Results of the Analysis of Soil Samples from Near Decatur, Alabama for Fluorinated Organic Compounds II: Subsurface Soils

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Date

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Abstract: In March 2009, surface and subsurface soil samples were collected from six agricultural fields near Decatur, Alabama area where sludge from the Decatur Utilities had been applied for more than 12 years. Soil samples were also collected at one background field, an area where sludge had not been applied.

The results of analysis of the surface soils samples for selected perfluorinated chemicals (PFCs) and fluorotelomer compounds (FTCs) were provided in an earlier report. In this second report, we summarize results of PFC and FTC analyses for the subsurface soils.

Subsurface soil samples from the sludge-applied fields had soil concentrations of perfluorooctanoic acid (PFOA) at least 14-times that of the background field. The contrast in [PFOA] between sludge-applied and background subsurface soils exceeded that for most other perfluorocarboxylic acids (PFCAs). Perfluorooctane sulfonate (PFOS) concentrations in the subsurface soils of the sludge-applied fields ranged from about equal to background values to nearly 100-times that of the background field.

1. Introduction

For the last 12 years, Decatur Utilities, of Decatur, AL, has been authorized to apply sewage sludge on about 5000 acres of local agricultural land (Neill, 2009a). The United States Environmental Protection Agency (USEPA) collected (Neill, 2007) and analyzed (Washington et al., 2009a) a limited number of sludge and soil samples from this operation. The results documented the presence of several fluorotelomer alcohol (FTOH) and perfluorinated compounds (PFCs) in the land application soil samples.

The USEPA subsequently collected (November 13, 2008) and analyzed drinking water samples from a few Decatur, AL public drinking water supplies (Neill, 2009a). No levels of the perfluorooctanoic acid (PFOA) or perfluorooctane sulfonate (PFOS) were observed above the Provisional Health Advisories (USEPA, 2009) in these municipal drinking water samples (Washington et al., 2008a). In February 2009, the USEPA collected additional water samples from selected private wells, field wells, agricultural ponds, and surface waters located in and immediately around the land application fields. Elevated levels of some PFCs (Lindstrom et al., 2009) were found in some of these samples.

Additional samples of surface soils, subsurface soils were collected in March 2009 to characterize the extent and magnitude of the PFC contamination in the land application area (Neill, 2009c). These samples were analyzed for a variety of perfluorinated chemicals (PFCs) and fluorotelomer alcohols (FTOHs) using Standard Operating Procedures (SOPs) developed at ERD for the characterization of these analytes in surface soils. Quality control practices included as part of these SOPs, specifically, the recovery of mass-labeled internal standards, indicated that the PFCs in the subsurface soil samples were incompletely extracted using the SOP which was developed using surface soils. Consequently, only data for the surface soils were reported in our earlier report (Washington et al., 2009b) so that a method suitable for the analysis of PFCs in subsurface soils could be developed and, subsequently, all data for subsurface soils could be reported together. Now having developed a method for the PFC analysis in subsurface soils, here we report these subsurface-soil data as the second volume (Volume II) of a series with the original report (Volume I; Washington et al., 2009b); these reports are intended to be studied in tandem.

2. Methods

Volumes I (surface soils) and II (subsurface soils) share several common factors in their genesis: i) soil collection methods and transport to laboratory; ii) sample preparation for extraction; iii) extraction for analysis of fluorotelomer alcohols (FTOHs) and similar analytes on a gas chromatograph interfaced with a mass spectrometer (GC/MS); and iv) analytical methods for the extracts on both the GC/MS and a liquid chromatograph interfaced with a tandem mass spectrometer (LC/MS/MS). Consequently, in this volume, we describe only those procedures that differ from those employed for the surface soils, quality-control data that characterize the soundness of subsurface-soil data, and all analytical results for the subsurface soils. All procedures shared for the generation of both surface and subsurface soil data were reported in Volume I (Washington et al., 2009b).

2.1 Sample Collection

Soil samples were collected by USEPA Region 4 personnel during the period March 23-25, 2009 following the procedures described in the USEPA Region 4 Quality Assurance Project Plan (Neill, 2009b) and the corresponding trip report (Neill, 2009c).

The sampling equipment was supplied and prepared by scientists from Region 4's Science and Ecosystem Support Division (SESD) and the National Exposure Research Laboratory's Ecosystems Research Division (NERL/ERD), both located in Athens, GA. The sampling equipment was constructed of stainless steel materials and washed three times with Optima-grade methanol (MeOH) prior to and between uses. Subsurface soil samples were collected using hand augers and pans. Sample-depth intervals (Table 1) were chosen with the objective of collecting soil from the upper portion of the B horizons, as well as a deeper interval of within the B horizons. The soil samples were stored individually in certified-clean, 500 mL, wide-mouth high-density polyethylene (HDPE) containers. The sample containers and the MeOH-washed sampling equipment were determined to be free of contamination for the intended analytes before the sampling trip.

Quality-control samples taken to the field included Ottawa sand that has been shown to be low in the target analytes and a commercial top soil, the Cowart soil, for which the general range of concentrations of a variety of analytes has been documented (Washington et al., 2007, 2008b). Additionally, samples were collected from a background field that has not received any sludge application and is located near the sludge-application fields. Also, duplicate field samples were collected from selected locations in the sludge-applied fields.

2.2 Extraction of Subsurface Soil Samples for PFC Analyses on the LC/MS/MS

The surface-soil data for LC/MS/MS parameters were generated from extracts that were prepared in accordance with Standard Operating Procedure (SOP) PMB 59.0, an SOP that we developed specifically for sludge-treated soils using surface-soil samples collected from Decatur in the 2007/2008 reconnaissance survey (Neill, 2007; Washington et al., 2009a).

When we analyzed the 2009-survey subsurface soils using PMB 59.0, the recoveries of the mass-labeled internal standard uniformly were quite low indicating unsatisfactory extraction efficacy for the subsurface soils. In our attempts to overcome this artifact, we investigated extracting the soil samples on a Labquake rotisserie as opposed to the Eberbach shaker table that we used in the original extractions. The premise of this change was that subsurface soils generally have higher clay concentrations than do surface soils, and, consequently, subsurface soils might have a greater tendency to cohere and compact into relatively impermeable plugs. Whereas the shaker table oscillates back-and-forth with the soil plug remaining at the bottom of the tube, the rotisserie rotates the sample tubes end-over-end so that the relatively dense soil cascades through the extracting solution with each half revolution.

Because we were investigating causes for the low recovery of the subsurface soils, we sought to simplify our extraction to remove sources of uncertainty as to where the poor recovery arose. Given this incentive, we first analyzed our extracts on the LC/MS/MS without the ion-pairing cleanup step that we normally employ to subdue background noise on the mass chromatograms.

