

Table A7_2_1-5a: Cumulative Data for Total CO₂ Generated by Treated and Control Aerobic Non-Sterile Soils

Duration of Incubation (days)	Cumulative µgCO ₂ generated /g soil				
	Control	20 ppm Mancozeb*	10 ppm Mancozeb*	10 ppm ETU	5ppm 2,4-D
0	0.0	0.0	0.0	0.0	0.0
2	157.9	91.7	124.8	178.2	198.6
6	799.6	630.3	692.0	819.9	846.0
13	1360.6	1368.0	1354.3	1390.3	1463.6
23	2207.6	2245.3	2127.1	2116.3	2228.1
37	3151.4	3423.4	3014.8	2994.1	3132.3
66	4760.2	4982.6	4586.4	4569.9	4691.6
93	6784.2	6775.6	6500.4	6626.9	6704.6

* Data are the means from determinations of duplicate incubations

Table A7_2_1-5b: Cumulative Data for Total CO₂ Generated by Treated and Control Aerobic Sterile Soils

Duration of Incubation (days)	Cumulative µgCO ₂ generated /g soil				
	Control	20 ppm Mancozeb	10 ppm Mancozeb	10 ppm ETU	5ppm 2,4-D
0	0.0	0.0	0.0	0.0	0.0
2	0.0	45.8	32.1	36.7	22.9
6	0.0	96.2	36.7	68.8	32.1
13	50.4	142.0	36.7	91.7	73.4
23	77.9	201.6	50.5	105.5	87.2
31	87.1	265.8	55.1	119.3	101.0

Table A7_2_1-6: Cumulative Mineralisation of Mancozeb, ETU and 2,4-D in Soil under Aerobic, Non-sterile Conditions

Duration of Incubation (days)	Treatment			
	Mancozeb (20 ppm)	Mancozeb (10 ppm)	ETU (10 ppm)	2,4-D (5 ppm)
0	0	0	0	0
2	0.50	0.23	0.4	6.45
6	0.77	0.93	0.79	9.51
13	1.84	2.17	1.03	11.37
23	3.38	3.23	1.63	13.35
37	5.17	4.74	2.64	15.68
66	17.52	7.70	4.64	26.36
93	44.92	35.68	58.24	225.45

Note: These data are reported as ‘mineralisation’ on the basis of the radioactivity present in traps as CO₂ being expressed as a percentage of residual soil radioactivity remaining at that time for each time point. The cumulative data presented here were derived from this by adding the ‘% mineralisation’ for each timepoint to the running total for each flask.

Table A7_2_1-7a: Radioactivity and Evolved CS₂ Before and After Extraction in Non-Sterile Soil Treated with 20 ppm Mancozeb

Time Interval (days)	¹⁴C in soil before extraction (ppm) *	¹⁴C in soil after extraction (ppm) *	CS₂ evolved from soil before extraction (ppm) *	CS₂ evolved from soil after extraction (ppm) *
0	17.48	6.81	10.44	1.91
2	16.70	11.30	1.90	1.87
6	16.46	11.99	1.63	ND
13	16.51	12.14	0.77	ND 0.91
23	16.67	11.85	0.69	-
37	15.64	10.11	-	-
66	14.66	10.67	-	-
93	10.90	8.06	-	-

* data are the mean results from duplicate flasks

**Table A7_2_1-7b: Radioactivity and Evolved CS₂ Before and After Extraction in Non-Sterile Soil
Treated with 10 ppm Mancozeb**

Time Interval (days)	¹⁴C in soil before extraction (ppm)*	¹⁴C in soil after extraction (ppm)*	CS₂ evolved from soil before extraction (ppm) *	CS₂ evolved from soil after extraction (ppm) *
0	7.83	3.17	3.22	2.41
2	8.62	5.85	1.11	0.66
6	9.63	4.64	0.86	ND
13	8.27	5.65	ND	ND
23	8.34	7.26	ND	-
37	8.57	5.57	-	-
66	8.15	5.36	-	-
93	6.06	4.23	-	-

* data are the mean results from duplicate flasks

**Table A7_2_1-7c: Radioactivity Before and After Extraction in Non-Sterile Soil Treated with 10
ppm ETU**

Time Interval (days)	¹⁴C in soil before extraction (ppm)	¹⁴C in soil after extraction (ppm)
0	11.66	0.75
2	10.50	4.43
6	10.44	4.27
13	11.38	5.76
23	11.57	4.36
37	11.35	6.14
66	10.83	6.70
93	6.07	5.36

Table A7_2_1-7d: Radioactivity Before Extraction in Non-Sterile Soil Treated with 5 ppm 2,4-D

Time Interval (days)	¹⁴ C in soil before extraction (ppm)
0	5.60
2	5.64
6	5.33
13	5.37
23	5.19
37	5.28
66	4.50
93	1.24

**Table A7_2_1-8: Radioactivity and Evolved CS₂ Before and After Extraction in Sterile Soil
Treated with 20 and 10 ppm Mancozeb after 31 days Incubation**

Mancozeb Concentration	¹⁴C in soil before extraction (ppm) *	¹⁴C in soil after extraction (ppm) *	CS₂ evolved from soil before extraction (ppm) *	CS₂ evolved from soil after extraction (ppm) *
20 ppm	17.69	15.80	2.92	1.55
10 ppm	9.30	7.28	2.01	1.39

* data are the mean results from duplicate flasks

Table A7_2_1-9: Distribution of radioactivity between soil and water in Anaerobic soil

Mancozeb Concentration	Day	¹⁴ C in soil (ppm) *	¹⁴ C in water (ppm) *	Soil/Water Ratio *
20 ppm Mancozeb	27	12.41	5.41	2.3
	61	12.04	5.63	2.15
10 ppm Mancozeb	27	6.13	2.88	2.15
	61	5.37	2.43	2.2
10 ppm ETU	27	5.77	5.96	0.95
	61	5.72	5.69	1.0
5 ppm 2,4-D	27	2.74	3.19	0.85
	61	2.36	2.69	0.85

* data are the mean results from duplicate flasks

Section A7.2.2.4 Annex Point IIIA XII.1.1.		Other Soil Degradation studies	Official use only
JUSTIFICATION FOR NON-SUBMISSION OF DATA			
Other existing data [] Limited exposure []	Technically not feasible [] Other justification [X]	Scientifically unjustified []	
Detailed justification: The studies summarised elsewhere in section A7.2 are considered to adequately characterise the fate and behaviour of Zineb and its metabolites in soil for the purpose of assessing the level of risk associated with the proposed use. Consequently, it is considered that there is no necessity to conduct further studies to investigate other aspects of soil degradation.			
Undertaking of intended data submission []			
Evaluation by Competent Authorities			
<i>Use separate "evaluation boxes" to provide transparency as to the comments and views submitted</i>			
EVALUATION BY RAPPORTEUR MEMBER STATE			
Date	<i>Give date of action</i>		
Evaluation of applicant's justification	<i>Discuss applicant's justification and, if applicable, deviating view</i>		
Conclusion	<i>Indicate whether applicant's justification is acceptable or not. If unacceptable because of the reasons discussed above, indicate which action will be required, e.g. submission of specific test/study data</i>		
Remarks			
COMMENTS FROM OTHER MEMBER STATE (specify)			
Date	<i>Give date of comments submitted</i>		
Evaluation of applicant's justification	<i>Discuss if deviating from view of rapporteur member state</i>		
Conclusion	<i>Discuss if deviating from view of rapporteur member state</i>		
Remarks			

Section A7.2.3.1(1) Adsorption / Desorption screening test

Annex Point IIIA XII.1.2

IUCLID 3.3.2/01

Batch Soil Adsorption/Desorption of Mancozeb

		1 REFERENCE	Official use only
1.1	Reference	Yeh, S. M., (1986a) Batch Soil Adsorption/Desorption of Mancozeb, Rohm and Haas Company, 727 Norristown Road, Spring House, PA 19477, USA, Report No. 310-86-62, 10 November 1986.	
1.2	Data protection	Yes	
1.2.1	Data owner	Rohm & Haas	
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 Guideline 163-1.	
2.2	GLP	Yes	
2.3	Deviations	No.	
		3 MATERIALS AND METHODS	
3.1	Test material	¹⁴ C-Dithane M-45 containing 76.6% Mancozeb	
3.1.1	Lot/Batch number	541.04	
3.1.2	Specification	Deviating from specification given in section 2 as follows	
3.1.3	Purity	Dithane contained 76.6% Mancozeb The radiopurity was 88%, none of the formulation additives were radio-labelled.	
3.1.4	Further relevant properties	The specific activity of the formulated product was 9.61 µCi/mg	
3.1.5	Method of analysis	Refer to section 3.2.1 for methods of analysis.	
3.2	Degradation products	Degradation products tested: Yes	
3.2.1	Method of analysis for degradation products	Thin layer chromatography (TLC) of samples prepared from supernatant after 24 hours adsorption, supernatant after 24 hours desorption and soil after adsorption/desorption phases. Samples were spotted on Keisegel 60 F ₂₅₄ TLC plates and developed for ca 100 minutes using a solvent system of 65:25:10 ethyl acetate/isopropanol/water. Standard samples of ¹⁴ C-Ethylenethiourea (ETU) and unlabelled standards of ethylene urea (EU), ethylenediamine (EDA), Jaffe's base (JB), hydantoin (hyd) and ethylene bis-isothiocyanate sulfide (EBIS) were co-chromatographed on each plate. Components were visualised by use of spray reagents (fluorescamine, sodium nitroferricyanide, potassium ferricyanide and p-dimethylaminobenzaldehyde (Ehrlich's reagent)). Additionally, radiolabelled components were visualised and quantified using a Bioscan System 200 Imaging Scanner equipped with a Bioscan Autochanger 3000 and linked to an IBM PC-XT computer. To demonstrate the extent of breakdown of mancozeb, a specific method	

Section A7.2.3.1(1) Adsorption / Desorption screening test**Annex Point IIIA XII.1.2****IUCLID 3.3.2/01****Batch Soil Adsorption/Desorption of Mancozeb**

for the detection of intact mancozeb in study samples was applied to selected samples of soil and supernatant. The method detected CS₂, a quantitative breakdown product of Mancozeb. CS₂ was generated by reflux of the sample in dilute HCl in the presence of SnCl₂ then trapped before determination by gas chromatography using a flame photometric detector in sulphur mode.

A specific method was used for the analysis of ethylenethiourea (ETU) in soil samples. ETU residues were extracted from samples with methanol followed by partial clean-up of the extracts on alumina columns. Extracted ETU was derivatised with 1-bromobutane and the resulting derivative measured by gas chromatography with flame photometric detection.

3.3 Reference substance

No

3.3.1 Method of analysis for reference substance

Not applicable

3.4 Soil types

4 soil types were used:-

Soil 1: Georgia Sand (85E373)

Soil 2: Georgia Sandy Loam (85E372)

Soil 3: Pennsylvania Silt Loam (85E655)

Soil 4: Mississippi Clay loam (85E416)

Refer to table A7_1_3-1 for full details

3.5 Testing procedure**3.5.1 Test system**

The test system was comprised of non-sterile soil samples mixed with ¹⁴C-test material dissolved in 0.01M calcium chloride. The samples were held in 40 mL glass test vials with plastic lids and teflon cap liners which were incubated in the dark at 25-26°C on an orbital shaker set at ca 225rpm.

LSC of liquid samples was conducted using a Beckman LS3801 Liquid Scintillation Counter.

Radioactivity in soil samples was estimated by LSC following combustion in an R. J. Harvey Biological Materials Oxidizer. Counting efficiencies were calculated using factory standard quench sample data.

Supernatants were concentrated 3 fold prior to radio-profiling using a Buchi Rotavapor-R under vacuum at 36°C for 40-60 minutes.

Methanol extracts of soils were concentrated prior to radio-profiling by evaporation under nitrogen gas using an analytical N-EVAP (Organomation Assoc. Inc.).

Following TLC analysis, the location of standards and corresponding components from the sample study were visualised after treatment with reagents as detailed in section 3.2.1 and radiolabelled components were visualised and quantified using a Bioscan System 200 Imaging Scanner equipped with a Bioscan Autochanger 3000 and linked to an IBM PC-XT computer.

