GX071
(N-ethyl [1-^{14}C] perfluorooctanesulfonamide)

STUDY TITLE

Adsorption/Desorption Of ^{14}C-GX071

DATA REQUIREMENT

Environmental Fate Data Requirement
40 CFR 158
Subdivision N: Series 163-1

AUTHOR

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STUDY COMPLETED ON

January 16, 1991

PERFORMING LABORATORY

Agrisearch Incorporated
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LABORATORY PROJECT ID

Agrisearch Project No. 2515

SPONSOR

Griffin Corporation
Rocky Ford Road
Valdosta, GA 31601

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA §10 (d) (1) (A), (B), or (C).

Company Griffin Corporation

Company Representative L. Vernon White Date 1/28/91

Director, Regulatory Affairs
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Signature

These data are the property of Griffin Corporation, and as such, are considered to be confidential for all purposes other than compliance with FIFRA §10. Submission of these data in compliance with FIFRA does not constitute a waiver of any right to confidentiality which may exist under any other statute or in any other country.

CERTIFICATION OF GOOD LABORATORY PRACTICE

Project No. 2515 "Adsorption/Desorption Of ^{14}C-GX071", has been performed in compliance with current EPA Good Laboratory Practice Standards (40 CFR: 160) by Agrisearch Incorporated. However, the characterization by A & L Agricultural Laboratories of the soils used in this study was not performed under GLP's. All protocol deviations are detailed in APPENDIX A on page 47.

Submitted By:

William C. Spare, Study Director — Date 1/16/91
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QUALITY ASSURANCE
FINAL REPORT STATEMENT

Project No. 2515
Adsorption/Desorption Of ^{14}C-GX071

This project was inspected/audited by Quality Assurance according to Agrisearch Standard Operating Procedures and EPA's Good Laboratory Practice Standards (Ref. 1) and all findings were reported to the study director and management:

Type Of Inspection	Date Inspected/Audited	Date Reported
Protocol	9/18/89	9/18/89
Raw Data	1/05/90	1/05/90
Test Substance Preparation Test Substance Administration	2/07/90	2/07/90
Test System Sampling	8/08/90	8/08/90
Test System Preparation Test System Administration Test System Sampling	8/24/90	9/04/90
Raw Data	9/13,14/90	9/14/90
Draft Report	11/12/90	11/12/90
Final Report	1/10,11,14,15/91	1/15/91

Action has been taken in response to all items listed by Quality Assurance. It was concluded that the final report accurately reflects the raw data for this project.

Carolyn Ritchey
Carolyn Ritchey
Quality Assurance Officer

Date: 1/16/91

UNALTERED COPY STATEMENT

This report is a complete and unaltered copy of the report provided by the testing facility.

Company Griffin Corporation

Company Representative L. Vernon White Date 1/28/91

Director, Regulatory Affairs
Title

L. Vernon White
Signature

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STUDY IDENTIFICATION AND KEY PERSONNEL

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SPONSOR STUDY MONITOR:	Roger A. Novak, Ph.D.
EXPERIMENTAL START DATE:	January 2, 1990
EXPERIMENTAL TERMINATION DATE:	November 7, 1990
COMPLETION DATE:	January 16, 1991

All original raw data, the protocol, and final report are stored in the archives of Agrisearch Incorporated.

INTRODUCTION

The objective of this study was to predict the potential for movement of ^{14}C-GX071 to and dispersion in aquatic sites. Four soils were tested using batch equilibrium techniques (adsorption/desorption).

This study was performed to fulfill the requirements of the EPA "Pesticide Assessment Guidelines", Subdivision N, Chemistry: Environmental Fate For Leaching And Adsorption/Desorption Studies No. 163-1 (Reference 2).

MATERIALS/METHODS

Soil

Four types of soil (clay, sand, sandy loam, and loam) were used in the study. Soils were obtained by Agrisearch Incorporated. Soils were air-dried and sieved (2 mm mesh) prior to use in the study.

Soils were characterized by A & L Agricultural Laboratories, Incorporated. Results are summarized in TABLE I.

Test Substance

Analytical N-ethyl[1-^{14}C]perfluorooctanesulfonamide (GX071), 4.4 mCi, Lot No. 2271-131 at a specific activity of 14.1 mCi/mM (MW = 527) manufactured by New England Nuclear, Boston, Massachusetts was received from the University of Georgia on 9/14/89. The radioactive chemical was logged into the Agrisearch Incorporated test material receipt log book as number 89-046 and stored frozen at -20°C. Prior to study initiation the radioactive ^{14}C-GX071 was purified at Agrisearch by TLC and determined to be >99% pure by the use of two single dimension solvent systems: chloroform/methanol/formic acid/water 75:15:8:2 (v:v:v:v) and toluene/acetone 75:25 (v:v). The purity of the ^{14}C-GX071 was confirmed by GC (see APPENDIX B - Other Documents). GX071 has very limited solubility in water, less than 1 ppm at 25°C. See FIGURE 1 for nomenclature, structure, and ^{14}C label position of the test substance. See APPENDIX B for copies of other documents.

Test Solutions

The water used in this study was drinking water provided by the City of Frederick, Maryland. The drinking water was filtered, deionized, boiled and distilled. To this purified water 0.01N calcium ion was added (1.76 g calcium acetate, Baker Chemical #1266-1, per liter of water). Finally the water containing 0.01N calcium ion was filter sterilized by passing it through 0.2 micron Gelman membrane filters into autoclaved glassware. In this study reference to "water" refers to this purified, 0.01N calcium ion amended, sterile water.

For the range finding study, a stock solution of the radioactive compound (12.4 mg/ml) was prepared in methanol. A 0.01 ml volume of the radioactive stock was diluted in 200 ml of "water". Final GX071 nominal concentration was 0.6 ug/ml.

For the definitive test, a 0.036 ml volume of the radioactive stock was diluted to 3 ml in methanol. Each test system was established with soil and "water". To the "water" an appropriate volume of diluted ^{14}C-GX071 was added to achieve the following nominal concentrations: 0.3 ug/ml, 0.15 ug/ml, 0.075 ug/ml, 0.030 ug/ml, and 0.015 ug/ml. Additional "water" was used without GX071 addition as the 0.00 ug/ml control concentration.

Test System

The test system for this study was each capped centrifuge tube with soil, 0.01 N calcium ion solution and GX071. All test systems were identified by project number, soil type, replicate, concentration, dosing date, experimental start date, and technician initials. All test systems were shaken in the laboratory at 23 - 25°C.

Range Finding Method

Prior to the definitive testing, each soil was tested to establish the ratio of soil to stock solution and equilibration time for use in the adsorption and desorption phases of the study. Additionally, range finding was used to determine if adsorption to the test container or cap occurred. The range finding test was performed in duplicate using a concentration of 0.7 ug/ml of GX071.

Duplicate 1 g samples of each air dried and sieved soil type were placed in 50 ml Kimax borosilicate glass screw cap (Teflon^{R} lined caps) centrifuge tubes. Twenty ml of GX071 solution at approximately 0.7 ug/ml was added to each tube. All samples were shaken using a reciprocal Eberbach shaker with a 4 cm throw at approximately 175 to 200 rpm. At approximately 2, 4, 8, 24, and 48 hours of shaking, all samples were centrifuged (IEC centrifuge 1500 rpm ~1000 G) for 15 minutes and 100 ul of the supernatant was removed and analyzed by LSC.

An estimation of the Freundlich adsorption constant was obtained using $x/m = K_d\ C_e$ (see calculations). In order to evaluate the four soils equally, only one solution to soil ratio was selected for the definitive study. The soil to ^{14}C-GX071 solution ratio used in the definitive test was selected from the following table based on the estimated K_d.

Estimated K_d	Solution to Soil Ratio
≤10	5 ml:1 g
>10 ≤50	20 ml:1 g
>50 ≤400	100 ml:1 g
>400	1000 ml:1 g

Adsorption - Definitive Test

Based upon the equilibrium shaking time and solution to soil ratios, as determined by the range finding phase, the adsorption phase of the study was performed. Samples of prepared soil (0.25 g each) were placed in 50 ml Kimax glass centrifuge tubes in duplicate for each test solution concentration. Twenty five ml of calcium ion solution was added to each centrifuge tube. An appropriate amount of ^{14}C-GX071 was added to each tube to provide nominal concentrations of 0.015, 0.03, 0.075, 0.15, and 0.3 ug/ml. The soil to solution ratio was 1:100 g/mL. To check for adsorption to the test container, a complete set of "blank" tubes was tested with ^{14}C-GX071 at each dose level but without any soil. Each sample set was shaken for 2 hours using a Eberbach shaker at 175 to 200 rpm. Following centrifugation (~1000 G for 15 minutes), the equilibrium concentrations (C_e) of GX071 were determined in all solutions. Solutions remaining after adsorption were decanted and analyzed by LSC. Soil-less "blank" samples were not decanted. The actual concentration of GX071

adsorbed was determined by combustion of the residual soil (wet) with correction for the amount of radioactivity present in the solution remaining with the soil sample. Each sample set was then reshaken for 15 minutes at 300 to 400 rpm after addition of 25 ml of methanol to each tube. The methanol or methanol-blank samples were quantified by LSC.

Desorption - Definitive Test

Desorption of GX071 was performed using soils remaining from a separate definitive adsorption test at concentrations of 0.07, 0.14, 0.18, 0.22, and 0.70 ug/ml. The adsorption phase for preparing soils for desorption was performed as described above except that 50 ml of solution was used per 0.5 g of soil. Fifty ml of calcium ion solution at 0.01 N without GX071 was added to all tubes. Prior to addition of the calcium ion solution all soil samples (wet) were reweighed to correct for remaining adsorption solution in the final calculations. The entire sample set was shaken for 4 hours using the Eberbach shaker at 175 to 200 rpm. All samples were centrifuged (~1000 G for 15 minutes) and the supernatant analyzed by LSC to determine the equilibrium concentration (C_e). Soil concentration of GX071 was determined by combustion of the residual soil and corrected as noted in the adsorption section.

ANALYTICAL METHOD

Radiocarbon Analysis

All solutions were analyzed by direct LSC in Scint-A (Packard Inst. Co.) scintillation cocktail. Soils were oxidized using R.J. Harvey Instruments Corporation Biological Materials Oxidizer (BMO). BMO generated $^{14}CO_2$ was trapped in National Diagnostics Oxosol-CO_2 trapping fluor.

All quantification was performed using 2 channel counting for 5 minutes with a Beckman LS3801 Liquid Scintillation Spectrophotometer. Obtained counts per minute (cpm) were converted to disintegrations per minute (dpm) using the internal standard feature (H#) of the instrument.

Samples for LSC combustion were analyzed the day of adsorption or desorption. No samples were stored prior to analysis.

Calculations

The Freundlich equation is applicable to the adsorption/desorption of materials at intermediate concentrations and is commonly used to evaluate batch equilibrium data. The equation is an empirical relationship which may be expressed as:

$$x/m = K_d C_e^{1/n} \text{ or } \ln x/m = \ln K_d + 1/n \ln C_e$$

where x/m is the soil equilibrium concentration in ug/g, C_e is the aqueous phase equilibrium concentration in ug/ml, K_d is the Freundlich adsorption constant and n is a constant.

The values of ln C_e versus ln x/m were plotted for adsorption and desorption. The constants K_d and n were determined from the slope (1/n) and intercept (ln K_d) of the resultant straight line by linear regression.

The adsorption constant (K_d) was also expressed in terms of the soil organic carbon content using the following equation:

$$K_{oc} = (K_d \times 100) / \% \text{ O.C.}$$

where K_{oc} is the adsorption coefficient based on soil organic carbon content and % O.C. is the organic carbon content of the soil. The organic carbon content of the soil was calculated by dividing the organic matter content by 1.7 per protocol.

Statistical Methods

Linear regression, means, sums, and logs were used for data reduction. No other statistical methods were used.

RESULTS AND DISCUSSION

The results of the range finding study are presented in TABLES II-IV. Data in TABLE II show that equilibrium in all soil types was reached rapidly. Solution equilibrium was achieved by 2 hours with all subsequent aqueous samples at a plateau. Therefore, the equilibrium shaking time selected for the definitive study was 2 hours. The estimated K_d values from TABLE III indicated a K_d of $>50 \leq 400$ for all soils. A solution to soil ratio of 100 ml solution to 1 g soil (100:1) was used in the definitive study. Radiocarbon balance from the range finding study varied from 34.0% to 95.6% for all four soils (TABLE IV). This range finding study was performed to determine: solution to soil ratios, shaking time, and material balance during equilibration. Radiocarbon balance for the range finding test was determined by summation of total radioactivity in the equilibration solution plus total radioactivity in the combusted soils. Total radioactivity was divided by dose to determine balance. The range finding study did not show good radiocarbon balance. Poor balance was found to be caused by GX071 binding to the glass tubes and Teflon^R cap liners used in the study. Attempts to reduce this binding by silanization with methyltrimethoxysilane or by methylation using diazomethane did not result in lower binding of GX071 to the glass or Teflon^R (TABLE V). Therefore in the definitive test, glassware and caps were not pretreated and all soils were combusted to determine actual concentration.

