ADSORPTION

V 2649G (V 2648G)

THE SOAP AND DETERGENT ASSOCIATION (SDA)
New York, NY

FINAL REPORT

WESTON Study № 96-005

WESTON W.O. № 05821-007-001

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PROJECT PARTICIPANTS

The following staff members of The Soap and Detergent Association (SDA) and Roy F. Weston, Inc. had major roles in the execution of this testing program:

Alvaro J. DeCarvalho Principal Investigator	The Soap and Detergent Association
Drew McAvoy, Ph.D. Alternate Principal Investigator	The Soap and Detergent Association
Jon Doi, Ph.D. Laboratory Manager	Roy F. Weston, Inc.
Kay H. Marks Study Director	Roy F. Weston, Inc.
Kathleen C. Crapo Quality Assurance Coordinator	Roy F. Weston, Inc.
John Hash Associate Project Scientist	Roy F. Weston, Inc.

COMPLIANCE STATEMENT

WESTON Study Nº 96-005

Referenced Study: Adsorption: V 2649G (V 2648G)

Compliance Statement

The above-referenced study was conducted in accordance with all pertinent United States Environmental Protection Agency (USEPA) Good Laboratory Practice (GLP) Regulations (40 CFR, Part 792) with the following exceptions: the Sponsor accepted full responsibility for appropriate characterization and stability verification of the test substances in addition to collection (with appropriate documentation) of the test system. The dosing solutions prepared by WESTON were assayed by liquid scintillation counting (LSC), but were not analyzed for stability or uniformity. The purity of a dosing solution prepared from the primary solution of the radiolabelled material was determined by WESTON.

Kay H. Marks 6/14/96

Kay H. Marks Date
Study Director

Quality Assurance Statement

Type of Audit	Date of Audit	Date Findings Reported to Management and Study Director
Calculations	12 March 1996	12 March 1996
Spike test vessels	13 March 1996	13 March 1996
Raw data/draft report	6 May 1996	6 May 1996

Results: The Quality Assurance Unit reviewed this report for accuracy and adherence to EPA GLP Regulations and laboratory SOPs. The Quality Assurance Review indicates that the final report accurately presents the raw data as developed during Study Nº 96-005.

Kathleen C. Crapo

Kathleen C.Crapo
Quality Assurance Coordinator

6/14/96

Date

1.0 EXECUTIVE SUMMARY

Roy F. Weston, Inc. (WESTON®) conducted an adsorption study on test substance V 2649G (V 2648G) using three distinct soil samples collected from the Jacksonville, Florida SDA septic tank field study site as the test system. In this test a known quantity of test substance was added to a series of test vessels that contained upgradient groundwater and soil. The vessels were agitated to achieve equilibrium between adsorbed and dissolved test substance. The water and soil were separated by centrifugation and the amount of test substance remaining in water was determined by liquid scintillation counting (LSC).

The test system consisted of soil (core) samples collected and shipped to WESTON by Ayres Associates, 3901 Coconut Palm Drive, Tampa, Florida 33619. The chain of custody accompanying the samples identified the collection sites as follows:

- Soil from Site 1, consisting of cores SC1-A through D, was collected one foot below the trench (gravel).

- Soil from Site 2, consisting of cores SC2-A through D, was collected four feet below the trench.

- Soil from Site 3, consisting of cores SC3-A through D, was collected down gradient 20 feet and four feet below ground surface.

The samples from each designated site were composited, passed through a 2 mm sieve, and refrigerated at ~4°C. An aliquot of each composited sample was sent to A & L Great Lakes Laboratories, Inc., 3505 Conestoga Drive, Fort Wayne, Indiana 46808-4413 for characterization.

The test apparatus for the adsorption study consisted of 38 Teflon 30-mL centrifuge tubes. All tubes, except the test substance controls (no soil added), received seven grams dry weight equivalent of the appropriate soil and seven milliliters upgradient groundwater. The test substance controls received seven milliliters upgradient groundwater. The test substance was dosed to duplicate tubes (for each soil) at concentrations of 40, 200, 700, and 2,000 μg/L using the radiolabelled and unlabelled (as required) forms of V 2649G. No test substance was added to the soil blank tubes.

The test vessels were equilibrated for three hours on a shaker table rotating at ~180 rpm. The vessels were then centrifuged for 40 minutes at ~5600 x g. Triplicate aliquots of each centrate were assayed by liquid scintillation counting (LSC). The soil from selected vessels was extracted with MeOH to determine the mass of test substance present after the experiment was terminated. The summary of the test results that follows includes calculation of K_d and K_{oc} using measured data (solution and solids) and using measured solution and solids by difference:

Site 1

Vessel #	Conc., (μg/L)	Centrate[1] % Recovered	Soil % Recovered	K_d[2] (measured)	K_{oc}[3] (measured)	K_d (by difference)	K_{oc} (by difference)
3	40	31.67	39.05	1.23	946	2.11	1623
4	40	28.96	33.28	1.15	885	2.40	1846
5	200	23.23	na	nc	nc	2.97	2285
6	200	23.43	65.42	2.79	2146	3.18	2446
7	700	19.48	na	nc	nc	4.04	3108
8	700	18.33	64.64	3.53	2715	4.36	3354
9	2000	17.22	na	nc	nc	4.68	3600
10	2000	17.27	69.37	4.02	3092	4.66	3585

na= not analyzed; nc = not calculated

[1] Centrate: liquid phase following centrifugation
[2] Kd = distribution adsorption coefficient
[3] The adsorption coefficient was calculated as a function of the organic carbon content of the soil.

Site 2

Vessel #	Conc., (µg/L)	Centrate % Recovered	Soil % Recovered	K_d (measured)	K_{oc} (measured)	K_d (by difference)	K_{oc} (by difference)
13	40	52.56	na	nc	nc	0.78	2600
14	40	54.04	49.96	0.92	3067	0.73	2433
15	200	39.17	na	nc	nc	1.34	4467
16	200	43.18	37.51	0.87	2900	1.12	3733
17	700	37.47	na	nc	nc	1.50	5000
18	700	39.42	43.06	1.09	3633	1.38	4600
19	2000	41.34	na	nc	nc	1.31	4367
20	2000	41.80	37.30	0.89	2967	1.29	4300

Site 3

Vessel #	Conc., (µg/L)	Centrate % Recovered	Soil % Recovered	K_d (measured)	K_{oc} (measured)	K_d (by difference)	K_{oc} (by difference)
23	40	41.75	na	nc	nc	1.13	>11300
24	40	39.36	32.04	0.81	>8100	1.26	>12790
25	200	37.83	na	nc	nc	1.27	>12700
26	200	37.88	32.03	0.85	>8500	1.27	>12700
27	700	40.95	na	nc	nc	1.17	>11700
28	700	39.10	32.62	0.83	>8300	1.28	>12800
29	2000	52.84	na	nc	nc	0.74	>7400
30	2000	55.77	23.84	0.43	>4300	0.65	>6500

A summary of K (adsorption coefficient) and 1/n (constant describing the degree of nonlinearity of the isotherm) follows for each soil:

Test System	K (measured data)	1/n (measured data)	K (solids by difference)	1/n (solids by difference)
Site 1	0.55	1.37	1.32	1.23
Site 2	0.89	1.01	0.54	1.15
Site 3	1.38	0.86	1.90	0.88

2.0 INTRODUCTION

WESTON conducted an adsorption test on test substance V 2649G (V 2648G) using three distinct soil samples collected from the Jacksonville, Florida SDA septic tank field study site as the test system. The test was designed to determine the partitioning of the test substance between each soil and upgradient groundwater collected from the same location. This test is based on the procedures outlined in the Organization for Economic Co-Operation and Development (OECD) Guidelines for Testing of Chemicals, Method 106 (1981).

Sorption is a general expression for a process in which a test substance moves from one phase to be accumulated in another. Some of the factors influencing the relative distribution of a test substance between sorbed and solution phases include physical and chemical parameters of the molecule, properties of the soil, and properties of the water in which the test substance is dissolved.

The distribution of a test substance between sorbed and solution phases is the tendency to reach equilibrium. Sorption equilibrium has been generally described by an isotherm. An isotherm is a diagram that depicts the distribution of the test substance between a solid sorbent and the solution in equilibrium with it over a range of concentrations. The values of constants K and 1/n are calculated using the Freundlich equation. The individual K_d and K_{oc} values are calculated using the ratio of test substance sorbed to the test substance in solution at equilibrium.

3.0 OBJECTIVE

The objective of this study was to determine the partitioning of the test substance between soil and water.

4.0 EXPERIMENTAL PROTOCOL

The adsorption test was conducted in accordance with the following protocol and referred test methods:

WESTON protocol entitled "Adsorption," signed by Mr. Alvaro J. DeCarvalho (Sponsor) on 1 March 1996 and by Ms. Kay H. Marks (Study Director) on 12 March 1996.

Ms. Marks' protocol amendment #1 dated 3 May 1996: Additional study information.

The water content of the soil was performed per Methods of Soil Analysis, Volume 1, Physical and Mineralogical Properties, Including Statistics of Measurement and Sampling. Ed. C.A. Black, Am. Soc. Agron., Madison, Wisconsin, pages 82-96.

Samples generated during testing were counted by LSC per WESTON SOP 16-16-FE007.

Combustions were performed per WESTON SOP 16-16-FE051.

RAD-TLC analysis was performed per WESTON SOP 16-16-FE031.

5.0 TEST SUBSTANCE

5.1 RADIOLABELLED

CLIENT TSIN	**V 2648G**[a]
Test substance name	dodecylbenzene sulfonic acid, sodium salt [Ring-^{14}C(U)]
Specific activity, μCi/mg	77.0
Radiochemical concentration, μCi/mL	93.3
Mass concentration, mg/mL	1.2
Purity, %	>92[b]
CAS number	25155-30-0
Molecular formula	$C_{18}H_{29}SO_3Na$
Molecular weight	348 mg/mmole
Lot number	2599-075 (8/3/89)
Sample form	Methanol solution (clear)
Storage conditions	Refrigerate (~4°C) in the dark in a closed container
Date received	12/06/95
Expiration date	01/01/2000
WESTON TSIN	FE95-0140

[a] Provided by Vista Chemical Company, Austin, Texas.
[b] RAD-TLC analysis performed by WESTON on 7 February 1996.

5.2 UNLABELLED

CLIENT TSIN	V 2649G[a]
Test substance name	dodecylbenzene sulfonic acid, sodium salt
Percent active	54.08
Sample form	White slurry
Storage conditions	Refrigerate (~4°C) in the dark in a closed container
Lot number	VO-382-167-2
Date received	12/06/95
Expiration date	01/01/2000
WESTON TSIN	FE95-0139

6.0 TEST SUBSTANCE PREPARATION

6.1 V 2649G

A stock solution of the unlabelled form of the test substance (V 2649G) was prepared in deionized (ASTM Type II)[1] water at a concentration of 10,000 mg active/L as follows:

- 1.8496 grams V 2649G was weighed into a tared glass beaker.
- The test substance was transferred to a 100-mL volumetric flask and diluted to volume with deionized water.

6.2 DOSING SOLUTIONS

- Solution 1, used to dose the 40 µg/L vessels, was prepared by adding 9.3 µL of the primary solution of V 2648G to a vial, evaporating the solvent with nitrogen, and diluting the test substance to two milliliters with deionized water.

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[a] Provided by Vista Chemical Company, Austin, Texas.

[1] "Standard Specification for Reagent Water," ASTM Committee D-19 on Water, ASTM Designation D1193-77, (Reapproved 1983).

- Solution 2, used to dose the 200 μg/L vessels, was prepared by adding 9.3 μL of the primary solution of V 2648G to a vial, evaporating the solvent with nitrogen, adding 4.5 μL of the V 2649G solution, and diluting to two milliliters with deionized water.

- Solution 3, used to dose the 700 μg/L vessels, was prepared by adding 9.3 μL of the primary solution of V 2648G to a vial, evaporating the solvent with nitrogen, adding 18.5 μL of the V 2649G solution, and diluting to two milliliters with deionized water.

- Solution 4, used to dose the 2,000 μg/L vessels, was prepared by adding 9.3 μL of the primary solution of V 2648G to a vial, evaporating the solvent with nitrogen, adding 54.9 μL of the V 2649G solution, and diluting to two milliliters with deionized water.

Each of the dosing solutions was assayed by LSC on the days of preparation and test initiation. The latter results were used to determine the initial dpm added to the test vessels.

