Study Title

Hydrolysis Reactions of 2-(*N*-Methylperfluoroctanesulfonamido)-Ethyl Alcohol (*N*-MeFOSE Alcohol)

Data Requirement:

Based on OPPTS: 835.2110

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Study Completion Date

March 30, 2001

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Project Identification

3M Laboratory Report No: W1880

Total Number of Pages

71

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Statement of Non-Compliance

Study Title: Hydrolysis Reactions of 2-(*N*-Methylperfluoroctanesulfonamido)-Ethyl Alcohol (*N*-MeFOSE Alcohol)

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This study does not fully comply with the requirements of the US EPA Good Laboratory Practices (GLP) Standards at 40 CFR Part 792 (TSCA). However, many GLP standards were used in the development of the analytical method (Appendix A), and the quality assurance procedures followed in this study were based on the practices described in the GLP documentation.

This is a revised report in that the statistics on the study data and the discussion were changed from the initial study report. Changes to these interpretive sections were made to better represent the experimental results of the study.

Study Director — Date 03/30/01

Sponsor Representative — Date 03/30/01

Quality Assurance Statement

Study Title: Hydrolysis Reactions of 2-(*N*-Methylperfluoroctanesulfonamido)-Ethyl Alcohol (*N*-MeFOSE Alcohol)

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The following table provides details of the audits performed by the 3M Environmental Laboratory Quality Assurance Unit (QAU).

Inspection Dates	Phase	Date Reported to	
		Management	Study Director
10/4-6/2000	Data and Draft Report	10/06/00	10/06/00
3/19, 20/2001	Draft Report	3/21/01	3/121/01

Casey Howell — Apr 2, 2001

QAU Representative — Date

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Location of Archives

The 3M Environmental Laboratory will retain the original data documents and digital copies of the original data related to this work for at least 10 years following the effective date of any related final ruling. Information may be obtained through written inquiry addressed as follows:

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Summary

We report here the results of our study of the hydrolysis of 2-(*N*-methylperfluorooctane-sulfonamido)-ethyl alcohol (hereafter, *N*-MeFOSE Alcohol). Our methods are described below and in Appendix A to this work; our results are based on the observed concentrations of *N*-MeFOSE alcohol and its potential hydrolysis product perfluorooctane sulfonate (PFOS) in buffered aqueous solutions as a function of time. The chosen analytical technique was high performance liquid chromatography with mass spectrometry detection (HPLC/MS). Tables 1 and 2 summarize the results of the study.

During this study, we prepared and examined samples at six different pH levels from 1.5 to 11.0 over a period of 49 days, and our results indicate no dependence of the degradation rate of *N*-MeFOSE alcohol on the sample pH level. Our results based on the *N*-MeFOSE alcohol concentrations, pooled over the observed pH levels, are presented in Table 1.

Table 1. Summary of Results Based on *N*-MeFOSE Alcohol Concentrations

Observed Rate Constant at 50° C (day^{-1})	Calculated Rate Constant at 25° C (day^{-1})	Calculated Half Life at 25° C (years)	Calculated (2σ) Half Life Range at 25° C (years)
0.0030	0.00030	6.3	3.8 to 19.4

We also monitored the concentration of one of the potential hydrolysis products (perfluorooctane sulfonate, PFOS), but never observed this compound at levels above its limit of quantification (LOQ, equal to 12.8 ng/mL). Assuming PFOS to be the only hydrolysis product of *N*-MeFOSE Alcohol, this LOQ (and other experimental data) provide a second estimate of the *N*-MeFOSE alcohol half-life, presented in Table 2.

Table 2. Summary of Results Based on PFOS Limit of Quantification

Maximum Possible Rate Constant at 50° C (day^{-1})	Maximum Calculated Rate Constant at 25° C (day^{-1})	Calculated Half Life at 25° C (years)
7.0×10^{-4}	7.0×10^{-5}	≥ 27

According to the data available from this study, the half-life estimate of Table 2 represents the minimum possible half-life of the compound *N*-MeFOSE alcohol under the assumption that it hydrolyzes to form *only* the compound PFOS.

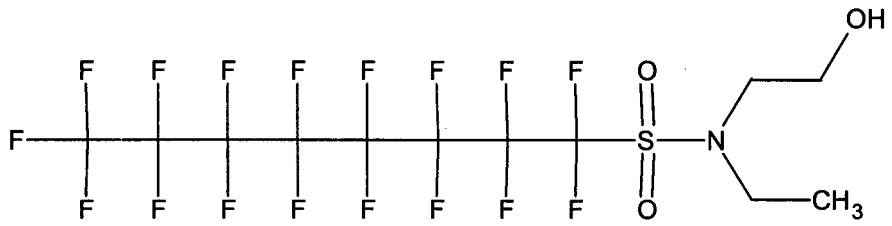
Introduction

Three primary chemical routes of environmental degradation are hydrolysis, photolysis, and biodegradation. Studies of these routes provide information on the environmental persistence of both the "parent" compounds and their reaction products, and are ideally carried out over the range of chemical conditions pertinent to both environmental and metabolic processes.

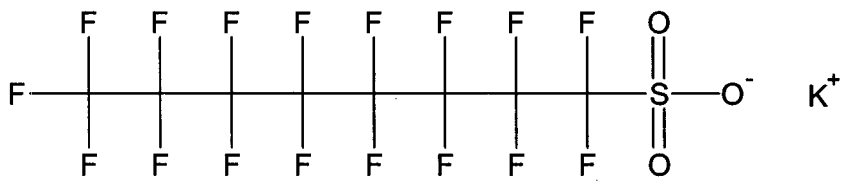
The hydrolysis of *N*-MeFOSE alcohol (or, more generally, its degradation in the presence of H_2O) is addressed in this report. Structures of the "parent" compound *N*-MeFOSE alcohol and the potassium salt of possible hydrolysis product perfluorooctane sulfonate (PFOS) are illustrated in Figure 1.

Figure 1. Structures of *N*-MeFOSE Alcohol and the Potassium Salt of PFOS

***N*-MeFOSE Alcohol**



Potassium Salt of PFOS



Summary of Kinetics Model

A full mathematical description of the kinetics model employed in this study is presented in Appendix B. The study data allow two independent estimates of the hydrolytic half-life of *N*-MeFOSE Alcohol.

The first estimate (see Table 1) is based on the observed degradation of the "parent" compound *N*-MeFOSE alcohol in dilute, appropriately buffered aqueous solutions. Equation 1 describes the estimated half-life $\left(\hat{T}^{1/2}_{p}\right)_1$ in terms of the estimated total parent hydrolysis rate $\hat{k}_P$ (see Appendix B, Equation B10):

$$\left(\hat{T}^{1/2}_{p}\right)_1 = \frac{\ln(2)}{\hat{k}_P} \qquad \text{Eq. 1}$$

We determined the quantity $\hat{k}_P$ from the experimental data as described in Appendix B. At each pH level, we used the earliest study data meeting the data quality objectives to determine the relevant concentration ratios (see Equation B8).

The measured concentrations of the potential hydrolysis product PFOS (also obtained during the experiments described here) provide a second estimate (see Table 2) of the parent half-life. During the course of this study, we did not detect PFOS above its limit of quantitation (LOQ), and related studies[1] show that PFOS is itself hydrolytically stable. Assuming also that PFOS is the only hydrolytic product of the parent compound *N*-MeFOSE alcohol, these PFOS analyses provide the following estimate $\left(T^{1/2}_{p}\right)_2$ of the minimum *N*-MeFOSE alcohol half-life (see Equations B32 and B33):

$$\left(T^{1/2}_{p}\right)_2 \geq \frac{\Delta t\ [P_0]\ln(2)}{A^{LOQ}_{PFOS}}\ , \qquad \text{Eq. 2}$$

where

$[P_0]$= the initial *N*-MeFOSE alcohol molar concentration,

Δt = the time interval over which the study was conducted (49 days), and

A^{LOQ}_{PFOS} = the molar limit of quantitation for the compound PFOS.

All the samples used in this study were maintained at a reaction temperature of 50° C. The quoted results, valid for the reaction temperature of 25° C, were calculated from our experimental results according to methods described in Appendix B (Eq. B38 and B39).

Materials and Methods

Details of the characteristics of the test materials, sample preparation techniques, and analytical methods are presented in Appendix A (ETS-8-178.0, "Preparation of 2-(*N*-Methylperfluoroctanesulfonamido) Ethyl Alcohol (*N*-MeFOSE Alcohol) Hydrolysis Samples and Analysis by High Performance Liquid Chromatography with Mass Spectrometry Detection.") A summary of these items is provided below, as well as a description the known deviations from the procedures of Appendix A. 3M prepared and analyzed the samples included in this study between March 13 and October 5, 1999.

Chemical Characterizations

Table 3 describes the sources and properties of the materials used in this work. These materials were used to prepare both the samples and the quantitative standards used to quantify them. For this reason, and because Equation B8 (see Appendix B) involves only ratios of parent concentrations, the resulting rate and half-life estimates are largely independent of the material purity levels.

Table 3. Characterizations of Test and Reference Substances

	N-EtFOSE Alcohol[c]	PFOS	THPFOS[b]	*N*-MeFOSE Alcohol
Source	3M Specialty Chemistry	3M Specialty Chemistry	ICN Biomedicals	3M ICP/PCP Division
Chemical Lot Number[a]	TN-A-1284	TN-A-2130 SD108	TN-A-1339 (Lot #59909, SD028)	TN-A-1282 SD 015
Physical Description	Off-white powder	White powder	Light brown powder	Amber waxy solid
Molecular Weight (gm mole^{-1})	570.9	498.9	428.0	556.9

[a] The "S" and "TNA" designations are based on reference numbers in two redundant databases maintained by 3M.
[b] 3, 3, 4, 4, 5, 5, 6, 6, 7, 7, 8, 8, 8-tridecafluorooctane sulfonic acid.
[c] 2-(*N*-ethylperfluoroctanesulfonamido)-ethyl alcohol.

Sample Preparation

We prepared three 1.0-mL aqueous buffer samples (a sample, a duplicate, and a "matrix spike") at each of six pH levels (1.5, 3, 5, 7, 9 and 11) for analysis at eight time intervals (0, 7, 14, 21, 28, 35, 42 and 49 days). Buffered solutions containing the analyte *N*-MeFOSE alcohol and THPFOS (3, 3, 4, 4, 5, 5, 6, 6, 7, 7, 8, 8, 8-tridecafluorooctane sulfonic acid), the latter serving as a surrogate for the compound PFOS, formed the basis of all these samples. The chosen buffer solutions are described fully in Appendix A.

We prepared all the samples simultaneously, and placed all but the "Day 0" samples in an orbital incubator/shaker maintained at 50° (± 3°) C. After at least three minutes of agitation, we diluted the "Day 0" samples 10:1 with methanol, added solutions of the internal standard 2-(*N*-ethylperfluoroctanesulfonamido)-ethyl alcohol (*N*-EtFOSE

alcohol), spiked the samples (as required) with a solution of *N*-MeFOSE alcohol, and then refrigerated them. After the appropriate incubation times, subsets of the sample vials were removed from the incubator and then diluted, spiked, and stored as described immediately above. Except during the relatively short periods of time required to prepare them, the samples were shielded from light.

In all the samples, the resulting THPFOS and *N*-EtFOSE alcohol concentrations were 403 and 311 ng/ml, respectively. In the samples spiked with the anlatye *N*-MeFOSE alcohol, the resulting analyte levels were 221 ng/ml above those in the unspiked samples.

Six calibration standards containing *N*-MeFOSE alcohol (78.9 to 789 ng/ml), *N*-EtFOSE alcohol (311 ng/ml), THPFOS (403 ng/ml) and PFOS (3.1 to 94 ng/ml) served as the quantitative basis of the study. All these standards were prepared at the appropriate pH levels using the chosen buffer solutions (see Appendix A.)

Sample Analysis

The equipment we used for the HPLC/MS analysis was a Hewlett Packard model 1100 equipped with a Dionex IonPac® NG-1 HPLC column (aqueous ammonium acetate/methanol solvent gradient) and an ALS Model G1322A degassing module. An ALS Model G1315A column heater maintained the column temperature at 40 °C, a quaternary pump supplied a column flow rate of 0.3 mL/min, and an ALS Model G1313A auto-sampler provided 5 µL sample injections. The detector was a Hewlett Packard MSD mass spectrometer, operated in negative-mode electrospray ionization mode; anions of PFOS, THPFOS, and acetate adducts of *N*-MeFOSE alcohol and *N*-MeFOSE alcohol were detected at the charge-to mass ratio (m/z) values 499, 427, 616, and 630 respectively.

We processed the resulting data using the computer program *HP ChemStation for LC* (Rev.A.06.0). The calibration curves and analytical results are based on the measured area ratios for the analyte/internal standard pairs; *N*-EtFOSE alcohol served as the internal standard for *N*-MeFOSE alcohol, and THPFOS served as the internal standard for PFOS. Additional analytical details, including the gradient elution program, instrument and detector parameters, and performance specifications, are presented in Appendix A.

Deviations

The gradient parameters and column temperature were incorrectly entered on the instrument parameters spreadsheet dated 12/17/99. The correct parameters are listed in the data acquisition method file "FOSESIM.m."

Through either human or mechanical error, samples for "Day 42," pH 1.5 were not analyzed.

The mass spectrometer detector gain was set to 2.0 for samples analyzed on 12/20/99. Earlier sets were analyzed with gain = 1.0.

The pH 3 data were reanalyzed using the correct quantitative ion at m/z = 616; an earlier analysis incorrectly employed the "monitor" ion value m/z = 617.

The actual concentration of the THPFOS solution added as a surrogate (21,030 µg/mL) was incorrectly entered as 20,130 µg/mL in some documents. This error has no effect on the accuracy of the analytical results, but has been noted on the reagent preparation logs.

The analytical results for one set of samples, intended for the pH 1.5, "Day 49" analysis, indicated much lower concentrations of *N*-MeFOSE than for any other similar sample prepared for this study. All other analytical results were quite consistent, so it is highly likely that these samples were not properly prepared.

Incorrect amounts of either the surrogate or internal standard solutions were added to the following pH = 1.5 samples: MFA-002, 127, 128, and 129. The same is true of the following pH = 11 samples: MFA-016, 017, 018, 034, 035, 036, 124, 125, and 126. In many cases, these samples were rejected on the basis of the data quality objectives (see the section immediately below). The resulting analytical values for the surrogate and internal standards were also excluded from evaluations of the consistency of the related results (see the "Data Summary and Discussion" section below).

Results and Discussion

Data Quality Objectives (DQO's)

Below is a brief description of the data quality objectives applied in this study. A full description is presented in Appendix A. With the exceptions of the anomalous results noted below, all the DQO's were met. Appendix C presents the results for each sample set, organized by pH level.

Calibrations. The minimum acceptable coefficient of determination (r^2) for linear fits to calibration data is 0.990. The acceptance criterion for individual calibration points is that their values fall within ± 25% of the linear fit value; data outside this range are excluded and the calibration curve is recalculated. No more than two points may be rejected from a calibration data set. Data for the high or low calibration standards may be rejected, though this results in a smaller effective calibration range.

Continuing Calibration Verification (CCV). Identical calibration samples are examined at the beginning and end of each sample run. Results of the second calibration run may not deviate by more than ± 25% of the first run for any analyte. The average results of the calibration runs are used to calculate the analyte concentrations.

Matrix Spikes. The acceptable percent spike recovery range is 75% to 125%. Analyte specificity is demonstrated by acceptable analyte spike recoveries.

Sample Duplicates. Duplicate pairs with relative percent deviation (RSD) greater than 25% may be accepted at the analyst's discretion, but must be noted.

Solvent Blanks. Concentration results for solvent blanks may exceed neither 5% of the highest calibration standard nor 25% of the lowest calibration level.

System Suitability. Suitability was demonstrated by either an abbreviated mass-to-charge (m/z) check-tune or performance of a full auto-tune routine.

Anomalous Analytical Results

Calibrations. Of the 144 calibration results obtained, 13 individual values failed to meet the stated DQO and were rejected. We rejected fewer than two values for any particular calibration run and compound.

Spike Recoveries. Results for the following sample pairs failed to meet this data quality objective and are excluded from the following analyses: MFA-127-128 ("Day 49", pH = 1.5); MFA-004-005 ("Day 0", pH = 3.0); MFA-016-017 ("Day 0", pH = 11); MFA-034-035 ("Day 7", pH = 11); and MFA-124-125 ("Day 42", pH = 11).

Sample Duplicates. The following sample pairs failed to meet this data quality objective and are excluded from the following analyses: MFA-001-002 ("Day 0", pH = 1.5) and MFA-040-041 ("Day 14", pH = 3.0).

Solvent Blanks. PFOS results for two of the 12 solvent blanks (the initial blanks performed at pH levels 3.0 and 7.0) exceeded 25% of the calibration standards values below 15.7 ng/ml. As a result, the detection limit for PFOS quoted in this study is 15.7 ng/ml (see Appendix A).

Statistical Methods and Calculations

Using functions provided in Microsoft® Excel® software, we calculated means, standard deviations, and first-order rate constants (see Appendix B, Equation B8) for various subsets of the acquired data. Our linear regressions included the determination of constant terms, that is, we did not force the regression fits to pass through the origin.

As described in Appendix B (Equations B38 and B39), rates measured at 50°C were extrapolated to 25°C by dividing by a factor of 10; this approximation is valid for reactions with Arrhenius heats of activation near 18 Kcal/mole.[2]

Data Summary and Discussion

The LOQ is defined as the concentration of the lowest (accepted) standard in the calibration set for which the known concentration exceeds 400% of the indicated solvent blank level (see Appendix A). During this study, the LOQ's for *N*-MeFOSE alcohol and PFOS were 78.9 ng/mL and 15.7 ng/mL, respectively.

Excluding those samples noted above in the "Deviation" section, our results for the surrogate compound (THPFOS) and the internal standard (*N*-EtFOSE alcohol) were quite consistent throughout the study. The percent relative standard deviations of the measured THPFOS values, calculated for each pH level, ranged from 2.1% to 10.2%. The corresponding values for *N*-EtFOSE alcohol ranged from 4.7% to 11.3%.

Figure 2 illustrates the observed *N*-MeFOSE alcohol concentrations at the six pH levels (at 50°C), and Table 4 presents the results of the slope determinations (see Equation B8) for the same data.

Table 4. Observed (50° C) Degradation Rates of *N*-MeFOSE Alcohol in Aqueous Buffered Solutions and at Various pH Levels.

PH	Observed Rate (day^{-1})	Percent (2σ) Rate Uncertainty
1.5	4.26×10^{-3}	67
3.0	-4.42×10^{-4}	582
5.0	5.68×10^{-3}	25
7.0	4.83×10^{-3}	24
9.0	3.97×10^{-3}	34
11	2.98×10^{-3}	25

These degradation rates, with the exception of the pH = 3.0 data, are generally well determined, with percent relative 2σ (95% confidence) uncertainties in the range 24% to 67%. The data do not indicate any clear dependence of the degradation rate on the sample pH.

In the absence of a clear trend relating the degradation rate to sample pH, it is appropriate to "pool" the data from all pH levels and to determine the degradation rate using the entire data set. Figure 3 illustrates the results of this pooled analysis according to Equation 1, and Table 5 summarizes the results of the analysis.

Table 5. Degradation Rate and Half Life of *N*-MeFOSE Alcohol in Aqueous Buffered Solutions Using Data Pooled Over pH Levels.

