Data Evaluation Report on the adsorption-desorption of halcomid in soil

PMRA Submission Number {.....} EPA MRID Number 45369733

Data Requirement: PMRA Data Code:
EPA DP Barcode: D284964
OECD Data Point:
EPA Guideline: 163-1

Test material:
Common name: Halcomid.
Chemical name
IUPAC: N,N-Dimethyldecanoic acid amide.
CAS name: Not reported.
CAS No: Not reported.
Synonyms: Not reported.
SMILES string: O=C(CCCCCCCCC)N(C)C.

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Company Code:
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CITATION: Morgenroth, U. and S. Völkl. 1995. Adsorption/desorption of N,N-dimethyldecanoic acid amide on four soils. Unpublished study performed by RCC Umweltchemie AG; sponsored by Bayer AG, Monheim, Germany; and submitted by The C.P. Hall Company, Chicago, IL. Study Project No.: RCC Project 340356. Experiment initiation date August 3, 1993, and completion date March 18, 1994 (p. 16). Final report issued December 20, 1995.

ADMINISTRATIVE CONCLUSIONS

1) This study is scientifically valid, and satisfies Subdivision N Guideline criteria for adsorption/desorption (mobility, §163-1). No additional sorption data are needed at this time.

2) The submitter should note the comments given in Section IV of this Data Evaluation Report, and consider their applicability to the acceptability of future submissions.

SCIENTIFIC CONCLUSIONS

Sorption coefficients derived from this study are tabulated as follows (see Tables 8a and 8b for definitions of symbols and equations used for calculations):

Adsorption and Desorption Values for [^{14}C]Halcomid in Soils[1]

Soil	Freundlich Adsorption (regression values)				Freundlich Desorption (regression values)				Simple Adsorption Average[2] (approximate ranges)
	K_F	1/N	R^2	K_{Foc}	K_F	1/N	R^2	K_{Foc}	Kd / Koc
Soil I Sandy loam (California)	2.84	0.80	0.9990	351	4.26	0.83	0.9987	526	3.15 / 389 (0.86-4.8) / (110-590)
Soil II Loamy sand (Illinois)	3.84	0.83	0.9996	630	5.70	0.89	0.9999	934	4.00 / 656 (1.5-5.8) / (240-950)
Soil III Silt loam (Illinois)	6.03	0.78	1.00	569	9.16	0.83	0.9991	864	8.29 / 782 (2.7-13.2) / (250-1200)
Soil IV Loam (Iowa)	11.4	0.80	1.00	559	14.6	0.83	0.9995	716	16.1 / 789 (6.5-24) / (320-1200)

[1]Data were obtained from study report Tables 3 and 4, pps. 45-46; and Table 6, p. 48.
[2]Average of Kds for four test concentrations with amount of a.i. equivalent to approximately 0.2, 1, 5, and 29 mg a.i/kg soil at soil:solution ratio of 1:5 (w/v) for all soils.
K_d -Simple adsorption coefficient.
K_F - Freundlich adsorption and desorption coefficients
1/N -Slope of Freundlich adsorption/desorption isotherms.
R^2 - Square of regression coefficient.
K_{oc} - Simple adsorption coefficient per fraction of organic carbon [(K_d x 100)/(% organic carbon)].
K_{Foc} - Freundlich sorption coefficient per fraction of organic carbon [(K_F x 100)/(% organic carbon)]

Sorption appeared to correlate relatively well with soil organic carbon (within a narrow range of approximately 0.8 to 2%) and clay content, but not with pH.

EXECUTIVE SUMMARY:

The adsorption/desorption characteristics of [1-^{14}C]-labeled N,N-dimethyldecanoic acid amide (halcomid) were studied in a sandy loam soil [Soil I; pH 7.73, organic carbon 0.81%] from California, a loam soil [Soil IV; pH 7.3, organic carbon 2.04%] from Iowa, and a loamy sand soil [Soil II; pH 4.8, organic carbon 0.61%] and silt loam soil [Soil III; pH 6.7, organic carbon 1.06%], each from Illinios, in a batch equilibrium experiment. The experiment was conducted in accordance with the USEPA Pesticide Assessment Guidelines, Subdivision N, Section §163-1, and in compliance with the USEPA GLP Volume 54 No. 158.

The adsorption phase of the study was carried out by equilibrating air-dried and sieved (2 mm) soil with [^{14}C]halcomid at nominal concentrations of 0.2, 1.0, 5.0, and 25.0 mg a.i./kg soil for all test soils. The soils were equilibrated in the dark at approximately 20°C for 6 hours. The equilibrating solution used was a mixture of 0.01M $CaCl_2$ + 1% NaN_3 (bactericide), with soil:solution ratios of 1:5 (w:v) for all test soils. The desorption phase of the study was carried out by replacing the adsorption solution with an equivalent volume of pesticide-free 0.01M $CaCl_2$ + 1% NaN_3 solution, and equilibrating the solution in the dark at approximately 20°C for 6 hours. One desorption step was conducted for all test soils.