The adsorption phase of the study was repeated twice. In the first adsorption trial, GX071 concentrations in the soil after adsorption were calculated from the difference in the aqueous phase concentrations before and after adsorption with corrections for glass binding. These soil samples were then used for the desorption phase of the study as reported below. The adsorption phase data are not reported here due to erratic mass balances. The second trial of the adsorption phase utilized direct combustion of the adsorbed soils to determine bound GX071 rather than a calculation by difference. In the second adsorption phase of the study, samples were equilibrated with solution at 0.25 g soil to 25 ml solution. Solutions were counted by LSC after centrifugation (TABLE VI) and soils were combusted for adsorbed GX071 (TABLE VII). After soil combustion, 25 ml of methanol was added to each sample tube. The tubes were shaken and the methanol quantified for radiocarbon. Total balance was the sum of percent dose in the water after adsorption plus the soil combustion and the methanol rinse (TABLE VIII). Total average radiocarbon balance was 83.6 to 95.9% for soils and 100.9% for soil-less blanks.

In the desorption phase of the study, soils from the first adsorption trial were used. These soils had been adsorbed with GX071 in solution at 0.07 to 0.7 ug/ml, centrifuged, decanted, and fresh prepared "water" was added. Following four hours of desorption shaking samples were again centrifuged, "waters" sampled for LSC (TABLE IX), decanted and the remaining soils combusted for remaining GX071. Correction for solution remaining in the soil after adsorption (carryover radioactivity) provided no detectable desorbed radiocarbon (TABLE X). Radiocarbon balance for the desorption samples averaged 87.2 to 107.5% of dose for soils and 80.7% for soil-less blanks.

Parts per million (ppm) values were determined from radioactivity of each sample divided by the specific activity (SA) of ^{14}C-GX071 used in the study (SA=59400 dpm/ug). The obtained values from adsorption were evaluated using the empirical Freundlich isotherm. All desorption tests were inappropriate for evaluation by the Freundlich equation. Logarithmic plots of C_e (equilibrium solution concentration) versus x/m (equilibrium soil concentrations) are presented as straight lines for adsorption in FIGURES 2 and 3. Calculation of the Freundlich equations by regression analysis is presented in TABLE XI for adsorption. Desorption values are presented in TABLE XII and were estimated using the simple ratio of $x/m = K_dCe$ with the assumption that $n = 1$. The adsorption constants (K_d) were found to be between 2118 and 2296 with estimated desorption K_d values from 4445 to 11320.

Following completion of adsorption, aliquots of the highest solution concentrations were analyzed by thin-layer chromatography in two solvent systems. Since no detectable desorption was observed, no TLC of desorption solutions was attempted. Analysis showed GX071 at 97.9 to 99.9% in adsorption solutions (See APPENDIX B - Other Documents).

CONCLUSION

Although binding of GX071 to test vessels did occur and was significant, it did not affect the calculation of K_d (adsorption or desorption) since soil bound GX071 was determined by direct combustion of treated soils and the aqueous phases were counted directly. The desorption data also showed that once GX071 was bound to soil and/or glass, it did not desorb back into solution.

The following K_d values were determined for this study.

SOIL TYPE	ADSORPTION	DESORPTION*
MS-Clay	633	8,155
MD-Sand	118	4,445
MD-Sandy Loam	2296	11,320
CA-Loam	1257	9,775

* Estimated K_d based on n = 1 and x/m = K_dCe using detection limits.

TABLE I: SOIL CHARACTERIZATION. SOILS USED FOR GX071 ADSORPTION AND DESORPTION.

SOURCE	MISSISSIPPI	MARYLAND	MARYLAND	CALIFORNIA
Texture	Clay	Sand	Sandy Loam	Loam
Series	Sharkey	Sassafras	Sequatchie	Hesperia
% Sand	25.2	95.6	63.2	44.0
% Silt	32.8	2.2	20.0	47.0
% Clay	42.0	2.2	16.8	9.0
Organic Matter %	4.8	0.9	1.9	0.8
pH	5.9	6.5	7.5	6.7
Field Capacity %	35.9	3.8	15.8	11.7
Cation Exchange Capacity (meq/100g)	24.3	1.8	6.1	4.3
Bulk Density (g/ml)	1.22	1.65	1.28	1.57

Soil characterization was performed by A & L Agricultural Laboratories, Inc. Bulk density determined at Agrisearch Incorporated, Frederick, Maryland.

TABLE II: AVERAGE CONCENTRATION OF GX071 IN SOIL AND AQUEOUS SOLUTIONS DURING RANGE FINDING AT A WATER TO SOIL RATIO OF 20:1 (mL:g). EXPRESSED AS ppm GX071.

Equilibrium Shaking Time	Rep.	SOIL TYPE: Mississippi Clay	Maryland Sand	Maryland Sandy Loam	California Loam	Blank
Solution (ug/ml)						
2 Hours	1	0.029	0.061	0.042	0.047	0.136
	2	0.030	0.063	0.038	0.055	0.116
	x	0.030	0.062	0.040	0.051	0.126
4 Hours	1	0.031	0.056	0.040	0.049	0.102
	2	0.027	—	0.039	0.057	0.071
	x	0.029	0.056	0.040	0.053	0.087
8 Hours	1	0.028	0.060	0.044	0.051	0.321 *
	2	0.031	0.050	0.041	0.059	0.223 *
	x	0.030	0.055	0.043	0.055	0.272
24 Hours	1	0.026	0.064	0.043	0.053	0.159
	2	0.028	0.052	0.039	0.058	0.113
	x	0.027	0.058	0.041	0.056	0.136
48 Hours	1	0.026	0.059	0.049	0.056	0.233 *
	2	0.026	0.047	0.050	0.057	0.265 *
	x	0.026	0.053	0.050	0.057	0.249
Soil (ug/g)						
48 Hours	1	10.62	5.12	9.61	9.42	
	2	10.66	3.70	11.41	7.99	NA
	x	10.64	4.41	10.51	8.71	

* = Samples not centrifuged.
NA = Not applicable, no soil.
— = Tube broken, sample disturbed.

TABLE III: ESTIMATED K_d VALUES FROM RANGE FINDING ($x/m = K_dC_e$)*.

Equilibrium Shaking Time	SOIL TYPE			
	Mississippi Clay	Maryland Sand	Maryland Sandy Loam	California Loam
2 Hours	355	71	263	171
4 Hours	367	79	263	164
8 Hours	355	80	244	158
24 Hours	394	76	256	156
48 Hours	409	83	210	153

* Assumes 1/n = 1 in the Freundlich adsorption isotherm. Soil concentration (x/m) determined by oxidation of soils at 48 hours.

TABLE IV: RADIOCARBON BALANCE FOR RANGE FINDING. SOILS COMBUSTED AFTER 48 HOURS OF ADSORPTION SHAKING. BALANCE DETERMINED BY SUMMATION OF PERCENT OF DOSE* ON SOIL AND PERCENT OF DOSE IN SOLUTION FOR EACH REPLICATE OF EACH SOIL TYPE

Soil Type	Rep	TOTAL dpm**		PERCENT OF DOSE		
		Soil ($\times 10^5$)	Solution ($\times 10^4$)	Soil	Solution	Total
Mississippi Clay	1	6.72	3.09	80.6	3.7	84.3
	2	6.89	3.11	82.7	3.7	86.4
Maryland Sand	1	3.31	7.02	39.8	8.4	48.2
	2	2.27	5.60	27.3	6.7	34.0
Maryland Sandy Loam	1	6.36	5.76	76.4	6.9	83.3
	2	7.36	5.97	88.4	7.2	95.6
California Loam	1	6.07	6.63	72.8	8.0	80.8
	2	4.90	6.8	58.8	8.2	67.0

* Dose = 8.33×10^5 dpm per replicate

** Determined by direct liquid scintillation counting of solutions and by oxidation of soils

TABLE V: RADIOCARBON BALANCE FOR RANGE FINDING USING TREATED GLASSWARE. SOILS COMBUSTED AFTER 6 HOURS OF ADSORPTION SHAKING. BALANCE DETERMINED BY SUMMATION OF PERCENT OF DOSE ON SOIL AND PERCENT OF DOSE IN SOLUTION FOR EACH REPLICATE OF EACH SOIL TYPE

SOIL TYPE	REP	TOTAL DPM		PERCENT OF DOSE		
		SOLUTION ($x10^4$)	SOIL ($x10^5$)	SOLUTION	SOIL	TOTAL
Methylated Glassware (Dose = 2.01 x 10^5 dpm per replicate)						
Mississippi Clay	1	1.14	1.47	5.7	73.3	79.0
	2	1.21	1.22	6.0	60.8	66.8
Maryland Sand	1	3.02	1.11	15.0	55.2	70.3
	2	2.71	1.29	13.5	64.2	77.7
Maryland Sandy Loam	1	1.76	1.38	8.8	68.8	77.6
	2	1.81	1.25	9.0	62.3	71.3
California Loam	1	2.16	1.38	10.8	68.6	79.4
	2	2.24	1.32	11.2	65.2	76.4
Silanized Glassware (Dose = 4.46 x 10^5 dpm per replicate)						
Mississippi Clay	1	2.28	3.62	5.1	81.0	86.1
	2	2.31	3.42	5.2	76.7	81.9
Maryland Sand	1	6.01	4.50	13.5	101.0	114.5
	2	5.06	2.34	11.3	52.5	63.8
Maryland Sandy Loam	1	3.65	3.17	8.2	71.0	79.2
	2	3.93	2.69	8.8	60.3	69.1
California Loam	1	4.46	3.28	10.0	73.5	83.5
	2	4.64	2.86	10.4	64.1	74.5

TABLE VI: MEAN MEASURED CONCENTRATION (C_e) OF GX071 IN SOLUTION AS ug/ml (ppm) AND AVERAGE FOR THE DUPLICATE SAMPLES AT EACH CONCENTRATION IN THE ADSORPTION PHASE OF THE STUDY

SECOND ADSORPTION TEST SOL. CONC.	Rep	SOIL TYPE Mississippi Clay	Maryland Sand	Maryland Sandy Loam	California Loam
0 ug/ml		0.0	0.0	0.0	0.0
0.01	1	0.002	0.004	0.002	0.003
	2	0.002	0.003	0.002	0.004
	$\bar{x}$	0.002	0.004	0.002	0.004
0.03	1	0.003	0.006	0.004	0.005
	2	0.003	0.005	0.004	0.004
	$\bar{x}$	0.003	0.006	0.004	0.005
0.06	1	0.006	0.012	0.008	0.011
	2	0.009	0.015	0.007	0.014
	$\bar{x}$	0.008	0.014	0.008	0.013
0.13	1	0.012	0.017	0.013	0.017
	2	0.012	0.024	0.012	0.019
	$\bar{x}$	0.012	0.021	0.013	0.018
0.26	1	0.018	0.044	0.019	0.022
	2	0.019	0.077	0.019	0.029
	$\bar{x}$	0.019	0.061	0.019	0.026

TABLE VII: MEAN MEASURED CONCENTRATION (x/m) OF GX071 IN SOIL AS ug/g (ppm) AND AVERAGE FOR THE DUPLICATE SAMPLES AT EACH CONCENTRATION IN THE ADSORPTION PHASE OF THE STUDY. CONCENTRATION DETERMINED BY COMBUSTION.