7.0 TEST SYSTEM/SOLVENT

The adsorption study was conducted using field-moist soil samples added on a dry weight basis at a ratio of one part solvent to one part soil (7 mL upgradient groundwater/7 g soil).

7.1 SOIL

The test system used in this study consisted of soil samples obtained from the saturated and unsaturated zones at the Jacksonville, Florida SDA septic tank field study site. The samples were collected on 16 January 1996 and shipped overnight to WESTON by Ayres Associates. Per a chain of custody included with the samples, WESTON treated the soils as follows:

- Cores SC1-A through SC1-D, collected one foot below the trench, were screened, composited, and identified as Site 1.

- Cores SC2-A through SC2-D, collected four feet below the trench, were screened, composited, and identified as Site 2.

- Cores SC3-A through SC3-D, collected down gradient 20 feet and 4 feet below the ground surface, were screened, composited, and identified as Site 3.

The dry weight fraction of the sieved soils was determined in percent to be 78.0, 77.2, and 77.8 for Sites 1, 2, and 3, respectively. The soils were stored at approximately 4°C.

An approximately one kilogram aliquot of each composited soil was sent to A & L Great Lakes Laboratories, Inc., 3505 Conestoga Drive, Fort Wayne 46808-4413 for the following analyses: pH, cation exchange capacity (CEC), organic matter (O.M.), water holding capacity (WHC), bulk density, textural classification (Table 7).

7.2 SOLVENT

The solvent used in this study was upgradient (background) groundwater (BW1-5) obtained from the Jacksonville, Florida SDA septic tank field study site. The water was collected on 16 January 1996 and shipped overnight to WESTON by Ayres Associates. The water was refrigerated at ~4°C until used in this study.

8.0 TEST DESIGN

Vessel #	Conc., µg/L	Soil Site #	Soil[1] g	Upgradient Water[2] mL	Test Substance[3] µL	Deionized Water µL
1	-	1	9.0	5	-	50
2	-	1	9.0	5	-	50
3	40	1	9.0	5	50	-
4	40	1	9.0	5	50	-
5	200	1	9.0	5	50	-
6	200	1	9.0	5	50	-
7	700	1	9.0	5	50	-
8	700	1	9.0	5	50	-
9	2000	1	9.0	5	50	-
10	2000	1	9.0	5	50	-
11	-	2	9.1	4.9	-	50
12	-	2	9.1	4.9	-	50
13	40	2	9.1	4.9	50	-
14	40	2	9.1	4.9	50	-
15	200	2	9.1	4.9	50	-
16	200	2	9.1	4.9	50	-
17	700	2	9.1	4.9	50	-
18	700	2	9.1	4.9	50	-
19	2000	2	9.1	4.9	50	-
20	2000	2	9.1	4.9	50	-
21	-	3	9.0	5	-	50
22	-	3	9.0	5	-	50
23	40	3	9.0	5	50	-
24	40	3	9.0	5	50	-
25	200	3	9.0	5	50	-
26	200	3	9.0	5	50	-
27	700	3	9.0	5	50	-
28	700	3	9.0	5	50	-
29	2000	3	9.0	5	50	-
30	2000	3	9.0	5	50	-
31	40	-	-	7	50	-
32	40	-	-	7	50	-
33	200	-	-	7	50	-
34	200	-	-	7	50	-
35	700	-	-	7	50	-
36	700	-	-	7	50	-
37	2000	-	-	7	50	-
38	2000	-	-	7	50	-

[1] Wet weight of soil
[2] The volume was adjusted to reflect the water associated with the field-moist soil.
[3] Volume of the respective dosing solution.

9.0 DEFINITIVE ISOTHERM EXPERIMENT

9.1 TEST SET-UP

Thirty-eight Teflon 30-mL centrifuge tubes were used as the test vessels for this study. Duplicate tubes were required for each treatment (test substance concentration/test system). Duplicate tubes were required as test substance controls for each concentration. Duplicate tubes were used as soil blanks for each soil.

9.2 SOIL ADDITION

Seven grams dry weight equivalent of the appropriate soil was added to all centrifuge tubes with the exception of the test substance control vessels.

9.3 SOLVENT ADDITION

Five milliliters of upgradient water was added to the tubes containing soil from sites 1 and 3. Tubes containing Site 2 soil received 4.9 mL upgradient groundwater. The tubes used as test substance controls received seven milliliters upgradient groundwater. The volume of water was adjusted based on the water content of the field-moist soil samples.

9.4 TEST SUBSTANCE ADDITION

Fifty microliters of the appropriate dosing solution was added to each tube with the exception of the soil blanks. The soil blanks received 50 μL deionized water.

9.5 EXPERIMENTAL PROCEDURE

The centrifuge tubes were placed on a shaker table and rotated at ~180 rpm for three hours. At the end of this time period, the tubes were centrifuged at ~5600 x g for

~40 minutes. The centrates were transferred to a separate container and triplicate 1 mL aliquots were cocktailed in Ultima Gold™ (Packard catalog # 6013329) and assayed by LSC.

A mass balance was determined on one tube for each concentration from each soil site using the following procedure:

- The solids were transferred to a clean centrifuge tube.
- 20 mL MeOH was added to the solids; the tubes were vortexed for ~20 minutes.
- The tubes were centrifuged for ~40 minutes @ ~5600 x g (7460 rpm).
- The centrate was transferred to a graduated cylinder; the volume was documented.
- Triplicate 1 mL aliquots of the centrate were cocktailed in Ultima Gold and assayed by LSC.
- The extraction procedure and LSC assay were repeated one additional time.

The following procedures were performed to improve the mass balance percents:

- 20 mL MeOH was added to the original centrifuge tubes for each treatment extracted above; the tubes were vortexed for ~20 minutes; triplicate 1-mL aliquots of the MeOH were cocktailed with Ultima Gold and assayed by LSC.
- Test vessel 3 (Site 1 at 40 μg/L) was submitted to the extraction procedure to confirm the results obtained from test vessel 4.

- Test vessel 30 (Site 3 at 2,000 μg/L) was extracted a third and fourth time to determine if the MeOH extraction procedure was efficient in removing all sorbed test substance from the solids.

- Aliquots of the solids pellet from test vessels 3, 4, and 24 were combusted to determine if any test substance remained in the soil following extraction with MeOH (Table 5).

- The test substance recovered from the test substance control vessels ranged from 55 to 76 percent. To determine the reason for the low recoveries in these tubes, container rinses using 20 mL MeOH were conducted (Table 1). When the test substance that was found to be sorbed to the container was included, the recoveries ranged in percent from 87 to 102.

10.0 MISCELLANEOUS STUDY INFORMATION

10.1 FACILITY AND PERSONNEL

The testing was performed at WESTON's Laboratory, 254 Welsh Pool Road, Lionville, Pennsylvania 19341-1345. Ms. Marks was the Study Director and conducted the study. Mr. Hash sieved and composited the soils.

10.2 STUDY DATES

Soil collected: 16 January 1996
Sorption test initiated: 13 March 1996
Final data collected: 10 April 1996

10.3 DATA STORAGE

Test data may be found in WESTON Laboratory Notebook FE582. A copy of the final report, protocol, and all pertinent raw data and documentation will be maintained at Roy F. Weston, Inc., 254 Welsh Pool Road, Lionville, Pennsylvania 19341-1345.

11.0 RESULTS

The summary of the test results that follows includes calculation of K_d and K_{oc} using measured data (solution and solids) and using measured solution and solids by difference:

Site 1

Vessel #	Conc., (µg/L)	Centrate % Recovered	Soil % Recovered	K_d (measured)	K_{oc} (measured)	K_d (by difference)	K_{oc} (by difference)
3	40	31.67	39.05	1.23	946	2.11	1623
4	40	28.96	33.28	1.15	885	2.40	1846
5	200	23.23	na	nc	nc	2.97	2285
6	200	23.43	65.42	2.79	2146	3.18	2446
7	700	19.48	na	nc	nc	4.04	3108
8	700	18.33	64.64	3.53	2715	4.36	3354
9	2000	17.22	na	nc	nc	4.68	3600
10	2000	17.27	69.37	4.02	3092	4.66	3585

Site 2

Vessel #	Conc., (µg/L)	Centrate % Recovered	Soil % Recovered	K_d (measured)	K_{oc} (measured)	K_d (by difference)	K_{oc} (by difference)
13	40	52.56	na	nc	nc	0.78	2600
14	40	54.04	49.96	0.92	3067	0.73	2433
15	200	39.17	na	nc	nc	1.34	4467
16	200	43.18	37.51	0.87	2900	1.12	3733
17	700	37.47	na	nc	nc	1.50	5000
18	700	39.42	43.06	1.09	3633	1.38	4600
19	2000	41.34	na	nc	nc	1.31	4367
20	2000	41.80	37.30	0.89	2967	1.29	4300

Site 3

Vessel #	Conc., (µg/L)	Centrate % Recovered	Soil % Recovered	K_d (measured)	K_{oc} (measured)	K_d (by difference)	K_{oc} (by difference)
23	40	41.75	na	nc	nc	1.13	>11300
24	40	39.36	32.04	0.81	>8100	1.26	>12790
25	200	37.83	na	nc	nc	1.27	>12700
26	200	37.88	32.03	0.85	>8500	1.27	>12700
27	700	40.95	na	nc	nc	1.17	>11700
28	700	39.10	32.62	0.83	>8300	1.28	>12800
29	2000	52.84	na	nc	nc	0.74	>7400
30	2000	55.77	23.84	0.43	>4300	0.65	>6500

A summary of K (adsorption coefficient) and 1/n (constant describing the degree of nonlinearity of the isotherm) follows for each soil:

Test System	K (measured data)	1/n (measured data)	K (solids by difference)	1/n (solids by difference)
Site 1	0.55	1.37	1.32	1.23
Site 2	0.89	1.01	0.54	1.15
Site 3	1.38	0.86	1.90	0.88

12.0 TREATMENT OF RESULTS

For the isotherm experiment, a graph of the Freundlich isotherm was prepared by plotting log x/m vs log C_e. The Freundlich isotherm is described by the following equation:

$$\text{Log } x/m = \log K + (1/n) \log C_e$$

where: x = the amount of test substance sorbed

m = the amount of sorbent (i.e. soil)

C_e = equilibrium concentration of the test substance in solution

K = adsorption coefficient

n = a constant describing the degree of nonlinearity of the isotherm

The value of the constants K and 1/n were calculated. When the constant n in the Freundlich isotherm experiment is equal to 1, the Freundlich adsorption coefficient, K, can

be used as a distribution adsorption coefficient, K_d. The distribution adsorption coefficient is described by the equation:

$$K_d = \frac{x/m}{C_e}$$

If the value of n does not equal 1, K_d is calculated from the above equation.

