Study Title

Soil Adsorption/Desorption Study of Potassium Perfluorooctanesulfonate (PFOS)

Data Requirement

Based on OECD 106

Author

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Study Initiation Date

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Study Completion Date

Date of Signing

Performing Laboratory

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Project Identification

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GLP Compliance Statement

Study Title: Soil Adsorption/Desorption Study of Potassium Perfluorooctanesulfonate (PFOS)

Study Identification Number: E00-1311

This study was conducted in compliance with Toxic Substances Control Act (TSCA) Good Laboratory Practice (GLP) Standards with the exceptions listed below:

Exceptions to GLP compliance:

- 40 CFR 972.130(e): There is not an electronic audit trail of corrections. The authenticated hardcopy printouts are considered the original raw data.

Mark E. Ellefson 06/04/01

Mark E. Ellefson, Study Director Date

William K. Reagen 06/04/01

William K. Reagen, Testing Facility Management Date

Quality Assurance Statement

Study Title: Soil Adsorption/Desorption of Potassium Perfluorooctanesulfonate (PFOS)

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This study was audited by the 3M Environmental Laboratory Quality Assurance Unit (QAU), as indicated in the following table. The findings were reported to the study director and laboratory management.

Inspection Dates	Phase	Date Reported to	
		Management	Study Director
9/19/00	Protocol	9/20/00	9/20/00
11/07/00	In-phase	11/07/00	11/07/00
4/18/01	Data (Tier I)	4/18/01	4/18/01
5/11/01	Data (Tier II&III)	5/11/01	5/11/01
5/14/01	Data (Tier II&III)	5/14/01	5/14/01
5/17/01	Data (Tier II&III)	5/17/01	5/17/01
5/22/01	Data (Tier II&III)	5/22/01	5/22/01
5/25/01	Final Report	5/25/01	5/25/01

Ken J. Lantz — 6-4-01

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Location of Archives

All original raw data and the report have been archived at the 3M Environmental Laboratory. The test materials and analytical reference standard reserve samples, as well as the samples pertaining to the analytical phase of this study are archived at the 3M Environmental Laboratory.

Reserve samples, digital copies of original data and all original paper data will be retained in the archives of 3M Environmental Laboratory for a period of at least 10 years following the effective date of the applicable final test rule.

Summary

A preliminary, screening, and advanced study of the soil adsorption/desorption of Potassium Perfluorooctanesulfonate was performed to better understand partitioning under a variety of environmental conditions. Specifically, three types of soil, one sediment, and one sludge were tested. Analyses were conducted as described by 3M Environmental Laboratory Analytical Methods ETS-8-159 "Preparation of Soil Samples for Preliminary (Tier I) Sorption Studies for Fluorochemicals as the Test Substance" and ETS-8-160 "Preparation of Soil Samples for Screening (Tier II) and Advanced (Tier III) Sorption Studies for Fluorochemicals as the Test Substance" (Appendix A), according to OECD Guideline 106 "Adsorption – Desorption Using a Batch Equilibrium Method".

Results of the study are presented in the following tables:

Table 1. Summary Table of PFOS Adsorb/Desorb Tier I Studies

Question	Conclusion
Is the analytical method appropriate/adequate for the study?	The methods ETS-8-159 and ETS-8-160 provide sufficient recovery of the test substance.
What is the best test vessel to use?	Polypropylene tubes will be used throughout this study.
What is the equilibrium time and amount adsorbed at equilibrium?	The 1:5 soil:solution ratio shows a >50% adsorption for both soils tested. The equilibrium time is 48 hours.
What is a suitable desorption solvent?	Methanol is a suitable desorption solvent.
What is the optimal soil:solution ratio?	The optimal soil:solution ration is 1:5
Is the test substance sufficiently stable during the study period?	The test substance is stable during the course of the study.

Table 2. Summary Table of PFOS Adsorb/Desorb Tier II Studies

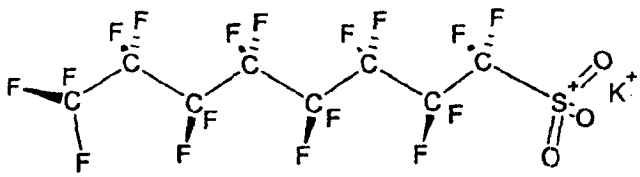
Soil Type	Average Distribution Coefficient, K_d, L/kg	Percentage of Organic Carbon in Soil, %oc	Average Organic Carbon Normalized Adsorption Coefficient, K_{oc}, L/kg
Clay (ST-1)	18.3	2.6	704
Clay Loam (ST-2)	9.72	2.6	374
Sandy Loam (ST-3)	35.3	2.8	1260
River Sediment (ST-Sed)	7.42	1.3	571
Domestic Sludge (ST-Slg)	<120	NA	NA

Table 3. Summary Table of PFOS Adsorb/Desorb Tier III Studies

Soil Type	Desorption Coefficient, $K_{d(des)}$, L/kg	K^{ads}_{f}, L/kg	K^{des}_{f}, L/kg
Sandy Loam	34.9	28.2	94.0
Clay Loam	15.8	14.0	60.2
Clay	47.1	25.1	105
River Sediment	10.0	8.70	44.6
Domestic Sludge	<237	338	3130

Note: The units for the isotherms assume that the term n=1. More accurately, the units are $(\mu g^{1-1/n}\ (L)^{1/n}\ kg^{-1})$

Introduction



POTASSIUM PERFLUOROOCTANESULFONATE (PFOS)

CAS Number: 2795-39-3

Chemical Formula: $C_8F_{17}SO_3K$ Molecular Weight: 538.22 g/mol

Purpose

Adsorption/desorption studies are useful for generating essential information on the mobility of chemicals and their distribution in the soil, water, and air compartments of our biosphere. They can be used in the prediction or estimation of the availability of a chemical for degradation, transformation and uptake by organisms; soil leaching profile; volatility from soil; and run-off from land surfaces into natural waters. Adsorption data can also be used for comparative and modeling purposes.

