Study Title

Hydrolysis Reactions of 2-(*N*-ethylperfluoroctanesulfonamido)-Ethyl Alcohol (*N*-EtFOSE Alcohol)

Data Requirement:

Based on OPPTS: 835.2110

Author

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Study Completion Date

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Performing Laboratory

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Project Identification

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Statement of Non-Compliance

Study Title: Hydrolysis Reactions of 2-(*N*-ethylperfluoroctanesulfonamido)-Ethyl Alcohol (*N*-EtFOSE 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 03/23/01

Sponsor Representative — Date 03/23/01

Quality Assurance Statement

Study Title: Hydrolysis Reactions of 2-(*N*-ethylperfluoroctanesulfonamido)-Ethyl Alcohol (*N*-EtFOSE 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
9/21, 22, 25/2000	Data and Draft Report	10/03/00	10/03/00
10/2/2000	Data and Draft Report	10/03/00	10/03/00
3/14, 15/2001	Draft Report	3/15/01	3/15/01

Casey Howell — *QAU Representative*

Mar 26, 2001 — Date

Table of Contents

List of Tables

List of Figures

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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*-ethylperfluorooctane-sulfonamido)-ethyl alcohol (hereafter, *N*-EtFOSE alcohol). Our methods are described below and in Appendix A to this work; our results are based on the observed concentrations of *N*-EtFOSE 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*-EtFOSE alcohol on the sample pH level. Our results based on the *N*-EtFOSE alcohol concentrations, pooled over the observed pH levels, are presented in Table 1.

Table 1. Summary of Results Based on *N*-EtFOSE 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)
0.00262	0.000262	7.3	5.3 to 11.5

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*-EtFOSE alcohol, this LOQ (and other experimental data) provide a second estimate of the *N*-EtFOSE 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)
6.1×10^{-4}	6.1×10^{-5}	≥ 31

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*-EtFOSE alcohol under the assumption that it hydrolyzes to form *only* the compound PFOS.

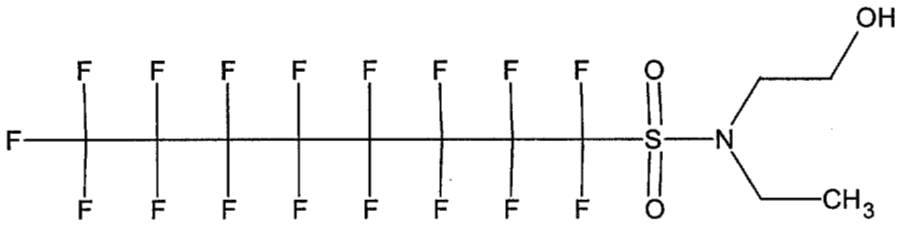
Introduction

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.

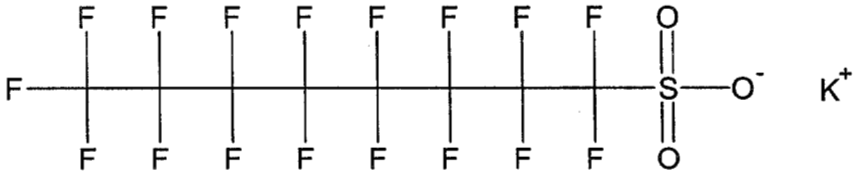
The hydrolysis of *N*-EtFOSE alcohol (or, more generally, its degradation in the presence of H_2O) is addressed in this report. Structures of the "parent" compound *N*-EtFOSE alcohol and the potassium salt of possible hydrolysis product perfluorooctane sulfonate (PFOS) are illustrated in Figure 1.

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

***N*-EtFOSE 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*-EtFOSE alcohol.

The first estimate (see Table 1) is based on the observed degradation of the "parent" compound *N*-EtFOSE 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. The data corresponding to "Day 0" (t = 0) were used to determine the relative concentration ratios (see Equations B8 and B9).

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*-EtFOSE alcohol, these PFOS analyses provide the following estimate $\left(T^{1/2}_{p}\right)_2$ of the minimum *N*-EtFOSE 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*-EtFOSE 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-8-179.0, "Preparation of 2-(*N*-ethylperfluoroctanesulfonamido) Ethyl Alcohol (*N*-EtFOSE 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 Equations 3 and 6 involve only ratios of the parent and product 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	PFOS (Potassium Salt)	THPFOS	*N*-MeFOSE Alcohol
Source	3M Specialty Chemistry	3M ICP/PCP Division	ICN Biomedicals	3M Specialty Chemistry
Chemical Lot Number[a]	S398-332	Batch # 171	Batch # 53406	S398-331
Physical Description	Off-white powder	Light colored powder	Brown waxy solid	White powder
Molecular Weight (gm mole^{-1})	570.9	538	428	556.9

[a] The "S" and "TNA" designations are based on reference numbers in two redundant databases maintained by 3M.

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 455 ng/mL of the analyte *N*-EtFOSE alcohol and 403 ng/mL of 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.

All the samples were prepared simultaneously, and all but the "Day 0" samples were placed in an orbital incubator/shaker maintained at 50° (± 3°) C. After at least three minutes of agitation, the "Day 0" samples were spiked (as required) with the PFOS solution, diluted 10:1 with methanol containing the internal standard THPFOS, and refrigerated. After the appropriate incubation times, subsets of the sample vials were removed from the incubator and then spiked, diluted, and stored as described

immediately above. Except during the relatively short periods of time required to prepare them, the samples were shielded from light.

Eight calibration standards containing N-MeFOSE alcohol (316 ng/ml), *N*-EtFOSE alcohol (155 to 932 ng/ml), THPFOS (403 ng/ml) and PFOS (1.3 to 64 ng/ml) served as the quantitative basis of the study. All these standards were prepared at the appropriate pH levels using the buffer solutions described above.

Sample Analysis

The equipment we used for the HPLC/ITMS 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*-EtFOSE alcohol were detected at the charge-to mass ratios 499, 427, 616, and 630 respectively. We processed the resulting data using the computer program *HP ChemStation for LC* (Rev.A.06.0). Further analytical details, including the gradient elution program, instrument and detector parameters, and performance specifications, are presented in Appendix A.