The K_{oc} constant was calculated for each soil type. The K_{oc} constant relates the amount of material sorbed to the soil organic carbon content and is described by the equation:

$$K_{oc} = \frac{K_d}{\text{fraction organic carbon}}$$

Table 1: Definitive Isotherm: Test Substance Control Vessels
Study: 96–005

Percent Recovery Data

Vessel #	Conc., µg/L	Centrate rep 1 dpm/500 µL	Centrate rep 2 dpm/500 µL	Centrate rep 3 dpm/500 µL	Centrate total (7 mL) dpm	Centrate % Recovered	Container dpm 1 mL of 20	Container total dpm	Container % Recovered	Total % Recovered
31	40	1299	1301	1283	18121	54.75	787	15740	47.55	102.30
32	40	1435	1428	1385	19824	59.89	687	13740	41.51	101.40
33	200	1757	1798	1798	24981	65.91	683	13660	36.04	101.95
34	200	1611	1634	1637	22783	60.11	765	15300	40.37	100.48
35	700	1903	1867	1951	26698	69.53	417	8340	21.72	91.24
36	700	1855	1904	1907	26441	68.86	357	7140	18.59	87.45
37	2000	2310	2383	2302	32643	76.45	252	5040	11.80	88.25
38	2000	2240	2339	2298	32093	75.16	260	5200	12.18	87.34

Test substance dosed (based on LSC assay of the individual dosing solutions):

40 µg/L vessels: 33,100 dpm per tube
200 µg/L vessels: 37,900 dpm per tube
700 µg/L vessels: 38,400 dpm per tube
2000 µg/L vessels: 42,700 dpm per tube

Table 2: Definitive Isotherm using Test System Site 1
Study: 96–005

Test system: Site 1 (sand: 93.2%; organic carbon: 0.13%)

Vessel #	Conc., µg/L	Centrate rep 1 dpm/500 µL	Centrate rep 2 dpm/500 µL	Centrate rep 3 dpm/500 µL	Centrate total (7 mL) dpm	Centrate % Recovered	Soil (ext: 1) % Recovered	Soil (ext: 2) % Recovered	Soil total % Recovered	Container dpm 1 mL of 20	Container total dpm	Container % Recovered	Total % Recovered	Kd	Koc
1	–	10	outlier	0	70	na									
2	–	0	0	11	51	na									
3	40	782	777	687	10481	31.67	37.65	1.40	39.05	25	500	1.51	72.23	1.23	946
4	40	688	684	682	9585	28.96	32.78	0.50	33.28	24	480	1.45	63.69	1.15	885
5	200	640	629	618	8806	23.23									
6	200	635	636	632	8881	23.43	63.70	1.72	65.42	37	740	1.95	90.80	2.79	2146
7	700	513	528	562	7481	19.48									
8	700	509	502	497	7037	18.33	60.54	4.10	64.64	34	680	1.77	84.74	3.53	2715
9	2000	524	531	521	7355	17.22									
10	2000	520	553	507	7373	17.27	66.76	2.61	69.37	47	940	2.20	88.84	4.02	3092

Vessel #	Conc., µg/L	Soil (ext: 1) rep 1: dpm 1 mL of 20	Soil (ext: 1) rep 2: dpm 1 mL of 20	Soil (ext: 1) rep 3: dpm 1 mL of 20	Soil (ext: 1) total dpm	Soil (ext:1) adjusted for centrate remaining	Soil (ext: 1) % Recovered	Soil (ext: 2) rep 1: dpm 1 mL of 20	Soil (ext: 2) rep 2: dpm 1 mL of 20	Soil (ext: 2) rep 3: dpm 1 mL of 20	soil (ext: 2) total dpm	Soil (ext: 2) adjusted for extrate remaining	Soil (ext: 2) % Recovered
3	40	715	753	738	14707	12462	37.65	100	97	93	1933	463	1.40
4	40	714	705	722	14273	10851	32.78	75	89	75	1593	166	0.50
6	200	1359	1344	1394	27313	24141	63.70	186	210	214	4067	653	1.72
8	700	1273	1310	1281	25760	23248	60.54	179	167	180	3507	1575	4.10
10	2000	1541	1532	1598	31140	28508	66.76	200	208	226	4227	1113	2.61

Vessel #	Conc., µg/L	Centrate % Recovered	Soil total % recovered by extraction	Soil % recovered combustion	Container % Recovered	Mass Balance %
3	40	31.67	39.05	10.52	1.51	82.75
4	40	28.96	33.28	23.40	1.45	87.09

Table 3: Definitive Isotherm using Test System Site 2
Study: 96–005

Test system: Site 2 (sand: 93.2%; organic carbon: 0.03%)

Vessel #	Conc., µg/L	Centrate rep 1 dpm/500 µL	Centrate rep 2 dpm/500 µL	Centrate rep 3 dpm/500 µL	Centrate total (7 mL) dpm	Centrate % Recovered	Soil (ext: 1) % Recovered	Soil (ext: 2) % Recovered	Soil total % Recovered	Container dpm 1 mL of 20	Container total dpm	Container % Recovered	Total % Recovered	Kd	Koc
11	–	6	6	0	56	na									
12	–	4	0	0	19	na									
13	40	1250	1261	1217	17397	52.56									
14	40	1269	1302	1262	17887	54.04	47.81	2.15	49.96	108	2160	6.53	110.53	0.92	3067
15	200	1055	1062	1064	14845	39.17									
16	200	1133	1182	1192	16366	43.18	34.85	2.66	37.51	159	3180	8.39	89.08	0.87	2900
17	700	1035	990	1058	14387	37.47									
18	700	1068	1080	1096	15139	39.42	43.06	0.00	43.06	118	2360	6.15	88.63	1.09	3633
19	2000	1256	1267	1260	17654	41.34									
20	2000	1274	1294	1257	17850	41.80	37.30	0.00	37.30	94	1880	4.40	83.51	0.89	2967

Vessel #	Conc., µg/L	Soil (ext: 1) rep 1: dpm 1 mL of 20	Soil (ext: 1) rep 2: dpm 1 mL of 20	Soil (ext: 1) rep 3: dpm 1 mL of 20	Soil (ext: 1) total dpm	Soil (ext: 1) dpm adjusted for centrate remaining	Soil (ext: 1) % Recovered	Soil (ext: 2) rep 1: dpm 1 mL of 20	Soil (ext: 2) rep 2: dpm 1 mL of 20	Soil (ext: 2) rep 3: dpm 1 mL of 20	soil (ext: 2) total dpm	Soil (ext: 2) dpm adjusted for extract remaining	Soil (ext: 2) % Recovered
14	40	1130	1095	1107	22213	15825	47.81	147	148	145	2933	712	2.15
16	200	966	949	943	19053	13208	34.85	84	110	100	1960	1007	2.66
18	700	1097	1118	1076	21940	16533	43.06	88	115	98	2007	−736	0.00
20	2000	1114	1141	1090	22300	15925	37.30	95	138	99	2213	−17	0.00

Table 4: Definitive Isotherm using Test System Site 3
Study: 96–005

Test system: Site 3 (sand: 93.2%; organic carbon: <0.01%)

Vessel #	Conc., µg/L	Centrate rep 1 dpm/500 µL	Centrate rep 2 dpm/500 µL	Centrate rep 3 dpm/500 µL	Centrate total (7 mL) dpm	Centrate % Recovered	Soil (ext: 1) % Recovered	Soil (ext: 2) % Recovered	Soil total % Recovered	Container dpm 1 mL of 20	Container total dpm	Container % Recovered	Total % Recovered	Kd	Koc
21	–	5	4	5	65	na									
22	–	4	12	0	75	na									
23	40	980	987	994	13818	41.75									
24	40	942	931	919	13029	39.36	31.03	1.01	32.04	182	3640	11.00	82.40	0.81	>8100
25	200	1039	1005	1028	14336	37.83									
26	200	1012	1024	1040	14355	37.88	30.11	1.92	32.03	266	5320	14.04	83.94	0.85	>8500
27	700	1112	1143	1115	15727	40.95									
28	700	1059	1108	1050	15013	39.10	31.97	0.65	32.62	212	4240	11.04	82.76	0.83	>8300
29	2000	1580	1645	1610	22563	52.84									
30	2000	1695	1719	1689	23814	55.77	22.31	1.53	23.84	168	3360	7.87	87.48	0.43	>4300

Vessel #	Conc., µg/L	Soil (ext: 1) rep 1: dpm 1 mL of 20	Soil (ext: 1) rep 2: dpm 1 mL of 20	Soil (ext: 1) rep 3: dpm 1 mL of 20	Soil (ext: 1) total dpm	Soil (ext: 1) adjusted for centrate remaining	Soil (ext: 1) % Recovered	Soil (ext: 2) rep 1: dpm 1 mL of 20	Soil (ext: 2) rep 2: dpm 1 mL of 20	Soil (ext: 2) rep 3: dpm 1 mL of 20	soil (ext: 2) total dpm	Soil (ext: 2) adjusted for extract remaining	Soil (ext: 2) % Recovered
24	40	698	708	693	13993	10271	31.03	51	56	48	1033	334	1.01
26	200	816	837	828	16540	11413	30.11	79	71	83	1553	726	1.92
28	700	884	884	878	17640	12278	31.97	81	85	70	1573	250	0.65
30	2000	917	911	877	18033	9528	22.31	103	92	106	2007	654	1.53

Vessel #	Conc., µg/L	Soil (ext: 3) rep 1: dpm 1 mL of 20	Soil (ext: 3) rep 2: dpm 1 mL of 20	Soil (ext: 3) rep 3: dpm 1 mL of 20	Soil (ext: 3) total dpm	Soil (ext: 3) adjusted for centrate remaining	% Recovered	Soil (ext: 4) rep 1: dpm 1 mL of 20	Soil (ext: 4) rep 2: dpm 1 mL of 20	Soil (ext: 4) rep 3: dpm 1 mL of 20	soil (ext: 4) total dpm	Soil (ext: 4) adjusted for extract remaining	Soil (ext: 4) % Recovered
30	2000	17	19	20	373	173	0.40	4	9	3	107	69	0.16

Vessel #	Conc., µg/L	Centrate % Recovered	Soil total % recovered by extraction	Soil % recovered by combustion	Container % Recovered	Mass Balance %
24	40	39.36	32.60	9.24	11.00	92.2

Table 5: Combustion Data
Study: 96−005

vessel #	cup #	aliquot wgt,mg	total dpm/aliquot	dpm/mg	wet weight total mg	dpm/soil pellet	% 14C	average %14C
3	27	164.1	71	0.43	9000	3870	11.69	
	28	125.2	43	0.34	9000	3060	9.24	
	29	137.6	62	0.45	9000	4050	12.24	
	30	141.8	58	0.41	9000	3690	11.15	
	31	167.2	79	0.47	9000	4230	12.78	
	7	165.2	83	0.50	9000	4500	13.60	
	8	195.9	66	0.34	9000	3060	9.24	
	9	209.9	58	0.28	9000	2520	7.61	
	10	176.9	46	0.26	9000	2340	7.07	
	11	195.8	78	0.40	9000	3600	10.88	10.55
24	22	144.8	96	0.66	9000	5940	17.95	
	23	169.1	69	0.41	9000	3690	11.15	
	24	175.2	41	0.23	9000	2070	6.25	
	25	174.0	40	0.23	9000	2070	6.25	
	26	144.6	24	0.17	9000	1530	4.62	9.24
4	12	227.5	178	0.78	9000	7020	21.21	
	13	198.1	189	0.95	9000	8550	25.83	
	14	190.6	179	0.94	9000	8460	25.56	
	15	162.8	137	0.84	9000	7560	22.84	
	16	196.8	155	0.79	9000	7110	21.48	23.4

Table 6

LS Counts of Radiolabelled Dosing Solutions: Mean ± Std Dev

V 2648G

TSIN	Aliquot (µL)	(dpm)[a]	Mean ± Std Dev	% RSD
Solution 1: for 40 µg/L treatment	20	14494 14246 13909	14216 ± 294	2.1
Solution 2: for 200 µg/L treatment	20	14502 15037 14974	14838 ± 294	2.0
Solution 3: for 700 µg/L treatment	20	14221 15085 15357	14888 ± 593	4.0
Solution 4: for 2,000 µg/L treatment	20	16426 16369 16435 15788	16255 ± 312	1.9
Solution 1: for 40 µg/L treatment	20	13004 13486 13245	13245 ± 241	1.8
Solution 2: for 200 µg/L treatment	20	15320 14943 15235	15166 ± 198	1.3
Solution 3: for 700 µg/L treatment	20	15474 15214 15409	15366 ± 135	0.9
Solution 4: for 2,000 µg/L treatment	20	17161 16913 17176	17083 ± 148	0.9

RSD = Relative Standard Deviation

[a] Counted in Ultima Gold cocktail.

Table 7

Soil Characterization Data

Sample ID	Site 1	Site 2	Site 3
pH	6.1	6.3	6.9
CEC (meq/100g)	0.99	0.75	0.78
O.C. (%)	0.13	0.03	<0.01
O.M. (%)	0.22	0.05	<0.01
WHC (%) 1/3 Bar	2.12	1.34	1.22
WHC (%) 15 Bar	1.03	0.82	0.37
Sand (%)	93.2	93.2	93.2
Silt (%)	4.0	4.0	4.0
Clay (%)	2.8	2.8	2.8
Soil Classification	Sand	Sand	Sand
Bulk Density (g/cc)	1.45	1.51	1.47
Iron Oxide (%)[1]	0.022	0.009	0.009

CEC = cation exchange capacity
O.C. = organic carbon
O.M. = organic matter
WHC = water holding capacity

[1] Represents the % of Fe in soil as free Fe oxides.

APPENDIX A

FREUNDLICH GRAPHS

The following text outlines the steps required to determine K, 1/n, K_d, and K_{oc} for each test system:

1) Average centrate dpm/500 μL is obtained as raw data from the scintillation counter.