The distribution of a chemical between soil and aqueous phases is a complex process depending on a number of different factors: the chemical nature of the substance, the characteristics of the soil, and climatic factors such as rainfall, temperature, sunlight and wind. Thus, the numerous phenomena and mechanisms involved in the process of adsorption of a chemical by soil cannot be completely defined by a simplified laboratory model such as the present guideline. However, even if this attempt cannot address all of the environmental possibilities, it provides valuable information on the environmental relevance of the adsorption of a chemical.[1]

OECD guideline 106 is aimed at estimating the adsorption/desorption behavior of a substance on soils. The goal is to obtain a sorption value that can be used to predict partitioning under a variety of environmental conditions. Therefore, equilibrium adsorption coefficients for a chemical onto various soils are determined as a function of soil characteristics. Different soil types must be used in order to emulate the interactions of a given substance with naturally occurring soils. The soil parameters that are believed most important for adsorption are pH, organic carbon content, clay content, and soil texture. The procedures outlined in the guideline are designed to evaluate the adsorption of a chemical on different soil types that have a varying range of pH, organic carbon content, and soil texture. The guideline is comprised of three tiers:

1.1 Tier I: The Preliminary Study

1.1.1 The preliminary study is designed to determine:

a) a suitable analytical method

b) the adsorption of the test substance onto the surfaces of the test vessels

c) the equilibration time for adsorption and the amount of test substance adsorbed at equilibrium

d) a suitable desorption solvent

e) the soil:aqueous solution ratio

f) the stability of the test substance during the study period

1.1.2 The preparatory methodology for Tier I is described in the 3M Environmental Laboratory Method ETS-8-159: Preparation of Soil Samples for Preliminary (Tier I) Sorption Studies for Fluorochemicals as the Test Substance (based on OECD Guideline 106).

1.2 Tier II: Screening Study:

1.2.1 The screening study is designed to study the adsorption of the test substance in three different soil systems, one sediment, and one dried sludge by means of:

a) adsorption kinetics at a single concentration

b) determination of distribution coefficients K_d and K_{oc}

1.2.2 The preparatory methodology for Tier II is described in the 3M Environmental Laboratory Method ETS-8-160: Preparation of Soil Samples for Screening (Tier II) and Advanced (Tier III) Sorption Studies for Fluorochemicals as the Test Substance (based on OECD Guideline 106).

1.3 Tier III: Adsorption Isotherms and Desorption Kinetics/Desorption Isotherms

1.3.1 The advanced study is designed to:

a) determine the Freundlich adsorption isotherms that, in turn, determine the

[1] According to OECD Guideline 106

influence of concentration on the extent of adsorption onto the soils, sediment and sludge.

b) study desorption by means of determining desorption kinetics/Freundlich desorption isotherms

1.3.2 The preparatory methodology for Tier III is described in the 3M Environmental Laboratory Method ETS-8-160: Preparation of Soil Samples for Screening (Tier II) and Advanced (Tier III) Sorption Studies for Fluorochemicals as the Test Substance (based on OECD Guideline 106).

Materials and Methods

Chemical Characterization

Table 4. Characterization of the Test Substance, Test System, and Analytical Reference Substances

Test Substance	PFOS	THPFOS
Source	3M Specialty Chemicals, Bldg. 236-1B-10	SynQuest Labs
Expiration Date	8/31/2001	none
Storage Conditions	Frozen	Frozen
TCR Identification Number	TCR-00017-046	TCR-00017-047
Physical Description	White Powder	White Powder
Purity	97.9%	TBD

Solvent	PFOS Solubility (Corrected for Purity)
ASTM Type I Water	680 ug/mL *
Natural Seawater	12.7 ug/mL **
Aqueous Solution of 3.5% Sodium Chloride	20.2 ug/mL **
n-Octanol	56.0 ug/mL ***

** As reported by 3M Environmental Laboratory Report #E00-1716 Phase: Solubility of PFOS in Water, ** Phase: Solubility of PFOS in Natural Seawater and an Aqueous Solution of 3.5% Sodium Chloride, and *** Phase: Solubility of PFOS in n-Octanol.*

All solubility determinations of PFOS TCR-00017-046 were conducted at 22-25 °C.

For PFOS stability information refer to 3M Environmental Laboratory report W-1878 (PFOS hydrolysis study).

