

US EPA ARCHIVE DOCUMENT

**Data Evaluation Report on the adsorption-desorption of XDE-742 (pyroxsulam) in soil**

PMRA Submission Number 2006-4727

EPA MRID Number 46908332

**Data Requirement:** : PMRA DATA CODE: 8.2.4.2a  
 EPA DP Barcode: 332118  
 OECD Data Point: IIA 7.4.1, IIA 7.4.2  
 EPA Guideline: Subdivision N, § 163-1 Leaching and Adsorption/Desorption

**Test material:**

Common name: XDE-742 (pyroxsulam)  
 Chemical name:  
 IUPAC: N-(5,7-dimethoxy[1,2,4]triazolo[1,5- $\alpha$ ]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide  
 CAS name: N-(5,7-dimethoxy[1,2,4]triazolo[1,5- $\alpha$ ]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide  
 CAS No: 422556-08-9  
 Synonyms: DE-742, XR-742  
 Smiles string: c1(c(ccnc1OC)C(F)(F)F)S(Nc2nn3c(n2)nc(cc3OC)OC)(=O)=O

**Primary Reviewer:** J.D. Whall (PMRA)

**Signature:**

**Date:** March 4, 2007

**Secondary Reviewer:** Hemendra Mulye (PMRA)

**Date:** March 8, 2007

**Greg Orrick (USEPA)**

**Date:** June 18, 2007

**David McAdam (AUS DEW)**

**Date:** 30 May 2007

**PMRA Company Code:** DWE

**PMRA Active Code:** JUA

**PMRA Use Site Category:** 13, 14

**EPA PC Code:** 108702

**CITATION:** Smith, J.K., 2004, Soil Batch Equilibrium Adsorption/Desorption of 14C-XDE-742, Dow AgroSciences LLC, 9330 Zionsville Road, Indianapolis, IN 46268, 030069, M. D. Culy, 7 April 2004.



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### EXECUTIVE SUMMARY:

The adsorption/desorption characteristics of radiolabelled XDE-742 were studied in twenty soils of varying textures, organic matter contents and pHs in a batch equilibrium experiment. The experiment was conducted in accordance with the OECD 106, FIFRA 163-1, SETAC Part 1 Section 4, and OPPTS 835.1220 guidelines, and to meet the GLP standards 40 CFR part 160. A preliminary (Tier 2) study was conducted using 16 European soils, 2 U. S. soils, and 2 Canadian soils to determine  $K_d$  values. Based on the results of the preliminary test, the definitive (Tier 3) isotherm test was conducted at a 1:5 soil:solution ratio with 10 European soils. The adsorption phase of the definitive isotherm study was carried out by equilibrating fresh soil with XDE-742 at 0.025, 0.050, 0.125, 0.250 and 0.500  $\mu\text{g a.i./g soil}$  (or, 0.005, 0.010, 0.025, 0.05 and 0.1  $\mu\text{g a.i./mL}$ ) in the dark at 25 °C for 72 hours. The equilibration solution used was 0.01 M  $\text{CaCl}_2$ , with a soil:solution ratio of 1:5. The desorption phase of the study was carried out by adding approximately the amount of 0.01 M  $\text{CaCl}_2$  removed for adsorption and equilibrating in the dark at 25 °C for 24 hours. The samples were desorbed once. The supernatant solution after adsorption and desorption was separated by centrifugation and the XDE-742 residues were analyzed by HPLC with fraction collection. The fractions were then assayed by LSC. The soils were extracted three times with 90:10 acetone:0.1 N HCl. The extracts were concentrated using a turbo evaporator and analyzed by HPLC fitted with a fraction collector. The  $^{14}\text{C}$  residue remaining in the soil after extraction was determined by combustion.

For the definitive isotherm study,  $K_d$  and  $K_{OC}$  values were re-calculated by the PMRA by combining data from both replicates into a single adsorption isotherm and by using single-point desorption isotherms from the highest test concentration. For the adsorption phase, the average  $K_d$  value for the ten soils was 0.57 mL/g (range 0.19 to 1.76 mL/g); the corresponding average  $K_{OC}$  values were 30.0 mL/g (range 7.1 to 54.3 mL/g). Following a single desorption cycle, the average  $K_d$  value for the ten soils was 0.42 mL/g (range 0.13 to 1.27 mL/g); the corresponding average  $K_{OC-des}$  value was 22.3 mL/g (range 5.0 to 46.0 mL/g).

When adsorption  $K_{oc}$  values are plotted against the pH of the soil, the inverse relationship between  $K_{oc}$  and pH is apparent. Since  $K_{oc}$  is simply  $K_d$ /soil organic carbon content, this shows that pH is a good indicator of XDE-742 adsorption provided that the influence of organic carbon is also considered. In other words, adsorption of XDE-742 is influenced by both pH and soil organic carbon content, as the soil pH decreases the  $K_{oc}$  value increases.

Freundlich adsorption isotherm plots were also generated by the PMRA reviewer for the definitive isotherm data. Freundlich adsorption correlation coefficients ranged from 0.809 to 0.995, and  $1/n$  values from 0.93 to 1.21. Adsorption  $K_F$  values ranged from 0.18 to 1.60  $\mu\text{g}^{1-1/n} \text{ mL}^{1/n} \text{ g}^{-1}$ , and corresponding  $K_{FOC-ads}$  values ranged from 7.2 to 68.0  $\mu\text{g}^{1-1/n} \text{ mL}^{1/n} \text{ g}^{-1}$ , respectively. Freundlich desorption correlation coefficients ranged from 0.883 to 0.999, and  $1/n$  values ranged from 0.33 to 0.86. Desorption  $K_F$  values ranged from 0.04 to 0.51  $\mu\text{g}^{1-1/n} \text{ mL}^{1/n} \text{ g}^{-1}$ , and corresponding  $K_{FOC-des}$  values ranged from 1.0 to 18.0  $\mu\text{g}^{1-1/n} \text{ mL}^{1/n} \text{ g}^{-1}$ , respectively.

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Adsorption of XDE-742 in the range of soils tested is generally linear with respect to concentration (i.e., the majority of the slopes of the Freundlich adsorption coefficients  $[1/n]$  fall within the range of 0.9 - 1.1). Therefore, adsorption can be described using non-Freundlich  $K_{OC-ads}$  values. **Based on the PMRA-calculated adsorption coefficients in the ten soils used in the definitive study (average  $K_{OC-ads} = 29.97$  mL/g [range 7.09 to 54.26 mL/g]), XDE-742 Technical can be considered very highly mobile according to the classification criteria of McCall et al. (1981) and considered mobile to highly mobile according to the FAO classification scheme (FAO, 2000). Desorption coefficients (average  $K_{OC-des} = 22.3$  mL/g [range 5.0 to 46.0 mL/g]), indicate that XDE-742 does not bind irreversibly with soil, and can readily desorb. This study is scientifically sound and satisfies the DACO requirements for an adsorption/desorption study with the active ingredient (DACO 8.2.4.2).**