For the specific detection of intact mancozeb in study samples, a method

Section A7.2.3.1(1) Adsorption / Desorption screening test**Annex Point IIIA XII.1.2****IUCLID 3.3.2/01****Batch Soil Adsorption/Desorption of Mancozeb**

relying on the evolution of carbon disulphide with subsequent GC analysis was applied.

For the specific analysis of ethylenethiourea (ETU) in soil samples, a method relying on the derivitisation of extracted ETU with 1-bromobutane with subsequent GC analysis was applied.

3.5.2 Test solution and Test conditions

A fresh stock solution of ^{14}C Dithane was prepared on each occasion of use by dissolving TS in 0.01M calcium chloride to give a stock solution of nominally 50 $\mu\text{g/mL}$ solution. Sonication was used to ensure dissolution of the TS. This was verified by repeated LSC of the stock until reproducible figures consistent with the theoretical concentration were achieved. The stock solution was diluted with 0.01M calcium chloride to give a range of nominal concentrations of 0, 0.5, 1.0, 2.0 and 5.0 $\mu\text{g/mL}$ test solutions as required for the described experiments. Test solutions were used immediately after preparation.

Incubations were conducted using 30 mL of test solutions with 6 g soil to give a soil/solution ratio of 1/5 (w/v) with a final nominal TS concentration range of 0.05 – 5 $\mu\text{g/mL}$.

Samples were incubated in the dark at *ca* 25-26°C with shaking to ensure mixing of the soil and solution phases for the duration of the tests.

3.6 Test performance

3.6.1 Preliminary test

According to (a)“OECD 106”: Yes

To define conditions for optimal adsorption, 30 mL of nominal 5 $\mu\text{g/mL}$ Dithane test solution were incubated with 6 g of each soil type in the dark in an orbital shaker with continuous shaking at *ca* 225 rpm in 40 mL glass test vials. After 2, 4, 8 and 24 hours incubation, samples were removed from the shaker, and centrifuged to separate soil from solution. Aliquots of the supernatant were taken for LSC, the tubes were shaken by hand to ensure dispersion of the soil and the samples were returned to the shaker. The concentration of radioactivity in solution through the experiment was assessed to determine if and when equilibrium was reached to determine the duration of incubation for the adsorption phase.

3.6.2 Screening test: Adsorption

According to (a)“OECD 106”: Yes

^{14}C -Dithane in 30 mL of 0.01M calcium chloride was added to 6 g (dry weight equivalents) of soil in 40 mL glass test vials with plastic lids and teflon cap liners. Four concentrations of Dithane (0.44-4.64 $\mu\text{g/mL}$) were prepared in triplicate, the concentrations of each being confirmed by Liquid Scintillation Counting (LSC). All samples were mixed continuously for 24 hours in a horizontal position in an orbital shaker at 225 RPM at 25.6°C in the dark, after which each sample was centrifuged to separate the soil from solution. The supernatant was removed and subject to LSC to determine the quantity of TS adsorbed by the soil at each concentration studied for each soil type.

3.6.3 Screening test: Desorption

According to (a)“OECD 106”: Performed

Samples from the adsorption phase were mixed with 30 mL of fresh 0.01M calcium chloride solution and the vials were returned to the shaker. After 2 hours desorption on the shaker, each sample was centrifuged to separate the soil from solution and the supernatant was removed and subject to LSC to determine the quantity of TS desorbed from the soil. A fresh 30 mL 0.01M calcium chloride was added to each

Section A7.2.3.1(1) Adsorption / Desorption screening test**Annex Point IIIA XII.1.2****IUCLID 3.3.2/01****Batch Soil Adsorption/Desorption of Mancozeb**

vial and they were returned to the shaker. This was repeated 4, 8 and 24 hours after initiation of the desorption phase to give data on desorption periods of 2, 4, 8 and 24 hours at each concentration studied for each soil type.

3.6.4 HPLC-method

According to (a) "OECD-HPLC-method"¹: No

3.6.5 Other test

To allow estimation of the mean recovery of applied radioactivity, all residual soil samples from the desorption tests were frozen alongside the adsorption/desorption supernatants. Residual radioactivity in each soil sample was later determined by combustion in a sample oxidiser followed by LSC.

4 RESULTS**4.1 Preliminary test**

Equilibrium was essentially achieved in all soil types after 4-8 hours incubation. 24 hours was selected as the duration time for the adsorption phase. (See tables A7_1_3-2a - A7_1_3-2d and fig A7_2_3_1-1).

4.2 Screening test: Adsorption

See tables A7_1_3-3a - A7_1_3-3d.

4.3 Screening test: Desorption

See tables A7_1_3-4a - A7_1_3-4d.

4.4 Calculations

4.4.1 K_a , K_d

Soil 1: Georgia Sand (85E373) $K_a = 11.67$, $K_d = 52.71$

Soil 2: Georgia Sandy Loam (85E372) $K_a = 9.89$, $K_d = 40.84$

Soil 3: Pennsylvania Silt Loam (85E655) $K_a = 7.26$, $K_d = 27.82$

Soil 4: Mississippi Clay Loam (85E416) $K_a = 10.13$, $K_d = 41.42$

K_a and K_d figures quoted are for 24 hour adsorption and desorption phases.

4.4.2 $K_{a_{oc}}$, $K_{d_{oc}}$

Soil 1: Georgia Sand (85E373) $K_{a_{oc}} = 2334$, $K_{d_{oc}} = 10542$

Soil 2: Georgia Sandy Loam (85E372) $K_{a_{oc}} = 618$, $K_{d_{oc}} = 2552$

Soil 3: Pennsylvania Silt Loam (85E655) $K_{a_{oc}} = 363$, $K_{d_{oc}} = 1391$

Soil 4: Mississippi Clay Loam (85E416) $K_{a_{oc}} = 675$, $K_{d_{oc}} = 2761$

$K_{a_{oc}}$ and $K_{d_{oc}}$ figures quoted are for 24 hour adsorption and desorption phases.

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).

Section A7.2.3.1(1) Adsorption / Desorption screening test

Annex Point IIIA XII.1.2

IUCLID 3.3.2/01

Batch Soil Adsorption/Desorption of Mancozeb**4.5 Degradation product(s)**

Specific analysis of selected samples demonstrated that at least 90% of mancozeb, the active ingredient of Dithane, was decomposed over the 48 hour duration of the adsorption/desorption test.

Ethylenethiourea (ETU) comprised 21-47% of the adsorption supernatant radioactivity after equilibration with soil for 24 hours. No detectable ethylene urea (EU) was found in adsorption supernatants from Sand or Sandy Loam, but EU made up 28% and 12% of the radioactivity in the adsorption supernatants from Silt Loam and Clay Loam respectively.

Specific analysis for ETU in residual soil samples showed that this accounted for <1% of the initial radioactivity.

In addition to ETU and EU, TLC analyses suggested the possible presence of ethylenediamine, Jaffe's base, ethylenebisithiocyanate sulfide in the analyses of degradation products.

The material balance for test systems containing soil ranged from 93-106% of the initial dose indicating that essentially all of the dithane was accounted for in the profiling experiments.

5 APPLICANT'S SUMMARY AND CONCLUSION**5.1 Materials and methods**

A batch soil adsorption/desorption study on Mancozeb using ¹⁴C radiolabelled Dithane was conducted at a range of concentrations (0.5, 1.0, 2.0 and 5.0 ppm Dithane M-45) in sand, sandy loam, silt loam and clay loam soils in a series of individual experiments. In each experiment, 30 mL of test solution were applied to 6 g of soil. On the basis of preliminary experiments to establish time taken to reach the adsorption equilibrium, based on uptake of radioactivity into the soil, the adsorption phase was conducted for 24 hours. Subsequently 4 desorptions with fresh CaCl₂ solution (30 mL on each occasion) were conducted over a second 24 hour period. The amount of radioactivity was determined in each adsorption or desorption supernatant by liquid scintillation counting and in each soil after the final desorption step by combustion radioassay.

A separate series of experiments were conducted with 5 ppm of formulated product in each soil type to identify the quantity of Mancozeb and the number and nature of its degradates. These employed specific analysis for Mancozeb and ETU using GC and HPLC methods and profiling by TLC analyses.

5.2 Results and discussion

Due to the rapid decomposition of mancozeb under the test conditions, the calculated Freundlich constants and derived data were determined on the assumption that the radioactivity present represented only mancozeb, when in fact it represents the overall behaviour of mancozeb and its degradates.

Overall, using radioactivity as a guide, mancozeb and at least several of its degradates were strongly adsorbed. Freundlich K values for adsorption ranged from 7.3 (medium mobility) to 11.7 (slight mobility) where mobility was classified according to the McCall mobility index. Freundlich K values for desorption range from 17.8 to 103.6

5.2.1 Adsorbed a.s. [%]

Soil 1: Georgia Sand (85E373) 68.5-81.4%

Soil 2: Georgia Sandy Loam (85E372) 62.5-78.2%

Section A7.2.3.1(1) Adsorption / Desorption screening test**Annex Point IIIA XII.1.2****IUCLID 3.3.2/01****Batch Soil Adsorption/Desorption of Mancozeb**

Soil 3: Pennsylvania Silt Loam (85E655) 53.3-78.2%

Soil 4: Mississippi Clay Loam (85E416) 63.6-79.1%

Data are reported as ranges of % radioactivity adsorbed for the range of concentrations tested for each soil. These data were not included in the report but were calculated from reported results. Refer to tables A7_1_3-3a - A7_1_3-3d for details.

5.2.2 K_a

Soil 1: Georgia Sand (85E373) $K_a = 11.67$

Soil 2: Georgia Sandy Loam (85E372) $K_a = 9.89$

Soil 3: Pennsylvania Silt Loam (85E655) $K_a = 7.26$

Soil 4: Mississippi Clay Loam (85E416) $K_a = 10.13$

5.2.3 K_d

2 hours desorption

Soil 1: Georgia Sand (85E373) $K_d = 26.44$

Soil 2: Georgia Sandy Loam (85E372) $K_d = 24.55$

Soil 3: Pennsylvania Silt Loam (85E655) $K_d = 17.83$

Soil 4: Mississippi Clay Loam (85E416) $K_d = 25.60$

4 hours desorption

Soil 1: Georgia Sand (85E373) $K_d = 103.63$

Soil 2: Georgia Sandy Loam (85E372) $K_d = 36.57$

Soil 3: Pennsylvania Silt Loam (85E655) $K_d = 35.92$

Soil 4: Mississippi Clay Loam (85E416) $K_d = 43.41$

8 hours desorption

Soil 1: Georgia Sand (85E373) $K_d = 77.88$

Soil 2: Georgia Sandy Loam (85E372) $K_d = 35.93$

Soil 3: Pennsylvania Silt Loam (85E655) $K_d = 50.25$

Soil 4: Mississippi Clay Loam (85E416) $K_d = 53.58$

24 hours desorption

Soil 1: Georgia Sand (85E373) $K_d = 52.71$

Soil 2: Georgia Sandy Loam (85E372) $K_d = 40.84$

Soil 3: Pennsylvania Silt Loam (85E655) $K_d = 27.82$

Soil 4: Mississippi Clay Loam (85E416) $K_d = 41.42$

5.2.4 $K_{a_{oc}}$

Soil 1: Georgia Sand (85E373) $K_{a_{oc}} = 2334$

Soil 2: Georgia Sandy Loam (85E372) $K_{a_{oc}} = 618$

Soil 3: Pennsylvania Silt Loam (85E655) $K_{a_{oc}} = 363$

Soil 4: Mississippi Clay Loam (85E416) $K_{a_{oc}} = 675$

Section A7.2.3.1(1) Adsorption / Desorption screening test**Annex Point IIIA XII.1.2****IUCLID 3.3.2/01****Batch Soil Adsorption/Desorption of Mancozeb**