Other than these two deviations in practice, i.e., rotisserie vs. shaker table, no ion-pairing cleanup, all extraction procedures for the subsurface soils are identical to those described in PMB 59.0. Because results of this effort proved satisfactory for our quality-control criteria, no other extraction procedures were investigated. Descriptions of the samples extracted for this effort are given in Table 1.

2.3 Sample Analysis

As indicated above, LC/MS/MS analytical procedures were identical to those described for the surface soils in Volume I. Since limits of quantitation (LOQs) for this method are defined uniquely using data for each sample run, we summarize the LC/MS/MS-run parameters here in Tables 2 and 3.

3. Quality Control

The sample analytical processes included prescribed quality control (QC) procedures to document data quality and analytical performance as defined in the ERD Quality Assurance Project Plan (March 26, 2009) and as described below.

3.1 Field Blanks

Field blanks were used to check for contamination that might arise from any source in the study. Study field blanks consisted of commercially purchased Ottawa sand which has been documented to have low or no detectable levels of the targeted analytes (Washington et al., 2007; Ellington et al., 2009). Two field blanks were prepared by pouring the Ottawa sand into the cleaned HDPE soil-sample containers, labeling the containers, and transporting the containers to and from the field without opening. Upon return to the laboratory, the field blanks were handled, extracted and analyzed exactly like all the soil samples. For field blanks, repeated extractions should fall within 15% of each other or be less than the method level of quantitation (LOQ).

3.2 Field Duplicates

Duplicate soil samples provided a metric of the repeatability of the combined effects from heterogeneity of the distribution of analytes at the sampling scale in the field, variation in sampling or laboratory techniques, variation in sample containers or reagents, and/or analytical uncertainty. These data reflect the heterogeneity of the sample material in the field and provide a measure of the reproducibility of results for samples collected with the objective of being morphologically and geographically identical at the field scale.

3.3 Background Field Samples

Background field soil samples served as a means for characterizing the local or regional soils PFC and/or FTOH contributions, if any. Background field sample values were compared with the land application area soil samples to characterize the extent and magnitude of PFC and FTOH contamination. Background soil samples were collected from an agricultural field (S101) where sludge was not applied. This background field was located near the sludge-treated fields. For background field samples, repeated extractions should fall within ±15% of each other or be less than the LOQ.

3.4 Laboratory Procedure Blanks

The full extraction procedure was performed in empty polypropylene copolymer (PPCO) tubes identical to those used to extract the soil samples. These data should fall below the LOQ and are used to document that the extraction solvents and containers are free of the targeted analytes.

3.5 Laboratory Solvent Blanks

Laboratory solvent blanks, consisting of 60/40 ACN/H_2O, with and without matrix internal standards, were injected periodically during the sample runs. These data were used to demonstrate the eluents were free of the target analytes and that there was no sample "carry-over" from incomplete elution off the analytical column.

3.6 Laboratory Fortified Soil Extracts

Fortification of samples with target analytes provided data for verifying that the observed peaks are attributed correctly and that the quantitation is accurate. After initial LC/MS/MS analyses were completed, six samples were selected, split into a second pre-weighed autosampler vial, reweighed and fortified with a weighed amount of a standard. These fortified samples were then analyzed and the analytical concentrations compared to the theoretical concentrations, with the quality criterion being that measured values should fall within ± 30% of the calculated values.

3.7 GC/MS Identification of FTOHs in Soil Extracts

Targeted FTOH analytes were identified via GC/MS analysis using fragment ions and by the comparison to standards retention times (Tables 4 & 5; Figure 3 of Volume I). Both the GC/MS quantification ion and qualification ion were monitored for confirmation. Derivatizations with trimethylsilylimidazole (TMSI) were performed on selected samples to confirm the results and to ensure there were no interfering compounds and/or peaks (Figure 4 of Volume I).

3.8 Laboratory Recovery Internal Standards

Internal standards were used to document extraction recovery efficacy and the overall analytical accuracy. Known amounts of mass-labeled internal standards were added to all soil samples before extraction. Since mass-labeled recovery standards commonly contain small amounts of the unlabeled molecule, care was taken to avoid contaminating the samples with unlabeled analyte (Washington et al., 2007). Care was also taken to ensure the internal standards for the sample extracts requiring several-fold dilutions for calibration weren't diluted below the LOQ. The recovery internal standard added to LC/MS/MS analysis samples was $^{13}C_8$-labeled perfluorooctanoic acid ($^{13}C_8$-PFOA). The recovery internal standard added to GC/MS analysis samples was $^{2}H_2{}^{13}C_2$-8:2 FTOH. Using these internal standards, mean back-predicted values for all standards used to generate the calibration curves should fall within the acceptable quality criterion of ± 30% of the nominal values (Tables 6 and 7).

4. Results

4.1. Sample Completeness

All the subsurface soil and QC samples planned for this study were collected, analyzed, and reported (Table 8). A total of six sludge-applied subsurface soil samples were collected from the one field where sludge had been applied. Two subsurface soil samples were collected at the background site. Of these samples, all the collected subsurface soil samples were analyzed for PFCs and FTOHs, and reported upon herein.

4.2. Standard Curve Back-Prediction

Tables 6 and 7 summarize the mean back-calculated values for the calibration curve standards for each PFC and FTOH, respectively. Mean back-calculated values for all standards above the LOQ are within the quality criterion of ± 30% of the calculated value.

4.3. Blanks Taken to the Field, and Background Fields

Tables 9 and 10 document the expected low to non-detect PFC and FTOH analyte levels for the QC sand and QA soil samples. For the sand blanks, analyte values all were less than the LOQ values except for C8 which was detected slightly above the LOQ (Tables 9 and 10).

Tables 11 and 12 document the expected low to non-detect PFC and FTOH analyte levels for the background field samples. Most background sample analytes fell below the LOQs and the few detected analytes fell moderately above the LOQ. Out of the six low-level detections in these background samples, one fell outside of the acceptable quality criterion for repeated extractions (± 15% of each other). These few non-compliances likely reflect the challenge of detecting values near the LOQ as well as heterogeneity in the sludge-applied soil samples.

4.4. Field Duplicates

Tables 13 and 14 summarize the field duplicate sample PFC and FTOH results (respectively) and associated percent relative difference (%RD). Duplicate sample results for the targeted acid species, PFOS, and the majority of the FTOH species are considered very good (<50% RD). Some of the duplicate sample results for 7:2s FTOH, 12:2 FTOH and 14:2 FTOH, however, exceeded the general range of other analytes, 50% RD. Consequently Table 14 includes within-sample FTOH results as well. The results of analysis on two extractions of the same sample yielded uniformly good results (<30%). Therefore, the high variability seen between different duplicate sample FTOH analytes most likely reflect true heterogeneity in the field samples.