Deviations

No deviations from the procedures defined in the analytical method (Appendix A) were noted during the study. As noted in the following sections, some calibration and sample data failed to meet data quality objectives and were rejected.

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 linear fit 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 288 calibration results obtained, 22 individual values failed to meet the stated DQO and were rejected. No more than two values for any compound were rejected for any particular calibration run.
- **Spike Recoveries.** Results for three sample pairs (EFA -136 and -137, at pH = 7.0; EFA-31 and -32 at pH = 9.0; and EFA -139 and -140, at pH = 9.0) failed to meet the related DQO and were rejected.

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, the regression fits were not forced 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, such as these, 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*-EtFOSE alcohol and PFOS were 155 ng/mL and 12.8 ng/mL, respectively

Results for the surrogate compound (THPFOS) and the internal standard (*N*-MeFOSE alcohol) were very consistent throughout the study. The percent relative standard deviations of the measured values, calculated for each pH level, ranged from 1.8% to 7.4%.

Table 4 presents the results of the rate determinations at six pH levels and 50°C.

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

PH	Observed Rate (day^{-1})	Percent (2σ) Rate Uncertainty (day^{-1})
1.5	0.0028	66
3.0	0.0024	115
5.0	0.0027	56
7.0	0.0030	36
9.0	0.0022	95
11	0.0023	54

These degradation rates are generally only poorly determined; their percent relative 2σ (95% confidence) uncertainties range from 36% to 115%. The data do not indicate any 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*-EtFOSE 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)
0.00262	37%	0.000262	7.3	5.3 to 11.5

Figure 2. Observed *N*-EtFOSE Alcohol Degradation for Various pH levels.

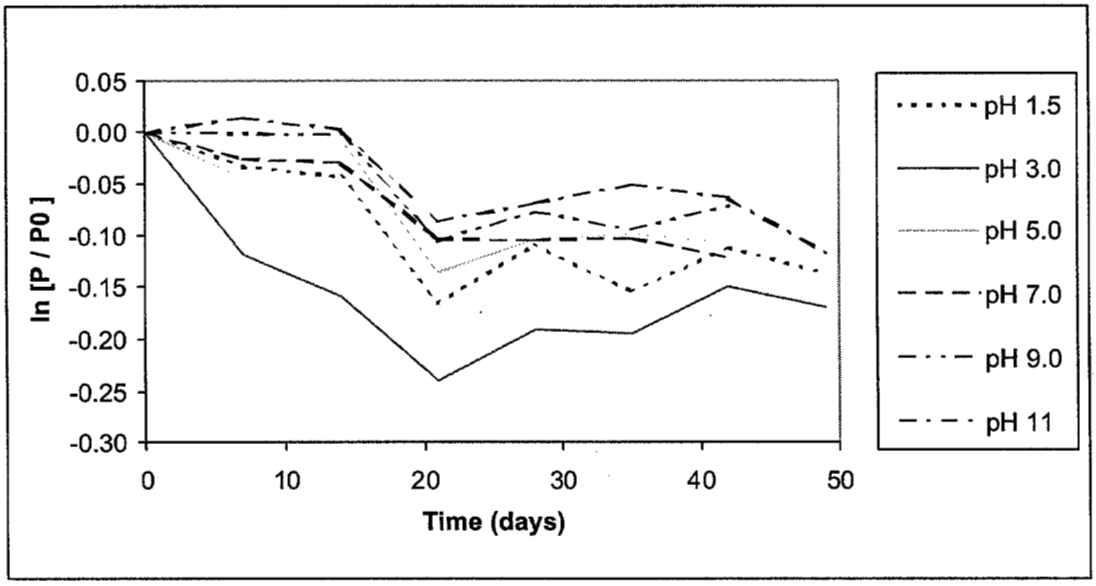
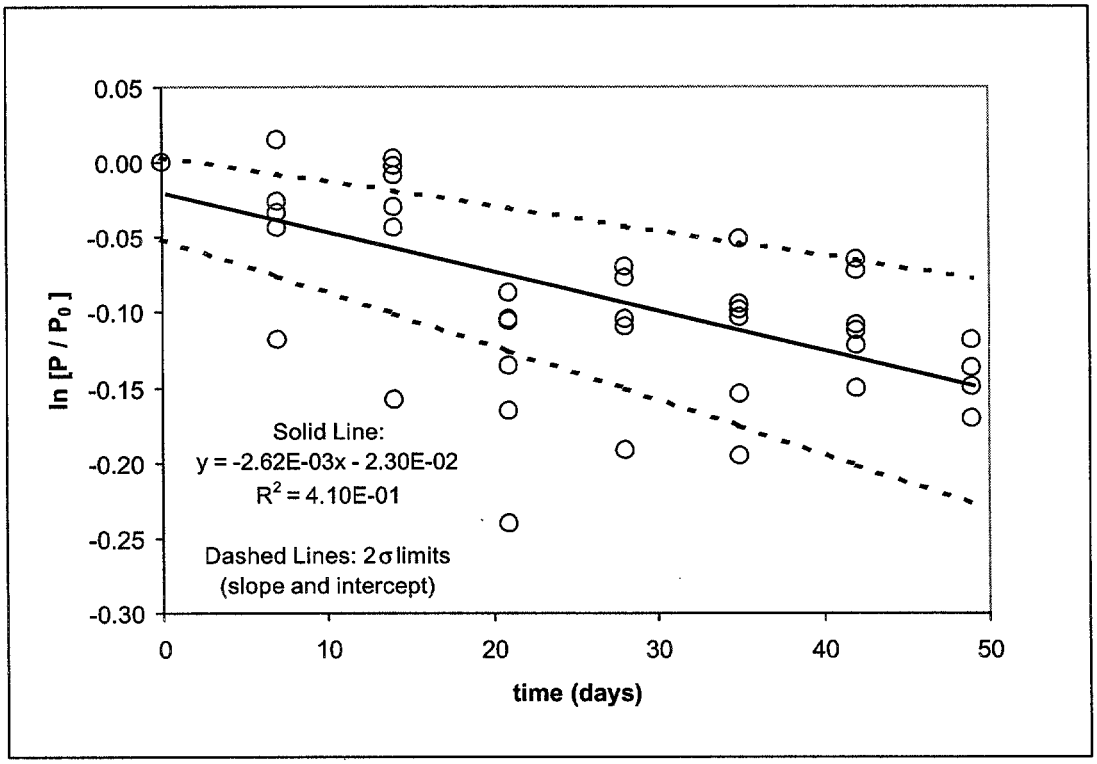


Figure 3. Pooled *N*-EtFOSE Alcohol Data and Slope Regression.



