Data Evaluation Record on the adsorption-desorption of AE C593600, a transformation product of clofentezine, in soil

PMRA Submission Number {.....} EPA MRID Number 47192104

Data Requirement: PMRA Data Code:
EPA DP Barcode: D342560
OECD Data Point:
EPA Guideline: 163-1

Test material:

Common name: AE C593600.

Chemical name:

IUPAC name: 2-Chlorobenzoic (2-chlorobenzylidene) hydrazide.
2-Chlorobenzoic acid (2-chlorobenzylidene)hydrazide.

CAS name: 2-Chlorobenzoic acid {(2-chlorophenyl)methylene}hydrazide.

CAS No.: Not reported.

Synonyms R1, M1, Compound III.

Smiles string: C1=CC=C(Cl)C(C=NNC(=O)C2=C(Cl)C=CC=C2)=C1 (EPI Suite v3.12 SMILES string from ISIS .MOL).

Primary Reviewer: Amy Barnes
Cambridge Environmental

Signature:
Date: 12/19/07

Secondary Reviewer: Joan Harlin
Cambridge Environmental

Signature:
Date: 12/19/07

QC/QA Manager: Joan Gaidos
Cambridge Environmental

Signature:
Date: 12/19/07

Final Reviewer: Lucy Shanaman
EPA Reviewer

Signature: Lucy Shanaman
Date: 2/20/08

Company Code:
Active Code:
Use Site Category:
EPA PC Code: 125501

CITATION: Deas, AHB. 1998. Clofentezine degradation product code: AE C593600 adsorption in three soils. Unpublished study performed by AgrEvo UK Limited, Essex, England; and sponsored and submitted by Makhteshim-Agan of North America, Inc. (location information not reported). Study Number: ENVIR/111J. Report Number: ENVIR\97\025. Experiment start and completion dates were not reported. Final report issued September 9, 1998.

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Date: 12/19/07

Secondary Reviewer: Joan Harlin
Cambridge Environmental

Signature: Joan Harlin
Date: 12/19/07

QC/QA Manager: Joan Gaidos
Cambridge Environmental

Signature:
Date: 12/19/07

Final Reviewer: Keara Moore
EPA Reviewer

Signature:
Date:

Company Code:
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Use Site Category:
EPA PC Code: 125501

CITATION: Deas, AHB. 1998. Clofentezine degradation product code: AE C593600 adsorption in three soils. Unpublished study performed by AgrEvo UK Limited, Essex, England; and sponsored and submitted by Makhteshim-Agan of North America, Inc. (location information not reported). Study Number: ENVIR/111J. Report Number: ENVIR\97\025. Experiment start and completion dates were not reported. Final report issued September 9, 1998.

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EXECUTIVE SUMMARY

The adsorption/desorption characteristics of [^{14}C]2-chlorobenzoic (2-chlorobenzylidene) hydrazide (label position not reported; AE C593600), a transformation product of clofentezine, were studied in a loam soil [pH 6.5, organic carbon 2.9%] and two sandy loam soils [pH 4.9, organic carbon 2.0% and pH 7.3, organic carbon 2.1%], each from England, in a batch equilibrium experiment. The experiment was conducted in accordance with European Commission Directive 95/36/EC. This study was conducted in compliance with OECD Principles of Good Laboratory Practice. The adsorption phase of the study was carried out by equilibrating air-dried soil with [^{14}C]AE C593600 at three nominal test concentrations: 0.2 mg a.i./kg, 1 mg a.i./kg (Shuttleworth and Abington soils), 2 mg a.i./kg (Terling soil), and 4 mg a.i./kg soil, for 2 hours; temperature and lighting conditions were not reported. The equilibrating solution used was 0.01M $CaCl_2$ solution, with a soil solution ratio of 1:25 (w:v) for all soils. A desorption phase was not conducted.

The supernatant solutions after adsorption were separated by centrifugation, and analyzed for total radioactivity using LSC. Following adsorption, soils were extracted once with acetone followed by centrifugation. Aliquots of the high-dose adsorption supernatants and soil extracts were analyzed for [^{14}C]AE C593600 and its transformation products using two-dimensional TLC analysis. The radioactivity in the soil residue after extraction was determined using LSC following combustion.

The experimental temperature employed during the study was not reported. The pH of the test solutions was not reported. In high-dose supernatants and soil extracts, [^{14}C]AE C593600 comprised 83.29-98.48% of the radioactivity, and the transformation product AE F092117 comprised 0.77-14.70%, based on two-dimensional TLC analysis. It was not stated that the test solutions were analyzed for parent compound prior to use in the study. Data were not provided.

Mass balances for duplicate soils at the end of the adsorption phase were 99.45 ± 1.2% (range 98.59-100.76%), 98.15 ± 0.8% (range 97.29-98.79%), and 97.08 ± 1.3% (range 95.58-97.99%) of the applied for the Shuttleworth sandy loam, Terling loam, and Abington sandy loam soils, respectively. A desorption phase was not conducted.