SECOND ADSORPTION TEST SOL. CONC.	Rep	SOIL TYPE: Mississippi Clay	Maryland Sand	Maryland Sandy Loam	California Loam
0 ug/ml		0.0	0.0	0.0	0.0
0.01	1	0.221	0.137	0.246	0.194
	2	0.213	0.111	0.190	0.234
	x	0.217	0.124	0.218	0.214
0.03	1	0.312	0.217	0.569	0.449
	2	0.656	0.229	0.504	0.406
	x	0.484	0.223	0.537	0.428
0.06	1	1.204	0.249	1.474	1.349
	2	1.298	0.991	1.274	1.627
	x	1.251	0.620	1.374	1.488
0.13	1	1.638	2.122	3.350	3.131
	2	2.105	1.978	3.129	2.873
	x	1.872	2.050	3.240	3.002
0.26	1	4.935	2.701	5.845	6.340
	2	3.959	3.536	5.899	5.450
	x	4.447	3.119	5.872	5.895

TABLE VIII: RADIOCARBON BALANCE FOR ADSORPTION OF GX071 IN SOIL Kd DETERMINATION

Blank Soil-less Samples

SAMPLE ID NOMINAL PPM	REP	PERCENT OF DOSE —ADSORPTION— WATER	SOIL	METHANOL RINSE	TOTAL
0.01	1	29.4	NA	75.3	104.7
	2	27.5	NA	79.4	106.9
0.03	1	18.9	NA	77.5	96.4
	2	37.2	NA	70.6	107.8
0.06	1	40.6	NA	52.2	92.8
	2	30.7	NA	72.3	103.0
0.13	1	19.5	NA	77.4	96.9
	2	35.3	NA	66.6	101.9
0.26	1	23.2	NA	74.2	97.4
	2	34.6	NA	66.2	100.8
			Avg ± 1 S.D. = 100.9 ± 4.9		

NA - no soil used

California Loam

SAMPLE ID NOMINAL PPM	REP	PERCENT OF DOSE —ADSORPTION— WATER	SOIL	METHANOL RINSE	TOTAL
0.01	1	21.4	28.2	41.8	91.4
	2	28.0	32.5	38.4	98.9
0.03	1	18.4	36.0	31.8	86.3
	2	17.2	31.1	33.2	81.5
0.06	1	16.5	38.7	43.5	98.7
	2	32.3	48.1	39.0	109.4
0.13	1	13.3	45.4	41.7	100.4
	2	14.8	38.0	46.5	99.4
0.26	1	8.4	46.2	42.4	97.0
	2	11.4	43.1	41.9	96.3
			Avg ± 1 S.D. = 95.9 ± 7.8		

Mississippi Clay

SAMPLE ID NOMINAL PPM	REP	PERCENT OF DOSE —ADSORPTION— WATER	SOIL	METHANOL RINSE	TOTAL
0.01	1	15.0	26.7	47.2	88.9
	2	14.9	26.7	34.0	75.6
0.03	1	12.9	19.6	50.6	83.0
	2	13.5	47.8	46.0	107.3
0.06	1	9.7	35.4	47.2	92.3
	2	14.1	34.7	52.9	101.6
0.13	1	9.1	26.7	46.1	81.8
	2	9.4	34.0	46.9	90.3
0.26	1	7.0	37.8	46.6	91.4
	2	7.3	23.9	56.0	87.2
			Avg ± 1 S.D. = 90.0 ± 9.3		

TABLE VIII: RADIOCARBON BALANCE FOR ADSORPTION OF GX071 IN SOIL (Cont'd) Kd DETERMINATION

Maryland Sand

SAMPLE ID NOMINAL PPM	REP	PERCENT OF DOSE ADSORPTION WATER	ADSORPTION SOIL	METHANOL RINSE	TOTAL
0.01	1	31.1	16.5	43.5	91.2
	2	25.9	15.6	36.7	78.2
0.03	1	22.5	14.7	40.1	77.3
	2	19.4	22.5	41.2	83.0
0.06	1	19.4	8.2	54.8	82.4
	2	23.0	21.6	36.4	81.0
0.13	1	13.0	38.5	34.8	86.2
	2	18.9	31.7	37.4	87.9
0.26	1	17.3	22.8	37.5	77.5
	2	29.8	29.7	32.3	91.8
			Avg ± 1 S.D. = 82.6 ± 5.4		

Maryland Sandy Loam

SAMPLE ID NOMINAL PPM	REP	PERCENT OF DOSE ADSORPTION WATER	ADSORPTION SOIL	METHANOL RINSE	TOTAL
0.01	1	16.4	34.0	41.0	91.4
	2	16.9	30.9	38.7	86.5
0.03	1	14.0	44.0	36.4	84.4
	2	15.4	37.8	32.9	86.1
0.06	1	12.6	48.0	39.4	100.0
	2	11.6	33.2	51.5	96.3
0.13	1	9.7	57.4	36.8	103.9
	2	9.0	40.2	53.5	102.8
0.26	1	7.5	49.7	34.6	91.8
	2	7.4	55.1	29.1	91.6
			Avg ± 1 S.D. = 94.5 ± 6.3		

TABLE IX: MEAN MEASURED CONCENTRATION (C_e) OF GX071 IN SOLUTION AS ug/ml (ppm) AND AVERAGE FOR THE DUPLICATE SAMPLES AT EACH CONCENTRATION IN THE DESORPTION PHASE OF THE STUDY.

FIRST ADSORPTION TEST SOL. CONC.	Rep	SOIL TYPE Mississippi Clay	Maryland Sand	Maryland Sandy Loam	California Loam
0 ug/ml		0.0	0.0	0.0	0.0
0.07	1	<0.0002	0.002	<0.0002	<0.0002
	2	<0.0002	<0.0002	<0.0002	<0.0002
0.14	1	<0.0002	<0.0002	<0.0002	<0.0002
	2	<0.0002	<0.0002	<0.0002	<0.0002
0.18	1	<0.0002	<0.0002	<0.0002	<0.0002
	2	<0.0002	<0.0002	<0.0002	<0.0002
0.22	1	<0.0002	<0.0002	<0.0002	<0.0002
	2	<0.0002	<0.0002	<0.0002	<0.0002
0.70	1	<0.0002	<0.0002	<0.0002	<0.0002
	2	<0.0002	<0.0002	<0.0002	<0.0002

Detection Limit = 0.0002 ppm

TABLE X: MEAN MEASURED CONCENTRATION (x/m) OF GX071 IN SOIL AS ug/g (ppm) AND AVERAGE FOR THE DUPLICATE SAMPLES AT EACH CONCENTRATION IN THE DESORPTION PHASE OF THE STUDY. CONCENTRATION DETERMINED BY COMBUSTION.

FIRST ADSORPTION TEST SOL. CONC.	Rep	SOIL TYPE: Mississippi Clay	Maryland Sand	Maryland Sandy Loam	California Loam
0 ug/ml		0.0	0.0	0.0	0.0
0.07	1	1.571	1.234	2.707	2.352
	2	1.691	0.544	1.821	1.557
	$\bar{x}$	1.631	0.889	2.264	1.955
0.14	1	5.199	1.703	6.660	5.227
	2	3.814	1.566	5.096	4.228
	$\bar{x}$	4.506	1.635	5.878	4.728
0.18	1	7.451	1.979	7.173	7.488
	2	5.470	2.185	--	5.961
	$\bar{x}$	6.461	2.082	7.173	6.725
0.22	1	8.100	2.450	7.129	8.049
	2	6.787	2.165	5.753	6.343
	$\bar{x}$	7.444	2.308	6.441	7.196
0.70	1	57.618	12.071	29.956	41.328
	2	68.221	19.130	55.028	58.540
	$\bar{x}$	62.920	15.601	42.492	49.934

-- Sample broken in lab accident.

TABLE XI: LINEAR REGRESSION ANALYSIS OF THE ADSORPTION DATA USING THE FREUNDLICH ISOTHERM ($\ln x/m = \ln K_d + 1/n \ln C_e$) FOR GX071.

SECOND ADSORPTION TEST SOL. CONC. (ug/ml)	Rep	SOIL TYPE: Mississippi Clay $\ln C_e$	Mississippi Clay $\ln x/m$	Maryland Sand $\ln C_e$	Maryland Sand $\ln x/m$	Maryland Sandy Loam $\ln C_e$	Maryland Sandy Loam $\ln x/m$	California Loam $\ln C_e$	California Loam $\ln x/m$
0.01	1	-6.247	-1.511	-5.521	-1.988	-6.160	-1.401	-5.894	-1.638
	2	-6.254	-1.546	-5.704	-2.198	-6.132	-1.659	-6.626	-1.454
0.03	1	-5.710	-1.165	-5.152	-1.526	-5.628	-0.568	-5.352	-0.801
	2	-5.661	-0.422	-5.301	-1.475	-5.530	-0.885	-5.420	-0.901
0.06	1	-5.077	0.185	-4.385	-1.392	-4.817	0.388	-4.546	0.299
	2	-4.701	0.260	-4.215	-0.009	-4.896	0.242	-4.246	0.486
0.13	1	-4.447	0.493	-4.093	0.752	-4.379	1.209	-4.067	1.141
	2	-4.420	0.744	-3.718	0.682	-4.457	1.141	-3.958	1.055
0.26	1	-4.011	1.596	-3.113	0.993	-3.947	1.766	-3.835	1.847
	2	-3.980	1.376	-2.568	1.263	-3.957	1.775	-3.529	1.696
LINEAR REGRESSION ANALYSIS									
Correlation		0.980		0.935		0.996		0.987	
Slope (1/n)		1.277		1.203		1.506		1.498	
Intercept		6.451		4.775		7.739		7.137	
n		0.783		0.831		0.664		0.667	
K_d		633		118		2296		1257	
K_{oc}		22,436		22,383		205,466		267,130	
% O.C.		2.8		0.53		1.1		0.47	

% O.C. = % Organic Carbon = % Organic Matter / 1.7

$K_{oc} = (K_d \times 100) / \%$ O.C.

Note: All natural log values were determined by computer spreadsheet and not the rounded numbers presented in TABLES VI and VII.

TABLE XII: LINEAR REGRESSION ANALYSIS OF THE DESORPTION DATA USING THE FREUNDLICH ISOTHERM ($\ln x/m = \ln K_d + 1/n \ln C_e$) FOR GX071.

FIRST ADSORPTION TEST SOL. CONC. (ug/ml)	Rep	SOIL TYPE: Mississippi Clay C_e	Mississippi Clay x/m	Maryland Sand C_e	Maryland Sand x/m	Maryland Sandy Loam C_e	Maryland Sandy Loam x/m	California Loam C_e	California Loam x/m
0.07	1	<0.0002	1.571	0.002	1.234	<0.0002	2.707	<0.0002	2.352
	2	<0.0002	1.691	<0.0002	0.544	<0.0002	1.821	<0.0002	1.557
0.14	1	<0.0002	5.197	<0.0002	1.703	<0.0002	6.560	<0.0002	5.227
	2	<0.0002	3.814	<0.0002	1.566	<0.0002	5.896	<0.0002	4.228
0.18	1	<0.0002	7.451	<0.0002	1.979	<0.0002	7.173	<0.0002	7.488
	2	<0.0002	5.470	<0.0002	2.185	<0.0002	—	<0.0002	5.961
0.22	1	<0.0002	8.100	<0.0002	2.450	<0.0002	7.129	<0.0002	8.049
	2	<0.0002	6.787	<0.0002	2.165	<0.0002	5.753	<0.0002	6.343
0.70	1	<0.0002	57.618	<0.0002	12.071	<0.0002	29.956	<0.0002	41.328
	2	<0.0002	68.221	<0.0002	19.130	<0.0002	55.028	<0.0002	58.540
LINEAR REGRESSION ANALYSIS									
Correlation		*		*		*		*	
Slope (1/n)		*		*		*		*	
Intercept		*		*		*		*	
n		1		1		1		1	
K_d		8,155		4,445		11,320		9,775	
K_{oc}		291,250		838,700		1,029,000		2,080,000	
% O.C.		2.8		0.53		1.1		0.47	

% O.C. = % Organic Carbon = % Organic Matter / 1.7

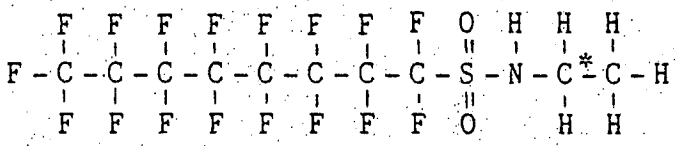
$K_{oc} = (K_d \times 100) / \%$ O.C.

Note: All natural log values were determined by computer spreadsheet and not the rounded numbers presented in TABLES VIII and IX.

* No calculation possible, estimated K_d based on n = 1, $x/m = K_d Ce$ using detection limit Ce = 0.0002, x/m = lowest average value for each soil from TABLE X.

— Sample lost, lab accident.