Observed Rate Constant at 50° C (day^{-1})	Percent (2σ) Rate Constant Uncertainty at 50° C (day^{-1})	Calculated Rate Constant at 25° C (day^{-1})	Calculated Half Life at 25°C (years)	Calculated (2σ) Half Life Range at 25° C (years)
0.0030	66	0.00030	6.3	3.8 to 19.4

Figure 2. Observed *N*-MeFOSE Alcohol Degradation for Various pH Levels

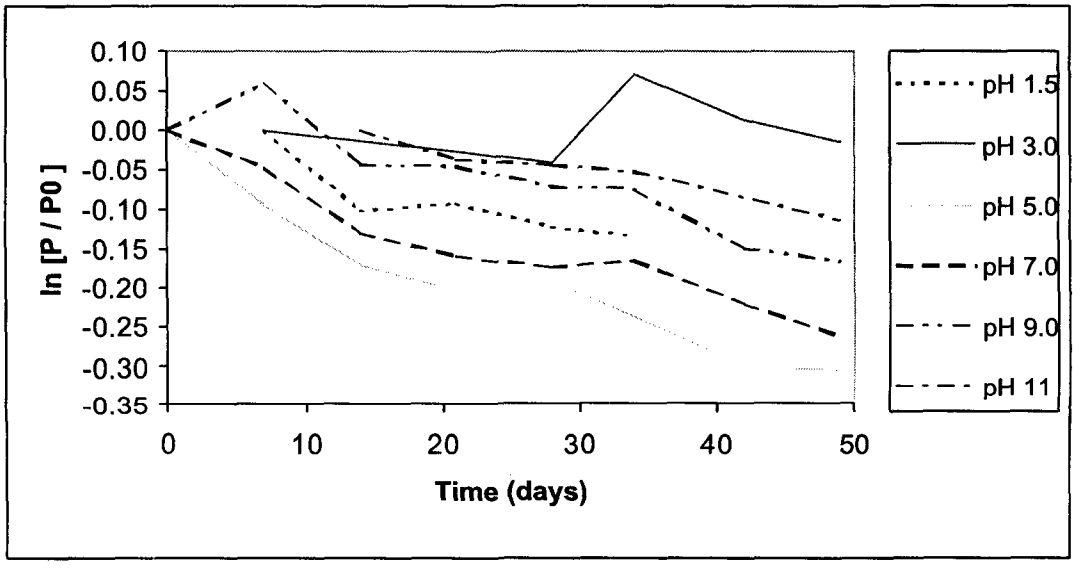
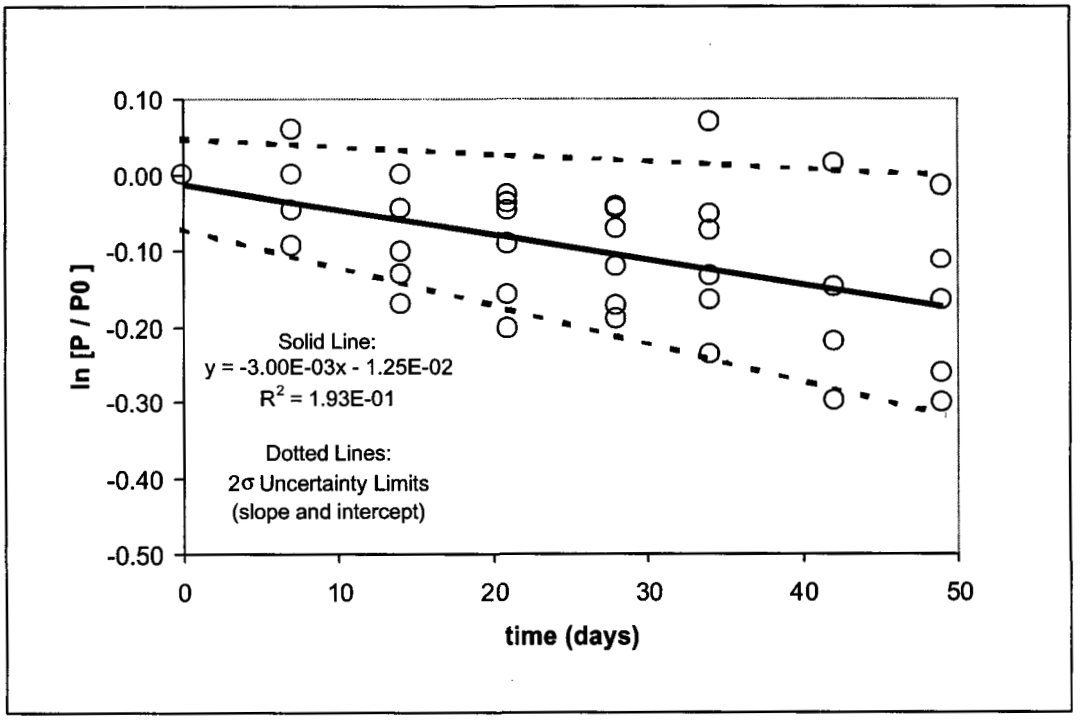


Figure 3. Pooled *N*-MeFOSE Alcohol Data and Slope Regression.



We also monitored the concentration of the hydrolysis product perfluorooctane sulfonate (PFOS), but never observed this compound at levels above its limit of quantification (LOQ, equal to 15.7 ng/mL). The initial *N*-MeFOSE alcohol concentration (473 ng/ml) and the PFOS LOQ provide a second estimate of the *N*-MeFOSE alcohol half-life (see in Appendix B, Equations B32 and B33). The maximum degradation rate is given by Equation 3:

$$k_P \le (k_P)_{max} = \frac{1}{P_0 \, \Delta t} \sum_{m=1}^{N} A_m^{LOQ} \qquad \text{Eq. 3}$$

and the minimum half-life is given by Equation 4

$$\left(T_P^{1/2}\right)_2 \ge \frac{\Delta t \, [P_0] \ln(2)}{A_{PFOS}^{LOQ}} \, . \qquad \text{Eq. 4}$$

We note that in both Equations 3 and 4, the initial *N*-MeFOSE alcohol concentration (P_0) and the PFOS LOQ (A_{PFOS}^{LOQ}) are molar quantities. Table 6 presents the results of the calculation.

Table 6. Degradation Rate and Half Life of *N*-MeFOSE Alcohol in Aqueous Buffered Solutions Based on PFOS Limit of Quantification

Δt (days)	$[P_0]$ (nm/ml)	A_{PFOS}^{LOQ} (nm/ml)	Maximum Observed Rate at 50° C (day^{-1})	Maximum Calculated Rate at 25° C (day^{-1})	Calculated Half Life at 25° C (years)
49	0.85	0.029	7.0×10^{-4}	7.0×10^{-5}	≥ 27

Conclusions

We have performed a study of the aqueous hydrolytic degradation 2-(*N*-ethylperfluorooctane-sulfonamido)-ethyl alcohol (*N*-MeFOSE Alcohol). Six different pH levels were included in the study, which were carried out at 50°C and extrapolated to 25°C. Our results based on direct observation of the *N*-MeFOSE alcohol concentration indicate no clear dependence of the degradation rate of *N*-MeFOSE alcohol on pH. From the data pooled over the six pH levels, we estimate that the hydrolytic half-life of *N*-MeFOSE alcohol at 25°C lies between 3.8 and 19.4 years, with the most likely value of 6.3 years.

The concentration of the compound PFOS, a likely hydrolytic product of *N*-MeFOSE Alcohol, was monitored throughout the study, but remained undetected above its limit of quantification (LOQ = 15.7 ng/mL). Using the LOQ for PFOS and the initial *N*-MeFOSE alcohol concentration (473 ng/ml), and assuming PFOS is the only hydrolytic product of *N*-MeFOSE Alcohol, the data indicate that the hydrolytic half-life of *N*-MeFOSE alcohol at 25°C is greater than or equal to 27 years.

References

[1] "Fate, Transport and Transformation Test Guidelines: 835.2110: Hydrolysis as a Function of pH," U.S. EPA Office of Prevention, Pesticides and Toxic Substances, publication number 712-C-98-057, January 1998.

[2] "Experimental Physical Chemistry", F. Daniels, et al., McGraw Hill Book Co. (New York), p. 131, 1962.

Signatures

Thomas L. Hatfield, Ph.D., Special Projects Team Leader — Date: 03/30/01

William K. Reagen, Ph.D., Laboratory Management — Date: 03/30/01

Appendix A: Analytical Method

ETS - 8-178.0, "Preparation of 2-(N-Methylperfluorooctanesulfonamido) Ethyl Alcohol (N-EtFOSE Alcohol) Hydrolysis Samples and Analysis by High Performance Liquid Chromatography with Mass Spectrometry Detection."

This Appendix presents the analytical method employed in this study.

3M ENVIRONMENTAL LABORATORY

METHOD

PREPARATION OF 2-(N-METHYLPERFLUOROOCTANESULFONAMIDO)-ETHYL ALCOHOL (N-MEFOSE ALCOHOL) HYDROLYSIS SAMPLES AND ANALYSIS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH MASS SPECTROMETRY DETECTION

Method Number: ETS-8-178.0

Adoption Date: 9/12/00

Revision Effective Date:

Exact Copy of Original

JST 9/13/00

Initial Date

Approved by:

Laboratory Manager — Date: Sept 12, 00

Team Leader — Date: Sept 14, 00

1.0 SCOPE AND APPLICATION

1.1 This procedure defines the steps for analysis of 2-(N-methylperfluorooctanesulfonamido)-ethyl alcohol (N-MeFOSE alcohol) hydrolysis samples by high performance liquid chromatography (HPLC) with mass spectrometry (MS) detection and quantitation. It is based on EPA OPPTS: 835.2110 (Reference 18.1). N-MeFOSE alcohol and the potential hydrolysis product perfluorooctane sulfonate (PFOS anion) are detected and quantified by this method. Internal standards for the method are 2-(N-ethylperfluorooctanesulfonamido)ethyl alcohol (N-EtFOSE alcohol) and 3, 3, 4, 4, 5, 5, 6, 6, 7, 7, 8, 8, 8-tridecafluorooctane sulfonic acid anion (THPFOS). This method may also be used to screen for the presence of FOSA (perfluorooctanesulfonamide). Representative structures are shown in Attachment A.

1.2 **Compatible analytes.** 2-(N-methylperfluorooctanesulfonamido)-ethyl alcohol (N-MeFOSE alcohol), perfluorooctanesulfonate (PFOS anion), 2-(N-ethylperfluorooctanesulfonamide)-ethyl alcohol (N-EtFOSE alcohol), 3, 3, 4, 4, 5, 5, 6, 6, 7, 7, 8, 8, 8-tridecafluorooctane sulfonic acid anion (THPFOS) and perfluorooctanesulfonamide (FOSA).

1.3 This is a performance-based method. Target analyte or surrogate matrix spike recoveries (100 ± 25%) are used for each sample matrix to evaluate method performance. (Refer to Section 10 for the quality control parameters to be analyzed by this method. Refer to Section 14 for the quality assurance evaluation criteria for this method.)

2.0 SUMMARY OF METHOD

2.1 Aliquots of N-MeFOSE alcohol stock solution are added to vials that contain buffers at pH 1.5, 3.0, 5.0, 7.0, 9.0 and 11.0. The vials are then placed in an orbital incubator/shaker set at 50.0 ± 3 °C. Sets of vials are removed at designated intervals and the date and time recorded. The aqueous sample from the hydrolysis of N-MeFOSE alcohol is diluted tenfold with methanol (MeOH). The parent compound, N-MeFOSE alcohol, and the PFOS hydrolysis product are separated on a Dionex IonPac® NG1 reversed-phase HPLC column using an ammonium acetate/MeOH solvent gradient, with detection/quantitation by electrospray ionization mass spectrometry in the negative mode.

3.0 DEFINITIONS

3.1 **Solvent blank.** A sample of analyte-free medium (for example, methanol) that is not taken through the sample preparation process. This blank is used to evaluate instrument contamination.

3.2 **Sample duplicates.** Two samples taken from and representative of the same sample source and separately carried through all steps of the extraction and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method, including sampling, extraction, and analysis.

3.3 **Matrix spike (MS).** Prepared by adding a known mass of target analyte to a specified amount of a sample matrix. This assumes that an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the matrix on method recovery efficiency.

3.4 **Calibration standard.** A dilution of various amounts of a stock, intermediate or purchased standard to achieve standard solutions in a concentration range of interest. Hydrolytic half-lives resulting from these analyses are calculated based on analytical ratios and not absolute numbers. Therefore, results do not depend on the purity of the standards used.

3.5 **Internal standard.** A known amount of a compound or element similar in analytical behavior to the compound(s) or element(s) of interest, added to all samples and standards, and carried through the entire measurement process (post-hydrolysis, after final dilution). It provides a reference for evaluating and controlling the precision and bias of the applied analytical method.

3.6 **Surrogate.** An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in the sample(s). In hydrolysis studies, surrogate is added to CCVs, samples, sample duplicates, and matrix spike samples along with the test analyte (pre-hydrolysis).

3.7 **Continuing calibration verification (CCV).** Standards analyzed during an analytical run to verify the continued accuracy of the calibration curve. This solution may or may not be prepared from a different source or lot number than the calibration curve standards.

3.8 **Dilution.** A step in the hydrolysis study procedure in which a solvent is added to the test analyte/buffer solution to prepare it for instrumental analysis. This step occurs after the vials are removed from incubation and before the samples are analyzed. If the solvent used is miscible with the test analyte/buffer solution, the diluting solvent is merely added and mixed. If the diluting solvent is non-miscible, a liquid-liquid extraction is performed.

3.9 **Limit of quantitation (LOQ).** The lowest concentration that can be reliably measured within specified limits of accuracy (see Sections 14.1 and 14.2) and precision (see Sections 14.3 and 14.4) during routine laboratory operating conditions. The LOQ is generally 5 to 10 times the minimum concentration with a 99% confidence limit that the concentration is greater than zero. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes, the LOQ is selected as the lowest non-zero standard in the calibration curve that is greater than 4 times the level of the solvent blanks. Sample LOQs are highly matrix-dependent.

3.10 **Accuracy.** The closeness of agreement between an experimentally determined value and an accepted reference value. When applied to a set of observed values, accuracy is a combination of a random (precision) and a common systematic (bias) component. For purposes of the study, the acceptance criterion is 75% to 125% of the nominal value.

4.0 WARNINGS AND CAUTIONS

4.1 Health and safety warnings

4.1.1 Wear the proper lab attire for all parts of this procedure. Wear gloves and proper eyewear at all times.

4.1.2 Handle all solvents in a hood for all parts of the described sample preparation procedure. Whenever possible and practical, dilute samples with solvent in a hood.

4.1.3 For potential hazards of each chemical used, refer to material safety data sheets, packing materials, and the 3M Environmental Laboratory Chemical Hazard Review.

4.2 Cautions

4.2.1 All glassware in which standards are prepared should be triple-rinsed with 1:1 acetone/MeOH to reduce the possibility of contamination.

4.2.2 Ensure that the HPLC mobile phases are prepared prior to beginning a run sequence, and that there is sufficient quantity to complete the run. Do not allow the pump to run dry.

4.2.3 Ensure that before starting the run sequence there is ample hard disk space on the computer to save all run data.

4.2.4 Ensure that there is enough nitrogen in the supply tank to complete sequence runs.

5.0 INTERFERENCE

5.1 Contaminants in solvents, reagents, glassware, and other sample processing or analysis hardware may cause interference. Use the routine analysis of laboratory solvent blanks to demonstrate that there is no such interference.

5.2 Contamination from columns, HPLC tubing, and detector components may cause interference at low detection levels. The routine analysis of solvent blanks must be used to demonstrate that there is no such interference.

6.0 EQUIPMENT

6.1 Analytical balance sensitive to 0.1 mg

6.2 Incubator/shaker capable of maintaining temperature at 50.0 ± 3 °C

6.3 Hewlett-Packard (HP) 1100 HPLC System, or equivalent

6.3.1 Pump, binary, Model G1312, or equivalent

6.3.2 Solvent degasser, Model G1322A or equivalent

6.3.3 Autosampler, ALS Model G1313A, variable injection volume capable

6.3.4 Column heater, Model G1316A

6.4 Dionex IonPac® NG1 Guard column, 4 x 35 mm, or equivalent

6.5 Mass spectrometer. Hewlett-Packard MSD Model G1946A, or equivalent, operating in electrospray-negative SIM mode

6.6 Clock, digital. Only one clock should be used, to insure unambiguous documentation of the correct performance of procedures.

6.7 pH meter. Corning Model 308 pH/Temperature Meter with 3-in-1 gel-filled combination electrode (pH/reference/temperature), or equivalent

6.8 Refrigerator capable of maintaining 4 ± 3 °C

6.9 Data system. A personal computer capable of controlling the HPLC system as well as recording and processing signals from the detector, Hewlett-Packard ChemStation® Version A.06.01 or later

7.0 SUPPLIES AND MATERIALS

7.1 Vials, 40 mL, VOA (I-Chem or equivalent)

7.2 Crimp cap autovials, 1.8 mL

7.3 Labels

7.4 Graduated pipets, glass, disposable, 1 mL to 10 mL

7.5 Pasteur pipets, glass, disposable

7.6 Hamilton Gastight® syringes (precision ±1% of total volume), 10 µL-1000µL
7.7 Volumetric flasks, various sizes
7.8 Beakers, glass, various sizes
7.9 Automatic pipettor, capable of dispensing 10–5000 µL

8.0 REAGENTS AND STANDARDS

8.1 **Methanol (MeOH).** HPLC/SPEC/GC grade from EM Science, or equivalent
8.2 **Acetone.** HPLC/SPEC/GC grade from EM Science, or equivalent
8.3 **18.0 MΩ water.** Water with lower resistance must not be used.
8.4 **Ammonium acetate,** 2 mM in water. This solution is chromatographic solvent A (see Section 12.3.1). (Example: An acceptable eluent solution is made by adding 0.15g ammonium acetate crystals to a 1-L volumetric flask containing about 500 mL 18.0 MΩ water, adding 10 mL of methanol, diluting to the mark with 18.0 MΩ water and mixing.)
8.5 **Stock, internal standard and calibration solutions**
All weights should be recorded to the nearest 0.0001 g in a standards preparation log:

8.5.1 N-MeFOSE alcohol prepared in acetone. (Example: A stock solution is prepared at a concentration of approximately 30,000 µg/mL by weighing 0.3 g of N-MeFOSE alcohol in a 10-mL volumetric flask and bringing to the mark with acetone. This solution is diluted in MeOH to make additional, appropriate standards.)
8.5.2 N-EtFOSE-alcohol internal standard prepared in acetone. (Example: A stock solution is prepared at a concentration of approximately 30,000 µg/mL by weighing 0.3 g of N-EtFOSE-OH in a 10-mL volumetric flask and bringing to the mark with acetone. This solution is diluted in MeOH to make additional, appropriate standards.)
8.5.3 Perfluorooctanesulfonate (PFOS) prepared in acetone. (Example: A stock solution is prepared at a concentration of approximately 3000 µg/mL by weighing 0.06 g of PFOS in a 20-mL volumetric flask and bringing to the mark with acetone. This solution is diluted in MeOH to make additional, appropriate standards.)
8.5.4 3, 3, 4, 4, 5, 5, 6, 6, 7, 7, 8, 8, 8-tridecafluorooctane sulfonic acid (THPFOS) internal standard prepared in MeOH. (Example: A stock solution is prepared at a concentration of approximately 20,000 µg/mL by weighing 0.2 g of THPFOS in a 10-mL volumetric flask and bringing to the mark with MeOH. This solution is diluted in MeOH to make additional, appropriate standards.)