4.5. Standard Addition and Precision of Analyses

The average recovery for the added concentration of PFC standards to the soil samples (Table 15) was within the acceptable range of ±30% of calculated values. The precision associated with two repeated injections of twelve FTOH sample extracts was <15% for all analytes (Table 16). These data confirm a satisfactory degree of analytical precision for both the acids and alcohols measured and reported for this study.

4.6 GC/MS Confirmation of FTOHs in Soil Extracts

Figure 3 of Volume I shows the GC/MS technique for identifying 14:2 FTOH analyte using the qualifying fragmentation ions and the elution shift after TMSI derivatization. Figure 4 of Volume I depicts the elution shift for the analytes, confirming their identities (e.g. the 8:2 FTOH peak disappears and the expected 8:2 FTOH derivative peak (–TMS replaces H) is detected in the TMSI treated extract).

4.7 Subsurface Soil Sample Results

Tables 17 and 18 summarize the results of analysis of the subsurface soil samples for the targeted PFCs and fluorotelomer alcohols, respectively. Subsurface soil PFOA concentrations ranged from 3-80 ng/g soil (ppb), one to two orders of magnitude above background (Table 11). Subsurface soil PFOS concentrations ranged from 0.2-20 ng/g soil (ppb), up to two orders of magnitude above background (Table 11). Generally the highest PFC mass concentrations were for PFOA (C8) and PFNA (C9). Of the S4-S8 sulfonates, PFOS was detected more commonly than all other sulfonates together. No unsaturated telomer acids were detected in the subsurface soil samples.

Recovery of the internal FTOH standard was satisfactory for all samples (Table 1). FTOHs and the 8:2 fluorotelomer acrylate (FTAc) mostly went undetected in the subsurface soil samples (Table 18). The only exception was 8:2 FTOH was detected at low levels in four of six subsurface soil samples.

5. Discussion

These sample-analysis results suggest that the majority of the Decatur subsurface soils in the land application area have concentrations of numerous PFCs above the background levels, but that FTOHs are not elevated or only marginally so relative to background values. In general, the highest mass-basis concentrations of the perfluorocarboxylic acids were the C8 through the C10 acids; values commonly fell in the 1-80 ng/g range. Among the analyzed sulfonates, only PFOS was detected, with mass-basis concentrations falling in the same general range as the C8 through C10 carboxylic acids.

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Table 1: Sample Descriptions, Extract Dilutions & Internal Standard Recovery for Subsurface-Soils Analytical Run

		Sample-Depth Interval	Nominal Soil Dilution Ratio		Recovery Standard % Recovery (1)	
Sample ID	Sample Description	(cm)	LC/MS/MS	GC/MS	LC/MS/MS	GC/MS
S14-1A2	Intermediate Subsurface Grab	36 - 51	1.1	12	85.8	101.9
S14-1A3	Deep Subsurface Grab	152 - 165	1.1	12	85.6	100.0
S14-10B2	Intermediate Subsurface Grab	41 - 56	1.1	12	92.9	96.9
S14-10B3	Deep Subsurface Grab	152 - 165	1.1	12	78.7	97.4
S14-10C2	Intermediate Subsurface Grab	41 - 56	1.1	12	76.4	108.8
S14-10C3	Deep Subsurface Grab	152 - 165	1.1	12	89.3	107.5
Background Field Samples						
S101-A2	Subsurface Grab	38 - 53	1.1	12	83.4	120
S101-B2	Subsurface Grab	38 - 53	1.1	12	78.8	104
Field Quality Controls						
QC-1	Ottawa Sand		1.1	12	85.2	116
QC-2	Ottawa Sand		1.1	12	85.3	102
Lab Quality Controls						
SB1	Ottawa Sand		1.1	12	89.3	109
TB1	Process Blank		1.1	12	88.3	