FIGURE 1: STRUCTURE AND NOMENCLATURE FOR GX-071



N-Ethyl perfluorooctanesulfonamide
* = ^{14}C

FIGURE 2: Freundlich Adsorption Isotherm For Maryland Sandy Loam And Maryland Sand

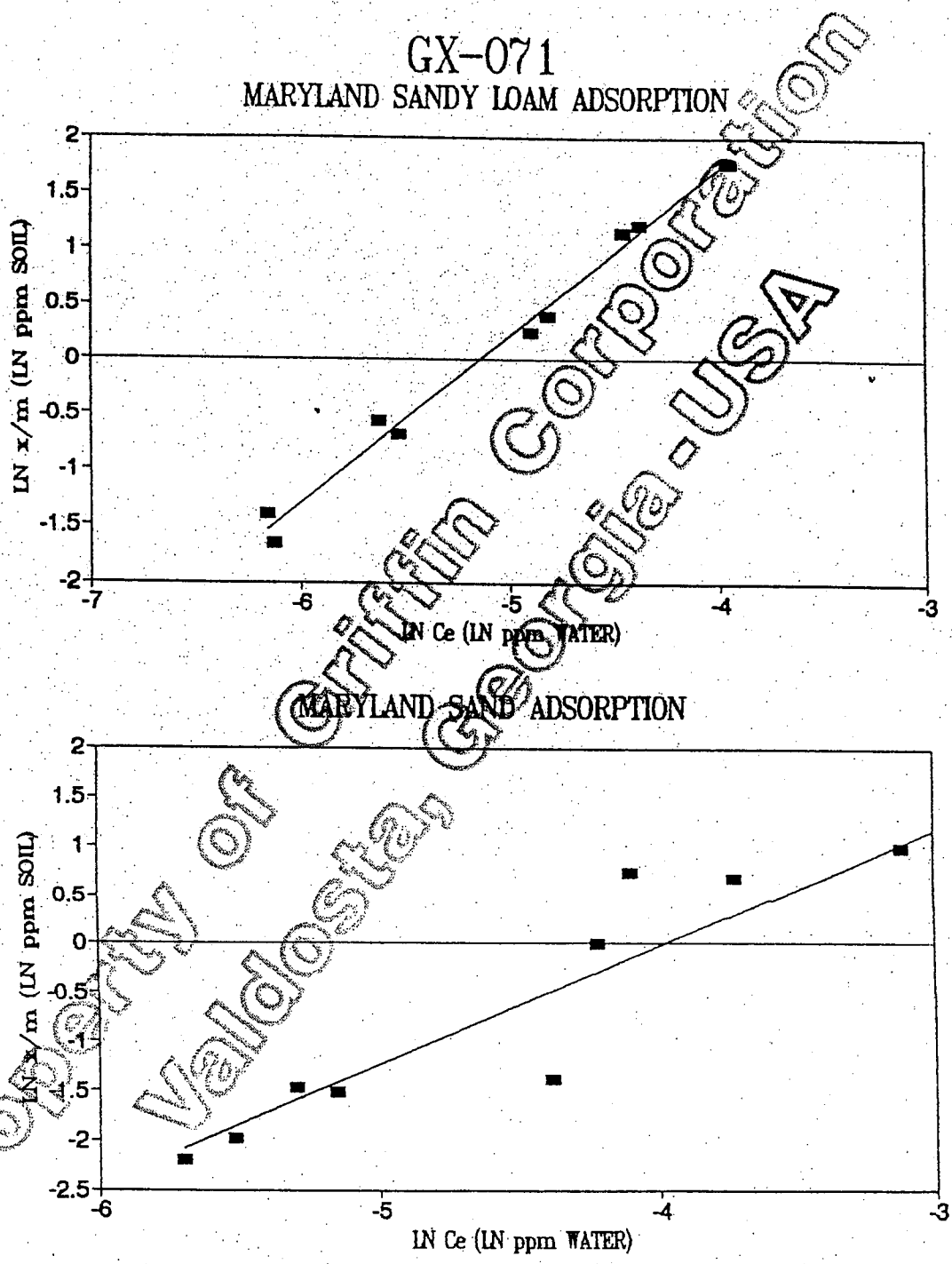
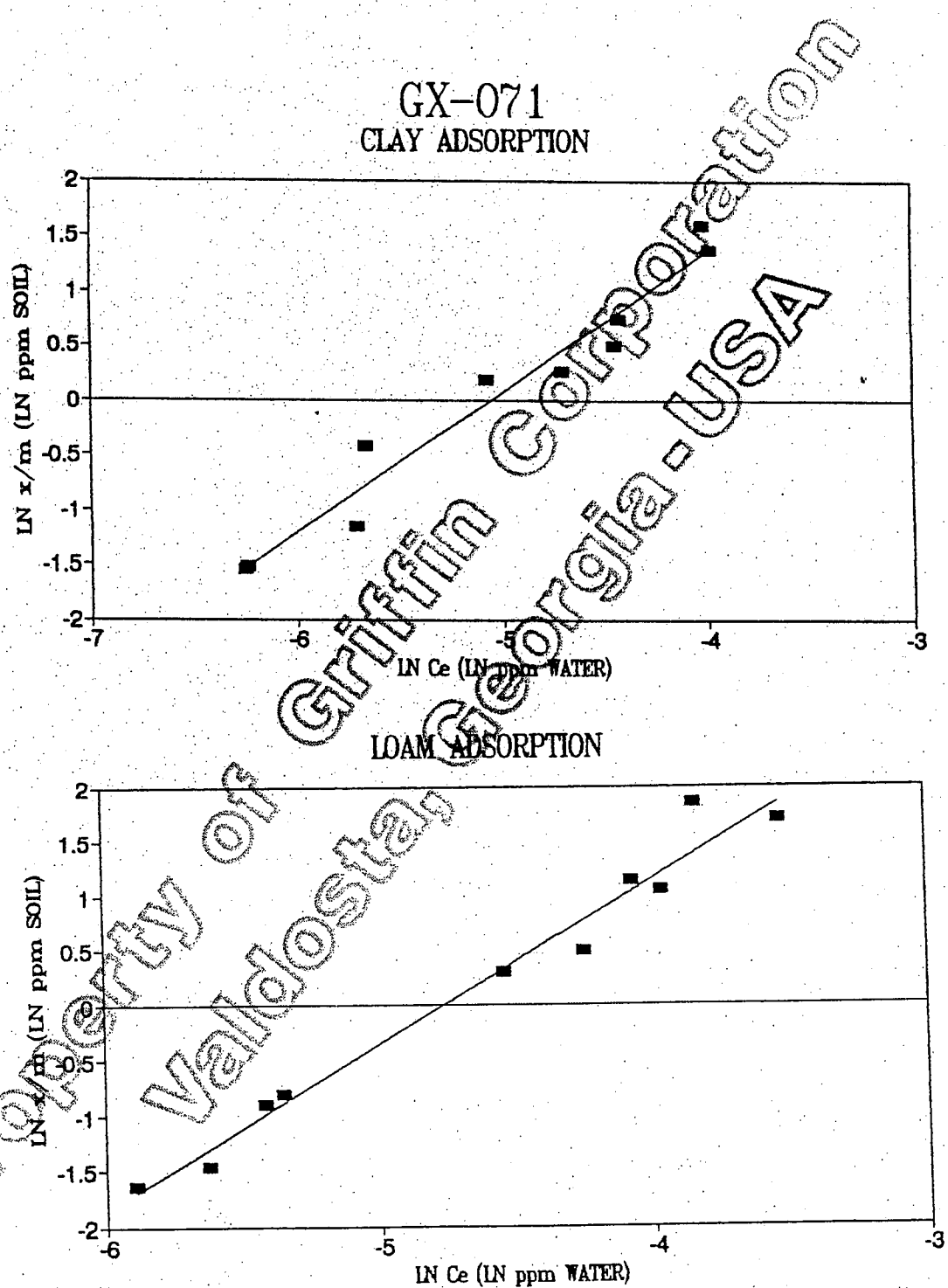


FIGURE 3: Freundlich Adsorption Isotherm For Mississippi Clay And California Loam



REFERENCES

(1) U.S. Environmental Protection Agency, August 17, 1989. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 54, No. 158: 34052-34074.

(2) "Pesticide Assessment Guidelines, Subdivision N Chemistry: Environmental Fate", U.S. Environmental Protection Agency, Office Of Pesticide And Toxic Substances, Washington, D.C., 20460: EPA 540/9-82-021, October 18, 1982. NTIS PB83-153973.

APPENDIX A

Protocol And Deviations

PROTOCOL APPROVAL

PROTOCOL: Adsorption/Desorption of ^{14}C-GX071

AGRISEARCH PROJECT NO: 2515

TEST MATERIAL: GX071 = n-ethylperfluro-octane-sulfonamide

EPA DATA REQUIREMENT: 40 CFR 158 Subdivision N: 163-1

PROPOSED START DATE: September 1989

PROPOSED TERMINATION DATE: November 1989

PROTOCOL ACCEPTANCE:

Sponsor:

Griffin Corporation
Rocky Ford Road
Valdosta, GA 31601

Roger A Novak 09.14.89

Roger Novak, Ph.D., Sponsor Study Monitor — Date
NPC, Incorporated
22636 Glen Drive, Suite 304
Sterling, VA 22170

Testing Facility:

William C Spare 9/14/89

William C. Spare, Study Director — Date
Agrisearch Incorporated
26 Water Street
Frederick, MD 21701

*** AGRISEARCH PROTOCOL ***

ADSORPTION/DESORPTION OF ^{14}C-GX071

OBJECTIVE

The objective of this study is to predict the potential for movement of ^{14}C-GX071 to and dispersion in aquatic sites. Four soil types are tested using batch equilibrium techniques (adsorption/desorption).

JUSTIFICATION FOR SELECTION OF TEST SYSTEM

Pesticide residues may move through the soil profile or be transported to and disperse in aquatic environments due to movement by leaching. This study is designed to predict the potential for leaching and movement of pesticides in the soil column and aquatic systems by the use of batch equilibrium technique with four soil types.

SUMMARY

Analytical grade ^{14}C-GX071 in solution is mixed with soil and allowed to equilibrate. The phases are separated by centrifugation and the solution concentration is determined by liquid scintillation counting (LSC). A portion of the soils (or sediment) is oxidized to determine adsorbed concentration. The soils (or sediment) are next desorbed with solution and desorbed concentrations are determined by LSC following centrifugation. Adsorption and desorption isotherms are graphically presented. Soil characteristics are reported. Data are evaluated using the Freundlich equation and n, K_d, and K_{oc} values are reported.

EXPERIMENTAL DESIGN

Soils

Four soil types: agricultural sand, sandy loam, silt loam, clay loam, or other soils representative of the proposed use area soils are used for this study. Soils are characterized for the following: source, texture (% sand, % silt, % clay), % organic matter, pH, 1/3 bar moisture (field capacity), cation exchange capacity and bulk density. All soils will have a pH within the range of 4 - 8. At least one of the soils will have an organic matter content less than or equal to one percent (sand or sandy loam).

All soils are dug to a depth not exceeding 15 cm. Each soil is mixed in a tray and sieved retaining the fraction less than 2 mm. Each sieved soil is finally air dried.

Test Material

The test material for this study is ^{14}C-GX071 at a specific activity of approximately 20 uCi/mg. The radioactive material is supplied by Griffin Corporation. See FIGURE 1 for structure of ^{14}C-GX071.

Stock solutions of ^{14}C-GX071 are prepared in 0.01N calcium ion solution using sterile distilled water. Solutions are prepared at 10, 5, 1, 0.5, 0.2 and 0.00 ppm and stored appropriately. All stock solution concentrations are confirmed by liquid scintillation counting (LSC). Due to the low water solubility of GX071, a saturated solution will be prepared as the highest test concentration. Serial dilution of this solution shall be used to provide four concentrations plus a blank for testing.

The purity of the test material will be confirmed prior to the start of the study. Additionally, the solution remaining after adsorption as well as after desorption (if possible) and demonstrating the highest concentration of test material will be tested for purity by thin-layer chromatography (TABLE 1).

Test System

The test system for this study is each capped container of soil, solution, and ^{14}C-GX071. All test systems are identified by project number, soil type, replicate, concentration, date, and technician initials.

Range Finding

Prior to definitive testing, each soil is tested to establish the ratio of soil to stock solution for use in the adsorption and desorption phases of the study. The range finding test is performed in duplicate with the highest concentration of test material to be tested.

Duplicate 1 g sample of dry soil are placed in 50 ml glass screw cap centrifuge tubes. Twenty ml of the test material stock solution is transferred to each tube and capped. The samples are shaken, centrifuged at greater than 1000 G for 15 minutes to pellet the soil, and sampled. A 100 ul sample of the supernatant is removed and counted by LSC after approximately 2, 4, 8, and 24 hours of shaking at the same temperature as the aerobic soil metabolism study. If equilibrium has not been obtained by 24 hours, shaking is continued for 24 hour periods until equilibrium is reached. The adsorbed concentration is determined at the final equilibrium analysis. If material balance data demonstrates a greater than 90% recovery in the range finding, then no combustion of pelleted soil is performed in the definitive study. Determination of soil concentrations will be made by difference.

Based upon the estimated K_d as determined from $x/m = K_d C_{eq}$ (see CALCULATIONS), the following ratios of test material solution to soil are used for the adsorption and desorption.