5.2.5	Ka/Kd	Soil 1: Georgia Sand (85E373)	Ka/Kd = 0.22
		Soil 2: Georgia Sandy Loam (85E372)	Ka/Kd = 0.24
		Soil 3: Pennsylvania Silt Loam (85E655)	Ka/Kd = 0.26
		Soil 4: Mississippi Clay Loam (85E416)	Ka/Kd = 0.24
		These data were not included in the report but were calculated here on the basis of the 24 h adsorption/desorption Ka/Kd.	
5.2.6	Degradation products (% of a.s.)	Soil 1: Georgia Sand (85E373)	ETU = 47%
		Soil 2: Georgia Sandy Loam (85E372)	ETU = 21%
		Soil 3: Pennsylvania Silt Loam (85E655)	ETU = 26%
		Soil 4: Mississippi Clay Loam (85E416)	ETU = 26%
5.3	Conclusion	Overall, using radioactivity as a guide, mancozeb and several of its degradates were strongly adsorbed. Freundlich K values for adsorption ranged from 7.3 (medium mobility) to 11.7 (slight mobility) when classified according to the McCall mobility index.	
		Freundlich K values for desorption ranged from 17.8 to 103.6.	
		The Freundlich linearity coefficients deviated from the ideal value of 1.0, which may be a reflection of the multiple compounds and modes of adsorption thereof: the total radioactivity data give a composite representation for the adsorption/desorption behaviour of mancozeb and its subsequent breakdown products.	
5.3.1	Reliability	1	
5.3.2	Deficiencies	No	

Evaluation by Competent Authorities

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

EVALUATION BY RAPPORTEUR MEMBER STATE

Date	<i>Give date of action</i>
Materials and Methods	<i>State if the applicants version is acceptable or indicate relevant discrepancies referring to the (sub) heading numbers and to applicant's summary and conclusion.</i>
Results and discussion	<i>Adopt applicant's version or include revised version. If necessary, discuss relevant deviations from applicant's view referring to the (sub)heading numbers</i>
Conclusion	<i>Adopt applicant's version or include revised version</i>
Reliability	<i>Based on the assessment of materials and methods include appropriate reliability indicator</i>
Acceptability	acceptable / not acceptable <i>(give reasons if necessary, e.g. if a study is considered acceptable despite a poor reliability indicator. Discuss the relevance of deficiencies and indicate if repeat is necessary.)</i>

Section A7.2.3.1(1) Adsorption / Desorption screening test

Annex Point IIIA XII.1.2

IUCLID 3.3.2/01

Batch Soil Adsorption/Desorption of Mancozeb

Remarks	
	COMMENTS FROM ...
Date	<i>Give date of comments submitted</i>
Materials and Methods	<i>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</i>
Results and discussion	<i>Discuss if deviating from view of rapporteur member state</i>
Conclusion	<i>Discuss if deviating from view of rapporteur member state</i>
Reliability	<i>Discuss if deviating from view of rapporteur member state</i>
Acceptability	<i>Discuss if deviating from view of rapporteur member state</i>
Remarks	

Table A7_1_3-1: Classification and physico-chemical properties of soils used as adsorbents

	Soil 1 85E373	Soil 2 85E372	Soil 3 85E655	Soil 4 85E416
Soil order	Not reported	Not reported	Not reported	Not reported
Soil series	Not reported	Not reported	Not reported	Not reported
Classification	Sand	Sandy Loam	Silt Loam	Clay Loam
Location	Georgia	Georgia	Pennsylvania	Mississippi
Horizon	Not reported	Not reported	Not reported	Not reported
Sand [%]	88	78	14	22
Silt [%]	8	10	66	50
Clay [%]	4	12	20	28
Organic carbon [%]	0.5	1.6	2.0	1.5
Carbonate as CaCO ₃	Not reported	Not reported	Not reported	Not reported
insoluble carbonates [%]	Not reported	Not reported	Not reported	Not reported
pH (1:1 H ₂ O)	5.7	5.9	6.4	7.4
Cation exchange capacity (MEQ/100 g)	3.5	5.7	9.6	12.9
Extractable cations (MEQ/100 g)	-	-	-	-
Ca	Not reported	Not reported	Not reported	Not reported
Mg	Not reported	Not reported	Not reported	Not reported
Na	Not reported	Not reported	Not reported	Not reported
K	Not reported	Not reported	Not reported	Not reported
H	Not reported	Not reported	Not reported	Not reported
Special chemical/mineralogical features	None reported	None reported	None reported	None reported
Clay fraction mineralogy	Not reported	Not reported	Not reported	Not reported

Table A7_1_3-2: Results of preliminary test:

Test substance	¹⁴ C-Dithane
Sample purity	The radiopurity was 88%
Weighed soil	6 g
Volume of CaCl ₂ solution	30 mL
Nominal concentration of a.s. final solution	5.0 µg/mL
Analytical concentration final of a.s. solution	Data not reported
Concentration of the test solution (show calculation)	Data not reported
Details of the analytical method used:	
Method	Liquid Scintillation Counting (LSC) of Supernatant
Recovery rate	No figures reported, refer to fig 1 for summary
Detection limit	Data not reported

Table A7_1_3-3a: Results of screening test – adsorption for Soil 1 (85E373):

	Nominal 5.0 µg/mL		Nominal 2.0 µg/mL		Nominal 1.0 µg/mL		Nominal 0.5 µg/mL	
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
Concentration of test material [mg/l]	1.44	0.09	0.50	0.02	0.20	0.01	0.08	0.01
After contact of....hours with soil	24		24		24		24	
Correction for blank with soil	The study was conducted with radiolabelled material, therefore no correction of the concentration of test material was necessary for blanks either with or without soil. Final corrected concentration therefore = measured concentration							
Correction for blank without soil								
Final corrected concentration [mg/l]	1.44	0.09	0.50	0.02	0.20	0.01	0.08	0.01
Initial concentration of test solution [mg/l]	4.64		1.80		0.88		0.44	
Decrease in concentration [mg/l]	Not reported							
Quantity adsorbed [µg]	Not reported							
Quantity of soil [g of oven-dried equivalent]	6		6		6		6	
Quantity adsorbed [µg] per gram of soil	15.89	0.53	6.60	0.01	3.43	0.04	1.79	0.03
Test material adsorbed [%] *	68.5*	-	73.3*	-	78.0*	-	81.4*	-
Temperature [°C]	25.6		25.6		25.6		25.6	
Volume of solution recovered after centrifugation [ml]	Not reported							
Volume of solution not recovered [ml]	Not reported							
Corresponding quantity of test substance [mg]	Not reported							

*Not detailed in report, but calculated here by multiplying quantity adsorbed per g of soil by 6 (g of soil in incubation) and expressing this as a percentage of µg TS present in 30 mL of solution as determined from the starting concentration.

Table A7_1_3-3b: Results of screening test – adsorption for Soil 2 (85E372):

	Nominal 5.0 µg/mL		Nominal 2.0 µg/mL		Nominal 1.0 µg/mL		Nominal 0.5 µg/mL	
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
Concentration of test material [mg/l]	1.73	0.07	0.53	0.04	0.23	0.01	0.10	0.00
After contact of....hours with soil	24		24		24		24	
Correction for blank with soil	The study was conducted with radiolabelled material, therefore no correction of the concentration of test material was necessary for blanks either with or without soil. Final corrected concentration therefore = measured concentration							
Correction for blank without soil								
Final corrected concentration [mg/l]	1.73	0.07	0.53	0.04	0.23	0.01	0.10	0.00
Initial concentration of test solution [mg/l]	4.64		1.80		0.88		0.44	
Decrease in concentration [mg/l]	Not reported							
Quantity adsorbed [µg]	Not reported							
Quantity of soil [g of oven-dried equivalent]	6		6		6		6	
Quantity adsorbed [µg] per gram of soil	14.51	0.27	6.39	0.21	3.31	0.02	1.72	0.01
Test material adsorbed [%] *	62.5*	-	71.0*	-	75.2*	-	78.2*	-
Temperature [°C]	25.6		25.6		25.6		25.6	
Volume of solution recovered after centrifugation [ml]	Not reported							
Volume of solution not recovered [ml]	Not reported							
Corresponding quantity of test substance [mg]	Not reported							

* Not detailed in report, but calculated here by multiplying quantity adsorbed per g of soil by 6 (g of soil in incubation) and expressing this as a percentage of µg TS present in 30 mL of solution as determined from the starting concentration.

Table A7_1_3-3c: Results of screening test – adsorption for Soil 3 (85E655):

	Nominal 5.0 µg/mL		Nominal 2.0 µg/mL		Nominal 1.0 µg/mL		Nominal 0.5 µg/mL	
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
Concentration of test material [mg/l]	2.34	0.02	0.68	0.06	0.30	0.01	0.13	0.01
After contact of....hours with soil	24		24		24		24	
Correction for blank with soil	The study was conducted with radiolabelled material, therefore no correction of the concentration of test material was necessary for blanks either with or without soil. Final corrected concentration therefore = measured concentration							
Correction for blank without soil								
Final corrected concentration [mg/l]	2.34	0.02	0.68	0.06	0.30	0.01	0.13	0.01
Initial concentration of test solution [mg/l]	4.64		1.80		0.88		0.44	
Decrease in concentration [mg/l]	Not reported							
Quantity adsorbed [µg]	Not reported							
Quantity of soil [g of oven-dried equivalent]	6		6		6		6	
Quantity adsorbed [µg] per gram of soil	12.36	0.32	6.06	0.34	3.20	0.04	1.72	0.04
Test material adsorbed [%] *	53.3*	-	67.3*	-	72.7*	-	78.2*	-
Temperature [°C]	25.6		25.6		25.6		25.6	
Volume of solution recovered after centrifugation [ml]	Not reported							
Volume of solution not recovered [ml]	Not reported							
Corresponding quantity of test substance [mg]	Not reported							

* Not detailed in report, but calculated here by multiplying quantity adsorbed per g of soil by 6 (g of soil in incubation) and expressing this as a percentage of µg TS present in 30 mL of solution as determined from the starting concentration..

Table A7_1_3-3d: Results of screening test – adsorption for Soil 4 (85E416):

	Nominal 5.0 µg/mL		Nominal 2.0 µg/mL		Nominal 1.0 µg/mL		Nominal 0.5 µg/mL	
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
Concentration of test material [mg/l]	1.87	0.02	0.49	0.01	0.24	0.00	0.12	0.01
After contact of....hours with soil	24		24		24		24	
Correction for blank with soil	The study was conducted with radiolabelled material, therefore no correction of the concentration of test material was necessary for blanks either with or without soil. Final corrected concentration therefore = measured concentration							
Correction for blank without soil								
Final corrected concentration [mg/l]	1.87	0.02	0.49	0.01	0.24	0.00	0.12	0.01
Initial concentration of test solution [mg/l]	4.64		1.80		0.88		0.44	
Decrease in concentration [mg/l]	Not reported							
Quantity adsorbed [µg]	Not reported							
Quantity of soil [g of oven-dried equivalent]	6		6		6		6	
Quantity adsorbed [µg] per gram of soil	14.75	0.12	7.08	0.10	3.48	0.05	1.72	0.08
Test material adsorbed [%] *	63.6*	-	78.7*	-	79.1*	-	78.2*	-
Temperature [°C]	25.6		25.6		25.6		25.6	
Volume of solution recovered after centrifugation [ml]	Not reported							
Volume of solution not recovered [ml]	Not reported							
Corresponding quantity of test substance [mg]	Not reported							

*Not detailed in report, but calculated here by multiplying quantity adsorbed per g of soil by 6 (g of soil in incubation) and expressing this as a percentage of µg TS present in 30 mL of solution as determined from the starting concentration.