Soil Type	Clay	Barnes Loam	Clay Loam	Sandy Loam	River Sediment	Domestic Sludge
Source	Agvise	Agvise	Agvise	Agvise	Agvise	NIST
Expiration Date	12/31/2015	12/31/2015	12/31/2015	8/1/2015	12/31/2015	10/31/2005
Storage Conditions	Room Temperature	Room Temperature	Room Temperature	Room Temperature	Room Temperature	Room Temperature
Chemical Lot Number	00-2407	00-2404	00-2405	99-2564	00-2046	2781
Physical Description	Dried and sieved	Dried and sieved	Dried and sieved	Dried and sieved	Dried and sieved	Dried and sieved
% Organic Carbon	2.6%	4.9%	2.6%	2.8%	1.3%	N/A
% Sand	16%	39%	21%	58%	39%	N/A
% Silt	22%	50%	46%	22%	42%	N/A
% Clay	62%	11%	33%	20%	19%	N/A

Method Summaries

- **ETS-8-159 "Preparation of Soil Samples for Preliminary (Tier I) Sorption Studies for Fluorochemicals as the Test Substance"**
  - **Suitable Analytical Method:**

    A soil of high adsorbability (high organic carbon and clay content) is agitated with an appropriate volume of 0.01 M $CaCl_2$ solution at a 1:5 (w:v) ratio for a minimum of 4 hours. The mixture is centrifuged and the aqueous phase filtered, if necessary. This "matrix solution" is then prepared by adding 100uL of 500mg/L PFOS in a 10ml volumetric flask to reach a nominal concentration within the concentration range that is likely to occur during the test. This "study sample" is then analyzed using HPLC/MS. After the study sample has been analyzed, a determination is made as to whether HPLC/MS is a suitable analytical method for the test substance.

  - **Suitable (Minimally Adsorbing) Container**

    Up to five containers comprising a variety of materials are exposed to a 0.01 M $CaCl_2$ aqueous solution dosed with the test substance at a concentration of 0.10mg/L and 1.0mg/L for a minimum of 24 hours. The resulting solution is analyzed for the test substance using HPLC/MS. In addition, an extraction of the container walls is made with methanol. This extract is analyzed as well. Any container that adsorbs less than 10% of the test substance onto its walls is considered suitable for use as a study container. Alternatively, the container demonstrating the least amount of test substance absorption is chosen from those tested.

  - **Selection of Optimal Soil: Aqueous Solution Ratio, Determination of Equilibration Time, and Stability of the Test Substance Under the Conditions of the Study**

    The optimal soil: aqueous ratio is determined by using two types of soils and three soil: solution ratios. An aqueous solution in contact with the soil is spiked with 125uL of

500mg/L PFOS to a concentration of 5.0mg/L. Sufficient tubes are prepared such that tubes can be removed at intervals (spanning 0 to 48 hr) and the aqueous portion analyzed. Equilibrium is determined by plotting the adsorption of the test substance over time. The optimal soil: solution ratio is determined by comparing of the amount of adsorption of the test substance at equilibrium for the various soil: solution ratio study samples. Acceptable values are soil: solution ratios that give a depletion of the test chemical greater than 20% and preferably greater than 50% at equilibrium. Stability is determined by a mass balance determination following a methanol extraction of the soil. The mass balance determination is conducted on the one soil:solution ratio per soil that gives a depletion of the test chemical above 20% and preferably above 50% at equilibrium.

- **Suitable Desorption Solvent:**

  Methanol was investigated as the suitable desorption solvent for the test substance. A high clay content soil was dosed with the test substance at two levels (0.75 µg, 7.5 µg). The test substance was then extracted from the study samples three times with methanol. The combined methanol extracts are then prepared for analysis by HPLC/MS.

  In all cases, adequate quality assurance samples were prepared and analyzed. The equilibration steps were performed at 19 to 30 degrees C.

- **ETS-8-160 "Preparation of Soil Samples for Screening (Tier II) and Advanced (Tier III) Sorption Studies for Fluorochemicals as the Test Substance"**

  - **Tier II: Adsorption Kinetics (One concentration):**

    Appropriate soils and/or sediments and/or sludges were selected for the study. Replicate study samples containing the soils (or sediments or sludges) are equilibrated by shaking for at least 12 hours at room temperature with 0.01 M $CaCl_2$. Study samples are dosed with the test substance at approximately 0.5 mg/L and placed on an orbital shaker. Replicate sets of these study samples are removed at designated time points throughout a 48 hour time period. Study samples are then prepared and analyzed for the target analyte. The adsorption kinetics are determined using this data. The last set of study samples (48 hour) are saved and used for the desorption kinetics portion of the method.

  - **Tier III: Desorption Kinetics (One concentration):**

    After the adsorption kinetics experiment, the 48 hour study samples are centrifuged and the aqueous phase removed. The volume of solution removed is replaced by an equal volume of 0.01 M $CaCl_2$ without test substance. The new mixture is agitated until the desorption equilibrium is reached. During a 48 hour period, at defined time intervals, small aliquots of the aqueous phase are removed and analyzed for the target analyte. The experiment then continues with the original mixture. The desorption kinetics are determined using this data.

  - **Tier III: Adsorption Isotherms (Five concentrations):**

    Five test substance concentrations are used, covering two orders of magnitude. Study samples containing soil (or sediments or sludges) in contact with 0.01 M $CaCl_2$ are equilibrated for a minimum of 12 hours. After equilibration, the study samples are dosed with test substance. The samples are then gently agitated until adsorption equilibrium is reached. Sample sets are removed from the orbital shaker at designated time intervals. The study samples are then prepared and analyzed for the target analyte. The adsorption

isotherms are calculated using this data. The study samples from the last time interval are saved for the desorption isotherm study.