The supernatant solution after adsorption and desorption was separated by centrifugation and aliquots were analyzed for total radioactivity using LSC. Following desorption, the soils were combusted and duplicate aliquots were analyzed for total radioactivity using LSC. Following adsorption and desorption, supernatant samples from the highest (25.0 mg a.i./kg) and lowest (0.2 mg a.i./kg) test concentrations were analyzed for [^{14}C]halcomid using HPLC.

Based on HPLC analysis, [^{14}C]halcomid was stable in aqueous solution, comprising ≥99.2% of the recovered radioactivity in the low- and high-dose adsorption and desorption supernatants. The mass balance at the end of the adsorption phase was not reported. Mass balances at the end of the desorption phase were 32.79-47.90%, 32.33-43.28%, 18.60-32.71%, and 11.84-25.45% of the applied for the sandy loam, loamy sand, silt loam, and loam soils, respectively.?????

After 6 hours of equilibration 26.4-51.1%, 34.9-57.1%, 44.2-73.5%, and 61.6-84.0% of the applied [^{14}C]halcomid was adsorbed to the sandy loam, loamy sand, silt loam, and loam soils, respectively. Reviewer-calculated average, simple adsorption coefficient (K_d) values for each soil (average of the experimentally measured Kd values for the four test concentrations) were 3.15, 4.00, 8.29, and 16.09 for the sandy loam, loamy sand, silt loam, and loam soils, respectively. Freundlich K_{ads} values were 2.84, 3.84, 6.03, and 11.41 for the sandy loam, loamy sand, silt loam, and loam soils, respectively; corresponding Freundlich adsorption K_{oc} values were 350.6, 629.5, 568.9, and 559.3. At the end of desorption, 32.8-47.8%, 33.2-42.3%, 18.9-32.7%, and 12.0-25.0% of the applied [^{14}C]halcomid was desorbed from the sandy loam, loamy sand, silt loam, and loam soils, respectively. Freundlich K_{des} values were 4.26, 5.70, 9.16, and 14.62 for the sandy loam, loamy sand, silt loam, and loam soils, respectively; corresponding Freundlich desorption K_{oc} values were 525.9, 934.4, 864.2, and 716.7. In all cases, Freundlich

1/n exponent values were approximately 0.8 and the square of all regression correlation coefficients were >0.99 (see Table 8b).

The reviewer-calculated r^2 value for the relationship of Kd vs. % organic carbon is 0.9511, for Kd vs. pH is 0.1048, and for Kd vs. % clay is 0.9706.

Results Synopsis

The reviewer calculated simple adsorption K_d values for each soil at each experimental concentration using the following equation (see Table 8a for symbol definitions):

$$Kd = \frac{\left[\frac{(C_0 V_0 - C_{eq} V_0)}{m}\right]}{C_{eq}}$$

The following results for each soil are the average of the simple Kds for each of the four test concentrations, each of which had duplicate samples:

Soil type: Soil I Sandy loam
Adsorption K_d: 3.15

Soil type: Soil II Loamy sand
Adsorption K_d: 4.00

Soil type: Soil III Silt loam
Adsorption K_d: 8.29

Soil type: Soil IV Loam
Adsorption K_d: 16.1

Adsorption K_d values using the Freundlich adsorption isotherm

$$\log (X/m) = \log K + 1/n \log Ce$$

are as follows (see Table 8b for symbol definitions and a complete listing of values, including regression correlation r^2 values, all of which were >0.99):

Soil type: Soil I Sandy loam
Amount adsorbed: 26.4-51.1% of the applied
Freundlich K_{ads}: 2.84. 1/n: 0.80
Freundlich adsorption K_{oc}: 350.6
Amount desorbed: 32.8-47.8% of the adsorbed
Freundlich K_{des}: 4.26. 1/n: 0.83
Freundlich desorption K_{oc}: 525.9

Soil type: Soil II Loamy sand
Amount adsorbed: 34.9-57.1% of the applied
Freundlich K_{ads}: 3.84. 1/n: 0.83
Freundlich adsorption K_{oc}: 629.5
Amount desorbed: 33.2-42.3% of the adsorbed
Freundlich K_{des}: 5.70. 1/n: 0.89
Freundlich desorption K_{oc}: 934.4

Soil type: Soil III Silt loam
Amount adsorbed: 44.2-73.5% of the applied
Freundlich K_{ads}: 6.03. 1/n: 0.78
Freundlich adsorption K_{oc}: 568.9
Amount desorbed: 18.9-32.7% of the adsorbed
Freundlich K_{des}: 9.16. 1/n: 0.83
Freundlich desorption K_{oc}: 864.2

Soil type: Soil IV Loam
Amount adsorbed: 61.6-84.0% of the applied
Freundlich K_{ads}: 11.41. 1/n: 0.80
Freundlich adsorption K_{oc}: 559.3
Amount desorbed: 12.0-25.0% of the adsorbed
Freundlich K_{des}: 14.62. 1/n: 0.83
Freundlich desorption K_{oc}: 716.7

Study Acceptability: This study is classified **acceptable** and fulfills the Subdivision N Guideline §163-1 data requirements for a mobility study using unaged soil.