8.6 **Buffers for calibration of pH meter**
Purchased pH calibration standards of pH 4.0, 7.0, and 10.0 (suppliers vary).
8.7 **Buffer solutions for hydrolysis study.** Prepare buffer solutions of pH 1.5, 3.0, 7.0, 9.0 and 11.0 using guidelines from CRC Handbook of Chemistry and Physics (Reference 18.2). Prepare buffer solution of pH 5.0 using guidelines from Fate, Transport and Transformation Test Guidelines (Reference 18.2). Prepare the buffer solutions in 1-liter quantities. Calibrate a portable pH/temperature meter using purchased pH calibration standards of pH 4.0, 7.0, and 10.0, and measure the pH of all buffer solutions. Prepare buffer solutions of pH 1.5, 3.0, 5.0, 7.0, 9.0 and 11.0 at ambient room temperature. The

concentrations are given below. Record final pH measurements of all buffers. Store buffers in sealed glass containers.

8.7.1 pH 1.5

8.7.1.1 207 mL of 0.1 N HCl (reagent grade)
8.7.1.2 125 mL of 0.2 M KCl (reagent grade)
8.7.1.3 Add 18.0 MΩ water to about 900 mL total volume
8.7.1.4 Adjust pH to 1.5 with additional 1 N HCl
8.7.1.5 Bring to a final volume of 1 L with 18.0 MΩ water

8.7.2 pH 3.0

8.7.2.1 223 mL of 0.1 M HCl (reagent grade)
8.7.2.2 500 mL of 0.1 M potassium hydrogen phthalate (reagent grade)
8.7.2.3 Add 18.0 MΩ water to about 900 mL total volume
8.7.2.4 Adjust pH to 3.0 with 1 N HCl or 1 N NaOH
8.7.2.5 Bring to a final volume of 1 L with 18.0 MΩ water

8.7.3 pH 5.0

8.7.3.1 467 mL of 0.1M NaOH (reagent grade)
8.7.3.2 500 mL of 0.1 M monopotassium citrate (reagent grade)
8.7.3.3 Add 18.0 MΩ water to about 900 mL total volume
8.7.3.4 Adjust to pH of 5.0 with 1 N NaOH or 1 N HCl
8.7.3.5 Bring to a final volume of 1 L with 18.0 MΩ water

8.7.4 pH 7.0

8.7.4.1 500 mL 0.1 M KH_2PO_4 buffer (reagent grade)
8.7.4.2 291 mL 0.1 N NaOH (reagent grade)
8.7.4.3 Adjust to pH 7.0 with either 1 N HCl or 1 N NaOH
8.7.4.4 Bring to a final volume of 1 L with 18.0 MΩ water.

8.7.5 pH 9.0

8.7.5.1 500 mL 0.025 M sodium borate decahydrate (reagent grade)
8.7.5.2 46 mL of 0.1 N HCl (reagent grade)
8.7.5.3 Add 18.0 MΩ water to approximately 900 mL
8.7.5.4 Adjust to pH 9.0 with either 1 N HCl or 1 N NaOH
8.7.5.5 Bring to a final volume of 1 L with 18.0 MΩ water.

8.7.6 pH 11.0

8.7.6.1 500 mL 0.05 M $NaHCO_2$ (reagent grade)
8.7.6.2 227 mL 0.1 N NaOH (reagent grade)
8.7.6.3 Add 18.0 MΩ water to approximately 900 mL
8.7.6.4 Adjust pH to 11.0 with 1N NaOH
8.7.6.5 Bring to a final volume of 1 L with 18.0 MΩ water

8.8 Test analyte and spike solutions:

8.8.1 N-MeFOSE alcohol test analyte solution with THPFOS surrogate. [Example: An analyte solution of N-MeFOSE alcohol, TN-A-1282, at approximately 500 µg/mL and THPFOS at approximately 400 µg/mL is used (a dilution in MeOH of the solutions prepared in Sections 8.5.1 and 8.5.4). A 10-µL aliquot of this solution added to 1 mL buffer (step found in Section 12.1.5) results in a final concentration of approximately 500 ng/mL N-MeFOSE alcohol and

approximately 400 ng/mL THPFOS after MeOH dilution (step found in Section 12.1.13)].

8.8.2 N-EtFOSE alcohol internal standard solution. [Example: An analyte solution of N-EtFOSE alcohol, TN-A-1284, at approximately 30 µg/mL is used (a thousand-fold dilution in MeOH of the solution prepared in Section 8.5.2). A 100-µL aliquot of this solution added to 1mL buffer (step found in Section 12.1.5) results in a final concentration of approximately 300 ng/mL after MeOH dilution (step found in Section 12.1.13)].

8.8.3 Spiking solution. [Example: A spiking solution is prepared by adding 10-µL of the N-MeFOSE alcohol stock solution (Section 8.5.1) to a 10-mL volumetric flask and diluting to the mark with methanol. A 70-µL aliquot of this solution added to the 1 mL sample (Section 12.1.15) results in a final spike concentration of approximately 210 ng/mL of N-MeFOSE alcohol].

9.0 SAMPLE HANDLING

9.1 Record times of initial preparation and dilution on the fluorochemical degradation (hydrolysis) analysis sample preparation sheet (Attachment B).

9.2 For Time 0 samples, aliquot only the 1mL of buffer into the vials. DO NOT spike with test analyte. Store the vials at room temperature until ready to analyze. Then proceed from Section 12.1.12.

9.3 Once the 9.0 mL of diluting solvent has been added to the hydrolysis mixtures, the samples should be analyzed as soon as possible. Alternatively, aliquots of the methanol-diluted samples should be refrigerated at 4 ± 3 °C until analysis can be performed.

10.0 QUALITY CONTROL

10.1 Sample Duplicates. Prepare and analyze all samples in duplicate to provide a measure of the precision of analysis.

10.2 Matrix spikes. Prepare a post-hydrolysis matrix spike sample (Section 8.8.3 and 12.1.12) for each interval and pH level used in the study. Concentrations of the spike should be approximately equal to a mid-range calibration standard. The matrix spike sample should be analyzed immediately following the sample duplicates to which it corresponds. The analyst shall accept percent spike recoveries of 100 ± 25%. Spike recoveries outside of this range should be noted. Appropriate steps must be taken to correct the problem before analysis is allowed to proceed. Before the analysis is allowed to proceed, consult with the Team Leader or designee for direction and final acceptance or rejection of the analytical run.

10.3 Solvent blank. Solvent blanks should be run before and after every calibration curve, CCV, and after no more than 20 injections. Acceptable values for the blank are values less than 25% of the LOQ standard. If analyte carryover is a problem, use back-to-back solvent blanks.

10.4 Continuing Calibration Verification (CCV). A standard analyzed periodically during an analytical run to verify the continued accuracy of the calibration curve and is run in tandem with the solvent blank. This solution may be prepared from a different source or lot number than the calibration curves standards.

10.5 Internal standard. Internal standard is added post-hydrolysis (after final dilution) to all standards, samples, and matrix spikes at a constant concentration.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Standard preparation. Prepare six calibration standards containing N-MeFOSE alcohol, N-EtFOSE alcohol, PFOS and THPFOS in 9:1 MeOH:buffer for each pH level. Standards from approximately 75 ng/mL to 750 ng/mL of N-MeFOSE alcohol and 3 ng/mL to 100 ng/mL of PFOS are suggested. A level of approximately 400 ng/mL THPFOS surrogate and 300 ng/mL of N-EtFOSE alcohol internal standard in each calibration standard is suggested.

11.2 Calibration standards. Analyze the calibration standards at the beginning and end of the run. Average the peak area response from both curves. Use the data reduction software program for linear regression calculations to relate the analyte peak area ratio versus amount ratio, using internal standard calibration. Use N-EtFOSE alcohol as the internal standard for N-MeFOSE alcohol quantification, and THPFOS surrogate as the "internal standard" for PFOS quantitation. Quadratic regression may be used if data review shows this to be a consistent and more accurate representation of the instrument response. Consult with the Team Leader for direction prior to performing the quadratic calibration methodology.

12.0 PROCEDURES

12.1 Sample and spike preparation

12.1.1 Before spiking with any of the stock standards, transfer approximately 1 mL of the solution to an autovial and cap the vial. Use this smaller volume for spiking to minimize the effects of evaporation from stock solutions and to prevent contamination of the larger volume of stock solution.

12.1.2 Determine the number of time intervals that will be analyzed. Each interval will have three vials for each pH, multiplied by the number of pHs analyzed. One vial at each level will be labeled as sample, duplicate, and spike.

12.1.3 Obtain the appropriate number of 40-mL VOA vials with caps and cardboard boxes. Prepare appropriate sample preparation worksheets, create labels, and affix them to the vials. The labels should include the sample number and i.d., temperature, pH, time interval, test analyte, and date of preparation. Record the pH of each buffer solution.

12.1.4 Remove the cap of the VOA vial and add 1 mL of the appropriate buffer solution to all of the pre-labeled vials. Always replace the cap immediately after any addition to minimize evaporation.

12.1.5 Put "Time 0" samples aside at this point. For all other samples, continue on to section 12.1.6.

12.1.6 To all of the vials, add 10 µL of the combined N-MeFOSE alcohol and THPFOS analyte solution (Section 8.8.1) with a 25-µL Hamilton Gastight® syringe.

12.1.7 Make sure that the cap has been firmly tightened and place the samples back in the cardboard case.

12.1.8 Place the case into a pre-warmed incubator/shaker for the appropriate time. Record the time, temperature, and rate of shaking. The temperature is determined by the conditions of the experiment. Continue to manually monitor the incubator temperature daily during the entire incubation. Record the temperature on the sample preparation sheet (Attachment B).

12.1.9 Store **"Time 0"** samples at room temperature until the time of analysis.

12.1.10 Remove the case from the incubator at the designated preset time.

12.1.11 Remove the vials from the case and place in racks. Allow the vials to cool for approximately 15 minutes to room temperature.

12.1.12 While vials from the first time point are cooling, spike the stored "Time 0" samples with test analyte solution (Section 12.1.6). Then continue on to section 12.1.13 with all samples.

12.1.13 Add 9 mL of methanol to each vial.

12.1.14 Using a 100-µL gas tight syringe, add 100 µL of N-EtFOSE alcohol internal standard solution (Section 8.8.2) to each sample and spike vial.

12.1.15 Using a 100-µL gas tight syringe, add 70 µL of N-MeFOSE alcohol spiking solution (Section 8.8.3) to the sample spike vials. Shake the vials for three minutes by hand or Vortex mixer to mix the contents and extract any analytes that may have adsorbed to the vial.

12.1.16 Aliquot approximately 1 mL of each sample to the appropriately labeled autovial, cap, and refrigerate at 4 ± 3 °C until analysis.

12.2 Instrument set up

12.2.1 Check that the appropriate HPLC column is in the instrument for analysis.

12.2.2 Check that the correct eluent solutions are in bottles to be used and that enough is available to complete the sequence run.

12.2.3 Place the samples in the autosampler tray and construct a sequence table with appropriate calibration standards, calibration check standards and solvent blanks.

12.2.4 Verify that all samples and standards are positioned correctly. Enter sequence information (sample or standard ID, method name). Use one injection per sample.

12.2.5 Save sequence as analysis date and instrument letter (e.g. on March 14, 1999, save sequence table as 031499.s). Save all data to a subdirectory labeled with analysis date. (e.g. 031499).

12.2.6 Set post-sequence command macro to shut down system after the run is completed (Example: "STANDBY" on HP1100/MSD systems).

12.3 HPLC set up:

12.3.1 Analysis of N-MeFOSE alcohol hydrolysis samples in buffers at pH levels 1.5, 3.0, 5.0, 7.0, 9.0 and 11.0.

Column: Dionex IonPac® NG1 Guard column, 4 x 35 mm, or equivalent

Solvent A: Ammonium Acetate 2mM in water (with 1% MeOH).

Solvent B: Methanol

Recommended Solvent Gradient:

TIME (MIN)	%A	%B	FLOW RATE
0.0	60	40	0.3 mL/min
1.0	60	40	0.3 mL/min
4.0	5	95	0.3 mL/min
11.0	5	95	0.3 mL/min

Post time: 6 minutes, column temperature: 40°C.

12.4 Recommended mass spectrometer set up*:

MSD:	Ionization mode	API-ES
	Polarity	Negative
	Acquisition mode	SIM
	SIM resolution	High
	Time filter	Enabled
	Peakwidth	0.25 min
	Gain	1.0
	Fragmentor	70
	Dwell time	183 msec
	Capillary voltage	3500
	Drying gas	Nitrogen
	Nebulizer pressure	30 psig
	Drying gas flow	8 L/min
	Drying gas temp	300° C

*Example conditions are applicable to HP1100/MSD equipment only.

12.5 Auto-sampler setup*:

AUTO-SAMPLER:	ALS Model G1313A
AUTO-SAMPLER PROGRAM:	None
INJECTION VOLUME:	5.0 µL

*Example conditions are applicable to Hewlett Packard 1100 only

12.6 Ions used for identification and quantification:

APPROX. RETENTION TIME (MIN)	COMPONENT NAME	DESCRIPTION	QUANTIFICATION ION	MONITOR ION
8.4	MeFOSE-OH	Test Analyte	616	617
8.6	EtFOSE-OH	Internal Standard	630	631
6.2	PFOS	Potential Degradation Product	499	500

5.9	$THPFOS^-$	Surrogate	427	None
*	FOSA	Qualitative use only	None	498

*No calibration standards analyzed, so no retention times available.

12.7 Sample analysis

12.7.1 Enter the standard, sample, and QC information into the sequence table. Analyze calibration standards first, then up to 20 injections, followed by the calibration standards. If more than 20 injections are to be run, analyze a continuing calibration standard (CCV) after every 20 injections and run the calibration standards again at the end of the sequence. Run solvent (or method) blanks after the highest calibration standard, before and after the CCV, and after the set of samples to check for any analyte carryover.

12.7.2 Place standards, samples, and QC (matrix spikes, sample duplicates, and blanks) into the autosampler tray according to the order they are listed in the sequence.

12.7.3 Identify the electronic acquisition files with an appropriate prefix (e.g. MeFOS). Do not exceed five characters if the sequence contains more than 99 lines.

12.7.4 Save sequence as analysis date (e.g. on March 14, 1999, save sequence table as 031499.s). Save all data to a subdirectory labeled with analysis date (e.g. 031499).

12.7.5 Start the sequence.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 **Peak Evaluation.** Peaks must be symmetric in shape and identified by extracting compound-specific ions. Peaks considered for calibration must have peak heights greater than 5 (five) times the baseline noise for that region of the chromatogram. Peak area integration is from baseline to baseline using automatic or manual integration.

13.2 **Calculation of Rate Constant (*k*).** Calculate the test analyte concentrations in each of the pH matrices using the curves obtained from the calibrations. Assuming first-order kinetics a rate constant (*k*) can be determined by plotting:

$$Ln\left(\frac{[\text{N-MeFOSE-OH}]_t}{[\text{N-MeFOSE-OH}]_0}\right)$$ versus minus elapsed time (*-t*). The subscripts *t* and *0* refer to analyte concentrations determined at some elapsed time *t* and at $t = 0$, respectively. The slope of the resulting line is *k*. The r^2 value for this plot should be > 0.80. For r^2 values less than this, consult the Team Leader or designee.

13.3 **Target analyte concentrations.** Calculate the Me-FOSE alcohol and PFOS concentrations in each of the pH matrices using the curves obtained from the calibrations.

13.4 **Matrix spikes.** Calculate the percent recovery for each of the matrix spikes. Calculate the matrix spike percent recoveries using the following equation:

$$\% \text{ Recovery} = \frac{(\text{observed spiked sample result} - \text{observed sample result})}{\text{Actual amount spiked}} \times 100$$

Using the observed matrix spike recoveries, calculate the average spike recovery.

13.5 Sample Duplicates. Calculate the relative percent deviation (RPD) for the duplicate samples:

$$RPD = \frac{|A-B|}{(A+B)/2} \times 100\%$$

Where A= the concentration measured in the sample
B= the concentration measured in the sample duplicate

14.0 METHOD PERFORMANCE

14.1 Accuracy. For purposes of the study, the acceptance criterion is 75% to 125% of the nominal value.

14.2 Coefficient of determination (r^2). The coefficient of determination (r^2) for the calibration curves should be 0.99 or greater. The curves should be examined closely for linearity and intercept, particularly for accuracy of quantitation at the low and high ends of the curve. The accuracy of all standards used for calibration must be within 75-125%. On occasion it may be necessary to use exponential or quadratic fits of the data, usually when broad range curves (greater than 3 orders of magnitude between the low and high concentration standards) are used. Document in the raw data the technical justification for using quadratic equations.

14.3 Matrix spikes. The analyst shall accept percent spike recovery values of 100 ± 25%. Spike recoveries outside of this range should be noted. Consult with the Team Leader or designee for direction, and for final acceptance or rejection of the data. Data that are used in final report that is deemed out of control will be required to have a technical justification for why the data are being used, documented in the final report and raw data.

14.4 Sample duplicates. The analyst shall accept %RPD (See Section 13.5) values < 25%. %RPD values > 25% should be noted. RPD values of 25% or greater should be noted. Appropriate steps must be taken to correct the problem before analysis is allowed to proceed (e.g. sample re-runs, additional blanks, etc.). Consult with the Team Leader or designee for direction, and for final acceptance or rejection of the data.

14.5 Continuing calibration verification (CCV). If the percent difference for the amount of quantitated analyte is greater than 25% from the true value relative to the initial standard curve, stop the run. Only those samples analyzed before the last acceptable calibration check standard will be used. Consult with the Team Leader or designee for direction, and for final acceptance or rejection of the data.

14.6 Internal standard and Surrogate. Review of the internal standard and surrogate can be performed by averaging the area response throughout the analytical run and calculating the relative standard deviation (%RSD). %RSD values >10% should be noted. Inconsistencies in the internal standard peak area may indicate instrumental changes over time. Inconsistencies in the surrogate peak area may indicate instrumental changes, changes in the test-system, or hydrolysis of the surrogate over time. Consult with the Team Leader or designee for direction and final acceptance or rejection of the analytical run.

14.7 **Limit of Quantitation (LOQ).** The LOQ is equal to the lowest standard in the calibration curve that is greater than 4 times the level of the solvent blanks. If blank contamination is present, it may be necessary to delete the lowest standard of the calibration curve.

14.8 **Solvent blanks.** Solvent blanks should show no more than a 5% carryover from a high standard or calibration check standard. If so, two solvent blanks may be necessary to rule out instrumental contamination. If peaks with greater than 25% of the peak area of a low standard value are observed in sequential solvent blanks, it is indicative of instrument contamination. The instrument shall be serviced by thoroughly cleaning the electrospray source, and replacing/cleaning columns, tubing, etc.

14.9 **Specificity.** Analyte specificity is demonstrated by acceptable post-hydrolysis analyte spike recoveries.

14.10 **System Suitability.** Without performing a method validation, system suitability can be demonstrated by acceptable instrumental checks (e.g. abbreviated m/z check-tune, or full auto-tune routines). Consult the appropriate instrumental manuals (Reference 18.3).

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

15.1 Dispose of sample waste by placing in high or low BTU containers as appropriate. Use broken glass containers to dispose of glass pipettes.

15.2 Collect HPLC solvent waste in the satellite accumulation can. Empty into the flammable storage drum in the hazardous waste collection area on the 2nd floor.

15.3 Use smaller bore columns when possible to minimize waste generation.

16.0 RECORDS

16.1 Print hard copies of all graphics and data analysis summaries for archiving.

16.2 Sign and date all data packages and label with instrument ID.

16.3 Fill out the hydrolysis sample preparation worksheet completely, making sure to include all initials and dates.

16.4 Print out the sample sequence table, reduce the size with photocopying and tape the photocopy into the instrument log. Keep the original copy for the raw data files package.