Estimated K_d	Solution: Soil Ratio
$\leq$ 10	5 mls: 1 g
> 10 $\leq$ 50	20 mls: 1 g
> 50 $\leq$ 400	100 mls: 1 g
> 400	1000 mls: 1 g

Adsorption

Based upon the equilibrium shaking time and solution to soil ratios as determined by range finding, the adsorption phase of the study is performed. Duplicate samples of dry soil are placed in appropriate containers. Each test material stock solution is transferred to appropriate containers and capped. The entire sample set is shaken for the time determined by range finding and at the same temperature. The equilibrium concentrations are determined in all solutions and soils after centrifugation.

Desorption

Following adsorption, desorption is performed on the soil samples remaining from adsorption. Calcium ion solution at 0.01N without test material is added to the soil remaining after adsorption in the same ratio as used for adsorption. The entire sample set is shaken until equilibrium is reached (same time as adsorption study). Supernatant is decanted and analyzed by LSC. Concentration of test material in soils is determined.

RADIOCARBON ANALYSIS

All solutions are analyzed by direct LSC in appropriate scintillation fluor. Soils are oxidized using an R.J. Harvey Instruments Corporation Biological Materials Oxidizer (BMO). BMO generated $^{14}CO_2$ is trapped in appropriate scintillation fluor and quantified by LSC.

All quantification is performed using 2 channel counting for five minutes with a Beckman Liquid Scintillation Spectrophotometer Model LS3801. Obtained counts per minute (cpm) are converted to disintegrations per minute (dpm) using the internal standard ratio (H#) feature of the instrument.

CALCULATIONS

The Freundlich equation is applicable to the adsorption/desorption of materials at intermediate concentrations and is commonly used to evaluate batch equilibrium data. The equation is an empirical relationship which may be expressed as:

$$C_{ads} = KC_{eq}^{1/n}$$

where C_{ads} is the adsorbed concentration (ug/g), C_{eq} is the solution equilibrium concentration (ug/ml) and K and n are constants. Another relationship which assumes that n equals one is the simple proportionality:

$$x/m = K_d C_{eq}$$

where x/m is the adsorbed concentration (ug/g), C_{eq} is the solution equilibrium concentration (ug/ml) and K_d is the distribution coefficient.

Both the adsorption and desorption phases are subjected to evaluation using the Freundlich equation. The adsorption coefficient K_d and the constant 1/n are calculated for each soil type. Additionally, K_{oc} (adsorption coefficient based on organic carbon) is determined from the following equation:

$$\% \text{ O.C.} = \% \text{ Organic Carbon} = \% \text{ Organic Matter} / 1.7$$

$$K_{oc} = (K_d \times 100) / \% \text{ O.C.}$$

STATISTICAL METHODS

No statistical methods will be used. Mathematical methods of linear regression, means, sums, and logs will be used for data reduction.

REPORT

A full report will be issued in draft form after completion of the study. The report will meet the reporting requirements under Subdivision N (160-5 and 163-1), EPA SEPs, and will include, but not be limited to:

(1) description of test material

(2) soil characterization

(3) description of all procedures and analytical methods

(4) concentration of radioactivity (ug/g soil or ug/ml solution) in all soils and equilibrium solutions

(5) examples of calculations for 1/n and K_d

(6) radiocarbon material balance

(7) tabulated values of K_d, 1/n, and K_{oc}

(8) graphic presentation of adsorption and desorption isotherms

Subsequently, a copy of the final report will be made available after completion of sponsor review of the draft.

Records To Be Maintained

All original raw data, documentation, records, and the original approved protocol and final report will be maintained in the archives of Agrisearch Incorporated, 26 Water Street, Frederick, Maryland 21701.

QUALITY ASSURANCE

Agrisearch Incorporated has established a Quality Assurance Unit (QAU) as a commitment to performing laboratory studies in compliance with current Good Laboratory Practice (GLP) Standards as established by the Environmental Protection Agency. The independent QAU monitors each study in progress to assure conformance with applicable regulations.

All final reports issued by Agrisearch Incorporated are signed by the Quality Assurance Officer. This signature on the final report documents study examination by the QAU. Additionally, each study may be subject to one or more unannounced in progress inspections. In progress inspections ensure that the study methods conform to GLP's and the protocol.

It is agreed that this is the official protocol for the conduct of this study. Any amendments will contain the reason for the amendment and be signed and dated by the study director.

REFERENCES

1) "Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate", U.S. Environmental Protection Agency, Office of Pesticide and Toxic Substances, Washington, D.C., 20460; EPA 540/9-82-021, October 18, 1982. NTIS PB83-153973.

2) U.S. Environmental Protection Agency. 1983. Pesticide Programs; Good Laboratory Practice Standards; Final Rule (40 CFR, Part 160). Federal Register, Vol. 48, No. 230: 53946-53969.

TABLE 1: THIN-LAYER CHROMATOGRAPHY (TLC)

Silica Gel Plates:

Precoated glass backed silica gel 60 F254 (EM Merck) layer thickness of 0.25 mm

Solvent Systems:

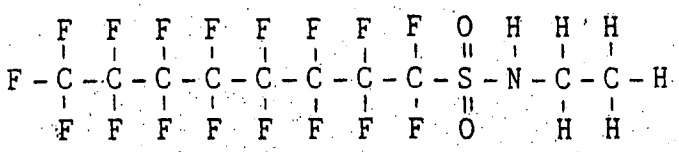
(1)

To be provided by Sponsor

(2)

Visualization: UV - Light at 254 nm

FIGURE 1: STRUCTURE AND NOMENCLATURE FOR GX-071



N-Ethyl perfluorooctanesulfonamide

Protocol Deviations
Project No. 2515
Adsorption/Desorption Of ^{14}C-GX071

The method of dosing test systems specified in the protocol was found to be unsatisfactory for GX071. For this study definitive stock solutions were not prepared in calcium ion solution but test solutions were directly dosed with ^{14}C-GX071 stock. This change was made due to the plating problems associated with GX071. The protocol stated that all samples would be centrifuged. Blank samples were not centrifuged in the definitive test since no soil particles were present to be centrifuged out. None of these deviations were expected to adversely impact the performance or results of the study.

William C. Spare

William C. Spare, Study Director
Agrisearch, Inc.
26 Water Street
Frederick, MD 21701

1/15/91
Date

APPENDIX B

Other Documents

Agricultural Chemicals Group

Confidential

GX-071

Experimental Product Information Sheet

Physical and Chemical Properties:

Molecular Weight		527
Chemical Formula		$C_8F_{17}SO_2N(H)CH_2CH_3$
Color		White
Odor		None
Physical State		Solid
Melting Point	Meniscus Point	95.5°C.
	Liquefaction Point	96.5°C.
	Melting Range	93.0 - 96.5°C
Boiling Point @ 2mm Hg		≈ 110°C.
Bulk Density @ 25°C.		0.1485 g/mL
Solubility @ 25°C.	Water	Nil
	Hexane	1.4%
	Methylene Chloride	1.86%
	1-Octanol	7.09%
	Methanol	83.3%
Vapor Pressure @ 25°C.		4.3 x 10E -7
Dissociation Constant (K_A)		3.16 x 10E -10
Seta Flash cc		> 200° F.

Stability: GX-071 is thermally stable and resistant to hydrolysis.

Mode of Action: Preliminary data indicate that GX-071 is an uncoupler of oxidative phosphorylation in isolated mitochondria.

Intended Use: A low rate of GX-071 is incorporated in a bait that is specifically formulated to promote feeding by pest insects and minimize the influence on non-target organisms.

Griffin Corporation

POSTAL ADDRESS: P.O. Box 1847, Valdosta, GA 31603-1847 U.S.A. • LOCATION: Rocky Ford Road, Valdosta, GA 31601, U.S.A.
TELEPHONE: [illegible] • TELEX U.S.: 552 [illegible] GRIFFIN • TELEX INTL.: 296-5694 GRIFINTL • FAX: 912-244-5813

GRIFFIN CORPORATION
Rocky Ford Rd., Valdosta, GA 31603
(912) 242-8635

Material Safety Data Sheet

Issue Date
November 8, 1988

Trade Name
Sulfluramid

Chemical Family
Fluoroaliphatic sulfonamide

1 **Ingredients** — Percent

$C_8F_{17}SO_2N(C_2H_5)H$
N-Ethyl Perfluorooctanesulfonamide ~98
Related Fluorochemical Compounds

2 **Physical State**

Solubility in Water	Nil
Bulk Density (packed)	0.45 g/mL
Percent Volatile (at ambient temp.)	0
Sublimation in closed containers @ 50°C	0%
in open containers @ 40°C	0.007%/minute
in open containers @ 60°C	0.03%/minute
pH	NA

Appearance and Odor
White color needle-like crystals. Bland odor.

3 **Fire and Explosion Hazard Data**

Flash point (Test Method)
Seta Flash CC >200°F

Extinguishing Media
Water, Foam, CO_2, Dry Chemical

Special Fire Fighting Hazards
None Known

Unusual Fire and Explosion Hazards
Toxic by-products, including Hydrogen Fluoride, may be formed.

4 **Environmental Information**

Spill Response
Collect spilled material. Clean up residue.

Recommended Disposal
Mix with flammable material and incinerate. HF is a combustion product.
Disposal Alternate: Dispose of waste at approved chemical waste facility.

Sulfluramid page 2.

5 **Health Hazard Data**

Eye Contact
Non-irritating ocularly.

Skin Contact
Non-irritating ⟷ mildly irritating, dermally.

Inhalation
Animal studies indicate inhalation hazard is low.

Ingestion
Acute oral LD50 (rat) >500 mg/kg. Preliminary studies in dogs suggest that continued ingestion of high doses may arrest spermatogenesis.

Suggested First Aid
Eye Contact: Immediately flush with plenty of water.
Skin Contact: Wash affected area with soap and water.
Inhalation: Remove to fresh air.
Ingestion: Give two glasses of water. Induce vomiting by placing finger in back of throat. Call a physician or Poison Control Center. Never give anything by mouth to an unconscious person.

6 **Reactivity Data**

Stable. Hazardous polymerization will not occur.
Thermal decomposition may produce toxic materials including Hydrogen Fluoride

7 **Special Protection Information**

Eye Protection: Safety Glasses
Skin Protection: Rubber Gloves and full clothing.
Ventilation: General ventilation is adequate. If chemical is subjected to heat or in dusty areas wear respirator approved under NIOSH TC-21C-132.

8 **Department of Transportation**

DOT Proper Shipping Name:
Chemicals N.O.S.

9 **Environmental Data**

COD=.002 g/g; BOD5=Nil; BOD20=Nil; 96-Hr. LC50 Fathead Minnow >34 mg/l.
48-hr. EC50 Waterflea (D. magna) >10 mg/l;
Concentration inhibiting respiration of waste treatment microorg. >1000 mg/l.

Notice of Warranty

All statements, technical data and recommendations contained herein are based on tests that I believed to be reliable, but the accuracy or completeness thereof is not guaranteed. The following is made in lieu of all warranties, express or implied.

Prepared By:	Title:	Approved By:	Title:
[signature]	Business Mgr: Insecticides		

AGRISEARCH INCORPORATED
TEST MATERIAL LOG

Agrisearch #: 89-046 Data Received: 9/14/89

Sponsor: DuPont (NEN) Data Logged: 9/14/89

TEST MATERIAL

Name/ID#: N-Ethylperfluorooctane Sulfonamide [N-Et-FOSA-1-^{14}C]

Lot/Batch #: 2271-131

Amount Received per Sponsor: 204.7 mg = ~~5.5 mCi~~ See Description @ Study

Description: S.A. = 14.1 mCi/mM
Liquid (yellow) in Ind. Polyethylene Glycol m.w. 400
@ 4.4 mg/in vial

Shipped Via: Federal Express

Storage: ~~RT~~ Temp. Refrigerate Freezer

Test Substance Metabolite/degradate Analytical standard

Initials SCA

AGI-011

THE UNIVERSITY OF GEORGIA
COLLEGE OF VETERINARY MEDICINE
ATHENS, GEORGIA 30602

Office of the Dean
(404) 542-3461

SAMPLE ACCOUNTABILITY FORM

89-046

Sample Number: 2271-131	Sample Name: [ethyl-1-14C] N-ethylperfluorooctane sulfonamide
Date Shipped: 11 September 1989	Shipped By: Feliz Burgess
Date Received:	
Description of Shipment: 1 vial containing 4 mCi in PEG 400 at 5 uCi/ul	

Sample Custody			
Date	Amount	From	To
11 Sept 1989	~1 ml	R. O. Manning	Bill Spare (AgriSearch) mD 21013-01

September 11, 1989

Dear Sir:

The N-Ethylperfluorooctane Sulfonamide (N-Ethyl-1-^{14}C) was originally put into suspension in 1.0 ml Polyethylene Glycol M.W. 400. This resulted in a suspension with 5.5 millicuries per ml. Of the original solution approximately 200 µl have been removed from the vial, leaving 4.4 millicuries in the vial.