I. MATERIALS AND METHODS

Note: The reviewer referenced the page numbers located in the lower right-hand corner of the study report pages.

GUIDELINE FOLLOWED: The study was conducted in accordance with the USEPA Pesticide Assessment Guidelines, Subdivision N, Section §163-

1 (October 1982) and the OECD Guideline for Testing Chemicals No. 106 (May 1981; pp. 18, 20). No significant deviations from Subdivision N guidelines were noted.

COMPLIANCE: The study was conducted in compliance with the OECD Principles of Good Laboratory Practice (1981), the Swiss Principles of Good Laboratory Practice (March 1986), and the USEPA GLP Volume 54 No. 158 (August, 1989; pp. 9, 18). Signed and dated Data Confidentiality, GLP, and Compliance, and Quality Assurance statements were provided (pp. 2-4, 8-9). A Certificate of Authenticity was not provided.

A. MATERIALS:

1. Test Material [1-^{14}C]-labeled N,N-dimethyldecanoic acid amide (halcomid; p. 24).

Chemical Structure: See DER Attachment.

Description: Colourless liquid (p. 23).

Purity:
Radiolabelled:
Analytical purity: Not reported.
Radiochemical purity: 99.4% (p. 24).
Batch No.: A 387.
Specific activity: 100.5 Mci/g (3.72 MBq/mg).
Location of the label: 1-Carbon (carbonyl carbon).

Non-radiolabelled:
Analytical purity: 98.8% (p. 23).
Batch No.: 930129ELBO2.

Storage conditions of test chemicals: The radiolabeled test substance was stored frozen in the dark (p. 24). The unlabeled test substance was stored in a refrigerator in the dark (p. 23).

Physico-chemical properties of halcomid.

Parameter	Values	Comments
Water solubility	270 mg/L	At 20°C.
Organic solvent solubility (e.g. acetone, DMF, toluene, hexane, 2-propanol, acetonitrile, methylene chloride)	>200 g/L	
Vapour pressure	Not reported	
UV absorption	Not reported	
Molecular Formula	$C_{12}H_{25}NO$	
Molecular Weight	Not reported	
Melting point	Not reported	
Bulk density	Not reported	
pK_a	Not reported	
K_{ow}	Not reported	
Stability of Compound at room temperature	Not reported	

Data were obtained from pp. 23-24 of the study report.

2. Soil Characteristics

Table 1: Description of soil collection and storage.

Description	Soil I Sandy loam	Soil II Loamy sand	Soil III Silt loam	Soil IV Loam
Geographic location	Porterville, California	Farm Eldon Weber, RR1, Geneseo, Illinois	Alvey Laboratory, Belleville, Illinois	Agri-Growth-Research, RR1, Kelley, Iowa
Pesticide use history at the collection site	Not reported	Not reported	Not reported	Not reported
Collection procedures	Not reported	Not reported	Not reported	Not reported
Sampling depth (cm)	Not reported	0-15.24	0-15.24	0-15.24
Storage conditions	Stored in boxes in layers of *ca.* 10 cm; stored outdoors during the vegetation period and indoors during the winter period.	Stored in sealed plastic containers at room temperature.	Stored in sealed plastic containers at room temperature.	Stored in sealed plastic containers at room temperature.

Description	Soil I Sandy loam	Soil II Loamy sand	Soil III Silt loam	Soil IV Loam
Storage length[1]	*ca.* 20 months	*ca.* 20 months	*ca.* 15 months	*ca.* 20 months
Soil preparation	Air-dried; sieved, 2 mm.	Air-dried; sieved, 2 mm.	Air-dried; sieved, 2 mm.	Air-dried; sieved, 2 mm.

Data were obtained from pp. 21-22 of the study report.
[1] Storage length was determined by the reviewer to be the interval from soil sampling (December 1991 for Soil I sandy loam, Soil II loamy sand, and Soil IV loam and May 1992 for Soil III silt loam) to study initiation (August 1993).

Table 2: Properties of the soils.

Property	Soil I	Soil II	Soil III	Soil IV
Soil Texture	Sandy loam	Loamy sand	Silt loam	Loam
% sand	67.2	83.1	24.1	41.2
% silt	24.6	9.4	59.1	33.6
% clay	8.2	7.5	16.8	25.2
pH water 0.1M KCl	 7.90 7.73	 Not reported 4.8	 Not reported 6.7	 Not reported 7.3
Organic carbon (g/100 g soil)	0.81	0.61	1.06	2.04
Organic matter (%)[1]	1.39	1.05	1.82	3.51
CEC (meq/100 g soil)	5.3	7.4	12.6	31.0
Moisture at 1/3 atm (%)	Not reported	Not reported	Not reported	Not reported
Field capacity (g/100 g soil)	11.42	Not reported	Not reported	Not reported
Bulk density (gm/cc)	Not reported	Not reported	Not reported	Not reported
Biomass (mg microbial C/100 g)	Not reported	Not reported	Not reported	Not reported
Soil taxonomic classification	Not reported	Not reported	Not reported	Not reported
Soil mapping unit (for EPA)	Not reported	Not reported	Not reported	Not reported

Data were obtained from pp. 21-22 of the study report.
[1] Percent organic matter was determined by the reviewer as follows: % organic carbon × 1.72.