After 2 hours of equilibration, 58.4-67.0%, 60.5-65.5%, and 53.0-56.2% of the applied [^{14}C]AE C593600 residues was adsorbed to the Shuttleworth sandy loam, Terling loam, and Abington sandy loam soils, respectively. Calculated Freundlich adsorption K_d values, based on three test concentrations per test soil, were 21.7 for the Shuttleworth sandy loam soil, 24.7 for the Terling loam soil, and 15.6 for the Abington sandy loam soil. Corresponding Freundlich K_{oc} values were 1084, 851, and 742. Calculated adsorption K_d values were 22.98, 25.09, and 14.72 for the Shuttleworth sandy loam, Terling loam, and Abington sandy loam soils, respectively. Corresponding K_{oc} values were 1149, 865, and 701. A desorption phase was not conducted.

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Results Synopsis:

Soil type:	Shuttleworth Sandy loam.
Amount adsorbed:	58.4-67.0% of the applied.
Adsorption K_d:	23.0
Adsorption K_{oc}:	1150
Freundlich adsorption K_d:	21.7
Freundlich adsorption K_{oc}:	1080
Amount desorbed:	Not applicable.
Desorption K_d:	Not applicable.
Desorption K_{oc}:	Not applicable.
Freundlich desorption K_d:	Not applicable.
Freundlich desorption K_{oc}:	Not applicable.

Soil type:	Terling Loam.
Amount adsorbed:	60.5-65.5% of the applied.
Adsorption K_d:	25.1
Adsorption K_{oc}:	865
Freundlich adsorption K_d:	24.7
Freundlich adsorption K_{oc}:	851
Amount desorbed:	Not applicable.
Desorption K_d:	Not applicable.
Desorption K_{oc}:	Not applicable.
Freundlich desorption K_d:	Not applicable.
Freundlich desorption K_{oc}:	Not applicable.

Soil type:	Abington Sandy loam.
Amount adsorbed:	53.0-56.2% of the applied.
Adsorption K_d:	14.7
Adsorption K_{oc}:	701
Freundlich adsorption K_d:	15.6
Freundlich adsorption K_{oc}:	742
Amount desorbed:	Not applicable.
Desorption K_d:	Not applicable.
Desorption K_{oc}:	Not applicable.
Freundlich desorption K_d:	Not applicable.
Freundlich desorption K_{oc}:	Not applicable.

Study Acceptability: This study is classified as **supplemental.** No significant deviations from good scientific practices were noted. The study was conducted using only three soil types. It could not be determined if the English test soils were comparable to soils found in typical use areas in the United States. The experimental temperature and lighting conditions were not

reported. The study was conducted using a transformation product of clofentezine, rather than the parent compound. Desorption was not studied.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: This study was conducted in accordance with European Commission Directive 95/36/EC amending Council Directive 91/414/EC, with reference to the OECD Guidelines 106 (1981), the draft revision OECD Guidelines 106 (1996) and the guidelines given in SETAC-Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides (p.9). The following significant deviations from the objectives of Subdivision N guidelines were noted:

The study was conducted using only two soil types.

It could not be determined if the English test soils used in the study were typical of the pesticide use area in the U.S.

The study was conducted using a transformation product of clofentezine, rather than the parent compound.

The experimental temperature and lighting conditions were not reported.

Desorption was not studied.

COMPLIANCE: This study was conducted in compliance with OECD Principles of Good Laboratory Practice (1981; p.3). Signed and dated Data Confidentiality, GLP, and Quality Assurance statements were provided (pp.2-4). A Certificate of Authenticity was not provided; an Approvals page was provided (p.5).

A. MATERIALS:

1. Test Material [^{14}C]AE C593600 (p.9).

Chemical Structure: See DER Attachment 1.

Description: Technical grade (radiolabeled test material).

Purity:

Radiolabeled

Radiochemical purity: 98.8% (p. 10; Appendix 1, p.32).
Batch No.: Not reported.
Analytical purity: Not reported.
Specific radioactivity: 273 µCi/mg (p.10).
Location of the label: Not reported

Unlabeled

Analytical purity: 97.0% (Appendix 1, p.32).
Batch No.: R000164.

Storage conditions of test chemicals:

Storage conditions were not reported.

Physico-chemical properties of AE C593600:

Parameter	Value	Comment
Molecular weight	Not reported.	
Molecular formula	Not reported.	
Water Solubility	0.32 mg/L	At 20°C.
Vapor Pressure/Volatility	Not reported.	
UV Absorption	Not reported.	
pKa	5.35-10.35	At 20°C.
K_{ow}/log K_{ow}	Not reported.	
Stability of compound at room temperature, if provided	Not reported.	

Data were obtained from p.10; Appendix 2, p.34 of the study report.

2. Soil Characteristics

Table 1: Description of soil collection and storage.

Description	**Sandy loam**	**Loam**	**Sandy loam**
Geographic location	Shuttleworth Farm Estate, Old Warden, Bedfordshire.	Terling Hall, Hatfield Peverel, Essex.	Land Settlement, Abington, Cambs.
Site description	Not reported.		
Pesticide use history at the collection site	No previous pesticide treatment.		
Collection procedures	Not reported.		
Sampling depth (cm)	Not reported.		
Storage conditions	Stored in the dark at 20°C.		
Storage length (days)	Not reported.		
Soil preparation	Air-dried and sieved (2 mm).		