2) The dpm/500 μL is normalized to the dpm in the total centrate (7 mL) and converted to the percent ^{14}C recovered in the centrate using the measured activity added to the respective test vessels.

3) Average soil extract dpm/1 mL is obtained as raw data from the scintillation counter.

4) The dpm/1 mL is normalized to the dpm in the total extract (20 mL) and adjusted for the volume of centrate not removed in the centrate transfer. The adjusted value is converted to the percent ^{14}C recovered in the extract. Results of the two soil extractions are presented as % recovered in soil.

5) The % recovered values are used to calculate the concentration of test substance in the liquid and the solid phases.

6) The logarithm (log) of each concentration value is taken and used to graph the Freundlich isotherm by plotting log x/m (log solids) vs log C_e (log solution).

The equation used to describe the Freundlich isotherm follows:

$$\log x/m = \log K + (1/n) \log C_e$$

which is equivalent to the equation $y = a+bx$ as seen on the graphs that follow.

therefore,

parameter a = log K [adsorption coefficient or constant (y-axis intercept)]

parameter b = 1/n [X coefficient: constant describing the degree of nonlinearity of the isotherm (slope)]

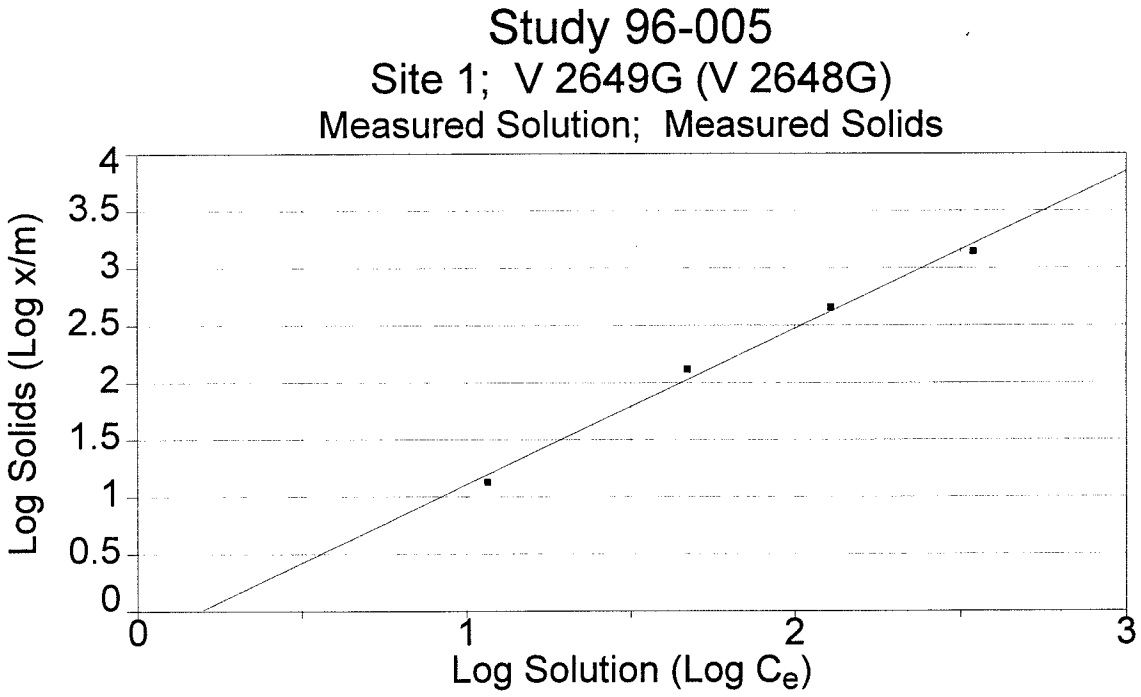
therefore, for reporting purposes

K = antilogarithm of parameter a (log k)

1/n = parameter b

K_d = chemical sorbed divided by chemical in solution at equilibrium

K_{oc} = K_d divided by the fraction organic carbon



Rank 1 Eqn 8001 [UDF 1] y= a+bx (a,b)

r^2 Coef Det	DF Adj r^2	Fit Std Err	F-value
0.9914709923	0.9744129768	0.0978535313	232.49386662

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	-0.26339902	0.172553691	-1.52647569	-0.97886847	0.452070435
b	1.367315991	0.089673298	15.24774956	0.995498357	1.739133625

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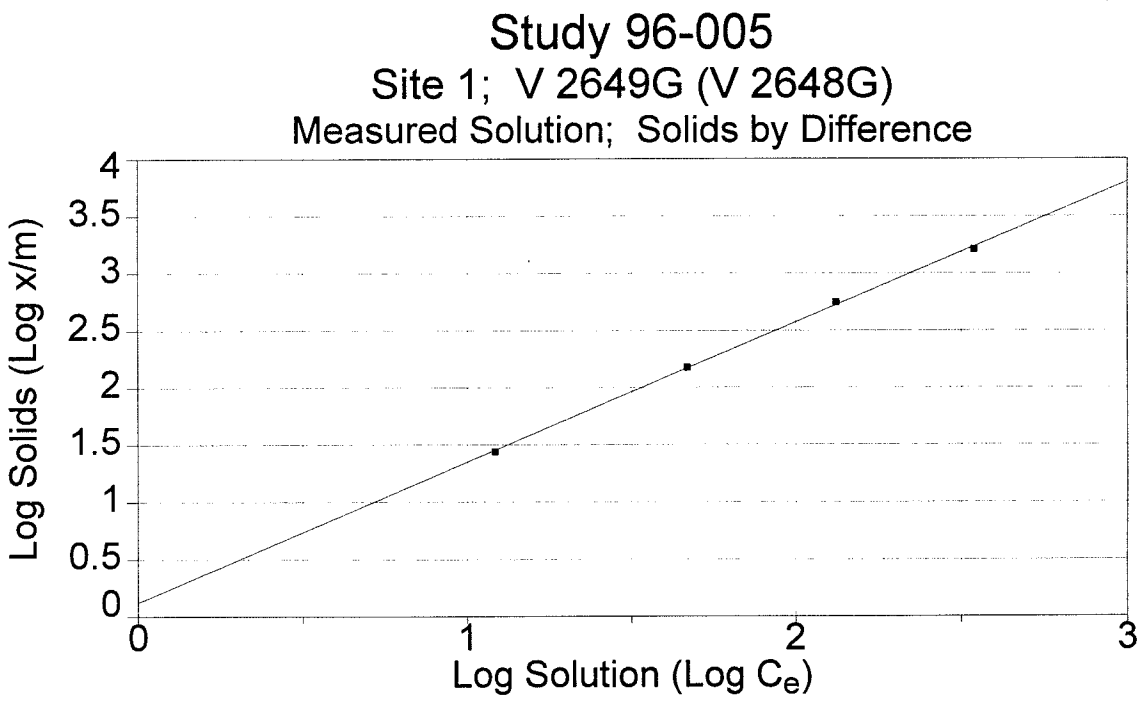

Rank 1 Eqn 8001 [UDF 1] y= a+bx (a,b)

r^2 Coef Det	DF Adj r^2	Fit Std Err	F-value
0.9991397684	0.9974193053	0.0274517233	2322.9553778

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	0.121074072	0.049062131	2.467770354	-0.08235511	0.324503251
b	1.225145871	0.025419521	48.1970474	1.119747425	1.330544317

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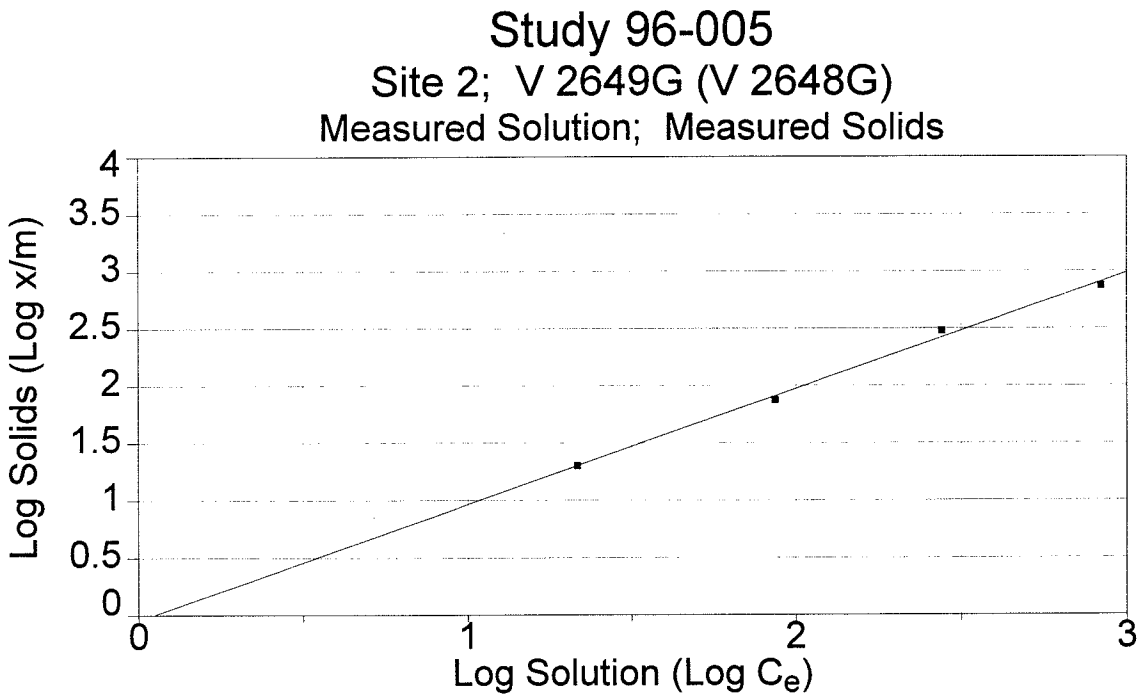

Rank 1 Eqn 8001 [UDF 1] y= a+bx (a,b)

r^2 Coef Det	DF Adj r^2	Fit Std Err	F-value
0.9959056688	0.9877170064	0.054036913	486.48026947

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	-0.04970403	0.1025356	-0.48474897	-0.47485337	0.375445321
b	1.010691912	0.04582328	22.05629789	0.82069217	1.200691654

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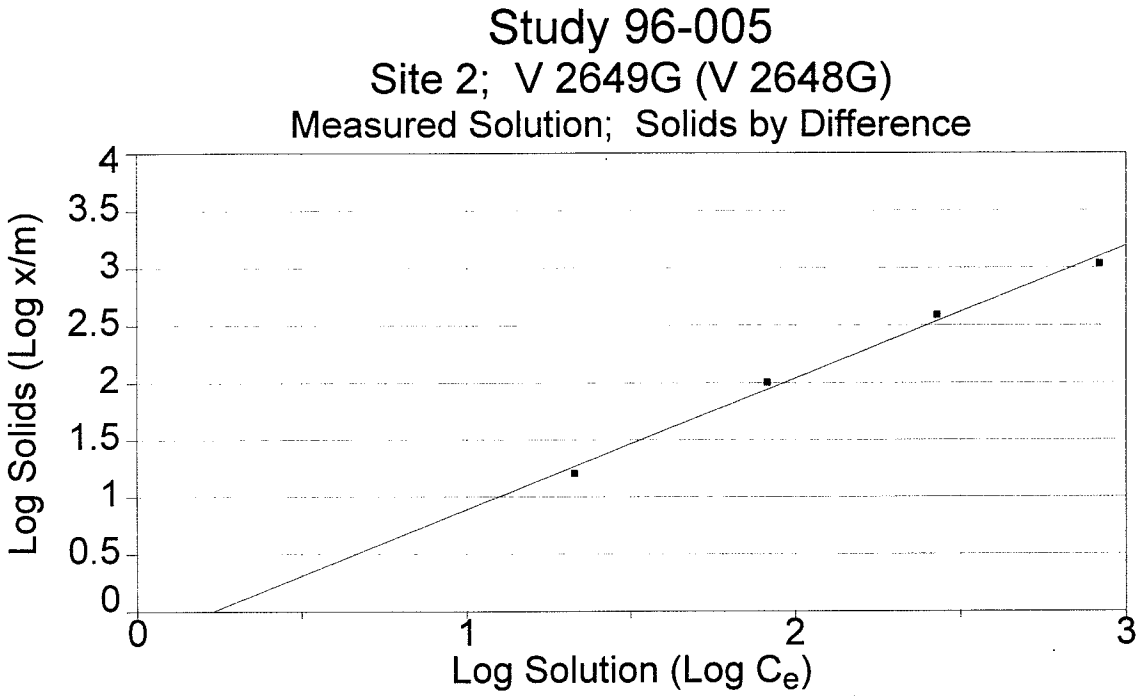