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 12.8 ng/mL). The initial *N*-EtFOSE alcohol concentration (455 ng/ml) and the PFOS LOQ provide a second estimate of the *N*-EtFOSE alcohol half-life (see in Appendix B, Equations B32 and B33). The maximum degradation rate is given by Equation 3:

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

and the minimum half-life is given by Equation 4

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

We note that in both Equations 3 and 4, the initial *N*-EtFOSE 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*-EtFOSE Alcohol in Aqueous Buffered 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	0.80	0.024	6.1×10^{-4}	6.1×10^{-5}	≥ 31

Conclusions

We have performed a study of the aqueous hydrolytic degradation 2-(*N*—ethylperfluoro-octanesulfonamido)-ethyl alcohol (*N*-EtFOSE 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*-EtFOSE alcohol concentration indicate no clear dependence of the degradation rate of *N*-EtFOSE alcohol on pH. From the data pooled over the six pH levels, we estimate that the hydrolytic half-life of *N*-EtFOSE alcohol at 25°C lies between 5.3 and 11.5 years, with the most likely value of 7.3 years.

The concentration of the compound PFOS, a likely hydrolytic product of *N*-EtFOSE alcohol, was monitored throughout the study, but remained undetected above its limit of quantification (LOQ = 12.8 ng/mL). Using the LOQ for PFOS and the initial *N*-EtFOSE alcohol concentration (455 ng/ml), and assuming PFOS is the only hydrolytic product of *N*-EtFOSE alcohol, the data indicate that the hydrolytic half-life of *N*-EtFOSE alcohol at 25°C is greater than or equal to 25 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 03/23/01

William K. Reagen, Ph.D., Laboratory Management — Date 03/23/01

Appendix A: Analytical Method

ETS - 8-179.0, "Preparation of 2-(N-ethylperfluorooctanesulfonamido) 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*-ETHYL PERFLUOROOCTANESULFONAMIDO)-ETHYL ALCOHOL (*N*-ETFOSE ALCOHOL) HYDROLYSIS SAMPLES AND ANALYSIS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH MASS SPECTROMETRY DETECTION

Method Number: ETS-8-179.1

Adoption Date: 9/14/00

Effective Revision Date: 3/19/01

Approved by:

Laboratory Manager — Date: 03/23/01

Team Leader — Date: March 23 01

1.0 SCOPE AND APPLICATION

1.1 This procedure defines the steps for analysis of 2-(*N*-ethyl perfluorooctanesulfonamido)-ethyl alcohol (*N*-EtFOSE 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*-EtFOSE alcohol and the potential hydrolysis product perfluorooctane sulfonate (PFOS anion) are detected and quantified by this method. *N*-EtFOSE alcohol is quantified using *N*-MeFOSE alcohol (2-(*N*-methylperfluorooctanesulfonamido)-ethyl alcohol) as an internal standard. The surrogate THPFOS (the anion of 3, 3, 4, 4, 5, 5, 6, 6, 7, 7, 8, 8, 8-tridecafluorooctane sulfonic acid) is used to quantify PFOS. Representative structures are shown in Attachment A.

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

1.3 **Compatible matrices for analysis.** Aqueous solutions at various buffered pH levels.

1.4 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 frequency of quality control parameters to be performed in 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*-EtFOSE alcohol stock solution containing THPFOS surrogate 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*-EtFOSE alcohol is diluted tenfold with methanol (MeOH) and spiked with *N*-MeFOSE alcohol internal standard. The parent compound, *N*-EtFOSE 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 **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.

3.2 **Calibration Curve.** The graphical relationship between known values, such as concentration of a series of calibration standards and their instrumental response.

3.3 **Internal Standard Calibration.** Process of establishing a relationship between the ratio of the target analyte(s) response to internal standard or surrogate response and a known concentration of the target analyte(s). The ratio of analyte to internal standard response is used to generate the calibration curve and determine unknown concentrations.

3.4 **Correlation Coefficient (r).** A measure of the degree of correlation between two variables. This term is generally used to evaluate the linearity of a Least Squares Linear regression. An r value of 0.98 is at the lower bounds of what is considered linear. Values of r may range from –1 to +1. A value of +1 denotes perfect direct functional relationship between two variables. A value of –1 also denotes a perfect inverse relationship. When r = 0, there is no effect of one variable upon the other variable.

3.5 **Coefficient of Determination (r^2).** The square of the correlation coefficient. It is the proportion of the variation in the dependent variable that is accounted for by the independent variable.

3.6 **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.

3.7 **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).

3.8 **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.

3.9 **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.

3.10 **Limit of Quantitation (LOQ).** The lowest concentration that can be reliably measured within specified limits of accuracy 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.

3.11 **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.

3.12 **Relative Percent Difference (RPD).** A measure of precision defined as the absolute value of the difference of two values divided by the average of the two values and multiplied by 100.

3.13 **Matrix Spike (MS).** Prepared by adding a known mass of target analyte to a specified amount of a sample matrix prior to analysis. 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.

3.14 **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.

3.15 **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.