### PMRA Results Synopsis:

		Adsorption - PMRA Values						
		Freundlich				Non-Freundlich		
Soil	pH <sup>a</sup>	r <sup>2</sup>	1/n	K <sub>F-ads</sub> <sup>b</sup>	K <sub>FOC-ads</sub> <sup>b</sup>	r <sup>2</sup>	K <sub>d-ads</sub> <sup>c</sup>	K <sub>OC-ads</sub> <sup>c</sup>
M641	6.2	0.990	1.01	0.50	55.4	0.990	0.49	54.3
M642	7.8	0.984	0.94	0.24	9.7	0.993	0.29	11.8
M644	7.7	0.837	0.93	0.18	22.7	0.422	0.22	27.8
M645	7.8	0.809	1.21	0.29	22.6	0.800	0.20	15.0
M646	5.9	0.979	0.93	1.04	38.6	0.957	1.32	48.9
M649	7.6	0.948	0.98	0.27	7.1	0.810	0.27	7.1
M650	5.4	0.995	0.96	1.60	43.3	0.992	1.76	47.7
M660	6.3	0.913	0.97	0.29	28.9	0.684	0.28	28.5
M661	5.7	0.963	1.11	0.88	68.0	0.989	0.67	51.2
M662	7.9	0.931	0.98	0.19	7.4	0.894	0.19	7.5

<sup>a</sup> soil pH

<sup>b</sup>  $\mu\text{g}^{-1-1/n} \text{mL}^{1/n} \text{g}^{-1}$

<sup>c</sup>  $\text{mLg}^{-1}$

		Desorption - PMRA Values						
		Freundlich				Non-Freundlich		
Soil	pH <sup>a</sup>	r <sup>2</sup>	1/n	K <sub>F-des</sub> <sup>b</sup>	K <sub>FOC-des</sub> <sup>b</sup>	r <sup>2</sup>	K <sub>d-des</sub> <sup>c</sup>	K <sub>OC-des</sub> <sup>c</sup>

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M641	6.2	0.986	0.50	0.15	17	0.997	0.41	46
M642	7.8	0.998	0.86	0.21	8	0.990	0.28	11
M644	7.7	0.996	0.34	0.04	4	0.998	0.13	16
M645	7.8	0.883	0.47	0.06	4	0.841	0.18	14
M646	5.9	0.987	0.35	0.25	9	0.977	0.81	30
M649	7.6	0.982	0.33	0.05	1	0.947	0.18	5
M650	5.4	0.998	0.54	0.51	14	0.996	1.27	34
M660	6.3	0.999	0.36	0.05	5	0.997	0.18	18
M661	5.7	0.998	0.56	0.24	18	0.998	0.56	43
M662	7.9	0.983	0.37	0.04	2	0.933	0.15	6

<sup>a</sup> soil pH

<sup>b</sup>  $\mu\text{g}^{1-1/n} \text{mL}^{1/n} \text{g}^{-1}$

<sup>c</sup>  $\text{mLg}^{-1}$

**Study Acceptability:** This study is classified as acceptable and satisfies the guideline requirement for an adsorption/desorption study in soil.

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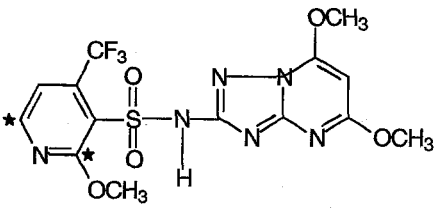
## I. MATERIALS AND METHODS

**GUIDELINE FOLLOWED:** This study was conducted to fulfill U.S. Environmental Protection Agency (EPA) requirements for aerobic soil metabolism as outlined in the EPA Pesticide Registration Guidelines, Subdivision N, §163-1, Leaching and Adsorption/Desorption. There were no deviations reported from the guidelines.

**COMPLIANCE:** This study was also conducted to meet Good Laboratory Practices standards, 40 CFR Part 160. Signed and dated GLP, Quality Assurance and Data Confidentiality statements were provided.

### A. MATERIALS:

#### 1. Test Material XDE-742 (pyroxsulam)

Chemical Structure	
Common Name	XDE-742-pyridine-2,6- <sup>14</sup> C
Description	Radiolabeled test material, radiolabel denoted with “*”
Radiochemical Purity	98.4%
Inventory #	INV1905
Specific Activity	43.7 mCi/mmol
Storage Conditions	Stable at -20 °C

#### Physico-chemical properties of XDE-742 (pyroxsulam):

Parameter	Values	Comments
Water solubility		(1)
pH 4	0.0164 g/L at 20 °C	
pH 7	3.20 g/L at 20 °C	
pH 9	13.7 g/L at 20 °C	
Unbuffered	0.0626 g/L at 20 °C	
Vapor Pressure	<1E-7 Pa	(2)
pKa	N/A	not available at time of report
Log D		(3)
pH 4	1.080	
pH 7	-1.010	
pH 9	-1.600	
Stability of Compound at Room Temperature	N/A	not available at time of report

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### 2. Soil Characteristics

Table 1: Description of soil collection and storage

Soil ID	Collection Date <sup>a</sup>	Pesticide Use History <sup>b</sup>	Sampling Depth (cm)
M640	March 18	Glyphomax Plus; Fallow	0-7 inches
M641	March 19	Glypohsate	18
M642	February 25	None	20
M643	February 25	None	20
M644	February 25	None; elemental Sulfur	20
M645	March 21	None	5-15
M646	March 20	None; not indicated	8-11
M647	March 20	MCPA and 2, 4-D; None	4-5 + 9 cm
M648	March 26	None	6 inches
M649	March 24	None	10
M650	March 26	None for 5 yrs	10
M651	March 26	None for 5 yrs	10
M652	March 24	None for several years	10
M655	April 15	None	3-5 inches
M657	April 21	Glyphosate	0-6 inches
M658	April 10	Isoxaflutole; none	0-20
M659	April 17	Oxadiazon, molinate, bensulfuron methyl	0-20
M660	March 25	Prochloraz, Epoxiconazol, Fenpropimorph, Kersoxim, Iodosulfuron, Mefenpyr, Cinidon, Chlormequat; Pendimethalin, Flufenact, Bentazon, Dichlorprop-P	0-25
M661	March 6	None	0-25
M662	February 17	None since 1997	0-25

<sup>a</sup> Dates are all 2003

<sup>b</sup> Pesticide Use History is previous two years unless otherwise stated.

The soils were sieved through a 2-mm mesh screen before using. Agvise Laboratories, Inc. characterized the soils. Soil moistures (based on the dry soil weight) were determined using a Denver IR Moisture Analyzer.



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Table 2: Properties of the soils used in the definitive adsorption/desorption study.