- **Tier III: Desorption Isotherms:**

  The study samples from the adsorption isotherm study are used for the desorption isotherm study. These samples are centrifuged and the aqueous layer removed. An equal volume of fresh 0.01 M $CaCl_2$ solution containing no test substance is added to each sample. The samples are placed in the orbital shaker and equilibrated for 48 hours. The samples are then prepared and analyzed for the target analyte. (These study samples are saved and used to determine mass balance.) The desorption isotherms are determined using this data.

- **Tier III: Mass Balance:**

  The study samples from the desorption isotherm study are used for the mass balance study. The study samples are centrifuged and the aqueous layer removed. Three portions of methanol are added to the study samples. With each addition of methanol, the study samples are agitated, centrifuged, and the methanol removed and placed in a second container. These subsequent (methanol) study samples are then prepared and analyzed for the target analyte. Mass balance is determined using this data.

  In all cases, adequate quality control samples are prepared and analyzed. Additionally, the equilibration steps were carried out at between 19 and 30 degrees C.

Table 5. Test System Distribution for Study LRN-E00-1311

Population		Total	Selected for LRN-E00-1311
Tier I "Suitable Analytical Method"	Test Substance Solutions	45	45
	Control Blanks	15	15
Tier I "Suitable Desorption Solvent"	Test Substance Solutions	18 + 6 Matrix Spikes	18 + 6 Matrix Spikes
	Control Blanks	9 + 3 matrix spikes	9 + 3 matrix spikes
Tier I "Suitable (Minimally Adsorbing) Container"	Test Substance Solutions	60 + 16 Matrix Spikes	60 + 16 Matrix Spikes
	Control Blanks	30 + 8 matrix spikes	29 + 8 matrix spikes
Tier I "Selection of Optimal Soil: Solution Ratios"	Test Substance Solutions	171 + 57 Matrix Spikes	171 + 57 Matrix Spikes
	Control Blanks	63 + 21 matrix spikes	63 + 21 matrix spikes
Tier II "Adsorption Kinetics"	Test Substance Solutions	144 + 48 Matrix Spikes	139 + 48 Matrix Spikes
	Control Blanks	36 + 12 matrix spikes	36 + 12 matrix spikes
Tier III "Desorption Kinetics"	Test Substance Solutions	126 + 42 Matrix Spikes	115 + 42 Matrix Spikes
	Control Blanks	126 + 42 Matrix Spikes	126 + 42 Matrix Spikes

Tier III "Adsorption Isotherms"	Test Substance Solutions	180 + 60 Matrix Spikes	180 + 60 Matrix Spikes
	Control Blanks	36 + 12 matrix spikes	36 + 12 matrix spikes
Tier III "Desorption Isotherms"	Test Substance Solutions	90 + 30 Matrix Spikes	89 + 30 Matrix Spikes
	Control Blanks	18 + 6 matrix spikes	18 + 6 matrix spikes
Tier III "Mass Balance"	Test Substance Solutions	90 + 30 Matrix Spikes	89 + 30 Matrix Spikes
	Control Blanks	18 + 6 matrix spikes	18 + 6 matrix spikes

Specimen Collection and Analysis

During the course of the study, aliquots of the test systems were collected at predetermined time intervals. Some sample aliquots were diluted with methanol. The following table describes the sampling regimen information:

Table 6. Sample Collection and Preparation for Study LRN-E00-1311

Step	Procedure	Replicates	Date(s) Performed
Tier I "Suitable Analytical Work"	Each of the three soils, the sediment, and the sludge are equilibrated with 0.01M $CaCl_2$ in triplicate. This "matrix solution" is then removed and dosed with the test substance, and the resulting solution analyzed.	Each "matrix solution" is dosed in triplicate, and one control sample is prepared.	Samples aliquotted 11/02/00
Tier I "Suitable Container"	Four types of test vessels, each of a different material, are exposed to the test substance for 24 hours. The resulting solution is analyzed for the test substance to measure any loss to the container. The test vessel is then extracted to measure any test substance that may have adsorbed onto the sides if the container.	Each type of container is dosed at two concentrations, and a set of controls is also prepared. Every vessel is extracted with methanol after the test substance has been removed.	Aqueous samples aliquotted 11/14/00 Methanol extractions aliquotted 11/15/00
Tier I "Suitable Desorption Solvent"	Two types of soil are dosed with the test substance, and the soil is extracted with methanol three times. The extracts are combined and analyzed to measure the suitability of the solvent.	Each type of soil is dosed at two concentrations, and a set of controls is also prepared.	Methanol extractions aliquotted 1/22/01
Tier I "Selection of Optimal Soil: Solution Ratios and Mass Balance"	Two types of soil are prepared in three soil:solution ratios to determine the optimal ratio to use in the remainder of the study. Samples are prepared at several time points to determine the time required for the solution to reach equilibrium, and a mass balance calculation is performed.	Each soil/ratio/time combination is prepared in triplicate, one matrix spike is performed, and one control is prepared.	Aqueous samples aliquotted 11/29/00-12/4/00 Methanol extractions aliquotted 12/7/00