Data were obtained from p.10; Table 1, p.17 of the study report.

Table 2: Properties of the soils.

Property	Shuttleworth	Terling	Abington
Soil texture (USDA)[1]	Sandy loam	Loam	Sandy loam
% Coarse sand (2000-600 µm)	2.21	8.75	1.69
% Medium sand (600-212 µm)	50.52	20.82	30.06
% Fine sand (212-63 µm)	19.20	10.98	30.09
% Coarse silt/fine sand (63-53 µm)	0.47	5.66	2.29
% Coarse silt (53-20 µm)	8.30	16.62	10.40
% Fine silt (20-2 µm)	12.59	13.50	9.37
% Clay (<2 µm)	6.70	23.68	16.10
pH (1:5) in 0.01M $CaCl_2$	4.9	6.5	7.3
pH (1:5) in water	5.7	6.7	8.3
Organic carbon (%)	2.0	2.9	2.1
Organic matter (%)[2]	3.4	5.0	3.6
CEC (meq/100 g soil)	11.3	16.3	18.9
$CaCO_3$ (%)	Not reported.		
Moisture at 1/3 atm (%)	Not reported.		
Bulk density (g/mL)	Not reported.		
Biomass (mg microbial C/100 g or CFU or other)	Not reported.		
Soil taxonomic classification	Not reported.		
Soil mapping unit (for EPA)	Not reported.		

Data were obtained from Table 1, p.17 of the study report.
1 Textural classifications were confirmed using the NRCS soil texture calculator http://soils.usda.gov/technical/aids/investigations/texture/ which calculates texture based on the percent sand and clay.
2 Calculated as % organic carbon x 1.72.

C. STUDY DESIGN:

1. Preliminary study: Preliminary experiments were conducted to determine the water solubility and pK_a of AE C593600, the appropriate adsorption equilibrium time to be used in the definitive study, and adsorption of the AE C593600 to the test vessels, and to assure the stability of AE C593600 during the selected exposure time to be used in the definitive study (pp.10-11). A preliminary experiment to determine the optimal soil:solution ratio to be used in the definitive study was not reported.

Preparation of stock solutions for preliminary studies was not reported.

The water solubility and pK_a of AE C593600 at 20°C were determined. The experimental details were not reported (pp.10-11, Appendix 2, p.34). It was reported that AE C593600 has a solubility of 0.32 mg/L, and two dissociation constants: $pK_1 = 5.35$ and $pK_2 = 10.35$.

To determine the adsorption equilibration time, (2 x 2 g) of the Shuttleworth sandy loam and Terling loam test soils were added to screw top centrifuge tubes and suspended in *ca.* 50 mL of [^{14}C]AE C593600 in 0.01M $CaCl_2$ solution at a nominal test concentration of 0.16 mg/L (p.11). The samples were shaken on an end-over-end shaker for 6, 24, and 48 hours; temperature and lighting conditions were not reported. Following each sampling, the samples were centrifuged and triplicate aliquots of the supernatants were analyzed for total radioactivity using LSC. Aliquots were analyzed by TLC. Acetone (*ca.* 10 mL) was added to the test soils and the samples were shaken for 1 hour followed by centrifugation; temperature and lighting conditions were not reported. Triplicate aliquots of the soil extracts were analyzed using LSC. The remaining soil was air-dried and analyzed by LSC following combustion. The highest K_d values were obtained for samples shaken for 6 hours. For samples shaken for 24 or 48 hours, the concentration of the radioactivity in the supernatant increased, resulting in lower distribution coefficients. AE C593600 was determined to be unstable in the test system, comprising 75.49-82.63% of the radioactivity in the supernatants following 6 hours of shaking; the transformation product AE F092117 comprised 14.05-22.33% of the radioactivity (p.12; Appendices 3a-3c, pp,35-37).

To determine adsorption of the test substance to the glass test vessels, treated aqueous samples (50 mL) without soil were exposed for 24 hours and analyzed by LSC (p.11). No adsorption to the test tubes was observed.

Based on the results of the preliminary studies, an adsorption equilibration time of 2 hours was selected for use in the definitive study (p.12).

2. Definitive study experimental conditions:

Table 3: Study design for the adsorption phase.

Parameters		**Shuttleworth Sandy loam**	**Terling Loam**	**Abington Sandy loam**
Condition of soil (air dried/fresh)		Air-dried.		
Have these soils been used for other laboratory studies? (specify which)				
Soil (g/replicate)		*ca.* 2		
Equilibrium solution used (eg: 0.01N $CaCl_2$)		0.01M $CaCl_2$		
Control used (with salt solution only) (Yes/No)		No.		
Test material concentrations [1]	Nominal application rates (mg a.i./kg soil)	0.25, 1, 4	0.25, 2, 4	0.25, 1, 4
	Analytically measured concentrations (mg a.i./kg soil)	Not reported.		
Identity and concentration of co-solvent, if any		Acetone, 0.1% (v:v)		
Soil:solution ratio (w:v)		1:25		
Initial pH of the equilibration solution, if provided		Not reported.		
No. of replications	Controls	None.		
	Treatments	Duplicate.		
Equilibration	Time (hours)	2		
	Temperature (°C)	Not reported.		
	Darkness (Yes/No)	Not reported.		
	Shaking method	End-over-end shaker.		
	Shaking time (hours)	2		
Method of separation of supernatant (eg., centrifugation)		Centrifugation.		
Centrifugation	Speed (rpm)	4000		
	Duration (min)	60		
	Method of separation of soil and solution	Not reported.		