16.5 Print chromatograms and quantification reports for all analyses.

16.6 Print calibration tables and curve information and store in the raw data file.

16.7 Store hydrolysis sample preparation worksheets in the raw data file.

16.8 Enter all standard preparation information in the standards preparation logbook. Make a photocopy of the logbook page and include the copy in the raw data file.

16.9 Archive electronic data to appropriate media when necessary.

17.0 ATTACHMENTS

17.1 Attachment A. Representative chemical structures

17.2 Attachment B. Hydrolysis sample logsheet

18.0 REFERENCES

18.1 Fate, Transport and Transformation Test Guidelines Office of Prevention, Pesticides and Toxic Substances (OPPTS) 835.2110 Hydrolysis as a Function of pH, EPA 712-C-98-057, January 1998.

18.2 *CRC Handbook of Chemistry and Physics, 1st Student Edition*, "Buffer Solutions Operational Definitions of pH," Robert C. Weast, Ph.D., 1988, p. D-87.

18.3 HP 1100 Series LC/MSD Reference Collection, Rev. A.00.01 June 1997 CD/ROM

19.0 AFFECTED DOCUMENTS

19.1 None.

20.0 REVISIONS

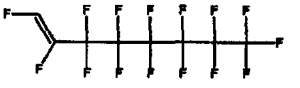
Revision number	Reason for revision	Date of Revision

Attachment A. Representative Chemical Structures

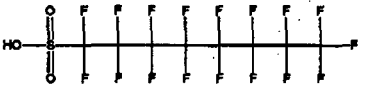
N-MeFOSE Alcohol FW = 557.23

N-EtFOSE Alcohol FW = 571.25

FOSA FW=499.14

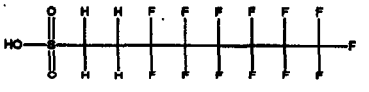


C_8 Olefin FW=382.07



PFOS FW=500.13

C_8 Hydride FW=420.07



THPFOS FW=428.17

Attachment B. Hydrolysis Sample Logsheet

Fluorochemical Degradation (Hydrolysis) Analysis

TEST ANAYLTE: _____ Incubator ID: _____ Freezer ID: _____

Lab Request Number: _____ Start Date: _____ Time: _____ Start Date: _____ Time: _____

Nominal Interval: _____ Stop Date: _____ Time: _____ Stop Date: _____ Time: _____

Nominal Temperature: _____ °C Incubation Interval: _____ days _____ hours _____ min

					Buffer		Test Analyte		Spike Solution		Dilution Solvent		Internal Standard		Comment
				Date:											
				Time:											
				Initials:											
Sample No.	Rep	Int	Temp	pH	Solution ID	Amount (mL)	Solution ID	Amount (µL)	Solution ID	Amount (µL)	Solution ID	Amount (mL)	Solution ID	Amount (µL)	
	r1			1.5					--	--					
	r2			1.5					--	--					
	r3			1.5					--	--					
	ms			1.5											
	blk			1.5			--	--	--	--					
	r1			3					--	--					
	r2			3					--	--					
	r3			3					--	--					
	ms			3											
	blk			3			--	--	--	--					
	r1			5					--	--					
	r2			5					--	--					
	r3			5					--	--					
	ms			5											
	blk			5			--	--	--	--					
	r1			7					--	--					
	r2			7					--	--					
	r3			7					--	--					
	ms			7											
	blk			7			--	--	--	--					
	r1			9					--	--					
	r2			9					--	--					
	r3			9					--	--					
	ms			9											
	blk			9			--	--	--	--					
	r1			11					--	--					
	r2			11					--	--					
	r3			11					--	--					
	ms			11											
	blk			11			--	--	--	--					

Component	Conc. (µg/mL)	Component	Conc. (µg/mL)	Component	Conc. (µg/mL)	Component	Conc. (µg/mL)

Comments:

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Appendix B: Kinetics Model

This Appendix includes a mathematical description of the kinetics model employed in the study.

Kinetics Model

B1. Reaction Components and Rates

The arguments below are based on the following idealized set of reactions representing the hydrolysis of a parent compound P and its hydrolysis products A_m, which number N. The actual hydrolysis reactions that occur under neutral, acidic, and basic conditions are subsumed in these equations, and are assumed to proceed with pseudo-first order rates k_{Pm} (for the parent) and k_{Am} (for the parent's hydrolysis products).

$$P + H_2O \overset{k_{Pm}}{\Leftrightarrow} n_m A_m + Y_{m1} \qquad (m = 1 \text{ to } N) \tag{B1}$$

$$A_m + H_2O \overset{k_{Am}}{\Leftrightarrow} Y_{m2} \qquad (m = 1 \text{ to } N) \tag{B2}$$

where the general symbols Y_{m1} and Y_{m2} represent all the other hydrolysis products.

B2. Parent Compound Concentrations

Equation B1 indicates that the pseudo-first order differential change in the parent concentration P is given by

$$dP = -P\left(\sum_{m=1}^{N} n_m k_{Pm}\right)dt \tag{B3}$$

which is equivalent to the separable differential equation

$$\frac{dP}{P} = -\left(\sum_{m=1}^{N} n_m k_{Pm}\right)dt \tag{B4}$$

Equation B4 may be directly integrated to obtain the general solution

$$\ln[P] = \left(-\sum_{m=1}^{N} n_m k_{Pm} t\right) + C \tag{B5}$$

With the initial condition $P(t=0) \equiv P_0$, the specific solution to Equation B4 is

$$P = P_0 \exp\left(-\sum_{m=1}^{N} n_m k_{Pm} t\right) \equiv P_0 e^{-k_P t} \tag{B6}$$

using the additional definition of the total parent hydrolysis rate

$$k_P \equiv \sum_{m=1}^{N} n_m \, k_{Pm} \quad . \tag{B7}$$

Equation B6 can be re-written in a form that allows a least-squares estimate of the total parent hydrolysis rate:

$$k_P t = -\ln\left(\frac{P}{P_0}\right) \tag{B8}$$

Using the initial $(t = 0)$ measured value of the parent concentration P_0 and later values P measured at later times t, one can calculate and plot the (linear) quantity $[-\ln (P/P_0)]$ versus time and obtain a least -squares estimate of the slope of the line. The resulting slope is the least-squares estimate $\hat{k}_P$ of the total parent hydrolysis rate.

Equation B6 indicates that over a period of time $T^{1/2}_p$ (the parent hydrolysis half-life) the parent concentration P is reduced through hydrolysis by a factor of two, where

$$T^{1/2}_p = \frac{\ln(2)}{k_P} \tag{B9}$$

A least squares estimate $\hat{T}^{1/2}_p$ of the parent hydrolysis half-life is therefore available from

$$\hat{T}^{1/2}_p = \frac{\ln(2)}{\hat{k}_P} \quad . \tag{B10}$$

B3. Product Compound Concentrations

The pseudo-first order differential changes in the product concentrations A_m (using Equations B2 and B6) are

$$dA_m = \left(n_m k_{Pm} P - k_{Am} A_m\right) dt = \left(n_m k_{Pm} P_0 \, e^{-k_P t} - k_{Am} A_m\right) dt \tag{B11}$$

and the (first order, non-separable) differential equation governing the product concentrations is

$$\frac{dA_m}{dt} + k_{Am} A_m = n_m k_{Pm} P_0 \, e^{-k_P t} \quad . \tag{B12}$$

The "standard form" of Equation B12 is

$$A'_m + S(t) A_m = Q(t) \tag{B13}$$

where the "function" $S(t)$ is actually a constant:

$$S(t) = k_{Am} \tag{B14}$$

and

$$Q(t) = n_m k_{Pm} P_0 \, e^{-k_P t} \quad . \tag{B15}$$

The general solution A_m to Equation B12 is contained in

$$A_m \, e^{\int S(t)\,dt} = \int Q(t) \, e^{\int S(t')\,dt'} \, dt + C \tag{B16}$$

where

$$e^{\int S(t)dt} = e^{\int S(t')dt'} = e^{k_{Am} \int dt} = e^{k_{Am} t} \tag{B17}$$

and

$$\int Q(t) \, e^{\int S(t')dt'} \, dt \; + \; C \; = \; n_m k_{Pm} P_0 \int e^{k_{Am} t} \, e^{-k_P t} dt \; + \; C \tag{B18}$$

There are two cases of Equation B18 to consider. In the circumstance that $k_{Am} = k_P$, which occurs only when the hydrolysis rate of the m[th] product is identical to the total parent hydrolysis rate, the general solution to Equation B18 is

$$\text{(for } k_{Am} = k_P \text{)}$$

$$A_m \, e^{k_P t} = n_m k_{Pm} P_0 \, t + C \tag{B19}$$

and, using the initial condition $A_m(t = 0) = A_{m0}$, the specific solution to Equation18 is

$$\text{(for } k_{Am} = k_P \text{)}$$

$$A_m = \left(n_m k_{Pm} P_0 \, t + A_{m0} \right) e^{-k_P t} \quad . \tag{B20}$$

We note that when $k_{Am} = k_P = 0$ (that is, when both the parent and potential product are hydrolytically stable), Equation B7 requires (also) that $k_{Pm} = 0$, so Equation B20 becomes

$$A_m = A_{m0} \qquad \text{(B21)}$$

indicating, as required, that the product concentration does not change with time.

The circumstance $k_{Am} = k_P$ is highly improbable, and is neglected in the remainder of this discussion. However, the reader should bear in mind that the expressions derived below do not hold when the parent hydrolysis rate k_P and the product hydrolysis rate k_{Am} approach each other.

In the more probable case, for which $k_{Am} \neq k_P$ (i.e. that the hydrolysis rate of the m^{th} product is different from the total parent hydrolysis rate), the general solution to Equation B18 is

$$A_m e^{k_{Am}t} = \frac{n_m k_{Pm} P_0}{k_{Am} - k_P} e^{(k_{Am} - k_P)t} + C \qquad \text{(B22)}$$

and the specific solution to Equation B18 with the initial condition $A_m(t = 0) = A_{m0}$ is

$$A_m = \left(A_{m0} + \frac{n_m k_{Pm} P_0}{k_P - k_{Am}} \right) e^{-k_{Am} t} - \frac{n_m k_{Pm} P_0}{k_P - k_{Am}} e^{-k_P t} \quad . \qquad \text{(B23)}$$

Of greatest interest here is the case in which the product compounds are known to be hydrolytically stable, that is, when $k_{Am} = 0$ for all m. In this case, Equation B23 becomes

(for hydrolytically stable products)

$$A_m = A_{m0} + \frac{n_m k_{Pm} P_0}{k_P} \left(1 - e^{-k_P t} \right) \quad . \qquad \text{(B24)}$$

B4. Relationships Between the Parent and Compound Concentrations

Equations B7 and B24 can be combined to obtain

(for hydrolytically stable products)

$$k_P = \sum_{m=1}^{N} n_m k_{Pm} = \frac{k_P}{\left(1 - e^{-k_P t} \right)} \sum_{m=1}^{N} \frac{(A_m - A_{m0})}{P_0} \qquad \text{(B25)}$$

so that

(for hydrolytically stable products)

$$\left(1-e^{-k_P t}\right)=\sum_{m=1}^{N}\frac{\left(A_m-A_{m0}\right)}{P_0} \qquad \text{(B26)}$$

or

(for hydrolytically stable products)

$$k_P t=-\ln\left[1-\sum_{m=1}^{N}\frac{\left(A_m-A_{m0}\right)}{P_0}\right] \qquad \text{(B27)}$$

If the changes in the product concentrations are all small compared to the original parent concentration, that is, if

$$\left|\sum_{m-1}^{N}\frac{A_m-A_{m0}}{P_0}\right|<<1, \qquad \text{(B28)}$$

we may use the expression (valid for $-1 \le X \le 1$)

$$\ln(1+X)=X-\frac{1}{2}X^2+\frac{1}{3}X^3-\frac{1}{4}X^4+..... \qquad \text{(B29)}$$

and Equation B23 becomes

(for hydrolytically stable products and $\left|\sum_m A_m-A_{m0}\right|<<P_0$)

$$k_p t\cong-\left(-\sum_{m=1}^{N}\frac{A_m-A_{m0}}{P_0}\right) \qquad \text{(B30)}$$

or

(for hydrolytically stable products and $\left|\sum_m A_m - A_{m0}\right| << P_0$)

$$k_p t \cong \sum_{m=1}^{N} \frac{A_m - A_{m0}}{P_0} \quad . \tag{B31}$$

B5. Parent Half-Life Estimates Based on Limits of Quantification of the Products

In every experimental determination of k_P, there is some set of values A_m^{LOQ} (the "limits of quantitation") below which the product concentrations A_m cannot be reliably measured. If during an experiment carried out over the period of time Δt all the product concentrations A_m remain below their limits of quantitation, then the maximum possible value of the rate k_P is obtained by assuming (for all the products) that 1) $A_{m0} = 0$ and 2) at time $t = \Delta t$, the product concentrations have increased to the values $A_m = A_m^{LOQ}$. With these assumptions, the experimental data indicate that the reaction rate k_P is less than some maximum value $(k_P)_{max}$ as follows:

(for hydrolytically stable products at concentrations below the limits of quantitation)

$$k_P \le (k_P)_{max} = \frac{1}{P_0 \, \Delta t} \sum_{m=1}^{N} A_m^{LOQ} \, . \tag{B32}$$

Under the same circumstances and assumptions, the experimental data indicate that the parent half-life $T_p^{1/2}$ (see Equation B9) is greater than the value $(T_p^{1/2})_{min}$ as follows:

(for hydrolytically stable products at concentrations below the limits of quantitation)

$$T_p^{1/2} \ge (T_p^{1/2})_{min} = \frac{\ln(2)}{(k_P)_{max}} = \Delta t \; P_0 \ln(2) \left[\sum_{m=1}^{N} A_m^{LOQ}\right]^{-1} \tag{B33}$$

The reader should note that Equations B32 and B33 are valid only when both 1) the products are hydrolytically stable and 2) the concentrations of *all* the potential products are measured. Otherwise, the quantity $(k_P)_{max}$ in Equation B32 may not actually represent the maximum possible value of the rate constant k_P, and the related result in Equation B33 for $(T_p^{1/2})_{min}$ is also questionable.

B6. Parent Half-Life Estimates Based on Limits of Quantification and Experimental Precision of Product Concentrations

In certain experiments, some hydrolysis products are present at quantifiable but essentially constant concentrations over the time (Δ t) of the experiment. In this case, it is the experimental precision of the measured product concentrations, rather than the limits of quantitation, which contribute to the estimate of the maximum value of the parent hydrolysis rate k_P. If the set of concentrations measured for the m^{th} product have the mean value μ_m and standard deviation σ_m, the data do not exclude the possibility that the product concentration increased from the initial value $\sigma_m - \mu_m$ to the value $\sigma_m + \mu_m$ at time t = Δ t. Taking this possibility to be the actual case for the measured products, the maximum value of the quantity $(A_m - A_{m0})$ is $2\sigma_m$. This reasoning suggests that the following estimate of the maximum parent hydrolysis rate is appropriate:

(for hydrolytically stable products at either 1) constant measured concentrations with standard deviation σ_m or 2) concentrations below the limits of quantitation)

$$k_P \le (k_P)_{max} = \frac{1}{P_0\ \Delta t}\left[\sum_{\text{Below LOQ}} A_m^{LOQ} + \sum_{\text{Constant}} 2\sigma_m\right]. \qquad \text{(B34)}$$

Under these circumstances and assumptions, the experimental data indicate that the parent half-life $T_p^{1/2}$ is greater than the value $(T_p^{1/2})_{min}$ as follows:

(for hydrolytically stable products at either 1) constant measured concentrations with standard deviation σ_m or 2) concentrations below the limits of quantitation)

$$T_p^{1/2} \ge (T_p^{1/2})_{min} = \frac{\ln(2)}{(k_P)_{max}} = \Delta t\ \ P_0\ \ln(2)\left[\sum_{\text{Below LOQ}} A_m^{LOQ} + \sum_{\text{Constant}} 2\sigma_m\right]^{-1}. \qquad \text{(B35)}$$

The reader should note that Equations B34 and B35 are valid only when both 1) the products are hydrolytically stable and 2) the concentrations of *all* the potential products are measured.

B6. Parent Half-Life Estimates Based on the Experimental Precision of Parent Concentrations

In certain experiments, the hydrolytic parent remains at an essentially constant concentration over the time (Δ t) of the experiment. In this case, it is the experimental precision of the measured parent concentrations that determines the maximum value of the parent hydrolysis rate k_P. If the set of concentrations measured for the parent have the mean value μ_P and standard deviation σ_P, the data do not exclude the possibility

that the product concentration increased from the initial value $\mu_P - \sigma_P$ to the value $\mu_P + \sigma_P$ at time t = Δ t. This reasoning suggests that the following estimate of the maximum parent hydrolysis rate is appropriate:

(for essentially constant parent concentrations with mean value μ_P and standard deviation σ_P)

$$k_P \leq (k_P)_{max} = \frac{2\sigma_P}{\mu_P \, \Delta t}. \qquad \text{(B36)}$$

Under these circumstances and assumptions, the experimental data indicate that the parent half-life $T^{1/2}_p$ is greater than the value $(T^{1/2}_p)_{min}$ as follows:

(for essentially constant parent concentrations with mean value μ_P and standard deviation σ_P)

$$T^{1/2}_p \geq (T^{1/2}_p)_{min} = \frac{\ln(2)}{(k_P)_{max}} = \frac{\mu_P \, \Delta t \ln(2)}{2\sigma_P}. \qquad \text{(B37)}$$

B8. Temperature Dependence of the Reaction Rate and Half-Life

In order to increase the speed of the reactions of interest, we conducted this experimental study using samples maintained at the temperature 50°C = 323 K. Of greater interest are the corresponding results for the environmentally important temperature 25°C = 298 K.

When the Arrhenius activation energy for a reaction is ΔH_a, Equation B38 [B1] provides the following relationship between the hydrolysis rates (k_1 and k_2) for that reaction at two different absolute temperatures (T_1 and T_2):

$$\frac{k_1}{k_2} = \exp\left\{\frac{\Delta H_a}{R}\left[\frac{1}{T_2} - \frac{1}{T_1}\right]\right\} \qquad \text{(B38)}$$

where $R = 1.99 \times 10^{-3}$ Kcal mole^{-1} K^{-1} is the ideal gas constant. Using the value[B2] ΔH_a =18 Kcal/mole, the rate ratio k_1/k_2 at the corresponding temperatures T_1 =298 K and T_2 =323 K is

$$\frac{k_1}{k_2} = \exp\left\{\frac{18}{1.99\times10^{-3}}\left[\frac{1}{323} - \frac{1}{298}\right]\right\} = \exp(-2.35) = 0.095 \qquad \text{(B39)}$$

Equation B39 indicates that the hydrolysis reactions of interest proceed approximately ten times more slowly at 25°C than at the chosen experimental temperature of 50°C. Accordingly, the rate reactions reported here for the temperature 25°C are ten times lower than those measured at 50°C, and the hydrolysis half-life estimates reported here for 25°C samples are ten times longer than those calculated from the 50°C experimental data.

References to Appendix B:

[B1] I. N Levine, "Physical Chemistry," McGraw-Hill (New York), pp. 498-501 (1978).

[B2] F. Daniels, et al., "Experimental Physical Chemistry", McGraw Hill (New York), p.131 (1962).

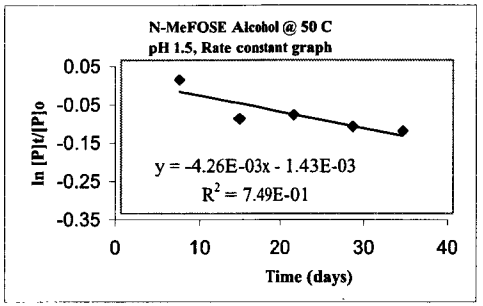
Appendix C: Selected Analytical and Kinetics Results

This Appendix includes selected sample data and their related kinetics results.