Rank 1 Eqn 8001 [UDF 1] y= a+bx (a,b)

r^2 Coef Det	DF Adj r^2	Fit Std Err	F-value
0.992219691	0.9766590729	0.0853462487	255.05919762

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	-0.2668426	0.160733925	-1.66015107	-0.93330306	0.399617864
b	1.151889692	0.072125759	15.97057287	0.852830444	1.45094894

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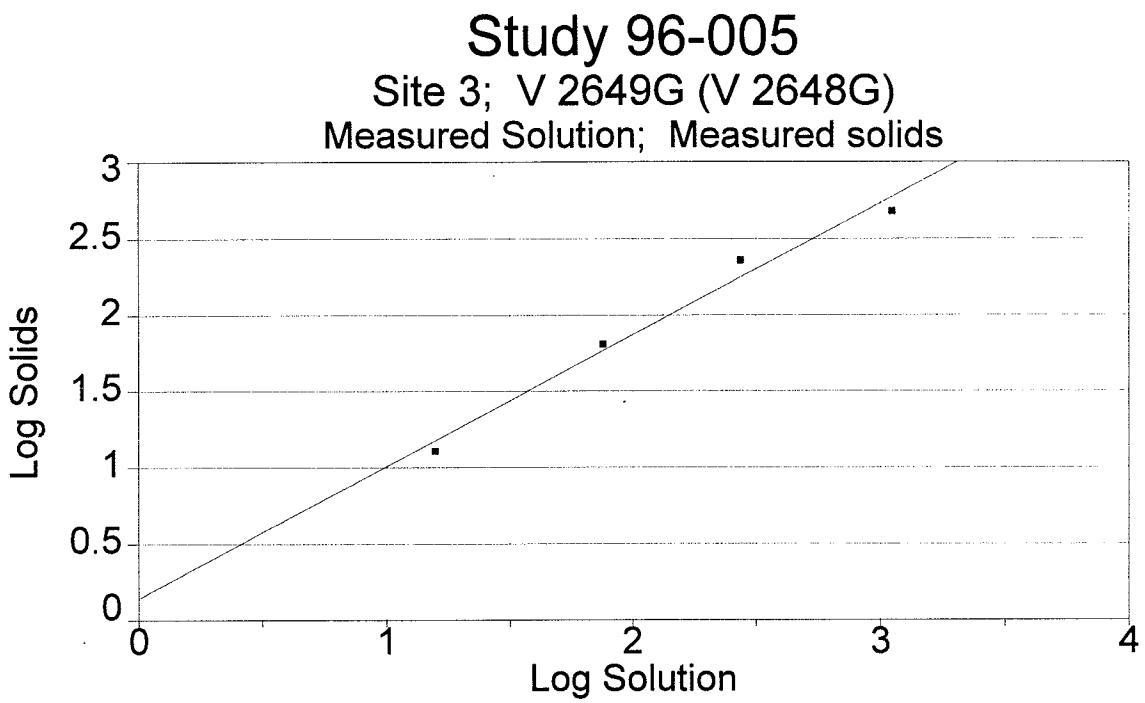

Rank 5 Eqn 1 y=a+bx

r^2 Coef Det	DF Adj r^2	Fit Std Err	F-value
0.9803722734	0.9411168201	0.1181052061	99.896670876

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	0.139780024	0.194103486	0.720131445	-0.6650426	0.944602648
b	0.863473141	0.08639196	9.994832209	0.505261112	1.221685169

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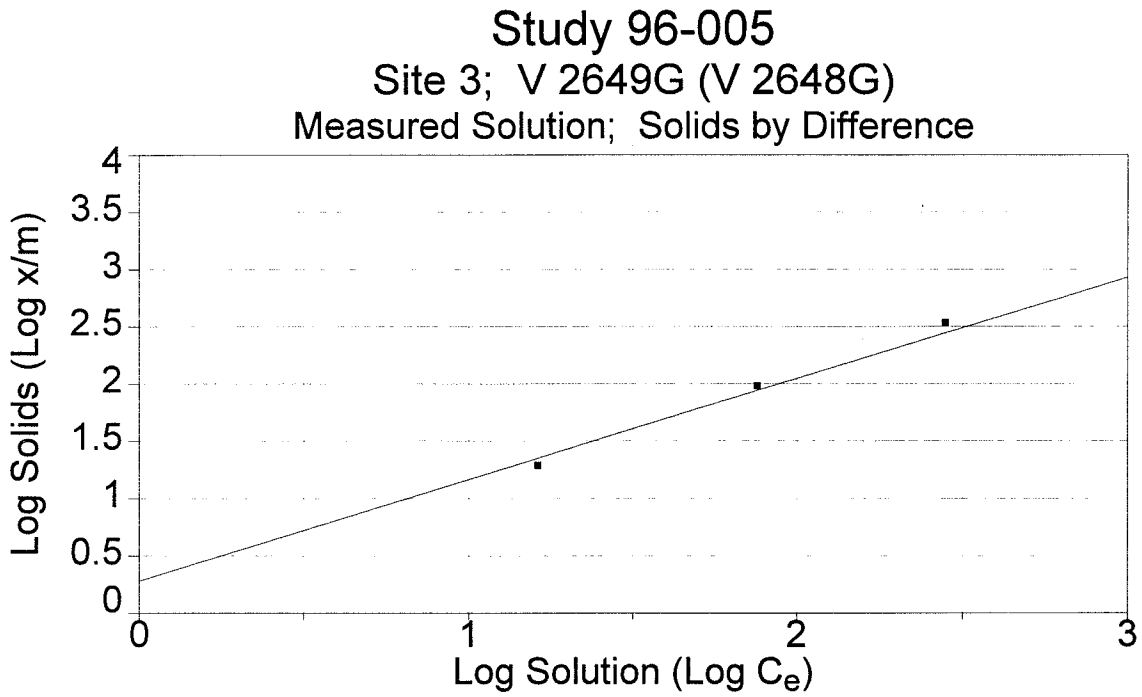

Rank 1 Eqn 8001 [UDF 1] y= a+bx (a,b)

r^2 Coef Det	DF Adj r^2	Fit Std Err	F-value
0.9854210997	0.956263299	0.1027834012	135.18455809

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	0.277658839	0.170760624	1.626012092	-0.43037591	0.985693586
b	0.883450137	0.075983361	11.62688944	0.568395889	1.198504385

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APPENDIX B

PROTOCOL
PROTOCOL AMENDMENT
CHAIN OF CUSTODY: SOIL COLLECTION
TEST SUBSTANCE IDENTIFICATION SHEETS

WESTON Study No. 96-005

PROTOCOL

SPONSOR: The Soap and Detergent Association (SDA)
475 Park Avenue South
New York, NY 10016

LABORATORY: Roy F. Weston, Inc.
Environmental Fate & Effect Laboratory
254 Welsh Pool Road
Lionville, PA 19341-1345

TITLE: Adsorption

1.0 PRINCIPLE OF THE TEST METHOD

A known quantity of test substance is added to a series of test vessels that contain water and soil. The vessels are agitated to achieve equilibrium between adsorbed and dissolved test substance. The water and soil are separated by centrifugation and the amount of test substance remaining in the water is determined by liquid scintillation counting (LSC).

2.0 OBJECTIVE

The objective of the study is to determine the partitioning of the test substance between soil and water.

3.0 JUSTIFICATION FOR TEST SYSTEM

This test method is based on the procedures outlined in the OECD Guidelines for Testing of Chemicals, Method 106 (1981).

4.0 GOOD LABORATORY PRACTICE STANDARDS

The test will be conducted in accordance with United States Environmental Protection Agency (USEPA) Good Laboratory Practice (GLP) Regulations (40 CFR Part 792). The Sponsor accepts full responsibility for appropriate characterization and stability verification of the test substance in addition to collection (with appropriate documentation) of the test system (soil). Stock solution(s) of the test substance will be analyzed by Roy F. Weston, Inc., (WESTON®) by LSC, as noted in this protocol, but the stock solution(s) will not be analyzed for stability or uniformity.

5.0 TEST SUBSTANCE(S)

5.1 RADIOLABELLED

TSIN[1]: V 2648G (Radioactive C_{12} LAS)

Specific Activity (μCi/mg): 77 μCi/mg

Radiochemical Purity (%): >92%

Mass concentration, mg/mL (if applicable): 1.2 mg/mL

Radiochemical concentration, μCi/mL (if applicable): 93.3 μCi/mL

Sample form: methanol solution (clear)

Solubility in water: ~35%

Storage Conditions: keep in the dark, refrigerated (~4°C), closed container

Safe Handling Precautions: see MSDS

Expiration Date: 1/1/2000

5.2 UNLABELLED

TSIN: V 2649G (non radioactive C_{12} LAS)

Percent active: 54.08

Sample form: white slurry

Solubility in water: ~35%

Storage Conditions: keep in the dark, refrigerated (~4°C), closed container

Safe Handling Precautions: see MSDS

Expiration Date: 1/1/2000

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[1] TSIN = Test Substance Identification Number/Name.

6.0 SOLVENT AND STOCK SOLUTION PREPARATION

Prepare a stock solution of the test substance in deionized (ASTM Type II[1]) water. Determine the concentration of the test substance in the stock solution by LSC.

7.0 TEST PROCEDURE

7.1 TEST SYSTEM

The test system was provided by the Sponsor as follows (see WESTON Study 96-001):

Three samples were collected from each of three sites within the septic tank discharge plume. Site #1 was directly below the trench (½ - 1 foot below the gravel), in the infiltration zone (in the biomat), in what would historically be the unsaturated zone. Site #2 was directly below Site #1 and in the saturated zone at approximately 4 foot depth. Site #3 was slightly down gradient of and in between well points TW-4 and TW-5 in the saturated zone at 4 foot depth. Samples were shipped overnight to WESTON.

The soils were sieved through a 2 mm screen. Samples of each location were composited and identified as Site 1, Site 2, and Site 3, respectively. The percent moisture content of each "site" sample was determined. An aliquot of each sample was shipped to A & L Great Lakes Laboratories, Fort Wayne, Indiana for the following analyses: pH, cation exchange capacity, organic matter, water holding capacity, bulk density, textural classification.

The soils were stored under refrigeration. Conduct the adsorption study using field-moist soil samples added on a dry weight basis. Use a ratio of one part solvent to one part soil (7 mL upgradient groundwater/7 g soil). Conduct the tests in Teflon centrifuge tubes using upgradient groundwater from the sampling site as the solvent.

Conduct the study at 20 $\pm$ 2 °C.

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[1] "Standard Specification for Reagent Water," ASTM Committee D-19 on Water, ASTM Designation D1193-77, (Reapproved 1983).

8.0 DEFINITIVE ISOTHERM EXPERIMENT

Soil Type	No. Replicates	Test Substance Concentration
None	2	40 μg/L
1	2	40 μg/L
2	2	40 μg/L
3	2	40 μg/L
None	2	200 μg/L
1	2	200 μg/L
2	2	200 μg/L
3	2	200 μg/L
None	2	700 μg/L
1	2	700 μg/L
2	2	700 μg/L
3	2	700 μg/L
None	2	2000 μg/L
1	2	2000 μg/L
2	2	2000 μg/L
3	2	2000 μg/L
1	2	no test sub
2	2	no test sub
3	2	no test sub

Set up the test vessels according to the test design given above using upgradient groundwater as the aqueous phase (record the amount of groundwater and soil added to each vessel). Place the test vessels on a shaker table and agitate @ ~180 rpm for 3 hours. Agitation should be sufficient to maintain the soil in suspension. At the end of this time period separate the aqueous phase by centrifugation @ ~5600 x g for ~ 40 minutes. Assay triplicate aliquots of the centrates by LSC in Ultima Gold™ cocktail (Packard catalog # 6013329).

For one of each of the test substance concentrations from each soil site, extract the soil in one of the duplicate test vessels to determine the mass of test substance present in the soil after the experiment is terminated. Determine the mass balance of the test substance in these vessels. The extraction procedure follows:

- Transfer the solids to clean centrifuge tubes.
- Extract the solids two times with 20 mL MeOH. The solvent is added to the tubes which are vortexed vigorously for ~20 minutes.
- Centrifuge the test tubes @ ~5600 g for ~40 minutes .
- Assay triplicate 1 mL aliquots of the centrate in Ultima Gold™ cocktail by LSC.