Tier II "Adsorption Kinetics"	Three soils, one sediment, and one sludge are prepared in the soil:solution ratio indicated in the Tier I study. The samples are dosed with the test substance and placed on a shaker, samples are then pulled at several time points.	Each soil/time combination is prepared in triplicate, one matrix spike is performed, and a set of controls is prepared.	Aqueous samples aliquotted 2/1/01-2/7/01
Tier III "Desorption Kinetics"	The aqueous layer is removed from the 48 hour samples from the above step. Fresh $CaCl_2$ is added to the test vessel and the vessel is placed on a shaker. Small samples are taken at several time points, and additional aqueous solution is added to replace the sample taken.	Each 48 hour sample vessel from the above step produces 9 samples over the course of the desorption (seven time points and two matrix spikes).	Aqueous samples aliquotted 2/13/01-2/15/01
Tier III "Adsorption Isotherms"	Three soils, one sediment, and one sludge are prepared in the soil:solution ratio indicated in the Tier I study. The samples are dosed with the test substance (at one of 5 concentrations) and equilibrated for either 0 or 48 hours.	Each soil/concentration/time point combination is prepared in triplicate, one matrix spike is performed.	Aqueous samples aliquotted 2/14/01-2/15/01
Tier III "Desorption Isotherms"	The aqueous layer is removed from the 48 hour samples from the above step. Fresh aqueous $CaCl_2$ solution is added to the test vessel and the vessel is placed on a shaker for 48 hours.	Each 48 hour vessel produces one sample, and every third sample is also prepared as a matrix spike.	Aqueous samples aliquotted 2/21/01
Tier III "Mass Balance Determination"	The aqueous solution is removed from the 48 hour samples in the above step. The remaining soil is then extracted with three portions of methanol, each time the solution is decanted into the same vessel.	Each 48 hour vessel produces one sample, and every third sample is also prepared as a matrix spike.	Methanol extractions aliquotted 2/22/01-2/26/01

Diluted samples (i.e. sample extracts) containing 250 ng/mL THPFOS as internal standard were analyzed using high-performance liquid chromatography/mass spectrometry (HPLC/MS) in the negative ion mode. PFOS levels were evaluated versus standard curves ranging in concentration from 2.5-1000 ng/mL PFOS and 250 ng/mL THPFOS. Internal Standard quantification was used to best fit and report the data. Target ions were deprotonated PFOS (m/z = 499), and deprotonated THPFOS (m/z = 427).

Analytical details are included in the file for this study that is maintained by the 3M Environmental Laboratory; the study folder is located in the 3M archives. All study samples were generated and analyzed at the 3M Environmental Laboratory. No chain of custody forms were generated or required.

Reserve samples of reference materials from the 3M Environmental Laboratory will be stored at the 3M Environmental Laboratory for a period not less than 10 years following the effective date of the final test rule, or until the quality of the preparation no longer affords evaluation. Reserve samples of the reference materials from the contract laboratory will be returned to the 3M Environmental Laboratory for retention and archiving.

Results and Discussion

Data Quality Objectives

The following data quality objectives are from the methods used in the present study. The data quality objectives were met, except as documented in raw data.

- **Coefficient of Determination (r^2).** The coefficients of determination (r^2) for the calibration curves were 0.985 or greater. The curves were examined closely for accuracy of quantitation at the low and high ends of the curve. Quadratic curve fit was applied to calibration standards and sample data to improve quantitation over the concentration range appropriate to the data.
- **Method Blanks.** Method blanks (applicable sample matrix taken through the entire sample preparation, dilution, and analysis process) provided a measure of laboratory contamination. Acceptable values for the blanks were less than 50% of the limit of quantitation (LOQ), defined herein as the concentration of the lowest standard.
- **Matrix Spike Recoveries.** A post-preparatory matrix spike sample for each of the sample groups used in the study was prepared by adding aliquots of test-analyte solution according to 3M Method ETS-8-159 and 160. The spike recoveries for >80% of the samples were acceptable (100 ± 30%). Instances where spike recoveries outside of the control range were observed are indicated in Appendix E.
- **Sample Triplicates.** The Relative Standard Deviations of triplicate samples were acceptable when at least 80% of them were less than or equal to 30%. Q-tests were used to exclude outliers in data sets.
- **Continuing Calibration Verification.** If the percent difference for the amount of quantified analyte was greater than 30% from the true value relative to the initial standard curve, only those samples analyzed before the last acceptable calibration check standard were used. The remaining samples were analyzed with a new curve.
- **Limit of Quantitation (LOQ).** The LOQ was equal to the lowest acceptable standard in the calibration curve.
- **Calibration Standards.** Eleven standards ranging in concentration from approximately 2.5 to 1000 ng PFOS/mL methanol were used for the calibration curves. Calibration curves were run before and after every analytical sequence.
- **Solvent Blanks.** Solvent (Methanol) blanks provide a measure of instrumental contamination. Acceptable values for the blanks were less than 50% of the limit of quantitation, defined herein as the lowest calibration standard.
- **System Suitability.** Without performing a method validation, system suitability was demonstrated by acceptable instrumental checks (e.g. abbreviated m/z check-tune, or full auto-tune routines).

Statistical Methods and Calculations

Means and standard deviations were calculated using functions provided in Microsoft® Excel® software. Calculations with the raw data were made using the equations described in the OECD Method 106 (see Appendix B).

