permethric acid as a function of pH. Bayer AG, Institute for Formulation development and Analysis. D-51368 Leverkusen, Germany. Bayer Report No.: 145000921 BES Ref.: M-043185-01-1 Report date: 16 June 1997. Unpublished
1.2	**Data protection**	Yes
1.2.1	Data owner	Bayer CropScience AG
1.2.2		
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes, OECD Guidelines No. 111
2.2	**GLP**	Yes
2.3	**Deviations**	No.

3 MATERIALS AND METHODS

3.1	**Test material**	
3.1.1	Lot/Batch number	920622ELB03 and 920622ELB04 – used as test and reference substances. The chemical identity of both materials was previously established by 1 H-NMR- and mass spectral analysis and they were further characterized by HPLC. Their purity had been certified in both cases to be 99.8% w/w.
3.1.2	Specification	
3.1.3	Purity	
3.1.4	Further relevant properties	3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane-carboxylic acid. Molecular Formula: $C_8 H_{10} Cl_2 0_2$ Molar Mass: 209.1 g/mol
3.2	**Reference substance**	Structural Formula: 
3.2.1	Initial concentration of reference substance	
3.3	**Test solution**	
3.4	**Testing procedure**	
3.4.1	Test system	The study was performed according the OECD Guideline No. 111, as stated in the addendum in the monograph from the PPP dossier. No deviations from this guideline were noted.
3.4.2	Temperature	

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Hydrolysis as a function of pH and identification of breakdown products

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Annex Point VII 7.6.2.1

3.4.3	pH	The hydrolysis of *cis*- and *trans*-permethric acid was performed at 50 °C in 0.01 M aqueous buffer solution adjusted to pH 4 (citrate), 7 (phosphate) and 9 (borate) under sterile conditions in the dark. As the substance was found to be stable over 1 week under these conditions the study was finished after the preliminary test. The initial concentration of the test substance was: approx. 100 mg/1 (half-saturated aqueous solution). Two replicates were sampled at each pH.
3.4.4	Duration of the test	
3.4.5	Number of replicates	
3.4.6	Sampling	
3.4.7	Analytical methods	Analysis was by HPLC (column LiChrospher 60 RP select B) with UV detection (212 nm). The method was acceptably validated.
3.5	**Preliminary test**	
		4 RESULTS
4.1	**Concentration and hydrolysis values**	The decrease of the concentration was in all cases found to be < 2 % after 1 week at 50 °C and pH 4, 7 and 9. This is not significant with regard to the analytical error and does not indicate a degradation of the test substance due to hydrolysis.
4.2	**Hydrolysis rate constant (k_h)**	
4.3	**Dissipation time**	See Table A7.1.1.1.1/03-1
4.4	**Concentration – time data**	The corresponding half-lives of cis- and trans-permethric acid are expected to be > 1 year at 25°C.
4.5	**Specification of the transformation products**	
		5 APPLICANT'S SUMMARY AND CONCLUSION
5.1	**Materials and methods**	Test substance: cis- and trans-permethric acid (2,2-dimethyl-cyclopropanecarboxylic acid (DCVA)), HPLC certified purity: 99.8 % for each isomer The hydrolysis of cis- and trans-permethric acid was performed according the OECD Guideline No. 111, at 50 °C in 0.01 mol aqueous buffer solution adjusted to pH 4 (citrate), 7 (phosphate) and 9 (borate) under sterile conditions in the dark. As the substance was found to be stable over 1 week under these conditions the test was finished after the preliminary test.
5.2	**Results and discussion**	The decrease of the concentration was in all cases found to be < 2 % after 1 week at 50 °C and pH 4, 7 and 9. This is not significant with regard to the analytical error and does not indicate a degradation of the test substance due to hydrolysis. The corresponding half-lives of *cis*- and *trans*-permethric acid are expected to be > 1 year at 25°C.
5.2.1	k_H	
5.2.2	DT_{50}	
5.2.3	r^2	
5.3	**Conclusion**	*Cis*- and *trans*-permethric acid is stable to hydrolysis (DT50 estimated > 1 year at 25°C). Validity criteria are considered as fulfilled. No deviations from this guideline were noted

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Annex Point VII 7.6.2.1

5.3.1	Reliability	1
5.3.2	Deficiencies	No

Evaluation by Competent Authorities	
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	2006/09/25
Materials and Methods	Applicant's version is acceptable.
Results and discussion	Applicant's version is acceptable.
Conclusion	Applicant's version is acceptable.
Reliability	1
Acceptability	Original study and study summary are acceptable.
Remarks	-
	COMMENTS FROM ...
Date	*Give date of comments submitted*
Materials and Methods	*Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant's summary and conclusion.* *Discuss if deviating from view of rapporteur member state*
Results and discussion	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	

Table A7.1.1.1.1/03-1: Comparison of concentration of cis and trans permethric acid at t_0 and 1 week

pH	cis-Permethric acid (mg/l)			trans-Permethric acid (mg/l)		
	initial	1 week 50°C	difference	initial	1 week 50°C	difference
pH 4	88.28	86.94	1.35	91.85	90.09	1.77
pH 7	95.93	95.48	0.45	101.07	100.63	0.44
pH 9	100.54	100.32	0.22	113.02	112.83	0.19

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			Official use only
		1 REFERENCE	
1.1	**Reference**	Gronberg, R.R. (1987). Photodecomposition of [phenyl-UL-14C]Baythroid in aqueous solution by sunlight. Mobay Chemical Corporation, Agricultural Chemicals Division. Bayer Report No.: 88598. BES Ref.: M-040090-01-1 Report date: Original report 18 October 1984; revised report April 30 1987. Unpublished With supplementary information from: Puhl, R.J., Hurley, J. B. and Dime R. A. (1983). Photodecomposition of BAYTHROID-14C in Aqueous Solution and on Soil. Mobay Chemical Corporation, Agricultural Chemicals Division. Bayer Report No.: 86182. BES Ref.: M-077776-01-1 Report date: 2 December 1983. Unpublished	
1.2	**Data protection**	Yes	
1.2.1	Data owner	Bayer CropScience AG	
1.2.2			
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I	
		2 GUIDELINES AND QUALITY ASSURANCE	
2.1	**Guideline study**	No (not required, as study started before 30 June 1988).	
2.2	**GLP**	No. When the study was performed, GLP was not compulsory (as study started before June 30 1988).	
2.3	**Deviations**	Not applicable	
		3 MATERIALS AND METHODS	
3.1	**Test material**	Cyfluthrin	
3.1.1	Lot/Batch number	Vial No.: C-70B	
3.1.2	Specification	As given in section 2	
3.1.3	Purity	Radiochemical purity >94%.	
3.1.4	Radiolabelling	[Phenyl-U-^{14}C]cyfluthrin. Radiochemical purity >94%. Specific activity 21.75 mCi/mmole	X
3.1.5	UV/VIS absorption spectra and absorbance value	Not available.	

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Phototransformation in water including identity of transformation products

3.1.6	Further relevant properties	-	
3.2	**Reference substances**	No information available	
3.3	**Test solution**	The methodology was stated to be from a previous experiment (Puhl et al, 1983). The only exception was that the samples were irradiated outdoors with natural sunlight That methodology is provided below. A solution of 1% acetonitrile in pH 5 sterile phosphate buffer was used to obtain a cyfluthrin concentration of 5 µg/l. This was placed in a screw cap borosilicate glass vessel and placed outdoors in natural Kansas, USA (38°N) sunlight on 15 August 1984. The tubes were tilted at a 30 degree angle with respect to the ground's surface, so that the sun's rays would be perpendicular to the samples Control samples were wrapped in aluminium foil and placed with the test samples. (See Table A7.1.1.2/01-1).	
3.4	**Testing procedure**		
3.4.1	Test system	A 1% acetonitrile in pH 5 sterile buffer solution was used to obtain a cyfluthrin concentration of 5 µg/l. This was placed in a screw cap borosilicate glass vessel. . (See Table A7.1.1.2/01-2).	
3.4.2	Properties of light source	Natural Kansas, USA (38°N) sunlight on 15 August 1984.	
3.4.3	Determination of irradiance	Light intensity measurements were taken daily at 8:30 am, 11:30 am and 4:30 pm with a Black-Ray Ultra violet meter, model J-221. Range : 1130-4950 µW/cm²	X
3.4.4	Temperature	max 89°F-94°F equivalent to 28,3°C-34,4°C min 60°F-68°F equivalent to 15,6°C-20°C	X
3.4.5	pH	Initial pH was 5. Final pH was not measured but expected to be close to 5 given that the solution was a buffer.	
3.4.6	Duration of the test	Natural sunlight outside over a maximum period of 14 days	
3.4.7	Number of replicates	Two	
3.4.8	Sampling	Test samples were taken at 1, 3, 7 and 14 days. Control samples were taken at 7 and 14 days. Sample storage before analysis was not stated.	
3.4.9	Analytical methods	Aqueous samples were extracted three times with dichloromethane: acetonitrile (2:1 v/v). The organic fractions were evaporated to dryness, dissolve in acetonitrile and analysed by TLC using toluene (100%) or toluene- dichloromethane-acetic acid (25:10:1), or benzene-ethyl acetate-acetic acid (50:10:1) as solvent systems. Radioactive zones on TLC-plates were located by autoradiography and scrapped off into vials for liquid scintillation counting. Identification was performed by co-chromatography of standards on TLC-plates, under UV-light or by autoradiography.	
3.5	**Transformation**	Transformation products were not tested: However they were identified	

	products	as part of the study with the parent molecule.	
3.5.1	Method of analysis for transformation products	Transformation products were analysed by TLC as for parent compound Reverse phase was also used with acetonitrile:MeOH:0.5 M aqueous sodium chloride 2:2:1). Phenol was examined using hexane-diethyl ether (1:1) and dichloromethane. A 2-dimensional tic of isolated FPBacid was carried out with DCM-methanol-ammonium hydroxide (70:25:1).	
		4 RESULTS	
4.1	**Screening test**	Not performed	
4.2	**Actinometer data**	Not used	
4.3	**Controls**	Initial amount: 89% cyfluthrin. Final (14 day) amount: 84% cyfluthrin	
4.4	**Photolysis data**		
4.4.1	Concentration values	The concentration values of the test substance at the start of the photolysis experiment and each time point in given in table A7.1.1.2/01-3	
4.4.2	Mass balance	See table-A7.1.1.2/01-3. Losses were postulated to be due to absence of trapping systems for volatile compounds	
4.4.3	k^c_p	DT_{50} <1 day, K^c_p >0.693 day^{-1}.	
4.4.4	Kinetic order	By observation	
4.4.5	k^c_p / k^a_p	Not relevant	
4.4.6	Reaction quantum yield (ϕ^c_E)	Not calculated.	
4.4.7	k_{pE}	No data.	
4.4.8	Half-life ($t_{1/2E}$)	DT_{50} under the natural sunlight conditions of the experiment (38°N) was <1 day.	X
4.5	**Specification of the transformation products**	The compound was radiolabelled in the phenyl –ring and the metabolites FPB-ald and FPB-acid were identified at a maximum of 18 and 37% respectively. (See Table A7.1.1.2/01-4). No other metabolites exceeded 10%.	
		5 APPLICANT'S SUMMARY AND CONCLUSION	
5.1	**Materials and methods**	A solution of 1% acetonitrile in pH 5 sterile buffer was used to obtain a cyfluthrin concentration of 5 µg/l. This was placed in a screw cap borosilicate glass vessel and placed outdoors in natural Kansas, USA (38°N) sunlight on 15 August 1984. Control samples were wrapped in aluminium foil and placed with the test samples.	
5.2	**Results and discussion**	Cyfluthrin was rapidly degraded with a DT_{50} of <1 day. The major metabolites were FPB-ald (max. 18%) and FPB-acid (max. 37%) which	

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Phototransformation in water including identity of transformation products

		were formed sequentially
5.2.1	k^c_p	
5.2.2	K_{pE}	
5.2.3	ϕ^c_E	
5.2.4	$t_{1/2E}$	
5.3	**Conclusion**	Photolysis of [phenyl-UL-14C] cyfluthrin by natural sunlight resulted in rapid cleavage of cyfluthrin's ester bond and formation of 4-fluoro-3-phenoxybenzaldehyde (FPB-ald) and 4-fluoro-3-phenoxybenzoic acid (FPB-acid) as the major photoproducts. The resulting half-life was 1 day.
5.3.1	Reliability	2
5.3.2	Deficiencies	Yes The study is not conducted to modern standards and guidelines. However this does not affect the major conclusion stated above This study is appropriate to demonstrate the rapid photolytic degradation of cyfluthrin and formation of the FPB-ald and FPB-acid metabolites following radiolabelling in the phenyl ring. Hence the validity of the study is not compromised.

Evaluation by Competent Authorities

EVALUATION BY RAPPORTEUR MEMBER STATE

Date 2006/09/26

Materials and Methods The applicant's version is acceptable with minor restrictions.

Comments

Due to study accomplishment by breach of GLP standards as well as posterior developed transformation test guidelines few discrepancies should be mentioned. Submitted test results should correspond to light intensities and spectral distribution form regions between 40 and 65° northern latitude during spring and autumn. Appropriate results should be given, e.g. by extrapolation.

Radiolabelled test substances should have radiochemical and chemical purities of ☐95 %.

Temperature during photolysis studies should be maintained within the range of 20 to 30 °C.

Discrepancies in the time schedule of light measurements attract attention as follows:

Original study report Appendix II: 12:30 pm

Summary original study report: 1:30 pm

This document subheading 3.4.3.: 11:30 am

Document IIIA/ Section 7.1.1.1.2 — Phototransformation in water including identity of transformation products

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Results and discussion	The applicant's version is acceptable with minor restrictions. Comments Calculation of measured photolysis rate constant is not traceable, thus equal for the experimental half-life. Indication of calculation method and correlation coefficient are missing. In 4.4.8 the sunlight half-life of the test substance in water for both the summer and winter session should be calculated.
Conclusion	The applicant's version is adopted.
Reliability	2
Acceptability	Acceptable
Remarks	As mentioned above the original study misses GLP standards and reference to approved test guidelines. Hence, partly the identification of major photoproducts is an accepted study result.
	COMMENTS FROM ...
Date	*Give date of comments submitted*
Materials and Methods	*Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant's summary and conclusion.* *Discuss if deviating from view of rapporteur member state*
Results and discussion	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	

Table A7.1.1.2/01-1: Description of test solution and controls

Criteria	Details
Purity of water	Not stated

Preparation of test chemical solution	A solution of 1% acetonitrile in pH 5 sterile phosphate buffer
Test concentrations (mg a.s./L)	Cyfluthrin concentration of 5 µg/l.
Temperature (°C)	max 83°F-94°F equivalent to 28,3°C-34,4°C *min 60°F-68°F equivalent to 15,6°C-20°C*
Preparation of a.s. solution	Not relevant
Controls	Identical solutions maintained in darkness
Identity and concentration of co-solvent	1% v/v acetonitrile

Table A7.1.1.2/01-2: Description of test system

Criteria	**Details**
Laboratory equipment	Screw cap borosilicate glass vessel placed outdoors in natural) sunlight
Test apparatus	none
Properties of artificial light source:	Not relevant
Nature of light source	
Emission wavelenght spectrum	
Light intensity	
Filters	
Properties of natural sunlight:	Natural sunlight used
Latitude	38°N
Hours of daylight	Not stated (outdoor condition)
Time of year	August
Light intensity	1150-4950 µW/cm²
Solar irradiance (L_λ)	Not stated

Table A7.1.1.2/01-3: Concentration values of the test substance and transformation products

	Day 0	Day 1	Day 3	Day 7	Day 14
Cyfluthrin	89	27	15	12	6
FPBald	2	10	16	18	12
FPBacid	2	21	28	33	37
TLC origin	<1	5	5	6	6
Other[1]	5	3	3	2	2
Diffuse	1	4	2	6	5
Aqueous	1	6	9	5	9
Total	**100**	**76**	**78**	**82**	**77**

1. Two unknowns also seen in the [phenyl-U-14C] cyfluthrin standard.

Table A7.1.1.2/01-4: Specification and amount of transformation products

CAS-Number	CAS and/or IUPAC Chemical Name(s)	Amount [%] of parent compound measured at
		pH_5
Not stated	4-Fluoro-3-phenoxybenzaldehyde (FPBald)	18
Not stated	4-Fluoro-3-phenoxybenzoic acid (FPBacid)	37

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1 REFERENCE

1.1	**Reference**	Puhl, R.J., Hurley, J. B. and Dime R. A. (1983). Photodecomposition of BAYTHROID-^{14}C in Aqueous Solution and on Soil. Mobay Chemical Corporation, Agricultural Chemicals Division. Report No.: 86182. BES N° M-072776-01-1 December 2, 1983. unpublished
1.2	**Data protection**	Yes
1.2.1	Data owner	Bayer CropScience AG
1.2.2		
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	No (not required, as study started before 30 June 1988).
2.2	**GLP**	No, When the study was performed, GLP was not compulsory.
2.3	**Deviations**	Not applicable

3 MATERIALS AND METHODS

3.1	**Test material**	Cyfluthrin:
3.1.1	Lot/Batch number	Vial N°.: C-2118 Vial N°.: C-276
3.1.2	Specification	As given in section 2
3.1.3	Purity	Radiochemical purity >97%.
3.1.4	Radiolabelling	[Phenyl-U-^{14}C] cyfluthrin. Radiochemical purity >97%. Specific activity 21.75 mCi/mmole [fluorophenyl-UL-^{14}C] cyfluthrin. Radiochemical purity >97%. Specific activity 21.75 mCi/mmole
3.1.5	UV/VIS absorption spectra and absorbance value	Not available.
3.1.6	Further relevant properties	None
3.2	**Reference substances**	4-Fluoro-3-phenoxybenzaldehyde-phenyl-UL-^{14}C (FPBald) Radiochemical purity: 98+ %, 3.92 mCi/mmole phenol-UL-^{14}C Radiochemical purity: 95+ %, 22.0 mCi/mmole
3.3	**Test solution**	A solution of 1% acetonitrile in pH 5 sterile phosphate buffer was used to obtain a cyfluthrin concentration of 5 µg/l.

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3.4	**Testing procedure**		
3.4.1	Test system	Photolysis apparatus : Merry-go-round reactor Light source : medium pressure Hg-lamp suspended in a borosilicate glass immersion well Control samples were wrapped in aluminium foil and placed with the test samples.	
3.4.2	Properties of light source	The intensity of the light source during the aqueous study was about 6700 $\mu W/cm^2$ at the sample surface, which is more than twice the average intensity of a July day in Kansas City, MO (2745 $\mu W/cm^2$).	
3.4.3	Determination of irradiance	The intensity distribution was measured with a scanning photometer; Schoeffel M-460).	
3.4.4	Temperature	The temperature in the vicinity of the samples was 25-28°C.	
3.4.5	pH	Initial pH was 5. Final pH was not measured but expected to be close to 5 given that the solution was a buffer.	
3.4.6	Duration of the test	Up to 144 hrs	
3.4.7	Number of replicates	Two	
3.4.8	Sampling	Aqueous samples (irradiated and controls)were taken at intervals of 0, 24, 48, 72, and 144 hr.	
3.4.9	Analytical methods	Aqueous samples were extracted three times with dichloromethane: acetonitrile (2:1 v/v). The organic fractions were evaporated to dryness, dissolve in acetonitrile and analysed by TLC.	
3.5	**Transformation products**	Transformation products were identified as part of the study with the parent molecule.	
3.5.1	Method of analysis for transformation products	Transformation products were analysed by TLC as for parent compound Standards were developed with the radioactive samples where appropriate. Standards were located by viewing the plates under UV light, while radioactive zones were located by autoradiography.	
		4 RESULTS	
4.1	**Screening test**	Not performed	
4.2	**Actinometer data**	Not used	
4.3	**Controls**	Radiocarbon in control solution at t_0 was 90.8% of applied radioactivity Radiocarbon in control solution at t = 144 hrs was 81.3% of applied radioactivity	X
4.4	**Photolysis data**		
4.4.1	Concentration values	The concentration values of the test substance at the start of the photolysis experiment and each time point is given in table A7.1.1.2/02-1	
4.4.2	Mass balance	See table A7.1.1.2/02-2. Losses were postulated to be due to absence of trapping systems for volatile compounds	
4.4.3	k^c_p	$K^c_p = 0.00236$ hrs^{-1}.	