Table A7_1_3-4a: Results of screening test – 24 hour desorption for Soil 1 (85E373):

	Nominal 5.0 µg/mL	Nominal 2.0 µg/mL	Nominal 1.0 µg/mL	Nominal 0.5 µg/mL
	Mean	Mean	Mean	Mean
Temperature [°C]	25.6	25.6	25.6	25.6
Concentration in combined washings [mg/l] ¹	0.75	0.22	0.12	0.06
Corresponding quantity of test material [mg]	0.0225	0.0066	0.0036	0.0018
Quantity desorbed [µg] ²	25.32	6.06	3.36	1.62
[%] of adsorbed test material, which is desorbed ³	26.6	15.3	16.3	15.1
[%] of adsorbed test material, which is not desorbed ⁴	73.4	84.7	83.7	84.9

¹ Not detailed in report. Quantity desorbed was calculated here from addition of concentrations in desorption supernatant after, 2, 4, 8 and 24 hours

² Not detailed in report. Quantity desorbed was calculated here from: ((µg/g in soil at end of adsorption phase) – (µg/g in soil at end of desorption phase)) x 6

³ Not detailed in report. % desorbed was calculated here from:
$$\frac{(\mu\text{g/g in soil at end of adsorption phase} - \mu\text{g/g in soil at end of desorption phase})}{(\mu\text{g/g in soil at end of adsorption phase})} \times 100$$

⁴ Not detailed in report. % not desorbed was calculated here from:
$$\frac{(\mu\text{g/g in soil at end of desorption phase})}{(\mu\text{g/g in soil at end of adsorption phase})} \times 100$$

Table A7_1_3-4b: Results of screening test – 24 hour desorption for Soil 2 (85E372):

	Nominal 5.0 µg/mL	Nominal 2.0 µg/mL	Nominal 1.0 µg/mL	Nominal 0.5 µg/mL
	Mean	Mean	Mean	Mean
Temperature [°C]	25.6	25.6	25.6	25.6
Concentration in combined washings [mg/l] ¹	0.92	0.34	0.14	0.06
Corresponding quantity of test material [mg]	0.0276	0.0102	0.0042	0.0018
Quantity desorbed [µg] ²	27.00	9.24	3.78	1.56
[%] of adsorbed test material, which is desorbed ³	31.0	24.1	19.0	15.1
[%] of adsorbed test material, which is not desorbed ⁴	69.0	75.9	81.0	84.9

¹ Not detailed in report. Quantity desorbed was calculated here from addition of concentrations in desorption supernatant after, 2, 4, 8 and 24 hours

² Not detailed in report. Quantity desorbed was calculated here from: $((\mu\text{g/g in soil at end of adsorption phase}) - (\mu\text{g/g in soil at end of desorption phase})) \times 6$

³ Not detailed in report. % desorbed was calculated here from:
$$\frac{(\mu\text{g/g in soil at end of adsorption phase} - \mu\text{g/g in soil at end of desorption phase})}{(\mu\text{g/g in soil at end of adsorption phase})} \times 100$$

⁴ Not detailed in report. % not desorbed was calculated here from:
$$\frac{(\mu\text{g/g in soil at end of desorption phase})}{(\mu\text{g/g in soil at end of adsorption phase})} \times 100$$

Table A7_1_3-4c: Results of screening test – 24 hour desorption for Soil 3 (85E655):

	Nominal 5.0 µg/mL	Nominal 2.0 µg/mL	Nominal 1.0 µg/mL	Nominal 0.5 µg/mL
	Mean	Mean	Mean	Mean
Temperature [°C]	25.6	25.6	25.6	25.6
Concentration in combined washings [mg/l] ¹	0.85	0.28	0.13	0.05
Corresponding quantity of test material [mg]	0.0255	0.0084	0.0039	0.0015
Quantity desorbed [µg] ²	24.06	7.68	3.54	1.2
[%] of adsorbed test material, which is desorbed ³	32.4	21.1	18.4	11.6
[%] of adsorbed test material, which is not desorbed ⁴	67.6	78.9	81.6	88.4

¹ Not detailed in report. Quantity desorbed was calculated here from addition of concentrations in desorption supernatant after, 2, 4, 8 and 24 hours

² Not detailed in report. Quantity desorbed was calculated here from: ((µg/g in soil at end of adsorption phase) – (µg/g in soil at end of desorption phase)) × 6

³ Not detailed in report. % desorbed was calculated here from:
$$\frac{(\mu\text{g/g in soil at end of adsorption phase} - \mu\text{g/g in soil at end of desorption phase})}{(\mu\text{g/g in soil at end of adsorption phase})} \times 100$$

⁴ Not detailed in report. % not desorbed was calculated here from:
$$\frac{(\mu\text{g/g in soil at end of desorption phase})}{(\mu\text{g/g in soil at end of adsorption phase})} \times 100$$

Table A7_1_3-4d: Results of screening test – 24 hour desorption for Soil 4 (85E416):

	Nominal 5.0 µg/mL	Nominal 2.0 µg/mL	Nominal 1.0 µg/mL	Nominal 0.5 µg/mL
	Mean	Mean	Mean	Mean
Temperature [°C]	25.6	25.6	25.6	25.6
Concentration in combined washings [mg/l] ¹	0.97	0.31	0.16	0.07
Corresponding quantity of test material [mg]	0.0291	0.0093	0.0048	0.0021
Quantity desorbed [µg] ²	27.1	8.52	4.14	1.74
[%] of adsorbed test material, which is desorbed ³	30.6	20.1	19.8	16.9
[%] of adsorbed test material, which is not desorbed ⁴	69.4	79.9	80.2	83.1

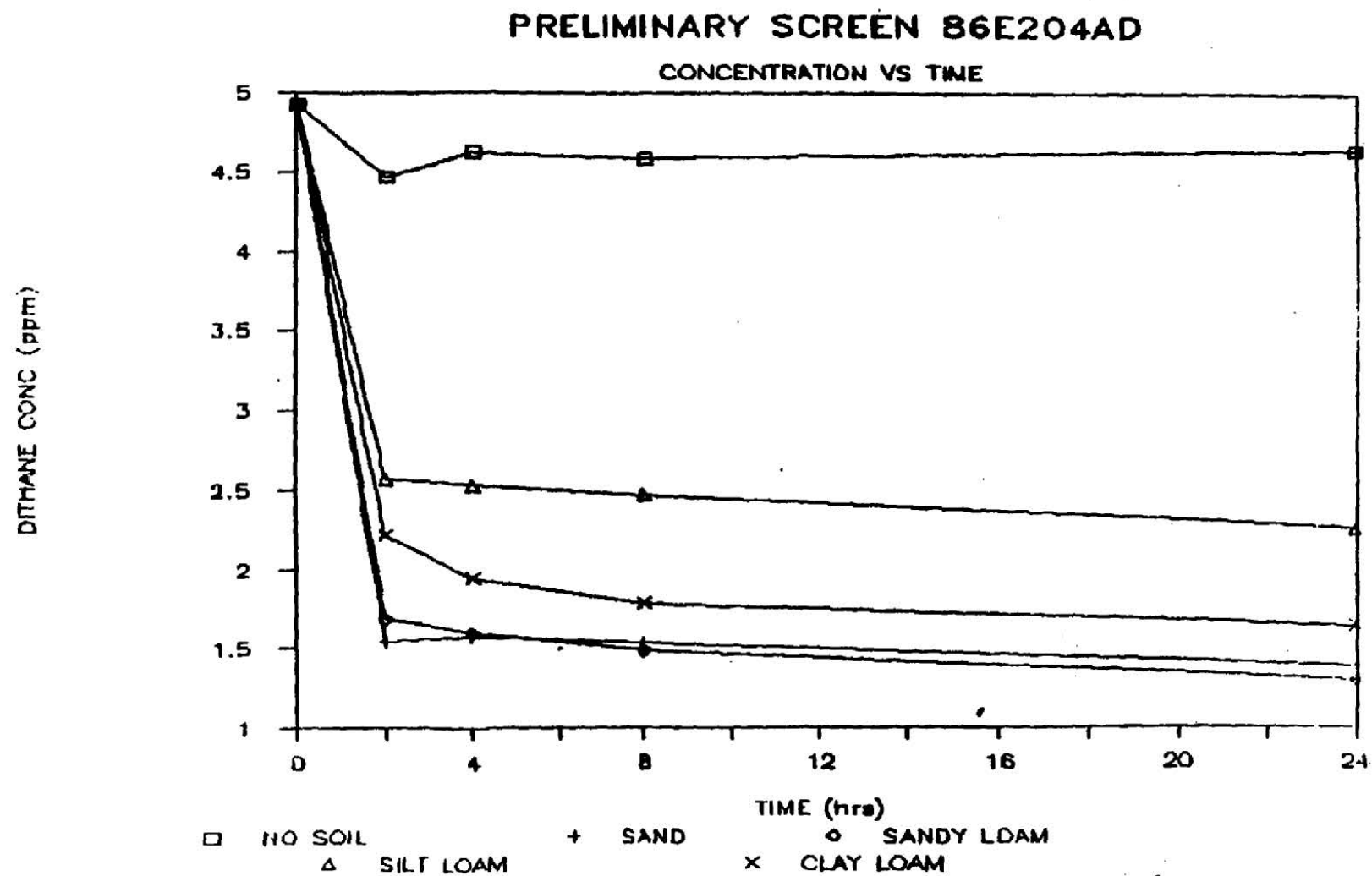
¹ Not detailed in report. Quantity desorbed was calculated here from addition of concentrations in desorption supernatant after, 2, 4, 8 and 24 hours

² Not detailed in report. Quantity desorbed was calculated here from: $((\mu\text{g/g in soil at end of adsorption phase}) - (\mu\text{g/g in soil at end of desorption phase})) \times 6$

³ Not detailed in report. % desorbed was calculated here from:
$$\frac{(\mu\text{g/g in soil at end of adsorption phase} - \mu\text{g/g in soil at end of desorption phase})}{(\mu\text{g/g in soil at end of adsorption phase})} \times 100$$

⁴ Not detailed in report. % not desorbed was calculated here from:
$$\frac{(\mu\text{g/g in soil at end of desorption phase})}{(\mu\text{g/g in soil at end of adsorption phase})} \times 100$$

Figure A7_1_3-1: Results of Preliminary Screening Test for Soils 1-4



Section A7.2.3.1(2)

Annex Point IIIA XII.1.2

IUCLID 3.3.2/03

Adsorption / Desorption screening test

¹⁴C)-Ethylenethiourea, a metabolite of Mancozeb:
Adsorption/Desorption in SoilOfficial
use only**1 REFERENCE****1.1 Reference**

Yeh, S.M., (1986b), Determination of Soil Adsorption/Desorption constants for Ethylene thiourea, performed by Biospherics, Inc., 4928 Wyaconda Road, Rockville, MD 20852, USA, for Rohm and Haas Company, 727 Norriston Road, Spring House, PA 19477, Technical Report No. 310-86-63, December 17, 1986.

1.2 Data protection

Yes

1.2.1 Data owner

Cerexagri B.V./Dow/BASF

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**

EPA Adsorption – Desorption Using a Batch Equilibrium Method

2.2 GLP

Yes

2.3 Deviations

No

3 MATERIALS AND METHODS**3.1 Test material**¹⁴C)-Ethylenethiourea (¹⁴C-ETU)

3.1.1 Lot/Batch number

¹⁴C : 541.01

3.1.2 Specification

The test substance is a metabolite of Mancozeb

3.1.3 Purity

¹⁴C : 99%

3.1.4 Further relevant properties

Specific activity of ¹⁴C radiolabelled material was 10.08 mCi/mg

3.1.5 Method of analysis

TLC methodPlate: Merck Kieselgel 60 F₂₅₄

Solvent: ethylacetate: methanol: water (65:25:10 v/v)

Radiolabelled compounds were detected by preparation of a radioluminogram of the TLC plate using a Bioscan Autochanger 3000 with Bioscan System 200 Imaging Scanner.

Samples were co-chromatographed with non-radiolabelled reference standard, which was visualised with fluorescamine, FN reagent and Ehrlich's reagent.