Data Summary and Discussion

Appendices D and E summarize individual sample data. Representative chromatograms are presented in Appendix G. All concentrations and calculations can be found in the data tables, along with the log plots used to determine the isotherm values. These data are used in all subsequent tables and figures in this report.

Results of the Tier I study are presented in table 7 through 11:

Table 7. Suitable Analytical Method Determination

Soil Type	Soil:0.01M $CaCl_2$ Solution ID	Average Recovery of PFOS from matrix solution, %	%RSD
Barnes Loam (ST-1)	E00-1311-0001, -0002, -0003	115%	7.51
Clay Loam (ST-2)	E00-1311-0004, -0005, -0006	115%	4.92
Clay (ST-3)	E00-1311-0007, -0008, -0009	99.8%	21.8
River Sediment (ST-Sed)	E00-1311-0010, -0011, -0012	116%	8.88
Domestic Sludge (ST-Slg)	E00-1311-0013, -0014, -0015	88.3%	16.0

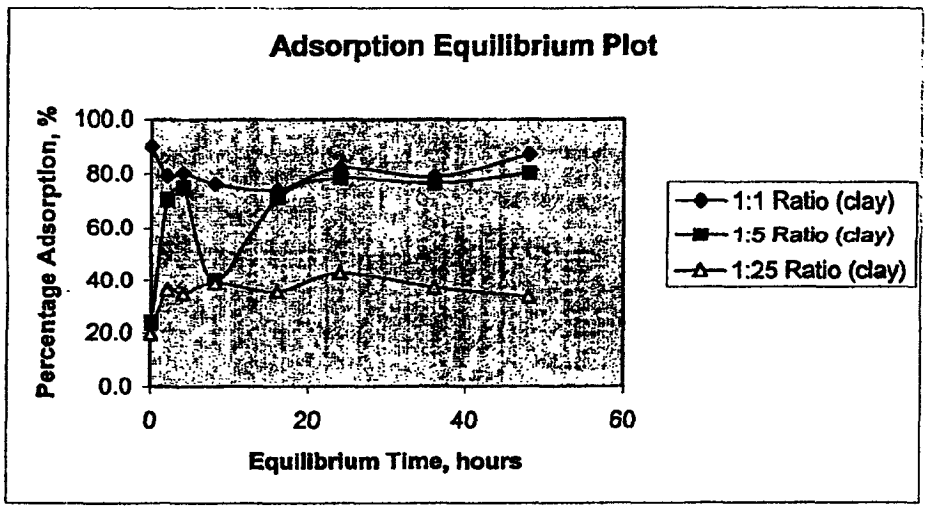
Table 8. Suitable Test Vessel Determination

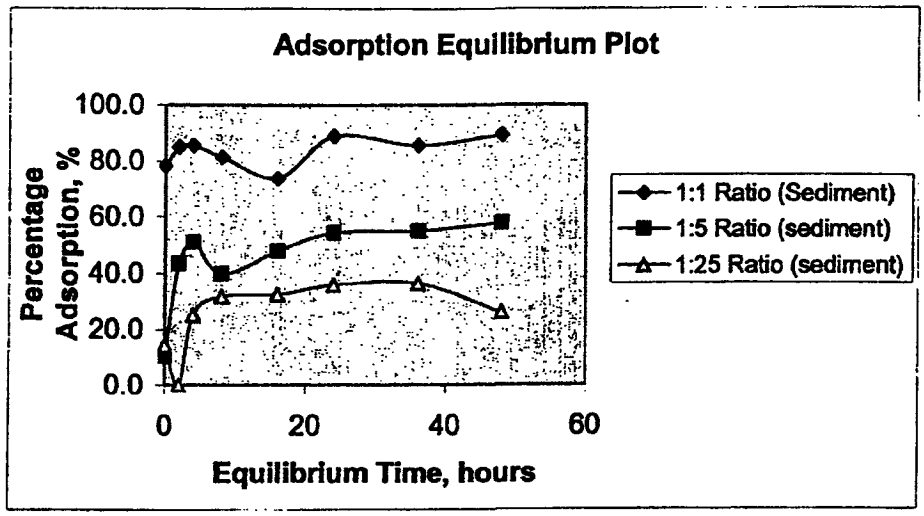
Centrifuge Tube Type	Average Recovery of 1.0mg/L PFOS After Adsorb Step, %	Average Concentration of PFOS Extracted From Test Vessels During Desorb Step, PPB	Average Matrix Spike Recovery for Adsorb and Desorb Steps, %
Polypropylene	96.2%	244ppb	116%
Polystyrene	94.5%	172ppb	203%
Glass	96.7%	398ppb	206%
Teflon	89.5%	94.4ppb	176%

It was determined that the polypropylene centrifuge tubes were suitable for this study. These tubes had the least amount of extractable PFOS, and were the only tubes with acceptable matrix spike recoveries.