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Phototransformation in water including identity of transformation products

4.4.4	Kinetic order	By observation	
4.4.5	k^c_p / k^a_p	Not relevant	
4.4.6	Reaction quantum yield (ϕ^c_E)	Not calculated.	
4.4.7	k_{pE}	No data.	
4.4.8	Half-life ($t_{1/2E}$)	DT_{50} = 12.2 days.	
4.5	**Specification of the transformation products**	The major product detected in the aqueous study was 4-fluoro-3-phenoxybenzoic acid (FPB-acid) at a maximum of 8.5% of the radioactivity recovered from irradiated control when 4-fluoro-3-phenoxybenzaldehyde (FPB-ald) was also identified at a maximum of 3% of the radioactivity recovered from irradiated control. (See Table A7.1.1.2/02-3).	

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1	**Materials and methods**	Irradiation of a sterile aqueous solutions of cyfluthrin buffered at pH 5 was performed for 144 hours using a medium pressure mercury vapour lamp Control samples were wrapped in aluminium foil and placed with the test samples.	
5.2	**Results and discussion**	Cyfluthrin was rapidly degraded with a DT_{50} of 12.2 days. The major metabolites detected were FPB-ald (max. 3%) and FPB-acid (max. 8.5%).	
5.2.1	k^c_p	K^c_p = 0.00236 hrs^{-1}.	
5.2.2	K_{pE}		
5.2.3	ϕ^c_E		
5.2.4	$t_{1/2E}$		X
5.3	**Conclusion**	Photolysis of [phenyl-UL-^{14}C] cyfluthrin by medium pressure Hg-lamp resulted in rapid cleavage of the ester bond and formation of 4-fluoro-3-phenoxybenzaldehyde (FPB-ald) and sequentially 4-fluoro-3-phenoxybenzoic acid (FPB-acid) as the major photoproducts. The resulting half-life was 12.2 day	
5.3.1	Reliability	2	
5.3.2	Deficiencies	Yes The study is not conducted to modern standards and guidelines . However this does not affect the major conclusions stated above Hence the validity of the study is not compromised.	

Evaluation by Competent Authorities	
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	2006/09/27
Materials and Methods	Despite minor deficiencies applicant's version is acceptable. Comments: Total mass balance should fall within that range 90% to 110% of the total applied radioactivity. Percentage represented by each Diastereoisomer of BAYTHROID should be provided.
Results and discussion	Despite minor deficiencies applicant's version is acceptable. Comments: The original test study provides sunlight half-life $t_{1/2}$ = 60 d of the test substance due to extrapolations between light intensity of the mercury light source (6700 µW/cm²) and the sunlight of a July day at Kansas City (2745 µW/cm²).
Conclusion	Applicant's version is adopted.
Reliability	2
Acceptability	acceptable
Remarks	As mentioned above the original study misses GLP standards and reference to approved test guidelines. Hence partly the identification of major photoproducts including percent of parent compound is an accepted study result.
	COMMENTS FROM:
Date	*Give date of comments submitted*
Materials and Methods	*Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant's summary and conclusion.* *Discuss if deviating from view of rapporteur member state*
Results and discussion	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	

Table A7.1.1.2/02-1: Radiocarbon in solution during photolysis of aqueous solution of ^{14}C-cyfluthrin

	^{14}C recovered, as percent of applied	
Hours	irradiated	Dark control
0	90.8	90.8
24	79.0	75.0
48	74.0	78.5
72	72.5	71.5
144	74.2	81.3

Table A7.1.1.2/02-2: Radiocarbon recoveries during photolysis of aqueous solution of ^{14}C-cyfluthrin

	^{14}C recovered[a], as percent of applied[b]	
Hours	irradiated	Dark control
0	96.3	96.3
24	93.2	94.8
48	87.1	89.4
72	87.6	90.6
144	83.4	91.3

[a] **Recoveries were determined following extraction and include organosoluble and water soluble radioactivity**

[b] **Average of two replicates**

Table A7.1.1.2/02-3: Concentration values of the test substance and transformation products

	^{14}C as % recovered from irradiated control[a]				
hours	0	24	48	72	144
Cyfluthrin	95.3	84.4	81.1	77.7	66.1
FPBacid	0.8	4.0	5.8	6.6	8.5
FPBald	-	-	1.9	2.3	3.0
TLC origin	1.3	4.1	3.8	4.7	3.5
Other[1]	1.8	4.5	3.5	4.1	4.3
subtotal	**99.2**	**97.0**	**96.1**	**95.4**	**85.4**
Aqueous	0.8	1.3	1.3	1.3	5.9
Lost	-	1.7	2.6	3.3	8.7
Total	100	100	100	100	100

[a] for recoveries see table A7_1_1_2-2

Document IIIA/ Section 7.1.1.1.2/03 — **Other Soil degradation** — X

BPD Data set IIA/ Annex Point VII.7.6.2.2 — Phototransformation in soil including identity of transformation products

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1 REFERENCE

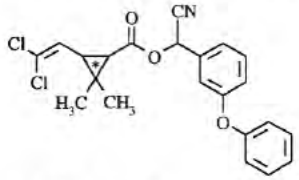
1.1	**Reference**	Takahashi, N., Mikami, N., Matsuda, T. and Miyamoto, J (1985). Photodegradation of the pyrethroid insecticide cypermethrin in water and on soil surface. Journal of Pesticide Sci., 10, 629-642. Laboratory of Biochemistry and Toxicology, Sumitomo Chemical Co., BES Ref.: M-072742-01-1 Published paper This study on cypermethrin was summarised to give an information on the fate and behaviour of the cyclopropyl moiety, which is in common with cyfluthrin
1.2	**Data protection**	No
1.2.1	Data owner	Published study
1.2.2	Criteria for data protection	No data protection claimed

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	No
2.2	**GLP**	No, GLP was not compulsory at the time the study was performed (as study started before June 30 1988).
2.3	**Deviations**	None

3 MATERIALS AND METHODS

3.1	**Test material**	Cypermethrin
3.1.1	Lot/Batch number	Not specified
3.1.2	Specification	*Cis-trans* cypermethrin isomers labelled either in the cyclopropyl, cyano or benzyl ring positions, with specific activity ranging from 22mCi/mmol to 28 mCi/mmol..
3.1.3	Purity	The radiochemical purity of each preparation was >99%.
3.1.4	Radiolabelling	Radiolabelling at the cyclopropyl position (^{14}C-*P1)* is shown below:



Radiochemical purity = 99%, 22mCi/mmol

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		For preparation radiolabelled at the cyano position (^{14}CN), radiochemical purity is also >99%, 28 mCi/mmol For preparation radiolabelled at the benzyl position (benzyl-^{14}C-*P1*), radiochemical purity is also >99%, 25.2 mCi/mmol	
3.1.5	UV/VIS absorption spectra and absorbance value	Not applicable	
3.1.6	Further relevant properties	The compound used was a mixture of 8 isomers with 2 chiral centers: cyclopropyl C-1 and C-3 and the benzyl methane carbon atom.	
3.2	**Reference substances**	See Table 7.1.1.1.2/03-1 for the list of reference substances used and the corresponding R_f values on pre-coated TLC plates on different solvent systems.	
3.3	**Test solution**	aqueous solutions: 50 ppb soils: 1.1 µg/cm^2	
3.4	**Testing procedure**		
3.4.1	Test system	Test water:	Distilled water, 2% aqueous acetone, natural river water (pH 8.7), natural seawater (pH 8.3); humic acid (1 ppm) aqueous solution; All solutions contained Tween 85 (no absorption above 290 nm) to prevent cypermethrin from sticking to the glass wall. Water samples were sterilized through 0.1µm filter paper immediately before use.
		Soil samples:	Kodeira light clay: (pH 5.5; clay loam*) Katano sandy loam (pH 4.6; sand*) Azuchi sandy clay loam (pH 6.3; sandy loam*) (* UK ADAS classification system)

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	Test procedure:	Aqueous: Each flask was exposure to natural sunlight for 10 days (*ca* 8 hours per day). Sunlight intensity with wavelengths between 300 and 400 nm was approx. 290, 1180, and 239 µW/cm^2 at the beginning, middle, and end of the day, respectively. Volatile ^{14}C was trapped in NAOH solution. Control samples were kept in the dark. Soil: Onto soil TLC plate, ^{14}C-*P1* in diethyl ether was evenly applied at the rate of 1.1 µg/cm^2 and exposed to natural sunlight. Sunlight intensity with wavelengths between 300 and 400 nm was approximately 1010, 1640, and 270 µW/cm^2 at the beginning, middle, and end of the day, respectively. Control samples were kept in the dark.
3.4.2 Duration of the test	10 days	
3.4.3 Number of replicates	Two replicates per sampling day for the aqueous samples; one sample each for the soil samples.	
3.4.4 Sampling	Aqueous solutions: 0, 1, 2, 4, 7 and 10 days (except 2 % acetone: 0.5 days); Soils: 0, 1, 2, 3, 5 and 7 days	
3.4.5 Extraction	Aqueous suspensions: Samples were acidified prior to extraction with ethyl acetate to release $^{14}CO_2$ and H^{14}CN which were then either precipitated out or trapped as volatiles for determination together with the substances trapped during photolysis. Soil suspensions: The soil thin layer was scraped off and extracted with acetone/distilled water and centrifuged.	
3.4.6 ^{14}C determination and quantification:	Liquid samples and extracts of soil samples were radioassayed by liquid scintillation and the remainder evaporated and determined by TLC and chiral-HPLC with UV detection (230 nm). For TLC analysis, the following four separate solvent systems were used: hexane/diethyl ether (20/1); hexane/toluene/acetic acid (3/15/2); benzene saturated with formic acid/diethyl ether (10/3) and toluene/diethyl ether/acetic acid (75/25/1). Radioactive zones on TLC-plates were located by autoradiography. Unextracted soil samples were combusted to determine $^{14}CO_2$ and also fractionated into fulvic, humic acids and humins.	
3.4.7 Identification	Co-chromatography of standards on TLC-plates (silica gel, 60 F 254). Visualisation under UV-light or by autoradiography; HPLC-UV, IR-spectra, NMR spectra, EI-MS spectra and GC-MS	
3.4.8 Determination of quantum yield	Quantum yields were determined by comparing the photolysis rate with that of parathion as a reference compound using a merry-go-	

Document IIIA/ Section 7.1.1.1.2/03 — **Other Soil degradation** — x

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round apparatus.

4 RESULTS

4.1	**Screening test**	Not performed	
4.2	**Radioactive distribution after photodegradation in test system**	The photodegradation of cyclopropyl-14C-cypermethrin by sunlight in distilled water, 2% aqueous acetone, river water and humic acid aqueous solution revealed that in all cases DCVA (*cis*- and *trans*-, resulting from isomerisation) was the major degradation product resulting from experiments with the cyclopropyl label. 3-phenoxybenzoic acid was also a major degradate, which was observed with the benzyl label. Except from the artificial system "aqueous acetone" where some minor additional degradation products were formed, no other compound could be detected'. On average after 10 days DCVA accounted for 13.5%, 17.0 % and 39.9 % of the applied radioactivity in distilled water, humic acid aqueous solution and river water, respectively. 3-phenoxybenzoic acid accounted for 21.8%, 63.5% and 29.6% in distilled water, humic acid aqueous solution and river water, respectively. Less than 3 % were volatiles and 20.7 %, 22.3 % and 7.6 %, respectively, were not identified. No other degradate individually accounted for >10% applied radioactivity (Table A7.1.1.1.2/03-2). After photolysis on soil levels of DCVA were very small (< 0.1 % of the applied radioactivity), however PB-acid, the second metabolite resulting from ester cleavage of cypermethrin, was found in amounts form 3 - 12 % of the applied radioactivity. The major metabolite detected 7 days after treatment was the amide analogue of cypermethrin (NH2CO-cypermethrin), no other metabolites retaining the ester bond could be detected. Depending on soil type up to 20 % of the applied radioactivity could not be detected and a max. of 47.3 % was not extractable from soil. In addition, losses occurred, probably due to volatile compounds (Table A7.1.1.1.2/03 -3).	X
4.3	**Mean balance of radioactivity**	The total amount of radioactivity recovered in the water samples ranged from 82.5% to 97.8% for the *cis*-isomer and 78.3% to 95.8% for the *trans*-isomer. Recovery of applied radioactivity in soil surfaces was much lower, ranging from 61.6% to 85.8% for both isomers.	
4.4	**Effect of pH**	Both isomers were fairly stable in the solutions tested (in the dark), but in slightly basic media such as river water, both isomers were gradually degraded. After 7 days in the dark, the recovered *cypermethrin* amounted to 55.3-59.6% and 83.9 – 88.7% of the applied radioactivity in river water and see water respectively.	X
4.5	**Half-life**	The *cis* isomer was photodecomposed 1.4 to 1.7 times faster in sunlight than the trans-isomer, in water. The half-life of the *cis*-isomer was 2.3 and 2.6 days in 1 ppm humic acid aqueous solution and in distilled water, respectively. The half life of the cis isomer was 0.6 – 0.7 day in natural river water, and <0.5 days in 2%	

Document IIIA/ Section 7.1.1.1.2/03 — **Other Soil degradation** — x

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aqueous acetone.

From an additional experiment the half-lives for *cis*- and *trans*-DCVA were calculated to be 22.3 and 32.9 days.

On three types of soil samples, both isomers were rapidly photodegraded with the initial half-life of 0.6 – 1.9 days.

4.6 Formation and identity of transformation products

The photoreactions involved were: 1R/1S and cis/trans isomerisation of the cyclopropane ring, cleavage of the ester or diphenyl ether linkage, oxidation of the CHO group to the COOH group, hydration of the CN group to $CONH_2$, hydrolysis of the $CONH_2$ to COOH group, oxidative cleavage of the halogenated side chain, dehalogenation, intramolecular cyclisation to form γ- or δ-lactone, and photomineralisation of the cyclopropyl C-1, cyano- and benzyl-ring to $^{14}CO_2$.

4.7 Degradation pathway

The proposed degradation pathway is shown below.

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1 Materials and methods

Photodegradation of (1R, *cis*, αRS)- and (1R, trans, αRS)- α-cyano-3- phenoxybenzyl (1RS)-cis, *trans*-3-[2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate] in water and on soil surface was studied, using 14C preparation labelled separately at the cyclopropyl C-1, cyano or benzyl ring.

Water samples (distilled water, 2 % aqueous acetone, river water, humic acid (1 ppm) aqueous solution) and soil samples (light clay, sandy loam, and sandy clay loam) were exposed to natural sunlight for 10 and 7 days (*ca* 8 hours per day), respectively. After extraction, liquid samples and extracts of soil samples were radioassayed by liquid scintillation and the remainder evaporated and components determined by TLC and HPLC. Radioactive zones on TLC-plates were located by autoradiography. Soil samples were combusted to determine $^{14}CO_2$

5.2 Results and discussion

This study on cypermethrin was summarised to give an information on the fate and behaviour of the cyclopropyl moiety, which is in common with cyfluthrin

The *cis* isomer was photodecomposed 1.4 to 1.7 times faster in sunlight than the *trans*-isomer in water. The half-life of the *cis*-isomer was 2.3 and 2.6 days in 1 ppm humic acid aqueous solution and in distilled water, respectively. The half life of the cis isomer was 0.6 – 0.7 day in natural river water, and <0.5 days in 2% aqueous acetone. X

On three types of soil samples, both isomers were rapidly photodegraded with the initial half-life of 0.6 – 1.9 days.

The only significant metabolite resulting from the cyclopropyl moiety was DCVA.

Document IIIA/ Section 7.1.1.1.2/03	**Other Soil degradation**	x
BPD Data set IIA/ Annex Point VII.7.6.2.2	Phototransformation in soil including identity of transformation products	

5.3 Conclusion	It can be concluded that cypermethrin in aqueous solutions or adsorbed to soil will be rapidly degraded if exposed to natural sunlight. The only significant metabolite resulting from the cyclopropyl moiety is DCVA.	
5.3.1 Reliability	2	
5.3.2 Deficiencies	None	

Evaluation by Competent Authorities

EVALUATION BY RAPPORTEUR MEMBER STATE

Date	2006/09/28
Materials and Methods	Applicant's version is acceptable.
Results and discussion	Despite some deficiencies applicant's version is acceptable. Comments: Both data and result recapitulation of the above mentioned original study by Takahashi et al. is not always retraceable, e.g. data in 4.2 and Table A7.1.1.1.2/03-2. Values for distribution of radioactivity after photolysis in river water and humic acid solution seem to be interchanged. In Table A7.1.1.1.2/03-2, values for the photodegradation product cis- and trans-DCVA are consolidated whereas in 4.2. a selection of results is discussed. After 7 days in the dark, the recovered cypermethrin amounted to 55.3-59.6 % (trans-cypermethrin) and 88.7-83.9 % (cis-cypermethrin) of the applied radioactivity in river water and sea water respectively. (4.4) Applying sunlight, the cis-isomer was photodecomposed 1.4 to 1.7 times faster than the trans-isomer in natural waters and humic acid solutions. Furthermore, the photodegradation of both isomers in river and sea water is 3-4 times as rapid as in distilled water.
Conclusion	Additionally, relevant degradation products by photolysis are 3-phenoxy-benzoic acid (recovers 25.6, 9.5, 67.8 and 29.8 % of applied ^{14}C in distilled water, acetone solution, river water and humic acid solution, respectively) and 3-phenoxybenzaldehyde (up to 8.4 % of applied ^{14}C in distilled water).
Reliability	3
Acceptability	Acceptable with restrictions (see above).
Remarks	A photodegradation study of cypermethrin was summarised to give information about fate and behaviour of cyfluthrin (additional fluorid bond at the phenoxy group).

COMMENTS FROM ...

Date	*Give date of comments submitted*

Document IIIA/ Section 7.1.1.1.2/03 — Other Soil degradation

X

BPD Data set IIA/ Annex Point VII.7.6.2.2 Phototransformation in soil including identity of transformation products

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

Table A7.1.1.1.2/03-1: Reference substances used and the corresponding R_f values on pre-coated TLC plates on different solvent systems

Chemical	Rf Values			
	A	B	C	D
(1RS, cis, α*RS)-P1*	0.43, 0.48	0.84	0.67	0.68
(1RS, trans, α*RS)-P1*	0.36, 0.40	0.84	0.67	0.68
cis-P2	0.0	0.28, 0.31	0.25, 0.28	0.14, 0.16
trans-P2	0.0	0.27, 0.29	0.23, 0.26	0.13, 0.15
cis-P3	0.0	0.34, 0.37	0.57	0.17, 0.20
trans-P3	0.0	0.33, 0.34	0.54	0.17, 0.19
cis-P4	0.0	0.31, 0.34	0.50	0.43, 0.45
trans-P4	0.0	0.30, 0.32	0.50	0.43, 0.45
P5	0.0	0.05	0.16	0.04
cis-P6	0.02	0.26	0.57	0.45
Trans-P6	0.01	0.26	0.57	0.38
P7	0.0	0.26	0.20	0.15
P8	0.02	0.48	0.42	0.39
P9	0.09	0.47	0.44	0.44
P10	0.07	0.44	0.41	0.42
P11	0.13	0.56	0.51	0.49
P12	0.0	0.15	0.31	0.18
P13	0.43	0.45	0.69	0.61
P14	0.0	0.29	0.52	0.32
P15	0.0	0.28	0.34	0.17
P16	0.0	0.16	0.30	0.09
P17	0.18	0.66	0.61	0.52
P18	0.0	0.18	0.19	0.04
P19	0.0	0.42	0.46	0.19
P20	0.02	0.65	0.59	0.60
P21	0.02	0.28	0.49	0.38
P22	0.0	0.06	0.14	0.02
P23	0.0	0.07	0.20	0.02

A: hexane/diethy ether (20/1), B: hexane/toluene/acetic acid (3/15/2); C: benzene saturated with formic acid/diethyl ether (10/3); D: toluene/diethyl ether/acetic acid (75/25/1).