3.2 Degradation products

Degradation products tested: Ethyleneurea

3.2.1 Method of analysis for degradation products

TLC method above

3.3 Reference substance

Ethylene urea (not labelled) supplied by Rohm and Haas

Section A7.2.3.1(2)**Adsorption / Desorption screening test****Annex Point IIIA XII.1.2****(¹⁴C)-Ethylenethiourea, a metabolite of Mancozeb:
Adsorption/Desorption in Soil****IUCLID 3.3.2/03**

3.3.1	Method of analysis for reference substance	Not applicable
3.4	Soil types	<p>4 soil types, classified as per USDA, were used:-</p> <p>Soil 1: Georgia sand</p> <p>Soil 2: Georgia sandy loam</p> <p>Soil 3: Pennsylvania silt loam</p> <p>Soil 4: Mississippi clay loam</p> <p>Analyses were performed by the Soil Testing Laboratory , University of Maryland , College park , Maryland</p> <p>Refer to table A7_1 _3-1 for full details</p> <p>At the initiation of the experiment moisture content of the soil samples were determined.</p>
3.5	Testing procedure	
3.5.1	Test system	<p>The test system was comprised of soils, mixed with ¹⁴C test material dissolved in 0.01M calcium chloride. The samples were held in centrifuge tubes and incubated in the dark at 25.8 °C whilst being mixed on an orbital shaker.</p> <p>LSC of liquid samples was conducted using a Beckman LS-3801 Liquid Scintillation Counter (LSC). Counting was performed for 5 min per sample. Counting efficiencies were computed using factory standard quench sample data.</p> <p>Radioactivity in solid residues was determined by LSC following combustion in a R.J.Harvey Biological Materials Oxidizer and trapping of the resulting ¹⁴CO₂ in Harvey ¹⁴C Cocktail.</p>
3.5.2	Test solution and Test conditions	<p>A stock solution of ¹⁴C ethylenethiourea was prepared by dissolving 49.6 mg ¹⁴C-ETU in 10 mL of 0.01M calcium chloride to give a 4.96 µg/mL solution. The stock was stored frozen and thawed for each use.</p> <p>Dose solutions were prepared at 2.0 ppm for the preliminary screen and 0., 0.2, 0.5, 1.0 and 2.0 ppm for the adsorption study. Concentrations of the stock and dose solutions were confirmed with LSC measurements. Samples were incubated in the dark at 25.8 °C and shaken continuously for the duration of the tests, except when aliquots were taken for LSC.</p>
3.6	Test performance	
3.6.1	Preliminary test	No
3.6.2	Screening test: Adsorption	<p>According to (a)"EPA????": Yes</p> <p>To determine the time necessary to reach dynamic equilibrium during the adsorption phase concentrations of 2 ppm ETU was tested over 24 hours. Aliquoting was performed at 2, 4, 8, and 24 hours after dosing. Samples were removed from the shaker , centrifuged at 1000 rpm for 5 mi, 100 µl aliquots were added to 10 ml Maxifluor (Baker), shaken and counted by LSC. The equilibrium was reached between 4 and 8 hrs. 24 hours was selected as duration time of the adsorption phase.</p>
3.6.3	Screening test:	No

Section A7.2.3.1(2)**Annex Point IIIA XII.1.2****IUCLID 3.3.2/03****Adsorption / Desorption screening test****(¹⁴C)-Ethylenethiourea, a metabolite of Mancozeb:
Adsorption/Desorption in Soil**

	Desorption	
3.6.4	TLC-method	According to TLC method chapter 3.1.5
3.6.5	Other test	<p>The solubility of ethylene urea in 0.01 M calcium chloride at 5 µg/mL was confirmed by LSC after centrifugation to remove any potentially undissolved material.</p> <p>The potential for ethylene urea to adsorb onto containers was assessed by incubation of 25 mL of 0.05 µg/mL ethylene urea in 0.01 M calcium chloride in both polypropylene and teflon tubes.</p> <p>Tests to assess the effect of various incubation periods of up to 48 hours for the conduct of the adsorption/desorption phases were conducted.</p> <p>The stability of ethylene urea in each soil type was assured by the incubation of 30 mL of 2 ppm ethylenethio urea in 0.01 M calcium chloride with 6 g of soil for 24 hours, after which the supernatant and soil were separated. The soil was washed with 0.01 M calcium chloride and thoroughly extracted with acetonitrile/water, acetonitrile, methanol and water, after which all of the extracts were combined with the supernatants and the total recovery was assessed. For soils 1 and 4 (SK 961089 and SK 920191 respectively) an additional series of extractions with aqueous trifluoroacetic acid (0.1%) was applied because the recovery of total radioactivity was less than 90% to this point. The combined washings and supernatants were concentrated to a small volume for radio-profiling by HPLC as detailed in section 3.1.5.</p> <p>To allow estimation of the mean recovery of applied radioactivity, all residual soil samples from the desorption tests were air dried prior to quantification by combustion in a sample oxidiser followed by LSC.</p>

4 RESULTS

4.1	Preliminary test	A 1:5 ratio of soil/aqueous (w/v, 6g/30ml) was selected on the basis of providing the maximum adsorption for the adsorption/desorption tests. See tables A7_1_3-2a - A7_1_3-2d.	
4.2	Screening test: Adsorption	See tables A7_1_3-3a - A7_1_3-3d	
4.3	Screening test: Desorption	See tables A7_1_3-4a - A7_1_3-4d	
4.4	Calculations		
4.4.1	K _a , K _d	Soil 1: Clay loam (SK 961089)	K _a = 0.22, K _d = 0.40
		Soil 2: Loam (SK 179618)	K _a = 0.16, K _d = 0.20
		Soil 3: Loamy sand (SK 566696)	K _a = 0.15, K _d = 0.29
		Soil 4: Clay loam (SK 920191)	K _a = 0.22, K _d = 0.42
4.4.2	K _{aoc} , K _{doc}	Soil 1: Clay loam (SK 961089)	K _{aoc} = 5, K _{doc} = 9
		Soil 2: Loam (SK 179618)	K _{aoc} = 4, K _{doc} = 5
		Soil 3: Loamy sand (SK 566696)	K _{aoc} = 19, K _{doc} = 36
		Soil 4: Clay loam (SK 920191)	K _{aoc} = 11, K _{doc} = 20

Section A7.2.3.1(2)

Annex Point IIIA XII.1.2

IUCLID 3.3.2/03

Adsorption / Desorption screening test**(¹⁴C)-Ethylenethiourea, a metabolite of Mancozeb:
Adsorption/Desorption in Soil****4.5 Degradation product(s)**

A test for breakdown products was conducted in advance of the preliminary test (see section 5.2). No degradation products were characterised for any of the soils under the conditions of the test (see section 5.2.6).

5.1 Materials and methods**5 APPLICANT'S SUMMARY AND CONCLUSION**

A GLP-compliant study was carried out in which the adsorption/desorption characteristics of (¹⁴C)-ethylene urea were determined in four soil types. The study was conducted in accordance with the requirements of OECD Guideline 106.

All experiments were performed in the dark at 20±2°C using autoclaved 0.01M CaCl. The air-dried soils were sterilised by gamma irradiation and preconditioned by mixing overnight with 0.01M CaCl.

Following a preliminary test, a full adsorption/desorption test was carried out by adding aliquots of (¹⁴C)-ethylene urea in 0.01M CaCl at concentrations of 5, 2.5, 0.5, 0.25 and 0.05 µg/ml to duplicate samples of the preconditioned soils and 0.01 M CaCl. The concentration of radioactivity in the doses was determined by LSC and used to calculate the quantity of ethylene urea applied to each sample.

Samples were mixed for 24 hours and centrifuged to separate the soil and solution. Radioactivity in the supernatant was determined by LSC. The supernatant was then replaced by fresh 0.01 M CaCl for the desorption phase, mixed for 24 hours, centrifuged and the radioactivity in the supernatant determined by LSC.

Soil residues were air-dried prior to quantification by combustion. The mean recovery of applied radioactivity for each soil type was in the range 97 to 102%.

5.2 Results and discussion

The solubility of ethylene urea in 0.01 M calcium chloride at 5 µg/mL was confirmed.

No adsorption to either polypropylene or teflon tubes was observed on the basis of LSC determinations following incubation of 25 mL of 0.05 µg/mL ethylene urea in 0.01 M calcium chloride in both types of tube. Polypropylene tubes were used throughout the study.

24 Hours incubation was selected as suitable for the conduct of the adsorption/desorption phases.

Ethylene urea was stable under the conditions of the test for at least 48 hours.

Freundlich adsorption constants (K) calculated by linear regression analysis for soils SK 961089 (clay loam), SK 179618 (loam), SK 566696 (loamy sand) and SK 920191 (clay loam) were 0.22, 0.16, 0.15, and 0.22 respectively.

Freundlich adsorption constants related to organic carbon content (K) for soils SK 961089 (clay loam), SK 179618 (loam), SK 566696 (loamy sand) and SK 920191 (clay loam) were 5, 4, 19 and 11 respectively.

Adsorption and desorption isotherms showed a strong positive monotonic relationship between log C and log X/m, indicated by the correlation coefficients being close to 1. Mass balance results

Section A7.2.3.1(2)

Adsorption / Desorption screening test

Annex Point IIIA XII.1.2

¹⁴C)-Ethylenethiourea, a metabolite of Mancozeb:
Adsorption/Desorption in Soil

IUCLID 3.3.2/03

demonstrated that mean recoveries of applied radioactivity from each soil type were in the range 97 to 102%.

The desorption equilibrium was attained within twice the time of the adsorption equilibrium but the total desorbed was <75% of the amount adsorbed, therefore the adsorption is not considered to be reversible.

5.2.1	Adsorbed a.s. [%]	Soil 1: Clay loam (SK 961089)	13.4-19.1%
		Soil 2: Loam (SK 179618)	9.8-13.0%
		Soil 3: Loamy sand (SK 566696)	10.3-16.6%
		Soil 4: Clay loam (SK 920191)	16.3-44.7%*

Data are reported as ranges of mean % ethylene urea adsorbed for the range of concentrations tested for each soil. Refer to tables A7_1_3-3a - A7_1_3-3d for full details.

* The upper range for SK 920191 may be misleading because of poor duplication: replicates were 20.0% and 69.3%. The latter figure was discounted in the regression analysis to calculate the adsorption constant, but included in the reported means.

5.2.2	K_a	Soil 1: Clay loam (SK 961089)	$K_a = 0.22$
		Soil 2: Loam (SK 179618)	$K_a = 0.16$
		Soil 3: Loamy sand (SK 566696)	$K_a = 0.15$
		Soil 4: Clay loam (SK 920191)	$K_a = 0.22$

5.2.3	K_d	Soil 1: Clay loam (SK 961089)	$K_d = 0.40$
		Soil 2: Loam (SK 179618)	$K_d = 0.20$
		Soil 3: Loamy sand (SK 566696)	$K_d = 0.29$
		Soil 4: Clay loam (SK 920191)	$K_d = 0.42$

5.2.4	$K_{a_{oc}}$	Soil 1: Clay loam (SK 961089)	$K_{a_{oc}} = 5$
		Soil 2: Loam (SK 179618)	$K_{a_{oc}} = 4$
		Soil 3: Loamy sand (SK 566696)	$K_{a_{oc}} = 19$
		Soil 4: Clay loam (SK 920191)	$K_{a_{oc}} = 11$

5.2.5	Ka/Kd	Soil 1: Clay loam (SK 961089)	$K_a/K_d = 0.55^*$
		Soil 2: Loam (SK 179618)	$K_a/K_d = 0.80^*$
		Soil 3: Loamy sand (SK 566696)	$K_a/K_d = 0.52^*$
		Soil 4: Clay loam (SK 920191)	$K_a/K_d = 0.52^*$

*Derived from reported data

5.2.6	Degradation products (% of a.s.)	Soil 1: Clay loam (SK 961089)	<9.7%**
		Soil 2: Loam (SK 179618)	<4.8%**
		Soil 3: Loamy sand (SK 566696)	<8.3%**
		Soil 4: Clay loam (SK 920191)	<11.4%**

**Not reported, but inferred as a worst case on the basis of % applied radioactivity not recovered in the stability test. This fraction of

Section A7.2.3.1(2)

Adsorption / Desorption screening test

Annex Point IIIA XII.1.2

¹⁴C)-Ethylenethiourea, a metabolite of Mancozeb:
Adsorption/Desorption in Soil

IUCLID 3.3.2/03

radioactivity was not characterised and could represent irreversibly adsorbed TS. Of the recovered radioactivity, radio-HPLC analysis of breakdown products not associated with ethylene urea gave figures of 0.01%, 1.24%, 2.05% and 0.24% for soils 1, 2, 3 and 4 respectively, none of which was present as a discrete chemical entity.

5.3 Conclusion

The validity criteria of the study are considered as being fulfilled.

Adsorption and desorption isotherms showed a strong positive monotonic relationship between the log of the concentrations of ethylene urea adsorbed to the soil, indicated by correlation coefficients close to 1.

The desorption equilibrium was attained within twice the time of the adsorption equilibrium but the total desorbed was <75% of the amount adsorbed, therefore the adsorption is not considered to be reversible.

5.3.1 Reliability

1

5.3.2 Deficiencies

No

Evaluation by Competent Authorities

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

EVALUATION BY RAPPORTEUR MEMBER STATE

Date

Give date of action

Materials and Methods

State if the applicants version is acceptable or indicate relevant discrepancies referring to the (sub) heading numbers and to applicant's summary and conclusion.

Results and discussion

Adopt applicant's version or include revised version. If necessary, discuss relevant deviations from applicant's view referring to the (sub)heading numbers

Conclusion

Adopt applicant's version or include revised version

Reliability

Based on the assessment of materials and methods include appropriate reliability indicator

Acceptability

acceptable / not acceptable

(give reasons if necessary, e.g. if a study is considered acceptable despite a poor reliability indicator. Discuss the relevance of deficiencies and indicate if repeat is necessary.)