Table 9. Equilibrium Time for Adsorption of PFOS

Soil Type	Soil:Solution Ratio	Time Point, Hours	Average Recovery of PFOS (in solution)%
Clay	1:1	24	17.6%
		36	21.0%
		48	12.7%
	1:5	24	21.6%
		36	23.5%
		48	19.7%
	1:25	24	57.4%
		36	60.2%
		48	66.8%
Sediment	1:1	24	11.0%
		36	14.3%
		48	10.6%
	1:5	24	45.9%
		36	45.0%
		48	42.2%
	1:25	24	65.3%
		36	67.3%
		48	68.0%





The test substance appeared to reach equilibrium after 24 hours in all of the soil:solution ratios. The clay appeared to take more time to reach equilibrium, therefore the 48 hour time point was used as the equilibrium time for the remainder of the study. This ensured that all five of the soils were given ample time to equilibrate.

Table 10: Suitable Desorb Solvent, Methanol

Soil Type	Theoretical Test Substance Concentration In Extract, ug/L	Average Recovery, ug/L	AVERAGE RECOVERY OF PFOS%
Clay	0.0	<25.0	N/A
	100	71.2	71.2%
	1000	591	59.1%
Sediment	0.0	<25.0	N/A
	100	96.0	96.0%
	1000	601	60.1%

Despite using methanol, an excellent solvent for PFOS, the average percent recovery of PFOS ranged from zero to 96%. It is unlikely that a different solvent would yield better recovery. Previous results in this study have indicated that the test substance is readily and rapidly adsorbed on the soil, and is not likely to be completely extracted from it.

The optimal soil:solution ratio was determined to be 1:5 (refer to Table 9 for applicable data). This ratio demonstrated average adsorption percentages of PFOS ranging from approximately 75% in clay to 55% in sediment.

Table 11. Stability of PFOS during the Study Period

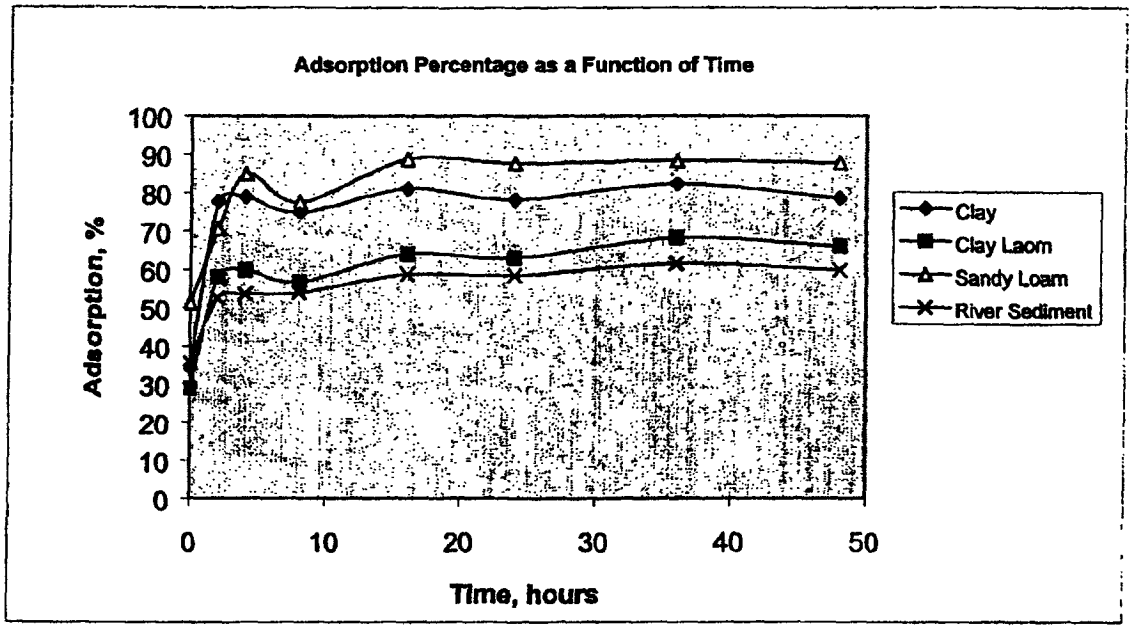
Soil Type	Soil:Solution Ratio, Time Point	Time Point, hours	Total Recovery of PFOS %
Clay	1:1	48	46.2
		48	53.2
		48	71.3
	1:5	48	34.3
		48	35.6
		48	Tube Broke
	1:25	48	83.2
		48	91.2
		48	96.3
Sediment	1:1	48	43.7
		48	48.1
		48	24.7
	1:5	48	57.6
		48	78.7
		48	59.5
	1:25	48	73.6
		48	87.4
		48	83.1

According to OECD guideline 106, the mass balance calculation should show at least 90% recovery of the test substance for the test substance to be considered stable over the course of the study. The recovery of the test substance was lower than the OECD guideline, likely due to the incomplete desorption of the test substance from the soil. Hydrolysis studies of PFOS conducted at the 3M Environmental Laboratory have demonstrated test substance stability in aqueous matrices.