Extraction of the original container wall may be performed at the discretion of the Study Director.

9.0 CALCULATIONS

For the isotherm experiment, prepare a graph of the Freundlich isotherm by plotting log x/m vs log C_e. The Freundlich isotherm is described by the following equation:

$$\text{Log } x/m = \log K + (1/n) \log C_e$$

where: x = the amount of test substance sorbed
m = the amount of sorbent (i.e. soil)
C_e = equilibrium concentration of the test substance in solution
K = adsorption coefficient
n = a constant describing the degree of nonlinearity of the isotherm

Calculate the value of the constants K and 1/n. When the constant n in the Freundlich isotherm experiment is equal to 1, the Freundlich adsorption coefficient, K, can be used as a distribution adsorption coefficient, K_d. The distribution adsorption coefficient is described by the equation:

$$K_d = \frac{x/m}{C_e}$$

If the value of n does not equal 1, calculate K_d from the above equation.

Calculate the K_{oc} constant for each soil type. The K_{oc} constant relates the amount of material sorbed to the soil organic carbon content and is described by the equation:

$$K_{oc} = \frac{K_d}{\text{fraction organic carbon}}$$

10.0 RECORDS TO BE MAINTAINED

All records necessary to reconstruct the study and demonstrate adherence to the Protocol are maintained.

11.0 PROTOCOL CHANGES

If changes in the approved protocol become necessary, verbal agreement should be made between the Study Director and Sponsor's Principal Investigator. As soon as practical thereafter, these changes and the reasons for them should be put in writing, approved by both persons, and attached as amendments to the protocol.

12.0 REPORTING

The final report should conform to all requirements outlined in Section 792.185 Sub-part J, Good Laboratory Practice Standards. It is to be signed and dated by the Study Director, Quality Assurance Officer, and Laboratory Manager, and is to include but is not limited to the following:

1. Identification of test substance by test substance identification number (TSIN), percent active, color, form, specific activity, and date received.

2. Soil characterization data.

3. a. Graphs of log x/m vs C_e for each soil type tested.

   b. K, K_d, 1/n, K_{oc}.

4. Reference to the protocol (title, P.I., date) and amendment(s) if made, test methods, and any analytical or standard operating procedures used.

5. Any protocol deviations and their scientific implications.

6. Reference to laboratory notebook or other files containing raw or processed data.

7. Dates test was conducted and persons responsible for the execution of the study.

8. Laboratory study number

9. Description of the test method and all calculations.

10. Temperature range recorded during test period.

11. Description of the quality assurance methods used to insure the quality of the data.

APPROVED

DREW McAVOY — MARCH 1, 1996 — (513) 627-5570
Alternate Principal Investigator — Date — Phone

ALVARO J. DE CARVALHO
Alvaro J de Carvalho — MARCH 1, 1996 — (212) 725-1262
Sponsor's Principal Investigator — Date — Phone

TO BE COMPLETED BY LABORATORY

Laboratory Study No. 96-005

Estimated Experimental Start Date 3/13/96 Defined as: Spike test substance

Estimated Experimental Termination Date 3/14/96

Estimated Reporting Date 8/13/96

Date Test Substance(s) Received 12/6/95

APPROVED

Kay H Mercke — 3/12/96 — 1-610 7016 170
Study Director — Date — Phone

Sponsor: The Soap and Detergent Association (SDA), 475 Park Avenue South, New York, NY 10016

Laboratory: Roy F. Weston, Inc., 254 Welsh Pool Road, Lionville, PA 19341-1345

PROTOCOL AMENDMENT № 1

Study №: 96-005

Study Title: Adsorption

Test Substance: V 2649G (V 2648G)

Protocol Amendment:

1) Per a conversation with SDA members on 2 February 1996:

Purity of V 2648G (dosing solution prepared for Study 96-001 from the primary stock solution provided by the Sponsor) determined using RAD-TLC. The mobile phase used was chloroform/methanol/water/formic acid (80:25:3:1). This was a qualitative procedure to determine whether the parent material was intact.

2) Protocol Section 8.0: Definitive Isotherm Experiment, page 5

The following procedures (beyond the scope of the protocol) were performed:

- 20 mL MeOH was added to the original centrifuge tubes from each treatment extracted as specified in the protocol; the tubes were vortexed for ~20 minutes; triplicate one milliliter aliquots of the MeOH were cocktailed with Ultima Gold and assayed by liquid scintillation counting (LSC).

- Test vessel 3 (Site 1 at 40 μg/L) was submitted to the extraction procedure to confirm the results obtained from test vessel 4.

- Test vessel 30 (Site 3 at 2,000 μg/L) was extracted a third and fourth time to determine if the MeOH extraction procedure was efficient in removing the sorbed test substance from the solids.

- Aliquots of the solids pellet from test vessels 3, 4, and 24 were combusted to determine if any test substance remained in the soil following extraction with MeOH.

Protocol Amendment
(continued)

3) Protocol Section 8.0: Definitive Isotherm Experiment, page 5

Container rinses were conducted on the test substance control vessels.

Reason for Protocol Amendment:

1) Available purity data was from 1989.

2) To improve the mass balance percents.

3) To determine if test substance sorbed to the Teflon centrifuge tubes.

Kay H Marks — Study Director — Date 5-13-96

Alvaro J De Carvalho — Sponsor Approval — Date 5/20/96

AYRES ASSOCIATES

Engineers/Environmental Scientists
3901 Coconut Palm Drive, Suite 100
Tampa, FL 33619
Telephone: (813) 628-0742

CHAIN OF CUSTODY RECORD

PROJECT CODE: 00-9155.96 | SAMPLED BY: C&H/DLA

PROJECT NAME: SJDA St Johns Co.

SAMPLE FIELD ID	LAB ID	COLLECTION DATE/TIME	SAMPLE MATERIAL	NUMBER OF CONTAINERS	FIELD READINGS PH	COND	TEMP
BW1-5		1/16/96 855	H2O	1	7.23	897	70.2
TW21-6		1/16/96 1355	H2O	1	7.00	933	68.8
SC1-A		1/16/96 1200	Soil	1			
SC1-B		1/16/96 0915	Soil	1			
SC1-C		1/16/96 1210	Soil	1			
SC1-D		1/16/96 1215	Soil	1			
SC2-A		1/16/96 1000	Soil	1			
SC2-B		1/16/96 1025	Soil	1			
SC2-C		1/16/96 1240	Soil	1			
SC2-D		1/16/96 1250	Soil	1			
SC3-A		1/16/96 1056	Soil	1			
SC3-B		1/16/96 1030	Soil	1			
SC3-C		1/16/96 1110	Soil	1			
SC3-D		1/16/96 1125	Soil	1			

ANALYSES REQUESTED

COMMENTS

Background Surface Soil
H2O Sample Below Trench

SC1 A-D = 1 soil Sampling Location
composite these 4 samples
For your soil Needs (Gravel - 1' Below Trench)

SC2 A-D = 1 soil Sampling Location
Composite these 4 samples
For your Soil Needs (4' Below trench)

SC3A-D: 1 soil Sampling Location
Composite these 4 Samples
For your Soil Needs (Down Gradient 20' and 4' Below Ground Surface)

RELINQUISHED BY (SIGNATURE)	DATE/TIME	CARRIER	RECEIVED BY	DATE/TIME
	1-16-96 1500	FED EX		
RELINQUISHED BY (SIGNATURE)	DATE/TIME	CARRIER	RECEIVED BY	DATE/TIME
				1/17/96 10:15

REMARKS

PROJECT FILE COPY: WHITE CHAIN OF CUSTODY COPY: YELLOW LABORATORY FILE COPY: PINK LABORATORY CHECK IN COPY: GOLD

Laboratory : Please Return Gold Copy When Samples Are Checked In.

Vista Chemical Company

12024 Vista Parke Drive
Austin, Texas 78726-4050
(512) 331-2500

P.O. Box 200135
Austin, Texas 78720-0135
Fax (512) 331-2387

W.O. 05821-007-001

VISTA

December 4, 1995

Jon Doi, Ph.D.
Laboratory Manager
Environmental Fate and Effects Laboratory
Roy F. Weston, Inc.
254 Welsh Pool Road
Lionville, PA 19341-1345

Dear Jon:

We are sending you today by overnight courier the [^{14}C]- C_{12} LAS and "cold" LAS samples that are needed for the SDA biodegradation and adsorption coefficient studies. This letter contains the sample information that you requested in your September 28, 1995 letter to Al DeCarvalho.

95-0140
Rec'd 12/6/95 - A

RADIOACTIVE C_{12} LAS

1. Test Substance Identification Number (TSIN) - V 2648 G
2. Test Substance Name - Dodecylbenzene sulfonic acid, sodium salt [Ring - ^{14}C (U)]
3. Chemical Abstract Service (CAS) number - 25155-30-0
4. Molecular Formula - $C_{18}H_{29}SO_3Na$
5. Molecular Weight - 348 mg/mmole
6. Lot or Batch number - NEN Research Products - 2599-075(8/3/89)
7. Material Data Safety Data Sheet (MSDS) - Attached to letter - This MSDS is for Vista C-550 Slurry which contains C_{12} LAS homolog. All of the precautions required for handling C-550 slurry apply to handling of C_{12} LAS homolog.
8. Storage Conditions - Keep in the dark, refrigerated (4°C), in a well-closed container
9. Expiration Date - January 1, 2000. Our experience is that LAS solutions in methanol will store indefinitely if kept in the dark, in well-closed containers and at cool temperatures.
10. Solubility in water - Miscible
11. Color / Form - Dissolved in HPLC grade methanol/ clear solution

12. Percent chemical purity - Not available - TLC analysis by NEN Research Products (8/3/89) demonstrated that the radiochemical purity is greater than 97.5%. A copy of the analysis data provided by NEN Research Products is attached to this letter.
13. Percent active - 60.49% = 1.2117 ug LAS / (1.2117 ug LAS + 0.7914 ug methanol) x 100
14. Certificate of Analysis - See item 12.
15. Theoretical total organic carbon (TOC) content, in mg carbon/mg active = .6207 = 216 mg C/ 348 mg LAS.
16. Theoretical CO_2 (TCO_2), in mg CO_2/ mg active = 2.276 mg CO_2/ mg LAS = 18 C x 44 mg CO_2/ 348 mg LAS = 792 mg CO_2/ 348 mg LAS.
17. Quantities sent - volume of sample = 3917 uL ; mass of LAS in sample = 4746 ug ; radioactivity of LAS in sample = 365.5 u Ci

95-0139 216 195 21
Rec'd

NON RADIOACTIVE C_{12} LAS

1. Test Substance Identification Number (TSIN) - V 2649G
2. Test Substance Name - Dodecylbenzene sulfonic acid, sodium salt
3. Chemical Abstract Service (CAS) number - 25155 - 30-0
4. Molecular Formula - $C_{18}H_{29}SO_3Na$
5. Molecular Weight - 348 mg/mmole
6. Lot or Batch number VO - 382 - 167- 2
7. Material Data Safety Data Sheet (MSDS) - Attached to Letter - This MSDS is for Vista C-550 Slurry which contains C_{12} LAS homolog. All of the precautions required for handling Vista C-550 Slurry apply to handling the C_{12} LAS homolog.
8. Storage Conditions - Keep in the dark, in a well-closed container and at cool (4°C) temperatures
9. Expiration Date - January 1, 2000. Our experience is that LAS slurries (kept in well - closed bottles, in the dark , and at cool temperatures) will keep indefinitely without decomposition.
10. Solubility in water - Miscible
11. Color/Form - White slurry (solvent is water)
12. Percent chemical purity - Current desulfonation/ GC analysis shows it to be 95.81 % pure LAS and 100% of the LAS is C_{12} LAS homolog. GC analysis of LAB before sulfonation showed it to be > 99% pure LAS. The current analysis sheet is attached to this letter.
13. Percent active - 54.08 %
14. Certificate of Analysis - See item 12.
15. Theoretical total organic carbon (TOC) content is .6207 mg C/ mg LAS
16. Theoretical CO_2 (TCO_2) is 2.276 mg CO_2/mg LAS
17. Quantities sent - mass of sample = 92.75 grams of slurry; 50.158 grams of LAS

Please find attached to this letter all the appropriate paper work for shipment of the radioactive sample and the analyses mentioned above.