Table A7.1.1.1.2/03-2: Distribution of radioactivity after photolysis of cis- and trans cyclopropyl- ^{14}C-cypermethrin (50 ppb) in aqueous solutions (values are given in % of applied radioactivity and are average figures resulting from use of cis- and trans-cypermethrin)

	Distilled water	Humic acid solution	River water	2% aqueous acetone
Volatile ^{14}C	0.6	2.1	1.5	1.1
Extractable ^{14}C	85.6	89.3	88.8	82.5
DCVA (cis + trans)	26.9	79.8	33.9	9.1
Ident., retaining ester bond	38.0	1.9	31.4	4.5
Indent., ester bond cleaved	<0.1	<0.1	<0.1	3.9
Unidentified	20.7	7.6	23.5	65.1
Aqueous ^{14}C	4.8	3.9	4.7	5.2
Total ^{14}C	**91.0**	**95.3**	**95.0**	**88.8**

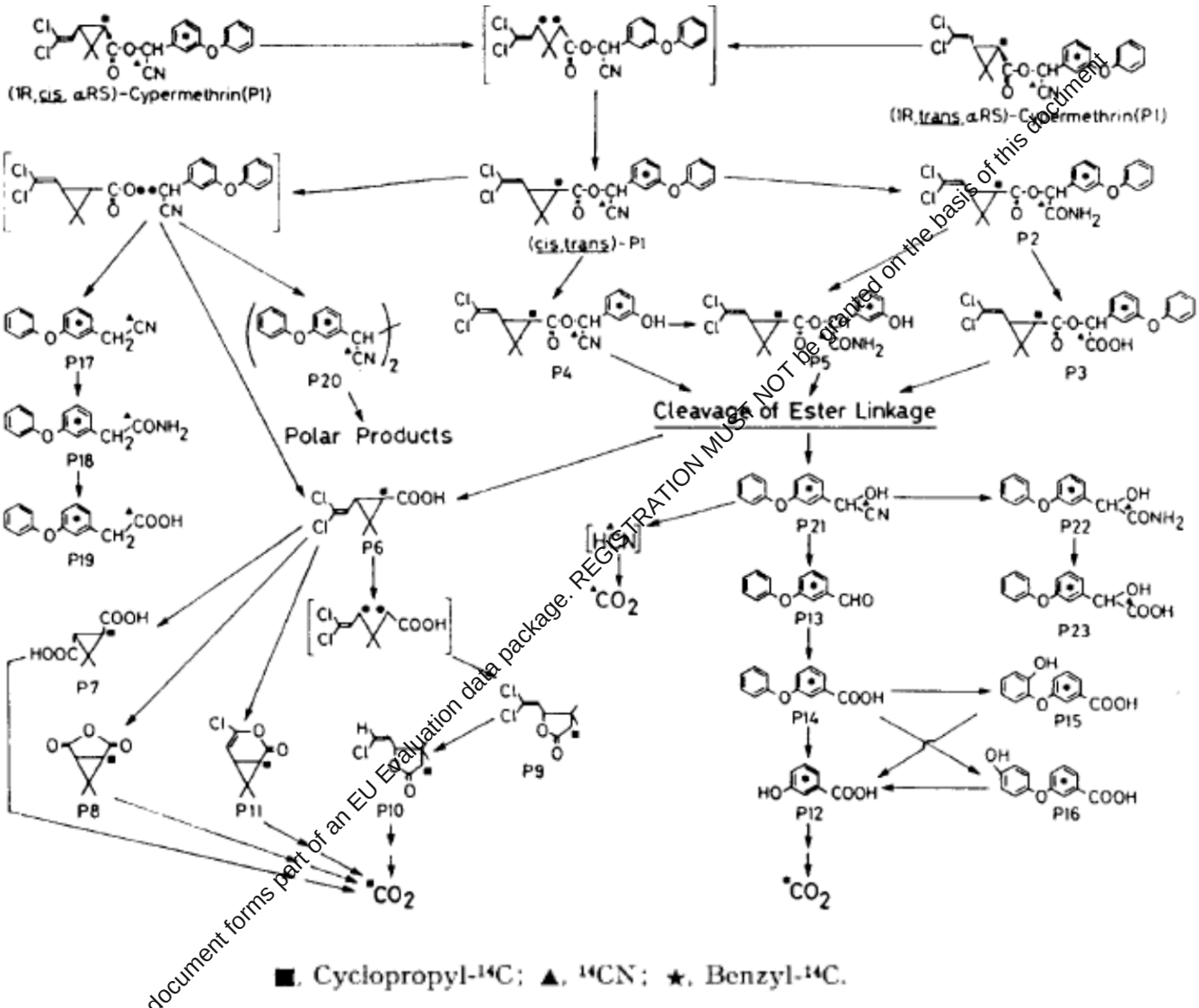
Table A7.1.1.1.2/03-3: Distribution of radioactivity after photolysis of cis cyclopropyl- ^{14}C-cypermethrin on soils (values are given in % of applied radioactivity)

	Kodeira	Azuchi	Katano
Extractable ^{14}C	47.3	65.6	57.7
DCVA (cis + trans)	<0.1	<0.1	<0.1
Amide-cypermethrin	25.1	31.2[1]	4.8
Metab. Retaining ester bond	3.8	43.2	54.5
Metab.: ester bond cleaved	<0.1	<0.1	<0.1
Unidentified	8.4	14.2	20.1
Bound ^{14}C	47.3	11.23	11.6
Humic acid	5.0	1.4	2.1
Humin	19.3	1.8	1.4
Fulvic acid	23.0	8.1	8.1
Total ^{14}C(dark control)[2]	**84.6 (103.3)**	**76.9 (88.2)**	**69.3 (97.5)**

1 original figure from the paper; however, the value is quite high and the sum of all metabolites exceeds 65.6%

2 benzyl label

Proposed Photodegradation Pathways for Cypermethrin (*P1)*



Document IIIA/ Section A7.1.1.1.2/04 — Phototransformation in water including identity of the products of transformation

BPD Data set IIA/ Annex Point VII.7.6.2.2

			Official use only
		1 REFERENCE	
1.1	**Reference**	Hellpointer, E. (1991). Determination of the quantum yield and assessment of the environmental half-life of the direct photodegradation of cyfluthrin in water. Bayer AG Crop protection-Research, Environmental research, Institute for Metabolism Research, Leverkusen Bayerwerk. Bayer AG Report No.: Pf 3555 BES Ref: M-073620-01-2 Report date: 4 September 1991 Unpublished	
1.2	**Data protection**	Yes	
1.2.1	Data owner	Bayer CropScience AG	
1.2.2	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I	
		2 GUIDELINES AND QUALITY ASSURANCE	
2.1	**Guideline study**	Yes, Phototransformation of chemicals in water, Part A: Direct phototransformation, UBA, Berlin, FRG (1990).	
2.2	**GLP**	Yes	
2.3	**Deviations**	None	
		3 MATERIALS AND METHODS	
3.1	**Test material**		
3.1.1	Lot/Batch number	800420ELB01	
3.1.2	Specification	As given in Section 2	
3.1.3	Purity	94.5%	X
3.1.4	Further relevant properties	None	
3.2	**Test method:**	ECETOC method based on the split-up of the polychromatic light into defined wavelength ranges. In the present test conception, groups comprising 5 nm were formed in the range of wavelengths from 295 to 400 nm and groups comprising 10 nm from 401 nm on. The quantum yield of a photochemical reaction is defined by that proportion of light quanta being absorbed by a substance which results in a reaction. The test was conducted in a merry-go-round irradiation apparatus, fitted with a mercury lamp (>295 nm). Light intensity was measured using the chemical actinometer uranyloxalate. Quantum yield was calculated using the QUANT (Lit. 10) program.	
3.3	**Test solution**	Acetonitrile/water (1:1, v:v)	

Document IIIA/ Section A7.1.1.1.2/04

Phototransformation in water including identity of the products of transformation

BPD Data set IIA/ Annex Point VII.7.6.2.2

3.4	**Testing procedure**			
3.4.1	Test system	Test water:	Acetonitrile/water (1:1, v:v)	
		Test conditions:	The test solution was filled in quartz cuvettes in a merry-go-round apparatus of Mangels Co. and irradiated with a mercury immersion lamp TQ 150 of Original Hanau Co. In the present test conception wavelengths between 295 and 400 nm were used.	
		Concentration:	5.10 or 5.14 mg/1 cyfluthrin as initial for the degradation experiments 39.5 mg/1 for the calculation of the quantum yield	
3.4.2	Sampling	0, 0.5, 1, 1.5, 2, 3, 4, 5, 6, 7, 8, 8.3, 8.6 hours		X
3.4.3	Identification and quantification	Reverse-phase HPLC-UV		
		4 RESULTS		
4.1	**Screening test**	Not performed		
4.1.1	UV-absorption properties	The UV-absorption spectrum of cyfluthrin in water/acetonitrile (1:1; v:v) (C = 29.5 mg/l) shows a main maximum under 200 nm with a band width up to about 220 nm and a small 2nd maximum at 268 nm (ε = 1854 l/mole) cm; band width up to about 280 nm). The absorption of cyfluthrin with e = 161 l/mole cm at 295 nm to e = 14 l/mole cm at 381 nm extends only relatively weakly but very far into the environmentally relevant range of wavelengths. Direct interactions of cyfluthrin in aqueous solution with the sunlight in the troposphere are therefore possible. The UV-absorption spectra of cyfluthrin which was absorbed on the silica gel surface, basically confirmed the extreme extent of absorption into the environmentally relevant range of wavelengths. Consequently it is also possible that the active ingredient which is present in water predominantly in the absorbed form interacts with the sunlight in the troposphere.		
4.1.2	Intensity of radiation	The actinometer determination showed a titration difference of 0.85 and 0.79 ml for the two degradation experiments. From this an intensity of the radiation absorbed by the actinometer of 7.756 and 7.208 x 10E16 photons/sec and 3 ml was calculated (in the range from 295 - 490 nm).		
4.1.3	Quantum yield	The photolytic half-lives of cyfluthrin were 5 - 6 hours, determined in two experiments. From the kinetic results of the two photo degradation experiments and the UV absorption data the quantum yield was calculated to be 0.0052.		
4.1.4	Environmental half-	The resulting quantum yield and UV absorption data in aqueous		

	lives	solution were used to estimate the environmental half-life of cyfluthrin in water by two different simulation models. According to the GC-SOLAR-program environmental half-lives of 2.8 to 58 days were calculated depending on the season and the geographical degree of latitude (30° - 60°) (See Table A7.1.1.1.2/04-1. Additional conditions: Pure water from close to the surface (0 -1 cm), 10th degree of longitude, clear sky, typical ozone concentrations in the atmosphere, half-lives integrated over the entire day. The model of Frank & Klöpffer resulted in half-lives of (minimum) 2.8 to 5.1 days and (maximum) 16 to 32 days between April and September (Table A7.1.1.1.2/04-2. Additional conditions: Pure water from close to the surface (0 - 1 cm), stagnant water, geographic and climatic conditions of Germany (50th degree of latitude), no contribution of another mono- or bimolecular elimination process, half-lives integrated over the entire day.
	5 APPLICANT'S SUMMARY AND CONCLUSION	
5.1	**Materials and methods**	The quantum yield of direct photodegradation of cyfluthrin in aqueous solution was determined according to the ECETOC-method in polychromatic light.
5.2	**Results and discussion**	From the UV absorption data and the kinetic results of two photodegradation experiments in a merry-go-round irradiation apparatus the quantum yield was calculated to be 0.0052. The resulting quantum yield and UV absorption data in aqueous solution were used to estimate the environmental half-life of cyfluthrin in water ranging from 3 to 58 days and 3.1 to 32 days by two different simulation models GC-SOLAR and Frank and Klöpffer – programs, respectively.
5.3	**Conclusion**	The model assessments show that an essential contribution to the elimination of cyfluthrin in the environment is to be expected from the direct photodegradation in the aqueous medium and that the half-life for direct photodegradation may range from 3 to 60 days.
5.3.1	Reliability	1
5.3.2	Deficiencies	None

Evaluation by Competent Authorities

EVALUATION BY RAPPORTEUR MEMBER STATE

Date	2006/09/28
Materials and Methods	Applicant's version is accepted. Comments: Non-radiolabelled test substances should have a chemical purity of 95% or greater. Percentage represented by each Diastereoisomer of Cyfluthrin should be provided. In 3.4.2 properties of light source should be described.
Results and discussion	Applicant's version is adopted.
Conclusion	Applicant's version is adopted.
Reliability	1
Acceptability	acceptable
Remarks	Applying ABIWAS 2.0 (based on model calculation by Frank and Klöpffer) and taking into account following assumption: – pure water from close to the surface (0 - 10 cm), – geographic and climatic conditions of Middle Europe (55th degree of latitude) a recalculation by CA shows comparable results of environmental half-lives between 5 and 90 days in dependence of season summer and winter, respectively.

COMMENTS FROM ...

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

Table A7.1.1.1.2/04-1: GC-Solar –Program

Season	Environmental half-lives (days)			
	30th	40th	50th	60th degree of latitude
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	20.0	58

Additional conditions: Pure water from close to the surface (0-1 cm), 10th degree of longitude, clear sky, typical ozone concentrations in the atmosphere, half-lives integrated over the entire day.

Table A7.1.1.1.2/04-2: Frank and Klöpffer - Program

Month	**Environmental half-lives (days)**		
	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

Additional conditions: Pure water from close to the surface (0 - 1 cm), stagnant water, geographic and climatic conditions of Germany (50th degree of latitude), no contribution of another mono- or bimolecular elimination process, half-lives integrated over the entire day.

Document IIIA/ Sections 7.1.1.2.1	Ready Biodegradability	
BPD Data Set IIA/ Annex Point VII.7.6.1.1		
	JUSTIFICATION FOR NON-SUBMISSION OF DATA	Official use only
Other existing data [ ]	Technically not feasible [ ] Scientifically unjustified [✓]	
Limited exposure [ ]	Other justification [ ]	
Detailed justification:	A ready biodegradability study on cyfluthrin was not performed since the test substance is regarded as "Not ready biodegradable in water" The higher tier water sediment studies (IIIAXII.2.1, A.7.1.2.2/01-02) clearly show that, while cyfluthrin is mineralized to CO_2 to a great extent at the termination of the studies, the portions of carbon dioxide which are formed at 28 days ranged between 7 and 43%. Thus, it would be expected that if a ready biodegradability study were to be conducted, the results would lead to the conclusion that cyfluthrin is not readily biodegradable in water. It should be noted that a ready biodegradability study can be performed with surface water as the inoculums (OECD-301), further supporting the fulfillment of this study by the water/sediment studies Conclusion: No ready and inherent biodegradability studies are available. However radiolabelled higher tier water/sediment simulation studies are available, which describe route and rate of both abiotic and biological degradation in natural water systems, thereby giving an indication of biodegradability of cyfluthrin. Furthermore ready biodegradation tests were not designed to generate degradation rates and it is considered that the radiolabelled higher tier water/ sediment simulation studies provide more meaningful data for the overall assessment of the environmental fate and biodegradability. Therefore these data from water/sediment systems are considered scientifically valid for evaluating the biodegradability and fate of the chemical in the environment. It is intended to submit the higher tier sediment/water study to meet the requirements of the ready and inherent biodegradability study requirement (please refer to Document IIIA, section 7.1.2.2.2). This approach has been agreed with the Competent Authority (BAuA).	
Undertaking of intended data submission [ ]		

Document IIIA/ Sections 7.1.1.2.1	Ready Biodegradability
BPD Data Set IIA/ Annex Point VII.7.6.1.1	
	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	2008/11/11
Evaluation of applicant's justification	Applicant's version is acceptable.
Conclusion	Applicant's justification is acceptable since higher tier simulation studies for the relevant environmental exposure compartments soil and freshwater/sediment are available. Cyfluthrin is regarded as "not readily biodegradable".
Remarks	-
	COMMENTS FROM OTHER MEMBER STATE (specify)
Date	*Give date of comments submitted*
Evaluation of applicant's justification	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Remarks	

Document IIIA/ Sections 7.1.1.2.2	Inherent Biodegradability	
BPD Data Set IIA/ Annex Point VII.7.6.1.2		
	JUSTIFICATION FOR NON-SUBMISSION OF DATA	Official use only
Other existing data [ ]	Technically not feasible [ ] Scientifically unjustified [✓]	
Limited exposure [ ]	Other justification [✓]	
Detailed justification:	Inherent biodegradability tests allow prolonged exposure of the test compound to microorganisms due to the lower compound/biomass ratio creating more favourable conditions for biodegradation. However as such, biodegradation under environmental conditions may not be assumed and the tests are not considered to provide adequate information for risk assessment purposes (TNsG, Chapter 3, section 7.0.2.2.2) "Core-data testing for inherent biodegradability may in general not be appropriate, since these tests do not provide adequate information for risk assessment purposes." No ready and inherent biodegradability studies are available. However radiolabelled higher tier water/sediment simulation studies are available, which describe route and rate of both abiotic and biological degradation in natural water systems, thereby giving an indication of biodegradability of cyfluthrin. Furthermore ready biodegradation tests were not designed to generate degradation rates and it is considered that the radiolabelled higher tier water/ sediment simulation studies provide more meaningful data for the overall assessment of the environmental fate and biodegradability. Therefore these data from water/sediment systems are considered scientifically valid for evaluating the biodegradability and fate of the chemical in the environment. It is intended to submit the higher tier sediment/water study to meet the requirements of the ready and inherent biodegradability study requirement (please refer to Document IIIA, section 7.1.2.2.1). This approach has been agreed with the Competent Authority (BAuA).	
Undertaking of intended data submission [ ]		

Document IIIA/ Sections 7.1.1.2.2	Inherent Biodegradability
BPD Data Set IIA/ Annex Point VII.7.6.1.2	
	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	2008/11/11
Evaluation of applicant's justification	Applicant's version is acceptable.
Conclusion	Applicant's justification is acceptable since higher tier simulation studies for the relevant environmental exposure compartments soil and freshwater/sediment are available. Cyfluthrin is regarded as "not inherently biodegradable".
Remarks	-
	COMMENTS FROM OTHER MEMBER STATE (specify)
Date	*Give date of comments submitted*
Evaluation of applicant's justification	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Remarks	

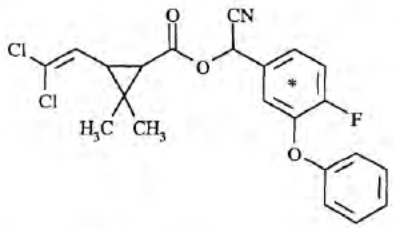
Document IIIA/ Sections 7.1.1.2.3 **BPD Data Set IIA/ Annex Point XII.2.1**	**Biodegradation in seawater**	
	JUSTIFICATION FOR NON-SUBMISSION OF DATA	Official use only
Other existing data [ ] **Limited exposure [ ]**	**Technically not feasible [ ]** **Scientifically unjustified [ ]** **Other justification [✓]**	
Detailed justification:	Cyfluthrin is not to be used or released in marine environments when its biocidal products are used according to the label recommendations. Therefore no biodegradation in seawater is necessary.	
Undertaking of intended data submission [ ]		
	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
	EVALUATION BY RAPPORTEUR MEMBER STATE	
Date	2007/05/09	
Evaluation of applicant's justification	The justification for non-submission of a test on biodegradation in seawater is acceptable. Neither use nor release in the marine environment is to be expected.	
Conclusion	Applicant's justification is acceptable.	
Remarks	-	
	COMMENTS FROM OTHER MEMBER STATE *(specify)*	
Date	*Give date of comments submitted*	
Evaluation of applicant's justification	*Discuss if deviating from view of rapporteur member state*	
Conclusion	*Discuss if deviating from view of rapporteur member state*	
Remarks		

Document IIIA/ Sections 7.1.2.1.1 BPD Data Set IIA/ Annex Point XII.2.1	Aerobic biodegradation	
	JUSTIFICATION FOR NON-SUBMISSION OF DATA	**Official use only**
Other existing data [ ] **Limited exposure [ ]**	**Technically not feasible [ ]** **Scientifically unjustified [✓]** **Other justification [ ]**	
Detailed justification:	No experimental studies on ready or inherent biodegradability of cyfluthrin were performed. Although a STP simulation test is recommended (TNsG 7.02.3.1), any simulation test should at least fulfill the following criteria: • give measured rates for primary and ultimate degradation of the parent compound. • allow for identification and quantification of metabolites formed during the test. The only laboratory EC STP (or the corresponding OECD STP) simulation test currently available is the coupled units test (EC method C.10 or the corresponding OECD test 303A). This test cannot distinguish between biological degradation and other elimination processes such as adsorption and volatilization, and therefore does not fulfill the criteria given above. However Bayer Environmental Science believes that the abiotic and biological degradation demonstrated in the water/sediment studies (IIIA 7.1.2.2.2/01 and /02), together with aerobic aquatic degradation (IIIA 7.1.2.2), radiolabelled hydrolysis (IIIA 7.1.1.1.1/01 and /02) and photo-transformation in water (IIIA 7.1.1.1.2/01 to /02) describe route and rate of both abiotic and biological degradation in natural water systems, thereby giving an indication of biodegradability of imidacloprid in the STP compartment. The water/sediment studies can be regarded as a realistic worst case for the evaluation of the degradation rate in an STP due to the lower microbial activity in the water/sediment system which simulates a natural pond or river. The water/sediment studies showed a comparatively fast degradation and thorough mineralization in all the four different systems tested. In general, the route of aerobic degradation is not expected to be different in a WWTP test compared to that in the natural sediments. An higher tiered study in water sediment (A7_1_2_2_2) is available and provides information on the aerobic biodegradation.	
Undertaking of intended data submission [ ]		