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 method 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–1000 µL
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 0.15 g ammonium acetate crystals to a 1-L volumetric flask containing about 500 mL water, adding 10 mL of methanol, diluting to the mark with 18.0 MΩ water and mixing.)
8.5 **Stock, internal standard, surrogate and calibration solutions**
All weights should be recorded to the nearest 0.0001 g in a standards preparation log:

8.5.1 *N*-EtFOSE 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*-EtFOSE 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*-MeFOSE-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 methanol. (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 methanol. 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) surrogate 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 methanol. This solution is diluted in methanol 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 Add 3.8777 g ammonium acetate (reagent grade) to 250 mL 18.0 MΩ water
8.7.3.2 Add 250 mL 0.052 M acetic acid (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 glacial acetic acid (approximately 0.5 mL)
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*-EtFOSE alcohol test analyte solution with THPFOS surrogate.** [Example: An analyte solution of *N*-EtFOSE alcohol at 500 µg/mL and THPFOS at 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 (the step performed in Section 12.1.6) results in a final concentration of 500 ng/mL *N*-EtFOSE alcohol and 400 ng/mL THPFOS after MeOH dilution (the step performed in Section 12.1.13)].

8.8.2 ***N*-MeFOSE alcohol internal standard solution.** [Example: An analyte solution of *N*-MeFOSE alcohol at 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 1 mL buffer (the step performed in Section 12.1.14) results in a final *N*-MeFOSE alcohol concentration of approximately 300 ng/mL.

8.8.3 Spiking solution. [Example: A spiking solution is prepared by adding 70 µL of the *N*-EtFOSE alcohol stock solution (Section 8.5.1) to a 10-mL volumetric flask and diluting to the mark with methanol. A 10-µL aliquot of this solution added to the 1.0 mL sample (the step performed in Section 12.1.15) results in a final spike concentration of 217 ng/mL of *N*-EtFOSE alcohol after MeOH dilution.

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 are ready to be analyzed. Alternatively, aliquots of the methanol-diluted samples should be refrigerated at 4 ± 3 °C or frozen until analysis can be performed.

10.0 QUALITY CONTROL

10.1 **Calibration Standards.** Calibration standards (Section 11) used to generate a calibration curve should be prepared in the same type of solvent or matrix as in the study samples. The number of calibration standards and the concentration levels should be sufficient to encompass the expected concentrations of the study samples. In general, a minimum of five calibration standards is required for fit of linear regression. Broad calibration ranges (greater that three orders of magnitude between low and high standards), may require use of a quadratic fit of the data and requires more points to adequately represent the calibration range.

10.2 **Internal standard (IS).** *N*-MeFOSE alcohol internal standard is added in a constant concentration to all standards, samples, and matrix spikes.

10.3 **Surrogate.** THPFOS surrogate is added in a constant concentration to CCVs, samples, sample duplicates, and matrix spike samples along with the *N*-EtFOSE alcohol test analyte (pre-hydrolysis).

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 **Solvent blank.** Solvent blanks (or clean method blanks) should be run before and after every calibration curve, CCV, method blank (if contamination is noted), and after batches of no more than 20 injections. Acceptable values for the blanks are values below the limit of quantitation (LOQ) of the instrument (Section 3.10). If analyte carryover is a problem (see Section 14.5), use back-to-back solvent blanks.

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

10.7 **Matrix spikes.** Prepare a post-hydrolysis matrix spike sample (the step performed in Section 12.1.15) 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.

11.0 CALIBRATION AND STANDARDIZATION

11.1 **Standard preparation.** Prepare six calibration standards containing *N*-EtFOSE alcohol, *N*-MeFOSE alcohol, PFOS and THPFOS in 9:1 MeOH:buffer for each pH level. Standards from approximately 150 ng/mL to 1000 ng/mL of *N*-EtFOSE alcohol and 1 ng/mL to 100 ng/mL of PFOS are suggested.

11.2 **Calibration standards.** Analyze the calibration standards at the beginning and end of the run. Individual calibration data points from both sets of calibration standards are used to generate a calibration curve. The calibration curve is then used by 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*-MeFOSE alcohol as the internal standard for *N*-EtFOSE alcohol 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 mixed *N*-EtFOSE alcohol analyte and THPFOS surrogate 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 each 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 are cooling, spike the stored "Time 0" samples with test analyte solution (Section 12.1.5) on Day 7. 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*-MeFOSE alcohol internal standard solution (Section 8.8.2) to each sample and spike vial.

12.1.15 Using a 25-µL gas tight syringe, add 10 µL of 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*-EtFOSE 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: 35°C.

12.4 Recommended mass spectrometer set up*:

MSD:	Ionization mode	API-ES
	Polarity	Negative
	Acquisition mode	SIM
	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
8.3	MeFOSE-OH	Internal Standard	616 ($M+OAc^-$)	617
8.6	EtFOSE-OH	Test Analyte	630 ($M+OAc^-$)	631
6.2	PFOS	Potential Degradation Product	499 ($M-H^+$)	500
5.9	THPFOS	Surrogate	427 ($M-H^+$)	No monitor
6.3	FOSA	Qualitative Purposes Only	498 ($M-H^+$)	No monitor

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. EtFOS). 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 4 (four) times the baseline noise for that region of the chromatogram. Peak area integration is from baseline to baseline using automatic or manual integration. *N*-EtFOSE alcohol concentrations are calculated using *N*-MeFOSE-OH as the internal standards. THPFOS surrogate is used as the "internal standard" to calculate PFOS concentrations. External standard calibration may be acceptable. Consult with the team leader for direction prior to performing the external calibration methodology. Document change in raw data and final report.

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:

$Ln\left(\frac{[\text{EtFOSE}]_t}{[\text{EtFOSE}]_0}\right)$ 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*.

13.3 Target analyte concentrations. Calculate the EtFOSE-OH 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 duplicate

14.0 METHOD PERFORMANCE

14.1 Coefficient of Determination (r^2). The coefficient of determination (r^2) for the calibration curves should be 0.990 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. Consult with the Team Leader or designee for direction and for final acceptance or rejection for the data.

14.2 Calibration Standards. The acceptance criterion for the calibration standards is that the accuracy of each standard is 75% to 125% of the nominal value. Calibration standards outside this range are to be considered outliers and excluded from the linear regression. 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 Internal Standard (IS) and Surrogate. Review of the internal standard and surrogate performance is performed by averaging the area response throughout the analytical run and calculating %RSD. 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.