If there are any questions, please call at 512-331-2461

Best Regards,

Allen M. Nielsen

Allen M. Nielsen
Senior Research Microbiologist
Research and Development
Vista Chemical Company

cc:
Vista - L.N. Britton, M.F. Cox, R.B. Martin
SDA - Al DeCarvalho

Preparation of Dodecylbenzenesulfonic acid [ring-^{14}C(U)], sodium salt
Lot No. 2599-075

Benzene [^{14}C(U)] was reacted with 1-dodecene and aluminum chloride to produce dodecylbenzene [ring-^{14}C(U)]. The product was reacted with fuming sulfuric acid to produce dodecylbenzenesulfonic acid [ring-^{14}C(U)]. The acid was then was converted to its sodium salt with sodium hydroxide and purified by column chromatography.

VISTA CHEMICAL COMPANY

Research & Development Department
Analysis Report

20-Nov-95

To A. NIELSEN Project Number 6940-05

Location _____ Reference V0926;60

Sample LAS SLURRY

Notes * % ACTIVE= MEQ/G*MW LAS/10 ** DESULF SENT FOR GC 11-20-95

ANALYSIS CODES:	ACTIVE	DESULF
METHOD (NUMBER OR TECHNIQUE):	MBAS	
ASSAY AND UNITS:	MEQ/G*	**

Analytical Number	Sample Number	= V 2649 G			
V0906-49-1		1.554			
		54.0792 % Active			

alyst (MEB) Mark Boss Approved _____

Alkylate Distribution by GC/MS

Reported by Charles Beall

SAMPLE AMN906-49-1 - V2649G
FILE B9630-2

POSITIONAL % TOTALS*		HOMOLOG DISTRIBUTION (LINEAR ONLY)*	
6,7 PHENYL	15.62	PRE C10	0.00
5 PHENYL	15.72	C10	0.00
4 PHENYL	15.96	C11	0.00
3 PHENYL	19.67	C12	100.00
2 PHENYL	33.03	C13	0.00
		C14	0.00
	100	POST C13	NA
		POST C14	NA
*NORMALIZED TO 100%			100

AVERAGE MOLECULAR WEIGHT	LINEARITY
246.00	95.81

PARAFFINS	0.00
PRE C10	0.00
POST C13	0.00
POST C14	0.00
BRANCHING AND TETRAL	4.19

AREAS

	C10	C11	C12	C13	C14
6,7 PHENYL		0	782509	0	0
5 PHENYL	0	0	787341	0	0
4 PHENYL	0	0	799255	0	0
3 PHENYL	0	0	985083	0	0
2 PHENYL	0	0	1654169	0	0
	0	0	5008357	0	0

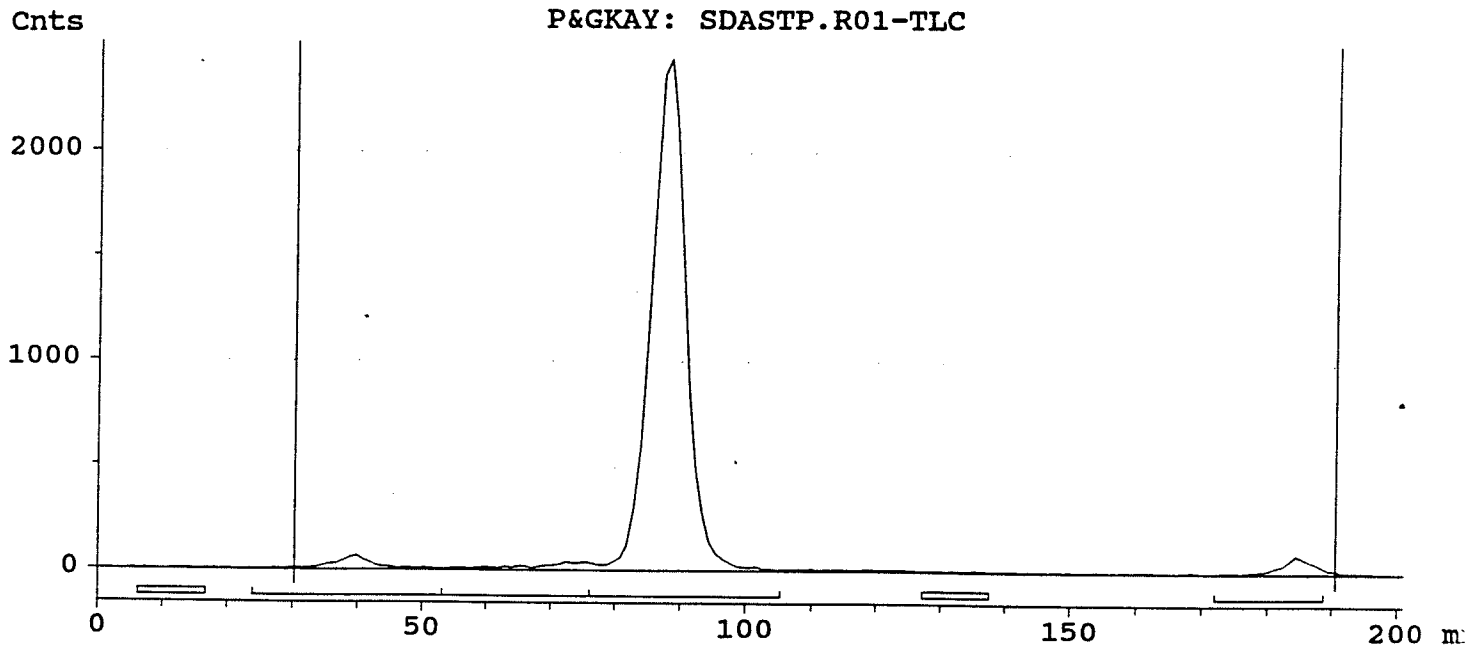
PARAFFINS	0		
PRE C10	0		
ALKYLATE	5227520		
POST C13	0		
POST C14	0		
GRAND TOTAL AREA	5227520	LESS PARAFFINS	5227520

AREA %

	C10	C11	C12	C13	C14	
6,7 PHENYL		0.00	14.97	0.00	0.00	
5 PHENYL	0.00	0.00	15.06	0.00	0.00	
4 PHENYL	0.00	0.00	15.29	0.00	0.00	
3 PHENYL	0.00	0.00	18.84	0.00	0.00	
2 PHENYL	0.00	0.00	31.64	0.00	0.00	
						TOTAL
TOTALS	0.00	0.00	95.81	0.00	0.00	95.81

APPENDIX C

PURITY DATA



tegrals: SDASTP.R01

Channel: TLC Detector:

Name	Start (mm)	End (mm)	Max (mm)	RF	Region Counts	Region CPM	% of Total	% of ROIs
Bkg 1	6.2	16.7	11.5	-0.118				
ROI 1	24.0	53.2	39.6	0.059	425	447.2	2.68	2.69
ROI 2	53.2	76.1	75.1	0.281	356	374.9	2.25	2.26
ROI 3	76.1	105.3	87.6	0.359	14553	15318.8	91.75	92.19
Bkg 2	127.2	137.7	132.5	0.641				
ROI 4	172.1	188.8	183.6	0.961	451	474.7	2.84	2.86
					15785	16615.6	99.51	100.00

Total Area = 15862 Counts = 16696.8 CPM
Bkg Area = 163 Counts = 171.6 CPM
Unallocated = 77 Counts (0.49%)

Study 96-001
Low concentration dosing solution prepared on 1/18/96 50 ug/kg planned to be dosed on 1/19/96 assayed.
REJ
JMW

Trace Parameters: SDASTP.R01 TLC

Trace Display Smoothing	: 0 chan		
Trace Display Shift	: 0.0 mm		
Trace Display Factor	: 1.000		
Channel Factor	: 1.000		
PW½HT Factor	: 6.3 mm	Area Reject	: 0
Sensitivity	: 100	Height Reject	: 0
Bkg Length	: 10.4 mm	Delay	: 0.0 mm
Smoothing	: 3 point moving average.		

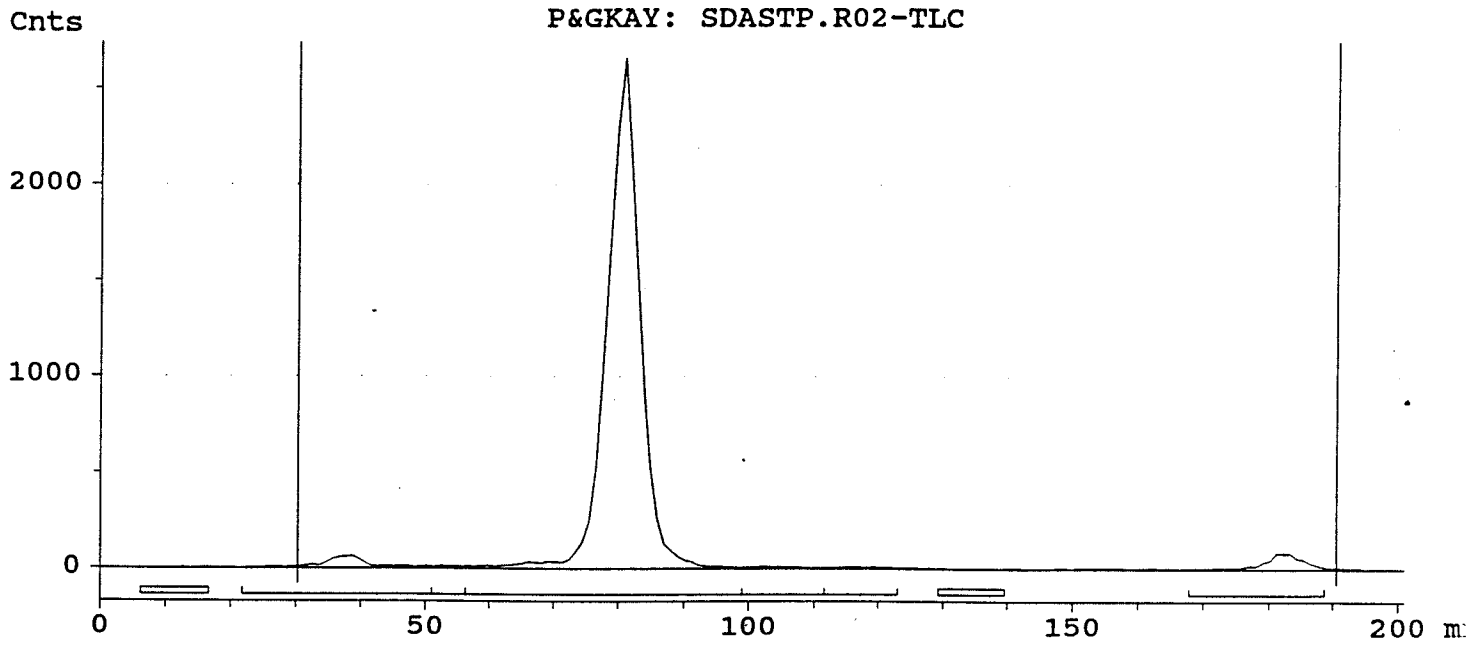
Comments: SDASTP.R01

Analysed : Wed Feb 07 17:44:13 1996

Instrument Parameters: SDASTP.R01

Origin : 30.2 mm Front : 189.8 mm

Collimator type	: Hi Effic	Width: 10 mm	
Resolution	: 256 chan	(0.1043 cm/chan)	
Lane Position	: 1	Run Time	: 1.0 min
Electronic res.	: Normal	Amp. Range	: 0 - 2047
Plate position	: Tab 1		