C. STUDY DESIGN:

1. Preliminary study: Preliminary experiments were conducted to determine the appropriate equilibration time and soil:solution ratio to be used in the definitive study, the stability of the test

substance in the test system, and adsorption of the test substance to the walls of the test vessels (p. 25).

Prior to the initiation of the preliminary experiments, application solution 1 was prepared by dissolving 980 µg of [1-^{14}C]-labeled halcomid in 0.01M $CaCl_2$ solution (p. 25). An aliquot of the application solution was analyzed for total radioactivity using LSC. The concentration of application solution 1 was determined to be 1.51 mg/300 mL. A second application solution was prepared by diluting 41 mL of application solution 1 to a volume of 53 mL with 0.01M $CaCl_2$ solution. A 0.25-g aliquot of NaN_3 was added as a bactericide to the application solution (application solution 2. An aliquot of the application solution was analyzed for total radioactivity using LSC. The concentration of halcomid in application solution 2 was determined to be 0.219 mg/53 mL.

To determine the appropriate equilibration time and soil:solution ratio to be used in the definitive study, duplicate 5-g aliquots (dry weight equivalent) of the Soil I sandy loam, Soil II loamy sand, Soil III silt loam, and Soil IV loam test soils were added to 25-mL aliquots of application solution 1 at a nominal test concentration of 5.0 mg/L (p. 25). Duplicate blank samples were also prepared without the addition of soil to measure adsorption of the test substance to the walls of the test vessels. After 1, 2, 3, 4, 5, 6, and 24 hours, 50-µL aliquots of the supernatants were removed for analysis using LSC. To determine the stability of the test substance in aqueous solution, the samples were analyzed using HPLC following each sampling interval. It was determined that equilibrium was established after 6 hours, with 35.8-71.0% of the applied radioactivity adsorbing to all the test soils (p. 38; Table 1, p. 43; Figure 19, p. 77). Based on HPLC analysis, it was determined that [^{14}C]halcomid was not stable in any of the test soils during incubation (Figure 4, p. 62).

A second preliminary test was conducted to determine the stability of the test substance in aqueous solution in the presence of a bactericide. Duplicate 5-g aliquots (dry weight equivalent) of Soil III silt loam were added to 25-mL aliquots of application solution 2 (p. 26). After 1, 2, 3, 4, 5, 6, and 24 hours, 50-µL aliquots of the supernatants were removed for analysis using LSC. To determine the stability of the test substance in aqueous solution, the samples collected at each sampling interval were analyzed by HPLC. It was determined that 53.3% and 56.5% of the applied radioactivity was adsorbed to the test soil after 6 and 24 hours, respectively (Table 1, **p.** 43). Based on HPLC analysis, [^{14}C]halcomid was stable in aqueous solution for up to 24 hours in the presence of 1% NaN_3 bactericide (p. 38; Figure 19, p. 77).

Based on the results of these preliminary studies, an equilibrium time of 6 hours and a soil:solution ratio of 1:5 (w:w) were selected for use in the definitive study (p. 37). It was determined that the bactericide 1% NaN_3 would be added to the equilibration solution used in the definitive study, to prevent degradation of the test substance. No significant adsorption of the test substance to the test vessels was observed (Table 2, p. 44).

2. Definitive study experimental conditions:

Table 3: Study design for the adsorption phase.

Parameters		Soil I Sandy loam	Soil II Loamy sand	Soil III Silt loam	Soil IV Loam
Condition of soil (air dried/fresh)[1]		Air-dried	Air-dried	Air-dried	Air-dried
Have these soils been used for other laboratory studies ? (specify which)		Yes MRIDs 45369734 45369735	No	No	No
Soil (g/replicate)		5	5	5	5
Equilibrium solution used (name and concentration; eg: 0.01N $CaCl_2$)		0.01M $CaCl_2$ + 1% NaN_3	0.01M $CaCl_2$ + 1% NaN_3	0.01M $CaCl_2$ + 1% NaN_3	0.01M $CaCl_2$ + 1% NaN_3
Control used (with salt solution only) (Yes/No)		Yes	Yes	Yes	Yes
Test material concentrations	Nominal application rates (mg a.i./kg soil)	0.2, 1.0, 5.0, 25.0	0.2, 1.0, 5.0, 25.0	0.2, 1.0, 5.0, 25.0	0.2, 1.0, 5.0, 25.0
	Analytically measured concentrations (mg a.i./kg soil)	0.21, 1.035, 5.08, 29.205	0.215, 1.05, 5.155, 29.635	0.21, 1.025, 5.04, 29.1	0.205, 0.995, 4.91, 28.135
Identity and concentration of co-solvent, if any		Acetone, <0.1%	Acetone, <0.1%	Acetone, <0.1%	Acetone, <0.1%
Soil:solution ratio		1:5	1:5	1:5	1:5
Initial pH of the equilibration solution, if provided		Not reported	Not reported	Not reported	Not reported
No. of replications	Controls	2	2	2	2
	Treatments	2	2	2	2
Equilibration	Time (hours)	6	6	6	6
	Temperature (°C)	*ca.* 20	*ca.* 20	*ca.* 20	*ca.* 20
	Darkness	Yes	Yes	Yes	Yes
	Shaking method	Laboratory shaker	Laboratory shaker	Laboratory shaker	Laboratory shaker
	Shaking time (hours)	6	6	6	6