Document IIIA/ Sections 7.1.2.1.1 BPD Data Set IIA/ Annex Point XII.2.1	Aerobic biodegradation
	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	2008/11/02
Evaluation of applicant's justification	Applicant's version is not completely acceptable as we can not follow his argumentation in all points. If the currently in force OECD 303A will be performed with radiolabelled test substance (as mentioned in Annex 7, paragraph 2), rates for primary and ultimate degradation of the parent compound as well identification and quantification of metabolites formed during the test would be possible. Water/sediment systems and sewage treatment plants systems are not comparable and the degradation rates can not be transfered from one system to the other. As no data on degradation in STP are available, for PEC estimation the degradation rate in STP was set to be zero as worst case assumption.
Conclusion	Applicant's justification is acceptable. The degradation rate in STP was set to be zero ($k_{STP} = 0\ h^{-1}$) for PEC estimation as worst case assumption.
Remarks	-
	COMMENTS FROM OTHER MEMBER STATE (specify)
Date	*Give date of comments submitted*
Evaluation of applicant's justification	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Remarks	

Document IIIA/ Section 7.1.2.2.1 — Aerobic Aquatic Degradation

BPD Data set IIIA/ Annex Point XII.2.1

			Official use only
		1 REFERENCE	
1.1	**Reference**	Anderson, C (1986). Degradation of ^{14}C-cyfluthrin in natural water, Bayer AG, Plant Protection Application Technology/CE, Metabolism Research Institute, Monheim, Germany Bayer Report PF 2542 BES Ref: M-073248-01-2 Report date: 26 February 1986 Unpublished	
1.2	**Data protection**	Yes	
1.2.1	Data owner	Bayer CropScience AG	
1.2.2			
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I	
		2 GUIDELINES AND QUALITY ASSURANCE	
2.1	**Guideline s tudy**	No	
2.2	**GLP**	No (not required, as study started before 30 June 1988).	
2.3	**Deviations**	None	
		3 MATERIALS AND METHODS	
3.1	**Test material**		
3.1.1	Lot/Batch number	Not provided	
3.1.2	Specification	[Fluorobenzene –UL-^{14}C]cyfluthrin with a specific radioactivity of 2.35 x 10^6 Bq/mg.	X
3.1.3	Purity	Radiochemical purity of >99%	
3.1.4	Radiolabelling	Radiolabelling was at the fluorobenzene ring of cyfluthrin:  * indicates label position	
3.1.5	UV/VIS absorption spectra and absorbance value	Not applicable	
3.1.6	Further relevant	None	

Document IIIA/ Section 7.1.2.2.1
BPD Data set IIIA/ Annex Point XII.2.1

Aerobic Aquatic Degradation

	properties		
3.2	**Reference substances**	FCR 3191 : FPB-acid FCR 1260 : FPB-aldehyde FCR 3145 : 4'OH-FPB-acid FCR 2728 : COOH-Cyfluthrin FCR 2978 : CONH2-Cyfluthrin R_f values of cyfluthrin and reference compounds on pre-coated TLC plates on different solvent systems are given in Table 7.1.2.2.1-1.	
3.3	**Test solution**	Stock solution A was prepared by dissolving 0.24 mg ^{14}C-cyfluthrin in 11 ml of acetonitrile; 10 ml of this was diluted 1:10 with acetonitrile.	
3.4	**Testing procedure**		
3.4.1	Test system	Test water: Filtered Rhine water (without sediment) Incubation system: Flasks with traps for volatile components Incubation conditions: laboratory, dark, 25 °C Concentration: 0.02 mg a.i./l Sampling 0, 1, 3, 7, 14 and 21 days (see details below) TLC and Co-chromatography of standards: one-dimension on silica gel plates and RP-18 plates, elution solutions are I: Toluene/ diethyl ether/ acetic acid, 100:5:1 II: Toluene/ethyl acetate/acetic acid 50:50:1 III: Methanol/ aceto-nitrile/0.5M NaCl in water 40:40:20 quantitative ^{14}C determination: liquid scintillation counting (fluids)or linear analyzer (plates) Non-radioactive Standards: densitometer	X
3.4.2	Properties of light source	Not applicable	
3.4.3	Determination of irradiance	Not applicable	
3.4.4	Temperature	25±2°C	
3.4.5	pH	Sample / pH at sampling / pH at end of test: Rhine water: 7.7 / 8.3 Sterile filtered Rhine water: 7.7 / 8.6 Rhine water controls: 7.7 / 9.3	
3.4.6	Duration of the test	21 days	
3.4.7	Number of replicates	Two replicated per sampling day, except on last day, when 6 samples	

Document IIIA/ Section 7.1.2.2.1
BPD Data set IIIA/ Annex Point XII.2.1

Aerobic Aquatic Degradation

		were taken.		
3.4.8	Sampling	Day 0 1 3 7 14 21 Flask 1,2 3,4 5,6 7,8 9,10 11, 12, 13, 14, 15, 16		X
3.4.9	Analytical methods	Water sample were extracted twice with dichloromethane. Where the aqueous fraction contained significant radioactivity, this was acidified to pH3 and further extracted with methyl acetate. Organic extracts analysed by thin-layer chromatography (silica gel 60 F_{254} plates, thickness 0.25 mm, and RP-18 $F_{254}S$ plates, thickness 0.25 mm) and developed in solvent systems I and II or III (See 3.2). Quantitation was by a linear analyser with detection of radioactive zones by autoradiography. The non-radiolabelled comparison compounds were detected by densitometer. Trapped CO_2 radioactivity was determined by LS measurement.		X
3.5	**Transformation products**	Transformation products were analysed: FCR 3191(FPB-acid), FCR 3145 (4'OH-FPB-acid), FCR 2728 (COOH-Cyfluthrin), each with its own R_f .: See table A7.1.2.2.1-1		
3.5.1	Method of analysis for transformation products	Thin-layer chromatography, with quantitation by densitometer.		
		4 RESULTS		
4.1	**Screening test**	not performed		
4.2	**Radioactive distribution after degradation in test system**	Radioactive distribution after degradation in test system are given in table 7.1.2.2.1-2		X
4.3	**Mean balance of radioactivity**	$^{14}CO_2$ was not detected during the incubation period. Other volatile compounds (up to 0.6% of the radioactivity) and radioactivity remaining in the aqueous phase after extraction with organic solvents accounted for <3% and were not further investigated. The mean balance of radioactivity was in the range of 79 to 104%		
4.4	**Effects of pH**	At the time of sampling, the Rhine water used had a pH of 7.7. At the end of the investigation the pH of solutions containing cyfluthrin was increased to a value of 8.3. From other experiments it is known that hydrolysis of cyfluthrin is faster under alkaline conditions. However, the acceleration of the hydrolysis of cyfluthrin during the study, which had to be expected due to the increase in pH, was not observed. Instead the degradation of cyfluthrin clearly slowed down. It was assumed that at this time cyfluthrin was adsorbed to a high extent to particles and that only a small amount of cyfluthrin was still		X

present in solution and available for further degradation.

4.5 Half-life

Cyfluthrin degraded rapidly during the first days of incubation under the described conditions. Assuming the reaction follows a pseudo first order kinetics, a **half-life of 4 days** can be calculated from degradation up to day 7.

4.6 Formation and identity of transformation products

FCR 3191 (FPB-acid) occurred at 70% of applied radioactivity. Via ester cleavage, FCR 1260 (FPB-aldehyde) is formed. However, FCR 1260 was not detected, but a separate trial revealed that it is rapidly converted to FCR 3191 (FPB-acid) in the Rhine water. Because the rate and route of degradation were similar in sterile and non sterile samples it is assumed that chemical processes predominate.

FCR 3145 (4'OH-FPB-acid) occurring up to 1.65%, is probably formed by microbial hydroxylation of FCR 3191.

Presence of small amounts of FCR 2728, COOH-Cyfluthrin (Up to 2.25%) indicates that cyfluthrin is also hydrolysed at the nitrile group giving rise to FCR 2728 via FCR 2978.

4.7 Degradation pathway

The proposed degradation pathway is shown below in Figure 7.1.2.2.1-1

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1 Materials and methods

The behaviour of ^{14}C-cyfluthrin in Rhine water was investigated under laboratory conditions. Samples were stored in the dark at 25°C.

Analysis of cyfluthrin and transformation products was by one-dimensional thin-layer chromatography. Quantitation was by a linear analyser with detection of radioactive zones by autoradiography. The non-radiolabelled comparison compounds were detected by densitometer. Trapped CO_2 radioactivity was determined by LS measurement.

5.2 Results and discussion

Cyfluthrin was degraded rapidly under the conditions of the test, A half-life of about 4 days was calculated according to pseudo first order kinetics until day 7, because the degradation clearly slowed down after 7 days. Assuming that the rate of hydrolysis increases linear with pH the degradation in Rhine water occurred more rapidly than indicated by the pH value of the water. This may be due to the presence of various inorganic and organic components in the Rhine water that might have accelerated the degradation of cyfluthrin. On the other hand, the acceleration of the hydrolysis expected from the increase in the pH during the course of the experiment was not observed. Instead the degradation rate decreased after day 7. It was assumed that at this time cyfluthrin was adsorbed to a high extent to fine particles reducing its availability for further degradation processes.

X

Document IIIA/ Section 7.1.2.2.1
BPD Data set IIIA/ Annex Point XII.2.1

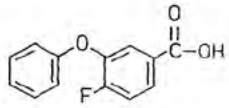
Aerobic Aquatic Degradation

The degradation was predominantly caused by abiotic chemical processes involving ester cleavage.

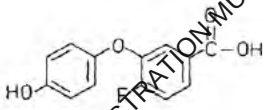
The main end product of degradation was FCR 3191 (FPB-acid), identified by co-chromatography. Small amounts of FCR 3145 (4'OH-FPB-acid), FCR 2728 (COOH-cyfluthrin) and unidentified polar metabolites were found.

Structure of transformation products:

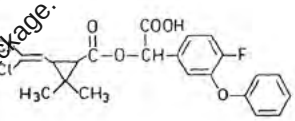
FCR 3191



FCR 3145



FCR 2728



5.3 Conclusion

The halflife of cyfluthrin was approx. 4 days according to pseudo first order kinetics. Under the given experimental conditions, degradation was predominantly abiotic with ester cleavage.

X

5.3.1 Reliability 2

5.3.2 Deficiencies None.

Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	2008/10/10
Materials and Methods	The applicant's version is acceptable apart from the following amendments: 3.1.2: The ratio of the diastereoisomer I, II, III, and IV was 22.7 : 19.7 : 28.8 : 28.8. 3.2: Structures of metabolites are given in Figure 7.1.2.2.1-1. 3.4.1: Incubation system: oil-coated quartz wool plugs were used as traps for volatile metabolites, soda lime was used for $^{14}CO_2$. Incubation conditions: samples were shaken, oxygen content at the end of the study was 80-90% saturation. Concentration: Flasks contained 0.9 % acetonitrile after addition of stock solution. 3.4.8: Flasks 1 – 12 contained test substance, flasks 13 & 14 were blanks (filtered Rhine water), flaks 15 & 16 were sterile controls (sterile filtered Rhine water and test substance) 3.4.9: Radioactivity content of the aqueous phase and the organic phases was determined by liquid scintillation counting (LS). To determine volatile metabolite, the oil-coated quartz wool plugs were extracted with acetic acid, radioactivity in the extract was determined by LS measurement. 5.1: Refer to comments point 3.4.9
Results and discussion	The applicant's version is acceptable apart from the following amendments: 4.2: Autoradiography of the TLC plates revealed 8 further zones in the test substance samples and further 3 zones in the sterile samples, because of low intensity, they were not investigated. Table 7.1.2.2.1-2: Table contains mean values of 2 replicates 4.4: The turbidity of the samples increased considerably with increasing incubation time. Parallel to this, an increase of several orders of magnitude was observed in the microbial count. Therefore it was assumed that cyfluthrin sorbed to the colloids causing turbidity. 4.5: Degradation up to day 7 seemed to be predominantly abiotic. After the first 7 days, the degradation clearly decelerated, maybe because of sorption to colloids.
Conclusion	The applicant's version is acceptable apart from the following amendments: 5.3: Refer to comments point 4.5. The DT_{50} value was re-calculated by RMS according FOCUS degradation kinetics report (2006) using ModelMaker 4.0, SFO model. A DT_{50} (for use as trigger) of 6.3 days was obtained [converted to average EU outdoor temperature of 12°C: DT_{50} = 17.8 days].
Reliability	2
Acceptability	acceptable
Remarks	-

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

Table 7.1.2.2.1-1: Rf values of cyfluthrin and reference compounds on Merck pre-coated TLC plates

Compound	Solvent I	Solvent II	Solvent III
Cyfluthrin	0.87	0.97	0.12
FCR 3191	0.08	0.53	0.47
FCR 1260	0.65	0.94	0.41
FCR 3145	0.01	0.40	0.63
FCR 2728	0.03	0.42	0.20
FCR 2978	0.03	0.53	0.22

I: Toluene/ diethyl ether/ acetic acid, 100:5:1
II: Toluene/ethyl acetate/acetic acid 50:50:1
III: Methanol/ aceto-nitrile/0.5M NaCl in water 40:40:20

Table 7.1.2.2.1-2: Distribution of the radioactivity between cyfluthrin, and its conversion products in the organic phases following the degradation of 14C-cyfluthrin in Rhine water.

Days after application	Cyfluthrin	FCR 3191	FCR 3145	FCR 2728	CO2
0	97.05	<0.1	<0.1	<0.1	< 0.001
1	90.10	8.30	<0.1	0.90	< 0.001
3	65.65	30	0.45	2.25	< 0.001
7	30.95	64.60	1.05	1.80	< 0.001
14	25.3	67.95	1.65	1.30	< 0.001
21	24.35	69.70	0.90	0.70	< 0.001
21(sterile)	20.30	70.10	0.4*		< 0.001

* Sum of FCR3145 and FCR2728.

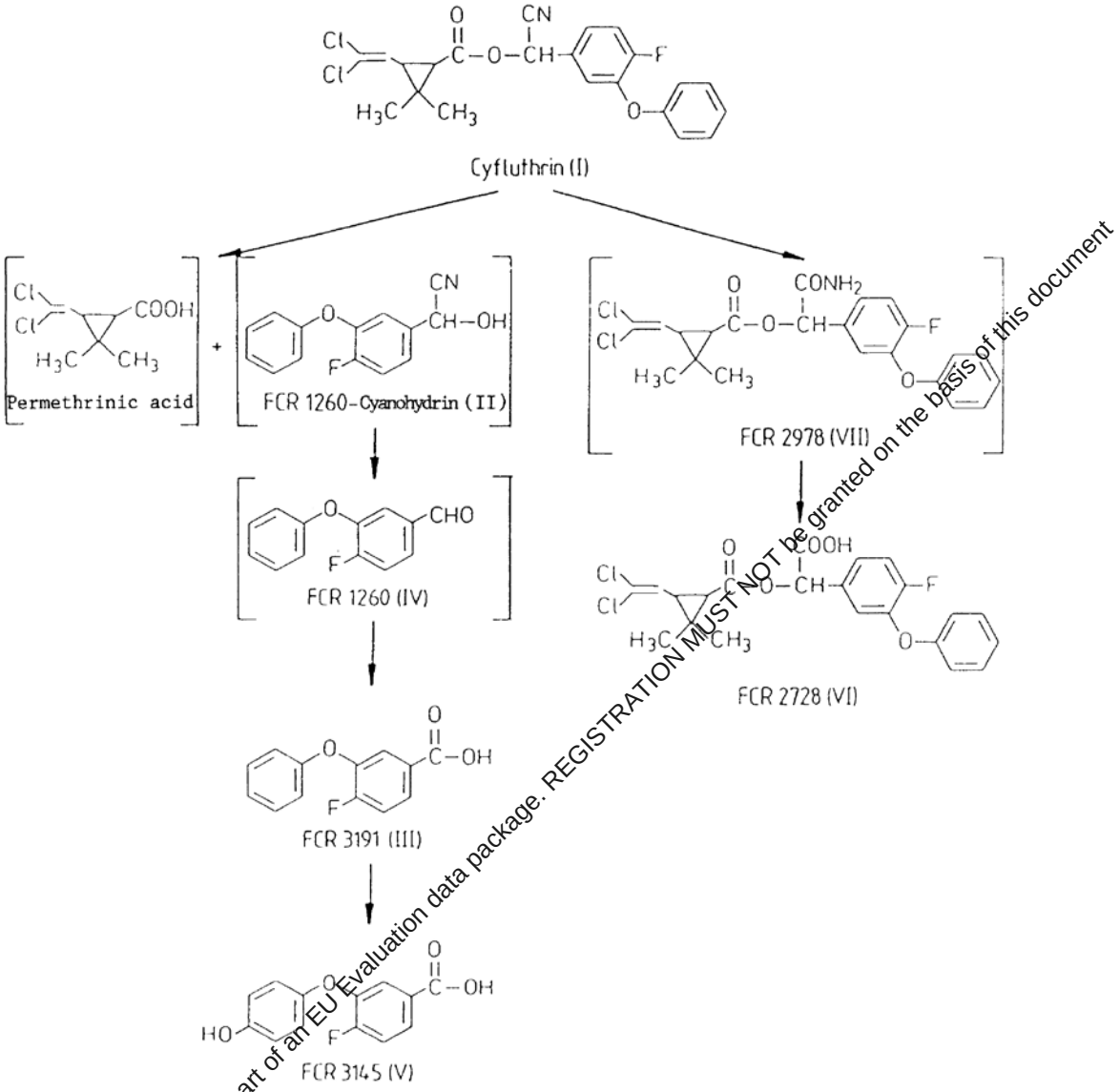


Figure 7.1.2.2.1-1 Degradation Pathway

Doc IIIA/Section A7.1.2.2.2/01 & 02

BPD Data Set IIIA/ Annex Point XII.2.1

Water/Sediment Degradation

Official use only

1 REFERENCE

1.1 Reference

Anderson, C (1987).
Degradation characteristics of cyfluthrin (Baythroid) in water/sediment systems. Bayer AG, Geschäftsbereich Pflanzenschutz Forschung ce Institut für Metabolismusforschung, 5090 Leverkusen.
Bayer Report No.: PF 2875, BES Ref: M-071937-01-2
Report date: 1 October 1987
Unpublished

Hammel, K. (2007)
Kinetic Evaluation of the Degradation of the Cyfluthrin Metabolites CONH2-cyfluthrin and CONH2-FPB-acid in Soil, and FPB-acid, FPB-ald and DCVA in Aquatic Systems. Bayer CropScience
Bayer Report MEF-07/235. BES Ref: M-288629-01-1
Report date: 31 May 2007
Unpublished

1.2 Data protection Yes

1.2.1 Data owner: Bayer CropScience AG

1.2.2

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

2 GUIDELINES AND QUALITY ASSURANCE

2.1 Guideline study Yes.

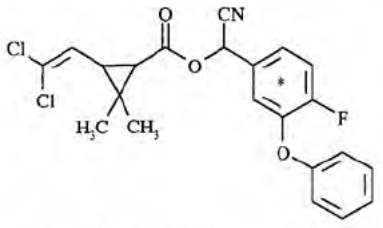
EPA guidelines on "Aerobic aquatic metabolism studies" (§ 162-4) and the requirements of the Dutch authorities. In corresponds with the FAO Annex to revised Guidelines on Environmental Criteria for the Registration of Pesticides.

2.2 GLP No, When the study was performed, GLP was not compulsory (as study started before 30 June 1988).

2.3 Deviations None

3 MATERIALS AND METHODS

3.1 Test material



* indicates label position

3.1.1 Lot/Batch number: The lot number was not stated

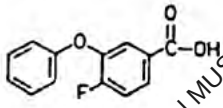
3.1.2 Specification: As described in Section 2.

