Section A7.1.3 Adsorption / Desorption screening test

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			Official use only
		1 REFERENCE	
1.1	**Reference**	Sommer, H., 2001, Estimation of the Adsorption Coefficient (Koc) of Dichlofluanid on Soil using High Performance Liquid Chromatography. Bayer AG, Institute for Metabolism Research and Residue Analysis, Monheim, Germany, Report No. MR-010/01, 2001-05-31	
1.2	**Data protection**	Yes	
1.2.1	Data owner	Bayer Chemicals AG	
1.2.2	Companies with letter of access	-	
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/IA	
		2 GUIDELINES AND QUALITY ASSURANCE	
2.1	**Guideline study**	Yes OECD Guideline 121 (Proposal for a New Guideline 121, January 2001)	
2.2	**GLP**	Yes	
2.3	**Deviations**	No	
		3 MATERIALS AND METHODS	
3.1	**Test material**	Dichlofluanid	
3.1.1	Lot/Batch number	[redacted]	
3.1.2	Specification	As given in section 2 of the dossier	
3.1.3	Purity	[redacted]	
3.1.4	Further relevant properties	-	X
3.1.5	Method of analysis	HPLC, fitted with a pulse-free pump and a suitable detection device according to OECD Guideline 121	
3.2	**Degradation products**	Degradation products tested: Yes; Main metabolite: dimethylaminosulfanilide (DMSA)	
3.2.1	Method of analysis for degradation products	HPLC	
3.3	**Reference substance**	Yes, 13 reference compounds were used to determine an average capacity factor k': acetanilide, N,N-dimethylbenzamide, Atrazine, Isoproturon, Aniline, Triadimenol, Linuron, Methiocarb, Fenthion, Pyrazophos, Phenantrene, Cyfluthrin and dimethylaminosulfanilide (DMSA). Sodium nitrate was used to determine the HPLC dead time (t_0).	X
3.3.1	Method of analysis for reference substance	HPLC	

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3.4	**Testing procedure**		X
3.4.1	Test system	HPLC (HP 1090 with DAD detector) is performed on analytical columns (Zorbax CN, length 250 mm, i.d. 4.6 mm) packed with a commercially available cyanopropyl solid phase containing lipophilic and polar moieties. As mobile phase methanol/0.01 M citrate-buffer pH 6.0 (55/45, v/v) was used. As a result of partitioning between mobile and stationary phases the test substance is retarded. The dual composition of the stationary phase having polar and non-polar sites allows for interaction of a molecule in the similar way as in the case for organic matter in soil. This enables the relationship between the retention time on the column and the adsorption coefficient on organic matter to be established.	
3.4.2	Test solution and Test conditions	According to guideline, maximum concentration of the test substance should not exceed 50% of the solubility in the solvent. Therefore the measurements were carried out at concentrations of approx. 5 mg/l. Stock solution: 11.68 mg dichlofluanid was weighed into a 10-ml volumetric flask and diluted to volume with methanol. Standard solution: 0.1 ml of stock solution was transferred into a 20-ml volumetric flask and diluted to volume with the mobile phase methanol/citrate buffer pH 6.0. HPLC parameters: Oven temperature 40 °C, Injection volume 250 µl, Flow rate 1.5 ml/min, run time 30 min.	
3.5	**Calculations**	**Kd:** Distribution coefficient is defined as the ratio of equilibration concentrations C of a dissolved test substance in a two phase system consisting of a sorbent (soil or sewage sludge) and an aqueous phase. It can be dimensionless or have the dimension ml/g. **Koc:** Distribution coefficient (Kd) or Freundlich adsorption coeffcient (Kf) normalised to the organic carbon content (foc) of a sorbent. Depending on the dimensions of Kd and Kf, Koc can be dimensionless or have the dimensions ml/g or µg/g organic matter, respectively. Using the HPLC estimation method Koc is deduced from the capacity factor (k') using a calibration plot of log k' versus log Koc of the selected reference compounds. Koc is an indicator for the extension of adsorption between a substance and the sorbent and allows comparisons to be made between different chemicals. **k':** Capacity factor = $(t_R - t_0)/t_0$; t_R = HPLC retention time of test and reference substances (min); t_0 = HPLC dead time (min). **log Koc** = Slope x log k' + Intercept; Slope and intercept derived from the linear regression of the reference standards using Koc.	X
		4 RESULTS	X
4.1	**Measurements**	HPLC retention time data for the reference compounds and dichlofluanid are given in table A7.1.3_1. The dead time t_0 was determined to be 1.536 min using sodium nitrate. Variability of the retention times from repetitive injections was low, confirming HPLC system stability throughout the analysis period.	X