N-MeFOSE Alcohol Buffer Hydrolysis Study at 50° C.

All concentrations in ng/ml

pH	Time (Days)	Conc.	ln([P]t/[P]o)
1.5	*0*		(A)
1.5	*42*		(A)
1.5	*49*		(A)
1.5	7	441	0.000
1.5	14	398	-0.102
1.5	21	402	-0.092
1.5	28	390	-0.122
1.5	34	385	-0.134



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.86541
R Square	0.74893
Adjusted R Square	0.66524
Standard Error	0.03064
Observations	5

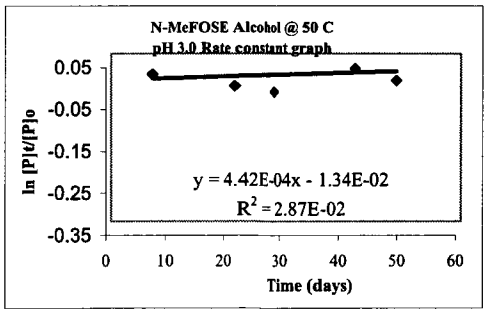
ANOVA

	df	*SS*
Regression	1	0.00840
Residual	3	0.00282
Total	4	0.01122

	Coefficients	*Standard Error*
Intercept	-0.00143	0.03264
X Variable 1	-0.00426	0.00142

% 2σ Slope Uncertainty
67%

pH	Time (Days)	Conc.	ln([P]t/[P]o)
3.0	*0*		(A)
3.0	*14*		(A)
3.0	7	348	0.000
3.0	21	338	-0.027
3.0	28	333	-0.042
3.0	34	373	0.069
3.0	42	353	0.014
3.0	49	343	-0.014



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.16934
R Square	0.02868
Adjusted R Square	-0.21415
Standard Error	0.04329
Observations	6

ANOVA

	df	*SS*
Regression	1	0.00022
Residual	4	0.00750
Total	5	0.00772

	Coefficients	*Standard Error*
Intercept	-0.01338	0.04261
X Variable 1	0.00044	0.00129

% 2σ Slope Uncertainty
582%

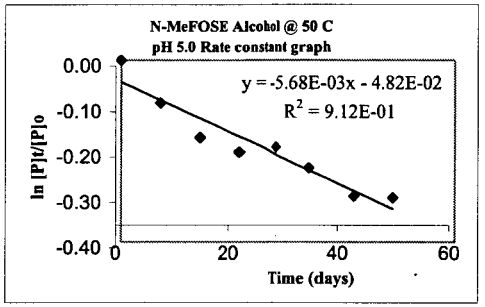
(A) Data in Italics have been rejected from the fits on the basis of the data quality objectives (see text).

BACK TO MAIN

N-MeFOSE Alcohol Buffer Hydrolysis Study at 50° C.

All concentrations in ng/ml

pH	Time (Days)	Conc.	ln([P]t/[P]o)
5.0	0	482	0.000
5.0	7	439	-0.094
5.0	14	407	-0.170
5.0	21	394	-0.202
5.0	28	398	-0.191
5.0	34	381	-0.236
5.0	42	358	-0.299
5.0	49	357	-0.302



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.95481
R Square	0.91167
Adjusted R Square	0.89695
Standard Error	0.03261
Observations	8

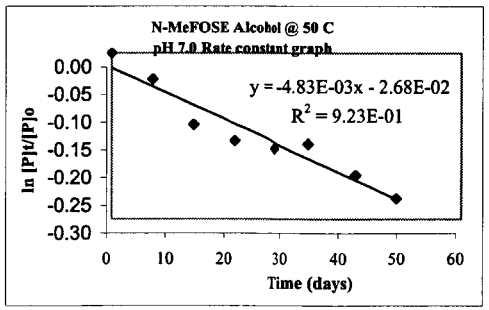
ANOVA

	df	*SS*
Regression	1	0.06584
Residual	6	0.00638
Total	7	0.07222

	Coefficients	*Standard Error*
Intercept	-0.04820	0.02104
X Variable 1	-0.00568	0.00072

% 2σ Slope Uncertainty
25%

pH	Time (Days)	Conc.	ln([P]t/[P]o)
7.0	0	467	0.000
7.0	7	446	-0.047
7.0	14	410	-0.130
7.0	21	399	-0.159
7.0	28	393	-0.173
7.0	34	396	-0.164
7.0	42	375	-0.220
7.0	49	360	-0.262



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.96050
R Square	0.92256
Adjusted R Square	0.90965
Standard Error	0.02578
Observations	8

ANOVA

	df	*SS*
Regression	1	0.04749
Residual	6	0.00399
Total	7	0.05147

	Coefficients	*Standard Error*
Intercept	-0.02680	0.01664
X Variable 1	-0.00483	0.00057

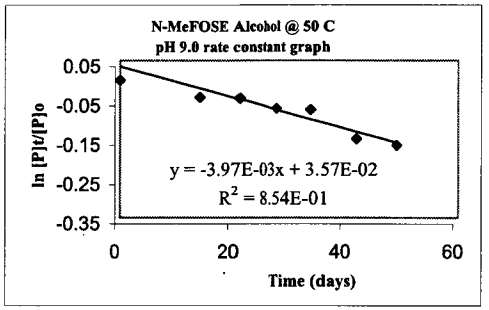
% 2σ Slope Uncertainty
24%

BACK TO MAIN

N-MeFOSE Alcohol Buffer Hydrolysis Study at 50° C.

All concentrations in ng/ml

pH	Time (Days)	Conc.	ln([P]t/[P]o)
9.0	0	412	0.000
9.0	7	437	0.060
9.0	14	394	-0.043
9.0	21	393	-0.046
9.0	28	383	-0.072
9.0	34	382	-0.075
9.0	42	355	-0.148
9.0	49	349	-0.164



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.92392
R Square	0.85363
Adjusted R Square	0.82924
Standard Error	0.03027
Observations	8

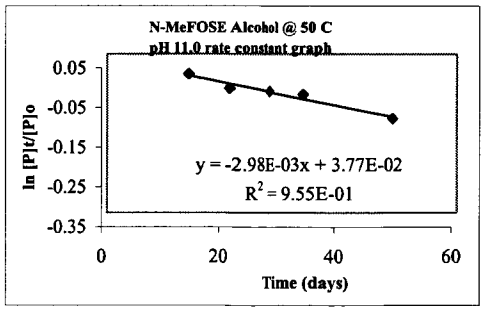
ANOVA

	df	*SS*
Regression	1	0.03207
Residual	6	0.00550
Total	7	0.03757

	Coefficients	*Standard Error*
Intercept	0.03566	0.01954
X Variable 1	-0.00397	0.00067

% 2σ Slope Uncertainty
34%

pH	Time (Days)	Conc.	ln([P]t/[P]o)
11.0	*0*	*480*	
11.0	*7*	*450*	
11.0	*42*	*413*	
11.0	14	413	0.000
11.0	21	398	-0.037
11.0	28	395	-0.044
11.0	34	392	-0.053
11.0	49	369	-0.113



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.97704
R Square	0.95460
Adjusted R Square	0.93947
Standard Error	0.01002
Observations	5

ANOVA

	df	*SS*
Regression	1	0.00634
Residual	3	0.00030
Total	4	0.00664

	Coefficients	*Standard Error*
Intercept	0.03775	0.01183
X Variable 1	-0.00298	0.00037

% 2σ Slope Uncertainty
25%

(A) Data in Italics have been rejected from the fits on the basis of the data quality objectives (see text).

BACK TO MAIN

MeFOSE Alcohol Hydrolysis Study
50C; pH 1.5
Retention times (RT) in minutes
All concentrations in ng/ml

			N-MeFOSE Alcohol						PFOS				THPFOS			N-EtFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc.	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	MeFOS004.D	92	0.0	0	0				6.3	0.0	0.0	0.0	6.0	0	0	8.6	0	0
99039-30-1	MeFOS006.D	1	8.4	159912	87	110%			6.3	10709	4.0	128%	6.0	327940	403	8.6	629180	311
99039-30-2	MeFOS007.D	2	8.4	302118	159	101%			6.2	20812	7.5	96%	6.0	335820	403	8.6	635756	311
99039-30-3	MeFOS008.D	3	8.4	583920	303	96%			6.2	39143	14.3	91%	6.0	329167	403	8.6	637107	311
99039-30-4	MeFOS009.D	4	8.4	882215	466	98%			6.2	93452	34.2	109%	6.0	325653	403	8.6	623482	311
99039-30-5	MeFOS010.D	5	8.4	1143693	610	97%			6.3	168595	62.1	99%	6.0	323376	403	8.6	616127	311
99039-30-6	MeFOS011.D	6	8.4	1499047	794	101%			6.3	260180	96.6	103%	6.0	320840	403	8.6	619682	311
MeOH Blank	MeFOS011.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0	8.6	0	0
MeOH Blank	EtFOS010.D	91	8.4	0	0				6.3	0	0.0		6.0	0	0	8.6	0	0
MFA-001	MeFOS011.D	11	8.4	901027	794	169%		(A)	6.3	0	0.0		6.0	340395	403	8.6	631887	311
MFA-002	MeFOS012.D	12	8.3	76714	0			(A,C)	6.3	9367	0.7		6.0	1843930	403	8.6	21628	311
MFA-003	MeFOS013.D	13	8.4	1272654	660			(A)	6.3	0	0.0		6.0	334722	403	8.6	633337	311
MFA-019	MeFOS014.D	14	8.4	818000	437	2%		Day 7	6.3	0	0.0		6.0	315749	403	8.6	616995	311
MFA-020	MeFOS015.D	15	8.4	830680	444			Day 7	6.3	0	0.0		6.0	319962	403	8.6	615930	311
MFA-021	MeFOS016.D	16	8.4	1196650	644		92%	Day 7	6.3	0	0.0		6.0	320529	403	8.6	610314	311
MFA-037	MeFOS017.D	17	8.4	824922	393	3%		Day 14	6.3	0	0.0		6.0	321851	403	8.6	692682	311
MFA-038	MeFOS018.D	18	8.4	804837	403			Day 14	6.3	0	0.0		6.0	311489	403	8.6	658945	311
MFA-039	MeFOS019.D	19	8.4	1204286	596		90%	Day 14	6.3	0	0.0		6.0	313734	403	8.6	663813	311
MFA-055	MeFOS020.D	20	8.4	818907	400	1%		Day 21	6.3	0	0.0		6.0	321434	403	8.6	675939	311
MFA-056	MeFOS021.D	21	8.4	823891	404			Day 21	6.3	0	0.0		6.0	321231	403	8.6	672256	311
MFA-057	MeFOS022.D	22	8.4	1209639	577		79%	Day 21	6.3	0	0.0		6.0	323500	403	8.6	689292	311
MFA-073	MeFOS023.D	23	8.4	796832	392	1%		Day 28	6.3	0	0.0		6.0	323192	403	8.6	670521	311
MFA-074	MeFOS024.D	24	8.4	803728	388			Day 28	6.3	0	0.0		6.0	315968	403	8.6	683560	311
MFA-075	MeFOS025.D	25	8.4	1213336	586		89%	Day 28	6.3	0	0.0		6.0	321240	403	8.6	679976	311
MeOH Blank	MeFOS028.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0	8.6	0	0
MeOH Blank	MeFOS030.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0	8.6	0	0
MFA-091	MeFOS031.D	26	8.4	793217	381	2%		Day 34	6.3	0	0.0		6.0	317397	403	8.6	687397	311
MFA-092	MeFOS032.D	27	8.4	793658	390			Day 34	6.3	0	0.0		6.0	316037	403	8.6	671514	311
MFA-093	MeFOS033.D	28	8.4	1194136	580		88%	Day 34	6.3	0	0.0		6.0	322198	403	8.6	677205	311
								(B)										
								(B)										
								(B)										
MFA-127	MeFOS034.D	29	8.6	20502				(C)	6.3	0	0.0		6.0	0	403	8.6	732926	311
MFA-128	MeFOS035.D	30	8.6	22336				(C)	6.3	0	0.0		6.0	0	403	8.6	772348	311
MFA-129	MeFOS036.D	31	8.4	410622	186			(C)	6.3	0	0.0		6.0	0	403	8.6	737726	311
MeOH Blank	MeFOS042.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0	8.6	0	0
MeOH Blank	MeFOS043.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0	8.6	0	0
99039-30-1	MeFOS044.D	1	8.4	160028	89	113%			6.3	11627	4.4	141%	6.0	321139	403	8.6	615234	311
99039-30-2	MeFOS045.D	2	8.4	296600	159	100%			6.3	20367	7.5	95%	6.0	329327	403	8.6	626824	311
99039-30-3	MeFOS046.D	3	8.4	569502	310	98%			6.3	40566	14.6	93%	6.0	332980	403	8.6	608282	311
99039-30-4	MeFOS047.D	4	8.4	862113	472	100%			6.3	80242	30.1	96%	6.0	318130	403	8.6	601758	311
99039-30-5	MeFOS048.D	5	8.4	1153879	614	97%			6.3	163639	61.1	98%	6.0	319076	403	8.6	617694	311
99039-30-6	MeFOS049.D	6	8.4	1507978	826	105%			6.3	251082	92.3	98%	6.0	323806	403	8.6	598749	311
Method ID:			Internal Standard quant: r^2=0.999						Internal Standard quant: r^2=0.999				mean	323030		mean	643498	
Original conc. (ng/ml) =		473	Curve Averaged, Linear, include origin						Curve Averaged, Linear, include origin				S.D.	6782		S.D.	30538	
Spike conc. (ng/ml) =		221	Calibration range: 79-789 ng/mL						Calibration range: 7.8-94 ng/mL				%S.D.	2.1%		%S.D.	4.7%	

(A) Data excluded on the basis of data quality objectives; see text.
(B) Samples not analyzed; see text.
(C) Internal standard omitted; see text.

BACK TO MAIN

MeFOSE Alcohol Hydrolysis Study
50C; pH 3
Retention times (RT) in minutes
All concentrations in ng/ml

			N-MeFOSE Alcohol						PFOS				THPFOS			N-EtFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc.	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	MeFOS002.D	91	0.0	0	0				6.2	12328	0.0	0.00	5.8	0	0	8.5	0	0
99039-31-1	MeFOS003.D	1	8.3	151736	82	104%			6.1	20171	3.2	101%	5.8	419594	403	8.5	636812	311
99039-31-2	MeFOS004.D	2	8.3	341079	155	98%			6.1	34709	7.8	100%	5.8	430655	403	8.5	744336	311
99039-31-3	MeFOS005.D	3	8.3	660722	301	95%			6.1	53143	13.6	87%	5.8	439792	403	8.5	738471	311
99039-31-4	MeFOS006.D	4	8.3	1127128	484	102%			6.2	106282	31.2	100%	5.9	438422	403	8.5	780745	311
99039-31-5	MeFOS007.D	5	8.3	1442949	639	101%			6.2	195877	59.4	95%	5.9	446792	403	8.5	757128	311
99039-31-6	MeFOS008.D	6	8.3	1796489	778	99%			6.2	288910	91.2	97%	5.9	435601	403	8.5	773265	311
MeOH Blank	MeFOS009.D	91	8.3	0	0				6.2	13023	0.0		5.8	0	0	8.5	0	0
MeOH Blank	MeFOS010.D	91	8.3	0	0				6.2	10891	0.0		5.8	0	0	8.5	0	0
MFA-004	MeFOS011.D	11	8.3	1134989	483	65%		(A)	6.2	14132	0.7		5.9	457701	403	8.5	788849	311
MFA-005	MeFOS012.D	12	8.3	390170	951			(A)	6.2	23260	0.0		5.9	2178603	403	8.5	137293	311
MFA-006	MeFOS013.D	13	8.3	1589221	683		-15%	(A)	6.2	11570	0.0		5.9	439722	403	8.5	779045	311
MFA-022	MeFOS014.D	14	8.3	829229	361	8%		(A)	6.2	21187	3.5		5.9	418052	403	8.5	771699	311
MFA-023	MeFOS015.D	15	8.3	764651	334			(A)	6.2	14049	1.0		5.9	425475	403	8.5	768972	311
MFA-024	MeFOS016.D	16	8.3	1403195	555		94%	(A)	6.2	13409	0.4		5.9	468367	403	8.5	847709	311
MFA-040	MeFOS017.D	17	8.3	866899	355	36%		(A)	6.2	13922	1.0		5.9	417398	403	8.5	820997	311
MFA-041	MeFOS018.D	18	8.3	597135	247			(A)	6.2	11887	0.3		5.9	418159	403	8.5	815246	311
MFA-042	MeFOS019.D	19	8.3	1297314	520		99%	(A)	6.2	14084	1.1		5.9	412958	403	8.5	837107	311
MFA-058	MeFOS020.D	20	8.3	825047	327	7%		Day 20	6.2	12959	0.7		5.9	420234	403	8.5	847509	311
MFA-059	MeFOS021.D	21	8.3	869828	349			Day 20	6.2	13631	0.8		5.9	426878	403	8.5	836796	311
MFA-060	MeFOS022.D	22	8.3	1317427	537		90%	Day 20	6.2	13975	1.2		5.9	408760	403	8.5	822995	311
MFA-076	MeFOS023.D	23	8.3	915705	367	20%		Day 28	6.2	12740	0.7		5.9	414108	403	8.5	839193	311
MFA-077	MeFOS024.D	24	8.3	738271	300			Day 28	6.2	12296	0.6		5.9	407935	403	8.5	827596	311
MFA-078	MeFOS025.D	25	8.3	1351582	553		100%	Day 28	6.2	13770	1.1		5.9	404854	403	8.5	819079	311
MeOH Blank	MeFOS026.D	91	8.3	0	0				6.2	12586	0.0		5.9	0	0	8.5	0	0
MeOH Blank	MeFOS028.D	91	8.3	0	0				6.2	11439	0.0		5.9	0	0	8.5	0	0
MFA-094	MeFOS029.D	26	8.3	901464	371	1%		Day 34	6.2	12253	0.6		5.9	406516	403	8.5	817359	311
MFA-095	MeFOS030.D	27	8.3	908936	375			Day 34	6.2	16034	2.0		5.9	402971	403	8.5	814894	311
MFA-096	MeFOS031.D	28	8.3	1346477	561		85%	Day 34	6.2	12839	0.9		5.9	396895	403	8.5	804552	311
MFA-112	MeFOS032.D	29	8.3	952582	359	4%		Day 42	6.2	12120	0.5		5.9	406709	403	8.5	890510	311
MFA-113	MeFOS033.D	30	8.3	907374	346			Day 42	6.2	13021	0.8		5.9	410407	403	8.5	881985	311
MFA-114	MeFOS034.D	31	8.3	1435453	545		87%	Day 42	6.2	13729	1.1		5.9	405375	403	8.5	882486	311
MFA-130	MeFOS035.D	32	8.3	948868	348	2%		Day 49	6.2	13388	0.9		5.9	416004	403	8.5	920660	311
MFA-131	MeFOS036.D	33	8.3	919227	339			Day 49	6.2	13132	0.8		5.9	410564	403	8.5	911546	311
MFA-132	MeFOS037.D	34	8.3	1428869	525		82%	Day 49	6.2	12775	0.7		5.9	408186	403	8.5	912801	311
MeOH Blank	MeFOS043.D	91	8.3	0	0				6.2	12279	0.0		5.9	0	0	8.5	0	0
MeOH Blank	MeFOS044.D	91	8.3	0	0				6.2	12022	0.0		5.9	0	0	8.5	0	0
99039-31-1	MeFOS045.D	1	8.3	168468	79	100%			6.2	21343	3.8	121%	5.9	406294	403	8.5	730702	311
99039-31-2	MeFOS046.D	2	8.3	336376	158	100%			6.2	33543	8.2	105%	5.9	403340	403	8.5	723068	311
99039-31-3	MeFOS047.D	3	8.3	654789	308	97%			6.2	53137	15.4	98%	5.9	400256	403	8.5	715896	311
99039-31-4	MeFOS048.D	4	8.3	1038617	487	103%			6.2	101279	33.3	107%	5.9	393427	403	8.5	715694	311
99039-31-5	MeFOS049.D	5	8.3	1350045	640	101%			6.2	189417	64.5	103%	5.9	399772	403	8.5	706581	311
99039-31-6	MeFOS050.D	6	8.3	1678778	781	99%			6.2	272461	97.1	103%	5.9	389466	403	8.5	719573	311

Method ID: MFO_0828. M — Internal Standard quant: r2 = 0.998 — Internal Standard quant: r2 = 0.997 — mean 417298 — mean 800052
Original conc. (ng/ml) = 473.4 — Curve averaged, Linear,include origin. — Curve averaged, Linear,include origin. — S.D. 18014 — S.D. 66857
Spike conc. (ng/ml) = 221 — Calibration range: 155-932 ng/ml — Calibration range: 15.7-94 ng/ml — %S.D. 4.3% — %S.D. 8.4%

(A) Data excluded on the basis of data quality objectives; see text.