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

Section A7.2.3.1(2)	Adsorption / Desorption screening test
Annex Point IIIA XII.1.2	(¹⁴C)-Ethylenethiourea, a metabolite of Mancozeb:
IUCLID 3.3.2/03	Adsorption/Desorption in Soil

Reliability	<i>Discuss if deviating from view of rapporteur member state</i>
Acceptability	<i>Discuss if deviating from view of rapporteur member state</i>
Remarks	

Table A7_2_3_1-1: Classification and physico-chemical properties of soils used as adsorbents

	Soil 1 SK 961089	Soil 2 SK 179618	Soil 3 SK 566696	Soil 4 SK 920191
Soil order	Not reported	Not reported	Not reported	Not reported
Soil series	Not reported	Not reported	Not reported	Not reported
Classification	Clay Loam (USDA)	Loam (USDA)	Loamy Sand (USDA)	Clay Loam (USDA)
Location	Chapel Hill Farm, Empingham, Rutland, UK	Kenslow Farm, Middleton, Derbyshire, UK	Grid Ref SK566696, Warsop, Nottinghamshire, UK	Grid Ref SK920191, South Witham Quarry, South Witham, Lincolnshire, UK
Horizon	15-30 cm	5-20 cm	12-20 cm	5-20 cm
Sand [%]	38 (USDA)	34 (USDA)	85 (USDA)	38 (USDA)
Silt [%]	28 (USDA)	46 (USDA)	4 (USDA)	26 (USDA)
Clay [%]	34 (USDA)	20 (USDA)	11 (USDA)	36 (USDA)
Organic carbon [%]	4.6	3.8	0.8	2.1
Carbonate as CaCO ₃	187.6 mg/kg	Not reported	Not reported	Not reported
insoluble carbonates [%]	Not reported	Not reported	Not reported	Not reported
pH (1:1 H ₂ O)	8.0	6.0	5.1	8.0
Cation exchange capacity (MEQ/100 g)	38.2	24.9	13.4	23.0
Extractable cations (MEQ/100 g)	-	-	-	-
Ca	Not reported	Not reported	Not reported	Not reported
Mg	Not reported	Not reported	Not reported	Not reported
Na	Not reported	Not reported	Not reported	Not reported
K	Not reported	Not reported	Not reported	Not reported
H	Not reported	Not reported	Not reported	Not reported
Special chemical/mineralogical features	None reported	None reported	None reported	None reported
Clay fraction mineralogy	Not reported	Not reported	Not reported	Not reported

Table A7_2_3_1-2a: Results of preliminary test for Soil 1 (SK 961089):

Test substance	(^{14}C) -Ethylene urea		
Sample purity	99.5%		
Weighed soil	10	5	1
Volume of CaCl_2 solution	10	25	25
Nominal concentration of a.s. final solution	5.0 $\mu\text{g/mL}$	5.0 $\mu\text{g/mL}$	5.0 $\mu\text{g/mL}$
Analytical concentration final of a.s. solution	Not reported	Not reported	Not reported
Concentration of the test solution (show calculation)	Not reported	Not reported	Not reported
Details of the analytical method used:			
Method	Liquid Scintillation Counting (LSC) of Supernatant		
Recovery rate	82.4*	94.1*	100.0*
Detection limit	1.5 x Background radioactivity		

* Determined from reported % of applied radioactivity adsorbed by soil

Table A7_2_3_1-2b: Results of preliminary test for Soil 2 (SK 179618):

Test substance	(^{14}C) -Ethylene urea		
Sample purity	99.5%		
Weighed soil	10	5	1
Volume of CaCl_2 solution	10	25	25
Nominal concentration of a.s. final solution	5.0 $\mu\text{g/mL}$	5.0 $\mu\text{g/mL}$	5.0 $\mu\text{g/mL}$
Analytical concentration final of a.s. solution	Not reported	Not reported	Not reported
Concentration of the test solution (show calculation)	Not reported	Not reported	Not reported
Details of the analytical method used:			
Method	Liquid Scintillation Counting (LSC) of Supernatant		
Recovery rate	89.3*	98.4*	100.0*
Detection limit	1.5 x Background radioactivity		

* Determined from reported % of applied radioactivity adsorbed by soil

Table A7_2_3_1-2c: Results of preliminary test for Soil 3 (SK 566696):

Test substance	(^{14}C) -Ethylene urea		
Sample purity	99.5%		
Weighed soil	10	5	1
Volume of CaCl_2 solution	10	25	25
Nominal concentration of a.s. final solution	5.0 $\mu\text{g/mL}$	5.0 $\mu\text{g/mL}$	5.0 $\mu\text{g/mL}$
Analytical concentration final of a.s. solution	Not reported	Not reported	Not reported
Concentration of the test solution (show calculation)	Not reported	Not reported	Not reported
Details of the analytical method used:			
Method	Liquid Scintillation Counting (LSC) of Supernatant		
Recovery rate	83.4*	98.1*	100.0*
Detection limit	1.5 x Background radioactivity		

* Determined from reported % of applied radioactivity adsorbed by soil

Table A7_2_3_1-2d: Results of preliminary test for Soil 4 (SK 920191):

Test substance	(^{14}C) -Ethylene urea		
Sample purity	99.5%		
Weighed soil	10	5	1
Volume of CaCl_2 solution	10	25	25
Nominal concentration of a.s. final solution	5.0 $\mu\text{g/mL}$	5.0 $\mu\text{g/mL}$	5.0 $\mu\text{g/mL}$
Analytical concentration final of a.s. solution	Not reported	Not reported	Not reported
Concentration of the test solution (show calculation)	Not reported	Not reported	Not reported
Details of the analytical method used:			
Method	Liquid Scintillation Counting (LSC) of Supernatant		
Recovery rate	80.7*	95.6*	100.0*
Detection limit	1.5 x Background radioactivity		

* Determined from reported % of applied radioactivity adsorbed by soil

Table A7_2_3_1-3a: Results of screening test – adsorption for Soil 1 (SK 961089):

	5.0 µg/mL		2.5 µg/mL		0.5 µg/mL		0.25 µg/mL		0.05 µg/mL	
	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean
Concentration of test material [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
After contact of....hours with soil	24	-	24	-	24	-	24	-	24	-
Correction for blank with soil	Not applied									
Correction for blank without soil	Not applied									
Final corrected concentration [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
Initial concentration of test solution [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
Decrease in concentration [mg/l]	Not reported									
Quantity adsorbed [µg]	9.2787 4.0506*	6.6647	3.8353 4.5748	4.2050	1.0112 0.9270	0.9691	0.3813 0.3576	0.3694	0.0674 0.0836	0.0755
Quantity of soil [g of oven-dried equivalent]	10	-	10	-	10	-	10	-	10	-
Quantity adsorbed [µg] per gram of soil	0.92787 0.40506*	0.66647	0.38353 0.45748	0.42050	0.10112 0.09270	0.09691	0.03813 0.03576	0.03694	0.00674 0.00836	0.00755
Test material adsorbed [%]	18.6 8.1*	13.4	15.3 18.3	16.8	20.6 18.9	19.8	15.3 14.3	14.8	13.5 16.7	15.1
Temperature [°C]	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-
Volume of solution recovered after centrifugation [ml]	Not detailed. Report states that as much of the supernatant solution was removed as was possible									
Volume of solution not recovered [ml]	Not detailed. Report states that as much of the supernatant solution was removed as was possible									
Corresponding quantity of test substance [mg]	Not reported									

* Data flagged as not used in Regression Analysis

Table A7 2 3 1-3b: Results of screening test – adsorption for Soil 2 (SK 179618):

	5.0 µg/mL		2.5 µg/mL		0.5 µg/mL		0.25 µg/mL		0.05 µg/mL	
	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean
Concentration of test material [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
After contact of....hours with soil	24	-	24	-	24	-	24	-	24	-
Correction for blank with soil	Not applied									
Correction for blank without soil	Not applied									
Final corrected concentration [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
Initial concentration of test solution [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
Decrease in concentration [mg/l]	Not reported									
Quantity adsorbed [µg]	6.7932 5.8705	6.3319	3.0549 2.9584	3.0066	0.5722 0.5228	0.5475	0.2728 0.2190	0.2459	0.0536 0.0762	0.0649
Quantity of soil [g of oven-dried equivalent]	10	-	10	-	10	-	10	-	10	-
Quantity adsorbed [µg] per gram of soil	0.67932 0.58705	0.63319	0.30549 0.29584	0.30066	0.05722 0.05228	0.05475	0.02728 0.02190	0.02459	0.00536 0.00762	0.00649
Test material adsorbed [%]	13.6 11.8	12.7	12.2 11.8	12.0	11.7 10.7	11.2	10.9 8.8	9.8	10.7 15.2	13.0
Temperature [°C]	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-
Volume of solution recovered after centrifugation [ml]	Not detailed. Report states that as much of the supernatant solution was removed as was possible									
Volume of solution not recovered [ml]	Not detailed. Report states that as much of the supernatant solution was removed as was possible									
Corresponding quantity of test substance [mg]	Not reported									

Table A7 2 3 1-3c: Results of screening test – adsorption for Soil 3 (SK 566696):

	5.0 µg/mL		2.5 µg/mL		0.5 µg/mL		0.25 µg/mL		0.05 µg/mL	
	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean
Concentration of test material [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
After contact of....hours with soil	24	-	24	-	24	-	24	-	24	-
Correction for blank with soil	Not applied									
Correction for blank without soil	Not applied									
Final corrected concentration [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
Initial concentration of test solution [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
Decrease in concentration [mg/l]	Not reported									
Quantity adsorbed [µg]	6.1783 4.1377	5.1580	2.7223 3.8707	3.2965	0.6407 0.7828	0.7118	0.3635 0.4680	0.4157	0.0579 0.0870	0.0725
Quantity of soil [g of oven-dried equivalent]	10	-	10	-	10	-	10	-	10	-
Quantity adsorbed [µg] per gram of soil	0.61783 0.41377	0.51580	0.27223 0.38707	0.32965	0.06407 0.07828	0.07118	0.03635 0.04680	0.04157	0.00579 0.00870	0.00725
Test material adsorbed [%]	12.4 8.3	10.3	10.9 15.5	13.2	13.1 16.0	14.5	14.5 18.7	16.6	11.6 17.4	14.5
Temperature [°C]	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-
Volume of solution recovered after centrifugation [ml]	Not detailed. Report states that as much of the supernatant solution was removed as was possible									
Volume of solution not recovered [ml]	Not detailed. Report states that as much of the supernatant solution was removed as was possible									
Corresponding quantity of test substance [mg]	Not reported									

Table A7_2_3_1-3d: Results of screening test – adsorption for Soil 4 (SK 920191):

	5.0 µg/mL		2.5 µg/mL		0.5 µg/mL		0.25 µg/mL		0.05 µg/mL	
	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean
Concentration of test material [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
After contact of....hours with soil	24	-	24	-	24	-	24	-	24	-
Correction for blank with soil	Not applied									
Correction for blank without soil	Not applied									
Final corrected concentration [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
Initial concentration of test solution [mg/l]	5.0	-	2.5	-	0.5	-	0.25	-	0.05	-
Decrease in concentration [mg/l]	Not reported									
Quantity adsorbed [µg]	8.4662	8.1252	4.4431	4.4670	3.3957*	2.1879	0.3849	0.4387	0.1085	0.0923
	7.7841		4.4910		0.9802		0.4924		0.0762	
Quantity of soil [g of oven-dried equivalent]	10	-	10	-	10	-	10	-	10	-
Quantity adsorbed [µg] per gram of soil	0.84662	0.81252	0.44431	0.44670	0.33957*	0.21879	0.03849	0.04387	0.01085	0.00923
	0.77841		0.44910		0.09802		0.04924		0.00762	
Test material adsorbed [%]	17.0	16.3	17.8	17.9	69.3*	44.7	15.4	17.5	21.7	18.5
	15.6		18.0		20.9		19.7		15.2	
Temperature [°C]	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-
Volume of solution recovered after centrifugation [ml]	Not detailed. Report states that as much of the supernatant solution was removed as was possible									
Volume of solution not recovered [ml]	Not detailed. Report states that as much of the supernatant solution was removed as was possible									
Corresponding quantity of test substance [mg]	Not reported									