tegrals: SDASTP.R02

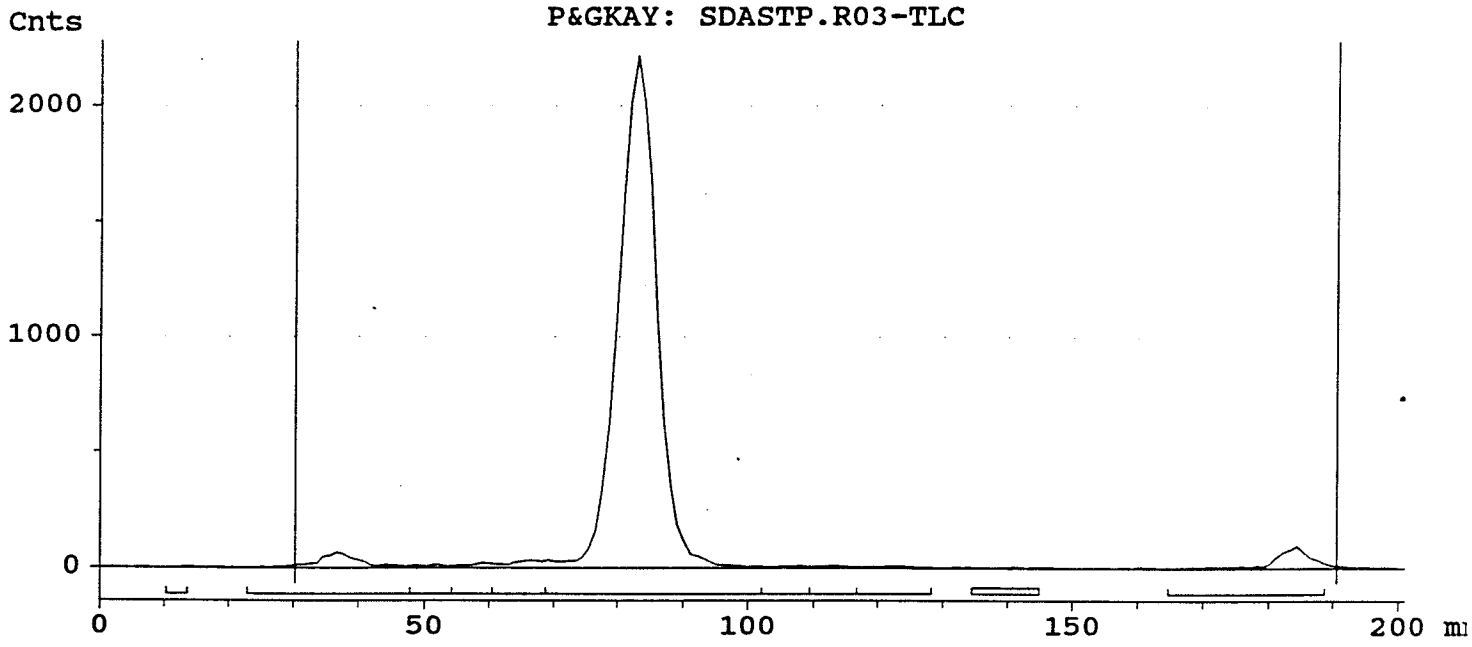
Channel: TLC Detector:

Name		Start (mm)	End (mm)	Max (mm)	RF	Region Counts	Region CPM	% of Total	% of ROIs
Bkg	1	6.2	16.7	11.5	-0.118				
ROI	1	21.9	51.1	38.6	0.052	457	517.4	2.86	2.87
ROI	2	51.1	56.3	52.1	0.137	42	47.5	0.26	0.26
ROI	3	56.3	99.1	80.3	0.314	14791	16744.9	92.63	92.77
ROI	4	99.1	111.6	102.2	0.451	63	71.6	0.40	0.40
ROI	5	111.6	123.1	115.8	0.536	42	47.8	0.26	0.27
Bkg	2	129.3	139.8	134.5	0.654				
ROI	6	167.9	188.8	180.4	0.941	548	619.8	3.43	3.43
						15943	18049.1	99.84	100.00

Total Area = 15968 Counts = 18077.4 CPM
Bkg Area = 55 Counts = 61.8 CPM
Unallocated = 25 Counts (0.16%)

Rep 2
KM

Method: P&GKAY File: SDASTP.R03



Integrals: SDASTP.R03

Channel: TLC Detector:

Name	Start (mm)	End (mm)	Max (mm)	RF	Region Counts	Region CPM	% of Total	% of ROIs
ROI 1	10.4	13.5	12.5	-0.111	-1	-1.4	-0.01	-0.01
ROI 2	22.9	48.0	36.5	0.039	425	463.6	2.68	2.68
ROI 3	48.0	54.2	52.1	0.137	49	52.9	0.31	0.31
ROI 4	54.2	60.5	58.4	0.176	74	80.2	0.46	0.46
ROI 5	60.5	68.8	66.7	0.229	165	180.0	1.04	1.04
ROI 6	68.8	102.2	82.4	0.327	14532	15853.1	91.77	91.70
ROI 7	102.2	109.5	107.4	0.484	30	32.5	0.19	0.19
ROI 8	109.5	116.8	112.6	0.516	34	36.8	0.21	0.21
ROI 9	116.8	128.3	121.0	0.569	30	32.5	0.19	0.19
Bkg 1	134.5	145.0	139.8	0.686				
ROI 10	164.8	188.8	183.6	0.961	511	557.2	3.23	3.22
					15847	17287.4	100.07	100.00

Total Area = 15835 Counts = 17274.8 CPM
Bkg Area = 160 Counts = 174.3 CPM
Unallocated = -12 Counts (-0.07%)

Rep 3

APPENDIX D

COPIES OF THE SOIL CLASSIFICATION REPORT

A & L GREAT LAKES LABORATORIES, INC.

3505 Conestoga Drive • Fort Wayne, Indiana 46808-4413 • Phone (219)483-4759 • FAX (219)483-5274

GLP SOIL CHARACTERIZATION

Report Number: F96031-016
Date of Report: 02/26/96

Protocol Number: 96-001

ROY F. WESTON , INC.
KAY H. MARKS
ENVIRONMENTAL FATE AND EFFECT LABORATORY
254 WELSH POOL ROAD
LIONVILLE PA 19341-1345

Sample ID:	A&L Contol	Site 1	Site 2	Site 3
Lab Number:	50720	50721	50722	50723
pH	6.6	6.1	6.3	6.9
CEC (meq/100g)	7.62	0.99	0.75	0.78
O.C. (%)	1.11	0.13	0.03	<0.01
O.M. (%)	1.90	0.22	0.05	<0.01
WHC (%) 1/3 Bar	18.03	2.12	1.34	1.22
WHC (%) 15 Bar	6.59	1.03	0.82	0.37
Sand (%)	45.2	93.2	93.2	93.2
Silt (%)	32.0	4.0	4.0	4.0
Clay (%)	22.8	2.8	2.8	2.8
Soil Classification	Loam	Sand	Sand	Sand
Bulk Density (g/cc)	1.52	1.45	1.51	1.47

Verified By: Kaylene M. Smith Date: 2-26-96

A & L GREAT LAKES LABORATORIES, INC.

3505 Conestoga Drive • Fort Wayne, Indiana 46808-4413 • Phone (219)483-4759 • FAX (219)483-5274

DUPLICATE ANALYSIS

Report Number: F96031-016
Date of Report: 02/26/96

Protocol Number: 96-001

ROY F. WESTON , INC.
KAY H. MARKS
ENVIRONMENTAL FATE AND EFFECT LABORATORY
254 WELSH POOL ROAD
LIONVILLE PA 19341-1345

	Sample I.D.	Original Analysis	Duplicate Analysis
pH	SITE 1	6.1	6.1
CEC (meq/100g)	SITE 3	0.78	0.71
O.C. (%)	SITE 2	0.03	0.06
O.M. (%)	SITE 2	0.05	0.11
WHC (%) 1/3 Bar	SITE 1	2.12	2.41
WHC (%) 15 Bar	SITE 3	0.37	0.38
Sand (%)	SITE 2	93.2	93.2
Silt (%)	SITE 2	4.0	4.0
Clay (%)	SITE 2	2.8	2.8
Soil Classification	SITE 2	Sand	Sand
Bulk Density (g/cc)	SITE 3	1.47	1.48

Verified By: Kay Jeane M. Smith Date: 2.27.96

A & L CONTROL DATA

Parameter	Control Value	Range
pH	6.6	6.5 - 6.7
CEC (meq/100g)	8.27	7.27 - 9.27
O.M. (%)	1.90	1.70 - 2.10
WHC (%) @ 1/3 Bar	18.15	15.15 - 21.15
WHC (%) @ 15 bar	6.62	5.62 - 7.62
Sand (%)	44.0	41.8 - 46.2
Silt (%)	31.1	29.1 - 33.1
Clay (%)	22.2	20.2 - 24.2
Bulk Density (g/cc)	1.51	1.43 - 1.59

Control sample values obtained fell within allowable variation established in analytical SOP GEN-0-001.

Runs Accepted:

Kaylene M. Smith

Kaylene M.Smith
Study Coordinator

2-26-90
Date

METHOD IDENTIFICATION

Parameter	A & L SOP Number
Acidity (pH)	AGL-10-001
Cation Exchange Capacity (CEC)	AGL-10-002
Organic Matter (OM) / Organic Carbon (OC)	AGL-10-003
Water Holding Capacity (1/3 & 15 Bar)	AGL-10-004
Particle Size Distribution	AGL-10-011
Bulk Density	AGL-10-006

SOIL pH

SOP AGL-10-001 uses a 1:1, soil to water, mixture. The mixture is stirred and allowed to stand for a period of time. After soaking, the sample is stirred again just before reading the pH. The pH is measured with an electronic meter and electrodes.

Reference: "Recommended Soil Test Procedures for the North Central Region", Number 499 (Revised), pp. 5-6, 1980. North Dakota State University.

CATION EXCHANGE CAPACITY (CEC)

SOP AGL-10-002 involves the saturation of the cation exchange sites present in the soil sample with ammonium ions obtained from a solution of ammonium acetate. After saturation, the excess ammonium acetate is washed from the soil using several portions of a 70% methanol solution. The washed soil is then mixed with an acidified 1N KCl solution to drive the ammonium ions off the exchange sites. In a closed environment, the soil mixture is mixed with MgO to raise the pH. The now basic solution is steam distilled and the ammonia is trapped in an acid catch solution and titrated to determine the CEC.

Reference: This method is a modification of the method described in: "Methods of Soil Analysis", Part 2, Chemical and Microbiological Properties, 1965, pp. 894-899. American Society of Agronomy.

SOIL ORGANIC MATTER (OM) BY TITRATION

SOP AGL-10-003 utilizes the heat of reaction generated from the combination of a mixture of the soil and a 1 N Dichromate solution with concentrated sulfuric acid to oxidize the organic matter in the soil. The oxidation process reduces a portion of the dichromate in solution. The remaining, unreduced dichromate, is titrated with ferrous ammonium sulfate to determine the % OM.

Reference: "Methods of Soil Analysis", Part 2, Chemical and Microbiological Properties, Second Edition, 1982, pp. 570-571. American Society of Agronomy.

WATER HOLDING CAPACITY (WHC)

SOP AGL-10-004 involves placement of saturated soil samples on a porous ceramic plate. The samples and the plates are placed in a pressure chamber. The ceramic plate is backed with rubber membrane and the space between the plate and the membrane is connected, by way of a small tube, to the outside of the tank. The tank can then be sealed and pressurized. The resulting pressure will then force the water from the soil through the ceramic plate into the space between the plate and the membrane, and from there to the outside of the tank. After equilibrium is achieved, as evidenced by lack of expressed water, the pressure is released. The soils are removed from the tank and the % moisture is determined.

Reference: Soil Moisture Equipment Company's modification to: "Methods of Soil Analysis", Part 1, Physical and Mineralogical Properties, Including Statistics of Measurement and Sampling, 1965, pp. 273-278. American Society of Agronomy.

PARTICLE SIZE ANALYSIS (TEXTURE)

SOP AGL-10-011 utilizes a hydrometer to determine the density changes in a soil solution as the particles settle to the bottom. Stokes Law can be applied to determine the percent sand, silt and clay sized particles remaining in solution at selected time intervals. A standard soil texture triangle is used to identify the texture class based on the values obtained for percent sand, silt and clay.

Reference: "Methods of Soil Analysis", Part 1, Physical and Mineralogical Properties, Including Statistics of Measurement and Sampling, 1965, pp. 549-566. American Society of Agronomy.

BULK DENSITY OF DISTURBED SOIL

SOP AGL-10-006 was developed by A & L to provide a method for determination of soil bulk density which could be used in the laboratory. Dry soil, ground to pass a ten mesh sieve, is placed into a tared graduated cylinder. The cylinder is then tapped on a solid surface and more soil added until the soil has reached the 100 cc mark and no further compaction is observed. The weight of the 100 cc of soil is obtained and a weight per unit volume is calculated.