Parameters		Soil I Sandy loam	Soil II Loamy sand	Soil III Silt loam	Soil IV Loam
Method of separation of supernatant (eg., centrifugation)		Centrifugation	Centrifugation	Centrifugation	Centrifugation
Centrifugation	Speed (g)	1720	1720	1720	1720
	Duration (min)	10	10	10	10
	Method of separation of soil and solution	Decanted	Decanted	Decanted	Decanted

Data were obtained from pp. 22, 27-28 of the study report.

[1] Prior to use in the definitive study, the dry weight for each test soil was determined by drying each soil at approximately 105°C for 3-16 hours under reduced pressure until a constant weight was reached (p. 22). The test soils were then pre-conditioned by shaking overnight with a 0.01M $CaCl_2$ solution containing 1% of NaN_3 as a bactericide (5:1, volume:mass). The soils were centrifuged and the supernatants were discarded.

[2] Test material concentrations were calculated by the reviewer by converting mg/L to mg a.i./kg using the following equation: test concentration (mg/L) x total volume of test material solution (mL)] ÷ amount of soil (g); eg. [0.04 mg/L x 25 mL) ÷ 5 g = 0.2 mg a.i./kg soil.

Table 4: Study design for the desorption phase.

Parameters		Soil I Sandy loam	Soil II Loamy sand	Soil III Silt loam	Soil IV Loam
Were the soil residues from the adsorption phase used? If not, describe the method for adsorption using a separate adsorption Table		Yes	Yes	Yes	Yes
Amount of test material present in the adsorbed state/adsorbed amount (mg a.i./kg soil)	0.2	0.120	0.133	0.171	0.196
	1.0	0.530	0.583	0.779	0.906
	5.0	2.086	2.549	3.259	4.172
	25.0	8.488	11.229	14.222	19.831
No. of desorption cycles		1	1	1	1
Equilibration solution and quantity used per treatment for desorption (eg., 0.01M $CaCl_2$)		0.01M $CaCl_2$	0.01M $CaCl_2$	0.01M $CaCl_2$	0.01M $CaCl_2$
Soil:solution ratio		1:5	1:5	1:5	1:5
Replications	Controls	2	2	2	2
	Treatments	2	2	2	2
Desorption equilibrium	Time (hours)	6	6	6	6
	Temperature (°C)	*ca.* 20	*ca.* 20	*ca.* 20	*ca.* 20
	Darkness	Yes	Yes	Yes	Yes
	Shaking method	Laboratory shaker	Laboratory shaker	Laboratory shaker	Laboratory shaker
	Shaking time (hours)	6	6	6	6
Centrifugation	Speed (rpm)	1720	1720	1720	1720
	Duration (min)	10	10	10	10
	Method of separation of soil and solution	Decanted	Decanted	Decanted	Decanted

Data were obtained from pp. 27-28 and Table 3, p. 45 of the study report.

3. Description of analytical procedures:

Extraction/clean up/concentration methods: Extraction/clean up/concentration methods were not employed in this study.

Total ^{14}C measurement: Following adsorption and desorption, aliquots (≤1.0 mL) of the supernatants were analyzed for total radioactivity using LSC (pp. 28-29). Following desorption,

soil samples (≤ 1.2 g, wet weight) were combusted in a stream of oxygen at 800°C using copper oxide as a catalyst; combustion efficiency was not reported. The liberated $^{14}CO_2$ was mixed with 10 mL of ethanolamine and ethylene glycol monomethyl ether (1:3, v:v), and duplicate aliquots were analyzed for total radioactivity using LSC.

Non-extractable residues, if any: Not applicable.

Derivatization method, if used: A derivatization method was not employed in the study.

Identification and quantification of parent compound: Following adsorption and desorption, supernatant samples from the highest (25.0 mg a.i./kg) and lowest (0.2 mg a.i./kg) test concentrations were analyzed for [^{14}C]halcomid by HPLC using the following operating conditions: Lichrospher 100 RP18 column (4.0 × 250 mm; 5-µm particle size), linear gradient mobile phase combining (Solvent A) water and (Solvent B) acetonitrile [%A:B at 0-5 min, 100:0; 5-25 min., 0:100; 25-28 min., 100:0; 28-35 min, 100:0], flow rate 1.5 mL/min., and UV detection (205 nm; pp. 27-28, 30-31).

Identification and quantification of transformation products, if appropriate: Samples were not analyzed for transformation products of halcomid.