* Data flagged as not used in Regression Analysis

Table A7_2_3_1-4a: Results of screening test – desorption for Soil 1 (SK 961089):

	5.0 µg/mL		2.5 µg/mL		0.5 µg/mL		0.25 µg/mL		0.05 µg/mL	
	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean
Temperature [°C]	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-
Concentration in combined washings [mg/l]	0.1833 0.2134	0.1984	0.0976 0.0856	0.0916	0.0167 0.0177	0.0172	0.0095 0.0095	0.0095	0.0018 0.0018	0.0018
Corresponding quantity of test material [mg]	0.001833 0.002134	0.001984	0.000976 0.000856	0.000916	0.0167 0.0177	0.000172	0.000095 0.000095	0.000095	0.000018 0.000018	0.000018
Quantity desorbed [µg] ¹	1.6923 2.1585	1.9254	0.8051 0.4002	0.6026	0.0612 0.1599	0.1106	0.0880 0.0667	0.0730	-0.0031 0.0095	0.0032
[%] of adsorbed test material, which is desorbed ²	18.2 53.3*	35.8	21.0 8.7	14.9	6.1 17.3	11.7	23.1 18.6	20.9	-4.7 11.3	3.3
[%] of adsorbed test material, which is not desorbed ³	81.8 46.7*	64.2	79.0 91.3	85.1	93.9 82.7	88.3	76.9 81.4	79.1	104.7 88.7	96.7

* Data flagged as not used in Regression Analysis

¹ Reported data for quantity desorbed was calculated from: (µg EU in soil at end of adsorption phase) – (µg EU in soil at end of desorption phase)

² Reported data for % EU desorbed was calculated from:
$$\frac{(\mu\text{g EU in soil at end of adsorption phase} - \mu\text{g EU in soil at end of desorption phase})}{(\mu\text{g EU in soil at end of adsorption phase})} \times 100$$

³ Reported data for % EU not desorbed was calculated from:
$$\frac{(\mu\text{g EU in soil at end of desorption phase})}{(\mu\text{g EU in soil at end of adsorption phase})} \times 100$$

Table A7_2_3_1-4b: Results of screening test – desorption for Soil 2 (SK 179618):

	5.0 µg/mL		2.5 µg/mL		0.5 µg/mL		0.25 µg/mL		0.05 µg/mL	
	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean
Temperature [°C]	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-
Concentration in combined washings [mg/l]	0.1728 0.1809	0.1769	0.0812 0.0853	0.0833	0.0171 0.0177	0.0174	0.0082 0.0088	0.0085	0.0018 0.0016	0.0017
Corresponding quantity of test material [mg]	0.001728 0.001809	0.001769	0.000812 0.000853	0.000833	0.000171 0.000177	0.000174	0.000082 0.000088	0.000085	0.000018 0.000016	0.000017
Quantity desorbed [µg] ¹	3.0210 3.3387	3.1799	0.6011 0.6896	0.6453	0.2223 0.3075	0.2649	0.0666 0.1143	0.0905	0.0271 0.0240	0.0256
[%] of adsorbed test material, which is desorbed ²	44.5 56.9	50.7	19.7 23.3	21.5	38.8 58.8	48.8	24.4 52.2	38.3	50.6 31.6	41.1
[%] of adsorbed test material, which is not desorbed ³	55.5 43.1	49.3	80.3 76.7	78.5	61.2 41.2	51.2	75.6 47.8	61.7	49.4 68.4	58.9

¹ Reported data for quantity desorbed was calculated from: (µg EU in soil at end of adsorption phase) – (µg EU in soil at end of desorption phase)

² Reported data for % EU desorbed was calculated from:
$$\frac{(\mu\text{g EU in soil at end of adsorption phase} - \mu\text{g EU in soil at end of desorption phase})}{(\mu\text{g EU in soil at end of adsorption phase})} \times 100$$

³ Reported data for % EU not desorbed was calculated from:
$$\frac{(\mu\text{g EU in soil at end of desorption phase})}{(\mu\text{g EU in soil at end of adsorption phase})} \times 100$$

Table A7_2_3_1-4c: Results of screening test – desorption for Soil 3 (SK 566696):

	5.0 µg/mL		2.5 µg/mL		0.5 µg/mL		0.25 µg/mL		0.05 µg/mL	
	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean
Temperature [°C]	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-
Concentration in combined washings [mg/l]	0.1713 0.1751	0.1732	0.0842 0.0860	0.0851	0.0158 0.0163	0.0161	0.0084 0.0082	0.0083	0.0018 0.0016	0.0017
Corresponding quantity of test material [mg]	0.001713 0.001751	0.001732	0.000842 0.000860	0.000851	0.000158 0.000163	0.000161	0.000084 0.000082	0.000083	0.000018 0.000016	0.000017
Quantity desorbed [µg] ¹	1.2566 0.8365	1.0465	0.3761 1.0407	0.7084	0.0279 0.169	0.0985	0.0967 0.1027	0.0996	0.0270 0.0158	0.0214
[%] of adsorbed test material, which is desorbed ²	20.3 20.2	20.3	13.8 26.9	20.4	4.4 21.6	13.0	26.6 21.9	24.3	46.5 18.2	32.4
[%] of adsorbed test material, which is not desorbed ³	79.7 79.8	79.7	86.2 73.1	79.6	95.6 78.4	87.0	73.4 78.1	75.7	53.5 81.8	67.6

¹ Reported data for quantity desorbed was calculated from: (µg EU in soil at end of adsorption phase) – (µg EU in soil at end of desorption phase)

² Reported data for % EU desorbed was calculated from:
$$\frac{(\mu\text{g EU in soil at end of adsorption phase} - \mu\text{g EU in soil at end of desorption phase})}{(\mu\text{g EU in soil at end of adsorption phase})} \times 100$$

³ Reported data for % EU not desorbed was calculated from:
$$\frac{(\mu\text{g EU in soil at end of desorption phase})}{(\mu\text{g EU in soil at end of adsorption phase})} \times 100$$

Table A7_2_3_1-4d: Results of screening test – desorption for Soil 4 (SK 920191):

	5.0 µg/mL		2.5 µg/mL		0.5 µg/mL		0.25 µg/mL		0.05 µg/mL	
	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean	Replicate 1 and 2	Mean
Temperature [°C]	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-	20±2°C	-
Concentration in combined washings [mg/l]	0.1755 0.1684	0.1720	0.0858 0.0844	0.0851	0.0064 0.0071	0.0068	0.0090 0.0088	0.0089	0.0016 0.0016	0.0016
Corresponding quantity of test material [mg]	0.001755 0.001684	0.001720	0.000858 0.000844	0.000851	0.000064 0.000071	0.000068	0.000090 0.000088	0.000089	0.000016 0.000016	0.000016
Quantity desorbed [µg] ¹	1.7314 0.9777	1.3546	0.4122 0.4255	0.4188	0.0499 -0.7721	-0.3612	0.0876 0.1040	0.0959	0.0186 -0.0059	0.0063
[%] of adsorbed test material, which is desorbed ²	20.5 12.6	16.5	9.3 9.5	9.4	1.5* -78.8*	-38.7	22.8 21.1	21.9	17.2 -7.8	4.7
[%] of adsorbed test material, which is not desorbed ³	79.5 87.4	83.5	90.7 90.5	90.6	98.5* 178.8*	138.7	77.2 78.9	78.1	82.8 107.8	95.3

* Data flagged as not used in Regression Analysis

¹ Reported data for quantity desorbed was calculated from: (µg EU in soil at end of adsorption phase) – (µg EU in soil at end of desorption phase)

² Reported data for % EU desorbed was calculated from:
$$\frac{(\mu\text{g EU in soil at end of adsorption phase} - \mu\text{g EU in soil at end of desorption phase})}{(\mu\text{g EU in soil at end of adsorption phase})} \times 100$$

³ Reported data for % EU not desorbed was calculated from:
$$\frac{(\mu\text{g EU in soil at end of desorption phase})}{(\mu\text{g EU in soil at end of adsorption phase})} \times 100$$

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Adsorption / Desorption screening test

Annex Point IIIA XII.1.2

⁽¹⁴C)-Ethylene urea, a metabolite of Mancozeb:
Adsorption/Desorption in Soil

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Official
use only**1 REFERENCE****1.1 Reference**

Cooke, J., (2003), (¹⁴C)-Ethylene urea, a metabolite of Mancozeb: Adsorption/Desorption in Soil, Covance Laboratories Ltd., Otley Road, Harrogate, North Yorkshire, HG3 1PY, England, Report No. 295/162-D2149, 23 July 2003.

1.2 Data protection

Yes

1.2.1 Data owner

Cerexagri B.V./Dow/BASF

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. Adsorption – Desorption Using a Batch Equilibrium Method

2.2 GLP

Yes

2.3 Deviations

No

3 MATERIALS AND METHODS**3.1 Test material**⁽¹⁴C)-Ethylene urea (¹⁴C-EU)

Ethylene urea

3.1.1 Lot/Batch number

¹⁴C : INV 1891

Unlabelled: 06403AO

3.1.2 Specification

Deviating from specification given in section 2 as follows

The test substance is a metabolite of Mancozeb

3.1.3 Purity

¹⁴C : 99.5% (from Certificate of Analysis)

Unlabelled: 100%

3.1.4 Further relevant properties

Specific activity of ¹⁴C radiolabelled material was 1.1 mCi/mmol (0.473 MBq/mg)

3.1.5 Method of analysis

The certified radiopurity was reconfirmed by high performance liquid chromatography (HPLC) and thin layer chromatography (TLC) and shown to be >98% at the start of the study.

The radiopurity in the formulations used to conduct the adsorption/desorption incubations was confirmed by HPLC and shown to be >98%.

HPLC method

Column: Inertsil ODS-3V 5 µm column (25 cm x 4.6 mm)

Solvent A: 10 mM Ammonium acetate at pH 10

Solvent B: Acetonitrile

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Gradient:	Time (min)	% A	% B
	0	100	0
	3	100	0
	15	90	10
	30	60	40

Flow rate: 1.0ml/min

UV detection: 210 nm

Radiolabelled compounds in the eluent were monitored using a flow-through radioactivity detector with liquid scintillant (3 mL/min) and a 500 µL liquid mixing cell.

Samples were co-chromatographed with non-radiolabelled reference standard, which was monitored using UV absorbance.

Chromatograms were evaluated using Laura (version 1.4a) software.

TLC method

Plate: Whatman K6F silica gel (20 x 20 cm)

Solvent: Chloroform: methanol: ammonia solution (10:1:1 v/v)

Radiolabelled compounds were detected by preparation of a radioluminogram of the TLC plate using a Fuji BAS L 500 Bio-image analyser.

Samples were co-chromatographed with non-radiolabelled reference standard, which was visualised with 1% w/v potassium permanganate spray.

Chromatograms were evaluated using Tina (version 2.09g) software.

3.2 Degradation products

Degradation products tested: No

3.2.1 Method of analysis for degradation products

Not applicable

3.3 Reference substance

No

3.3.1 Method of analysis for reference substance

Not applicable

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Adsorption / Desorption screening test**(¹⁴C)-Ethylene urea, a metabolite of Mancozeb:
Adsorption/Desorption in Soil****3.4 Soil types**

4 soil types, classified as per USDA, were used:-

Soil 1: Clay loam (SK 961089)

Soil 2: Loam (SK 179618)

Soil 3: Loamy sand (SK 566696)

Soil 4: Clay loam (SK 920191)

Refer to table A7_1_3-1 for full details

Soils were sterilised by gamma irradiation before use.

3.5 Testing procedure**3.5.1 Test system**

The test system was comprised of air-dried soils, sterilised by gamma irradiation and pre-conditioned by mixing overnight with 0.1M calcium chloride, mixed with ¹⁴C test material dissolved in 0.1M calcium chloride. The samples were held in 50 mL polypropylene centrifuge tubes and incubated in the dark at 20±2°C whilst being mixed on an end over end shaker (Stuart Scientific Rotator Drive STR4).

Soils were sterilised at Isotron, Bradford, West Yorkshire, UK.

LSC of liquid samples was conducted using a Packard Tricarb Model 900TR Liquid Scintillation Counter with facilities for computing quench corrected dpm.

Radioactivity in solid residues was determined after homogenisation in a vibrating cup mill by LSC following combustion in a Harvey OX-500 Biological Materials Oxidizer. Combustion efficiencies were checked throughout the period of use and on the basis of these being 99±4% efficient, no corrections were applied.