Data were obtained from pp.10-12; Tables 6-8, pp.22-24 of the study report.

1 Test material concentrations were calculated by converting mg/L to mg a.i./kg using the following equation: [test concentration (mg/L) × total volume of test material (mL)] ÷ amount of soil (g); eg. For the Shuttleworth sandy loam, lowest concentration, nominal application rate: [0.01 mg/L × 50 mL] ÷ 2 g = 0.25 mg a.i./kg soil.

Table 4: Study design for the desorption phase[1].

Parameters		Shuttleworth Sandy loam	Terling Loam	Abington Sandy loam
Were the soil residues from the adsorption phase used? If not, describe the method for adsorption using a separate adsorption				
Amount of test material present in the adsorbed state/adsorbed amount (mg a.i./kg soil)				
No. of desorption steps				
Equilibration solution and quantity used per treatment for desorption (eg., 0.01M $CaCl_2$)				
Soil:solution ratio (w:v)				
Replications	Controls			
	Treatments			
Desorption equilibration	Time (hours)			
	Temperature (°C)			
	Darkness			
	Shaking method			
	Shaking time (hours)			
Centrifugation	Speed (rpm)			
	Duration (min)			
	Method of separation of soil and solution			
Second desorption step				

1 A desorption phase was not conducted.

3. Description of analytical procedures:

Extraction/clean up/concentration methods: Following adsorption, soils were extracted once with acetone (*ca.* 10 mL) by shaking for 1 hour followed by centrifugation (pp.11-12).

Total ^{14}C measurement: Following adsorption and extraction, triplicate aliquots of the supernatants were analyzed for total radioactivity using LSC (pp.11-12). Mass balances were determined for soils by summing the radioactivity recovered in the adsorption supernatants, soil extracts, and unextracted radiocarbon (Tables 2-4, pp.18-20).

Non-extractable residues, if any: Following adsorption and extraction, triplicate aliquots of the soils were air-dried, combusted, and analyzed for total radioactivity using LSC (pp.12-13). Combustion efficiency was not reported.

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

Identification and quantification of parent compound: Aliquots of the high-dose adsorption supernatants and soil extracts were analyzed for [^{14}C]AE C592600 using two-dimensional TLC on silica gel plates (SilGUV254; 20 cm x 20 cm; 0.25-mm thickness) using Solvent 1 [chloroform:methanol:formic acid (98:2:0.5, v:v:v)] for the first dimension, and Solvent 2 [toluene:ethyl acetate:ethanol:acetic acid (80:5:10:0.5, v:v:v:v)] for the second dimension (p.13). Following development, areas of radioactivity were detected and quantified using autoradiography and a Rita-3200 Radio-TLC-Analyser. Unlabeled reference standards were visualized under ultraviolet light (254 nm). [^{14}C]AE C593600 was identified by comparison to the Rf value of an unlabeled reference standard of AE C593600 (purity 97.0%; Figures 1-3, pp.26-28; Appendix 1, p.32).

Identification and quantification of transformation products, if appropriate: Samples were analyzed for transformation products of AE C592600 as described above for the parent compound (p.13). The following reference standards were used (p.10; Appendix 1, p.32): clofentezine (AE B084866, purity 99.9%), 2-chlorobenzonitrile (AE F023666, purity 99.9%), and 2-chlorobenzamide (AE F092117, purity 98.0%).

Detection limits (LOD, LOQ) for the parent compound: Detection limits for LSC and TLC analyses were not reported.

Detection limits (LOD, LOQ) for the transformation products, if appropriate: Detection limits for LSC and TLC analyses were not reported.

II. RESULTS AND DISCUSSION

A. TEST CONDITIONS: The experimental temperature employed during the study was not reported. The pH of the test solutions was not reported. It was not stated that the test solution was analyzed for [^{14}C]AE C593600 prior to study initiation. In the high-dose adsorption supernatants and soil extracts, [^{14}C]AE C593600 comprised 83.29-98.48% of the radioactivity; the transformation product AE F092117 comprised 0.77-14.70%, based on two-dimensional TLC analysis (p.15; Table 5, p.21; Figures 1-3, pp.26-28).

B. MASS BALANCE: Mass balances for duplicate soils at the end of the adsorption phase were 99.45 ± 1.2% (range 98.59-100.76%), 98.15 ± 0.8% (range 97.29-98.79%), and 97.08 ± 1.3% (range 95.58-97.99%) of the applied for the Shuttleworth sandy loam, Terling loam, and Abington sandy loam soils, respectively (p.15; Tables 2-4, pp.18-20). A desorption phase was not conducted.

Table 5: Recovery of [^{14}C]AE C593600, expressed as percentage of applied radioactivity, in soils after adsorption/desorption (mean ± s.d.).