Sincerely,

Randall C. Manning

Randall O. Manning, Ph.D.

89-046

** ANALYSIS FILE ** 2:AN0301.

OPERATOR MJZ DATE 7/27/90

SAMPLE= GX071

PROJECT Purity CHECK

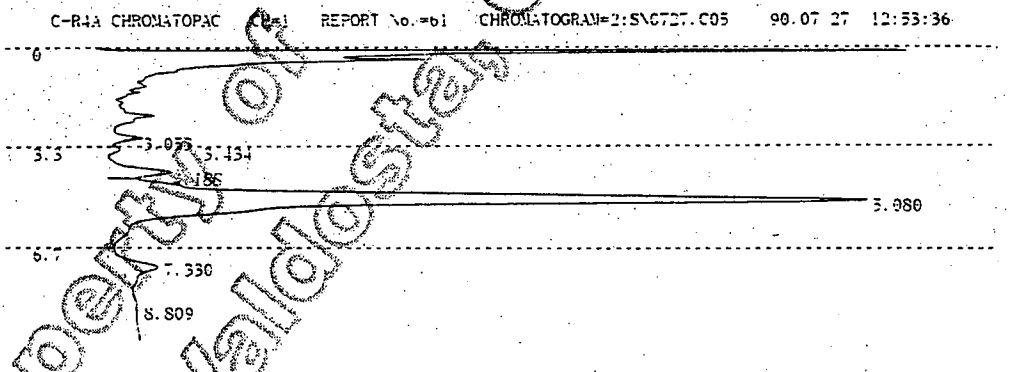
MATRIX TLC purified GX071

COLUMN
LENGTH: 15 M
DIA.: 0.53 mm I.D.
LIQUID PHASE: RTX-5
FILM: 1.00um
SUPPORT: CAPILLARY
MESH:
CARRIER GAS: HELIUM
ROTAMETER:
INLET PRESS.: 1.0 kg/cmxcm
RATE:
CHART SPEED: 5 mm/min
SAMPLE:
INJ. VOL.: 4 ul

DETECTOR: ECD
RANGE: 1
CURRENT: 0.5nA
FLOW RATES kg/cmxcm
HYDROGEN: AIR:
SCAVANGE:
SPLIT:
TEMPERATURE. CENTIGRADE
DET.: 200 INJ.: 200
COLUMN INITIAL: 70 for 1min
FINAL: 200
RATE: 30
SOLVENT:
CONC. (UNITS) ng

PROCESSING PARAMETERS

WIDTH (sec)	5	SLOPE (uV/min)	100
DRIFT (uV/min)	25000	MIN. AREA (Count)	50000
T.DBL (min)	0	STOP. TM (min)	25
ATTEN (2^X mV)	10	SPEED (mm/min)	5
METHOD (0~8)	4	W/B (0:WINDOW 1:BAND)	0
WINDOW (%)	5	SPL.WT	100
IS.WT		CAL'B POINTS (1~8)	4



** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME	
1	1	3.053	75303	11176					
	2	3.434	119761	10442					
	3	4.188	1323300	70109					
	4	5.08	25386776	855948	VE	1	1489.6021	89-057	95.4% GX-071
	5	7.33	1208396	41217		2	34.3114	89-058	
	6	8.809	53090	1122					
		TOTAL	26166622	990015			1523.9135		

SAMPLE= GX071 INJ.VOL. 0.2ul SAMPLE VOL.____ml ATTENUATION IS 10
COMMENTS 10pl of 5ml Final ACN

Purified GX071 Thin Layer Chromatography Ambis Scan

Purity Check GX071 (Rev) 7/25/90

SS: Chloroform/methanol/Formic Acid/water
7:1:1:2 v/v/v/v

99.0%

SS: Toluene/Acetone
7:2:5

99.4%

IMAGE
GX-071A

agrisearch
22 JUL 90
08:42 AM
ver: 2.01
ser#: 621

volt: 1677
time: 30 min

cnts: 28372
accptd: 67%
hi cnt: 97
CPMcm2: 41
Res plate: 2
(0.8 x 3.2)

DSCGMRREMOBVSFULP?

	0	1
	97	18
	1	1
	1	1
ZOOM	1.0	1.0
MONOCHROME		

AGRISEARCH 2515 - 56

7.25.90 Exp

** ANALYSIS FILE ** 2:AN0113.

OPERATOR JMJC DATE 10/30/90

SAMPLE# GX071 Def 3 Dose Stock

PROJECT 2515 @ 10/30/90 JMJC

MATRIX Hexane MeOH

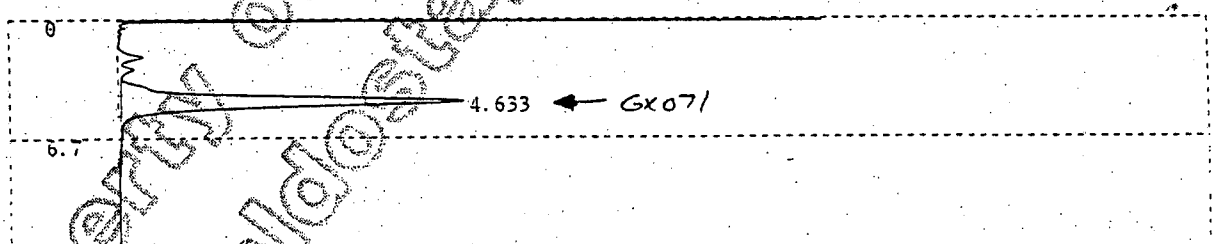
COLUMN
LENGTH: 60 M
DIA.: 0.53 mm I.D.
LIQUID PHASE: RTX-5
FILM: 1.00um
SUPPORT: CAPILLARY
MESH:
CARRIER GAS: HELIUM
ROTAMETER:
INLET PRESS.: 1.0 kg/cmxcm
RATE:
CHART SPEED: 5 mm/min
SAMPLE:
INJ. VOL.: 4 ul

DETECTOR: ECD
RANGE: 1
CURRENT: 0.5nA
FLOW RATES kg/cmxcm
HYDROGEN: AIR:
SCAVANGE:
SPLIT:
TEMPERATURE, CENTIGRADE
DET.: 250 INJ.: 250
COLUMN INITIAL: 70 15min
FINAL: 200
RATE: 30
SOLVENT:
CONC. UNITS: ng

PROCESSING PARAMETERS

WIDTH (sec)	5	SLOPE (uV/min)	500
DRIFT (uV/min)	25000	MIN.AREA (Count)	10000
T.DBL (min)	0	STOP.TIME (min)	25
ATTEN (2^X mV)	10	SPEED (mm/min)	5
METHOD (0~8)	4	W/B (0:WINDOW 1:BAND)	0
WINDOW (%)		SPL#	100
IS.WT		CAL'S POINTS (1~8)	5

C-R4A CHROMATOPAC CH=2 REPORT No.=19 CHROMATOGRAM=2:SN1030.C02 90/10/30 11:16:27



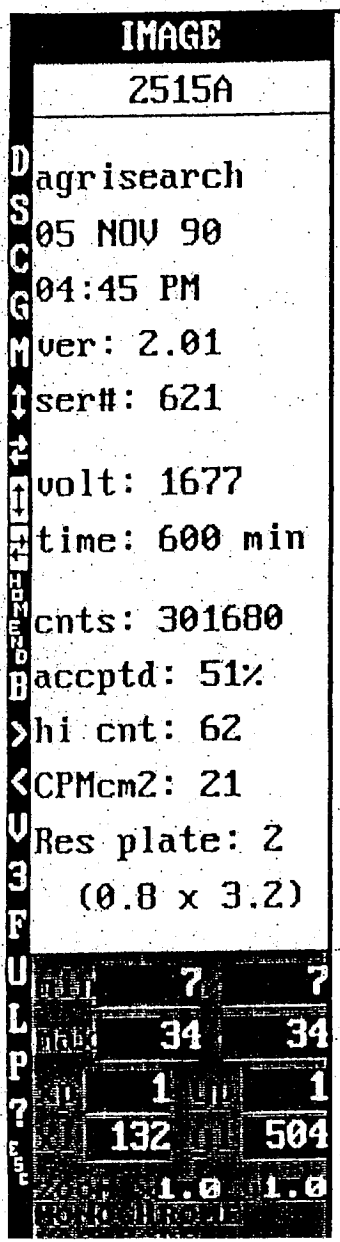
** CALCULATION REPORT **

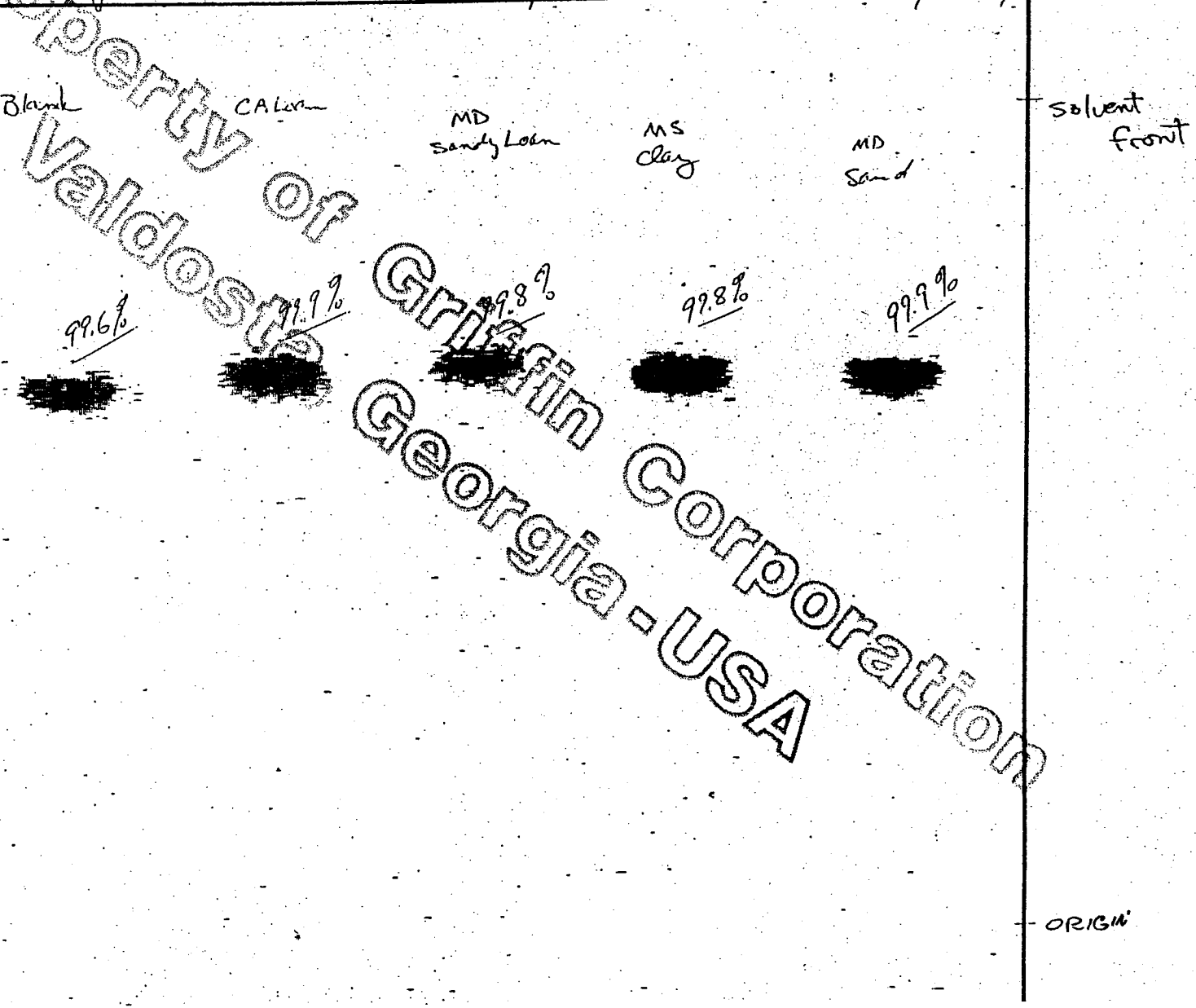
CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
2	1	4.633	13236853	321427		1	504.4618	89-057
	3	15.842	18189	529				
	4	17.677	508922	14045				
	5	19.388	540891	13196				
		TOTAL	14304852	349197			504.4618	