Table 2: Liquid Chromatograph and Mass Spectrometer Parameters

	Nominal Retention Time (RT)													
Compound	Apex RT (min)	Front RT (min)	Tail RT (min)	Delta T from Prev. Apex (min)	Number of Transitions	Number of Transitions Per Function	Parent Anion Mass (m/z)	Cone Potential (V)	Quan Ion Mass (m/z)	Quan Ion Collision Energy (eV)	Primary Qual Ion Mass (m/z)	Primary Qual Ion Collision Energy (eV)	2nd Qual Ion Mass (m/z)	2nd Qual Ion Collision Energy (eV)
Function 1 Time Interval 0 to 1.1 Min														
Perfluoropropanoic acid (C3)	0.65	0.4	0.9		2	5	162.80	14	118.80	11	69.80	25		
Perfluorobutanoic acid (C4)	0.70	0.4	1.0	0.05	1		212.85	13	168.80	10	Irregular response			
$^{13}C_4$-Perfluorobutanoic acid ((M+4)C4)	0.70	0.4	1.0	0.05	1		216.90	14	171.80	10	Irregular response			
Perfluoropentanoic acid (C5)	0.95	0.6	1.3	0.25	1		262.80	13	218.85	10	Irregular response			
Function 2 Time Interval 0.9 to 2.1 Min														
Perfluorohexanoic acid (C6)	1.35	1.0	1.7	**0.40**	2	7	312.80	13	268.85	10	118.80	20		
$^{13}C_2$-Perfluorohexanoic acid ((M+2)C6)	1.35	1.0	1.7	**0.40**	1		314.80	14	269.85	10	*119.30*	*20*		
Perfluorobutane sulfonate (PFBS)	1.50	1.2	1.8	0.15	2		298.90	40	79.85	30	98.85	40		
Perfluoroheptanoic acid (C7)	1.80	1.5	2.1	0.30	2		362.70	13	318.80	10	168.85	18		
Function 3 Time Interval 1.8 to 3.2 Min														
Perfluorooctanoic acid (C8)	2.30	1.9	2.7	**0.50**	2	11	412.70	14	368.75	10	168.85	18		
$^{13}C_4$-Perfluorooctanoic acid ((M+4)C8)	2.30	1.9	2.7	**0.50**	1		416.70	14	371.70	10	*171.85*	*18*		
$^{13}C_8$-Perfluorooctanoic acid ((M+8)C8)	2.30	1.9	2.7	**0.50**	1		420.70	13	375.70	11	*171.85*	*20*		
Perfluorohexane sulfonate (PFHxS)	2.50	2.1	2.9	0.20	2		398.90	50	79.85	40	98.85	40		
6:2 Fluorotelomer unsaturated acid (6:2FTUCA)	*2.60*	*2.2*	*3.0*	*0.10*	*1*		*357.00*	*16*	*293.00*	*17*				
$^{13}C_2$-6:2 Fluorotelomer unsaturated acid ((M+2)6:2FTUCA)	*2.60*	*2.2*	*3.0*	*0.10*	*1*		*359.00*	*16*	*294.00*	*17*				
Perfluorononanoic acid (C9)	2.75	2.4	3.1	0.15	2		462.70	15	418.70	11	218.85	18		
$^{13}C_5$-Perfluorononanoic acid ((M+5)C9)	2.75	2.4	3.1	0.15	1		467.70	15	422.70	12	*222.90*	*18*	*218.90*	*18*
Function 4 Time Interval 2.4 to 3.4 Min														
Perfluoroheptane sulfonate (PFHpS)	2.95	2.6	3.3	0.20	2	2	448.90	50	79.90	40	98.90	40		
Function 5 Time Interval 2.9 to 4.4 Min														
Perfluorodecanoic acid (C10)	3.35	3.0	3.7	**0.40**	2	11	512.90	15	468.70	11	218.85	20		
$^{13}C_2$-Perfluorodecanoic acid ((M+2)C10)	3.35	3.0	3.7	**0.40**	1		514.90	15	470.00	12				
Perfluorooctane sulfonate (PFOS)	3.55	3.2	3.9	0.20	2		498.90	60	79.85	50	98.85	40		
7:3 Fluorotelomer acid (7:3 FTCA)	*3.65*	*3.3*	*4.0*	*0.10*			*440.80*	*17*	*336.80*	*12*	*316.80*	*22*		
8:2 Fluorotelomer unsaturated acid (8:2FTUCA)	3.65	3.3	4.0	0.00	2		456.70	16	392.70	18	*342.70*	*40*		
$^{13}C_2$-8:2 Fluorotelomer unsaturated acid ((M+2)8:2FTUCA)	3.65	3.3	4.0	0.00	1		458.70	16	393.70	16	*343.70*	*40*		
Perfluoroundecanoic acid (C11)	3.90	3.6	4.2	0.25	2		562.70	15	518.70	12	218.85	20		
$^{13}C_2$-Perfluoroundecanoic acid ((M+2)C11)	3.90	3.6	4.2	0.25	1		564.90	15	520.00	13				
Function 6 Time Interval 4.0 to 15.0 Min														
Perfluorododecanoic acid (C12)	4.50	4.2	4.8	**0.60**	2	10	612.70	16	568.70	13	318.70	20		
$^{13}C_2$-Perfluorodecanoic acid ((M+2)C12)	4.50	4.2	4.8	**0.60**	1		614.90	16	570.00	13				
10:2 Fluorotelomer unsaturated acid (10:2FTUCA)	4.65	4.4	4.9	0.15	2		557.00	16	493.00	17	*443.00*	*38*		
$^{13}C_2$-10:2 Fluorotelomer unsaturated acid ((M+2)10:2FTUCA)	4.65	4.4	4.9	0.15	1		559.00	16	494.00	17				
Perfluorotridecanoic acid (C13)	5.15	4.9	5.4	0.50	2		662.75	16	618.70	13	318.70	22		
Perfluorotetradecanoic acid (C14)	5.80	5.5	6.1	0.65	2		712.75	18	668.70	14	318.70	24		

Italicized transitions are not monitored in 090428 MS Method

Most masses optimized by direct infusion on - 080707,8

Table 3: LC/MS/MS Integration and Optimization Parameters for Subsoils Analytical Run

Compound	Savitzky Golay Smoothing # Points; # Smooths	Quan. Qual. Ratio & *Tolerance* (%)	Standards Range (pg/g) (# Levels)	Internal Standard	1/x-Weighted Calibration Equation	Correlation Coefficient (r^2)	Limit of Quantitation (pg/g)	Limit of Quantitation (LOQ) Definition
Function 1 Time Interval 0 to 1.1 Min								
Perfluoropropanoic acid (C3)	5; 2							
Perfluorobutanoic acid (C4)	5; 2							
$^{13}C_4$-Perfluorobutanoic acid ((M+4)C4)	5; 2							
Perfluoropentanoic acid (C5)	0; 0							
Function 2 Time Interval 0.9 to 2.1 Min								
Perfluorohexanoic acid (C6)	5; 2	21 +/- *44%*	0.9 - 4800 (14)	(M+2)C6	0.011*[pg/g] + 0.018	0.997	18	≥LOQ within 20% tolerance
$^{13}C_2$-Perfluorohexanoic acid ((M+2)C6)	5; 2		Invariant		Matrix Internal Standard			
Perfluorobutane sulfonate (PFBS)	5; 2	4.8 +/- *44%*	9 - 4800 (12)	(M+2)C6	0.005*[pg/g] + 0.016	0.997	18	≥LOQ within 20% tolerance
Perfluoroheptanoic acid (C7)	0; 0	3.1 +/- *44%*	5 - 4800 (13)	(M+4)C8	0.011*[pg/g] + 0.006	0.995	18	≥LOQ within 20% tolerance
Function 3 Time Interval 1.8 to 3.2 Min								
Perfluorooctanoic acid (C8)	5; 2	3.31 +/- *44%*	0.9 - 4800 (14)	(M+4)C8	0.013*[pg/g] + 0.002	0.994	18	≥LOQ within 20% tolerance
$^{13}C_4$-Perfluorooctanoic acid ((M+4)C8)	5; 2		Invariant		Matrix Internal Standard			
$^{13}C_8$-Perfluorooctanoic acid ((M+8)C8)	5; 2		0.9 - 4800 (14)	(M+4)C8	0.013*[pg/g] + 0.002	0.997	5	LOQ within 30% tolerance, >LOQ within 20%
Perfluorohexane sulfonate (PFHxS)	5; 2	2.0 +/- *44%*	5 - 4800 (13)	(M+4)C8	0.008*[pg/g] - 0.012	0.992	38	LOQ within 30% tolerance, >LOQ within 20%
6:2 Fluorotelomer unsaturated acid (6:2FTUCA)	5; 2			*(M+2)6:2FTUCA*				
$^{13}C_2$-6:2 Fluorotelomer unsaturated acid ((M+2)6:2FTUCA)	5; 2		Invariant		Matrix Internal Standard			
Perfluorononanoic acid (C9)	5; 2	4.3 +/- *44%*	0.9 - 4800 (14)	(M+5)C9	0.011*[pg/g] + 0.015	0.996	18	≥LOQ within 20% tolerance
$^{13}C_5$-Perfluorononanoic acid ((M+5)C9)	5; 2		Invariant		Matrix Internal Standard			
Function 4 Time Interval 2.4 to 3.4 Min								
Perfluoroheptane sulfonate (PFHpS)	5; 2	1.5 +/- *44%*	5 - 4800 (13)	(M+5)C9	0.003*[pg/g] + 0.002	0.973	56	≥LOQ within 30% tolerance
Function 5 Time Interval 2.9 to 4.4 Min								
Perfluorodecanoic acid (C10)	5; 2	6.8 +/- *44%*	5 - 4800 (13)	(M+2)C10	0.011*[pg/g] + 0.016	0.992	18	≥LOQ within 20% tolerance
$^{13}C_2$-Perfluorodecanoic acid ((M+2)C10)	5; 2		Invariant		Matrix Internal Standard			
Perfluorooctane sulfonate (PFOS)	5; 2	1.32 +/- *44%*	5 - 4800 (12)	(M+2)C10	0.004*[pg/g] + 0.003	0.990	38	≥LOQ within 20% tolerance
7:3 Fluorotelomer acid (7:3 FTCA)	5; 2							
8:2 Fluorotelomer unsaturated acid (8:2FTUCA)	5; 2		5 - 4800 (13)	(M+2)8:2FTUCA	0.010*[pg/g] + 0.005	0.992	18	≥LOQ within 20% tolerance
$^{13}C_2$-8:2 Fluorotelomer unsaturated acid ((M+2)8:2FTUCA)	5; 2		Invariant		Matrix Internal Standard			
Perfluoroundecanoic acid (C11)	5; 2	8.8 +/- *44%*	5 - 4800 (13)	(M+2)C11	-2.66e-7*[pg/g]^2 + 0.010*[pg/g] - 0.010	0.997	18	≥LOQ within 20% tolerance
$^{13}C_2$-Perfluoroundecanoic acid ((M+2)C11)	5; 2		Invariant		Matrix Internal Standard			
Function 6 Time Interval 4.0 to 15.0 Min	5; 2							
Perfluorododecanoic acid (C12)	5; 2	10.8 +/- *44%*	0.9 - 4800 (14)	(M+2)C12	-7.54e-7*[pg/g]^2+ 0.010*[pg/g] + 0.006	0.998	38	≥LOQ within 20% tolerance
$^{13}C_2$-Perfluorodecanoic acid ((M+2)C12)	5; 2		Invariant		Matrix Internal Standard			
10:2 Fluorotelomer unsaturated acid (10:2FTUCA)	5; 2		0.9 - 4800 (14)	(M+2)10:2FTUCA	0.010*[pg/g] + 0.006	0.991	18	≥LOQ within 20% tolerance
$^{13}C_2$-10:2 Fluorotelomer unsaturated acid ((M+2)10:2FTUCA)	5; 2		Invariant		Matrix Internal Standard			
Perfluorotridecanoic acid (C13)	5; 2	12.9 +/- *44%*	5 - 4800 (13)	(M+2)C12	-8.86e-7*[pg/g]^2 + 0.013*[pg/g] + 0.033	0.997	18	≥LOQ within 20% tolerance
Perfluorotetradecanoic acid (C14)	5; 2	16.9 +/- *44%*	5 - 4800 (13)	(M+2)C12	-9.72e-7*[pg/g]^2 + 0.013*[pg/g] + 0.002	0.996	18	≥LOQ within 20% tolerance