14.4 Continuing Calibration Verification. If the percent difference for the amount of quantitated analyte is greater than 25% from the true value relative to the initial standard curve, the Team Leader should be consulted. 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 for the data.

14.5 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, the run should be stopped. This

is indicative of instrument contamination. The instrument shall be serviced by thoroughly cleaning the electrospray source, and replacing/cleaning columns, tubing, etc.

14.6 **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.

14.7 **Sample Duplicates.** The analyst shall accept %RPD (See Section 13.5) values < 25%. %RPD values > 25% 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 analytical run.

14.8 **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.

14.9 **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 graphics 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 Hewlett Packard 1100/MSD instruction CD/ROM

19.0 AFFECTED DOCUMENTS

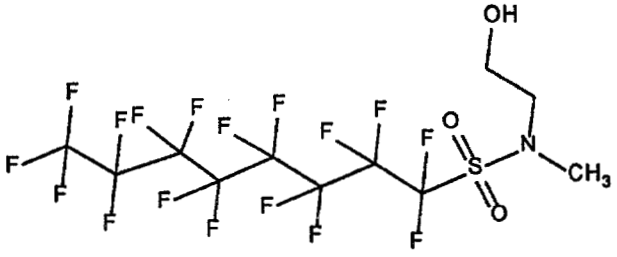
19.1 None.

20.0 REVISIONS

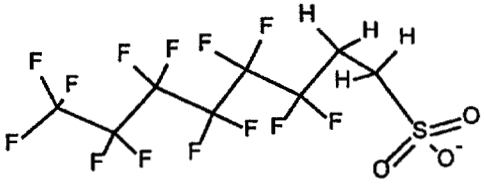
Revision number	Reason for revision	Date of Revision
001	Resizing of graphics and re-formatting to remove page orphans	03/19/01

Attachment A. Representative Chemical Structures

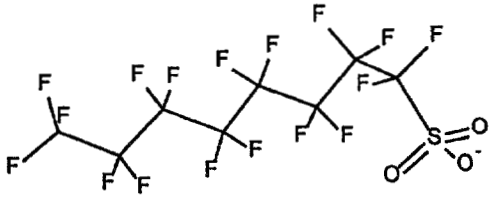
N-MeFOSE alcohol (FW = 557)



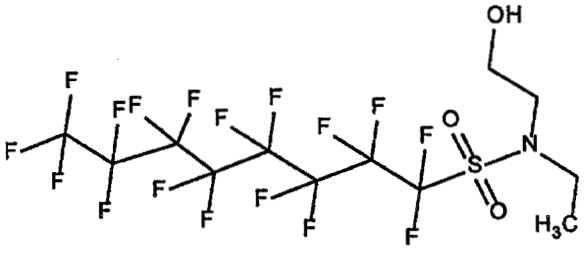
THPFOS (FW = 427 (anion))



PFOS (FW =499 (anion))



N-EtFOSE alcohol (FW = 526)



Counter cation = K^+ in this study, but may also be Li^+, Ca^{+2}, or DEA (diethanolamine)

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 I Amount (mL)	Solution ID Amount (µL)	Solution ID Amount (µL)	Solution 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:

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

(for $k_{Am} = k_P$)

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

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

(for $k_{Am} = k_P$)

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

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

$$A_m = A_{m0} \tag{B21}$$

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

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

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

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

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)

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

B4. Relationships Between the Parent and Compound Concentrations

Equations B7 and B24 can be combined to obtain

(for hydrolytically stable products)

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

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

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

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

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 (Δt) 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 $t = \Delta t$. 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 (Δt) 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)

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

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)

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

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).

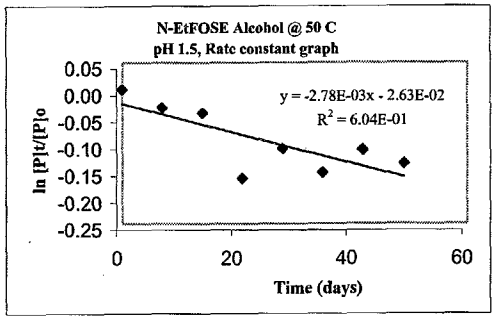
Appendix C: Selected Analytical and Kinetics Results

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

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

All concentrations in ng/ml.

pH	Time (Days)	Conc.	ln([P]t/[P]o)
1.5	0	490	0.000
1.5	7	474	-0.033
1.5	14	469	-0.043
1.5	21	415	-0.165
1.5	28	439	-0.110
1.5	35	420	-0.154
1.5	42	438	-0.112
1.5	49	427	-0.137



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.77717
R Square	0.60399
Adjusted R Square	0.53799
Standard Error	0.04167
Observations	8

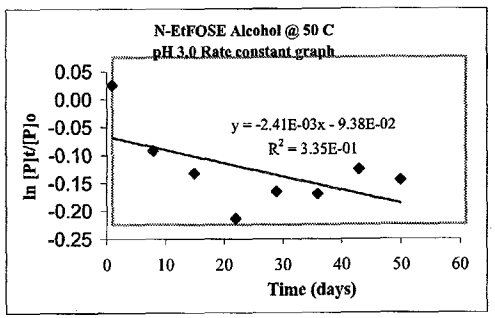
ANOVA

	df	*SS*
Regression	1	0.01589
Residual	6	0.01042
Total	7	0.02631

	Coefficients	*Standard Error*
Intercept	-0.02631	0.02690
X Variable 1	-0.00278	0.00092

% 2σ Slope Uncertainty
66%

pH	Time (Days)	Conc.	ln([P]t/[P]o)
3.0	0	461	0.000
3.0	7	409	-0.118
3.0	14	393	-0.158
3.0	21	362	-0.240
3.0	28	380	-0.191
3.0	35	379	-0.196
3.0	42	396	-0.151
3.0	49	388	-0.170



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.57897
R Square	0.33521
Adjusted R Square	0.22441
Standard Error	0.06292
Observations	8