BACK TO MAIN

MeFOSE Alcohol Hydrolysis Study
50C; pH 5
Retention times (RT) in minutes
All concentrations in ng/ml

			N-MeFOSE Alcohol						PFOS				THPFOS			N-EtFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc.	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	MeFOS051.D	92	8.4	0	0				6.3	0	0.0	0.0%	6.0	0	0.00	8.6	0	0
99039-32-1	MeFOS052.D	41	8.4	149060	82	104%			6.3	9155	3.5	111%	6.0	316040	402.60	8.6	598824	311
99039-32-2	MeFOS053.D	42	8.4	280351	159	101%			6.3	18855	7.4	94%	6.0	313687	402.60	8.6	589170	311
99039-32-3	MeFOS054.D	43	8.4	544208	306	97%			6.3	69313	27.2	174%	6.0	315295	402.60	8.6	597555	311
99039-32-4	MeFOS055.D	44	8.4	858881	485	103%			6.3	81289	31.6	101%	6.0	318179	402.60	8.6	596598	311
99039-32-5	MeFOS056.D	45	8.4	1154462	628	99%			6.3	178117	66.6	106%	6.0	331279	402.60	8.6	620432	311
99039-32-6	MeFOS057.D	46	8.4	1368379	765	100%			6.3	240605	93.4	99%	6.0	319232	402.60	8.6	588439	311
MeOH Blank	MeFOS058.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0.00	8.6	0	0
MeOH Blank	MeFOS059.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0.00	8.6	0	0
MFA-007	MeFOS060.D	51	8.4	966445	482	0%		Day 0	6.3	0	0.0		6.0	369039	402.60	8.6	676077	311
MFA-007	MeFOS061.D	51	8.4	972256	483			Day 0	6.3	0	0.0		6.0	358611	402.60	8.6	678936	311
MFA-009	MeFOS062.D	53	8.4	1262465	697		97%	Day 0	6.3	0	0.0		6.0	330072	402.60	8.6	611827	311
MFA-025	MeFOS063.D	54	8.4	764741	428	5%		Day 7	6.3	0	0.0		6.0	318812	402.60	8.6	602188	311
MFA-026	MeFOS064.D	55	8.4	801784	450			Day 7	6.3	0	0.0		6.0	321150	402.60	8.6	600482	311
MFA-027	MeFOS065.D	56	8.4	1155792	641		92%	Day 7	6.3	0	0.0		6.0	304415	402.60	8.6	608185	311
MFA-043	MeFOS066.D	57	8.4	778763	406	0%		Day 14	6.3	0	0.0		6.0	318966	402.60	8.6	645859	311
MFA-044	MeFOS067.D	58	8.4	818063	408			Day 14	6.3	0	0.0		6.0	327562	402.60	8.6	676091	311
MFA-045	MeFOS068.D	59	8.4	1165951	607		91%	Day 14	6.3	0	0.0		6.0	316845	402.60	8.6	647772	311
MFA-061	MeFOS069.D	60	8.4	791698	394	0%		Day 20	6.3	0	0.0		6.0	318539	402.60	8.6	676708	311
MFA-062	MeFOS070.D	61	8.4	798843	394			Day 20	6.3	0	0.0		6.0	321130	402.60	8.6	681943	311
MFA-063	MeFOS071.D	62	8.4	1174315	601		94%	Day 20	6.3	0	0.0		6.0	326528	402.60	8.6	659289	311
MFA-079	MeFOS072.D	63	8.4	773986	401	1%		Day 28	6.3	0	0.0		6.0	316457	402.60	8.6	650045	311
MFA-080	MeFOS073.D	64	8.4	768748	396			Day 28	6.3	0	0.0		6.0	314675	402.60	8.6	653653	311
MFA-081	MeFOS074.D	65	8.4	1157303	597		90%	Day 28	6.3	0	0.0		6.0	314393	402.60	8.6	654428	311
MeOH Blank	MeFOS075.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0.00	8.6	0	0
MeOH Blank	MeFOS077.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0.00	8.6	0	0
MFA-097	MeFOS078.D	66	8.4	768874	383	1%		Day 34	6.3	0	0.0		6.0	315754	402.60	8.6	676650	311
MFA-098	MeFOS079.D	67	8.4	750087	379			Day 34	6.3	0	0.0		6.0	313136	402.60	8.6	666126	311
MFA-099	MeFOS080.D	68	8.4	1142873	586		93%	Day 34	6.3	0	0.0		6.0	315673	402.60	8.6	658463	311
MFA-115	MeFOS081.D	69	8.4	750291	359	1%		Day 42	6.3	0	0.0		6.0	308799	402.60	8.6	702846	311
MFA-116	MeFOS082.D	70	8.4	771988	358			Day 42	6.3	0	0.0		6.0	323683	402.60	8.6	729469	311
MFA-117	MeFOS083.D	71	8.4	1161257	562		92%	Day 42	6.3	0	0.0		6.0	317017	402.60	8.6	697385	311
MFA-133	MeFOS084.D	72	8.4	765373	354	1%		Day 49	6.3	0	0.0		6.0	319485	402.60	8.6	727348	311
MFA-134	MeFOS085.D	73	8.4	779909	359			Day 49	6.3	0	0.0		6.0	320409	402.60	8.6	731061	311
MFA-135	MeFOS086.D	74	8.4	1157828	559		91%	Day 49	6.3	0	0.0		6.0	314997	402.60	8.6	699055	311
MeOH Blank	MeFOS092.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0.00	8.6	0	0
MeOH Blank	MeFOS093.D	92	8.4	0	0				6.3	0	0.0		6.0	0	0.00	8.6	0	0
99039-32-1	MeFOS094.D	41	8.4	146569	82	104%			6.3	8210	3.1	99%	6.0	317732	402.60	8.6	590591	311
99039-32-2	MeFOS095.D	42	8.4	276797	158	100%			6.3	18161	7.0	90%	6.0	316858	402.60	8.6	584803	311
99039-32-3	MeFOS096.D	43	8.4	535672	306	97%			6.3	67607	26.4	169%	6.0	315957	402.60	8.6	588851	311
99039-32-4	MeFOS097.D	44	8.4	839851	483	102%			6.3	77275	29.9	96%	6.0	319036	402.60	8.6	585887	311
99039-32-5	MeFOS098.D	45	8.4	1133812	627	99%			6.3	168159	63.7	102%	6.0	326660	402.60	8.6	610173	311
99039-32-6	MeFOS099.D	46	8.4	1354192	793	100%			6.3	241085	91.5	97%	6.0	326403	402.60	8.6	577012	311
Method ID:			Internal Standard quant: r^2=0.999						Internal Standard quant: r^2=0.999				mean	321175		mean	642778	
Original conc. (ng/ml) =		473.4	Curve Averaged, Linear, Include origin						Curve Averaged, Linear, Include origin				S.D.	11910		S.D.	46797	
Spike conc. (ng/ml) =		221	Calibration range: 79-789 ng/mL						Calibration range: 3-94 ng/mL				%S.D.	3.7%		%S.D.	7.3%	

BACK TO MAIN

MeFOSE Alcohol Hydrolysis Study
50C; pH 7
Retention times (RT) in minutes
All concentrations in ng/ml

			N-MeFOSE Alcohol						PFOS				THPFOS			N-EtFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc.	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	MeFOS052.D	91	8.3	0	0				6.2	12060	0.0	0.00	5.9	0	0	8.5	0	0
99039-33-1	MeFOS053.D	41	8.3	163904	74	94%			6.2	21926	3.4	110%	5.9	413061	403	8.5	722548	311
99039-33-2	MeFOS054.D	42	8.3	353900	162	103%			6.2	33291	7.3	94%	5.9	415745	403	0.0	726841	311
99039-33-3	MeFOS055.D	43	8.3	672851	313	99%			6.2	54104	14.6	93%	5.9	414516	403	0.0	721094	311
99039-33-4	MeFOS056.D	44	8.3	1043250	487	103%			6.2	120587	37.5	120%	5.9	416734	403	0.0	721008	311
99039-33-5	MeFOS057.D	45	8.3	1848028	863	137%			6.2	197863	64.7	103%	5.9	414375	403	0.0	722268	311
99039-33-6	MeFOS058.D	46	8.3	1678345	781	99%			6.2	283971	93.6	100%	5.9	418593	403	0.0	724772	311
MeOH Blank	MeFOS059.D	91	8.3	0	0				6.2	10884	0.0		5.9	0	0	0.0	0	0
MeOH Blank	MeFOS060.D	91	8.3	0	0				6.2	8447	0.0		5.9	0	0	0.0	0	0
MFA-010	MeFOS061.D	51	8.3	1059849	463	2%		Day 0	6.2	13752	0.4		5.9	431433	403	0.0	769705	311
MFA-011	MeFOS062.D	52	8.3	1035099	471			Day 0	6.2	13431	0.3		5.9	427482	403	0.0	738896	311
MFA-012	MeFOS063.D	53	8.3	1493105	693		102%	Day 0	6.2	12470	0.0		5.9	424804	403	0.0	726047	311
MFA-028	MeFOS064.D	54	8.3	950167	440	2%		Day 7	6.2	12076	0.1		5.9	406484	403	0.0	725630	311
MFA-029	MeFOS065.D	55	8.3	971370	451			Day 7	6.2	11626	0.0		5.9	412600	403	0.0	724750	311
MFA-030	MeFOS066.D	56	8.3	1421931	668		100%	Day 7	6.2	12182	0.1		5.9	408432	403	0.0	717606	311
MFA-046	MeFOS067.D	57	8.3	945561	409	1%		Day 14	6.2	11943	0.0		5.9	410627	403	0.0	777412	311
MFA-047	MeFOS068.D	58	8.3	955303	412			Day 14	6.2	10864	0.0		5.9	411793	403	0.0	780071	311
MFA-048	MeFOS069.D	59	8.3	1390135	604		88%	Day 14	6.2	11746	0.0		5.9	405115	403	0.0	775133	311
MFA-064	MeFOS070.D	60	8.3	960628	398	1%		Day 20	6.2	10395	0.0		5.9	415819	403	0.0	812219	311
MFA-065	MeFOS071.D	61	8.3	966872	400			Day 20	6.2	9801	0.0		5.9	425350	403	0.0	812637	311
MFA-066	MeFOS072.D	62	8.3	1429859	597		90%	Day 20	6.2	11029	0.0		5.9	416204	403	0.0	806256	311
MFA-082	MeFOS073.D	63	8.3	938910	387	3%		Day 28	6.2	10525	0.0		5.9	414343	403	0.0	816223	311
MFA-083	MeFOS074.D	64	8.3	938077	400			Day 28	6.2	10803	0.0		5.9	406804	403	0.0	789126	311
MFA-084	MeFOS075.D	65	8.3	1373974	590		89%	Day 28	6.2	10416	0.0		5.9	406568	403	0.0	784737	311
MeOH Blank	MeFOS076.D	91	8.3	0	0				6.2	10001	0.0		5.9	0	0	0.0	0	0
MeOH Blank	MeFOS078.D	91	8.3	0	0				6.2	10980	0.0		5.9	0	0	0.0	0	0
MFA-100	MeFOS079.D	66	8.3	917975	398	1%		Day 34	6.2	10737	0.0		5.9	396679	403	0.0	774961	311
MFA-101	MeFOS080.D	67	8.3	917551	395			Day 34	6.2	10892	0.0		5.9	401501	403	0.0	781448	311
MFA-102	MeFOS081.D	68	8.3	1364592	618		100%	Day 34	6.2	10866	0.0		5.9	395834	403	0.0	744222	311
MFA-118	MeFOS082.D	69	8.3	908151	380	3%		Day 42	6.2	10383	0.0		5.9	387918	403	0.0	802155	311
MFA-119	MeFOS083.D	70	8.3	875018	369			Day 42	6.2	11979	0.2		5.9	389550	403	0.0	796321	311
MFA-120	MeFOS084.D	71	8.3	1355262	565		86%	Day 42	6.2	10102	0.0		5.9	397888	403	0.0	808103	311
MFA-136	MeFOS085.D	72	8.3	894105	366	3%		Day 49	6.2	10966	0.0		5.9	392635	403	0.0	821314	311
MFA-137	MeFOS086.D	73	8.3	871427	353			Day 49	6.2	10552	0.0		5.9	392646	403	0.0	828208	311
MFA-138	MeFOS087.D	74	8.3	1360766	544		84%	Day 49	6.2	11994	0.1		5.9	404326	403	0.0	841482	311
MeOH Blank	MeFOS093.D	91	8.3	0	0				6.2	9877	0.0		5.9	0	0	0.0	0	0
MeOH Blank	MeFOS094.D	91	8.3	0	0				6.2	10936	0.0		5.9	0	0	mean	0	0
99039-33-1	MeFOS095.D	41	8.3	162533	75	95%			6.2	17446	1.9	62%	5.9	407992	403	mean	707642	311
99039-33-2	MeFOS096.D	42	8.3	338524	161	102%			6.2	29991	6.4	82%	5.9	407169	403	S.D.	700669	311
99039-33-3	MeFOS097.D	43	8.3	651730	310	98%			6.2	58016	16.2	103%	5.9	409832	403	mean	705419	311
99039-33-4	MeFOS098.D	44	8.3	1013215	480	101%			6.2	112160	35.3	113%	5.9	409148	403	S.D.	709949	311
99039-33-5	MeFOS099.D	45	8.3	1816530	853	135%			6.2	190397	61.9	99%	5.9	415661	403	mean	718262	311
99039-33-6	MeFOS100.D	46	8.3	1634822	788	100%			6.2	272629	90.2	96%	5.9	416714	403	S.D.	699829	311
Method ID:			Internal Standard quant: r2 = 0.998						Internal Standard quant: r2 = 0.997				mean	409510		mean	759304	
Original conc. (ng/ml) =		473.4	Curve averaged, Linear,include origin.						Curve averaged, Linear,include origin.				S.D.	10423		S.D.	43356	
Spike conc. (ng/ml) =		221	Calibration range: 155-932 ng/ml						Calibration range: 15.7-94 ng/ml				%S.D.	2.5%		%S.D.	5.7%	

BACK TO MAIN

MeFOSE Alcohol Hydrolysis Study
50C; pH 9
Retention times (RT) in minutes
All concentrations in ng/ml

			N-MeFOSE Alcohol						PFOS				THPFOS			N-EtFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc.	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	MeFOS011.D	92	8.4	0	0.0				6.2	0	0.0	0%	5.9	0	0.0	8.6	0	0.0
99039-34-1	MeFOS012.D	1	8.4	163460	80	102%			6.2	15569	5.5	175%	5.9	237029	402.6	8.6	699032	311
99039-34-2	MeFOS013.D	2	8.4	309997	146	92%			6.2	22667	8.7	111%	5.9	232506	402.6	8.6	706051	311
99039-34-3	MeFOS014.D	3	8.4	680067	304	96%			6.2	35291	13.9	89%	5.9	235554	402.6	8.6	726783	311
99039-34-4	MeFOS015.D	4	8.4	1006367	464	98%			6.2	76745	31.3	100%	5.9	236190	402.6	8.6	700715	311
99039-34-5	MeFOS016.D	5	8.4	1546780	692	110%			6.2	166823	67.2	107%	5.9	243630	402.6	8.6	719387	311
99039-34-6	MeFOS017.D	6	8.4	1664630	761	96%			6.2	248041	100.3	107%	5.9	244027	402.6	8.6	703692	311
MeOH Blank	MeFOS018.D	92	0.0	0	0				6.2	0	0.0		0.0	0	0.0	8.6	0	0
MeOH Blank	MeFOS019.D	92	0.0	0	0				6.2	0	0.0		0.0	0	0.0	8.6	0	0
MFA-013	MeFOS020.D	11	8.4	1028452	463	25%		Day 0	6.2	0	0.0		5.9	260732	402.6	8.6	717735	311
MFA-014	MeFOS021.D	12	8.4	847651	360			Day 0	6.2	0	0.0		5.9	213698	402.6	8.6	764466	311
MFA-015	MeFOS022.D	13	8.4	1569790	668		115%	Day 0	6.2	0	0.0		5.9	278506	402.6	8.6	759367	311
MFA-031	MeFOS023.D	14	8.4	996448	440	1%		Day 7	6.2	0	0.0		5.9	257996	402.6	8.6	733413	311
MFA-032	MeFOS024.D	15	8.4	964111	434			Day 7	6.2	0	0.0		5.9	260367	402.6	8.6	718160	311
MFA-033	MeFOS025.D	16	8.4	1413056	654		98%	Day 7	6.2	0	0.0		5.9	251637	402.6	8.6	696132	311
MFA-049	MeFOS026.D	17	8.4	1033316	396	1%		Day 14	6.2	0	0.0		5.9	281850	402.6	8.6	845429	311
MFA-050	MeFOS027.D	18	8.4	940771	392			Day 14	6.2	0	0.0		5.9	262960	402.6	8.6	777318	311
MFA-051	MeFOS028.D	19	8.4	1387782	588		88%	Day 14	6.2	0	0.0		5.9	257391	402.6	8.6	761035	311
MFA-067	MeFOS029.D	20	8.4	972226	393	0%		Day 20	6.2	0	0.0		5.9	268694	402.6	8.6	802228	311
MFA-068	MeFOS030.D	21	8.4	965677	394			Day 20	6.2	0	0.0		5.9	271896	402.6	8.6	795082	311
MFA-069	MeFOS031.D	22	8.4	1405487	570		80%	Day 20	6.2	0	0.0		5.9	268042	402.6	8.6	794941	311
MFA-085	MeFOS032.D	23	8.4	944313	383	0%		Day 28	6.2	0	0.0		5.9	269583	402.6	8.6	800204	311
MFA-086	MeFOS033.D	24	8.4	926910	384			Day 28	6.2	0	0.0		5.9	268863	402.6	8.6	783174	311
MFA-087	MeFOS034.D	25	8.4	1407593	576		87%	Day 28	6.2	0	0.0		5.9	272969	402.6	8.6	788327	311
MeOH Blank	MeFOS035.D	92	0.0	0	0				6.2	0	0.0		5.9	0	0.0	8.6	0	0
MeOH Blank	MeFOS037.D	92	0.0	0	0				6.2	0	0.0		5.9	0	0.0	8.6	0	0
MFA-103	MeFOS038.D	26	8.4	942289	378	2%		Day 34	6.2	0	0.0		5.9	287634	402.6	8.6	807638	311
MFA-104	MeFOS039.D	27	8.4	923555	386			Day 34	6.2	0	0.0		5.9	288790	402.6	8.6	776325	311
MFA-105	MeFOS040.D	28	8.4	1390550	561		81%	Day 34	6.2	0	0.0		5.9	283426	402.6	8.6	800018	311
MFA-121	MeFOS041.D	29	8.4	929686	361	3%		Day 42	6.2	0	0.0		5.9	285239	402.6	8.6	835761	311
MFA-122	MeFOS042.D	30	8.4	903803	349			Day 42	6.2	0	0.0		5.9	283638	402.6	8.6	841667	311
MFA-123	MeFOS043.D	31	8.4	1418215	541		84%	Day 42	6.2	0	0.0		5.9	291538	402.6	8.6	846199	311
MFA-139	MeFOS044.D	32	8.4	918039	345	2%		Day 49	6.2	0	0.0		5.9	293710	402.6	8.6	863218	311
MFA-140	MeFOS045.D	33	8.4	930094	353			Day 49	6.2	0	0.0		5.9	296728	402.6	8.6	855340	311
MFA-141	MeFOS046.D	34	8.4	1380184	536		85%	Day 49	6.2	0	0.0		5.9	293597	402.6	8.6	831130	311
MeOH Blank	MeFOS052.D	92	8.4	0	0				6.2	0	0.0		5.9	0	0.0	8.6	0	0
MeOH Blank	MeFOS053.D	92	8.4	0	0				6.2	0	0.0		5.9	0	0.0	8.6	0	0
99039-34-1	MeFOS054.D	1	8.4	171724	81	103%			6.1	19200	4.9	156%	5.9	321749	402.6	8.6	725918	311
99039-34-2	MeFOS055.D	2	8.4	314956	150	95%			6.2	25918	7.1	91%	5.9	316093	402.6	8.6	698921	311
99039-34-3	MeFOS056.D	3	8.4	693304	311	99%			6.2	43090	12.5	80%	5.9	317775	402.6	8.6	724207	311
99039-34-4	MeFOS057.D	4	8.4	1012133	466	99%			6.2	106004	32.4	104%	5.9	315713	402.6	8.6	701526	311
99039-34-5	MeFOS058.D	5	8.4	1499739	685	109%			6.2	192716	59.1	94%	5.9	319381	402.6	8.6	704933	311
99039-34-6	MeFOS059.D	6	8.4	1623048	744	94%			6.2	278633	87.0	93%	5.9	315350	402.6	8.6	701733	311
Method ID			Internal Standard quant: r^2 = 0.995						Internal Standard quant: r^2 = 0.995				Mean	274020		Mean	764089	
Original conc. (ng/ml) =		473.4	Curve averaged, linear, include origin						Curve averaged, linear, include origin				S.D.	27955		S.D.	54352	
Spike conc. (ng/ml) =		221	Calibration range : 155-932 ng/ml						Calibration range : 7.8-94 ng/ml				%S.D.	10.2%		%S.D.	7.1%	