Doc IIIA/Section A7.1.2.2.2/01 & 02
BPD Data Set IIIA/ Annex Point XII.2.1

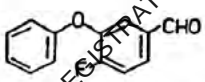
Water/Sediment Degradation

3.1.3	Purity	radioactive purity >98%	
3.1.4	Radiolabelling	Radiolabelled cyfluthrin, [Fluorobenzene-UL-^{14}C]cyfluthrin	
3.1.5	UV/VIS absorption spectra and absorbance value	none	X
3.1.6	Further relevant properties	Specific activity of 2.35 mBq/mg and was used.	X
3.2	**Reference substances**	Reference standards of possible transformation products were prepared: FCR 3191, FCR 1260, FCR 3145, FCR 2978, FCR 2728, FCR 2947, and FCR 1261. The structures are as follow:	X

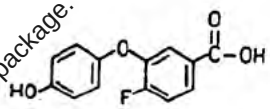
FCR 3191
FPB-acid



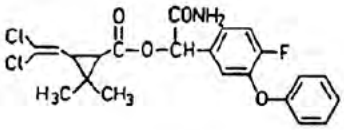
FCR 1260
FPB-ald



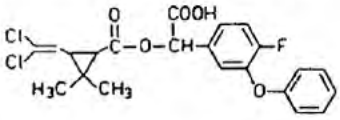
FCR 3145
4'OH-FPB-acid



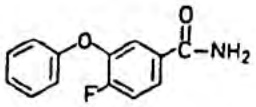
FCR 2978
COOH-Cyfluthrin



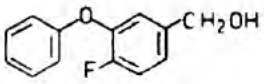
FCR 2728 CONH2-Cyfluthrin



FCR 2947
FPB-amide



FCR 1261



3.3	**Test solution**	Stock solution A: 0.1 g/ml of analytical standard in acetonitrile Stock solution B: 0.06 mg/ml of radiolabelled cyfluthrin in acetonitrile. (radiochemical purity = >98%) Reference solutions for TLC: 1 mg of each of FCR 3191, FCR 1260, FCR 3145, FCR 2978, FCR 2947, and FCR 1261, in 1 ml of acetonitrile. Stock solution K: 30 µL of stock solution A diluted to 50 ml with acetonitrile (0.06 mg/ml)	X

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3.4	Testing procedure	Criteria	Details	
3.4.1	Test system	Water/sediment source		
		Sample from an orchard drainage ditch at IJzendoorn (NL), loamy sand, pH 6.8, 0.51 % organic C		X
		Sample from a fishpond at Lienden (NL), loamy sand, pH 7.8, 1.08 % organic C		
		The test system contained 10% w/w sediment		
3.4.2	Temperature, pH	Darkness, 22°C		
3.4.3	Concentration	12 µg a.i./L		X
3.4.4	Duration of test	70 days		
3.4.5	Sampling	1) Ijzendoorn system: 2 samples taken after incubation periods of 1, 11, 28 days; 3 samples after 40 days and 4 samples after 70 days.		X
		2) Lienden system: 2 samples taken after incubation periods of 1, 11, 22 days; 3 samples after 40 days and 5 samples after 70 days		
3.4.6	Method of Analysis	The 1 day water samples were extracted with hexane but all subsequent time points ere extracted with ethyl acetate. The sediment was initially extracted with methanol. It was then extracted with HCl under reflux conditions, enriched with XAD and eluted with methanol. Organic extracts were then analysed. TLC analyses on one-dimension silica gel plates (layer thickness 0.25 mm); co-chromatography of standards.		X
		Solvent systems for TLC:		
		I: silica gel 60 F-254, toluene/ethyl acetate/acetic acid (50:50:1)		
		II: RP-18 F-254, methanol/acetonitrile/ 0.5 M NaCl (40:40:20)		
		III: silica gel 60 F-254, chloroform/ methanol (90:10)		
		Solvent system II was used for the quantitative determination of cyfluthrin and systems I and III for FCR 3191. FCR 1260 was determined by difference. FCR 2978 was determined using system I.		
		^{14}C determination and quantification :		
		- Solid samples: combustion to $^{14}C0_2$;		
		- Liquid samples: liquid scintillation counting.		
		- TLC: The distribution of radioactivity on the TLC plates was determined by measurement with linear analysers. Non-labelled standards were located by fluorescence quenching under a UV lamp or with a densitometer.		
3.4.7	Statistics	Kinetics evaluation of relevant metabolites has been conducted (Hammels, 2007).		
		Due to the scarcity of the experimental data the simple first-order (SFO) model was considered as only appropriate model.		
		The goodness of fit is assessed by visual inspection and a error criterion based on a chi-square (χ^2) significance test. The visual inspection focuses on the residuals which should not be distributed systematically but randomly.		

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		However in the case of systematic but sufficiently small deviations a fit is still qualified as visually acceptable. The χ^2 significance test which is described in the following evaluates the likeliness that a given model is a correct description of the values observed. Traditionally the coefficient of determination r2 was used to assess the goodness of fit.	
3.5	**Transformation products**	The following transformation products were tested by thin-layer chromatography: FCR 3191, FCR 1260, FCR 3145, FCR 2978, FCR 2728, FCR 2947, and FCR 1261. The individual zones were located by fluorescence quenching under a UV lamp or with a densitometer. Radioactive zones were visualised by taking autordiographs.	
		4 RESULTS	
4.1	**Distribution of radioactivity in water and soil sediment systems**	Table A7.1.2.2.2/01-1 summarises the distribution of radioactivity in water/sediment systems for cyfluthrin. The distribution of the radioactivity was very similar in the two systems. The radioactivity present in the aqueous phase decreased sharply;1 day after application it was already reduced to 20 % - 30 % of the amount applied and declined to < 2 % within 40 days. The corresponding amounts in the sediment at day 1 were 68 % to 75 %. At the end of the study the figures were 25 % - 32 %. During the course of the study the extractability of this portion decreased steadily. At the end of the experiment about 90 % of the radioactivity of the sediment (21 % - 29 % of applied) could not be extracted with methanol. Both systems were characterised by a very high degree of mineralisation. After 10 weeks more than 60 % of the applied radioactivity was found as $^{14}CO_2$	X
4.2	**Water/sediment degradation**	Cyfluthrin is translocated very rapidly from the aqueous phase into the sediment and degraded. After an incubation time of 1 day, only 13 % - 20 % of the applied cyfluthrin was still detectable. Ester cleavage and subsequent oxidation led to the formation of FCR 1260 and of the main metabolite FCR 3191. In addition FCR 2728, FCR 2978 and FCR 3145 were formed. There were also some unknown compounds but none of them ever reached a level of 10 % of the applied radioactivity. Table A7.1.2.2.2/01-2 summarises the degradation of cyfluthrin in two water/sediment systems.	X
4.3	**Degradation pathway**	The proposed metabolic pathway is shown below in Fig 7.1.2.2.2/01-1	
		5 APPLICANT'S SUMMARY AND CONCLUSION	
5.1	**Materials and methods**	The degradation of cyfluthrin in water/sediment systems was investigated in a laboratory study using radiolabelled active substance. The water and sediment samples came from an orchard drainage ditch (IJzendoorn) and a fishpond (Lienden) in the Netherlands. The systems were treated with 0.012 mg active substance/litre, based on the solubility of cyfluthrin in the water used. Samples, including reference standards, were analysed by TLC on one-dimension silica gel plates (layer thickness 0.25 mm). Radioactive zones on the TLC plates were visualised by taking auto radiograms. Non-labelled standards were located by fluorescence quenching under a UV lamp or with	X

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a densitometer. For solid samples, radioactivity was determined and quantified by combustion to $^{14}CO_2$ while for liquid samples, determination was made by liquid scintillation counting.

5.2 Results and discussion

Cyfluthrin is translocated very rapidly from the aqueous phase into the sediment and degraded. The radioactivity present in the aqueous phase decreased sharply; one day after application it was already reduced to 20 % to 30 % of the amount applied and declined to < 2 % within 40 days.

The corresponding amounts in the sediment at day 1 were 68 % to 75 %. At the end of the study the figures were 25 % - 32 %. During the course of the study the extractability of this portion decreased steadily; at the end of the experiment about 90 % of the radioactivity of the sediment (21 % - 29 % of applied) could not be extracted with methanol.

Both systems were characterised by a very high degree of mineralisation. After 10 weeks more than 60 % of the applied radioactivity was found as $^{14}CO_2$

Metabolites were formed by ester cleavage and subsequent oxidation. The main metabolite was FCR 3191. FCR 1260, FCR 2728, FCR 2978 and FCR 3145 were formed. There were also some unknown compounds but none of them ever reached a level of 10 % of the applied radioactivity. The proposed degradation pathway is shown below.

DT_{50} and DT_{90} were calculated and normalized to 12°C (Hammel, 2007) for FPB-acid (FCR 3191) and FPB-ald (FCR 1260) using the equation $DT50_2 = DT50_1 \cdot Q_{10}^{\frac{T_2 - T_1}{10}}$, where Q10 = 2.2 as given in FOCUS (2000). Detailed results are given in Table A7.1.2.2.2/01-3

X

5.3 Conclusion

Regardless of the source of the sludge, the active ingredient was very rapidly degraded in the total system. After an incubation period of 1 day, on average only 13 to 20 % of the applied radioactivity was still detectable as unchanged parent compound.

X

Using the equation for normalisation to 12°C (1), the DT_{50}(12°C) of cyfluthrin is 0.5 days and 0.8 days in the orchard drainage ditch (IJzendoorn) and the fishpond (Lienden) respectively

(1) $DT_{50}(12°C) = DT_{50}(T) \times e^{(0.08 \times (T-12))}$

5.3.1 Reliability: 1

5.3.2 Deficiencies: None

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Water/Sediment Degradation

Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	2008/10/14
Materials and Methods	The applicant's version is acceptable apart from the following amendments: 3.1.4: No information about the ratio of the diastereoisomers. 3.1.6: Specific activity was 2.35 x10^6 Bq/mg 3.2: Cyfluthrin (non-labelled, also called FCR 1272) was used as well. 3.3: Stock solution A contained non-labelled cyfluthrin. A reference solution of FCR 2728 was prepared as well. 3.4.1: Incubation system: Flasks with traps for volatile components and $^{14}CO_2$; oil-coated quartz wool plugs were used as traps for volatile metabolites, soda lime was used for $^{14}CO_2$. 3.4.3: Flasks contained <0.1 % acetonitrile after addition of stock solutions. 3.4.5: Two flasks containing radiolabelled cyfluthrin (each sampling day), one flask containing radiolabelled cyfluthrin and additional non-labelled cyfluthrin (day 40 and 70), one blank (day 70), one flask with non-labelled cyfluthrin (day 70). 3.4.6: Aqueous phase: Extraction with n-hexane (day 1) or with methyl acetate (the following sampling days). Volatile basic compounds were collected after addition of HCl to an aliquot of aqueous phase. During vacuum filtration, CO_2 was collected. Sediment after extraction was dried and combusted (automatic oxidizer followed by liquid scintillation counting (LS)). To determine volatile metabolites, the oil-coated quartz wool plugs were extracted with acetic acid. Trapped CO_2 was released by HCl addition to the soda lime and absorbed in ethanolamine/methanol. Radioactivity was determined by LS. 5.1: Refer to comments point 3.4.6.
Results and discussion	The applicant's version is acceptable apart from the following amendments: 4.1: In individual cases, the results of two replicates varied by more than 50%. 4.2: Data were missing in Table A7.1.2.2.2/01-2, a corrected version was attached to the end of the present document (see Annex 1, CA-Table 1). In individual cases, the results of two replicates of sediment extract varied by more than 50 %. The results of the sediment extract represent the methanol extract. In HCl extracts only small amounts of FCR 3191 and FCR 3145 were observed together with some unknown compounds of similar polarity. 5.2: Normalization to 12°C of half-lives of metabolites according to TGD results in slightly different values, a corrected version of Table A7.1.2.2.2/01-3 was attached to the end of the present document (see Annex 1, CA-Table 2).
Conclusion	The applicant's version is acceptable apart from the following amendments: 5.3: After an incubation period of 1 day, the results of two replicates varied by more than 50% (IJzendoorn A: 8.9%, B: 31.9%, Lienden A: 21.8%, B: 4.3%).

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	The calculation of the DT_{50} values mentioned (cyfluthrin, total system) was not presented in the study. The DT_{50} values were re-calculated by RMS according to FOCUS degradation kinetics report (2006) using ModelMaker 4.0, FOMC model. For Ijzendoorn system, a DT_{50} (for use as trigger) of 3.3 days was obtained [converted to average EU outdoor temperature of 12°C: DT_{50} = 7.3 days], for Lienden system, the DT_{50} was 1.95 days [converted to average EU outdoor temperature of 12°C: DT_{50} = 4.3 days].
Reliability	2
Acceptability	acceptable
Remarks	-

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

Table A7.1.2.2.2/01-1 **Behaviour of [fluorobenzene-UL-14C]cyfluthrin (12 ug/1) in two water/sediment systems: radioactivity balance in % of the applied radioactivity (mean values of two separate samples)**

Ijzendoorn	Days after application				
	1	11	28	40	70
Aqueous Phase	20.30	35.6	0.65	<0.1	<0.1
- Dissolved $^{14}CO_2$	0.75	4.1	7.0	5.05	3.1
Sediment					
- Methanol extract	67.00	25.8	10.9	7.45	3.85
- HCL-extract	1.40	12.75	18.6	19.65	16.90
- Bound $^{4}CO_2$	0.05	0.95	1.40	1.50	1.05
- Not extracted	6.50	8.35	21.60	16.65	11.65
- Total (sediment)	74.95	47.85	52.5	45.25	33.45
Volatile components	<0.01	<0.01	<0.01	<0.01	<0.01
- $^{14}CO_2$	0.05	9.15	37.1	44.85	57.15
Total radioactivity recovered	**96.0**	**96.7**	**97.25**	**95.15**	**93.7**
Lienden	**Days after application**				
	1	**11**	**23**	**40**	**70**
Aqueous Phase	30.25	11.1	0.95	1.8	0.9
- Dissolved $^{14}CO_2$	2.0	17.0	4.05	5.25	3.75
Sediment					
- Methanol extract	61	10.55	10.45	4.35	2.9
- HCL-extract	1.7	17.7	16.05	17.15	11.05
- Bound $^{4}CO_2$	0.03	0.8	0.7	1.2	1.35
- Not extracted	5.9	15	17.35	13	9.65
- Total (sediment)	68.63	53.05	44.55	35.7	24.95
Volatile components	<0.01	<0.01	<0.01	<0.01	<0.01
- $^{14}CO_2$	0.1	12.9	43.25	54.35	61.9
Total radioactivity recovered	100.9	**94.05**	**96.8**	**97.1**	**91.5**

Table A7.1.2.2.2/01-2 Degradation of ^{14}C-cyfluthrin in two water/sediment systems: distribution of metabolites at different sampling times

Location/ Phase	Days after Appl.	% applied radioactivity					
		FCR 1272	COE 538/ 78	FCR 1260	FCR 3145	FCR 2728	FCR 2978
Ijzendoorn	1	0.35	11.6	n.d.	n.d.	0.15	-
Water	11	<1.1	29.1	n.d.	n.d.	n.d.	-
	28	0.15	0.5	0.1	0.15	n.d.	-
	70	n.d.	n.d.	n.d.	n.d.	n.d.	
Ijzendoorn	1	20.1	16.45	15.65	n.d	3.9	4.55
Sediment-extract	11	6.3	15.4	<2.2	2.5	n.d.	n.d.
	28	1.1	1.75	1.35	<2.0	n.d.	2.6
	70	1.1	0.65	0.35	n.d.	n.d.	n.d.
Lienden	1	0.5	11.35	1.1	-	0.4	-
Water	11	n.d.	11.7	n.d.	-	n.d.	-
	70	n.d.	n.d.	n.d.	-	n.d.	-
Lienden	1	12.55	24.3	8.4	-	-	4.7
Sediment-extract	11	3.45	3.75	6.1	-	-	2.05
	70	0.85	0.5	0.25	-	-	n.d.

n.d. not detected

FCR 1272: cyfluthrin
COE 538/78 3-phenoxy-4-fluoro-benzoic acid (FPBacid)
FCR 1260 3-phenoxy-4-fluoro-benzaldehyde (FPBald)
FCR 3145 4'-OH-FPBacid
FCR 2728 COOH-cyfluthrin
FCR 2978 Alpha-carbamoyl-(4-fluoro-3-phenoxyphenyl)-methyl-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate (CONH2-cyfluthrin)

Table A7.1.2.2.2/01-3 DT_{50} and DT_{90} (total system)

Metabolites	System	DT_{50} (days)	DT_{90} (days)
FPB-acid	Ijzendoorn	8.8	29.3
FPB-acid	Lienden	17.8	59.3
FPB-ald	Ijzendoorn	7.3	24.1
FPB-ald	Lienden	22	22

Annex 1 Evaluation by Rapporteur Member State, CA-Tables

CA-Table 1 (revised 7.1.2.2.2/01-2): Degradation of ^{14}C-cyfluthrin in two water/sediment systems: distribution of metabolites at different sampling times

Location/ Phase	Days after Appl.	% applied radioactivity					
		Cyfluthrin	FCR 3191	FCR 1260	FCR 3145	FCR 2728	FCR 2978
Ijzendoorn	1	0.35	11.6	n.d.	n.d.	0.15	n.d.
Water	11	<1.1	29.1	n.d.	n.d.	n.d.	n.d.
	28	0.15	0.5	0.1	0.15	n.d.	n.d.
	40	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	70	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Ijzendoorn	1	20.1	16.45	15.65	n.d	3.9	4.55
Sediment-extract	11	6.3	15.4	<1.2	2.5	n.d.	n.d.
	28	1.1	1.75	1.35	<2.0	n.d.	2.6
	40	4.6	0.75	0.9	n.d.	n.d.	n.d.
	70	1.1	0.65	0.35	n.d.	n.d.	n.d.
Lienden	1	0.5	11.35	1.1	n.d.	0.4	n.d.
Water	11	n.d.	11.7	n.d.	n.d.	n.d.	n.d.
	22	n.d.	1.95	n.d.	n.d.	n.d.	n.d.
	40	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	70	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Lienden	1	12.55	24.3	8	n.d.	n.d.	4.7
Sediment-extract	11	3.45	3.75	0.1	n.d.	n.d.	2.05
	22	3.55	2.85	0.55	n.d.	n.d.	0.65
	40	1.55	0.45	1.55	n.d.	n.d.	0.25
	70	0.85	0.5	0.25	n.d.	n.d.	n.d.

n.d.	not detected
FCR 3191	3-phenoxy-4-fluoro-benzoic acid (FPBacid, COE 538/78)
FCR 1260	3-phenoxy-4-fluoro-benzaldehyde (FPBald)
FCR 3145	4'-OH-FPBacid
FCR 2728	COOH-cyfluthrin
FCR 2978	Alpha-carbamoyl-(4-fluoro-3-phenoxyphenyl)-methyl-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate (CONH2-cyfluthrin)

CA-Table 2 (revised 7.1.2.2.2/01-3): DT_{50} and DT_{90} values at test temperature and normalized to a temperature of 12 °C

Metabolites	System	DT_{50} (days) 22°C	DT_{50} (days) 12°C	DT_{90} (days) 22°C
FPB-acid	Ijzendoorn	4.0	8.9	13.3
FPB-acid	Lienden	8.1	18.0	26.9
FPB-ald	Ijzendoorn	3.3	7.3	11.0
FPB-ald	Lienden	10.0	22.3	33.3

