Section A7.1.3 Adsorption / Desorption screening test

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			Official use only
		1 REFERENCE	
1.1	**Reference**	Baltussen, E. (2008). Estimation of the adsorption coefficient (K_{OC}) of lactic acid 93% aq on soil and on sewage sludge using high performance liquid chromatography (HPLC). Notox Document 489046 GLP, Unpublished	
1.2	**Data protection**	Yes	
1.2.1	Data owner	Purac Biochem	
1.2.2	Companies with letter of access	No	
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing [a.s. / b.p.] for the purpose of its [entry into Annex I/IA / authorisation]	
		2 GUIDELINES AND QUALITY ASSURANCE	
2.1	**Guideline study**	Yes, OECD 121	
2.2	**GLP**	Yes	
2.3	**Deviations**	No	
		3 MATERIALS AND METHODS	

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3.1	**Test material**	Lactic acid 93% aqueous solution
3.1.1	Lot/Batch number	0712002519
3.1.2	Specification	As given in section 2
3.1.3	Purity	99.8% (solution was 92.8% lactic acid in water vs. nominal concentration of 93% lactic acid in water).
3.1.4	Further relevant properties	Substance is a strong acid; explicit instructions in the guideline apply.
3.1.5	Method of analysis	Not relevant. OECD is a direct screening test based on chromatography of the active substance; no analysis of samples from an experimental test is needed.
3.2	**Degradation products**	Degradation products tested: Not relevant; in the HPLC screening test no metabolites/degradates are formed. OECD 121 only tests the acsorption/desorption behaviour of the parent compound.
3.2.1	Method of analysis for degradation products	Not applicable
3.3	**Reference substance**	Yes; phenol
3.3.1	Method of analysis for reference substance	Not relevant. OECD is a direct screening test based on chromatography of the active substance; no analysis of samples from an experimental test is needed.
3.4	**Soil types**	Not relevant. OECD is a direct screening test based on chromatography of the active substance; no analysis of samples from an experimental test is needed.
3.5	**Testing procedure**	
3.5.1	Test system	Direct injection of solution of test substance onto HPLC column.
3.5.2	Test solution and Test conditions	Test at neutral pH Standard: Stock solution: 1 g/L solution of phenol in methanol. Test solution: 0.05 mL stock solution in 5 mL mobile phase. Test substance: Stock solution: 1.744 g/L solution of test substance in methanol. Test solution: 0.6 mL stock solution and 1.4 mL water in 5 mL mobile phase; final concentration test solution: 0.209 g/L. Test at pH 2.0 Standard: Stock solution: 1 g/L solution of phenol in methanol. Test solution: 0.05 mL stock solution in 5 mL mobile phase. Test substance: Stock solution: 1.128 g/L solution of test substance in methanol. Test solution: 0.6 mL stock solution and 1.4 mL phosphate buffer pH 2.0 in 5 mL mobile phase; final concentration test solution: 0.135 g/L.
3.6	**Test performance**	
3.6.1	Preliminary test	Not applicable
3.6.2	Screening test:	Not applicable

	Adsorption	
3.6.3	Screening test: Desorption	Not applicable
3.6.4	HPLC-method	Yes. HPLC: Alliance 2695 with UV detector 2487, Waters Column: Hypersil BDS-CN cyanopropyl mixed phase column, dp = 5 µm, Thermo Eluent: 30/70 v/v methanol/water (neutral pH); 30/70 v/v methanol/0.05 M phosphate buffer pH 2.0 (pH 2.0) Flow: 1 mL/min Injection volume: 10 µL Detection: 210 nm Data capture and calculations: Cary WinUV 3.1, Varian; Empower 5.00, Waters
3.6.5	Other test	

4 RESULTS

4.1	**Preliminary test**	see table A7_1 _3-2
4.2	**Screening test: Adsorption**	pKa of lactic acid (Perrin method) is 3.08. Therefore, HPLC test is done at neutral pH and at pH 2.0. R_t phenol is 2.98 minutes at neutral pH and 2.785 minutes at pH 2.0. R_t test substance is shorter than R_t phenol at neutral pH and at pH 2.0. K_{OC} test substance therefore < 20.9 (log K_{OC} < 1.32)
4.3	**Screening test: Desorption**	N.A.
	Calculations	
4.3.1	Ka , Kd	N.A.
4.3.2	Ka_{oc} , Kd_{oc}	N.A.
	Degradation product(s)	N.A.