Matrices	**Shuttleworth Sandy loam**	**Terling Loam**	**Abington Sandy loam**
At the end of the adsorption phase			
Supernatant solution	44.27 ± 3.2	44.29 ± 2.2	52.55 ± 3.1
Solid phase (extracted)	49.22 ± 2.7	48.03 ± 1.9	38.23 ± 2.7
Non-extractable residues in soil, if measured	5.96 ± 0.5	7.83 ± 0.9	6.30 ± 1.4
Total recovery	99.45 ± 1.2	98.15 ± 0.8	97.08 ± 1.3
At the end of the desorption phase[1]			
Supernatant solution			
Solid phase (total ^{14}C)			
Non-extractable residues in soil, if measured			
Total recovery			

Data were obtained from Tables 2-4, pp.18-20 of the study report.
1 A desorption phase was not conducted.

Table 6: Concentration of [^{14}C]AE C593600 residues in the solid and liquid phases at the end of adsorption equilibration period (mean ± s.d.; n = 2).

Concentration (mg a.i./kg soil)	Shuttleworth Sandy loam			Terling 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]
0.25	0.1540 ± 0.0	0.0046 ± 0.0	61.6 ± 0.0	0.1639 ± 0.0	0.0046 ± 0.0	65.5 ± 2.6
1, 2	0.6698 ± 0.0	0.0226 ± 0.0	67.0 ± 0.8	1.2090 ± 0.0	0.0401 ± 0.0	60.5 ± 1.8
4	2.3340 ± 0.0	0.0884 ± 0.0	58.4 ± 0.6	2.5615 ± 0.0	0.0873 ± 0.0	64.0 ± 0.1

Concentration (mg a.i./kg soil)	Abington Sandy loam		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% adsorbed[1]
0.25	0.1404 ± 0.0	0.0057 ± 0.0	56.2 ± 1.1
1	0.5295 ± 0.0	0.0251 ± 0.0	53.0 ± 1.0
4	2.1624 ± 0.1	0.1132 ± 0.0	54.1 ± 1.7

Data were obtained from Tables 6-8, pp.22-24 of the study report. Means and standard deviations were determined using Excel.

1 Percent adsorbed was calculated by dividing the amount in the soil by the nominal test concentrations x 100%; e.g. for the Shuttleworth sandy loam, lowest concentration [0.1540 mg a.i./kg ÷ 0.25 mg a.i./kg soil] x 100% = 61.6%.

Table 7: Concentration of [^{14}C]AE C593600 residues in the solid and liquid phases at the end of the desorption step (mean ± s.d.)[1].

Concentration (mg a.i./kg soil)	Shuttleworth Sandy loam			Terling Loam		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed
0.25						
1, 2						
4						

Concentration (mg a.i./kg soil)	Abington Sandy loam		
	on soil (mg a.i./kg)	in solution (µg a.i./mL)	% desorbed as % of the adsorbed
0.25			
1			
4			

1 A desorption phase was not conducted.

Table 8: Adsorption and desorption constants of [^{14}C]AE C593600 in the soils.

Soil	Adsorption						Desorption					
	K_d[1]	K_F[2]	1/n	r^2	K_{oc}	K_{Foc}	K_d	K_F	1/n	r^2	K_{oc}	K_{Foc}
Shuttleworth Sandy loam	22.98	21.7	0.92	1.000	1149	1084	NA	NA	NA	NA	NA	NA
Terling Loam	25.09	24.7	0.93	0.998	865	851	NA	NA	NA	NA	NA	NA
Abington Sandy loam	14.72	15.6	0.91	0.999	701	742	NA	NA	NA	NA	NA	NA

Data were obtained from p.15; Table 9, p.25; and Figures 4-6, pp.29-31 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.
NA = Not applicable.
1 Adsorption K_d values were calculated using data obtained from Tables 6-8, pp.22-24 of the study report and the following EPA-approved equation:

$K_d = [(C_0V_0 - CeqV_0) \div m] \div Ceq$ 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;
Ceq = the aqueous-phase equilibrium concentration; and
m = the dry mass of sorbent.

2 Freundlich K_F values were calculated by the study author using the following equation (p.14 of the study report):

$\log C_a = \log K_F + (1/n) \log (C_s)$, where

C_a = soil concentration after adsorption (µg/g);
C_s = concentration of supernatant after adsorption (µg/mL);
1/n = exponential constant or slope; and
K = Freundlich sorption constant.

C. ADSORPTION: After 2 hours of equilibration, 58.4-67.0%, 60.5-65.5%, and 53.0-56.2% of the applied [^{14}C]AE C593600 residues was adsorbed to the Shuttleworth sandy loam, Terling loam, and Abington sandy loam soils, respectively (Tables 6-8, pp.22-24). Calculated Freundlich adsorption K_d values, based on three test concentrations per test soil, were 21.7 for the Shuttleworth sandy loam soil, 24.7 for the Terling loam soil, and 15.6 for the Abington sandy loam soil; corresponding Freundlich K_{oc} values were 1084, 851, and 742 (p.15; Table 9, p.25). Adsorption K_d values were 22.98, 25.09, and 14.72 for the Shuttleworth sandy loam, Terling loam, and Abington sandy loam soils, respectively; corresponding K_{oc} values were 1149, 865, and 701.