Italicized transitions are not monitored in 090428 MS Method

Table 4: Gas Chromatograph/Mass Spectrometer Parameters for Fluorotelomer Alcohols Analysis

Compound of Interest	Formula & Molecular Weight	Acronym	Ions in PCI[1] (m/z)	Ions in NCI[2] (m/z)	PCI TMSI Derivatives[1,3] (m/z)
1H,1H,2H,2H-perfluorooctan-1-ol	$CF_3(CF_2)_5CH_2\text{-}CH_2\text{-}OH$ 364	6:2 FTOH	365*[4], 327	304, 284	437
1H,1H,2H,2H-perfluorodecan-1-ol	$CF_3(CF_2)_7CH_2\text{-}CH_2\text{-}OH$ 464	8:2 FTOH	465*, 427	404, 384	537
1^{2}H,1^{2}H,2H,2H-^{13}C$_2$-perfluorodecan-1-ol	$CF_3(CF_2)_7{}^{13}CH_2\text{-}{}^{13}CD_2\text{-}OH$ 468	M8:2 FTOH	469*,431		
1H,1H,2H,2H-perfluorododecan-1-ol	$CF_3(CF_2)_9CH_2\text{-}CH_2\text{-}OH$ 564	10:2 FTOH	565*, 527	504,484	637
1^{2}H,1^{2}H,2H,2H-^{13}C$_2$-perfluorododecan-1-ol	$CF_3(CF_2)_9{}^{13}CH_2\text{-}{}^{13}CD_2\text{-}OH$ 568	M10:2 FTOH	569*, 531		
1H,1H,2H,2H-perfluorotetradecan-1-ol	$CF_3(CF_2)_{11}CH_2\text{-}CH_2\text{-}OH$ 664	12:2 FTOH	665*, 627		737
1H,1H,2H,2H-perfluorohexadecan-1-ol	$CF_3(CF_2)_{13}CH_2\text{-}CH_2\text{-}OH$ 764	14:2 FTOH	765*, 727		837
1-Perfluoroheptylethanol	$[CF_3(CF_2)_6](CH_3)\text{-}CH\text{-}OH$	7:2 sFTOH	415*, 395, 377		487
1-Perfluorononylethanol	$[CF_3(CF_2)_8](CH_3)\text{-}CH\text{-}OH$	9:2 sFTOH	515*, 577		687
1-Perfluoroundecylethanol	$[CF_3(CF_2)_{10}](CH_3)\text{-}CH\text{-}OH$	11:2 sFTOH	615*, 677		787
1-Perfluorotridecylethanol	$[CF_3(CF_2)_{12}](CH_3)\text{-}CH\text{-}OH$	13:2 sFTOH	715*, 777		887
2-(Perfluorooctyl) ethyl acrylate	$F(CF_2)_8CH_2\text{-}CH_2\text{-}O\text{-}$ $C(O)CH{=}CH_2$	8:2 FT-acrylate	519*		
1H,1H-perfluoroundecan-1-ol	$CF_3(CF_2)_9CH_2\text{-}OH$ 550	10:1 FTOH	551*, 531		623

1. Positive Chemical Ionization
2. Negative Chemical Ionization
3. Extracts were treated with trimethylsilylimidazole
4. Asterisk denotes the principal ion for quantitation

Table 5. GC-MS Integration and Optimization Parameters for Fluorotelomer Alcohols Analysis