ANOVA

	df	*SS*
Regression	1	0.01198
Residual	6	0.02375
Total	7	0.03573

	Coefficients	*Standard Error*
Intercept	-0.09378	0.04061
X Variable 1	-0.00241	0.00139

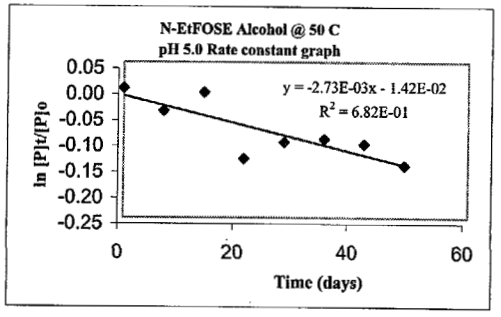
% 2σ Slope Uncertainty
115%

BACK TO MAIN

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

All concentrations in ng/ml.

pH	Time (Days)	Conc.	ln([P]t/[P]o)
5.0	0	477	0.000
5.0	7	456	-0.044
5.0	14	472	-0.009
5.0	21	416	-0.136
5.0	28	430	-0.104
5.0	35	432	-0.099
5.0	42	428	-0.108
5.0	49	410	-0.149



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.82601
R Square	0.68230
Adjusted R Square	0.62935
Standard Error	0.03444
Observations	8

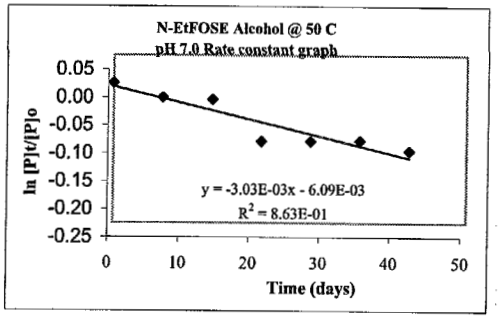
ANOVA

	df	*SS*
Regression	1	0.01528
Residual	6	0.00712
Total	7	0.02240

	Coefficients	*Standard Error*
Intercept	-0.01422	0.02223
X Variable 1	-0.00273	0.00076

% 2σ Slope Uncertainty
56%

pH	Time (Days)	Conc.	ln([P]t/[P]o)
7.0	0	468	0.000
7.0	7	456	-0.026
7.0	14	454	-0.029
7.0	21	421	-0.104
7.0	28	421	-0.105
7.0	35	422	-0.103
7.0	42	414	-0.122
7.0	*49*	*410*	(A)



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.92903
R Square	0.86309
Adjusted R Square	0.83571
Standard Error	0.02002
Observations	7

ANOVA

	df	*SS*
Regression	1	0.01263
Residual	5	0.00200
Total	6	0.01464

	Coefficients	*Standard Error*
Intercept	-0.00609	0.01364
X Variable 1	-0.00303	0.00054

% 2σ Slope Uncertainty
36%

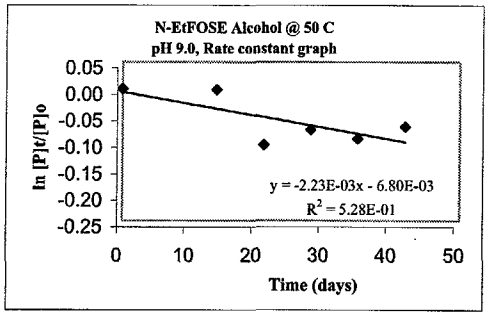
(A) Data in Italics have been rejected from the fits on the basis of the data quality objectives (see text).

BACK TO MAIN

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

All concentrations in ng/ml.

pH	Time (Days)	Conc.	ln([P]t/[P]o)
9.0	*7*	*365*	(A)
9.0	*49*	*392*	(A)
9.0	0	438	0.000
9.0	14	437	-0.002
9.0	21	394	-0.106
9.0	28	406	-0.078
9.0	35	399	-0.095
9.0	42	408	-0.072



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.72644
R Square	0.52771
Adjusted R Square	0.40964
Standard Error	0.03564
Observations	6

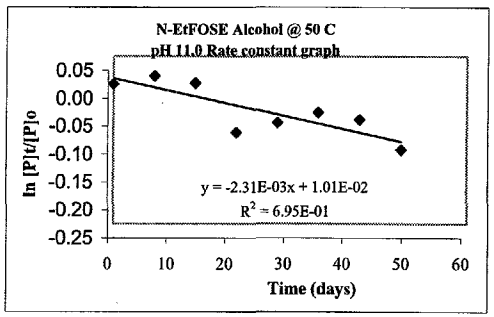
ANOVA

	df	*SS*
Regression	1	0.00568
Residual	4	0.00508
Total	5	0.01076

	Coefficients	*Standard Error*
Intercept	-0.00680	0.02858
X Variable 1	-0.00223	0.00105

% 2σ Slope Uncertainty
95%

pH	Time (Days)	Conc.	ln([P]t/[P]o)
11.0	0	412	0.000
11.0	7	419	0.015
11.0	14	413	0.002
11.0	21	378	-0.087
11.0	28	385	-0.069
11.0	35	392	-0.051
11.0	42	387	-0.064
11.0	49	367	-0.118



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.83358
R Square	0.69485
Adjusted R Square	0.64399
Standard Error	0.02836
Observations	8

ANOVA

	df	*SS*
Regression	1	0.01099
Residual	6	0.00483
Total	7	0.01581

	Coefficients	*Standard Error*
Intercept	0.01012	0.01830
X Variable 1	-0.00231	0.00063

% 2σ Slope Uncertainty
54%

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

BACK TO MAIN

EtFOSE Alcohol Hydrolysis Study
50C; pH 1.5
Reprocessed data on 9-5-00 using internal standard quantitation.