D. DESORPTION: A desorption phase was not conducted.

III. STUDY DEFICIENCIES

1. The study was conducted using a transformation product of clofentezine, rather than the parent compound.

2. The study was conducted using only three soils, representing only two soil types. Subdivision N Guidelines specify that a minimum of four test soils with characteristics that are sufficiently different so as to represent a full spectrum of the types of soils representative of the proposed use areas, be used in batch equilibrium studies.

3. It was not established that the English soils used in this study were comparable to soils that would be found at the intended use sites in the United States. Their FAO classifications were not provided.

4. Complete details of the experimental design were not reported. The experimental temperature and lighting conditions were not reported. Subdivision N guidelines specify that the temperature of the slurries should be maintained a temperature within the range of normal environmental parameters (18-30°C). The equilibration should be conducted in the dark to minimize photodegradation. During the experiment, AE C593600 degraded in the test solutions. In the adsorption high-dose supernatants and soil extracts, [^{14}C]AE C593600 accounted for 83.29-98.48% of the radioactivity, and the transformation product AE F092117 accounted for 0.77-14.70%, based on two-dimensional TLC analysis.

5. Desorption was not studied.

IV. REVIEWER'S COMMENTS

1. Calculated the Freundlich adsorption K_d and K_{oc} values based on the three test concentrations employed for each test soil. A minimum of four test concentrations should be used in batch equilibrium studies, in order to calculate accurate Freundlich K_d and K_{oc} values.

2. It was not stated that the test solutions were analyzed for [^{14}C]AE C593600 prior to study initiation, in order to confirm its stability in the test solution prior to use in the study. Data were not reported.

3. The position of the radiolabel of the test substance was not reported or depicted in the structure figure provided in the study report (p.9).

4. A preliminary experiment to determine the optimal soil:solution ratio to be used for each test soil in the definitive study was not reported.

5. The calculated r^2 value for Kads vs. % organic carbon was 0.3336, Kads vs. pH was 0.3792, and Kads vs. % clay was 0.0173.

6. AE C593600 is reported to have low mobility in soils (p.15).

7. The equilibration time preliminary experiment for the Abington sandy loam soil was started but abandoned due to instrument failure on the 48-hour samples (p.11).

8. For the Terling loam soil, the intended test concentration of 0.04 mg/L was made up at 0.08 mg/L as a result of a dilution error (p.12).

9. None of the test soils had an organic matter content of ≤1%, as recommended by Subdivision N guidelines.

10. Detection limits for LSC and TLC analyses were not reported.

11. The study author stated that although the Shuttleworth and Abington sandy loam soils are classified as having the same texture, the Shuttleworth soil is on the border with loamy sand (USDA and ADAS) soil or silty sand (BBA) soil (p.10). The author stated that the Shuttleworth and Abington sandy loam soils were selected for use in the definitive study due to their very different pH values, whereas their organic carbon contents are similar. The author added that this was a relevant comparison to make for AE C593600 due to its acid dissociation constant. AE C593600 has two acid dissociation constants: $pK_1 = 5.35$ and $pK_2 = 10.35$.

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.

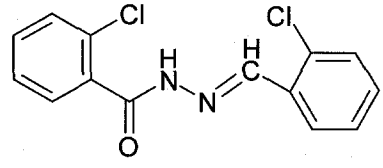
4. U.S. Environmental Protection Agency. 2003. Guidance for Calculating Sorption Coefficients in Batch Equilibrium Studies.

Attachment 1: Structure of Test Material

AE C593600 [R1, M1, Compound III]

IUPAC Name:	2-Chlorobenzoic (2-chlorobenzylidene) hydrazide. 2-Chlorobenzoic acid (2-chlorobenzylidene)hydrazide.
CAS Name:	2-Chlorobenzoic acid {(2-chlorophenyl)methylene}hydrazide.
CAS Number:	Not reported.
SMILES String:	C1=CC=C(Cl)C(C=NNC(=O)C2=C(Cl)C=CC=C2)=C1 (EPI Suite v3.12 SMILES string from ISIS .MOL).

Unlabeled



[^{14}C]AE C593600

Label position not reported.

* = Location of the radiolabel.

Attachment 2: Excel Spreadsheets

Chemical: AE C593600
PC Code: 125501
MRID: 47192104
Guideline No: 163-1

Table 4/6 Adsorption on soil

	Sandy loam	Loam	Sandy loam
0.01	0.1540	0.1684	0.1385
0.01	0.1540	0.1593	0.1423
AVG	0.1540	0.1639	0.1404
STDEV	0.0	0.0	0.0
0.04/0.08	0.6753	1.2344	0.5368
0.04/0.08	0.6643	1.1835	0.5222
AVG	0.6698	1.2090	0.5295
STDEV	0.0	0.0	0.0
0.16	2.3508	2.5650	2.2104
0.16	2.3171	2.5580	2.1143
AVG	2.3340	2.5615	2.1624
STDEV	0.0	0.0	0.1

Data were obtained from Tables 6-8, pp. 22-24 of the study report.