Detection limits (LOD, LOQ) for parent compound: For HPLC analyses, the LOD was 0.009 mg/L (or ppm); the LOQ was not reported (p. 30).

Detection limits (LOD, LOQ) for the transformation products: Samples were not analyzed for transformation products of halcomid.

II. RESULTS AND DISCUSSION

A. TEST CONDITIONS: The incubation temperature was reported to be approximately 20°C during the study; temperature records were not provided (p. 27). Following adsorption, the pH of the test solutions for the highest (25.0 mg a.i./kg) and lowest (0.2 mg a.i./kg) test concentrations ranged from 6.71-8.32 and 6.75-8.73, respectively (pp. 28, 37; Table 16, p. 58). Following desorption, the pH of the test solutions for the highest and lowest test concentrations ranged from 6.82-8.04 and 6.84-8.25, respectively. Based on HPLC analysis, [^{14}C]halcomid was stable in the high- and low-dose adsorption and desorption supernatants, comprising ≥ 99.2% of the recovered radioactivity (pp. 37-38; Table 15, p. 57; Figures 17-18, pp. 75-76).

B. MASS BALANCE: The mass balance at the end of the adsorption phase was not reported. Mass balances at the end of the desorption phase were 32.79-47.90%, 32.33-43.28%, 18.60-32.71%, and 11.84-25.45% of the applied for the sandy loam, loamy sand, silt loam, and loam soils, respectively (p. 40; Tables 7-10, pp. 49-52).

Table 5: Recovery of [^{14}C]halcomid, expressed as percentage of applied radioactivity, after adsorption/desorption (mean ± s.d.).

Matrices	**Soil I Sandy loam**	**Soil II Loamy sand**	**Soil III Silt loam**	**Soil IV Loam**
At the end of the adsorption phase				
Supernatant solution	53.9 ± 9.1	48.5 ± 8.2	35.2 ± 10.9	22.1 ± 7.9
Solid phase (total ^{14}C)	Not analyzed			
Non-extractable residues in soil, if measured	Not measured			
Total recovery	Not determined			
At the end of the desorption phase				
Supernatant solution	18.7 ± 0.7	19.9 ± 0.9	16.2 ± 1.3	13.0 ± 2.4
Solid phase (total ^{14}C)[1]	27.8 ± 8.7	28.6 ± 7.1	53.1 ± 14.9	61.2 ± 10.6
Non-extractable residues in soil, if measured	Not measured			
Total recovery	100.5 ± 2.8	96.99 ± 2.10	104.6 ± 3.4	96.4 ± 7.2

Data were obtained from Tables 11-14, pp. 53-56 of the study report. Means and standard deviations were calculated by the reviewer using Excel.

[1] All soils were combusted following desorption.

Table 6: Concentration of [^{14}C]halcomid in the solid and liquid phases at the end of adsorption equilibration period (**n** = 2; mean ± s.d.).

Concentration (mg a.i./kg soil)	Soil I Sandy loam			Soil II Loamy sand		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]
0.2	0.120 ± 0.0	0.021 ± 0.0	51.1 ± 0.3	0.133 ± 0.0	0.019 ± 0.0	57.1 ± 2.3
1.0	0.530 ± 0.0	0.111 ± 0.0	46.5 ± 2.7	0.583 ± 0.0	0.103 ± 0.0	51.2 ± 0.1
5.0	2.086 ± 0.0	0.633 ± 0.0	37.2 ± 0.2	2.549 ± 0.0	0.560 ± 0.0	45.5 ± 0.4
25.0	8.488 ± 0.4	4.270 ± 0.1	26.4 ± 1.3	11.229 ± 0.6	3.852 ± 0.1	34.9 ± 2.1

Concentration (mg a.i./kg soil)	Soil III Silt loam			Soil IV Loam		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]
0.2	0.171 ± 0.0	0.011 ± 0.0	73.5 ± 0.0	0.196 ± 0.0	0.007 ± 0.0	84.0 ± 0.2
1.0	0.779 ± 0.0	0.065 ± 0.0	68.4 ± 0.5	0.906 ± 0.0	0.041 ± 0.0	79.6 ± 0.4
5.0	3.259 ± 0.0	0.424 ± 0.0	58.1 ± 0.1	4.172 ± 0.1	0.253 ± 0.0	74.4 ± 1.7
25.0	14.222 ± 0.3	3.254 ± 0.0	44.2 ± 0.8	19.831 ± 0.0	2.168 ± 0.0	61.6 ± 0.0

Data were obtained from Table 3, p. 45 and Tables 7-10, pp. 49-52 of the study report. Means and standard deviations were calculated by the reviewer using Excel.

[1] Percent adsorbed as % of the applied.

Table 7: Concentration of [^{14}C]halcomid in the solid and liquid phases at the end of desorption (n = 2; mean ± s.d.).