BACK TO MAIN

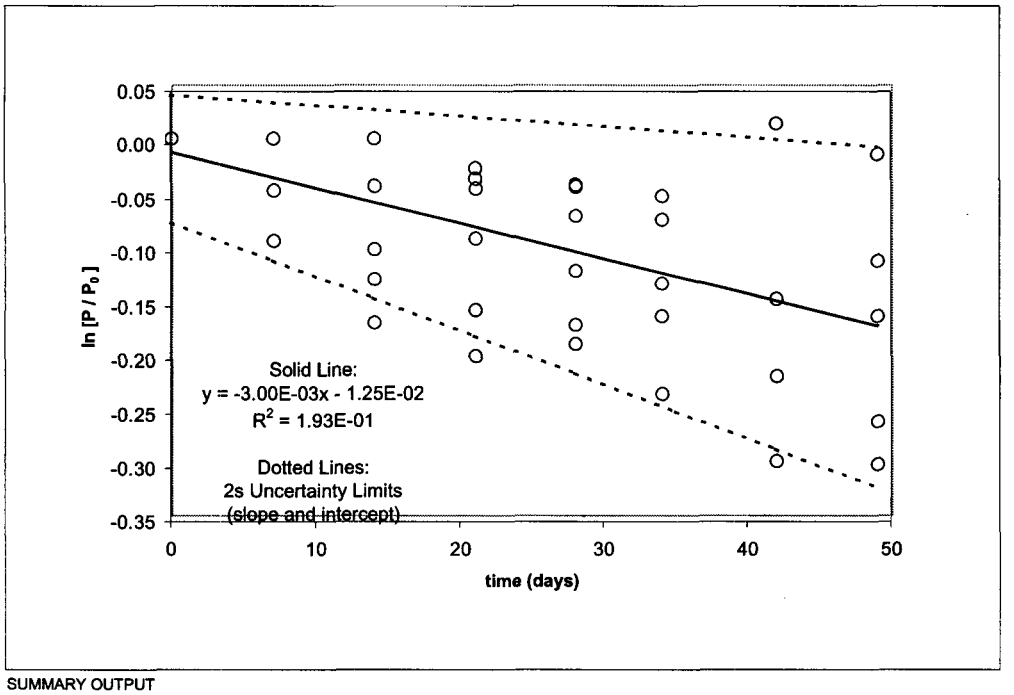
MeFOSE Alcohol Hydrolysis Study
50C; pH 11
Retention times (RT) in minutes
All concentrations in ng/ml

			N-MeFOSE Alcohol						PFOS				THPFOS			N-EtFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc.	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	MeFOS061.D	92	0.0	0	0				6.2	0	0.0	0.0	5.9	0	0	8.6	0	0
99039-35-1	MeFOS062.D	41	8.4	162927	81	103%			6.2	8592	2.4	76%	5.9	334451	403	8.6	693381	311
99039-35-2	MeFOS063.D	42	8.4	319287	145	92%			6.2	24325	7.8	99%	5.9	336584	403	8.6	748590	311
99039-35-3	MeFOS064.D	43	8.4	647404	308	98%			6.2	43465	14.2	91%	5.9	340261	403	8.6	706006	311
99039-35-4	MeFOS065.D	44	8.4	1035120	490	103%			6.2	97689	32.5	104%	5.9	341410	403	8.6	707232	311
99039-35-5	MeFOS066.D	45	8.4	1363926	650	103%			6.2	203483	67.1	107%	5.9	347910	403	8.6	701518	311
99039-35-6	MeFOS067.D	46	8.4	1661391	764	97%			6.2	297189	97.4	104%	5.9	350784	403	8.6	726509	311
MeOH Blank	MeFOS068.D	92	0.0	0	0				6.2	0	0.0		0.0	0	0	8.6	0	0
MeOH Blank	MeFOS069.D	92	0.0	0	0				6.2	0	0.0		0.0	0	0	8.6	0	0
MFA-016	MeFOS070.D	51	8.4	1015186	473	3%		(A)	6.2	0	0.0		5.9	361052	403	8.6	719209	311
MFA-017	MeFOS071.D	52	8.4	1001588	467			(A)	6.2	0	0.0		5.9	358929	403	8.6	688438	311
MFA-018	MeFOS072.D	53	8.4	1454983			-217%	(A)	6.2	0	0.0		5.9	365070	403	8.6	23244	311
MFA-034	MeFOS073.D	54	8.4	932168	448	1%		(A)	6.2	0	0.0		5.9	350786	403	8.6	696227	311
MFA-035	MeFOS074.D	55	8.4	933193	451			(A)	6.2	0	0.0		5.9	346018	403	8.6	693022	311
MFA-038	MeFOS075.D	56	8.4	1349755			-203%	(A)	6.2	0	0.0		5.9	351584	403	8.6	0	0
MFA-052	MeFOS076.D	57	8.4	1333450	410	2%		Day 14	6.2	0	0.0		5.9	518509	403	8.6	1090670	311
MFA-053	MeFOS077.D	58	8.4	916402	416			Day 14	6.2	0	0.0		5.9	349233	403	8.6	738040	311
MFA-054	MeFOS078.D	59	8.4	1330022	609		89%	Day 14	6.2	0	0.0		5.9	339650	403	8.6	729875	311
MFA-070	MeFOS079.D	60	8.4	924714	396	1%		Day 20	6.2	0	0.0		5.9	356993	403	8.6	783229	311
MFA-071	MeFOS080.D	61	8.4	933652	400			Day 20	6.2	0	0.0		5.9	362219	403	8.6	781943	311
MFA-072	MeFOS081.D	62	8.4	1341660	598		90%	Day 20	6.2	0	0.0		5.9	345470	403	8.6	750255	311
MFA-088	MeFOS082.D	63	8.4	923952	397	1%		Day 28	6.2	0	0.0		5.9	360186	403	8.6	780059	311
MFA-089	MeFOS083.D	64	8.4	886677	393			Day 28	6.2	0	0.0		5.9	352051	403	8.6	755853	311
MFA-090	MeFOS084.D	65	8.4	1320368	590		88%	Day 28	6.2	0	0.0		5.9	345627	403	8.6	747940	311
MeOH Blank	MeFOS085.D	92	0.0	0	0				6.2	0	0.0		0.0	0	0	8.6	0	0
MeOH Blank	MeFOS087.D	92	0.0	0	0				6.2	0	0.0		0.0	0	0	8.6	0	0
MFA-106	MeFOS088.D	66	8.4	895537	389	1%		Day 34	6.2	0	0.0		5.9	355343	403	8.6	772071	311
MFA-107	MeFOS089.D	67	8.4	889957	395			Day 34	6.2	0	0.0		5.9	353220	403	8.6	755961	311
MFA-108	MeFOS090.D	68	8.4	1330772	588		89%	Day 34	6.2	0	0.0		5.9	356886	403	8.6	756782	311
MFA-124	MeFOS091.D	69	8.4	900341	374	37%		(A)	6.2	0	0.0		5.9	361820	403	8.6	806499	311
MFA-125	MeFOS092.D	70	8.4	866673	543			(A)	6.2	0	0.0		5.9	354024	403	8.6	533536	311
MFA-126	MeFOS093.D	71	8.4	1299388	546		39%	(A)	6.2	0	0.0		5.9	348654	403	8.6	796217	311
MFA-142	MeFOS094.D	72	8.4	876044	367	1%		Day 49	6.2	0	0.0		5.9	357923	403	8.6	799439	311
MFA-143	MeFOS095.D	73	8.4	906488	370			Day 49	6.2	0	0.0		5.9	372805	403	8.6	820989	311
MFA-144	MeFOS096.D	74	8.4	1302131	544		79%	Day 49	6.2	0	0.0		5.9	358209	403	8.6	800015	311
MeOH Blank	MeFOS102.D	92	0.0	0	0				6.2	0	0.0		0.0	0	0	8.6	0	0
MeOH Blank	MeFOS103.D	92	0.0	0	0				6.2	0	0.0		0.0	0	0	8.6	0	0
99039-35-1	MeFOS104.D	41	8.4	160080	82	104%			6.2	8830	2.2	70%	5.9	364922	403	8.6	673354	311
99039-35-2	MeFOS105.D	42	8.4	305109	146	93%			6.2	24409	7.1	90%	5.9	368496	403	8.6	707289	311
99039-35-3	MeFOS106.D	43	8.4	621321	310	98%			6.2	44065	13.1	84%	5.9	372249	403	8.6	673339	311
99039-35-4	MeFOS107.D	44	8.4	984994	496	105%			6.2	122620	37.8	121%	5.9	369974	403	8.6	664376	311
99039-35-5	MeFOS108.D	45	8.4	1292328	636	101%			6.2	199728	61.8	99%	5.9	370582	403	8.6	679484	311
99039-35-6	MeFOS109.D	46	8.4	1570588	782	99%			6.2	282960	86.1	92%	5.9	377502	403	8.6	670949	311
Method ID:			Internal Standard quant: r2 = 0.999						Internal Standard quant: r2 = 0.995				Mean	359927		Mean	749151	
Original conc. (ng/ml) =		473.4	Curve averaged, Linear,include origin.						Curve averaged, Linear,include origin.				S.D.	29217		S.D.	84954	
Spike conc. (ng/ml) =		221	Calibration range: 155-932 ng/ml						Calibration range: 7.8-94 ng/ml				%S.D.	8.1%		%S.D.	11.3%	

(A) Data excluded on the basis of data quality objectives; see text.

BACK TO MAIN

Pooled N-MeFOSE Alcohol Data and Slope Regression



Solid Line:

Dotted Lines:
2s Uncertainty Limits
(slope and intercept)

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.438823641
R Square	0.192566188
Adjusted R Sq	0.17131793
Standard Erro	0.09318559
Observations	40

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	0.078696282	0.078696282	9.062681099	0.004617044
Residual	38	0.329975058	0.008683554		
Total	39	0.408671341			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-0.012516255	0.029261069	-0.427744267	0.671251755	-0.071752194	0.046719685	-0.071752194	0.046719685
X Variable 1	-0.002996307	0.000995309	-3.010428723	0.004617044	-0.005011205	-0.000981409	-0.005011205	-0.000981409

%2 s slope uncertainty
66.4

Appendix D: Selected Chromatograms

A representative set of chromatograms from the present study is included in this Appendix.

Method C:\HPCHEM\1\METHODS\1227_70I.M

Calibration Table

pH 7.0, FIRST STD. CURVE

Calib. Data Modified : Wednesday, December 29, 1999 9:20:34 AM

Calculate : Internal Standard
Based on : Peak Area

JJT 9/8/00

APPENDIX

THIS SERIES OF CHROMATOGRAMS IS A COPY OF MATERIAL PRESENTED IN 3M ARCHIVES FOR THIS PROJECT.

Rel. Reference Window : 10.000 %
Abs. Reference Window : 0.000 min
Rel. Non-ref. Window : 10.000 %
Abs. Non-ref. Window : 0.000 min
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 0.00000
Uncalibrated Peaks : not reported
Partial Calibration : Yes, identified peaks are recalibrated
Correct All Ret. Times: No, only for identified peaks

Curve Type : Linear
Origin : Included
Weight : Equal

SHOWN ARE:
- STD CURVE
- SOLVENT BLANK
- SAMPLE
- DUPLICATE
- MATRIX SPIKE

Recalibration Settings:
Average Response : Average all calibrations
Average Retention Time: Floating Average New 75%

Calibration Report Options :
Printout of recalibrations within a sequence:
Calibration Table after Recalibration
Normal Report after Recalibration
If the sequence is done with bracketing:
Results of first cycle (ending previous bracket)

Sample ISTD Information:

ISTD #	ISTD Amount [ppb]	Name
1	402.60000	THPFOS
2	310.80000	EtFOSE-OH

Signal 1: MSD1 427, EIC=426.7:427.7
Signal 2: MSD1 499, EIC=498.7:499.7
Signal 3: MSD1 630, EIC=629.7:630.7
Signal 4: MSD1 616, EIC=615.7:616.7

RetTime [min]	Sig	Lvl	Amount [ppb]	Area	Amt/Area	Ref	Grp	Name
5.870	1	1	402.60000	4.13061e5	9.74674e-4	I1		THPFOS
		2	402.60000	4.15745e5	9.68383e-4			
		3	402.60000	4.14516e5	9.71252e-4			
		4	402.60000	4.16734e5	9.66084e-4			
		5	402.60000	4.14375e5	9.71584e-4			
		6	402.60000	4.18593e5	9.61793e-4			
		11	402.60000	4.07992e5	9.86784e-4			

Method C:\HPCHEM\1\METHODS\1227_70I.M

RetTime [min]	Sig	Lvl	Amount [ppb]	Area	Amt/Area	Ref	Grp	Name
		12	402.60000	4.07169e5	9.88780e-4			
		13	402.60000	4.09832e5	9.82353e-4			
		14	402.60000	4.09148e5	9.83996e-4			
		15	402.60000	4.15661e5	9.68579e-4			
		16	402.60000	4.16714e5	9.66131e-4			
6.162	2	1	3.13000	2.19264e4	1.42750e-4	1		PFOS
		11	3.13000	1.74464e4	1.79406e-4			
		2	7.83000	3.32915e4	2.35195e-4			
		12	7.83000	2.99914e4	2.61075e-4			
		3	15.65000	5.41038e4	2.89259e-4			
		13	15.65000	5.80162e4	2.69752e-4			
		4	31.30000	1.20587e5	2.59563e-4			
		14	31.30000	1.12160e5	2.79065e-4			
		5	62.60000	1.97863e5	3.16381e-4			
		15	62.60000	1.90397e5	3.28787e-4			
		6	93.90000	2.83971e5	3.30667e-4			
		16	93.90000	2.72629e5	3.44424e-4			
8.306	4	1	78.90000	1.63904e5	4.81379e-4	2		MeFOSE-OH
		11	78.90000	1.62533e5	4.85439e-4			
		2	157.80000	3.53900e5	4.45889e-4			
		12	157.80000	3.38524e5	4.66141e-4			
		3	315.60000	6.72851e5	4.69049e-4			
		13	315.60000	6.51730e5	4.84249e-4			
		4	473.40000	1.04325e6	4.53774e-4			
		14	473.40000	1.01321e6	4.67226e-4			
		6	789.00000	1.67835e6	4.70106e-4			
		16	789.00000	1.63482e6	4.82621e-4			
8.535	3	1	310.80000	7.22548e5	4.30145e-4	I2		EtFOSE-OH
		2	310.80000	7.26841e5	4.27604e-4			
		3	310.80000	7.21094e5	4.31012e-4			
		4	310.80000	7.21008e5	4.31063e-4			
		5	310.80000	7.22268e5	4.30311e-4			
		6	310.80000	7.24772e5	4.28824e-4			
		11	310.80000	7.07642e5	4.39205e-4			
		12	310.80000	7.00669e5	4.43576e-4			
		13	310.80000	7.05419e5	4.40589e-4			
		14	310.80000	7.09949e5	4.37778e-4			
		15	310.80000	7.18262e5	4.32711e-4			
		16	310.80000	6.99829e5	4.44109e-4			