SAMPLE# GX071 INJ. VOL. 4 ul SAMPLE VOL. ____ml ATTENUATION IS 10
COMMENTS Def 3 2515

Ambis Scan of Thin Layer Chromatography

2515 Adsorption Solutions SS: Toluene/Acetone 75/25 v/v 11/7/90

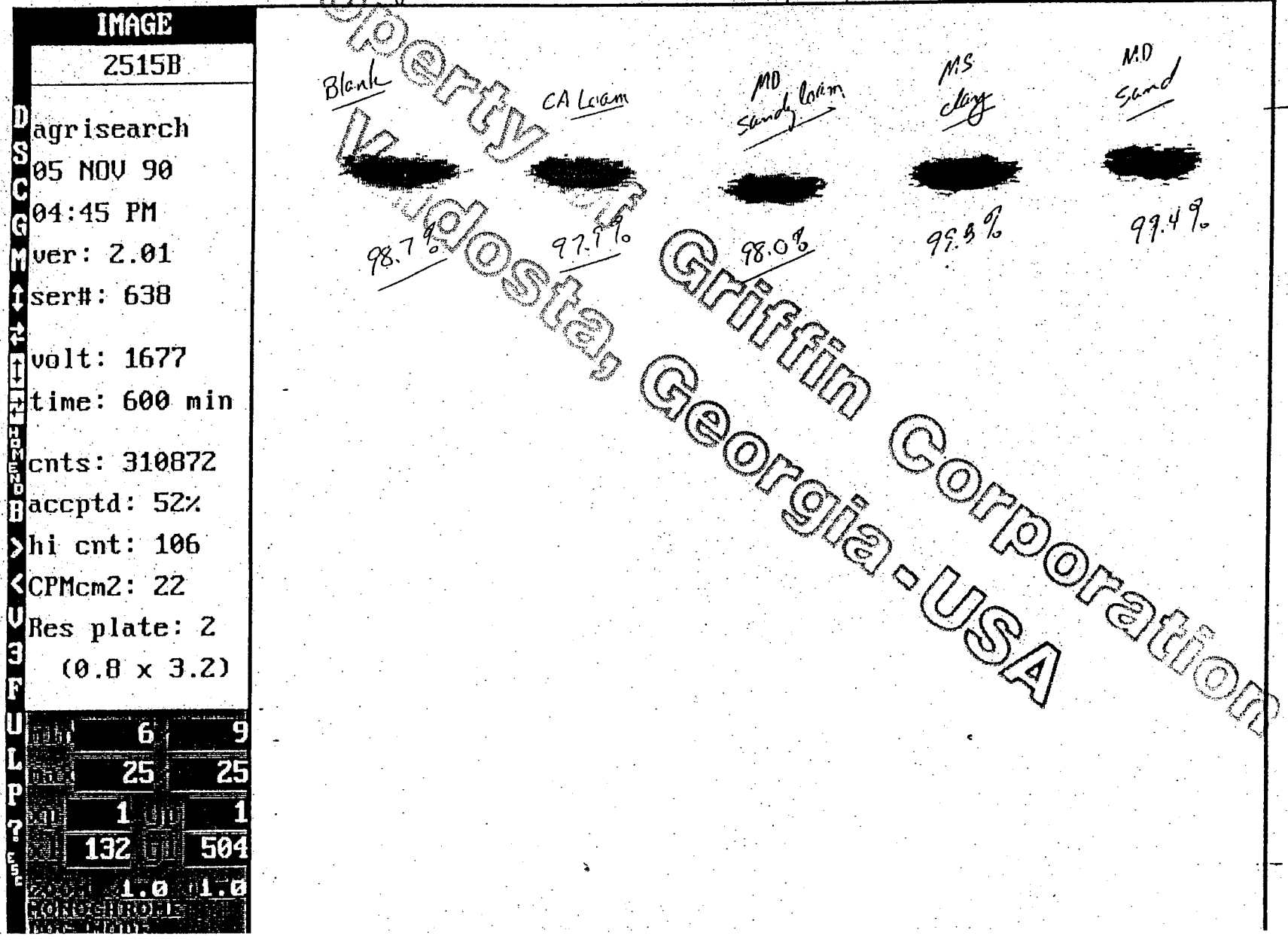




AMBIS Scan of Thin Layer Chromatography

25/15 Adsorption Solution ss: Chloroform / methanol / formic acid / water 75/15/8/2 v/v/v/v

11/7/90



APPENDIX C

Representative Data

AGRISEARCH INC.

DEFINITIVE 3

DOSE CALCULATIONS
SPECIFIC ACTIVITY 59400 DPM/ug

PROJECT # 2515

NOMINAL CONC.	LS3801 DPM	TOTAL DPM	AVERAGE DPM/ML	CALC. CONC.	ug/mL STOCK
0.02 ug/mL		19106	764	0.013 ug/mL	2.5
0.03 ug/mL		38213	1529	0.026 ug/mL	5.0
0.08 ug/mL	103640	95532	3821	0.064 ug/mL	12.5
	97201				
	91918				
	84260				
	100642				
0.15 ug/mL		191064	7643	0.129 ug/mL	25.0
0.30 ug/mL		382129	15285	0.257 ug/mL	50.0

Soil-Less Blanks Radiocarbon Balance

AGRISEARCH INC

11/12/90

DEFINITIVE ADSORPTION 3

PROJECT # 2515 SAMPLE SIZE: mL 25
S.A: 59400 DPM/ug

SAMPLE I.D.		DPM DOSED	ALIQ SIZE mL	LS3801 DPM	DPM/ml	AVG DPM/ml	TOTAL DPM	PPM WATER	% OF DOSE
BLANK									
ppm	REP								
0.00		0	2	27	14			0.0	
			2	60	30				
0.01	1	19106	2	447	224	225	6619	0.004	29.4
			2	452	226				
	2	19106	2	402	201	210	5250	0.004	27.5
			2	438	219				
0.03	1	38213	2	569	285	289	7225	0.005	18.9
			2	587	294				
	2	38213	2	1166	583	588	14206	0.010	37.2
			2	1107	554				
0.06	1	95532	2	3045	1523	1550	38750	0.026	40.6
			2	3155	1578				
	2	95532	2	2410	1205	1172	29306	0.020	30.7
			2	2279	1140				
0.13	1	191064	2	3049	1525	1489	37213	0.025	19.5
			2	2905	1453				
	2	191064	2	5325	2663	2701	67531	0.045	35.3
			2	5480	2740				
0.26	1	382129	2	7194	3597	3542	88556	0.060	23.2
			2	6975	3488				
	2	382129	2	10801	5401	5285	132125	0.089	34.6
			2	10339	5170				
							AVG =		29.7

METHANOL RINSE 25mL + INITIAL SOLUTION, ALIQUOT = 2mL

ppm	REP								
0.00		0	2	100	50			0.0	
			2	95	48				
0.01	1	19106	2	799	400	400	20013	0.007	104.7
			2	802	401				
	2	19106	2	800	400	409	20425	0.007	106.9
			2	834	417				
0.03	1	38213	2	1484	742	737	36850	0.012	96.4
			2	1464	732				
	2	38213	2	1620	810	824	41188	0.014	107.8
			2	1675	838				
0.06	1	95532	2	3543	1772	1772	88613	0.030	92.8
			2	3546	1773				
	2	95532	2	3902	1951	1968	98375	0.033	103.0
			2	3968	1984				
0.13	1	191064	2	7450	3725	3704	185188	0.062	96.9
			2	7365	3683				
	2	191064	2	7634	3817	3896	194775	0.066	101.9
			2	7948	3974				
0.26	1	382129	2	15174	7587	7445	372250	0.125	97.4
			2	14606	7303				
	2	382129	2	15361	7681	7708	385375	0.130	100.8
			2	15469	7735				

AVG TOTAL = 100.87
=======

AGRISEARCH INC.

DEFINITIVE 3 CA LOAM

PROJECT # : 2515 SAMPLE SIZE: mL 25
S.A: 59400 DPM/ug

SAMPLE I.D. ppm	REP	ADSORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.01	1	0.194	-1.638	0.003	-5.894
	2	0.234	-1.454	0.004	-5.626
0.03	1	0.449	-0.801	0.005	-5.352
	2	0.406	-0.901	0.004	-5.420
0.06	1	1.349	0.299	0.011	-4.546
	2	1.627	0.486	0.014	-4.246
0.13	1	3.131	1.141	0.017	-4.061
	2	2.873	1.055	0.019	-3.958
0.26	1	6.340	1.847	0.022	-3.835
	2	5.450	1.696	0.029	-3.529

Duplicates

LINEAR REGRESSION ANALYSIS

Regression Output:

Constant	7.137	CORRELATION	0.987
Std Err of Y Est	0.221	SLOPE (1/n)	1.498
R Squared	0.974	INTERCEPT	7.137
No. of Observations	10		
Degrees of Freedom	8		
X Coefficient(s)	1.498	n	0.667
Std Err of Coef.	0.087	Kd	1257
		Koc	267130
		% O.C.	0.471

1/4/91

AGRISEARCH INC.

DEFINITIVE 3 MD SANDY LOAM

PROJECT # : 2515 SAMPLE SIZE: mL 25
S.A: 59400 DPM/ug

SAMPLE I.D. ppm	REP	ADSORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.01	1	0.246	-1.401	0.002	-6.160
	2	0.190	-1.659	0.002	-6.132
0.03	1	0.569	-0.563	0.004	-5.628
	2	0.504	-0.685	0.004	-5.530
0.06	1	1.474	0.388	0.008	-4.817
	2	1.274	0.242	0.007	-4.896
0.13	1	3.350	1.209	0.013	-4.378
	2	3.129	1.141	0.012	-4.457
0.26	1	5.845	1.766	0.019	-3.947
	2	5.899	1.775	0.019	-3.957

Calculate

LINEAR REGRESSION ANALYSIS

Regression Output:

Constant	7.739		
Std Err of Y Est	0.126	CORRELATION	0.996
R Squared	0.991	SLOPE (1/n)	1.506
No. of Observations	10	INTERCEPT	7.739
Degrees of Freedom	8		
X Coefficient(s)	1.506	n	0.664
Std Err of Coef.	0.050	Kd	2296
		Koc	205466
		% O.C.	1.118

1/4/91 JCS

AGRISEARCH INC.

DEFINITIVE 3 MD SAND

PROJECT # : 2515 SAMPLE SIZE: mL 25

S.A: 59400 DPM/ug

SAMPLE I.D. ppm	REP	ADSORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.01	1	0.137	-1.988	0.004	-5.521
	2	0.111	-2.198	0.003	-5.704
0.03	1	0.217	-1.526	0.006	-5.152
	2	0.229	-1.475	0.005	-5.301
0.06	1	0.249	-1.392	0.012	-4.385
	2	0.991	-0.009	0.015	-4.215
0.13	1	2.122	0.752	0.017	-4.093
	2	1.978	0.682	0.024	-3.718
0.26	1	2.701	0.993	0.044	-3.113
	2	3.536	1.263	0.077	-2.568

LINEAR REGRESSION ANALYSIS

Regression Output:

Constant	4.775	CORRELATION	0.935
Std Err of Y Est	0.508	SLOPE (1/n)	1.203
R Squared	0.874	INTERCEPT	4.775
No. of Observations	10		
Degrees of Freedom	8		
		n	0.831
X Coefficient(s)	1.203	Kd	118
Std Err of Coef.	0.161	Koc	22383
		% O.C.	0.529

1/4/91

AGRISEARCH INC.

DEFINITIVE 3 CLAY

PROJECT # : 2515 SAMPLE SIZE: mL 25
S.A: 59400 DPM/ug

SAMPLE I.D. ppm	REP	ADSORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.01	1	0.221	-1.511	0.002	-6.347
	2	0.213	-1.546	0.002	-6.254
0.03	1	0.312	-1.165	0.003	-5.710
	2	0.656	-0.422	0.003	-5.661
0.06	1	1.203	0.185	0.006	-5.077
	2	1.298	0.260	0.009	-4.701
0.13	1	1.638	0.493	0.012	-4.447
	2	2.105	0.744	0.012	-4.420
0.26	1	4.935	1.596	0.018	-4.011
	2	3.959	1.376	0.019	-3.980

calculations

LINEAR REGRESSION ANALYSIS

Regression Output:

Constant	6.451		
Std Err of Y Est	0.242	CORRELATION	0.980
R Squared	0.960	SLOPE (1/n)	1.277
No. of Observations	10	INTERCEPT	6.451
Degrees of Freedom	8		
		n	0.783
X Coefficient(s)	1.277	Kd	633
Std Err of Coef.	0.098	Koc	22436
		% O.C.	2.824

1/4/91

AGRISEARCH INC.