Table 5 Adsorption solution

	Sandy loam	Loam	Sandy loam
0.01	41.02	40.35	49.17
0.04/0.08	44.43	41.82	53.22
0.16	47.36	44.70	55.27
AVG	44.27	42.29	52.55
STDEV	3.2	2.2	3.1

Data were obtained from Tables 2-4, pp. 18-20 of the study report.

Chemical: AE C593600
PC Code: 125501
MRID: 47192104
Guideline No: 163-1

Table 5 Extracted

	Sandy loam	Loam	Sandy loam
0.01	51.14	49.68	40.99
0.04/0.08	50.37	48.45	38.09
0.16	46.16	45.96	35.61
AVG	49.22	48.03	38.23
STDEV	2.7	1.9	2.7

Table 5 Non-extractable

	Sandy loam	Loam	Sandy loam
0.01	6.43	8.78	7.51
0.04/0.08	5.96	7.02	6.68
0.16	5.48	7.70	4.70
AVG	5.96	7.83	6.30
STDEV	0.5	0.9	1.4

Table 5 Recovery

	Sandy loam	Loam	Sandy loam
0.01	98.59	98.79	97.67
0.04/0.08	100.76	97.29	97.99
0.16	99.00	98.36	95.58
AVG	99.45	98.15	97.08
STDEV	1.2	0.8	1.3

Data were obtained from Tables 2-4, pp. 18-20 of the study report.

Chemical: AE C593600
PC Code: 125501
MRID: 47192104
Guideline No: 163-1

Table 6 Adsorption solution

	Sandy loam	Loam	Sandy loam
0.01	0.0046	0.0044	0.0058
0.01	0.0046	0.0048	0.0056
AVG	0.0046	0.0046	0.0057
STDEV	0.0	0.0	0.0
0.04/0.08	0.0223	0.0390	0.0248
0.04/0.08	0.0228	0.0411	0.0253
AVG	0.0226	0.0401	0.0251
STDEV	0.0	0.0	0.0
0.16	0.0876	0.0869	0.1113
0.16	0.0892	0.0876	0.1150
AVG	0.0884	0.0873	0.1132
STDEV	0.0	0.0	0.0

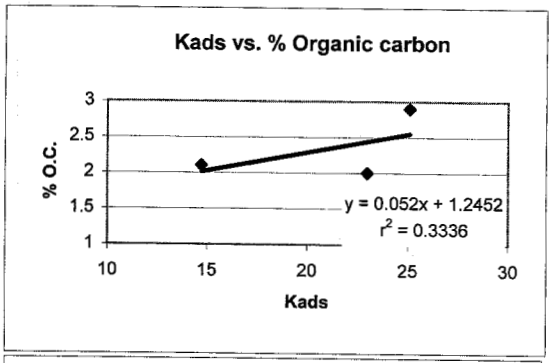
Table 6 % Adsorbed

	Sandy loam	Loam	Sandy loam
0.25	61.60	67.36	55.40
0.25	61.60	63.72	56.92
AVG	61.60	65.54	56.16
STDEV	0.0	2.6	1.1
1/'2	67.53	61.72	53.68
1/'2	66.43	59.18	52.22
AVG	66.98	60.45	52.95
STDEV	0.8	1.8	1.0
4	58.77	64.13	55.26
4	57.93	63.95	52.86
AVG	58.35	64.04	54.06
STDEV	0.6	0.1	1.7

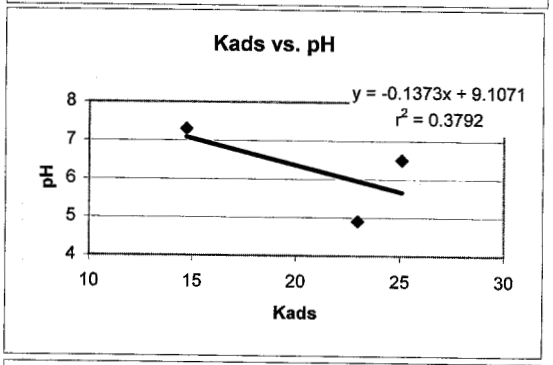
Data were obtained from Tables 6-8, pp. 22-24 of the study report.

Chemical: AE C593600
PC Code: 125501
MRID: 47192104
Guideline No: 163-1

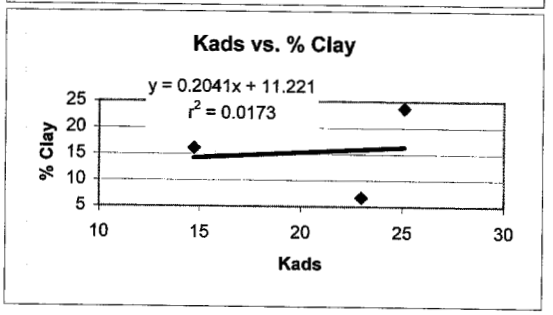
Soil	Kads	% organic carbon
Sandy loam	22.98	2
Loam	25.09	2.9
Sandy loam	14.72	2.1



Soil	Kads	pH
Sandy loam	22.98	4.9
Loam	25.09	6.5
Sandy loam	14.72	7.3



Soil	Kads	% clay
Sandy loam	22.98	6.7
Loam	25.09	23.68
Sandy loam	14.72	16.1



Data were obtained from Table 1, p. 17 of the study report. Kads values were reviewer-calculated using data obtained from Tables 6-8, pp. 22-24 of the study report.