Compound	Apex RT	Cycles /Sec	Standard Range (pg/mL)	Matrix Internal Standard	Equal-weighted calibration equation	Correlation Coefficient (r^2)	Limit of Quantitation (ng/g dry soil)[2]
Group 1 : Time Interval 4.5 to 8.5 min							
6:2 FTOH	7.986	2.37	200 – 5000 (4)	M10:2 FTOH	2.57×[pg/mL]	0.999	2.4
7:2s FTOH	8.133		200 – 5000 (4)	M10:2 FTOH	1.75×[pg/mL]	0.999	2.6
Group 2 : Time Interval 8.5 to 9.6 min							
M8:2 FTOH	9.239	1.21	100 – 5000 (5)	M10:2 FTOH	1.56×[pg/mL]	0.999	0.9
8:2 FTOH	9.256		100 – 5000 (5)	M10:2 FTOH	1.79×[pg/mL]	0.999	0.9
6:2 FT-acrylate	9.380		N.A.[1] (8:2 FT-acrylate)	M10:2 FTOH	2.45×[pg/mL]	0.999	0.9
9:2s FTOH	9.356		N.A. (8:2 FTOH)	M10:2 FTOH	1.79×[pg/mL]	0.999	0.9
Group 3 : Time Interval 9.6 to 10.6 min							
10:1 FTOH	9.758	0.98	200 – 5000 (4)	M10:2 FTOH	3.86×[pg/mL]	0.999	2.4
M10:2 FTOH	10.187		Invariant	-	Invariant	-	-
10:2 FTOH	10.207		200 – 5000 (4)	M10:2 FTOH	8.24×[pg/mL]	0.999	2.4
8:2 FT-acrylate	10.248		100 – 5000 (5)	M10:2 FTOH	2.45×[pg/mL]	0.999	0.9
11:2s FTOH	10.243		N.A. (10:2 FTOH)	M10:2 FTOH	1.75×[pg/mL]	0.999	2.4
Group 4 : Time Interval 10.6 to 12.4 min							
12:2 FTOH	10.973	1.21	N.A. (10:2 FTOH)	M10:2 FTOH	2.45×[pg/mL]	0.999	2.4
13:2s FTOH	10.959		N.A. (10:2 FTOH)	M10:2 FTOH	2.45×[pg/mL]	0.999	2.4
14:2 FTOH	11.580		N.A. (10:2 FTOH)	M10:2 FTOH	2.45×[pg/mL]	0.999	2.4
10:2 FT-acrylate	11.740		N.A. (8:2 FT-acrylate)	M10:2 FTOH	2.45×[pg/mL]	0.999	2.4

1) An analyte whose genuine standard was not available (N.A.), we quantified it using standard curve for similar compound in the homologous series.

2) Limit of Quantitation (LOQ) was defined as signal/noise ratio (S/N>3) and the lowest standard within ±30% of its theoretical value then multiplying by the average sample-dilution factors.

Table 6: Percent Deviation of Mean Back-Predicted Values for Perfluorinated Chemical Standard Curve Points for Subsurface-Soil Analytical Run [1]

Std Value pg/g	C6	C7	C8	C9	C10	C11	C12	C13	C14	S4	S6	S7	S8	U8	U10	M8C8
0.913	**108.1**	ND	ND	**53.3**	**42.4**	**151.9**	2.2	28.7	9.5	ND	ND	20.5	ND	**97.2**	16.8	**130.0**
4.502	8.3	12.2	9.4	-22.3	**-34.5**	**-54.8**	-12.6	**-31.7**	2.2	20.5	29.4	9.4	-25.6	7.7	13.3	-2.3
9.224	-6.2	17.9	0.6	6.2	-28.4	-25.2	-5.1	-4.3	-6.5	3.3	-8.9	-4.9	-20.0	**45.8**	7.3	-24.4
18.13	-6.9	7.8	6.2	-5.5	7.6	2.9	-1.1	-1.3	-4.6	-2.1	6.7	**-37.8**	13.3	-11.6	-9.1	1.2
38.07	4.8	1.3	-9.7	-19.9	13.9	-0.6	-4.1	-1.0	-0.4	-6.4	2.0	-1.9	9.7	-27.0	-2.9	-14.9
55.72	11.0	-5.9	-4.7	-1.4	6.5	2.3	5.4	6.4	7.4	5.6	-5.5	8.0	20.3	-27.7	1.3	-8.3
72.94	4.4	-2.1	-1.7	2.2	8.6	-2.9	3.9	-2.2	-10.8	-4.4	-1.5	16.8	0.3	13.0	-0.7	-12.2
93.02	-2.4	-4.6	4.8	-5.5	-0.8	9.5	6.2	2.1	2.0	-7.9	-5.3	-10.7	3.5	11.9	4.1	-5.7
231.5	-5.1	-6.3	-0.1	14.5	-7.1	2.2	2.4	0.8	2.1	-3.0	-1.3	16.3	0.9	-28.5	1.7	-7.4
483.9	-1.1	-9.2	-4.5	-1.6	3.0	-2.7	2.0	3.8	1.6	-4.7	-5.1	-0.1	-4.0	-16.4	11.7	-9.8
721	-3.1	-5.0	-1.8	-0.3	4.4	2.5	0.8	1.0	-6.2	-6.8	-9.7	-20.1	4.1	-23.8	4.0	-9.7
986.3	2.0	-7.0	0.8	-0.4	8.3	5.1	-0.3	-2.1	1.1	3.1	-5.2	7.2	24.4	-17.2	-7.3	-7.2
2380	1.6	-5.4	0.5	2.4	-4.8	4.2	-2.9	-1.8	2.8	0.0	-0.1	9.5	7.2	-20.7	7.6	-4.7
4763	-0.6	6.2	12.8	-1.3	-3.1	-3.5	3.3	3.7	-7.7	0.9	6.1	-4.2	-9.5	20.3	-4.5	7.2

[1] Bold values do not meet the quality criterion of being within 30% of the nominal standard concentration. Where present, the emboldened values fall below the limit of quantitation.

Table 7: Percent Deviation of Mean Back-Predicted Values for Fluorotelomer Alcohols Standard Curve Points

Std Value (pg/mL)	6:2 FTOH	7:2s FTOH	M8:2 FTOH	8:2 FTOH	10:1 FTOH	10:2 FTOH	8:2 FT-Acryl.
116	-17.3	-28.7	-13.3	3.4	-15.2	-11.2	-8.7
297	-18.7	-7.6	-2.2	-4.2	-5.8	-7.4	0.0
610	-18.1	2.4	-16.3	-13.1	-11.6	-8.7	-6.3
1205	-5.8	-8.9	-8.0	-4.9	-12.3	-10.3	-7.7
6085	3.8	4.0	3.7	3.5	4.1	3.6	3.6

Table 8: Proposed, Collected and Analyzed Samples Documenting Completeness

Sample Type	Planned	Collected	Analyzed	
			LC/MS/MS	GC/MS
Sludge-Applied Field Samples	30	30	30	30
Background Field Samples	4	4	4	4
Field Duplicates	2	2	2	2
Field Blanks	2	2	2	2
Field Reference Soil Samples	2	2	2	2
Laboratory Extract Spikes	5	5	5	0