The results of the Tier II study are presented in Table 12:

Table 12. Adsorption Kinetics of PFOS, 1:5 Soil:Solution Ratio, 48 Hour Time Point

Soil Type	Average Distribution Coefficient, K_d, L/Kg	Percentage of Organic Carbon in Soil, %	Average Organic Carbon Normalized Adsorption Coefficient, K_{oc}, L/kg
Clay (ST-1)	18.3	2.6	704
Clay Loam (ST-2)	9.72	2.6	374
Sandy Loam (ST-3)	35.3	2.8	1260
River Sediment (ST-Sed)	7.42	1.3	571
Domestic Sludge (ST-Slg)	< 120	NA	NA

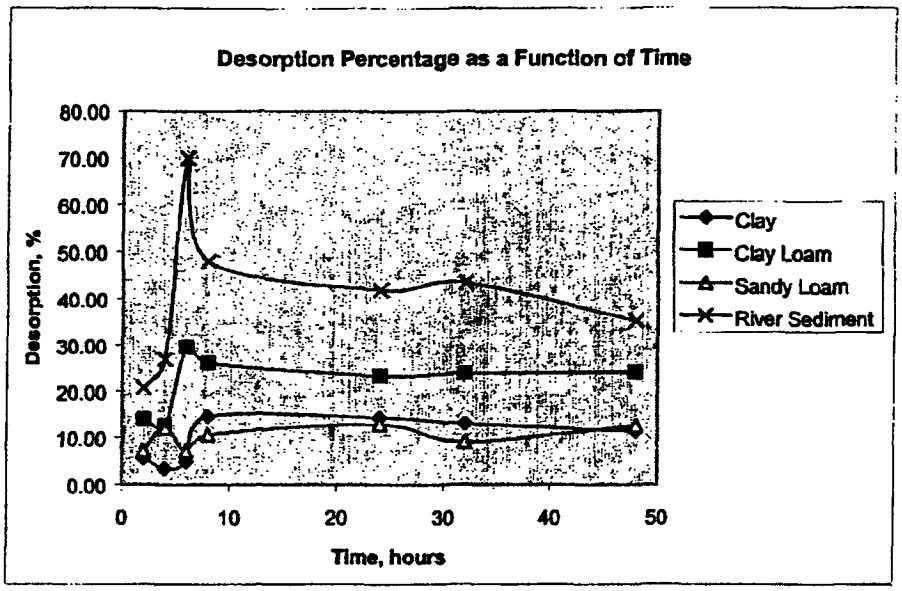


All of the soil/sediment/sludge matrices adsorbed the test substance to varying degrees. The sludge samples, in the adsorption portion of the kinetics study, demonstrated the most adsorption (>96%) and PFOS was not detected in the samples. The data indicates that adsorption occurred within the first few hours of exposure and the test substance concentration does not vary significantly after the 16 hour time point.

The results of the Tier III study are presented in Tables 13 through 15:

Table 13. Desorption Kinetics of PFOS, 1:5 Soil:Solution Ratio, 48 hour Time Point

Soil Type	Desorption Coefficient, K_{des}, L/Kg
Clay (ST-1)	47.1
Clay Loam (ST-2)	15.8
Sandy Loam (ST-3)	34.9
River Sediment (ST-Sed)	10.0
Domestic Sludge (ST-Slg)	<237



The test substance was poorly desorbed from the soil/sediment/sludge matrices during the 48-hour study period. The river sediment displayed the most desorption at only 39% after 48 hours. The sludge samples did not desorb a detectable amount of test substance. Desorption that did occur was accomplished rather quickly, after the 8 hour time point the test substance concentration did not vary significantly.

Table 14. Adsorption Isotherms

Soil Type	Log K^{ads}_F	K^{ads}_F, L/Kg	Regression Constant, 1/n	Regression Constant, n
Clay	1.40	25.1	0.884	1.13
Clay Loam	1.15	14.0	0.841	1.19
Sandy Loam	1.45	28.2	0.829	1.21
River Sediment	0.939	8.70	0.989	1.01
Domestic Sludge	2.53	338	1.26	0.795

Note: The units for the isotherm assume that the term n=1. More accurately, the units are $(\mu g^{1-1/n} (L)^{1/n} Kg^{-1})$

Table 15. Desorption Isotherms

Soil Type	Log K^{des}_{F}	K^{des}_{F}, L/Kg	Regression Constant, 1/n	Regression Constant, n
Clay	2.02	105	0.860	1.16
Clay Loam	1.78	60.2	0.954	1.05
Sandy Loam	1.97	94.0	0.985	1.02
River Sediment	1.65	44.6	1.02	0.981
Domestic Sludge	3.50	3130	0.977	1.02

Note: The units for the isotherm assume that the term n=1. More accurately, the units are $(\mu g^{-1/n}\ (L)^{1/n}\ Kg^{-1})$

Freundlich isotherms relate the amount of test substance adsorbed on the soil to the amount present in the aqueous solution at equilibrium. The values calculated for the regression constant indicate that the data obtained for the test substance over two orders of magnitude are slightly non-linear.

References

1. Adsorption-Desorption Using a Batch Equilibrium Method (OECD 106), January 2000.

Report Amendments

The organic carbon normalized adsorption coefficient (K_{oc}) values contained in tables 2 and 12, isotherm values contained in tables 3, 14 and 15 as well as raw spreadsheets on pages 205-233 were corrected following clarification of calculations described in OECD guideline 106. Table 13 had 2 values switched and was corrected. The conclusion section was removed. The title page was modified to indicate this report has been amended. The signature page was re-signed following the amendments.

Signatures

The amended final draft of this report is a true representation of the data developed in this study. It has been issued by:

Mark E. Ellefson, Study Director — Date: 05/23/02

William K. Reagan, Testing Facility Management — Date: 05/24/02