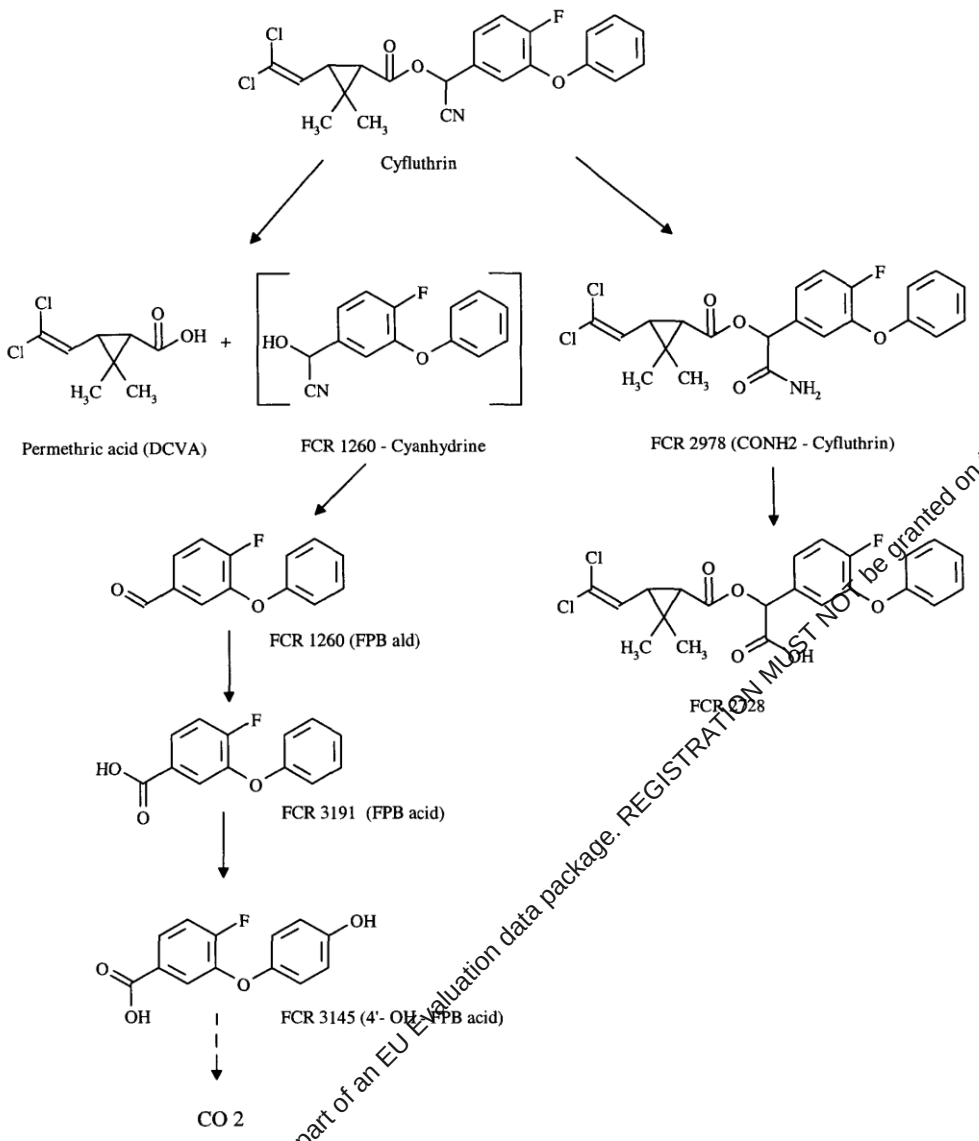

Figure A7.1.2.2.2/01-1 Degradation Pathway

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			Official use only
		1 REFERENCE	
1.1	**Reference**	Sneikus, J (2000). Aerobic aquatic degradation and metabolism of Cyfluthrin in water/sediment system, Bayer AG Crop Protection-Development, Institute for Metabolism Research, and Residue Analysis, D-51368 Leverkusen, Germany Bayer Report No.: MR 268/00, BES Ref: M-022319-02-1 Report date: 15 September 2000 Unpublished Hammel, K. (2007) Kinetic Evaluation of the Degradation of the Cyfluthrin Metabolites $CONH_2$-cyfluthrin and CO NH_2-FPB-acid in Soil, and FPB-acid, FPB-ald and DCVA in Aquatic Systems. Bayer CropScience Bayer Report MEF-07/235. BES Ref: M-288629-01-1 Report date: 31 May 2007 Unpublished	
1.2	**Data protection**	Yes	
1.2.1	Data owner	Bayer CropScience AG	
1.2.2			
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I	
		2 GUIDELINES AND QUALITY ASSURANCE	
2.1	**Guideline study**	Yes. BBA Guidelines for Testing of Plant Protectants in the Registration Process, Part IV, 5-1, Degradability and Fate of Plant Protectants in the Water/Sediment System, 1990 Commission Directive 95/36/EC, Placing Plant Protection Products on the Market: Official Journal of the European Communities, 14 July 1995 SETAC –European Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides, March 1995.	
2.2	**GLP**	Yes German Chemical Law, dated July 24, 1994: current version of attachment 1 and the current OECD Principle of Good Laboratory Practice, November 26, 1997 [C(97) 186/Final]	
2.3	**Deviations**	None	
		3 MATERIALS AND METHODS	
3.1	**Test material**	Radiolabelled cyfluthrin, [cyclopropane-1-^{14}C] cyfluthrin (ID TH 5108, Lot no.: 13050/2), with a specific activity of 1.55 mBq/mg and radiochemical purity >98% (sum of isomers) was used.	X

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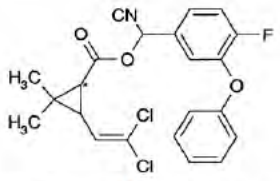
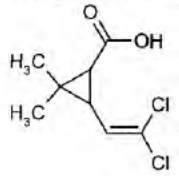


* indicates ^{14}C - position

3.2 Reference substances

Cyfluthrin analytical standard (non-radiolabelled) Purity 98,9% (cis 51,1%/trans 47,8%), and the 4 diastereoisomers, each with a purity >98%; X

Permethrinic acid (DCVA, 3-(2,2-dichlorovinyl) 2,2-dimethylcyclopropane carboxylic acid, with purity >98%. The structure is:



3.3 Test solution

Based on the recommended maximum single use rate of cyfluthrin is 25 g ai/ha, which translates to a concentrated volume of approx. 8.3 µg ai/l water. An aliquot containing 3.7 µg 14C-cyfluthrin was applied onto each vessel and after dilution to 450 ml, the actual concentration was 8.14 µg ai/l.

3.4 Testing procedure

3.4.1 Test system
3.4.2 Temperature, pH
3.4.3 Duration of test
3.4.4 Light source
3.4.5 Sampling
3.4.6 Method of analysis
3.4.7 Statistics

Water/sediment

1.)Water and sediment system collected from a small disused gravel-pit at Barmener See (Jülich, Germany), sand, pH 7.5, 0.48% org. C X

2.)Water and sediment system collected from a catchment basin in the course of the "Genkel Creek" at Genkel (Meinerzhagen, Germany), silt loam, pH 5.0, 4.91 % org. C

Each system was preincubated.

Characterisation of the water/sediment system is given in table 7.1.2.2.2/03-1

Test conditions:

Test conducted in an aquatic model ecosystem consisting of sediment and accompanying supernatant water (1:9 w/w) under aerobic conditions in the laboratory at a temperature of 20.3 ± 0.9°C, in the dark. X

Test system

Incubation flasks with trap attachments to collect CO_2 and other volatile metabolites. The trap attachments were permeable for oxygen, however, allowed to absorb CO_2 by soda lime and volatile metabolites by polyurethane foam. X

Concentration applied/ test system: 8.14µg ai/l corresponding to 25 g ai/ha.

Sampling:

0.5, 3 and 6 hours as well as 1, 2, 3, 7, 10, 14, 28, 56, 100 days post treatment

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Water/Sediment Degradation

	Processing of supernatant water: A small amount of supernatant water (50 ml) was decanted, adjusted to alkaline PH with 0.1N NaOH and used for determination of dissolved CO_2. The remaining supernatant was decanted and centrifuged. The aqueous phase was acidified and extracted with dichloromethane. The organic phase was concentrated, the residue taken up in isopropanol and aliquots used for TLC.	X
	Processing of sediment The sediment underwent a series of extraction with acetonitrile, filtration, and centrifugation, combining the extracts. A number of extractions with dichloromethane followed. The combined extracts were evaporated to dryness and the residue taken up in isopropanol for use in TLC. The sediment residue was allowed to air dry, then homogenized in a planet mill and weighed. Five portions were radioassayed by LSC.	X
	Identification TLC analyses on one-dimension RP-18 plates (layer thickness 0.25 mm); co-chromatography of standards. The radioactive zones on the TLC plates were visualised by digital autoradiography. Non-labelled standards were located by fluorescence quenching under a UV lamp.	
	^{14}C determination and quantification - Solid samples: combustion to $14CO_2$. - Liquid samples: liquid scintillation counting. At selected sampling intervals, extracts were analysed by normal phase HPLC and UV detection to determine ratio of diastereomers.	X
	Kinetics evaluation of relevant metabolites has been conducted (Hammels, 2007). Due to the scarcity of the experimental data the simple first-order (SFO) model was considered as only appropriate model. The goodness of fit is assessed by visual inspection and a error criterion based on a chi-square (χ^2) significance test. The visual inspection focuses on the residuals which should not be distributed systematically but randomly. However in the case of systematic but sufficiently small deviations a fit is still qualified as visually acceptable. The χ^2 significance test which is described in the following evaluates the likeliness that a given model is a correct description of the values observed. Traditionally the coefficient of determination r2 was used to assess the goodness of fit.	X
3.5 Degradation products	The main degradation product was permethrinic acid (DCVA) in both the supernatant water and the sediment. It was found with a maximum of 40% (Barmener See, day 2) and 48% (Genkel, day 28). The identity of permethrinic acid was confirmed by HPLC-MS, comparing the spectra with reference standards.	

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Water/Sediment Degradation

4 RESULTS

4.1 Distribution of radioactivity in water /soil sediment systems

Tables 7.1.2.2.2/03-2 and 7.1.2.2.2/032-3 summarise the behaviour of ^{14}C cyfluthrin and its metabolites in two water/sediment systems. One day after application the radioactivity in the water phase was already reduced to 40 % (Barmener See) or 26 % (Genkel) of the amount applied and stayed at this level within 100 days. The corresponding amounts in the sediment at day 1 were 53-73 %. At the end of the study the figures were 28-70 %. During the course of the study the extractability of this portion decreased steadily. At the end of the experiment 12-26 % of the applied radioactivity could not be extracted with acetonitrile and dichloromethane. X

4.2 Water/sediment degradation

Both systems were characterised by a high degree of mineralisation. After 100 days 37 % (Barmener See) and 14 % (Genkel) of applied radioactivity was found as $^{14}CO_2$.

4.3 Degradation products

The main degradation product was permethrinic acid (DCVA) in both the supernatant water and the sediment. It was found with a maximum of 40% (Barmener See, day 2) and 48% (Genkel, day 28). By study termination the content of permethrinic acid in the total system decreased to about 34% (Barmener See) and 35% (Genkel). There were also some unknown compounds but none of them ever reached a level of 10 % of the applied radioactivity.

4.4 Half-life (DT_{50})

The results showed that cyfluthrin was quickly eliminated from the water body, either via trans-location into the sediment or via degradation. The calculated DT_{50} values of cyfluthrin for the supernatant water phase were 2.4 and 3.8 hours (DT_{90} = 26.6 -41.7 hr) and for the total water/sediment system, 2.5-3.5 days (DT_{90} = 56.6 -66.8 day) for the Barmener See and Genkel systems, respectively.

Using the equation for normalisation to 12°C (1), DT_{50}(12°C) of cyfluthrin for the total water/sediment system were 4.7 days and 6.6 days for the Barmener See and Genkel systems, respectively while DT_{90}(12°C) were 107.3 days and 130.5 days for the Barmener See and Genkel systems, respectively. X

(1) $DT_{50}(12°C) = DT_{50}(T) \times e^{(0\,08\times(T-12))}$

DT_{50} and DT_{90} were evaluated and normalized to 12°C (Hammel, 2007) for DCVA in Genkel system only (DT_{50} = 304.8 days & DT_{90} = 1012.3 days). Values in Barmener See system cannot be evaluated due to excessive scatter. X

See table 7.1.2.2.2/03-4

4.5 Effect on isomer ratio

The degradation of cyfluthrin had no significant influence to the ratio of diastereomers. The ratio of the biological active diastereomers was nearly constant. X

4.6 Degradation pathway

The degradation pathway is shown below (Figure 7.1.2.2.2/03-1)

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Water/Sediment Degradation

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1	**Materials and methods**	The degradation and behaviour of [cyclopropane-1-^{14}C] cyfluthrin and its metabolites was investigated in two different water/sediment systems (Barmener See and Genkel) without plants. After dosing about 3.7 µg ai/ test vessel (corresponding to a direct spray of 25 g ai/ha), the test systems were incubated at 20°C under darkness for a maximum of 100 days. Aerobic conditions were maintained throughout the study in the supernatant water and relevant boundary layer between water and sediment. At each sampling interval the water and sediment phases were analysed by thin-layer chromatography (TLC). The ratio of the diastereomers of cyfluthrin in specimen samples was determined using normal phase HPLC. The content of radioactivity (RA) was determined by liquid scintillation measurement.	X
5.2	**Results and discussion**	The complete material balances found at all individual sampling intervals demonstrate that no relevant amount of RA dissipated from the systems during the entire testing period. During the entire study period a high formation of $^{14}CO_2$ was observed in the water-sediment systems used. Cyfluthrin was quickly eliminated from the water body, either via translocation into the sediment or via degradation. The calculated DT_{50} values of cyfluthrin for the supernatant water phase were 2.4 and 3.8 hours for the two systems respectively. The main degradation product was permethrinic acid (DCVA) in both the supernatant water and the sediment. There were also some unknown compounds but none of them ever reached a level of 10 % of the applied radioactivity. The degradation of cyfluthrin had no significant influence to the ratio of diastereomers. The ratio of the biological active diastereomers was nearly constant.	X
5.3	**Conclusion**	Cyfluthrin can be regarded as a well dissipating compound from the water phase of a water-sediment system. Cyfluthrin will be substantially and thoroughly degraded in an aquatic environment. The rate of total mineralisation in both systems is very high and there is no potential for persistence or accumulation of cyfluthrin in the aquatic environment. The half-lives of cyfluthrin in the total water/sediment system are estimated to be only 2.5 - 3.5 days and in the water phase, 2.4 - 3.8 hours. Normalized DT_{50} and DT_{90} to 12°C are summarized in table 7.1.2.2.2/03-4 Furthermore the degradation of cyfluthrin has no significant influence to the ratio of diastereomers. The ratio was more or less constant. The main degradation product is permethric acid (DCVA). The proposed degradation pathway is shown below. (Figure 7.1.2.2.2/03-1)	X
5.3.1	Reliability	1	
5.3.2	Deficiencies	None	

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Water/Sediment Degradation

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	Evaluation by Rapporteur Member State
Date	2008/10/16
Materials and Methods	The applicant's version is acceptable apart from the following amendments: 3.1: Specific activity was 1.55 x10^6 Bq/mg. The ratio of the diastereoisomer of radiolabelled cyfluthrin was I : II : III : IV = 26.6 : 18.7 : 32.5 : 22.2. 3.2: The ratio of the diastereoisomer of non-radiolabelled cyfluthrin was I : II : III : IV = 23.7 : 17.1 : 34.8 : 23.4. 3.4: Systems were pre-incubated for 4 weeks. Test conditions: water:sediment ratio 1:9 (w/w; wet weight:dry weight). Test system: a biometer apparatus was used. Processing of supernatant water: radioactivity in water was analysed by liquid scintillation counting (LSC) before and after organic extraction. Radioactivity in the organic extract was determined by LS, as well. Processing of sediment: After three acetonitrile extractions, a single dichloromethane extraction was done. Radioactivity was determined separately in acetonitrile (pooled) and dichloromethane extracts. TLC analyses were done separately as well. To determine volatile metabolites, the polyurethane plugs were extracted with ethylacetate. Trapped CO_2 was released by HCl addition to the soda lime and absorbed. Radioactivity was determined by LSC measurement. To determine ratio of diastereomers, water and sediment extracts were analysed using HPLC, UV detection and LSC. Kinetics evaluation: DT_{50} value of cyfluthrin was determined for water phase (using cyfluthrin content in water phase and 1st order function) and for the total system (using 1.5th or 2nd order function). 5.1: Refer to comments point 3.4.
Results and discussion	The applicant's version is acceptable apart from the following amendments: 4.1: Table 7.1.2.2.2/03-2: some values need to be corrected: Barmener See, 3 hours, sediment: 66.11%; sediment not extracted: 3.21% Barmener See, day 10, $^{14}CO_2$: 7.94% is the correct value. Table 7.1.2.2.2/03-3: some values need to be corrected: Barmener See, 3 hours, M1: 0.51 %. Barmener See, day 10, $^{14}CO_2$: 7.94%. Genkel, day 7, M3: 0.11%. Genkel, day 10, M3: 1.06%. 4.4: Cyfluthrin (total system, Genkel): DT_{80} was calculated instead of DT_{90}, the DT_{80} (20°C) was 56.6 days. Cyfluthrin (total system, Barmener See): the DT_{90} (20°C) was 66.8 days DCVA (total system, Genkel): the calculated DT_{50} (20°C) was 162.2 days (DT_{90}

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	(20°C) = 538.8 days), after conversion to average EU outdoor temperature according to TGD: DT_{50} (12°C): 307.6 days. Table 7.1.2.2.2/03-4 needs to be corrected (see comments above). 4.5: The ratio of diastereomers was more or less constant in the water extracts (determination up to day 1). In the sediment extracts the ratio changed from I : II : III : IV = 27.5 : 21.4 : 30.4 : 20.8 (day 0) to 49.5 : 29.5 : 11.8 : 9.3 (day 100, Barmener See) and 48.3 : 27.7 : 13.7 : 10.4 (day 100, Genkel). 5.2: Refer to comments point 4.5.
Conclusion	The applicant's version is acceptable apart from the following amendments: 5.3: Refer to comments point 4.1, 4.4 and 4.5. The DT_{50} values of cyfluthrin (total system) were re-calculated according to FOCUS degradation kinetics report (2006) (for use as trigger) by RMS using ModelMaker 4.0 and FOMC model. For Barmener See system, a DT_{50} (20°C) of 2.5 days was obtained [converted to average EU outdoor temperature of 12°C: DT_{50} = 4.7 days], for Genkel system, the DT_{50} (20°C) was 4.9 days [converted to average EU outdoor temperature of 12°C: DT_{50} = 9.3 days]. The DT_{50} values of DCVA (total system) were re-calculated according to FOCUS degradation kinetics report (2006) by RMS using ModelMaker 4.0 (FOMC model for cyfluthrin dissipation together with SFO model for metabolite dissipation). For Barmener See system, a DT_{50} (20°C) of 232.2 days was obtained [converted to average EU outdoor temperature of 12°C: DT_{50} = 440.4 days], for Genkel system, the DT_{50} (20°C) was 203.2 days [converted to average EU outdoor temperature of 12°C: DT_{50} = 385.4 days].
Reliability	1
Acceptability	acceptable
Remarks	-

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

Table 7.1.2.2.2/03-1 Characterisation of the water/sediment system:

System	**Barmener See**		**Genkel**	
Source	**Julich/Germany**		**Meinerzhagen/Germany**	
Collection	Freshly sampled from a small disused gravel pit		Freshly sampled from a catchment bassin in the course of the "Genkel creek"	
Sediment:				
pH (0.01 $CaCl_2/H_2O$)	6.9/7.5		4.6/5.0	
Organic carbon content (%)	0.48		4.91	
CEC(meq/100gDM)	3		15	
Total N (%)	0.18		0.43	
Total P (mg P/kg DM)	119		835	
Sand/silt/clay (%, DIN 19682)	94.7/5.3/<0.1		8.2/73.4/18.4	
Redox potential (mV) at time of collection	-224		-114	
Supernatant:	Start	End	Start	End
TOC (mg C/l)	2.7	15	1.5	12
DOC (mg C/l)	2.4	4	1.5	2
Total N (mg N/l)	1.95	10.0	3.12	<1
Total P (mg P/l)	0.1	0.36	0.01	0.27
pH	8.2		7.6	
Oxygen saturation (%)	103		88	
Redox potential (mV)	205		197	

Table 7.1.2.2.2/03-2 Behaviour of [cyclopropane-1-^{14}C] cyfluthrin in two water/sediment systems: % of the applied radioactivity (mean values of two separate samples)