			N-EtFOSE Alcohol						PFOS				THPFOS			N-MeFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc. (ng/ml)	% of Standard or % RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc. (ng/ml)	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	EtFOS002.D	91	8.5	0	0				6.2	0	0		5.9	0	0	8.3	0	0
L1 1.5, 155.40	EtFOS003.D	1	8.5	505275	160	103%			6.2	5973	2	124%	5.9	465165	403	8.3	880788	316
L2 1.5, 310.80	EtFOS004.D	2	8.5	957739	312	100%			6.2	42481	13	104%	5.9	456303	403	8.3	873132	316
L3 1.5, 466.20	EtFOS005.D	3	8.5	1396926	461	99%			6.2	88723	28	111%	5.9	447867	403	8.3	868287	316
L4 1.5, 621.60	EtFOS006.D	4	8.5	1869709	18066	(B)			6.2	112072	36	93%	5.9	451811	403	8.3	(B)	316
L5 1.5, 777.00	EtFOS007.D	5	8.5	2347115	789	102%			6.2	167565	(A)		5.9	(A)	403	8.3	856724	316
L6 1.5, 932.40	EtFOS008.D	6	8.5	2911551	947	102%			6.2	210524	66	103%	5.9	460976	403	8.3	887024	316
MeOH Blank	EtFOS009.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
MeOH Blank	EtFOS010.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
EFA-001	EtFOS011.D	11	8.6	1389788	487	1%		Day 0	6.2	0	0		5.9	455518	403	8.3	817105	316
EFA-002	EtFOS012.D	12	8.6	1373031	492			Day 0	6.2	0	0		5.9	451940	403	8.3	799340	316
EFA-003	EtFOS013.D	13	8.6	1966742	709		101%	Day 0	6.2	0	0		5.9	449589	403	8.3	797895	316
EFA-019	EtFOS014.D	14	8.6	1363242	474	0%		Day 7	6.2	0	0		5.9	466460	403	8.3	823152	316
EFA-020	EtFOS015.D	15	8.5	1358878	473			Day 7	6.2	0	0		5.9	465414	403	8.3	822557	316
EFA-021	EtFOS016.D	16	8.5	1810324	642		77%	Day 7	6.2	0	0		5.9	458389	403	8.3	811296	316
EFA-037	EtFOS017.D	17	8.5	1317957	473	1%		Day 14	6.2	0	0		5.9	443144	403	8.3	798883	316
EFA-038	EtFOS018.D	18	8.5	1327970	466			Day 14	6.2	0	0		5.9	460883	403	8.3	816320	316
EFA-039	EtFOS019.D	19	8.6	2009905	703		108%	Day 14	6.2	0	0		5.9	459552	403	8.3	822562	316
EFA-055	EtFOS020.D	20	8.5	1295532	409	3%		Day 21	6.2	0	0		5.9	458151	403	8.3	904583	316
EFA-056	EtFOS021.D	21	8.6	1393108	421			Day 21	6.2	0	0		5.9	471750	403	8.3	946257	316
EFA-057	EtFOS022.D	22	8.6	2057602	616		93%	Day 21	6.2	0	0		5.9	468384	403	8.3	959312	316
EFA-073	EtFOS023.D	23	8.6	1332265	449	5%		Day 28	6.2	0	0		5.9	464774	403	8.3	848397	316
EFA-074	EtFOS024.D	24	8.6	1282648	429			Day 28	6.2	0	0		5.9	456350	403	8.3	855992	316
EFA-075	EtFOS025.D	25	8.6	1805236	613		80%	Day 28	6.2	0	0		5.9	464731	403	8.3	846072	316
MeOH Blank	EtFOS026.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
L4 1.5, 621.60	EtFOS027.D	4	8.6	1932192	191348	(B)			6.2	113170	36	94%	5.9	453100	403	8.3	(B)	316
MeOH Blank	EtFOS028.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
EFA-091	EtFOS029.D	26	8.6	1287115	430	5%		Day 35	6.2	0	0		5.9	464732	403	8.3	855890	316
EFA-092	EtFOS030.D	27	8.6	1200913	409			Day 35	6.2	0	0		5.9	454761	403	8.3	838230	316
EFA-093	EtFOS031.D	28	8.6	1954046	649		105%	Day 35	6.2	0	0		5.9	459221	403	8.3	865946	316
EFA-109	EtFOS032.D	29	8.6	1284635	435	2%		Day 42	6.2	0	0		5.9	458673	403	8.3	845709	316
EFA-110	EtFOS033.D	30	8.6	1298904	441			Day 42	6.2	0	0		5.9	457220	403	8.3	842379	316
EFA-111	EtFOS034.D	31	8.6	2002610	667		105%	Day 42	6.2	0	0		5.9	466294	403	8.3	863388	316
EFA-127	EtFOS035.D	32	8.6	1295601	433	3%		Day 49	6.2	0	0		5.9	460311	403	8.3	855846	316
EFA-128	EtFOS036.D	33	8.6	1236840	421			Day 49	6.2	0	0		5.9	452048	403	8.4	840371	316
EFA-129	EtFOS037.D	34	8.6	2094856	694		123%	Day 49	6.2	0	0		5.9	454032	403	8.4	868271	316
MeOH Blank	EtFOS049.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.4	0	0
MeOH Blank	EtFOS050.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.4	0	0
L1 1.5, 155.40	EtFOS051.D	1	8.6	494514	164	106%			6.2	5778	1	114%	5.9	485811	403	8.4	841210	316
L2 1.5, 310.80	EtFOS052.D	2	8.6	949495	310	100%			6.2	41998	13	98%	5.9	474209	403	8.4	871122	316
L3 1.5, 466.20	EtFOS053.D	3	8.6	1416444	459	98%			6.2	83340	26	103%	5.9	456592	403	8.4	883822	316
L4 1.5, 621.60	EtFOS054.D	4	8.6	1838575	16370	(B)			6.2	110610	34	90%	5.9	461931	403	8.6	(B)	316
L5 1.5, 777.00	EtFOS055.D	5	8.6	2308742	777	100%			6.2	164483	(A)		5.9	(A)	403	8.4	855704	316
L6 1.5, 932.40	EtFOS056.D	6	8.6	2764328	912	98%			6.2	209470	64	101%	5.9	469641	403	8.4	874479	316
													mean	460164			mean	854060
													S.D.	8224			S.D.	36597
													%S.D.	1.8%			%S.D.	4.3%

Method ID: 1006_15.M

Internal Standard quant: r2 = 0.999
Curve averaged, Linear,include origin.
Calibration range: 155-932 ng/ml

Internal Standard quant: r2 = 0.997
Curve averaged, Linear,include origin.
Calibration range: 1.28-64 ng/ml

(A) Exluded on te basis of data quality objectives; see text.