Property	M641	M642	M644	M645
Location	Charentilly, France	Baden- Wurtemberg, Germany	Rhineland- Pfalz, Germany	Lincolnshire, England
Common Name	Charentilly	3A	LUFA 5M	Site I
USDA Textural Class	Silt Loam	Sandy Loam	Sandy Loam	Loamy Sand
% sand	17	55	59	79
% silt	56	28	28	12
% clay	27	17	13	9
International Textural Class	Light Clay	Clay Loam	Sandy Loam	Loamy Sand
% sand	45	63	73	85
% silt	28	20	14	6
% clay	27	17	13	9
ADAS Textural Class	Silty Clay Loam	Sandy Loam	Sandy Loam	Sandy Loam
% sand	13	51	53	75
% silt	60	32	34	16
% clay	27	17	13	9
German BBA Textural Class	Silty Loam	Sandy Loam	Loamy Sand	Loamy Sand
% sand	13	47	51	75
% silt	60	36	36	16
% clay	27	17	13	9
pH <sup>a</sup>	6.2	7.8	7.7	7.8
Organic Matter (%)	2.1	3.4	1.6	2.8
Organic Carbon (%) <sup>b</sup>	0.9	2.5	0.8	1.3
CEC (meq/100 g)	13.8	17.5	8.4	11.4
Moisture at 1/3 atm (%)	20.6	22.0	12.7	10.8
Bulk Density (g/cm <sup>3</sup> )	1.10	1.15	1.14	1.18
Biomass <sup>c</sup>	86.8	512.5	49.3	580.7

<sup>a</sup> 1:1 soil:water ratio

<sup>b</sup> LECO

<sup>c</sup> µg/g dry weight soil



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Table 2: Properties of the soils used in the definitive adsorption/desorption study (continued).

Property	M646	M649	M650	M660
Location	Derbyshire, England	Herts, England	Essex, England	Schifferstad, Germany
Common Name	Site D	Site 1	Site 7	Speyerer Wald
USDA Textural Class	Sandy Loam	Clay Loam	Loam	Sandy Loam
% sand	69	31	49	64
% silt	20	34	42	24
% clay	11	35	9	12
International Textural Class	Sandy Loam	Light Clay	Sandy Loam	Sandy Loam
% sand	77	43	71	72
% silt	12	22	20	16
% clay	11	35	9	12
ADAS Textural Class	Sandy Loam	Clay Loam	Sandy Silt Loam	Sandy Loam
% sand	67	27	47	62
% silt	22	38	44	26
% clay	11	35	9	12
German BBA Textural Class	Loamy Sand	Sandy Clay Loam	Silty Loamy sand	Loamy Sand
% sand	67	27	45	62
% silt	22	38	46	26
% clay	11	35	9	12
pH <sup>a</sup>	5.9	7.6	5.4	6.3
Organic Matter (%)	5.5	5.0	6.2	1.7
Organic Carbon (%) <sup>b</sup>	2.7	3.8	3.7	1.0
CEC (meq/100 g)	11.6	19.1	13.8	8.1
Moisture at 1/3 atm (%)	21.4	25.7	19.9	11.2
Bulk Density (g/cm <sup>3</sup> )	0.98	1.02	0.98	1.25
Biomass <sup>c</sup>	620.4	466.4	354.5	77.8

<sup>a</sup> 1:1 soil:water ratio

<sup>b</sup> LECO

<sup>c</sup> µg/g dry weight soil

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Table 2: Properties of the soils used in the definitive adsorption/desorption study (continued).

Property	M661	M662
Location	Borstel, Germany	Bruch West, Germany
Common Name	Borstel	Bruch West
USDA Textural Class	Loamy Sand	Sandy Loam
% sand	82	65
% silt	14	23
% clay	4	12
International Textural Class	Loamy Sand	Sandy Loam
% sand	89	76
% silt	7	12
% clay	4	12
ADAS Textural Class	Loamy Sand	Sandy Loam
% sand	82	62
% silt	14	26
% clay	4	12
German BBA Textural Class	Silty Sand	Loamy Sand
% sand	82	62
% silt	14	26
% clay	4	12
pH <sup>a</sup>	5.7	7.9
Organic Matter (%)	1.9	2.3
Organic Carbon (%) <sup>b</sup>	1.3	2.5
CEC (meq/100 g)	7.3	13.3
Moisture at 1/3 atm (%)	8.9	12.6
Bulk Density (g/cm <sup>3</sup> )	1.40	1.29
Biomass <sup>c</sup>	55.7	144.6

<sup>a</sup> 1:1 soil:water ratio

<sup>b</sup> LECO

<sup>c</sup> µg/g dry weight soil

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### **C. STUDY DESIGN:**

#### **1. Preliminary study:**

The solubility of  $^{14}\text{C}$ -XDE-742 in 0.01 M  $\text{CaCl}_2$  was verified by dosing at a nominal rate of 0.01  $\mu\text{g}/\text{mL}$ . The solution was shaken, and aliquots removed for determination of the concentration of  $^{14}\text{C}$ -XDE-742 in solution at twenty minutes and after overnight shaking. To determine container adsorption a nominal 0.01- $\mu\text{g}/\text{mL}$  dosing solution of the test substance in 0.01 M  $\text{CaCl}_2$  was prepared. Triplicate 1-mL aliquots of the dosing solution were analyzed by LSC. Aliquots (20-mL) of the dosing solution were then added to duplicate centrifuge tubes (glass, polysulfone and polycarbonate) and equilibrated on the shaker for a minimum of 16 hours. After equilibration, triplicate 1-mL aliquots of the equilibrated solution were analyzed by LSC. To determine matrix interference, an aliquot of two soils (one with high organic matter (M646) and one with high clay content and low organic matter (M659)) plus two controls (no soil) were equilibrated with 25-mL of 0.01 M  $\text{CaCl}_2$  by shaking in closed centrifuge tubes at room temperature for a minimum of 4 hours at a 1:5, w:w, soil:solution ratio. The aqueous phase was separated by centrifugation to remove soil particles and flocculent organic matter. A 0.5-mL aliquot of  $^{14}\text{C}$ -XDE-742 was added to the aqueous phase to create a nominal concentration of 0.01  $\mu\text{g}/\text{mL}$ . The aqueous phases for both soils matrices and the blanks were analyzed by LSC and HPLC.

A Tier I preliminary study was conducted to evaluate the approximate soil/solution ratios and equilibration time required for two soil matrices (high organic matter and low clay content (M646), and the other soil's low organic matter and high clay content (M659)). Duplicates of the two soil types were prepared for each time point at three soil:solution ratios. Weights of soils were 5 g, 5 g, and 2 g target dry soil weight for soil:solution ratios 1:2, 1:5, and 1:10, respectively. Aliquots of 0.01 M  $\text{CaCl}_2$ , (9 mL, 22.5 mL and 18 mL) were added to each soil volume. After equilibration the samples were dosed with 1 mL, 2.5 mL and 2 mL dose solution to achieve a nominal  $^{14}\text{C}$ -XDE-742 concentration of 0.01  $\mu\text{g}/\text{mL}$  and an overall soil:solution ratio of 1:2, 1:5, and 1:10, respectively. The dosing solution was analyzed in triplicate by LSC both before and after dosing to determine concentration and homogeneity of the solution. An aliquot of the dosing solution was analyzed by HPLC to determine radiopurity. In addition, the pH of an aliquot of the dosing solution in 0.01 M  $\text{CaCl}_2$  was measured. Duplicate controls containing test substance but no soil in 0.01 M  $\text{CaCl}_2$  solution were included. Duplicate blanks for each soil type containing soil and 0.01 M  $\text{CaCl}_2$  solution (soil: solution ratio of 1:2 only), but no test substance, were also included. The test, blank, and control samples were equilibrated in a darkened incubator on a reciprocating shaker. The incubator was set at 25 °C. At defined time intervals (approximately 2, 4, 8, 24, and 48 hours after initiation) duplicate test and control samples were removed from the shaker. The control samples were only

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analyzed by LSC at the 24-hour time interval. Blank samples were also removed from the shaker and triplicate aliquots were removed for LSC analysis, and the samples immediately returned to the shaker. The soil and solution phases in the test samples were separated by centrifugation and then decanted. The volume of the adsorption supernatant was measured (by weight) and triplicate aliquots of the supernatant were analyzed by LSC. HPLC was conducted on 1:5 soil:solution supernatant at 4, 8, 24 and 48 hours, and on the control samples at 24 hours. The radioactivity remaining on the soil pellet was determined by combustion to provide <sup>14</sup>C-mass balance. Extractions were performed on selected soils and the extract was analyzed by LSC.