Concentration (mg a.i./kg soil)	Soil I Sandy loam			Soil II Loamy sand		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed[1]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed[1]
0.2	0.080 ± 0.0	0.009 ± 0.0	32.8 ± 0.1	0.089 ± 0.0	0.010 ± 0.0	33.2 ± 1.2
1.0	0.344 ± 0.0	0.043 ± 0.0	35.1 ± 1.2	0.380 ± 0.0	0.046 ± 0.0	34.8 ± 1.1
5.0	1.261 ± 0.0	0.209 ± 0.0	39.6 ± 0.1	1.567 ± 0.0	0.229 ± 0.0	38.5 ± 0.6
25.0	4.429 ± 0.2	1.146 ± 0.0	47.8 ± 0.1	6.488 ± 0.5	1.185 ± 0.0	42.3 ± 1.4

Concentration (mg a.i./kg soil)	Soil III Silt loam			Soil IV Loam		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed[1]	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed[1]
0.2	0.140 ± 0.0	0.007 ± 0.0	18.9 ± 0.4	0.172 ± 0.0	0.005 ± 0.0	12.0 ± 0.2
1.0	0.617 ± 0.0	0.036 ± 0.0	20.7 ± 0.3	0.769 ± 0.0	0.028 ± 0.0	15.1 ± 0.3
5.0	2.448 ± 0.0	0.187 ± 0.0	24.9 ± 0.0	3.421 ± 0.1	0.158 ± 0.0	18.0 ± 0.1
25.0	9.576 ± 0.2	1.140 ± 0.0	32.7 ± 0.1	14.874 ± 0.1	1.092 ± 0.0	25.0 ± 0.6

Data were obtained from Table 5, p. 47 and Tables 7-10, pp. 49-52 of the study report. Means and standard deviations were calculated by the reviewer using Excel.

[1] Percent desorbed as % of the adsorbed.

Table 8a: Reviewer-calculated average* adsorption coefficients (Kd) of [^{14}C]halcomid in the soils. (*Averaged over the four test concentrations, each with duplicate samples.)

Soil	Kd
Soil I Sandy loam	3.15
Soil II Loamy sand	4
Soil III Silt loam	8.29
Soil IV Loam	16.09

Adsorption K_d values were reviewer-calculated using data obtained from Tables 3, p. 45 of the study report and the following equation:

$$Kd = \frac{\left[\frac{(C_0 V_0 - C_{eq} V_0)}{m}\right]}{C_{eq}}$$

where
S = the sorbed phase concentration with units of mass of sorbate per solid sorbent mass;
C_0 = the concentration in the water before sorption;
V_0 = the total water volume in the batch system;
C_{eq} = the aqueous-phase equilibrium concentration; and
m = the dry mass of sorbent.

Table 8b: Adsorption and desorption constants of [^{14}C]halcomid in the soils.

Soil	Adsorption[1]				Desorption			
	K_F	1/N	R^2	K_{oc}	K_F	1/N	R^2	K_{oc}
Soil I Sandy loam	2.84	0.80	0.9990	350.6	4.26	0.83	0.9987	525.9
Soil II Loamy sand	3.84	0.83	0.9996	629.5	5.70	0.89	0.9999	934.4
Soil III Silt loam	6.03	0.78	1.00	568.9	9.16	0.83	0.9991	864.2
Soil IV Loam	11.41	0.80	1.00	559.3	14.62	0.83	0.9995	716.7

Data were obtained from Table 4, p. 46 and Table 6, p. 48 of the study report.
K_d -Adsorption and desorption coefficients; K_F - Freundlich adsorption and desorption coefficients; 1/N -Slope of Freundlich adsorption/desorption isotherms.
K_{oc} - Coefficient adsorption per organic carbon (K_d or K x 100/% organic carbon).
R^2 - Regression coefficient of Freundlich equation.
[1] Freundlich adsorption isotherms were calculated by the registrant using the following equation (p. 33):
log (X/m) = log K + 1/n log Ce
where
X/m = equilibrium concentration of the test article in the soil phase after adsorption;
K = adsorption constant;
1/n = slope of the adsorption isotherm; and
Ce = equilibrium concentration of the test article in the aqueous phase after adsorption.

C. ADSORPTION: After 6 hours of equilibration 26.4-51.1%, 34.9-57.1%, 44.2-73.5%, and 61.6-84.0% of the applied [^{14}C]halcomid was adsorbed to the sandy loam, loamy sand, silt loam,

and loam soils, respectively (Tables 7-10, pp. 49-52). Adsorption K_d values (reviewer-calculated) were 3.15, 4.00, 8.29, and 16.09 for the sandy loam, loamy sand, silt loam, and loam soils, respectively (Table 3, p. 45). Freundlich K_{ads} values (registrant-calculated) were 2.84, 3.84, 6.03, and 11.41 for the sandy loam, loamy sand, silt loam, and loam soils, respectively; corresponding Freundlich adsorption K_{oc} values were 350.6, 629.5, 568.9, and 559.3 (Table 4, p. 46).