Barmener See	**Time after application**											
	0.5h	**3h**	**6h**	**1d**	**2d**	**3d**	**7d**	**10d**	**14d**	**28d**	**56d**	**100d**
Aqueous phase	46.62	36.64	31.21	39.98	44.26	35.93	45.49	44.43	33.71	44.91	42.28	37.15
$^{14}CO_2$	0.02	0.04	0.04	0.05	0.21	1.69	6.61	7.74	17.83	21.08	30.44	36.72
VOC*	0.17	0.22	-	0.22	0.12	0.02	0.20	0.14	0.38	0.28	0.03	0.08
Sediment	45.14	66.07	73.38	53.09	54.74	61.52	48.15	46.54	46.73	32.26	31.45	28.22
-extracted	43.24	62.90	70.24	50.61	49.07	46.77	31.66	30.81	18.03	16.40	16.08	16.03
-not extracted	1.90	3.17	3.14	2.48	5.67	14.75	16.49	15.73	28.70	15.86	15.37	12.19
Total	91.94	103.0	104.6	93.35	99.23	99.14	100.4	99.05	98.64	98.51	104.2	102.2
Genkel	**0.5h**	**3h**	**6h**	**1d**	**2d**	**3d**	**7d**	**10d**	**14d**	**28d**	**56d**	**100d**
Aqueous phase	28.80	45.49	26.43	25.90	18.47	29.06	31.93	36.23	32.80	45.03	34.82	18.97
$^{14}CO_2$	0.03	0.06	0.11	0.48	1.37	1.55	2.57	3.50	3.75	7.08	12.03	14.20
VOC*	0.28	0.56	0.29	0.35	0.04	0.10	0.19	0.38	0.35	0.22	0.15	0.61
Sediment	65.13	52.48	68.08	72.60	77.29	71.74	67.27	61.30	65.44	47.42	52.32	70.16
-extracted	61.27	49.19	64.01	64.93	66.81	60.13	56.23	47.60	53.80	36.80	39.26	44.13
-not extracted	3.86	3.29	4.07	7.67	10.48	11.61	11.04	13.70	11.64	10.62	13.06	26.03
Total	94.36	98.57	94.90	99.32	95.82	102.45	101.94	101.41	102.34	99.75	99.31	103.9

*VOC= Volatile organic compounds

Table 7.1.2.2.2/03-3 Distribution of metabolites after application of [cyclopropane- l-^{14}C] cyfluthrin to water/sediment system (% of applied radioactivity)

Type of study	Time after application	Cyfluthrin	DCVA	M1	M2	M3	$^{14}CO_2$
Water/	0.5h	82.07	n.d.	n.d.	n.d.	n.d.	0.02
Sediment	3h	85.13	3.92	n.d	n.d.	1.70	0.04
(Barmener See)	6h	90.90	5.73	0.64	0.62	n.d.	0.04
	1d	65.49	18.46	1.54	1.62	n.d.	0.05
	2d	43.98	40.42	n.d	2.91	n.d.	0.21
	3d	41.26	23.02	n.d	3.56	0.47	1.69
	7d	25.40	31.17	n.d	2.53	0.55	6.81
	10d	22.61	36.77	n.d	2.90	0.93	7.74
	14d	13.07	18.96	n.d	1.28	0.88	17.83
	28d	11.33	29.24	n.d	1.10	0.44	21.08
	56d	7.58	37.34	n.d	0.68	1.06	30.44
	100d	7.09	33.64	n.d	0.06	1.01	36.72
Water/	0.5h	85.31	n.d.	n.d.	n.d	n.d.	0.03
Sediment	3h	80.49	3.46	0.08	0.14	n.d.	0.06
(Genkel)	6h	80.95	8.18	0.17	1.04	n.d.	0.11
	1d	60.63	20.62	1.44	3.59	n.d.	0.48
	2d	58.72	18.41	n.d	5.48	0.18	1.37
	3d	47.20	28.44	n.d.	6.97	0.14	1.55
	7d	40.70	33.46	n.d	5.85	0.28	2.57
	10d	31.23	35.10	n.d.	5.40	1.40	3.50
	14d	37.20	31.97	n.d	4.77	0.44	3.75
	28d	19.13	47.63	n.d.	2.03	0.09	7.08
	56d	17.35	46.75	n.d	2.66	0.40	12.03
	100d	15.87	34.80	n.d.	1.87	2.15	14.20

n.d = not detected M1, M2, and M3 are unknowns

Table A7.1.2.2.2/03-4 DT_{50} and DT_{90} normalized to a temperature of 12 °C

	System	DT_{50} (days)	DT_{90} (days)
Cyfluthrin	Barmener See (water phase)	0.2	2.1
Cyfluthrin	Genkel (water phase)	0.3	3.3
Cyfluthrin	Barmener See (whole system)	4.7	107.3
Cyfluthrin	Genkel (whole system)	6.6	130.5
DCVA (2)	Genkel (whole system)	304.8	1012.3

(2) Values in Barmener See system cannot be evaluated due to excessive scatter. DT_{50} and DT_{90} were normalized to a temperature of T_2 = 12 °C using the Q_{10} formula

$$DT50_2 = DT50_1 \, Q_{10}^{\frac{T_1 - T_2}{10}}$$

and the default value Q_{10} = 2.2 as given by FOCUS [2000]

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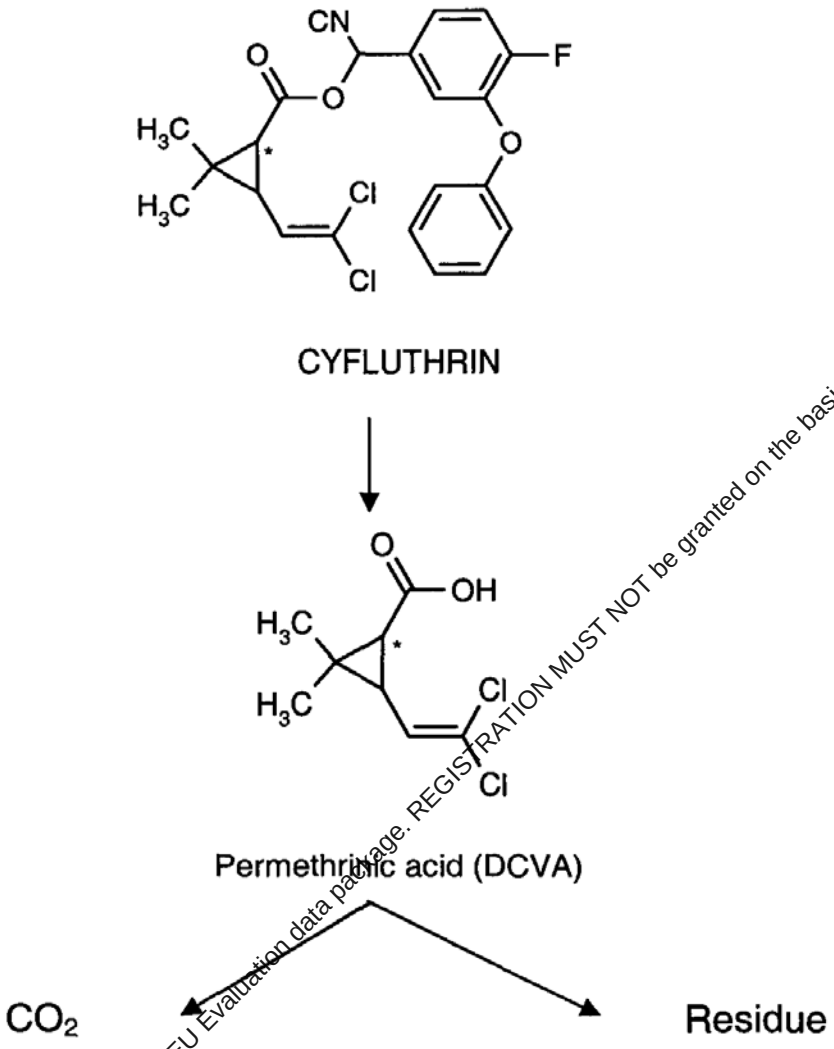

Figure 7.1.2.2.2/03-1 Degradation Pathway

Document IIA/ Section A7.1.3/01 — Adsorption / Desorption screening test

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Official use only

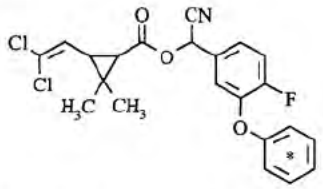
1 REFERENCE

1.1	**Reference**	Burhenne, J (1996). Adsorption/desorption of cyfluthrin on soils. University of Kassel, Agricultural and Ecological Chemistry, Nordbahnhofstralsse la, D-37213 Witzenhausen. Bayer Report No.: IM1972 BES Ref.: M-022224-01-1 Report date: 29 April 1996 Unpublished
1.2	**Data protection**	Yes
1.2.1	Data owner	Bayer CropScience AG
1.2.2		
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes OECD Guideline 106 (1981) EPA Guideline § 163-1 Leaching and Adsorption/ Desorption Studies EC Commission Directive 95/35 EC
2.2	**GLP**	Yes
2.3	**Deviations**	The Kads value was determined only with 1 concentration (and not via Freundlich Isotherm) because of the limiting water solubility for the high concentrations and the determination limit for radioactivity content in solution for the minimum concentration. Additionally the composition of the adsorbed cyfluthrin was determined.

3 MATERIALS AND METHODS

3.1	**Test material**	
3.1.1	Lot/Batch number	Test material used was [phenyl-UL-^{14}C]cyfluthrin, with radiochemical purity > 99 % and specific activity, 4.83 MBq/mg.
3.1.2	Specification	
3.1.3	Purity	
3.1.4	Further relevant properties	 * indicates label position Specification as given in section 2 Non-labelled cyfluthrin was also used: purity = >94.5%
3.1.5	Method of analysis	Purity of the test materials were checked by HPLC before the tests.
3.2	**Degradation products**	Not tested.
3.2.1	Method of analysis	Not applicable

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	for degradation products		
3.3	**Reference substance**	Non-radioactive cyfluthrin, with purity 94.5%	
3.3.1	Method of analysis for reference substance	Active substance content determined by HPLC	
3.4	**Soil types and characteristics**	Soil characteristics are given in table A7.1.3/01-1	X
3.5	**Testing procedure**		
3.5.1	Test system	Samples were taken from the top soil layers (0-30 cm).	X
3.5.2	Test solution and Test conditions	Application solution: 0.01 M CaCl2 with 1 % 2-propanol to increase the solubility of cyfluthrin Concentration: 5.9 µg/l Test conditions: Test conducted in the laboratory: Soil / water ratio 1g : 20ml, sterilised with $HgCl_2$ 43 ml Teflon tubes, shaking period 3 h, 20 °C, darkness Sampling: 2 replicates Analysis of extracts LiChrospher Si 60 column, isocratic with n-hexane/dioxane (975/25) ^{14}C determinations liquid scintillation (fluids) radioactivity monitor (HPLC)	
3.6	**Test performance**	The study was performed according the OECD Guideline No. 106, as stated in the addendum on the monograph from PPP dossier.	
3.6.1	Preliminary test	Not performed	
3.6.2	Screening test: Adsorption	According to (a)"OECD 106": Yes	
3.6.3	Screening test: Desorption	According to (a)"OECD 106": Yes	
3.6.4	HPLC-method	According to (a)" OECD-HPLC-method"[1]: No	
3.6.5	Other test		

4 RESULTS

4.1	**Adsorption**	The adsorption was investigated on four soils with a concentration of 5.9 µg/l in $CaCl_2$ solution. Cyfluthrin shows a high sorption and a low solubility
4.2	**Desorption**	

[1] OECD (1999) OECD-Guidelines for the Testing of Chemicals. Proposal for a new guideline 121: Estimation of the adsorption coefficient (K_{OC}) on soil and on sewage sludge using High Performance Liquid Chromatography (HPLC), Draft Document (August 1999).

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		in water. Due to this low solubility and high sorption no Freundlich-isotherms can be determined but only distribution coefficients (Ka or Kd) at one concentration (5.9 µg/l). Lower concentrations in the application solution lead to low radioactivity in the soil solutions below the limit of determination. The distribution coefficient calculated by the concentration in solution and at the soil as well as the soil carbon-based sorption coefficient Koc resulted in the values given in table A7.1.3/01-2. The extraction of the soils by organic solvents and the subsequent HPLC analysis of the extracts showed that the distribution of isomers of cyfluthrin at the soil remained nearly unchanged as shown in table A7.1.3/01-3	
4.3	**Calculations**		
4.3.1	Ka , Kd	Ka ranged from 1116-1793 in four soil types Kd ranged form 974-1705 in the four soil types above.	X
4.3.2	Ka_{oc} , Kd_{oc}	Ka_{OC} ranged from 73484 to 180290; Kd_{OC} ranged from 69877 to 160889	X
4.4	**Degradation product(s)**	No degradation products tested.	

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1	**Materials and methods**	The adsorption of [phenyl-UL-^{14}C]cyfluthrin was investigated in four soils, two originating from Germany, and two from the US, with a concentration of 5.9 µg/litre in CaCl2 solution. Samples were taken from the top soil layers (0-30 cm) and the test was conducted in the laboratory under the following conditions: soil /water -ratio 1g:20ml, sterilised with $HgCl_2$, shaking period 3 h, 20 °C, darkness. After extraction of the soils, the cyfluthrin content was determined by HPLC and the radioactivity by LSC.	
5.2	**Results and discussion**	Under the conditions of the test, the calculated Ka ranged from 1116-1793 while the Kd ranged form 974-1705 in the four soil types tested. The corresponding Koc values ranged from 73484-180290 (adsorption) and 69877 to 160889 (desorption). Extraction of the soils and subsequent analysis by HPLC showed that the distribution of isomers of cyfluthrin in the soil remained unchanged.	
5.3	**Conclusion**	Cyfluthrin is strongly absorbed to the soil.	
5.3.1	Reliability	1	
5.3.2	Deficiencies	None	

Evaluation by Competent Authorities	
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	2006/10/10
Materials and Methods	Despite minor deficiencies applicant's version is accepted. In table A7.1.3/01-1: Classification and physico-chemical properties of soils used as adsorbents lack of physico-chemical data is mentioned, particularly the content of organic carbon [%] for different soils, essential for calculation of K_{OC} (organic carbon normalised adsorption coefficient). 3.5.1. In accordance to EC method C.18 the horizon depth should not exceed 20 cm.
Results and discussion	The applicant's version is adopted. 4.3. The K_a, K_{OC}, K_d values should be given in cm^3/g.
Conclusion	Cyfluthrin is strongly adsorbed to the soil.
Reliability	2
Acceptability	acceptable
Remarks	According to the test guideline adsorption/desorption screening test should be studied in five different soil types.
	COMMENTS FROM:
Date	*Give date of comments submitted*
Materials and Methods	*Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant's summary and conclusion.* *Discuss if deviating from view of rapporteur member state*
Results and discussion	*Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	

Table A7.1.3/01-1: Classification and physico-chemical properties of soils used as adsorbents Soil types and characteristics

Soil	Laacher Hof	Borstel	Howe	Sable-91	Soil	Laacher Hof
Source	Rheinland, Germany	NiedersachsenGermany	Indiana, USA	Illinois, USA	Source	Rheinland, Germany
Soil type	Silt loam	Loamy sand	Loamy sand	Clay loam	Soil type	Silt loam
Clay/silt/sand (%) (DIN)	11.2/53.0/35.9	3.6/19.7/76.7	7.9/28.6/63.5	29/36/35	Clay/silt/sand (%) (DIN)	11.2/53.0/35.9
PH:H2O/$CaCl_2$	8.1/7.3	5.9/6.0	6.7/6.7	6.5/-	PH:H2O/$CaCl_2$	8.1/7.3

Table A7.1.3/01-2: Distribution coefficient and the soil carbon-based sorption coefficient Koc

Soil	**Adsorption**		**Desorption**	
	K_{ads}	**K_{oc} value**	**K_{ads}**	**K_{oc} value**
Laacher Hof	1116	124000	1448	160889
Borstel	1244	180290	974	141159
Howe	1321	117946	1307	116696
Sable-91	1793	73484	1705	69877

Table A7.1.3/01-3: Distribution of isomers of cyfluthrin in soil.

	Cyfluthrin isomer			
soil	**I**	**II**	**III**	**IV**
Laacher Hof	23.1%	18.1%	34.0%	24.9%
Borstel	24.5%	17.5%	31.4%	26.6%
Howe	25.0%	20.7%	33.5%	20.8%
Sable-91	25.1%	17.3%	34.4%	23.2%
Cyfluthrin specification	23-27%	17-21%	32-36%	21-25%

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1 REFERENCE

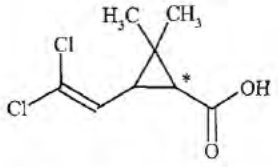
1.1	**Reference**	Slangen, P (1999), Adsorption/desorption of FCR 1272-permethric acid on soil, NOTOX B.V. Hambakenwetering 3, 5231 DD `s-Hertogenbosch, The Netherlands. Bayer AG, Bayer Report No.: IM 1983, BES Ref: M-015423-01-1 Report date: 30 August 1999 Unpublished
1.2	**Data protection**	Yes
1.2.1	Data owner	Bayer CropScience AG
1.2.2	Companies with letters of access	
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2007 on existing a.s. for the purpose of its entry into Annex I

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Yes, OECD Guideline 106 (1997, draft), EPA Guideline § 163-1 Leaching and Adsorption/ Desorption Studies, EC Commission Directive 95/35 EC, SETAC, 1995
2.2	**GLP**	Yes Ministry of Health, Welfare and Sport, State Supervisory Public Health Service,Veterinary Public Health Inspectorate, Rijswijk, The Netherlands
2.3	**Deviations**	None

3 MATERIALS AND METHODS

3.1	**Test material**	
3.1.1	Lot/Batch numbers	
3.1.2	Specification	
3.1.3	Purity	
3.1.4	Further relevant properties	The test materials were: 1) cyclopropane-l-^{14}C FCR 1272-permethric acid [cyclopropane-l-^{14}C]3-(2,2-dichlorovinyl)-2,2-dimethyl-cyclopropane carboxylic acid (DCVA), with radiochemical purity >99 %, isomer ratio of 53.7 % cis/46.3 % trans, specific activity of 3.22 MBq/mg



* = position of ^{14}C label

2) non-labelled FCR 1272-permethric acid, purity >98.9 % and isomer ratio of 51.1 % cis/47.8 % trans

3.1.5	Method of analysis	Radioactive purity was checked by TLC before the tests and stability of permethric acid was checked during the tests

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3.2	**Degradation products**	Not tested.	
3.2.1	Method of analysis for degradation products	Not applicable	
3.3	**Soil characteristics**	Soils characteristic are given in Table A7.1 .3/02-1	
3.4	**Testing procedure**		
3.4.1	Test system	Application solution:	0.01 M $CaCl_2$
		Test conditions: for adsorption kinetics:	Concentration: 1.00/1.02 µg/ml soil/water ratio: 1 : 2 at 22°C, 1 : 1 at 20 °C 50 ml polypropylene tubes shaking periods 4 ... 720
		Test conditions: for adsorption-desorption isotherms:	Concentrations: 0.04, 0.1, 0.5, 1.0 µg/ml soil/water ratio: 1:1 at 20 °C 50 ml polypropylene tubes, shaking period 24 h
		Sampling:	2 replicates
		Analysis by TLC:	Silica-60 F254 Chloroform : methanol: acetic acid (90:10:1 v/v/v)
		^{14}C determinations	Liquid scintillation (fluids) Electronic autoradiography instant imager
3.5	**Test performance**	The study was performed according the OECD Guideline No. 106, as stated in the addendum of the monograph from the PPP dossier.	
3.5.1	Preliminary test	Not performed	
3.5.2	Screening test: Adsorption	According to (a)"OECD 106": Yes	
3.5.3	Screening test: Desorption	According to (a)"OECD 106": Yes	
3.5.4	HPLC-method	According to (a)" OECD-HPLC-method"[1]: No	
3.5.5	Other test		

4 RESULTS

4.1 Adsorption kinetics: The adsorption equilibrium was reached in all soils within 24 h and was comparable for both soil/water ratios.

[1] OECD (1999) OECD-Guidelines for the Testing of Chemicals. Proposal for a new guideline 121: Estimation of the adsorption coefficient (K_{OC}) on soil and on sewage sludge using High Performance Liquid Chromatography (HPLC), Draft Document (August 1999).

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4.2	**Adsorption-Desorption isotherms:**	A significant adsorption-desorption hysteresis effect was observed in the adsorption and desorption isotherms. The isotherms could be described by the Freundlich equation. Correlation coefficients for the adsorption isotherm were >0.99 for all soils.
4.3	**Calculations**	The Freundlich adsorption isotherm parameters are given in Table A7.1.3/02-2: The Freundlich desorption isotherm parameters are given in Table A7.1.3/02-3.
4.4	**Degradation product(s)**	Permethric acid was stable during the experiment and hence no degradation products were formed.