A Tier II preliminary study was conducted as well to determine the required adsorption equilibration time for the remaining soils. Two replicated of all 20 soils were prepared for each time point at a 1:5 soil:solution ratio with 0.01 M CaCl<sub>2</sub> and dosed at a nominal concentration of 0.01 µg/mL. Test samples, controls, and blanks were equilibrated at 25 °C in a darkened incubator on a reciprocating shaker. At approximately 4, 8, 24, 48, and 72 hours after initiation, samples were removed from the shaker and analyzed under the same protocol as the Tier I preliminary study.

**2. Definitive study experimental conditions:**

For the definitive adsorption/desorption study, the following ten soil types were used: M641, M642, M644, M645, M646, M649, M650, M660, M661 and M662.

Table 3: Study design for the adsorption phase.

Parameter	Description	
Condition of soil (air dried/fresh)	Fresh	
Have these soils been used for other laboratory studies? (specify which)	Yes. Aerobic Soil Metabolism	
Soil (g/replicate)	5 g DWB	
Equilibration solution used	22.5 mL of 0.01 M CaCl <sub>2</sub>	
Control used (with salt solution only)	Yes (24 hours only)	
Test Material Concentrations	Nominal concentration (µg a.i./mL solution)	0.01 µg/mL
	Measured concentration (µg a.i./mL solution)	0.004 – 0.015 µg/mL
Identity and concentration of co-solvent:	Acetonitrile	
Soil: solution ratio	1:5	
Initial pH of the equilibration solution	N/A	
No. of replication	Controls	Duplicates
	Treatments	Duplicates per sample type

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Equilibration	Time	4, 8, 24, 48 and 72 hours
	Temperature (°C)	25 °C
	Darkness (Yes/No)	Yes
	Shaking method	Horizontal shaker at high speed
	Shaking time	72 hours for adsorption test
Method of separation of		Centrifugation
Centrifugation	Speed (rpm)	3500 rpm
	Duration (min)	30 minutes
	Method of separation of soil and solution	Decant

Table 4: Study design for the desorption phase.

Parameter		Description
Were the soil residues from the adsorption phase used?		Yes
Amount of test material present in the adsorbed state		0.01 µg/mL
Number of Desorption cycles		1
Equilibration solution used		22.8 mL of 0.01 M CaCl <sub>2</sub>
Soil solution Ratio		1:5
Replications	Controls	Duplicates
	Treatments	Duplicates
Desorption Equilibration	Time	2, 4, 8, 24 and 48 hours
	Temperature (°C)	25 °C
	Darkness (Yes/No)	Yes
	Shaking method	Horizontal shaker at high speed
	Shaking time	2, 4, 8, 24 and 48 hours for desorption test
Method of separation of supernatant		Centrifugation
Centrifugation	Speed (rpm)	3500 rpm
	Duration (min)	30 minutes
	Method of separation of soil and solution	Decant

### 3. Description of analytical procedures:

The soil pellets were extracted for purposes of <sup>14</sup>C mass balance and stability verification (as necessary).



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### *Extraction*

Soil pellets were extracted with 3 x 5 mL of the extraction solvent (90:10 v:v, acetonitrile:0.1N HCl). After addition of the extraction solvent, the tubes were capped and the soil pellets resuspended by shaking and vortexing. The samples were then placed on a shaker for 20 minutes at high speed. The samples were removed and the phases separated by centrifugation. The extracts were then decanted into 25-mL volumetric flasks. The process was repeated an additional two times and the extracts were combined. The final volume was adjusted to 25 mL using acetonitrile and transferred to a labeled vial. Triplicate 1-mL aliquots of each extract were analyzed by LSC for recovery calculations. Soils were allowed to air dry for combustion analysis.

### *Soil Extract Concentration*

Soil extracts were concentrated prior to HPLC analysis. For these samples, 10-mL aliquots of the soil extracts were transferred to a 15-mL conical glass vials. The extracts were reduced under nitrogen in a Turbo-Vap evaporator to <0.5 mL. The concentrates were then quantitatively transferred to 2-mL volumetric flasks with 95:5 v:v, water with 1% acetic acid: acetonitrile with 1% acetic acid. Duplicate 100- $\mu$ L aliquots of each concentrate were analyzed by LSC. The remaining samples were transferred to 2.0-mL HPLC auto sampler vials.

Radioactivity measurements were made using an LSC.  $^{14}\text{C}$ -Quench curves were generated for each instrument once every 6 months. The quench curve was used to resolve sample efficiency and convert the raw counts per minute (cpm) to disintegrations per minute (dpm). Aliquots of soil (approximately 0.5 g sub-samples) were combusted in triplicate to determine  $^{14}\text{C}$  mass balance. All combustion was performed using a Harvey Biological Oxidizer OX-500 using a scintillation cocktail (R.J. Harvey Carbon-14 Counting Cocktail) as a trapping agent and scintillation. The trapped  $^{14}\text{C}$  activity was measured by LSC analysis.

The oxidizer efficiency was determined by combusting known levels of  $^{14}\text{C}$  standard on cellulose and determining the amount of  $^{14}\text{C}$  activity recovered vs. the amount applied. The efficiency of the oxidizer was checked both before and after combustion of a sample set. Acceptable recoveries of 80 to 110 % were observed for all combustion sets.

HPLC was used as the primary analytical method. The following HPLC parameters were used to verify the radiopurity of the test substance, analyze dosing solutions, adsorption and desorption supernatants, and soil extracts. The HPLC system was equipped with a Zorbax 300 SB-C18 (4.6 x 250 mm, 5 micron packing) with an appropriate guard column. HPLC conditions consisted of the gradient using water with 1% acetic acid and acetonitrile with 1% acetic acid as mobile phase. The gradient conditions are summarized below:

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Time	% A <sup>a</sup>	% B <sup>b</sup>
0	95	5
5	95	5
20	5	95
24.2	5	95

<sup>a</sup> water with 1% acetic acid

<sup>b</sup> acetonitrile with 1% acetic acid

The flow rate was 1 mL/minute and the UV detector wavelength was 254 nm. Typically 1800 µL of sample was injected with 100 µL reference standards. Fraction collection began after 5 minutes (to account for HPLC void volume) and ended at 24.2 minutes for a total collection time of 19.2 minutes. Fractions of 0.1 mL were collected into two 96-well plates with 0.150 mL scintillation cocktail in each well.