DEFINITIVE 3 CA LOAM RADIOCARBON BALANCE

PROJECT # : 2515 SAMPLE SIZE 25 mL
S.A.: 59400 DPM/ug

SAMPLE I.D. ppm	REP	PERCENT OF DOSE: WATER ADSORPT	SOIL ADSORPT	METHANOL RINSE	TOTAL
0.01	1	21.4	28.2	41.8	91.4
	2	28.0	32.5	38.4	98.9
0.03	1	18.4	36.0	31.8	86.3
	2	17.2	31.1	33.2	81.5
0.06	1	16.5	38.7	43.5	98.7
	2	22.3	48.1	39.0	109.4
0.13	1	13.3	45.4	41.7	100.4
	2	14.8	38.0	46.5	99.4
0.26	1	8.4	46.2	42.4	97.0
	2	11.4	43.1	41.9	96.3
				AVG =	95.9
				1S.D.	7.84

AGRISEARCH INC.

DEFINITIVE 3 MD SANDY LOAM RADIOCARBON BALANCE

PROJECT # : 2515 SAMPLE SIZE 25 mL
S.A.: 59400 DPM/ug

SAMPLE I.D. ppm	REP	PERCENT OF DOSE WATER ADSORPT	SOIL ADSORPT	METHANOL RINSE	TOTAL
0.01	1	16.4	34.0	41.0	91.4
	2	16.9	30.9	38.7	86.5
0.03	1	14.0	44.0	36.4	94.4
	2	15.4	37.8	32.9	86.1
0.06	1	12.6	48.0	39.4	100.0
	2	11.6	33.2	51.5	96.3
0.13	1	9.7	57.4	36.8	103.9
	2	9.0	40.2	53.5	102.8
0.26	1	7.5	49.7	34.6	91.8
	2	7.4	55.1 *	29.1	91.6
				AVG =	94.5
				1S.D.	6.25

* Based on 2x of 25 sample due to spillage & bead.

AGRISEARCH INC.

DEFINITIVE 3 MD SAND RADIOCARBON BALANCE

PROJECT # : 2515 SAMPLE SIZE 25 mL
S.A.: 59400 DPM/ug

SAMPLE I.D. ppm		PERCENT OF DOSE WATER ADSORPT	SOIL ADSORPT	METHANOL RINSE	TOTAL
	REP			REPEATS	
0.01	1	31.1	16.5	43.5	91.2
	2	25.9	15.6	36.7	78.2
0.03	1	22.5	14.7	40.1	77.3
	2	19.4	22.5	41.2	83.0
0.06	1	19.4	8.2	54.8	82.4
	2	23.0	21.6	36.4	81.0
0.13	1	13.0	38.5	34.8	86.2
	2	18.9	31.7	37.4	87.9
0.26	1	17.3	22.8	37.5	77.5
	2	29.8	29.7	32.3	91.8
				AVG =	83.6
				1S.D.	5.42

AGRISEARCH INC.

DEFINITIVE 3 CLAY RADIOCARBON BALANCE

PROJECT # : 2515 SAMPLE SIZE 25 mL
S.A.: 59400 DPM/ug

SAMPLE I.D. ppm	REP	PERCENT OF DOSE WATER ADSORPT	SOIL ADSORPT	METHANOL RINSE	TOTAL
0.01	1	15.0	26.7	47.2	88.9
	2	14.9	26.7	34.0	75.6
0.03	1	12.9	19.6	50.6	83.0
	2	13.5	47.8	46.0	107.3
0.06	1	9.7	35.4	47.2	92.4
	2	14.1	34.7	52.8	101.6
0.13	1	9.1	26.7	46.1	81.8
	2	9.4	34.0	46.9	90.3
0.26	1	7.0	37.8	46.6	91.4
	2	7.3	23.9	56.0	87.2
				AVG =	90.0
				1S.D.	9.27

AGRISEARCH INC.

DEFINITIVE MD SANDY LOAM

PROJECT # : 2515 SAMPLE SIZE: mL 50
S.A: 59400 DPM/ug

SAMPLE I.D. ppm	REP	ADSORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.07	1	2.368	0.862	0.012	-4.400
	2	2.424	0.885	0.008	-4.805
0.14	1	4.496	1.503	0.017	-4.072
	2	4.026	1.393	0.013	-4.315
0.18	1	5.302	1.668	0.029	-3.543
0.22	1	4.692	1.546	0.032	-3.441
	2	6.748	1.909	0.033	-3.428
0.70	1	23.597	3.161	0.056	-2.881
	2	23.557	3.159	0.084	-2.473

By difference

LINEAR REGRESSION ANALYSIS
Regression Output:

Constant	5.632		
Std Err of Y Est	0.345	CORRELATION	0.925
R Squared	0.856	SLOPE (1/n)	1.038
No. of Observations	9	INTERCEPT	5.632
Degrees of Freedom			
		n	0.964
X Coefficient(s)	1.038	Kd	279.2
Std Err of Coef.	0.161	Koc	24983
		% O.C.	1.118

Part for Desorption
4 Dec 89

SAMPLE I.D. (PPM)	REP	DESORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.07	1	2.707	0.996	0.000	ERR
	2	1.821	0.600	0.000	ERR
0.14	1	6.660	1.896	0.000	ERR
	2	5.096	1.628	0.000	ERR
0.18	1	7.173	1.970	0.000	ERR
	2	LAB ACCIDENT - SAMPLE LOST			
0.22	1	7.129	1.964	0.000	ERR
	2	5.753	1.750	0.000	ERR
0.70	1	29.956	3.400	0.000	ERR
	2	55.028	4.008	0.000	ERR

LINEAR REGRESSION ANALYSIS — NO DESORPTION NO CALCULATION
Regression Output:

Constant			
Std Err of Y Est		CORRELATION	0.000
R Squared		SLOPE (1/n)	
No. of Observations		INTERCEPT	
Degrees of Freedom			
		n	ERR
X Coefficient(s)		Kd	
Std Err of Coef.		Koc	
		% O.C.	1.118

AGRISEARCH INC.

DEFINITIVE MD SAND

PROJECT # : 2515 SAMPLE SIZE: mL 50
S.A: 59400 DPM/ug

SAMPLE I.D. ppm	REP	ADSORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.07	1	1.422	0.352	0.014	-4.283
	2	2.611	0.960	0.013	-4.354
0.14	1	3.707	1.310	0.019	-3.961
	2	3.468	1.244	0.024	-3.726
0.18	1	4.209	1.437	0.036	-3.328
	2	4.907	1.591	0.028	-3.570
0.22	1	4.033	1.394	0.040	-3.219
	2	6.610	1.889	0.036	-3.328
0.70	1	21.953	3.089	0.086	-2.453
	2	19.282	2.959	0.095	-2.356

By difference

LINEAR REGRESSION ANALYSIS

Regression Output:

Constant	5.623		
Std Err of Y Est	0.320	CORRELATION	0.934
R Squared	0.871	SLOPE (1/n)	1.155
No. of Observations	10	INTERCEPT	5.623
Degrees of Freedom	8		
X Coefficient(s)	1.155	n	0.866
Std Err of Coef.	0.157	Kd	276.7
		Koc	52260
		% O.C.	0.529

SAMPLE I.D. (PPM)	REP	DESORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.07	1	1.234	0.210	0.002	-6.362
	2	0.544	-0.608	0.000	ERR
0.14	1	1.708	0.532	0.000	ERR
	2	1.566	0.449	0.000	ERR
0.18	1	1.979	0.683	0.000	ERR
	2	2.185	0.782	0.000	ERR
0.22	1	2.450	0.896	0.000	ERR
	2	2.165	0.772	0.000	ERR
0.70	1	12.071	2.491	0.000	ERR
	2	19.130	2.951	0.000	ERR

Data for Desorption
Not 1/4/91

LINEAR REGRESSION ANALYSIS NO DESORPTION
Regression Output: NO CALCULATION

Constant			
Std Err of Y Est		CORRELATION	0.000
R Squared		SLOPE (1/n)	
No. of Observations		INTERCEPT	
Degrees of Freedom			
X Coefficient(s)		n	ERR
Std Err of Coef.		Kd	
		Koc	
		% O.C.	0.529

AGRISEARCH INC.

DEFINITIVE CA Loam

PROJECT # : 2515 SAMPLE SIZE: mL 50
S.A: 59400 DPM/ug

SAMPLE I.D. ppm	REP	ADSORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.07	1	1.909	0.646	0.014	-4.271
	2	1.668	0.511	0.011	-4.536
0.14	1	2.640	0.971	0.027	-3.609
	2	3.437	1.234	0.017	-4.054
0.18	1	4.495	1.503	0.031	-3.471
	2	5.093	1.628	0.029	-3.539
0.22	1	4.376	1.476	0.059	-2.828
	2	7.354	1.995	0.032	-3.440
0.70	1	19.443	2.967	0.076	-2.575
	2	21.978	3.090	0.094	-2.367

LINEAR REGRESSION ANALYSIS

Regression Output:

Constant	5.409		
Std Err of Y Est	0.415	CORRELATION	0.895
R Squared	0.800	SLOPE (1/n)	1.097
No. of Observations	10	INTERCEPT	5.409
Degrees of Freedom	8		
		n	0.911
X Coefficient(s)	1.097	Kd	223.4
Std Err of Coef.	0.194	Koc	47475
		% O.C.	0.471

SAMPLE I.D. (PPM)	REP	DESORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.07	1	2.352	0.855	0.000	ERR
	2	1.557	0.443	0.000	ERR
0.14	1	5.227	1.654	0.000	ERR
	2	4.228	1.442	0.000	ERR
0.18	1	7.486	2.013	0.000	ERR
	2	5.961	1.785	0.000	ERR
0.22	1	8.049	2.086	0.000	ERR
	2	6.343	1.847	0.000	ERR
0.70	1	41.328	3.722	0.000	ERR
	2	58.540	4.070	0.000	ERR

LINEAR REGRESSION ANALYSIS — NO DESORPTION NO CALCULATION

Regression Output:

Constant			
Std Err of Y Est		CORRELATION	0.000
R Squared		SLOPE (1/n)	
No. of Observations		INTERCEPT	
Degrees of Freedom			
		n	ERR
X Coefficient(s)		Kd	
Std Err of Coef.		Koc	
		% O.C.	0.471

Data for Design
PD 1/4/91

AGRISEARCH INC.

DEFINITIVE CLAY

PROJECT # : 2515 SAMPLE SIZE: mL 50
S.A: 59400 DPM/ug

SAMPLE I.D. ppm	REP	ADSORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.07	1	2.705	0.995	0.007	-4.987
	2	3.451	1.239	0.007	-5.018
0.14	1	6.293	1.839	0.013	-4.348
	2	5.579	1.719	0.010	-4.585
0.18	1	5.827	1.762	0.018	-4.019
	2	5.669	1.735	0.019	-3.944
0.22	1	7.829	2.058	0.025	-3.707
	2	7.267	1.983	0.021	-3.870
0.70	1	24.023	3.179	0.059	-2.830
	2	22.270	3.103	0.064	-2.747

by Hyperquad

LINEAR REGRESSION ANALYSIS

Regression Output:

Constant	5.405		
Std Err of Y Est	0.204	CORRELATION	0.961
R Squared	0.924	SLOPE (1/n)	0.860
No. of Observations	10	INTERCEPT	5.405
Degrees of Freedom	8		
		n	1.162
X Coefficient(s)	0.860	Kd	222.5
Std Err of Coef.	0.087	Koc	7881
		% O.C.	2.824

SAMPLE I.D. (PPM)	REP	DESORPTION SOIL ppm	LN SOIL ppm	WATER ppm	LN WATER ppm
0.00		0.000		0.000	
0.07	1	1.571	0.452	0.000	ERR
	2	1.691	0.525	0.000	ERR
0.14	1	5.197	1.648	0.000	ERR
	2	3.814	1.339	0.000	ERR
0.18	1	7.451	2.008	0.000	ERR
	2	5.470	1.699	0.000	ERR
0.22	1	8.100	2.092	0.000	ERR
	2	6.787	1.915	0.000	ERR
0.70	1	57.618	4.054	0.000	ERR
	2	68.221	4.223	0.000	ERR

Detection Limit
32/59400/3
= 0.00002 ppm

LINEAR REGRESSION ANALYSIS — NO DESORPTION NO CALCULATION

Regression Output:

Constant			
Std Err of Y Est		CORRELATION	0.000
R Squared		SLOPE (1/n)	
No. of Observations		INTERCEPT	
Degrees of Freedom			
		n	ERR
X Coefficient(s)		Kd	
Std Err of Coef.		Koc	
		% O.C.	2.824

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