Table 9. Summary of Field Blanks (pg/g dry soil) for Perfluorinated Chemical Analysis for Subsurface Soil Analytical Run

Sample Type	C6	C7	C8	M8C8*	C9	C10	C11	C12	C13	C14	S4	S6	S7	S8	U8:2	U10:2
**Field Blanks	<LOQ	<LOQ	*37*	85.3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Field Blank (SD)			*14*	2.3												
% Precision (RSD)			*38*	2.7												

*M8C8 is an internal standard and reported in % recovered

**Mean of 4 determinations. QC Soil is Cowart, previously analyzed in multiple studies

NA designates 'not applicable' because the target analyte was not detected

Italicized values fall outside acceptable tolerance of 15% for repeated extractions, but are reported for completeness

Table 10. Summary of Field Blanks and QC Soil (ng/g dry soil) for Fluorotelomer Alcohol Analysis

Sample Type	6:2 FTOH	7:2s FTOH	8:2 FTOH	9:2s FTOH	10:2 FTOH	11:2s FTOH	8:2 FT-acrylate	12:2 FTOH	13:2s FTOH	14:2 FTOH	% M8:2 Rec.[2]
Sand	< 2.4[1]	< 2.6	< 0.9	< 0.9	< 2.4	< 2.4	< 0.9	< 2.4	< 2.4	< 2.4	109
Cowart Soil	< 2.4	< 2.6	< 0.9	< 0.9	< 2.4	< 2.4	< 0.9	< 2.4	< 2.4	< 2.4	105

1) Mean values of two replicated extractions were less than its respective Limit of Quantitation

2) Mass-labeled 8:2 FTOH was spiked before an extraction to monitor overall extraction efficiencies.

Table 11. Summary of Background Field Soils (pg/g dry soil) for Perfluorinated Chemical Analysis for Subsurface-Soil Analytical Run

Sample ID	C6	C7	C8	C9	C10	C11	C12	C13	C14	S4	S6	S7	S8	U8:2	U10:2	% Rec'y M8C8
S101A2 Mean	117	ND	64	ND	<LOQ	ND	<LOQ	ND	ND	ND	<LOQ	ND	<LOQ	<LOQ	ND	83
σ	*27*		*6*													*1*
S101B2 Mean	243	110	226	<LOQ	<LOQ	ND	<LOQ	ND	ND	ND	<LOQ	<LOQ	248	ND	ND	79
σ	*9*	*15*	*6*										*32*			*6*

*M8C8 is a recovery internal standard

Table 12. Summary of Background Field Soils (ng/g dry soil) for Fluorotelomer Alcohol Analysis

Sample ID		**6:2 FTOH**	**7:2s FTOH**	**8:2 FTOH**	**9:2s FTOH**	**10:2 FTOH**	**11:2s FTOH**	**8:2 FT-acrylate**	**12:2 FTOH**	**13:2s FTOH**	**14:2 FTOH**	**% M8:2* Rec.**
S101A1	Mean	< 2.4	< 2.6	< 0.9	< 0.9	< 2.4	< 2.4	< 0.9	< 2.4	< 2.4	< 2.4	116.1
	% RD	-	-	-	-	-	-	-	-	-	-	*2*
S101A2	Mean	< 2.4	< 2.6	< 0.9	< 0.9	< 2.4	< 2.4	< 0.9	< 2.4	< 2.4	< 2.4	119.5
	% RD	-	-	-	-	-	-	-	-	-	-	*9*
S101B1	Mean	< 2.4	< 2.6	< 0.9	< 0.9	< 2.4	< 2.4	< 0.9	< 2.4	< 2.4	< 2.4	104.4
	% RD	-	-	-	-	-	-	-	-	-	-	*9*
S101B2	Mean	< 2.4	< 2.6	< 0.9	< 0.9	< 2.4	< 2.4	< 0.9	< 2.4	< 2.4	< 2.4	103.6
	% RD	-	-	-	-	-	-	-	-	-	-	*12*

*Mass-labeled 8:2 FTOH was spiked before an extraction to monitor overall extraction efficiencies.

Table 13. Summary of Duplicate Field Samples (ng/g dry soil) for Perfluorinated Chemical Analysis

Sample ID	C6	C7	C8	M8C8*	C9	C10	C11	C12	C13	C14	S4	S6	S7	S8	U8:2	U10:2
090326-S17-1aA	19	38	190	4	91	684	199	396	65	70	1	1	2	127	0	1
0903261-S17-1aAD	27	53	269	4	132	986	233	526	81	114	0	3	0	189	0	1
Rel % Diff	35	35	34	11	37	36	16	28	21	48	NA	117	NA	39	NA	NA
09032-S1-4E-4	7	30	153	5	76	509	133	245	40	52	0	4	1	177	0	0
090326-S1-4ED-4	11	49	264	7	110	683	171	349	61	90	0	4	0	245	0	1
Rel % Diff	38	47	53	23	37	29	25	35	41	53		9	NA	32		200

*M8C8 is an internal standard

Table 14. Summary of Duplicate Field Samples (ng/g dry soil) for Fluorotelomer Alcohols Analysis

Sample ID		6:2 FTOH	7:2s FTOH	8:2 FTOH	9:2s FTOH	10:2 FTOH	11:2s FTOH	8:2 FT-acrylate	12:2 FTOH	13:2s FTOH	14:2 FTOH	% Rec.[2]
S17-1aA	Sub-mean	< 2.4 [1]	28.7	49.5	16.8	56.0	13.4	< 0.9	31.5	4.4	31.5	113.0
	% RD	-	3.6	17.6	22.1	14.7	17.7	-	27.1	3.9	12.8	3.3
S17-1aA-Duplicate	Sub-mean	< 2.4	43.1	80.1	25.0	95.8	20.5	1.0	50.3	6.9	52.4	109.6
	% RD	-	0.4	16.9	4.5	18.6	4.3	-	3.3	12.1	9.6	0.6
	Mean	< 2.4	35.9	64.8	20.9	75.9	17.0	< 0.9	40.9	5.7	41.9	111.3
	% RD	-	40.3	47.3	38.9	52.5	41.4	-	45.9	43.4	49.8	3.1
S1-4E	Sub-mean	< 2.4	12.7	27.7	9.0	30.8	4.5	< 0.9	10.9	< 2.4	10.2	107.4
	% RD	-	2.6	9.2	26.5	23.2	12.6	-	18.0	-	2.1	0.6
S1-4E-Duplicate	Sub-mean	< 2.4	27.8	42.7	16.2	52.1	9.6	< 0.9	21.8	3.3	26.3	109.6
	% RD	-	1.1	5.6	6.4	1.9	13.5	-	0.2	13.4	0.6	2.9
	Mean	< 2.4	20.3	35.2	12.6	41.5	7.1	< 0.9	16.4	-	18.3	108.5
	% RD	-	74.3	42.7	57.3	51.5	72.5	-	66.9	-	88.1	2.1

1) Mean values of two replicated extractions were less than its respective Limit of Quantitation

2) Mass-labeled 8:2 FTOH was spiked before an extraction to monitor overall extraction efficiencies.