Using the method of Currie (4), the limit of detection can be calculated from the expression:

$$LOD = \frac{2.71 + 4.65\sqrt{dpm_B \times T}}{T}$$

and the limit of quantitation can be calculated from the expression:

$$LOQ = \frac{50 \left( 1 + \sqrt{1 + \frac{dpm_B \times T}{12.5}} \right)}{T}$$

where LOD is the limit of detection (dpm), LOQ is the limit of quantitation (dpm), dpmB is the typical background (dpm) and T is the counting time (minutes). Samples were typically counted for 5 minutes while the blank was typically counted for 10 minutes. Typical background levels were 20 dpm. The resulting LOD was 10 dpm above background and LOQ was 40 dpm above background. The quantitation limit of <sup>14</sup>C for the subsamples (e.g., organic extracts, combustions) and HPLC analyses were <8.5% of applied radiocarbon for each process. Limits of quantitation and detection for each subsample as a percentage of the applied radiocarbon are given below.



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Sample Description	% of Applied <sup>14</sup> C	
	LOD	LOQ
Adsorption Supernatant-0.005 ppm	1.910	7.761
Adsorption Supernatant-0.01 ppm	0.972	3.951
Adsorption Supernatant-0.025 ppm	0.403	1.637
Adsorption Supernatant-0.05 ppm	0.196	0.799
Adsorption Supernatant-0.1 ppm	0.101	0.411
Desorption Supernatant-0.005 ppm	1.910	7.761
Desorption Supernatant-0.01 ppm	0.972	3.951
Desorption Supernatant-0.025 ppm	0.403	1.637
Desorption Supernatant-0.05 ppm	0.196	0.799
Desorption Supernatant-0.1 ppm	0.101	0.411
Organic Extracts-0.005 ppm	2.076	8.436
Organic Extracts-0.01 ppm	1.057	4.294
Organic Extracts-0.025 ppm	0.438	1.779
Organic Extracts-0.05 ppm	0.214	0.868
Organic Extracts-0.1 ppm	0.110	0.446
Soil Combustions-0.005 ppm	0.830	3.374
Soil Combustions-0.01 ppm	0.423	1.718
Soil Combustions-0.025 ppm	0.175	0.712
Soil Combustions-0.05 ppm	0.085	0.347
Soil Combustions-0.1 ppm	0.044	0.179
HPLC Analyses - Ads/Des-0.005 ppm	1.061	4.312
HPLC Analyses - Ads/Des-0.01 ppm	0.540	2.195
HPLC Analyses - Ads/Des-0.025 ppm	0.224	0.909
HPLC Analyses - Ads/Des-0.05 ppm	0.109	0.444
HPLC Analyses - Ads/Des-0.1 ppm	0.056	0.228
HPLC Analyses - Organic-0.005 ppm	0.369	1.500
HPLC Analyses - Organic-0.01 ppm	0.188	0.763
HPLC Analyses - Organic-0.025 ppm	0.078	0.316
HPLC Analyses - Organic-0.05 ppm	0.038	0.154
HPLC Analyses - Organic-0.1 ppm	0.020	0.079

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### II. RESULTS AND DISCUSSION

**A. TEST CONDITIONS:** The radiochemical stability of each test substance throughout the various tests was demonstrated by HPLC analysis of representative samples of the various phases.

**B. MASS BALANCE:** Mass balance was calculated for each definitive sample as the sum of the radioactivity recovered from the adsorption supernatant, the desorption supernatant (if applicable), the organic extract, and combustion of the extracted soil pellet

Table 5: Recovery of XDE-742 expressed as percentage of applied radioactivity, in soil after adsorption/desorption

Soil	Adsorption	Desorption	Extract	Soil Pellet	Recovery
0.005 mg/L Application Rate					
M641	89.8	3.5	2.0	0.7	96.0
M642	92.5	1.3	0.9	0.5	95.1
M644	94.6	0.5	0.6	0.5	96.2
M645	95.6	0.2	0.7	0.5	97.0
M646	75.8	8.5	8.9	3.6	96.9
M649	94.0	1.3	0.5	0.8	96.6
M650	71.8	12.8	8.8	1.9	95.4
M660	91.0	2.0	1.6	0.4	94.9
M661	87.1	5.4	3.5	0.8	96.8
M662	94.6	0.9	0.4	0.5	96.4
0.010 mg/L Application Rate					
M641	92.1	3.0	1.6	0.4	97.1
M642	95.1	1.3	0.9	0.3	97.7
M644	94.6	0.9	0.4	0.2	96.0
M645	98.3	0.6	0.2	0.2	99.3
M646	77.9	8.8	8.7	2.0	97.4
M649	94.6	0.8	0.6	0.3	96.3
M650	71.8	12.3	9.6	1.8	95.6
M660	94.9	2.5	1.1	0.2	98.7
M661	88.9	5.9	3.1	0.4	98.3
M662	96.7	0.8	0.3	0.2	98.0

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Soil	Adsorption	Desorption	Extract	Soil Pellet	Recovery
<b>0.025 mg/L Application Rate</b>					
M641	89.7	3.7	1.6	0.3	95.3
M642	94.7	1.8	0.7	0.4	97.6
M644	96.9	1.3	0.6	0.3	99.0
M645	93.8	0.9	0.5	0.2	95.6
M646	78.2	8.4	8.7	3.1	98.4
M649	94.2	1.2	0.7	0.5	96.6
M650	76.4	12.1	7.3	1.5	97.3
M660	94.2	2.7	1.1	0.3	98.2
M661	90.3	4.1	2.0	0.4	96.8
M662	96.6	1.0	0.5	0.2	98.3
<b>0.050 mg/L Application Rate</b>					
M641	89.9	3.4	1.6	0.5	95.3
M642	94.6	1.7	0.7	0.4	97.4
M644	88.1	1.8	0.5	0.3	90.7
M645	94.3	0.9	0.4	0.2	95.8
M646	76.1	8.9	8.5	3.0	96.4
M649	93.8	1.9	0.7	0.5	96.8
M650	72.8	14.2	8.9	1.5	97.5
M660	91.9	2.9	1.0	0.3	96.0
M661	89.6	6.0	2.4	0.4	98.3
M662	95.0	1.2	0.4	0.2	96.9
<b>0.10 mg/L Application Rate</b>					
M641	90.7	3.2	1.1	0.4	95.4
M642	94.0	1.4	0.8	0.3	96.5
M644	96.6	1.2	0.5	0.2	98.5
M645	95.1	0.9	0.0	0.2	96.2
M646	77.9	9.0	8.2	2.0	97.1
M649	95.1	1.5	0.6	0.5	97.6
M650	74.1	13.4	8.5	1.5	97.4
M660	95.8	1.8	0.8	0.3	98.7
M661	88.2	6.2	2.5	0.3	97.2
M662	96.1	0.8	0.4	0.2	97.4

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Table 6: Concentration of XDE-742 in the solid and liquid phases at the end of adsorption equilibration period