Peak Sum Table

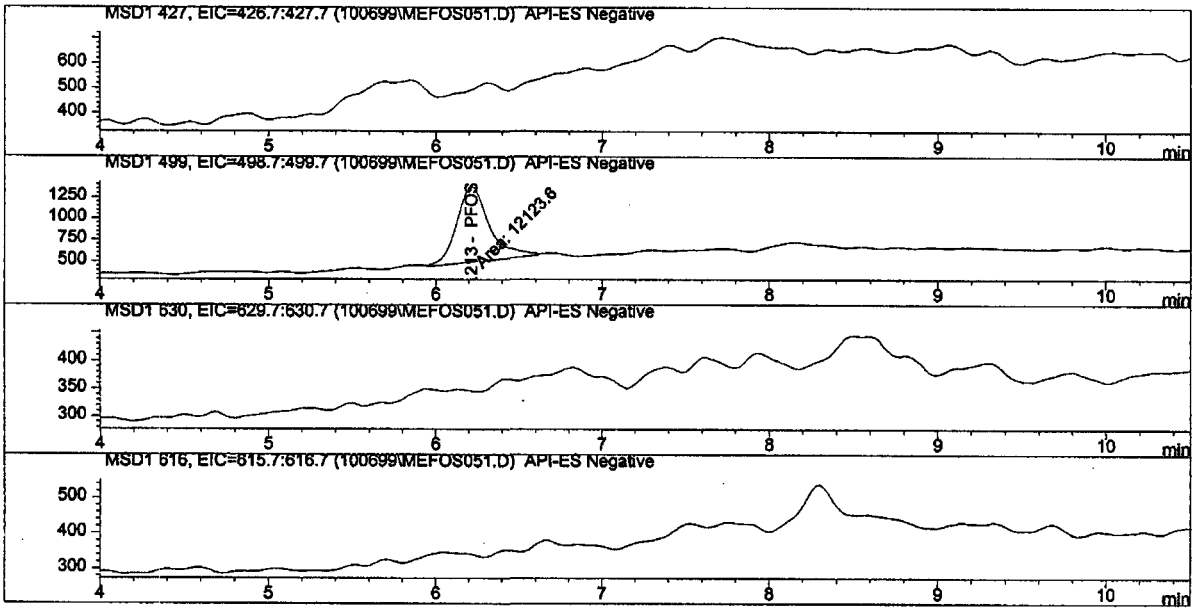
No Entries in table

Batch Run # 1 of 50
Data File C:\HPCHEM\1\DATA\100699\MeFOS051.D Sample Name: MeOH Blank

```
==========================================================================
Injection Date  : 10/7/99 5:09:30 AM            Seq. Line :  51
Sample Name     : MeOH Blank                        Vial :  91
Acq. Operator   : MTM                               Inj :   1
Acq. Method     : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed    : 10/6/99 1:56:00 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\1227_70I.M
Last changed    : 12/29/99 9:23:15 AM by MTM
                  (modified after loading)  (Results are from a previously s
SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using
4mmx35mm Dionex IonPac NG1 column, S/N 12879.
```

pH 7.0 Solvent Blank JJT 9/8/00

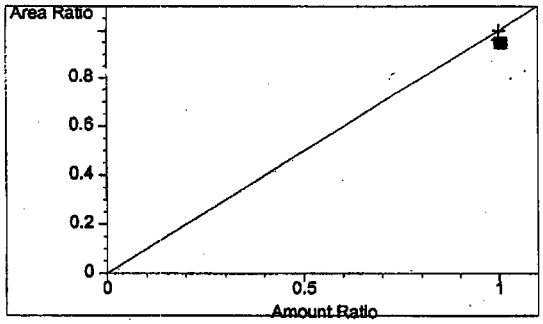
MTM



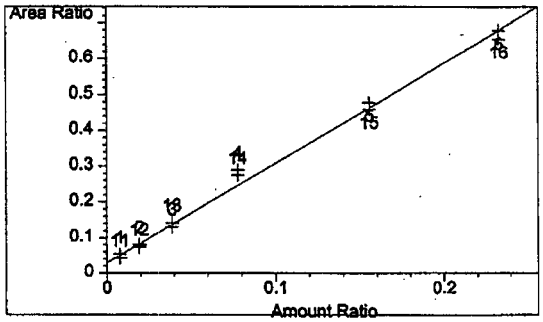

```
==========================================================================
                        Internal Standard Report
==========================================================================

Sorted By             :      Signal
Calib. Data Modified  :      12/29/99 9:23:15 AM
Multiplier            :      1.0000
Dilution              :      1.0000
Sample ISTD Information:
ISTD  ISTD Amount    Name
  #      [ppb]
----|-------------|-------------------------
  1    402.60000   THPFOS
  2    310.80000   EtFOSE-OH
```

Method C:\HPCHEM\1\METHODS\1227_70I.M

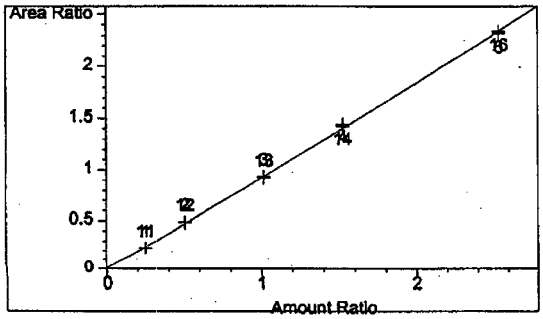
Calibration Curves



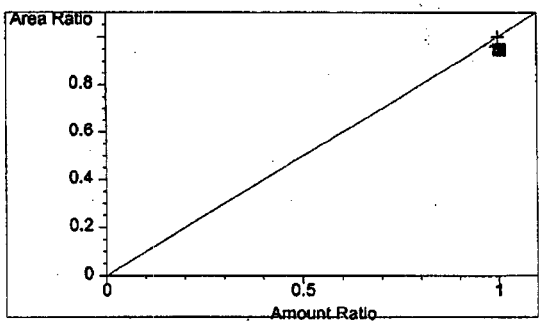
THPFOS at exp. RT: 5.870
MSD1 427, EIC=426.7:427.7
Correlation: 1.00000
Residual Std. Dev.: 0.00000
Formula: y = mx + b
m: 1.00000
b: 0.00000
x: Amount Ratio
y: Area Ratio



PFOS at exp. RT: 6.162
MSD1 499, EIC=498.7:499.7
Correlation: 0.99654
Residual Std. Dev.: 0.02056
Formula: y = mx + b
m: 2.79093
b: 2.92806e-2
x: Amount Ratio
y: Area Ratio



MeFOSE-OH at exp. RT: 8.306
MSD1 616, EIC=615.7:616.7
Correlation: 0.99973
Residual Std. Dev.: 0.01989
Formula: y = mx + b
m: 9.19192e-1
b: 6.85505e-3
x: Amount Ratio
y: Area Ratio



EtFOSE-OH at exp. RT: 8.535
MSD1 630, EIC=629.7:630.7
Correlation: 1.00000
Residual Std. Dev.: 0.00000
Formula: y = mx + b
m: 1.00000
b: 0.00000
x: Amount Ratio
y: Area Ratio

Batch Run # 1 of 50
Data File C:\HPCHEM\1\DATA\100699\MeFOS051.D Sample Name: MeOH Blank

Signal 1: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
5.870		-	-	-		THPFOS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
6.213	MM	1.21236e4	0.00000	0.00000		PFOS

Totals without ISTD(s) : 0.00000

Signal 3: MSD1 630, EIC=629.7:630.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
8.535		-	-	-		EtFOSE-OH

Totals without ISTD(s) : 0.00000

Signal 4: MSD1 616, EIC=615.7:616.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
8.306		-	-	-		MeFOSE-OH

Totals without ISTD(s) : 0.00000

2 Warnings or Errors :

Warning : ISTD compound(s) not found
Warning : Negative results set to zero (cal. curve intercept), (PFOS)

==========

*** End of Report ***

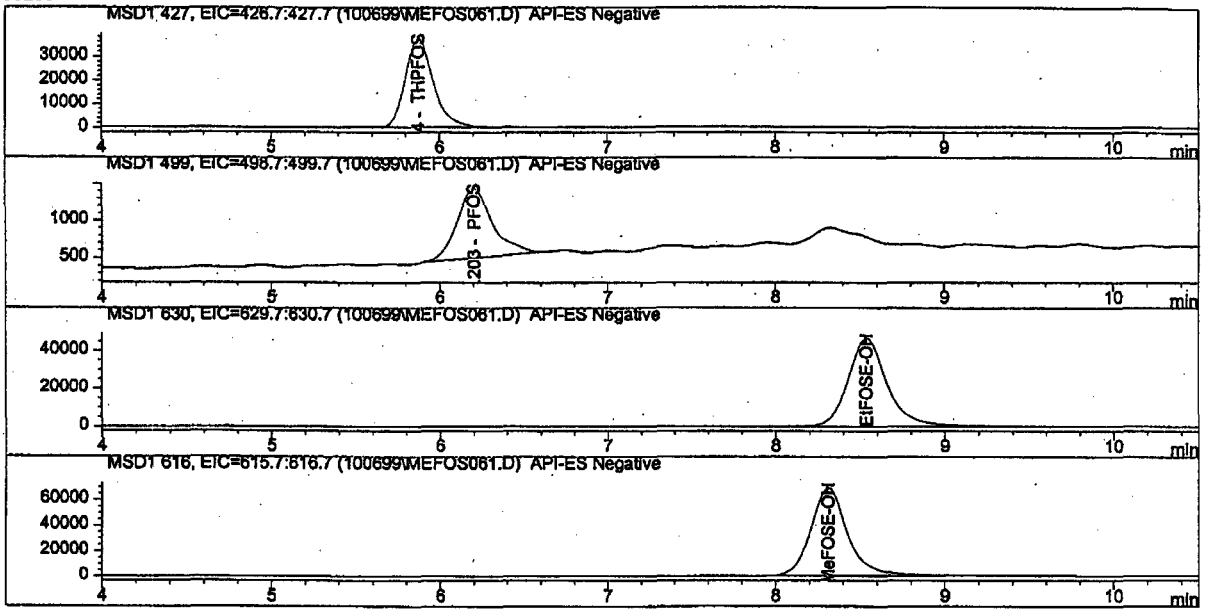
Batch Run # 11 of 50
Data File C:\HPCHEM\1\DATA\100699\MeFOS061.D Sample Name: MFA-010

Injection Date	: 10/7/99 8:11:41 AM	Seq. Line	: 61
Sample Name	: MFA-010	Vial	: 51
Acq. Operator	: MTM	Inj	: 1
Acq. Method	: C:\HPCHEM\1\METHODS\FOSESIM.M		
Last changed	: 10/6/99 1:56:00 PM by MTM		
Analysis Method	: C:\HPCHEM\1\METHODS\1227_70I.M		
Last changed	: 12/29/99 9:23:15 AM by MTM		

pH 7.0 SAMPLE JST 9/8/00

(modified after loading) (Results are from a previously s
SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using
4mmx35mm Dionex IonPac NG1 column, S/N 12879.

MTM



Internal Standard Report

Sorted By : Signal
Calib. Data Modified : 12/29/99 9:23:15 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount [ppb]	Name
1	402.60000	THPFOS
2	310.80000	EtFOSE-OH

Batch Run # 11 of 50
Data File C:\HPCHEM\1\DATA\100699\MeFOS061.D Sample Name: MFA-010

Signal 1: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type		Area	Amt/Area ratio	Amount [ppb]	Grp	Name
5.874	BB	I	4.31433e5	1.00000	402.60000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type		Area	Amt/Area ratio	Amount [ppb]	Grp	Name
6.203	PBA		1.37520e4	2.91660e-2	3.74287e-1		PFOS

Totals without ISTD(s) : 3.74287e-1

Signal 3: MSD1 630, EIC=629.7:630.7

RetTime [min]	Type		Area	Amt/Area ratio	Amount [ppb]	Grp	Name
8.538	VB	I	7.69704e5	1.00000	310.80000		EtFOSE-OH

Totals without ISTD(s) : 0.00000

Signal 4: MSD1 616, EIC=615.7:616.7

RetTime [min]	Type		Area	Amt/Area ratio	Amount [ppb]	Grp	Name
8.310	BV		1.05985e6	1.08250	463.26250		MeFOSE-OH

Totals without ISTD(s) : 463.26250

*** End of Report ***

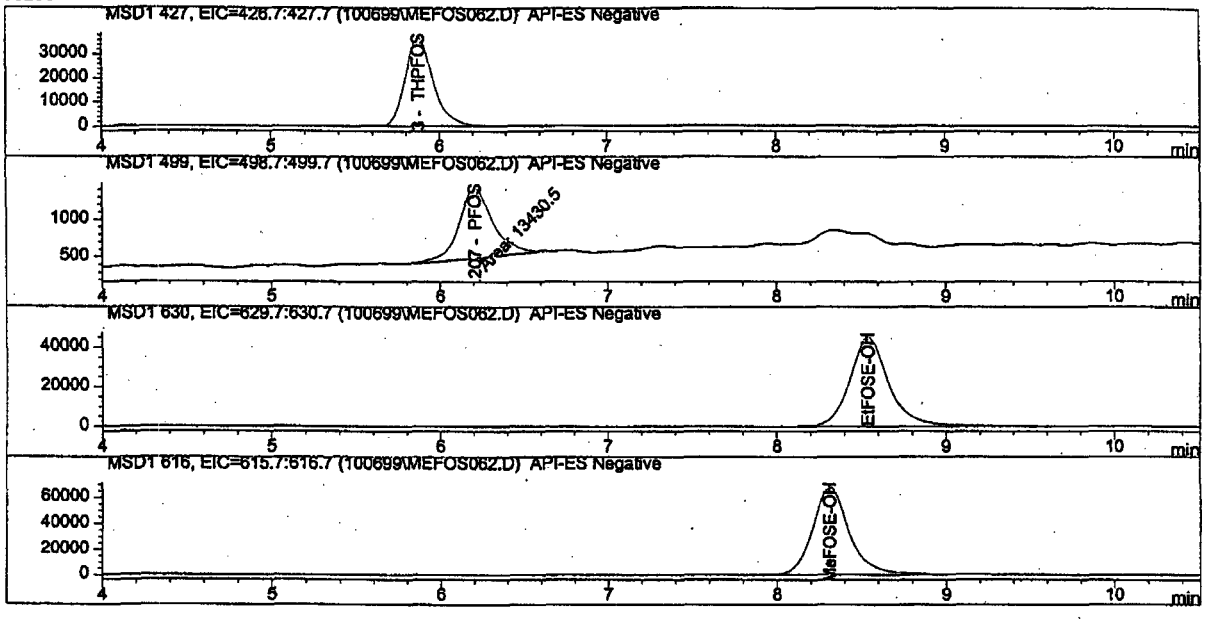
Batch Run # 12 of 50
Data File C:\HPCHEM\1\DATA\100699\MeFOS062.D Sample Name: MFA-011

Injection Date	: 10/7/99 8:29:54 AM	Seq. Line	: 62
Sample Name	: MFA-011	Vial	: 52
Acq. Operator	: MTM	Inj	: 1
Acq. Method	: C:\HPCHEM\1\METHODS\FOSESIM.M		
Last changed	: 10/6/99 1:56:00 PM by MTM		
Analysis Method	: C:\HPCHEM\1\METHODS\1227_70I.M		
Last changed	: 12/29/99 9:23:15 AM by MTM		

pH 7.0 DUPLICATE JJT 9/8/00

(modified after loading) (Results are from a previously s
SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using
4mmx35mm Dionex IonPac NG1 column, S/N 12879.

MTM



Internal Standard Report

Sorted By	: Signal
Calib. Data Modified	: 12/29/99 9:23:15 AM
Multiplier	: 1.0000
Dilution	: 1.0000

Sample ISTD Information:

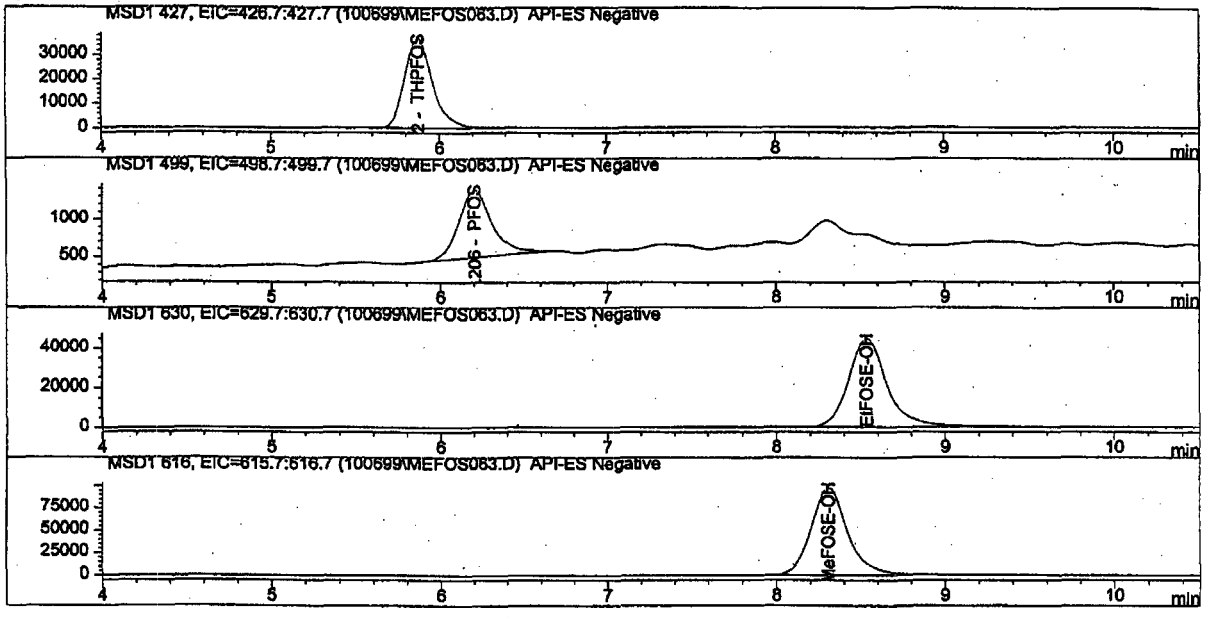
ISTD #	ISTD Amount [ppb]	Name
1	402.60000	THPFOS
2	310.80000	EtFOSE-OH

Batch Run # 13 of 50
Data File C:\HPCHEM\1\DATA\100699\MeFOS063.D Sample Name: MFA-012

Injection Date : 10/7/99 8:48:10 AM Seq. Line : 63
Sample Name : MFA-012 Vial : 53
Acq. Operator : MTM Inj : 1
Acq. Method : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed : 10/6/99 1:56:00 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\1227_70I.M
Last changed : 12/29/99 9:23:15 AM by MTM
(modified after loading) (Results are from a previously s
SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using
4mmx35mm Dionex IonPac NG1 column, S/N 12879.

pH 7.0
MATRIX SPIKE

MTM



Internal Standard Report

Sorted By : Signal
Calib. Data Modified : 12/29/99 9:23:15 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount [ppb]	Name
1	402.60000	THPFOS
2	310.80000	EtFOSE-OH

Batch Run # 12 of 50
Data File C:\HPCHEM\1\DATA\100699\MeFOS062.D Sample Name: MFA-011

Signal 1: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
5.873	BB I	4.27482e5	1.00000	402.60000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
6.207	MM	1.34305e4	2.43726e-2	3.08284e-1		PFOS

Totals without ISTD(s) : 3.08284e-1

Signal 3: MSD1 630, EIC=629.7:630.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
8.538	BB I	7.38896e5	1.00000	310.80000		EtFOSE-OH

Totals without ISTD(s) : 0.00000

Signal 4: MSD1 616, EIC=615.7:616.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
8.310	BV	1.03510e6	1.08259	471.34921		MeFOSE-OH

Totals without ISTD(s) : 471.34921

*** End of Report ***

Batch Run # 13 of 50
Data File C:\HPCHEM\1\DATA\100699\MeFOS063.D Sample Name: MFA-012

Signal 1: MSD1 427, EIC=426.7:427.7

RetTime [min]	Type		Area	Amt/Area ratio	Amount [ppb]	Grp	Name
5.872	PB	I	4.24804e5	1.00000	402.60000		THPFOS

Totals without ISTD(s) : 0.00000

Signal 2: MSD1 499, EIC=498.7:499.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
6.206	PBA	1.24702e4	9.10909e-4	1.07655e-2		PFOS

Totals without ISTD(s) : 1.07655e-2

Signal 3: MSD1 630, EIC=629.7:630.7

RetTime [min]	Type		Area	Amt/Area ratio	Amount [ppb]	Grp	Name
8.535	PB	I	7.26047e5	1.00000	310.80000		EtFOSE-OH

Totals without ISTD(s) : 0.00000

Signal 4: MSD1 616, EIC=615.7:616.7

RetTime [min]	Type	Area	Amt/Area ratio	Amount [ppb]	Grp	Name
8.307	BV	1.49310e6	1.08429	693.02691		MeFOSE-OH

Totals without ISTD(s) : 693.02691

*** End of Report ***