Chemical: AE C593600
PC Code: 125501
MRID: 47192104
Guideline No: 163-1

SUMMARY OUTPUT (% Organic carbon)

Regression Statistics	
Multiple R	0.57758967
R Square	0.33360982
Adjusted R Square	-0.33278035
Standard Error	0.56948212
Observations	3

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	0.162356781	0.162357	0.500622	0.607986771
Residual	1	0.324309886	0.32431		
Total	2	0.486666667			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	1.24523618	1.57259989	0.791833	0.57363	-18.73653999	21.22701	-18.73653999	21.22701235
X Variable 1	0.05198744	0.073475631	0.707547	0.607987	-0.88160897	0.985584	-0.88160897	0.985583854

SUMMARY OUTPUT (pH)

Regression Statistics	
Multiple R	0.61576997
R Square	0.37917266
Adjusted R Square	-0.24165469
Standard Error	1.36169172
Observations	3

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	1.132462337	1.132462	0.610754	0.577690122
Residual	1	1.85420433	1.854204		
Total	2	2.986666667			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	9.10705262	3.760251928	2.421926	0.249283	-38.67147823	56.88558	-38.67147823	56.88558348
X Variable 1	-0.13730145	0.175687971	-0.781507	0.57769	-2.369628774	2.095026	-2.369628774	2.095025879

Chemical: AE C593600
PC Code: 125501
MRID: 47192104
Guideline No: 163-1

SUMMARY OUTPUT (% Clay)

Regression Statistics	
Multiple R	0.13152254
R Square	0.01729818
Adjusted R Square	-0.96540364
Standard Error	11.9251418
Observations	3

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	2.503258809	2.503259	0.017603	0.916026853
Residual	1	142.2090079	142.209		
Total	2	144.7122667			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	11.2208012	32.93075596	0.340739	0.790933	-407.204126	429.6457	-407.204126	429.6457285
X Variable 1	0.20413436	1.538603743	0.132675	0.916027	-19.34567981	19.75395	-19.34567981	19.75394852

Chemical: AE C593600
PC Code: 125501
MRID: 47192104
Guideline No: 163-1

Shuttleworth Sandy loam - Adsorption

Initial soln concen (C_o) (ug/mL)	Volume of soln (V_o) (mL)	Concen in soln after equil (C_{eq}) (ug/mL)	Volume of soln (V_o) (mL)	Dry mass of sorbent (m) (g)	$[(C_oV_o)-(C_{eq}V_o)]$/soil mass	Kd	
0.01	50	0.0046	50	2	0.1350	29.35	
0.01	50	0.0046	50	2	0.1350	29.35	
0.04	50	0.0223	50	2	0.4425	19.84	
0.04	50	0.0228	50	2	0.4300	18.86	
0.16	50	0.0876	50	2	1.8100	20.66	
0.16	50	0.0892	50	2	1.7700	19.84	
						22.98	AVG

Terling Loam- Adsorption

Initial soln concen (C_o) (ug/mL)	Volume of soln (V_o) (mL)	Concen in soln after equil (C_{eq}) (ug/mL)	Volume of soln (V_o) (mL)	Dry mass of sorbent (m) (g)	$[(C_oV_o)-(C_{eq}V_o)]$/soil mass	Kd	
0.01	50	0.0044	50	2	0.1400	31.82	
0.01	50	0.0048	50	2	0.1300	27.08	
0.08	50	0.0390	50	2	1.0250	26.28	
0.08	50	0.0411	50	2	0.9725	23.66	
0.16	50	0.0869	50	2	1.8275	21.03	
0.16	50	0.0876	50	2	1.8100	20.66	
						25.09	AVG

Abington Sandy loam- Adsorption

Initial soln concen (C_o) (ug/mL)	Volume of soln (V_o) (mL)	Concen in soln after equil (C_{eq}) (ug/mL)	Volume of soln (V_o) (mL)	Dry mass of sorbent (m) (g)	$[(C_oV_o)-(C_{eq}V_o)]$/soil mass	Kd	
0.01	50	0.0058	50	2	0.1050	18.10	
0.01	50	0.0056	50	2	0.1100	19.64	
0.04	50	0.0248	50	2	0.3800	15.32	
0.04	50	0.0253	50	2	0.3675	14.53	
0.16	50	0.1113	50	2	1.2175	10.94	
0.16	50	0.1150	50	2	1.1250	9.78	
						14.72	AVG

Data were obtained from Tables 6-8, pp. 22-24 of the study report.

Koc

	Kd	Organic carbon %	Koc
Sandy loam	22.98	2.0	1149
Loam	25.09	2.9	865
Sandy loam	14.72	2.1	701

Data were obtained from Table 1, p. 17 and Tables 6-8, pp. 22-24.