Application Rate $\mu\text{g/mL}$	On soil ( $\mu\text{g a.i./g}$ )		In solution ( $\mu\text{g a.i./mL}$ )		% adsorbed	
	1	2	1	2	1	2
Soil M641						
0.005	0.0027	0.0022	0.005	0.005	10.2	8.4
0.010	0.0042	0.0051	0.010	0.010	7.9	9.6
0.025	0.0129	0.0098	0.023	0.023	10.3	7.8
0.050	0.0263	0.0204	0.047	0.047	10.1	7.8
0.100	0.0464	0.0445	0.094	0.093	9.3	8.9
Soil M642						
0.005	0.0020	0.0019	0.005	0.005	7.5	7.0
0.010	0.0025	0.0027	0.010	0.010	4.9	5.1
0.025	0.0067	0.0071	0.024	0.024	5.3	5.6
0.050	0.0138	0.0150	0.049	0.050	5.4	5.8
0.100	0.0301	0.0268	0.096	0.096	6.0	5.3
Soil M644						
0.005	0.0014	0.0017	0.005	0.005	5.4	6.6
0.010	0.0028	0.0017	0.010	0.010	5.4	3.3
0.025	0.0039	0.0043	0.025	0.025	3.1	3.5
0.050	0.0306	0.0148	0.046	0.049	11.9	5.8
0.100	0.0165	0.0160	0.098	0.097	3.4	3.2
Soil M645						
0.005	0.0012	0.0004	0.005	0.005	4.4	1.4
0.010	0.0009	0.0005	0.010	0.011	1.7	1.0
0.025	0.0077	0.0014	0.024	0.025	6.2	1.1
0.050	0.0146	0.0054	0.051	0.051	5.7	2.1
0.100	0.0247	0.0145	0.098	0.100	4.9	2.9
Soil M646						
0.005	0.0064	0.0061	0.004	0.004	24.2	23.0
0.010	0.0115	0.0122	0.008	0.008	22.1	23.6
0.025	0.0273	0.0263	0.019	0.019	21.8	21.0
0.050	0.0614	0.0384	0.038	0.043	23.9	15.0
0.100	0.1097	0.0999	0.077	0.079	22.1	19.9
Soil M649						
0.005	0.0016	0.0017	0.005	0.005	6.0	6.2
0.010	0.0028	0.0020	0.010	0.010	5.4	3.8
0.025	0.0074	0.0065	0.024	0.023	5.8	5.1

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0.050	0.0162	0.0222	0.047	0.047	6.2	8.5
0.100	0.0247	0.0191	0.094	0.094	4.9	3.8

Table 6: Concentration of XDE-742 in the solid and liquid phases at the end of adsorption equilibration period (continued)

Application Rate	On soil ( $\mu\text{g a.i./g}$ )		In solution ( $\mu\text{g a.i./mL}$ )		% adsorbed	
	1	2	1	2	1	2
Soil M650						
0.005	0.0074	0.0077	0.004	0.004	28.2	29.1
0.010	0.0146	0.0141	0.007	0.007	28.2	27.2
0.025	0.0294	0.0327	0.019	0.018	23.6	26.2
0.050	0.0698	0.0701	0.036	0.036	27.2	27.3
0.100	0.1292	0.1225	0.073	0.073	25.9	24.5
Soil M660						
0.005	0.0024	0.0019	0.011	0.005	9.0	7.2
0.010	0.0027	0.0036	0.010	0.010	5.1	6.9
0.025	0.0074	0.0076	0.024	0.024	5.8	6.0
0.050	0.0211	0.0261	0.047	0.047	8.1	10.0
0.100	0.0215	0.0228	0.096	0.096	4.2	4.5
Soil M661						
0.005	0.0034	0.0025	0.011	0.005	12.9	9.3
0.010	0.0058	0.0058	0.009	0.009	11.1	11.1
0.025	0.0122	0.0149	0.023	0.022	9.7	11.9
0.050	0.0267	0.0307	0.046	0.045	10.4	11.9
0.100	0.0591	0.0622	0.089	0.089	11.8	12.4
Soil M662						
0.005	0.0014	0.0013	0.012	0.005	5.4	4.7
0.010	0.0017	0.0023	0.010	0.010	3.3	4.4
0.025	0.0043	0.0064	0.025	0.025	3.4	5.1
0.050	0.0129	0.0118	0.050	0.050	5.0	4.6
0.100	0.0197	0.0142	0.099	0.098	3.9	2.8

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**Table 7: Concentration of XDE-742 in the solid and liquid phases at the end of desorption (NOTE: this table has been revised by the PMRA as per clarification from DOW AgroSciences to correct desorption solution concentrations).**

Application Rate	On soil ( $\mu\text{g a.i./g}$ )		In $\text{CaCl}_2$ solution ( $\mu\text{g a.i./mL}$ )		% desorbed as % of the adsorbed	
	1	2	1	2	1	2
Soil M641						
0.005	0.0007	0.0007	0.0002	0.0002	34.5	37.8
0.010	0.0011	0.0010	0.0003	0.0003	37.4	32.6
0.025	0.0024	0.0026	0.0009	0.0009	35.7	44.9
0.050	0.0053	0.0056	0.0018	0.0017	33.6	41.7
0.100	0.0073	0.0095	0.0032	0.0031	34.1	34.4
Soil M642						
0.005	0.0004	0.0003	0.0001	0.0000	17.0	13.7
0.010	0.0007	0.0004	0.0001	0.0001	26.5	25.8
0.025	0.0014	0.0010	0.0004	0.0004	33.7	27.5
0.050	0.0027	0.0030	0.0008	0.0007	31.3	24.1
0.100	0.0054	0.0055	0.0014	0.0014	23.1	26.3
Soil M644						
0.005	0.0003	0.0004	0.0000	0.0000	9.5	13.0
0.010	0.0003	0.0003	0.0001	0.0001	16.3	25.9
0.025	0.0011	0.0010	0.0003	0.0003	41.5	34.6
0.050	0.0021	0.0016	0.0009	0.0007	14.9	23.6
0.100	0.0034	0.0041	0.0012	0.0014	35.7	44.3
Soil M645						
0.005	0.0003	0.0002	0.0000	0.0000	4.5	79.1
0.010	0.0002	0.0003	0.0001	0.0000	35.5	19.3
0.025	0.0010	0.0008	0.0002	0.0002	15.0	70.2
0.050	0.0017	0.0019	0.0004	0.0003	15.1	32.2
0.100	0.0012	0.0030	0.0009	0.0007	18.7	24.9
Soil M646						
0.005	0.0033	0.0030	0.0004	0.0005	35.2	38.5
0.010	0.0056	0.0059	0.0009	0.0008	39.8	34.6
0.025	0.0148	0.0128	0.0021	0.0020	38.4	36.8
0.050	0.0294	0.0151	0.0044	0.0026	37.2	34.5
0.100	0.0509	0.0457	0.0087	0.0079	40.7	40.1
Soil M649						