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4.2	**Calculations**	Calculated adsorption parameter for the reference compounds and dichlofluanid are given in table A7.1.3.1_1.	
4.3	**Degradation product(s)**	DMSA as main metabolite was investigated as reference substance. For results see table A7.1.3.1_1.	X
		5 APPLICANT'S SUMMARY AND CONCLUSION	
5.1	**Materials and methods**	The adsorption koefficient Koc of dichlofluanid on soil was estimated using High Performance Liquid Chromatography (HPLC). The test was performed according to OECD Guideline 121 (Proposal for New Guideline, 2001). Thirteen reference standards of known Koc were analysed on a HPLC system to determine an average capacity factor k'. Sodium nitrate was used to determine the HPLC system dead time (t_0). A regression line was plotted with the determined k' values and the known Koc values (log k' versus log Koc).	
5.2	**Results and discussion**	Dichlofluanid was analysed on the same HPLC system during the same sample sequence as the reference substances and an average k' value of 1.415 was determined. The Koc value for dichlofluanid was estimated by interpolation from the reference substance regression line. The linear regression of measured k' values against literature Koc values yielded a line with a slope of 4.41, an intercept of 2.46 and a correlation coefficient R^2 of 0.893. The estimated Koc value for dichlofluanid is 1344.	
5.3	**Conclusion**	Based on classifications of Briggs (Proc. 7th British Insecticide and Fungicide Conference, Nottingham, UK, 83-86, 1973) and Verdam et al. (RIVM Report No. 728473001, NL, 1988) for the estimation of the mobility of plant protectants in soil based on Kd and/or Koc-values, dichlofluanid is to be classified as an immobile substance.	
5.3.1	Reliability	1	
5.3.2	Deficiencies	No	

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Evaluation by Competent Authorities	
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE (*)
Date	2/12/2004
Materials and Methods	Applicant's version is acceptable with the following comments: **3.1.4** The solubility of dichlofluanid in the HPLC solvents (methanol and citrate buffer) appears to have been considered, but not presented here. **3.3** and **4.3** DMSA was not used as a reference substance, according to the study report - this should read DMST (with the name also given in full). **3.4 and 3.5** The numbering and headings within these sections are incorrect. According to the Technical Notes for Guidance Section 3.4 should have been 'Soil Types' (this section has been omitted, but does not affect the study report conclusions as the study does not include soil), Section 3.5 should have been 'Testing Procedure' (and the required information has been given as Section 3.4) and Section 3.6 should have been 'Test Performance' (this section has been omitted, but the necessary information is given in Section 5.1).
Results and discussion	Applicant's version is acceptable with the following comments: **4.** The numbering and headings within this section do not follow the Technical Notes for Guidance, although the information required is given. Section 4.1 should be named 'Preliminary Test' but does include the necessary results, Sections 4.2 & 4.3 on screening tests are omitted and do not affect the study conclusions, and Sections 4.2 and 4.3 should be numbered 4.4 and 4.5 respectively. **4.1** The is a typing error in the mean retention time for cyfluthrin in table A7.1.3.1_1 which should read 7.496 The order in which the samples were analysed, and whether blank runs were included, would have been useful to rule out any likelihood that the aniline chromatogram contained carryover from acetanilide (identical t_r). The consistency in rounding, to two decimal points, k' and mean log k' values seems to be ignored for dichlofluanid compared to the reference compounds. **4.3** see above under 3.3.
Conclusion	Applicant's version is acceptable.
Reliability	1
Acceptability	Acceptable
Remarks	All endpoints and data presented in the summary and tables have been checked against the original study and are correct.
	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*

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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 A7.1.3.1_1: HPLC retention time data and Koc calculations

Substance	Mean Retention Time [min]	MeanDead Time [min]	Mean k'	Mean log k'	Mean Koc	Mean log Koc
Sodium nitrate	-	1.536	-	-	-	-
Acetanilide	2.485	1.536	0.62	-0.21	17.8	1.25
N,N-dimethylbenzamide	2.643	1.536	0.72	-0.14	33.1	1.52
Atrazine	2.746	1.536	0.79	-0.10	64.6	1.81
Isoproturon	2.966	1.536	0.93	-0.03	72.4	1.86
Aniline	2.485	1.536	0.62	-0.21	117	2.07
Triadimenol	3.044	1.536	0.98	-0.01	251	2.40
Linuron	3.323	1.536	1.16	0.07	389	2.59
Methiocarb.	3.012	1.536	0.96	-0.02	1,259	3.10
Fenthion	4.013	1.536	1.61	0.21	2,042	3.31
Pyrazophos,	4.010	1.536	1.61	0.21	4,467	3.65
Phenantrene	4.600	1.536	2.00	0.30	12,303	4.09
Cyfluthrin	7.7.496	1.536	3.88	0.59	64,300	4.81
Dimethylaminosulfanilide (DMSA)	2.693	1.536	0.76	-0.12	76.25	1.88
Dichlofluanid	**3.710**	**1.536**	**1.415**	**0.1507**	**1,344**	**3.13**