Table 15. Standard Addition of 100pg of Perfluorinated Chemicals to Selected Field Samples

Sample ID	pg C6	pg C7	pg C8	pg M8C8	pg C9	pg C10	pg C11	pg C12	pg C13	pg C14	pg S4	pg S6	pg S7	pg S8	pg U8	pg U10
	LCMSMS Analyzed Added Mass of Analyte															
S14-1A1-SA	111	104	106	104	78	113	95	111	135	136	113	100	124	167	109	116
S14-1A2-SA	118	116	106	105	118	135	107	103	116	124	119	109	142	119	114	112
S14-1A3-SA	117	99	111	107	96	107	98	115	116	114	115	109	111	98	107	131
S14-10B1-SA	115	115	127	109	93	124	84	90	109	115	103	121	119	116	109	93
S14-10C1-SA	107	131	146	116	101	167	65	115	142	130	131	109	127	189	118	109
	Actual Added Mass of Analyte															
	pg C6	pg C7	pg C8	pg M8C8	pg C9	pg C10	pg C11	pg C12	pg C13	pg C14	pg S4	pg S6	pg S7	pg S8	pg U8	pg U10
S14-1A1-SA	104	104	104	104	104	104	104	104	104	104	104	104	104	104	104	104
S14-1A2-SA	107	107	107	107	107	107	107	107	106	106	106	106	106	106	106	106
S14-1A3-SA	109	109	109	109	109	109	109	109	108	108	108	108	108	108	108	108
S14-10B1-SA	107	107	107	107	107	107	107	107	107	107	107	107	107	107	107	107
S14-10C1-SA	107	107	107	107	107	107	107	107	107	107	107	107	107	107	107	107
	Percent Recovery of Added Analyte via LCMSMS Analysis															
	C6	C7	C8	M8C8	C9	C10	C11	C12	C13	C14	S4	S6	S7	S8	U8	U10
S14-1A1-SA	107	100	102	100	75	108	92	106	130	131	109	96	119	161	105	112
S14-1A2-SA	111	109	99	99	111	127	100	97	109	116	111	102	134	112	107	105
S14-1A3-SA	108	91	102	99	88	99	90	106	107	105	106	100	102	90	98	120
S14-10B1-SA	107	107	118	102	87	116	79	84	102	108	96	113	112	109	102	88
S14-10C1-SA	100	122	136	108	95	156	61	108	133	121	122	102	119	177	111	102
Avg % Rec'y	107	106	112	102	91	121	84	100	116	116	109	103	117	130	105	105
SD % Rec'y	4.0	11.6	15.8	4.0	13.0	21.9	15.1	9.9	14.4	10.5	9.4	6.3	11.6	37.2	4.6	12.2

SA = Standard Addition of 50uL of 2500 pg/g mix of each analyte to 500uL of 200X diluted sample, which is equivalent to 100pg increment.

Values below LOQ assumed to be zero for calculation purposes.

Table 16. Precision of Repeated Injections for Fluorotelomer Alcohols (n=12)

Compound	6:2 FTOH	7:2s FTOH	8:2 FTOH	9:2s FTOH	10:2 FTOH	11:2s FTOH	8:2 FT-acrylate	12:2 FTOH	13:2s FTOH	14:2 FTOH	M8:2 FTOH
Mean % RD[1]	-[2]	7.0	9.5	10.0	10.6	13.8	-	15.5	23.3	14.5	5.7
s.d. (%)	-	5.8	4.2	6.7	7.9	10.5	-	5.8	14.2	7.1	4.7

1) Selected samples (n=12) were injected twice to evaluate the precision of GC/MS performance. The % RD (% relative difference) was calculated for each extract and, subsequently, mean and standard deviation (s.d.) were reported.

2) Values were less than its respective Limit of Detection.

Table 17. Concentrations of Perfluorinated Acids and Sulfonates in Subsurface Soils from Decatur, AL (pg/g dry soil)*

Sample ID	C6	C7	C8	C9	C10	C11	C12	C13	C14	S4	S6	S7	S8	U8:2	U10:2
S14-1A2	908	2140	21895	8184	5013	489	459	<LOQ	140	<LOQ	<LOQ	<LOQ	7373	<LOQ	<LOQ
S14-1A3	2510	5021	13567	1030	2046	613	1048	198	314	<LOQ	<LOQ	<LOQ	1780	<LOQ	<LOQ
S14-10B2	1147	3201	79620	36149	24063	3060	1606	266	251	<LOQ	227	264	20991	<LOQ	<LOQ
S14-10B3	1417	5247	9812	249	555	205	519	293	901	<LOQ	152	<LOQ	602	<LOQ	<LOQ
S14-10C2	4285	6240	15691	2594	5074	496	411	<LOQ	<LOQ	<LOQ	161	<LOQ	3390	<LOQ	<LOQ
S14-10C3	1917	2046	3167	231	343	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	240	<LOQ	<LOQ

*Mean values of two replicated extractions; LOQ = Limit of Quantization

Table 18. Concentrations of Fluorotelomer Alcohols in Subsurface Soil Samples from Decatur, AL (ng/g dry soil)*

Sample ID	6:2 FTOH	7:2s FTOH	8:2 FTOH	9:2s FTOH	10:2 FTOH	11:2s FTOH	12:2 FTOH	13:2s FTOH	14:2 FTOH	8:2 FTAc	% M8:2 FTOH Rec.
S14-1A2	<2.4	<2.6	0.4	<0.9	<0.9	<0.9	<2.4	<2.4	<2.4	<0.9	101.9
S14-1A3	<2.4	<2.6	0.8	<0.9	<0.9	<0.9	<2.4	<2.4	<2.4	<0.9	100.0
S14-10B2	<2.4	<2.6	1.0	<0.9	<0.9	<0.9	<2.4	<2.4	<2.4	<0.9	96.9
S14-10B3	<2.4	<2.6	<0.9	<0.9	<0.9	<0.9	<2.4	<2.4	<2.4	<0.9	97.4
S14-10C2	<2.4	<2.6	<0.9	<0.9	<0.9	<0.9	<2.4	<2.4	<2.4	<0.9	108.8
S14-10C3	<2.4	<2.6	1.2	<0.9	<0.9	<0.9	<2.4	<2.4	<2.4	<0.9	107.5

*Mean values of two replicated extractions