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Application Rate	On soil ( $\mu\text{g a.i./g}$ )		In $\text{CaCl}_2$ solution ( $\mu\text{g a.i./mL}$ )		% desorbed as % of the adsorbed	
	1	2	1	2	1	2
0.005	0.0003	0.0005	0.0001	0.0001	22.4	26.8
0.010	0.0005	0.0006	0.0001	0.0001	14.5	23.3
0.025	0.0014	0.0014	0.0003	0.0004	20.7	34.4
0.050	0.0030	0.0028	0.0009	0.0009	29.9	21.1
0.100	0.0051	0.0053	0.0014	0.0012	29.7	33.2
Soil M650						
0.005	0.0028	0.0032	0.0007	0.0007	45.6	49.0
0.010	0.0059	0.0058	0.0012	0.0013	43.8	46.4
0.025	0.0110	0.0142	0.0029	0.0033	51.1	51.6
0.050	0.0267	0.0272	0.0070	0.0069	52.3	50.4
0.100	0.0498	0.0491	0.0128	0.0127	51.6	53.4
Soil M660						
0.005	0.0005	0.0004	0.0001	0.0001	22.4	34.3
0.010	0.0007	0.0007	0.0003	0.0002	48.1	32.9
0.025	0.0017	0.0016	0.0007	0.0007	46.0	46.8
0.050	0.0033	0.0033	0.0015	0.0015	35.6	28.7
0.100	0.0054	0.0053	0.0019	0.0020	43.3	43.5
Soil M661						
0.005	0.0011	0.0007	0.0003	0.0002	41.4	45.6
0.010	0.0018	0.0017	0.0006	0.0006	53.2	54.5
0.025	0.0029	0.0043	0.0011	0.0016	42.6	52.4
0.050	0.0071	0.0073	0.0031	0.0034	57.1	55.1
0.100	0.0141	0.0148	0.0063	0.0078	52.5	60.0
Soil M662						
0.005	0.0002	0.0003	0.0000	0.0001	16.2	27.6
0.010	0.0003	0.0003	0.0001	0.0001	23.6	14.4
0.025	0.0009	0.0008	0.0002	0.0002	28.3	16.9
0.050	0.0017	0.0014	0.0006	0.0006	23.6	23.9
0.100	0.0027	0.0029	0.0008	0.0008	20.5	29.1



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Table 8: Study Author-calculated Adsorption and Desorption Constants of XDE-742 in the soils

Soil	pH <sup>a</sup>	Adsorption					Desorption				
		K <sub>F</sub> <sup>b</sup>	1/n	R <sup>2</sup>	K <sub>d</sub>	K <sub>oc</sub>	K <sub>F</sub> <sup>b</sup>	1/n	R <sup>2</sup>	K <sub>d</sub>	K <sub>oc</sub>
M641	6.2	0.48	1.00	0.9904	0.420-0.555	46.69-61.69	1.45	0.89	0.9867	6.20-10.25	689-1139
M642	7.8	0.25	0.95	0.9854	0.252-0.388	10.08-15.54	1.35	0.87	0.9588	10.02-32.53	401-1301
M644	7.7	0.19	0.94	0.8366	0.157-0.671	19.67-83.86	0.35	0.72	0.9173	6.20-46.06	775-5757
M645	7.8	0.33	1.25	0.8195	0.047-0.318	3.62-24.47	0.10	0.56	0.7425	1.32-27.99	102-2153
M646	5.9	1.03	0.93	0.9796	0.889-1.604	32.93-59.42	4.56	0.94	0.9950	7.45-9.78	276-362
M649	7.6	0.27	0.98	0.9488	0.200-0.475	5.25-12.51	1.04	0.82	0.9769	9.96-30.17	262-794
M650	5.4	1.55	0.96	0.9954	1.550-2.095	41.89-56.63	3.03	0.95	0.9975	4.50-6.60	121-178
M660	6.3	0.24	0.90	0.9284	0.223-0.552	22.34-55.19	1.06	0.87	0.9723	5.30-16.91	530-1691
M661	5.7	0.69	1.03	0.9893	0.505-0.740	38.88-56.94	1.00	0.86	0.9909	3.17-6.92	244-533
M662	7.9	0.16	0.93	0.9640	0.145-0.276	5.80-11.04	1.13	0.86	0.9755	12.03-29.09	481-1164

<sup>a</sup> soil pH

<sup>b</sup>  $\mu\text{g}^{1-1/n} \text{mL}^{1/n} \text{g}^{-1}$

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Table 9a: PMRA-calculated adsorption constants of XDE-742 in the soils.

		Adsorption - PMRA Values						
		Freundlich				Non-Freundlich		
Soil	pH <sup>a</sup>	R <sup>2</sup>	1/n	Kf-ads <sup>b</sup>	Kfoc-ads <sup>b</sup>	R <sup>2</sup>	Kd-ads <sup>c</sup>	Koc-ads <sup>c</sup>
M641	6.2	0.990	1.01	0.50	55.4	0.990	0.49	54.3
M642	7.8	0.984	0.94	0.24	9.7	0.993	0.29	11.8
M644	7.7	0.837	0.93	0.18	22.7	0.422	0.22	27.8
M645	7.8	0.809	1.21	0.29	22.6	0.800	0.20	15.0
M646	5.9	0.979	0.93	1.04	38.6	0.957	1.32	48.9
M649	7.6	0.948	0.98	0.27	7.1	0.810	0.27	7.1
M650	5.4	0.995	0.96	1.60	43.3	0.992	1.76	47.7
M660	6.3	0.913	0.97	0.29	28.9	0.684	0.28	28.5
M661	5.7	0.963	1.11	0.88	68.0	0.989	0.67	51.2
M662	7.9	0.931	0.98	0.19	7.4	0.894	0.19	7.5

<sup>a</sup> soil pH

<sup>b</sup>  $\mu\text{g}^{-1-1/n} \text{mL}^{1/n} \text{g}^{-1}$

<sup>c</sup>  $\text{mLg}^{-1}$

Table 9b: PMRA-calculated desorption constants of XDE-742 in the soils.

		Desorption - PMRA Values						
		Freundlich				Non-Freundlich		
Soil	pH <sup>a</sup>	R <sup>2</sup>	1/n	Kf-des <sup>b</sup>	Kfoc-des <sup>b</sup>	R <sup>2</sup>	Kd-des <sup>c</sup>	Koc-des <sup>c</sup>
M641	6.2	0.986	0.50	0.15	17	0.997	0.41	46
M642	7.8	0.998	0.86	0.21	8	0.990	0.28	11
M644	7.7	0.996	0.34	0.04	4	0.998	0.13	16
M645	7.8	0.883	0.47	0.06	4	0.841	0.18	14
M646	5.9	0.987	0.35	0.25	9	0.977	0.81	30
M649	7.6	0.982	0.33	0.05	1	0.947	0.18	5
M650	5.4	0.998	0.54	0.51	14	0.996	1.27	34
M660	6.3	0.999	0.36	0.05	5	0.997	0.18	18
M661	5.7	0.998	0.56	0.24	18	0.998	0.56	43
M662	7.9	0.983	0.37	0.04	2	0.933	0.15	6

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<sup>a</sup> soil pH  
<sup>b</sup>  $\mu\text{g}^{1-1/n} \text{mL}^{1/n} \text{g}^{-1}$   
<sup>c</sup>  $\text{mLg}^{-1}$