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1	**Materials and methods**	The adsorption behaviour of FCR 1272-permethric acid was investigated in three soils. Adsorption studies used the batch equilibrium method. Adsorption and desorption isotherms were determined over a range of concentrations of 0.04-1 µg/ml. Adsorption kinetics experiments at soil:solution ratio 1:2 were carried out at 22ºC; all other adsorption-desorption experiments were carried out at 20ºC.
5.2	**Results and discussion**	The adsorption equilibrium was reached in all soils within 24 h and was comparable for both soil/water ratios. A significant adsorption-desorption hysteresis effect was observed in the adsorption and desorption isotherms. The isotherms could be described by the Freundlich equation. Correlation coefficients for the adsorption isotherm were >0.99 for all soils.
5.3	**Conclusion**	FCR 1272-permethric acid can be considered to be moderately mobile in Speyer 2.1 soil and Cranfield 115 soil. In Cranfield 230 soil, FCR 1272-permethric acid is considered to be immobile, according to the classification scheme by Mensink, *et.al* .The mean $K_{f,oc}^{ads}$ was 133.71 (1/n = 0.904)
5.3.1	Reliability	1
5.3.2	Deficiencies	None

X

Evaluation by Competent Authorities

EVALUATION BY RAPPORTEUR MEMBER STATE

Date	2006/10/10
Materials and Methods	Applicant's version is accepted.
Results and discussion	Applicant's version is adopted.
Conclusion	Applicant's version is adopted.
Reliability	1
Acceptability	acceptable
Remarks	-

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

Table A7.1 .3/02-1: Classification and physico-chemical properties of soils used as adsorbents Soil types and characteristics

Soil	Speyer 2.1	Cranfield ll5	Cranfield 230
Source	Rheinland-Pfalz/ Germany	Netherton, Goodham/ UK	Tickvall, Derbyshire/ UK
Soil type	sand	Clay loam	Sandy loam
Horizon (cm)	0-20	0-20	10-20
Clay/silt/sand (%) (USDA)	2.5/8.0/89.5	32.2/23.1/44.9	10/18.9/71.2
pH $H_2O/CaCl_2$	6.9/6.0	8.1/7.5	5.1/4.3
Organic Carbon (%)	0.59	1.6	0.8
CEC (meq/l00g)	4	25.9	10.6

Table A7.1 .3/02-2: Freundlich adsorption isotherm parameters

Soil	**K_f^{ads} (cm^3/g)**	**$K_{f,oc}^{ads}$(cm^3/g)**	**l/n**
Speyer 2.1	0.184	31.05	0.884
Cranfield 115	0.224	13.95	0.871
Cranfield 230	2.893	356.15	0.957
Mean	**1.100**	**133.71**	**0.904**

Table A7.1 .3/02-3: Freundlich desorption isotherm parameters

Soil	**K_f^{des} (cm^3/g)**	**$K_{f,oc}^{des}$(cm^3/g)**
Speyer 2.1	0.676	114.19
Cranfield 115	0.498	31.11
Cranfield 230	5.678	699.17
Mean	**2.284**	**281.49**

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BPD Data Set IIA/ Annex Point VII.7.7

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1 REFERENCE

1.1 Reference

Oddy, A.; Brett, R.(2005)
[14C]-AE F105561: Adsorption to and desorption from five soils, Battelle UK Ltd., Ongar, United Kingdom
Report No.: CX/05/054, BES Ref: M-263792-01-1
Report date: 05 December 2005
Unpublished

1.2 Data protection Yes

1.2.1 Data owner: Bayer CropScience AG

1.2.2

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

2 GUIDELINES AND QUALITY ASSURANCE

2.1 Guideline study Yes

OECD Guideline 106 (1981)
EPA Guideline § 163-1 Leaching and Adsorption/ Desorption Studies
EC Commission Directive 95/36/EC Section 7.1.2
Canadian PMRA DACO Number 8.2.4.2 [

2.2 GLP Yes

2.3 Deviations none

3 MATERIALS AND METHODS

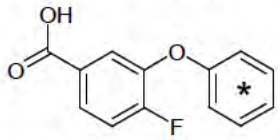
3.1 Test material 4-Fluoro-3-phenoxybenzoic acid (AE F105561, FPB-acid)

3.1.1 Lot/Batch number: Test material used was [^{14}C]-AE F105561, with radiochemical purity = 99 % and specific activity, 9.02 MBq/mg.

3.1.2 Specification

3.1.3 Purity

3.1.4 Further relevant properties



* indicated position of the [^{14}C]-label

Unlabelled 4-Fluoro-3-phenoxybenzoic acid (AE F105561; FPB-acid) was also used: purity > 94%

3.1.5 Method of analysis: -

3.2 Degradation products Not relevant

3.2.1 Method of analysis for degradation products: Not applicable

3.3 Reference substance none

3.3.1 Method of analysis: Not relevant

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	for reference substance	
3.4	**Soil types and characteristics**	Soil characteristics are given in table A7.1.3/03-1
3.5	**Testing procedure**	
3.5.1	Test system	Five agricultural soils were used for the study.; Pikeville sandy loam (USA, pH 5.3, % Organic carbon 1.0), Stanley clay loam (USA, pH 5.7, % Organic carbon 2.1), Höfchen silt loam (Germany, pH 6.5, % Organic carbon 2.07) Laacher Hof AXXa sandy loam (Germany, pH 6.1, % Organic carbon 1.64) Wurmwiese loam(Germany, pH 5.6, % Organic carbon 2.08)
3.5.2	Test solution and Test conditions	Application solution : 0.01 M $CaCl_2$ Nominal concentrations of FPB-acid: 0.5, 0.1, 0.05, 0.01 and 0.005 mg L-1 soil: solution ratio of 1:10 for the Stanley soil, 1:5 for the Höfchen,Pikeville and Wurmwiese soils and 1:3 for the Laacher Hof AXXa soil. Conditions : Adsorption : Pre-equilibration overnight (ca 16 hours); shaking period 2 h, Rotary shaker, 20 ± 2°C, darkness Desorption : shaking period 1 d, Rotary shaker, 20 ± 2°C, darkness Sampling: 2 replicates Analysis of extracts: Selected supernatants were analysed by HPLC Hichrom 5 C18, 250 x 4.6 mm i.d. Water + 0.1% Glacial Acetic Acid / Acetonitrile ^{14}C Detector Radiomatic 625 TR Scintillant Floscint III, flowrate 3 mL min-1 UV Wavelength 276 nm An unlabelled certified reference standard was included as a chromatographic marker ^{14}C determinations: Quantitative measurement of radioactivity in the aqueous supernatant, which was separated from soil by centrifugation, was carried out by liquid scintillation counting (LSC) after addition of a suitable LSC cocktail. After desorption, the soil was extracted twice with acetonitrile: water 4:1 v/v (with 1mL/L formic acid) and total radioactivity in the extracts was determined by LSC. Then the soil was combusted and the trapped $^{14}CO_2$ was quantified by LSC.
3.6	**Test performance**	The study was performed according the OECD Guideline No. 106
3.6.1	Preliminary test	Preliminary studies were carried out to determine the aqueous solubility, adsorption to the tubes, to determine any background radioactivity in the soil, to determine the soil/solution ratio to be used, and to determine the time required for the compound to equilibrate between soil and water under both adsorption and desorption conditions.
3.6.2	Screening test: Adsorption	According to (a)"OECD 106": Yes
3.6.3	Screening test: Desorption	According to (a)"OECD 106": Yes

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3.6.4	HPLC-method	According to (a)" OECD-HPLC-method"[1]: No	
3.6.5	Other test	none	

4 RESULTS

4.1 Preliminary test

4.1.1	Adsorption equilibrium time	In three of the soils the adsorption equilibrium of AE F105561 (FPB-acid) was reached after 24 hours, the exceptions being the Pikeville sandy loam and the Stanley clay loam in which the quantity of adsorbed radioactivity continued to rise up to 72 hours	
4.1.2	Parenthal mass balance	Parental mass balances were determined by LSC and HPLC analysis of the adsorption supernatants and solvent extracts. More than 90% of the applied radioactivity was found as AE F105561 (FPB-acid) in all soils up to 6 hours; Pikeville sandy loam 97.2%, Stanley clay loam 93.4%, Höfchen silt loam 92.3%, Laacher Hof AXXa sandy loam 93.9% and Wurmwiese loam 92.8%. 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 AE F105561 (FPB-acid). In order to ensure the stability of the test item an adsorption equilibrium time of 2 hours was therefore selected for the definitive study. Thus the adsorption phase of the definitive study was stopped before equilibrium had been reached and the determined Koc will therefore represent a lower limit. A pre-equilibration time of ca 16 hours was used.	
4.1.3	Desorption equilibrium time	The levels of radioactivity in the desorption supernatants were equivalent after 1 and 2 hours but then actually fell with longer desorption. This may be indicative of the adsorption phase being terminated before equilibrium had been achieved and thus resulting in the test item or degradate still being adsorbed during the desorption cycle. A desorption time of 1 hour for all soils was selected for the definitive study.	

4.2 Adsorption

The amount of applied test material adsorbed ranged from 23.5 to 53.6% in the Pikeville sandy loam, 22.7 to 72.4% in the Stanley clay loam, 27.5 to 78.2% in the Höfchen silt loam, 21.9 to 54.5% in the Laacher Hof AXXa sandy loam and 31.4 to 82.0% in the Wurmwiese loam

4.3 Desorption

At the end of the final desorption phase, the amount of test material desorbed, expressed as a percentage of the initial amount adsorbed, ranged from 27.5 to 67.8% for the Pikeville sandy loam, 15.6 to 74.3% for the Stanley clay loam, 13.6 to 72.5% for the Höfchen silt loam, 24.7 to 75.7% for the Laacher Hof AXXa sandy loam and 13.5 to 69.0% for the Wurmwiese loam.

4.4 Calculations

4.4.1	K_f , K_{des}	The Kf values ranged from 0.65 in the Laacher Hof AXXa sandy loam to 1.80 in the Stanley clay loam. See table A7.1.3/03-2 Kdes values obtained ranged from 0.89 in the Laacher Hof AXXa sandy loam to 2.32 in the Pikeville sandy loam. See table A7.1.3/03-2	X

[1] OECD (1999) OECD-Guidelines for the Testing of Chemicals. Proposal for a new guideline 121: Estimation of the adsorption coefficient (K_{OC}) on soil and on sewage sludge using High Performance Liquid Chromatography (HPLC), Draft Document (August 1999).

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4.4.2	K_{oc} , $Kd_{oc\ des}$	The values for Koc ranged from 39 in the Laacher Hof AXXa sandy loam to 123 in the Pikeville sandy loam, with a mean value of 73. The Kocdes values ranged from 54 in the Laacher Hof AXXa sandy loam to 232 in the Pikeville sandy loam, with a mean of 106.	
4.5	**Degradation product(s)**	AE F105561 (FPB-acid) was shown to be stable for the duration of the definitive study (5 hours) for all soils.	

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1	**Materials and methods**	The adsorption/desorption of AE F105561 (FPB-acid) was characterised in five soils using the batch equilibrium method in accordance with the OECD Guideline for the Testing of Chemicals No. 106. The adsorption phase of the study was carried out using pre-equilibrated soils with [14C]-AE F105561 at concentrations of approximately 0.5, 0.1, 0.05, 0.01 and 0.005 mg L-1 in the dark and at 20 ± 2°C for 2 hours for all soils. The desorption phase of the study was carried out for 1 hour per cycle with fresh 0.01M aqueous $CaCl_2$ applied to pre-adsorbed soil, for one desorption cycle, with the exception of the highest concentration, where three desorption cycles were performed.	
5.2	**Results and discussion**	AE F105561 (FPB-acid) was shown to be stable for the duration of the definitive study (5 hours) for all soils. Parental mass balances were determined by LSC and HPLC analysis was > 90% of the applied radioactivity found as AE F105561 (FPB-acid) in all soils. Due to lower extractability and a degree of instability of AE F105561 (FPB-acid) for periods longer than 6 hours, the adsorption period had therefore to be limited to 2 hours and thus was stopped before equilibrium had been reached. The determined Koc will therefore represent a lower limit. The overall material balances in the definitive study were determined by LSC and ~ 91% of applied radioactivity (AR) was found to be extractable prior to combustion in all soils. In the definitive adsorption test, the amount of applied test material adsorbed ranged from 23.5 to 53.6% in the Pikeville sandy loam, 22.7 to 72.4% in the Stanley clay loam, 27.5 to 78.2% in the Höfchen silt loam, 21.9 to 54.5% in the Laacher Hof AXXa sandy loam and 31.4 to 82.0% in the Wurmwiese loam. The calculated adsorption constants Kf of the Freundlich isotherms for the five test soils ranged from 0.65 to 1.80. The Freundlich exponents, 1/n, displayed a degree of non-linearity in all five soils tested with values ranging from 0.60 to 0.75, thus indicating an increased degree of adsorption at lower concentrations. At the end of the final desorption phase, the amount of test material desorbed, expressed as a percentage of the initial amount adsorbed, ranged from 27.5 to 67.8% for the Pikeville sandy loam, 15.6 to 74.3% for the Stanley clay loam, 13.6 to 72.5% for the Höfchen silt loam, 24.7 to 75.7% for the Laacher Hof AXXa sandy loam and 13.5 to 69.0% for the Wurmwiese loam. The desorption Kdes values ranged from 0.89 to 2.32 and were thus higher than the Kf values obtained in the adsorption phase, indicating stronger binding once adsorbed to soil.	
5.3	**Conclusion**	The mean determined Koc was 73 and the mean Koc des was 106. Thus, according to Briggs, AE F105561 (FPB-acid) can be classified as having intermediate mobility in soil. The determined Koc, however, represents a lower limit due to the necessary restriction on the adsorption period used in	X

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	the study.
5.3.1 Reliability	1
5.3.2 Deficiencies	None

Evaluation by Competent Authorities

EVALUATION BY RAPPORTEUR MEMBER STATE

Date	2006/10/11
Materials and Methods	Applicant's version is accepted.
Results and discussion	Applicant's version is adopted. Comments: The unity for values of k_f as well as K_{oc} is $\mu l\ g^{-1}$ or $cm^3 g^{-1}$.
Conclusion	Applicant's version is adopted. Comments: Due to the limited adsorption time, the applicant assumes higher K_{oc} as represented in this study. The K_{oc} values for Stanley, Höfchen, Laacher Hof and Wurmwiese soils are situated on the lower limit of Classification "moderately mobile" not effected by moderate increase of K_{oc} values. In contrast, even slight increase of the K_{oc} for FPBacid in Pikeville soil could involve a presentation in category "slightly mobile" in accordance to the classification systems for soil mobility by Briggs et al.
Reliability	1
Acceptability	acceptable
Remarks	-

COMMENTS FROM ...

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

Table A7.1.3/01-1: Classification and physico-chemical properties of soils used as adsorbents Soil types and characteristics

Soil Reference	05/012 Pikeville	05/013 Stanley	05/017 Hofchen	05/018 Laacher HofAXXa	05/019 Wurmiese
Source	703 North Avenue road, Pikeville NC	BRP, Stillwell, HS 66085, US	Hohenseh, Burscheid, Nordrhein-Westfalen, Germany	Monheim, Nordrhein-Westfalia, Germany	Laacherhof, Wurmeiese, Monheim, NRW, Germany
Soil Series		Osaka-Martin	n/a	n/a	n/a
Textural classification (USDA) Sand (50-2000 (im)	Sandy loam 68	Clay loam 36	Silt loam 11.6	Sandy loam 72.6	Is oam 47.3
Silt (2-50 (im)	20	36	70.5	17.1	36.9
Clay (<2 (im)	12	28	17.9	9.9	15.8
pH Deionised Water	6.1	6.4	7.24	6.8	6.4
0.01MCaCl2	5.3	5.7	6.5	6.1	5.6
1MKC1	n/a	n/a	6.0	6.3	5.9
Organic Carbon %	1.0	2.1	2.07	1.64	2.08
Organic Matter %	1.7	3.6	3.56	2.82	3.58
Cation Exchange Capacity (meq/lOOg)	5.4	23.6	12.8	7.3	9.6
Water holding capacity					
pF 0.05 -WHC max	n/a	n/a	57.1	43.6	54.1
pF2.0-WHCO.lbar	17.7	52.8	n/a	n/a	n/a
pF 2.5 -WHC 0.33 bar	11.5	38.7	n/a	n/a	n/a
pF 3.3 -WHC 2 bar	n/a	n/a	n/a	n/a	n/a
pF 4.2 -WHC 15 bar	3.4	23.0	n/a	n/a	n/a
40% WHC	n/a	n/a	25.3	19.4	21.6
50% WHC	n/a	n/a	31.6	24.3	27.1
60% WHC	n/a	n/a	37.9	29.2	32.5
75% 1/3 bar moisture	n/a	n/a	n/a	n/a	n/a
Bulk Density Particle Density	1.45 n/a	1.13 n/a	1.09 2.52	1.15 2.61	1.1 2.59
Soil Taxonomic Classification	n/a	Typil argindolls	n/a	n/a	n/a

n/a - data not available

Table A7.1.3/01-2: Freundlich Adsorption and desorption coefficient and constant of FPB-acid

	Adsorption				Desorption			
Soil type	Kf (mL/g)	l/n	R^2	Koc (mL/g)	Kf (mL/g)	l/n	R^2	Koc (mL/g)
Pikeville	1.23	0.749	0.999	123	2.32	0.777	0.999	232
Stanley	1.80	0.600	0.994	86	2.13	0.571	0.994	101
Höfchen	1.03	0.595	0.981	50	1.22	0.584	0.980	59
Laacher Hof AXXa	0.65	0.733	0.997	39	0.89	0.710	0.995	54
Wurmwiese	1.39	0.609	0.996	67	1.76	0.609	0.998	85
Mean	**1.22**	**0.657**	**0.993**	**73**	**1.66**	**0.65**	**0.993**	**106**

Document IIIA/ Sections 7.1.4.1 **BPD Data Set IIA/ Annex Point XII.2.1**	**Field study on accumulation in the sediment**	
	JUSTIFICATION FOR NON-SUBMISSION OF DATA	Official use only
Other existing data [ ] **Limited exposure [ ]**	**Technically not feasible [ ]** **Scientifically unjustified [✓]** **Other justification [ ]**	
Detailed justification:	Non-extractable residues formed in the water/sediment studies are not exceeding 70% of the applied dose and the mineralization rate in the water/sediment systems is more than 5% in 100 days. Therefore a field study on accumulation in the sediment is not required	
Undertaking of intended data submission [ ]		
	Evaluation by Competent Authoritiess	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
	EVALUATION BY RAPPORTEUR MEMBER STATE	
Date	2009/01/07	
Evaluation of applicant's justification	The applicant's justification is acceptable.	
Conclusion	Applicant's justification is acceptable.	
Remarks	The risk assessment shows no unacceptable risk for the water/sediment system applying risk mitigation measures, therefore the study is not required.	
	COMMENTS FROM OTHER MEMBER STATE *(specify)*	
Date	*Give date of comments submitted*	
Evaluation of applicant's justification	*Discuss if deviating from view of rapporteur member state*	
Conclusion	*Discuss if deviating from view of rapporteur member state*	
Remarks		

Document IIIA/ Sections 7.1.4 **BPD Data Set IIA/ Annex Point XII.2.2**	**Further studies on adsorption/desorption in water/sediment system**	
	JUSTIFICATION FOR NON-SUBMISSION OF DATA	Official use only
Other existing data [ ] **Limited exposure [ ]**	**Technically not feasible [ ]** **Scientifically unjustified [✓]** **Other justification [ ]**	
Detailed justification:	Non-extractable residues formed in the water/sediment studies are not exceeding 70% of the applied dose and the mineralization rate in the water/sediment systems is more than 5% in 100 days. Therefore a field study on accumulation in the sediment is not required	
Undertaking of intended data submission [ ]		
	Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted	
	EVALUATION BY RAPPORTEUR MEMBER STATE	
Date	2008/10/20	
Evaluation of applicant's justification	Applicant's justification refers to Doc III A 7.1.4.1 - Field study on accumulation in the sediment. Thus, the justification should be moved there. Further studies on adsorption/desorption in water/sediment studies are not required.	
Conclusion	The justification fails the required data and should be moved to "Field study on accumulation in the sediment".	
Remarks	-	
	COMMENTS FROM OTHER MEMBER STATE *(specify)*	
Date	*Give date of comments submitted*	
Evaluation of applicant's justification	*Discuss if deviating from view of rapporteur member state*	
Conclusion	*Discuss if deviating from view of rapporteur member state*	
Remarks		