D. DESORPTION: At the end of the desorption phase, 32.8-47.8%, 33.2-42.3%, 18.9-32.7%, and 12.0-25.0% of the applied halcomid was desorbed from the sandy loam, loamy sand, silt loam, and loam soils (Tables 7-10, pp. 49-52). Freundlich K_{des} values (registrant-calculated) were 4.26, 5.70, 9.16, and 14.62 for the sandy loam, loamy sand, silt loam, and loam soils, respectively; corresponding Freundlich desorption K_{oc} values were 525.9, 934.4, 864.2, and 716.7 (Table 6, p. 48).

III. STUDY DEFICIENCIES: No significant study deficiencies were noted.

IV. REVIEWER'S COMMENTS:

1. None of the test soils used in this study had an organic matter content ≤1%, as required by Subdivision N guidelines. However, the loamy sand soil had an organic matter content of 1.05%.

2. The diluted stock solution used to prepare the application solution for the definitive study was prepared by dissolving an aliquot (amount unspecified) of [^{14}C]halcomid in 15.75 mL of acetone (p. 26). The solution was concentrated to 2.0 mL under a stream of nitrogen and diluted to 350 mL with 0.01M $CaCl_2$ + 1% NaN_3 solution. The purity of the stock solution was determined to be 99.4%, based on HPLC analysis of duplicate samples.

3. The field application rate for halcomid was reported to be 600 g per ha (p. 23).

4. The physico-chemical properties of the test substance were incomplete; vapour pressure, UV adsorption, molecular weight, melting point, bulk density, pK_a, K_{ow}, and stability were not reported.

5. A complete description of the test soil collection and storage was not provided; pesticide use history at the collection site and collection procedures were not reported. The sampling depth at which the sandy loam soil was collected was not reported.

6. The definitive study temperature was reported to be approximately 20°C (p. 27). More detailed information was not provided. It is preferred that minimum, maximum, and average temperatures be reported. Any significant deviations from the average and their durations should be noted.

7. The moisture at 1/3 atm, bulk density, and microbial biomass were not reported for any of the test soils. Field capacity was reported only for the sandy loam soil.

8. It was not stated whether the test samples were stored prior to analysis. Storage intervals (if any) prior to sample analyses were not reported.

9. TLC analysis was used to determine the amount and purity of the test compound and to clean-up the test compound (pp. 31-32). TLC was performed using silica gel 60 F 254 coated plates (0.25 mm thickness) developed in chloroform:acetonitrile (50:50, v:v; Solvent System 1) and chloroform:acetonitrile:acetic acid (50:50:2, v:v:v; Solvent System 2). Radioactive areas were detected and quantified using a linear analyzer. The unlabeled test substance was visualized under UV light (254 nm). The Rf value of halcomid was determined to be 0.79 and 0.86 using Solvent Systems 1 and 2, respectively. The LOD for TLC analysis was 0.009 mg/L.

10. The radiochemical purity and stability of halcomid in the application solutions were determined to be ≥98.9%, based on HPLC analyses (p. 37; Table 15, p. 57; Figures 1-3, pp. 69-61).

11. An experimental protocol was not included in the study report.

12. Graphical computer printouts of the Freundlich adsorption and desorption isotherms are presented in Figures 5-8, pp. 63-66 and Figures 13-16, pp. 71-74 of the study report. Graphical computer printouts of the adsorption isotherms are presented in Figures 9-12, pp. 67-70 of the study report.

13. Many of the page numbers located at the upper right hand corner of the study report were illegible. Therefore, the reviewer referenced the handwritten page numbers located at the lower right hand corner of the study report pages.

V. REFERENCES:

1. U.S. Environmental Protection Agency. 1982. Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate, Section 163-1. Mobility studies. Office of Pesticide and Toxic Substances, Washington, DC. EPA 540/9-82-021.

2. U.S. Environmental Protection Agency. 1989. FIFRA Accelerated Reregistration, Phase 3 Technical Guidance. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 540/09-90-078.

3. U.S. Environmental Protection Agency. 1993. Pesticide Registration Rejection Rate Analysis - Environmental Fate. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 738.

4. U.S. Environmental Protection Agency. 2003. Guidance for Calculating Sorption Coefficients in Batch Equilibrium Studies.

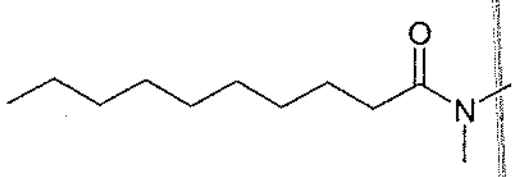
ATTACHMENT

Chemical Structure of Parent

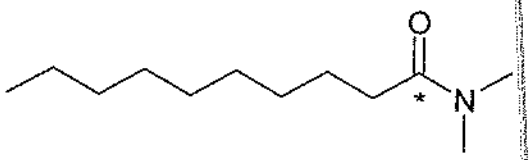
Halcomid

IUPAC name: N,N-Dimethyldecanoic acid amide.
CAS name: Not reported.
CAS No: Not reported.
SMILES string: O=C(CCCCCCCCC)N(C)C

Unlabeled



[1-^{14}C]Halcomid



* Position of radiolabel