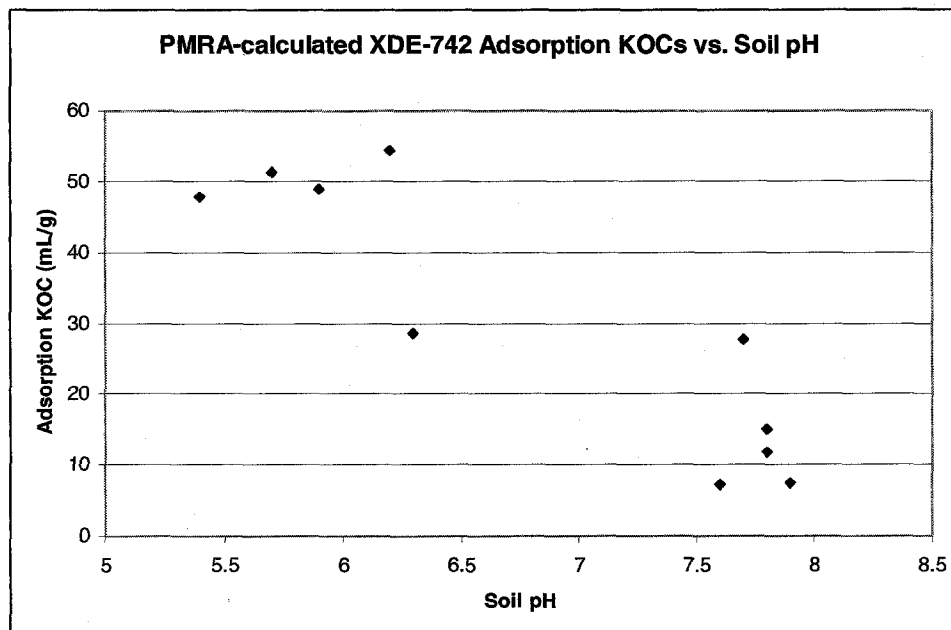


Figure 1. XDE-742 adsorption  $K_{OC}$  values (PMRA-derived) as a function of soil pH.

When adsorption  $K_{oc}$  values are plotted against the pH of the soil (Figure 1), the inverse relationship between  $K_{oc}$  and pH is apparent. Since  $K_{oc}$  is simply  $K_d$ /soil organic carbon content, this shows that pH is a good indicator of XDE-742 adsorption provided that the influence of organic carbon is also considered. In other words, adsorption of XDE-742 is influenced by both pH and soil organic carbon content, as the soil pH decreases the  $K_{oc}$  value increases.

**C. ADSORPTION AND DESORPTION:** An average of 89.7% (71.8%-98.3%) of the applied radioactivity remained in the adsorption solution, while an average of 3.8% (0.2%-14.2%) was recovered in the desorption solution. An average of 2.5% (0.0%-9.6%) of the applied radioactivity was recovered in the extract. The soil pellet contained an average of 0.7% (0.2%-3.6%) of the applied radioactivity.

For the definitive isotherm study,  $K_d$  and  $K_{OC}$  values were re-calculated by the PMRA by combining data from both replicates into a single adsorption isotherm and by using single-point desorption isotherms from the highest test concentration. For the adsorption phase, the average  $K_d$  value for the ten soils was 0.57 mL/g (range 0.19 to 1.76 mL/g); the corresponding average  $K_{OC}$  values were 30.0 mL/g (range 7.1 to 54.3 mL/g). Following a single desorption cycle, the

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average  $K_d$  value for the ten soils was 0.42 mL/g (range 0.13 to 1.27 mL/g); the corresponding average  $K_{OC-des}$  value was 22.3 mL/g (range 5.0 to 46.0 mL/g).

Freundlich adsorption and desorption isotherm plots were also generated by the PMRA reviewer for the definitive study. Freundlich adsorption correlation coefficients ranged from 0.809 to 0.995, and  $1/n$  values from 0.93 to 1.21. Adsorption  $K_F$  values ranged from 0.18 to 1.60  $\mu\text{g}^{1-1/n} \text{mL}^{1/n} \text{g}^{-1}$ , and corresponding  $K_{FOC-ads}$  values ranged from 7.2 to 68.0  $\mu\text{g}^{1-1/n} \text{mL}^{1/n} \text{g}^{-1}$ , respectively. Freundlich desorption correlation coefficients ranged from 0.883 to 0.999, and  $1/n$  values ranged from 0.33 to 0.86. Desorption  $K_F$  values ranged from 0.04 to 0.51  $\mu\text{g}^{1-1/n} \text{mL}^{1/n} \text{g}^{-1}$ , and corresponding  $K_{FOC-des}$  values ranged from 1.0 to 18.0  $\mu\text{g}^{1-1/n} \text{mL}^{1/n} \text{g}^{-1}$ , respectively.

Adsorption of XDE-742 in the range of soils tested is generally linear with respect to concentration (i.e., the majority of the slopes of the Freundlich adsorption coefficients [ $1/n$ ] fall within the range of 0.9 - 1.1). Therefore, adsorption can be described using non-Freundlich  $K_{oc-ads}$  values.

### III. STUDY DEFICIENCIES:

PMRA: This study was conducted with only one desorption cycle. The PMRA requires at least three desorption cycles to fully characterize potential mobility of bound residues. However, as an average of 90% of applied XDE-742 did not bind to the soils, and the compound is considered very highly mobile, the lack of additional desorption cycles does not invalidate the study.

USEPA: The calculated data provided in Appendix C often have too few significant figures. Raw and calculated data should preserve significant figures so that all study calculations can be represented.

### IV. REVIEWERS COMMENTS:

#### EAD comments:

The EAD reviewer-calculated non-Freundlich and Freundlich adsorption coefficients are generally in agreement with those from the study authors, and agrees with the study authors conclusions that XDE-742 is expected to be very highly mobile (average  $K_{OC} = 29.97$  mL/g [range 7.09 to 54.26 mL/g]) according to the classification scheme of McCall et al. (1981). The EAD however, recommends using the reviewer-calculated values as they represent a single isotherm and coefficients based on replicate data, rather than adsorption coefficients from individual data points.

The EAD determined single-point desorption isotherms rather than using the serial desorption isotherms generated by the study authors. An error was found in the raw desorption data reported by the study authors. The study authors confirmed via clarifax that in Appendix C, Table 10 (desorption supernatant concentrations), the authors reported the "Total amount in CaCl<sub>2</sub> desorption" (i.e., column AR) rather than " $\mu\text{g a.i./mL}$  in desorption" (i.e., column AU). The

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revised Table 10 is attached below. The slopes for the XDE-742 desorption isotherms are very similar to the adsorption isotherms, and  $K_{OC-des}$  values are lower than  $K_{OC-ads}$  values indicating that it is easily desorbed after a single cycle.

USEPA: The USEPA recommends using the PMRA-calculated statistics as well.

Australian Reviewer Comments. The single desorption cycle is acceptable given the low soil adsorption of the parent. The study author adsorption coefficients are given as ranges where as it is normal to present these as single values, thus DEW will use the reviewer calculated adsorption coefficients, noting that they are from replicated data points.

### EAD Conclusion:

Based on the adsorption coefficients in the ten soils used in this study (average  $K_{OC-ads} = 29.97$  mL/g [range 7.09 to 54.26 mL/g]), XDE-742 Technical can be considered very highly mobile according to the classification criteria of McCall et al. (1981). Desorption coefficients (average  $K_{OC-des} = 22.3$  mL/g [range 5.0 to 46.0 mL/g]), indicate that XDE-742 does not bind irreversibly with soil, and can readily desorb. This study is scientifically sound and satisfies the DACO requirements for an adsorption/desorption study with the active ingredient (DACO 8.2.4.2).

### V. REFERENCES:

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