3.5.2 Test solution and Test conditions

A stock solution of ¹⁴C ethylene urea was prepared by dissolving TS in 10 mL of 0.01M calcium chloride to give a 19.0 µg/mL solution. Sonication was used to ensure dissolution of the TS. This was mixed with unlabelled ethylene urea to create 5 formulation solutions ranging in concentration from 0.0189 – 1.91 mg/mL with corresponding specific activities of 0.473 – 0.0047 MBq/mg. 26 µL of each of these solutions was added to a mix of 10 g soil and 10 mL 0.01 M calcium chloride (ie a soil/solution ratio of 1/1 (w/v)) to give a final TS concentration range of 0.05 – 5 µg/mL.

Samples were incubated in the dark at 20±2°C for the duration of the tests.

3.6 Test performance**3.6.1 Preliminary test**

According to (a)“OECD 106”: Yes

To define conditions for optimal adsorption, 3 different ratios of soil to aqueous phase were assessed over 24 hours at 5 µg/mL in polypropylene tubes.

3.6.2 Screening test: Adsorption

According to (a)“OECD 106”: Yes

¹⁴C radiolabelled ethylene urea (prepared from stock radiolabelled TS diluted with unlabelled TS) in 10 mL of 0.01M calcium chloride was added to 10 g (dry weight equivalents) of soil in 50 mL polypropylene centrifuge tubes. All soil samples were pre-conditioned with 0.01M calcium chloride. Five concentrations of ethylene urea (0.05-5 µg/mL)

Section A7.2.3.1(3)**Adsorption / Desorption screening test****Annex Point IIIA XII.1.2****(¹⁴C)-Ethylene urea, a metabolite of Mancozeb:
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were prepared in duplicate, the concentrations of each being confirmed by Liquid Scintillation Counting (LSC). All samples were mixed continuously for 24 hours in an end over end shaker at a speed sufficient to ensure thorough mixing, after which each sample was centrifuged to separate the soil from solution. The supernatant was removed and subject to LSC to determine the quantity of TS adsorbed by the soil.

**3.6.3 Screening test:
Desorption**

According to (a)"OECD 106": Performed

Samples from the adsorption phase were mixed with a quantity of fresh 0.01M calcium chloride sufficient to replace the quantity of supernatant removed. The tubes were shaken vigorously to break up the compacted soil and then mixed continuously for 24 hours to allow desorption. Each sample was then centrifuged to separate the soil from solution and the supernatant was removed and subject to LSC to determine the quantity of TS desorbed from the soil.

3.6.4 HPLC-method

According to (a)" OECD-HPLC-method"¹: No

3.6.5 Other test

The solubility of ethylene urea in 0.01 M calcium chloride at 5 µg/mL was confirmed by LSC after centrifugation to remove any potentially undissolved material.

The potential for ethylene urea to adsorb onto containers was assessed by incubation of 25 mL of 0.05 µg/mL ethylene urea in 0.01 M calcium chloride in both polypropylene and teflon tubes.

Tests to assess the effect of various incubation periods of up to 48 hours for the conduct of the adsorption/desorption phases were conducted.

The stability of ethylene urea in each soil type was assured by the incubation of 10 mL of 5 µg/mL ethylene urea in 0.01 M calcium chloride with 10 g of soil for 48 hours, after which the supernatant and soil were separated. The soil was washed with 0.01 M calcium chloride and thoroughly extracted with acetonitrile/water, acetonitrile, methanol and water, after which all of the extracts were combined with the supernatants and the total recovery was assessed. For soils 1 and 4 (SK 961089 and SK 920191 respectively) an additional series of extractions with aqueous trifluoroacetic acid (0.1%) was applied because the recovery of total radioactivity was less than 90% to this point. The combined washings and supernatants were concentrated to a small volume for radio-profiling by HPLC as detailed in section 3.1.5.

To allow estimation of the mean recovery of applied radioactivity, all residual soil samples from the desorption tests were air dried prior to quantification by combustion in a sample oxidiser followed by LSC.

4 RESULTS**4.1 Preliminary test**

A 1:1 ratio of soil/aqueous (w/v, 10g/10ml) was selected on the basis of providing the maximum adsorption for the adsorption/desorption tests. See tables A7_1_3-2a - A7_1_3-2d.

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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Adsorption / Desorption screening test**(¹⁴C)-Ethylene urea, a metabolite of Mancozeb:
Adsorption/Desorption in Soil**

4.2	Screening test: Adsorption	See tables A7_1_3-3a - A7_1_3-3d
4.3	Screening test: Desorption	See tables A7_1_3-4a - A7_1_3-4d
4.4	Calculations	
4.4.1	Ka , Kd	Soil 1: Clay loam (SK 961089) Ka = 0.22, Kd = 0.40 Soil 2: Loam (SK 179618) Ka = 0.16, Kd = 0.20 Soil 3: Loamy sand (SK 566696) Ka = 0.15, Kd = 0.29 Soil 4: Clay loam (SK 920191) Ka = 0.22, Kd = 0.42
4.4.2	Ka _{oc} , Kd _{oc}	Soil 1: Clay loam (SK 961089) Ka _{oc} = 5, Kd _{oc} = 9 Soil 2: Loam (SK 179618) Ka _{oc} = 4, Kd _{oc} = 5 Soil 3: Loamy sand (SK 566696) Ka _{oc} = 19, Kd _{oc} = 36 Soil 4: Clay loam (SK 920191) Ka _{oc} = 11, Kd _{oc} = 20
4.5	Degradation product(s)	A test for breakdown products was conducted in advance of the preliminary test (see section 5.2). No degradation products were characterised for any of the soils under the conditions of the test (see section 5.2.6).

5 APPLICANT'S SUMMARY AND CONCLUSION**5.1 Materials and
methods**

A GLP-compliant study was carried out in which the adsorption/desorption characteristics of (¹⁴C)-ethylene urea were determined in four soil types. The study was conducted in accordance with the requirements of OECD Guideline 106.

All experiments were performed in the dark at 20±2°C using autoclaved 0.01M CaCl. The air-dried soils were sterilised by gamma irradiation and preconditioned by mixing overnight with 0.01M CaCl.

Following a preliminary test, a full adsorption/desorption test was carried out by adding aliquots of (¹⁴C)-ethylene urea in 0.01M CaCl at concentrations of 5, 2.5, 0.5, 0.25 and 0.05 µg/ml to duplicate samples of the preconditioned soils and 0.01 M CaCl. The concentration of radioactivity in the doses was determined by LSC and used to calculate the quantity of ethylene urea applied to each sample.

Samples were mixed for 24 hours and centrifuged to separate the soil and solution. Radioactivity in the supernatant was determined by LSC. The supernatant was then replaced by fresh 0.01 M CaCl for the desorption phase, mixed for 24 hours, centrifuged and the radioactivity in the supernatant determined by LSC.

Soil residues were air-dried prior to quantification by combustion. The mean recovery of applied radioactivity for each soil type was in the range 97 to 102%.

**5.2 Results and
discussion**

The solubility of ethylene urea in 0.01 M calcium chloride at 5 µg/mL was confirmed.

No adsorption to either polypropylene or teflon tubes was observed on

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Adsorption / Desorption screening test

Annex Point IIIA XII.1.2

¹⁴C)-Ethylene urea, a metabolite of Mancozeb:

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the basis of LSC determinations following incubation of 25 mL of 0.05 µg/mL ethylene urea in 0.01 M calcium chloride in both types of tube. Polypropylene tubes were used throughout the study.

24 Hours incubation was selected as suitable for the conduct of the adsorption/desorption phases.

Ethylene urea was stable under the conditions of the test for at least 48 hours.

Freundlich adsorption constants (K) calculated by linear regression analysis for soils SK 961089 (clay loam), SK 179618 (loam), SK 566696 (loamy sand) and SK 920191 (clay loam) were 0.22, 0.16, 0.15, and 0.22 respectively.

Freundlich adsorption constants related to organic carbon content (K) for soils SK 961089 (clay loam), SK 179618 (loam), SK 566696 (loamy sand) and SK 920191 (clay loam) were 5, 4, 19 and 11 respectively.

Adsorption and desorption isotherms showed a strong positive monotonic relationship between log C and log X/m, indicated by the correlation coefficients being close to 1. Mass balance results demonstrated that mean recoveries of applied radioactivity from each soil type were in the range 97 to 102%.

The desorption equilibrium was attained within twice the time of the adsorption equilibrium but the total desorbed was <75% of the amount adsorbed, therefore the adsorption is not considered to be reversible.

5.2.1	Adsorbed a.s. [%]	Soil 1: Clay loam (SK 961089)	13.4-19.1%
		Soil 2: Loam (SK 179618)	9.8-13.0%
		Soil 3: Loamy sand (SK 566696)	10.3-16.6%
		Soil 4: Clay loam (SK 920191)	16.3-44.7%*

Data are reported as ranges of mean % ethylene urea adsorbed for the range of concentrations tested for each soil. Refer to tables A7_1_3-3a - A7_1_3-3d for full details.

* The upper range for SK 920191 may be misleading because of poor duplication: replicates were 20.0% and 69.3%. The latter figure was discounted in the regression analysis to calculate the adsorption constant, but included in the reported means.

5.2.2	K _a	Soil 1: Clay loam (SK 961089)	K _a = 0.22
		Soil 2: Loam (SK 179618)	K _a = 0.16
		Soil 3: Loamy sand (SK 566696)	K _a = 0.15
		Soil 4: Clay loam (SK 920191)	K _a = 0.22
5.2.3	K _d	Soil 1: Clay loam (SK 961089)	K _d = 0.40
		Soil 2: Loam (SK 179618)	K _d = 0.20
		Soil 3: Loamy sand (SK 566696)	K _d = 0.29
		Soil 4: Clay loam (SK 920191)	K _d = 0.42
5.2.4	K _{aoc}	Soil 1: Clay loam (SK 961089)	K _{aoc} = 5
		Soil 2: Loam (SK 179618)	K _{aoc} = 4

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		Soil 3: Loamy sand (SK 566696) $K_{a_{oc}} = 19$
		Soil 4: Clay loam (SK 920191) $K_{a_{oc}} = 11$
5.2.5	K_a/K_d	Soil 1: Clay loam (SK 961089) $K_a/K_d = 0.55^*$
		Soil 2: Loam (SK 179618) $K_a/K_d = 0.80^*$
		Soil 3: Loamy sand (SK 566696) $K_a/K_d = 0.52^*$
		Soil 4: Clay loam (SK 920191) $K_a/K_d = 0.52^*$
		*Derived from reported data
5.2.6	Degradation products (% of a.s.)	Soil 1: Clay loam (SK 961089) $<9.7\%^{**}$
		Soil 2: Loam (SK 179618) $<4.8\%^{**}$
		Soil 3: Loamy sand (SK 566696) $<8.3\%^{**}$
		Soil 4: Clay loam (SK 920191) $<11.4\%^{**}$
		**Not reported, but inferred as a worst case on the basis of % applied radioactivity not recovered in the stability test. This fraction of radioactivity was not characterised and could represent irreversibly adsorbed TS. Of the recovered radioactivity, radio-HPLC analysis of breakdown products not associated with ethylene urea gave figures of 0.01%, 1.24%, 2.05% and 0.24% for soils 1, 2, 3 and 4 respectively, none of which was present as a discrete chemical entity.
5.3	Conclusion	The validity criteria of the study are considered as being fulfilled.
		Adsorption and desorption isotherms showed a strong positive monotonic relationship between the log of the concentrations of ethylene urea adsorbed to the soil, indicated by correlation coefficients close to 1.
		The desorption equilibrium was attained within twice the time of the adsorption equilibrium but the total desorbed was <75% of the amount adsorbed, therefore the adsorption is not considered to be reversible.
5.3.1	Reliability	1
5.3.2	Deficiencies	No

Evaluation by Competent Authorities

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

EVALUATION BY RAPPORTEUR MEMBER STATE**Date**

Give date of action

Materials and Methods

State if the applicants version is acceptable or indicate relevant discrepancies referring to the (sub) heading numbers and to applicant's summary and conclusion.

Results and discussion

Adopt applicant's version or include revised version. If necessary, discuss relevant deviations from applicant's view referring to the (sub)heading numbers

Conclusion

Adopt applicant's version or include revised version