5 APPLICANT'S SUMMARY AND CONCLUSION

5.1	**Materials and methods**	HPLC method is suitable for estimating the overall adsorption/desorption behaviour of organic acids, especially at low pH. Lactic acid is retained less by a cyanopropyl column than the reference substance phenol. Test is therefore valid.
5.2	**Results and discussion**	Lactic acid is retained less by a cyanopropyl column than the reference substance phenol. Peak shapes are not quite ideal but acceptable. Two major components are evident at R_t 1.21 minutes and 1.55 minutes at neutral pH; at pH 2.0 three major peaks are evident at R_t 2.08 minutes, R_t 2.20 minutes, and R_t 2.41 minutes. Individual peaks correspond to lactic acid and major oligomers. All peaks have shorter retention times than phenol. Log K_{OC} estimates are therefore all < 1.32.
5.2.1	Adsorbed a.s. [%]	N.A.
5.2.2	K_a	N.A.
5.2.3	K_d	N.A.
5.2.4	Ka_{oc}	N.A.
5.2.5	Ka/Kd	N.A.
5.2.6	Degradation products (% of a.s.)	N.A.
5.3	**Conclusion**	Results are valid within the constraints of the OECD 121 HPLC screening method for KOC. Applicability of OECD 121 may be subject to future validation investigations, but from the test results it appears that the test is relevant for lactic acid. Lactic acid is expected to be reasonably mobile in soil.
5.3.1	Reliability	1
5.3.2	Deficiencies	No

(If yes, discuss the impact of deficiencies and implications on results. If relevant, justify acceptability of study.)

	Evaluation by Competent Authorities
	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	2009/05/18
Materials and Methods	A HPLC-screening test according to the OECD test guideline (TG) No. 121 is submitted. As the substance is expected to be ionized for at least 10% within pH 5.5 to 7.5, the pKa-value was calculated and the HPLC-analysis was performed with both the ionized (measured at neutral pH) and the non-ionized form (measured at pH=2). In the test report it is described, how a calibration graph has to be prepared, but it is not documented that a calibration graph is performed. Instead of using a calibration graph, the retention time of the a.s. is compared with the retention time of phenol, one of the reference substances of the method with a low log Koc (1.32 L/kg). Although this value alone is outside of the range for which the method is applicable (log Koc 1.5 to 5 L/kg, see OECD TG), this approach can be accepted under consideration of all circumstances.
Results and discussion	Under the chromatographic conditions of the method, the retention time of the active substance is lower than the retention time of the reference substance phenol with the known log Koc of1.32 L/kg. Therefore it was concluded, that the log Koc of lactic acid at neutral pH, as well as at pH=2 is < 1.32 L/kg (Koc<20.9 L/kg).
Conclusion	Applicant's version can be adopted with constraints.
Reliability	2
Acceptability	Considering the properties of lactic acid (e.g. high water solubility, low log Kow, biodegradability) as a naturally occurring substance with low risk potential, the test can be accepted. Formally a test according to OECD TG No. 106 would have to be required; but it is not expected, that such a requirement will lead to considerably other results. In a literature study, performed by RMS, Sansone, FJ et al. (Geochim Cosmochim Acta 51: 1889-96 (1987)) reported, for example, an experimental estimated K_{OC}-value on a clastic mud for 5.7 L/kg and of 0.08 L/kg for a lateralic muddy sand.
Remarks	Our conclusion to accept the test, due to the circumstances mentioned above, is shared by the Swedish CA in the frame of an inquiry to the electronic discussion group.
	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*

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

Not applicable

Table A7_1 _3-2: Results of preliminary test:

Test substance	Lactic acid 93%
Sample purity	99.8%
Weighed soil	N.A.
Volume of $CaCl_2$ solution	N.A.
Nominal concentration of a.s. final solution	1.744 g/L ; 1.128 g/L
Analytical concentration final of a.s. solution	N.A.
Concentration of the test solution (show calculation)	0.209 g/L ; 0.135 g/L
Details of the analytical method used:	N.A.; HPLC screening method
Method	
Recovery rate	
Detection limit	

Table A7_1 _3-3: Results of screening test - adsorption:

Not applicable

Table A7_1 _3-4: Results of screening test - desorption:

Not applicable