(B) Excluded - no internal standard added.

BACK TO MAIN

EtFOSE Alcohol Hydrolysis Study
50C; pH 3
Reprocessed data on 8-31-00 using internal standard quantitation.

			N-EtFOSE Alcohol						PFOS				THPFOS			N-MeFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc. (ng/ml)	% of Standard or % RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc. (ng/ml)	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	EtFOS058.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0	8.3	0	0
L1 3.0, 155.40	EtFOS059.D	41	8.6	100572	31	(A)			6.2	11759	2.15	(A)	5.9	450603	403	8.3	796488	316
L2 3.0, 310.80	EtFOS060.D	42	8.6	993993	332	107%			6.2	46340	13.14	103%	5.9	457139	403	8.3	807861	316
L3 3.0, 466.20	EtFOS061.D	43	8.6	1469637	478	103%			6.2	84849	25.75	101%	5.9	451913	403	8.3	831972	316
L4 3.0, 621.60	EtFOS062.D	44	8.6	1837884	581	93%			6.2	125379	38.71	101%	5.9	453343	403	8.3	858143	316
L5 3.0, 777.00	EtFOS063.D	45	8.6	2351584	763	98%			6.2	211732	68.08	(A)	5.9	443115	403	8.3	837174	316
L6 3.0, 932.40	EtFOS064.D	46	8.6	2880052	932	100%			6.2	198183	63.05	99%	5.9	447053	403	8.3	839951	316
MeOH Blank	EtFOS065.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0	8.3	0	0
MeOH Blank	EtFOS066.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0	8.3	0	0
EFA-004	EtFOS067.D	51	8.6	1348761	461	0%		Day 0	6.2	0	0.00		5.9	456498	403	8.3	791432	316
EFA-005	EtFOS068.D	52	8.6	1306963	460			Day 0	6.2	0	0.00		5.9	444095	403	8.3	769975	316
EFA-006	EtFOS069.D	53	8.6	1981411	702		111%	Day 0	6.2	0	0.00		5.9	440492	403	8.3	766604	316
EFA-022	EtFOS070.D	54	8.6	1219370	402	4%		Day 7	6.2	0	0.00		5.9	466273	403	8.3	821287	316
EFA-023	EtFOS071.D	55	8.6	1235031	417			Day 7	6.2	0	0.00		5.9	453856	403	8.3	801078	316
EFA-024	EtFOS072.D	56	8.6	1672756	594		85%	Day 7	6.2	0	0.00		5.9	432375	403	8.3	764158	316
EFA-040	EtFOS073.D	57	8.6	1066222	376	9%		Day 14	6.2	0	0.00		5.9	442557	403	8.3	766340	316
EFA-041	EtFOS074.D	58	8.6	1134902	410			Day 14	6.2	0	0.00		5.9	435689	403	8.3	748177	316
EFA-042	EtFOS075.D	59	8.6	1643131	567		80%	Day 14	6.2	0	0.00		5.9	447337	403	8.3	786346	316
EFA-058	EtFOS076.D	60	8.6	1150307	380	10%		Day 21	6.2	0	0.00		5.9	432112	403	8.3	817457	316
EFA-059	EtFOS077.D	61	8.5	1102935	344			Day 21	6.2	0	0.00		5.9	445253	403	8.3	864520	316
EFA-060	EtFOS078.D	62	8.5	1706690	525		75%	Day 21	6.2	0	0.00		5.9	436986	403	8.3	881726	316
EFA-076	EtFOS079.D	63	8.5	1113117	371	5%		Day 28	6.2	0	0.00		5.9	442336	403	8.3	809837	316
EFA-077	EtFOS080.D	64	8.6	1146209	389			Day 28	6.2	0	0.00		5.9	450543	403	8.3	795653	316
EFA-078	EtFOS081.D	65	8.5	1622872	560		83%	Day 28	6.2	0	0.00		5.9	429690	403	8.3	785909	316
MeOH Blank	EtFOS082.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0	8.3	0	0
L4 3.0, 621.60	EtFOS083.D	44	8.6	1854835	612	98%			6.2	124667	29.10	102%	5.9	446589	403	8.3	823651	316
MeOH Blank	EtFOS084.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0	8.3	0	0
EFA-094	EtFOS085.D	66	8.6	1130406	378	0%		Day 35	6.2	0	0.00		5.9	443433	403	8.3	808420	316
EFA-095	EtFOS086.D	67	8.6	1142643	380			Day 35	6.2	0	0.00		5.9	445791	403	8.3	813785	316
EFA-096	EtFOS087.D	68	8.6	1775042	597		100%	Day 35	6.2	0	0.00		5.9	439114	403	8.3	806848	316
EFA-112	EtFOS088.D	69	8.6	1191578	394	1%		Day 42	6.2	0	0.00		5.9	454153	403	8.3	817322	316
EFA-113	EtFOS089.D	70	8.5	1208645	398			Day 42	6.2	0	0.00		5.9	435430	403	8.3	820921	316
EFA-114	EtFOS090.D	71	8.6	1824457	595		91%	Day 42	6.2	0	0.00		5.9	445709	403	8.3	831848	316
EFA-130	EtFOS091.D	72	8.5	1235171	397	4%		Day 49	6.2	0	0.00		5.9	447667	403	8.3	841331	316
EFA-131	EtFOS092.D	73	8.6	1172642	380			Day 49	6.2	0	0.00		5.9	435779	403	8.3	834320	316
EFA-132	EtFOS093.D	74	8.6	1932230	638		115%	Day 49	6.2	0	0.00		5.9	431328	403	8.3	821524	316
MeOH Blank	EtFOS105.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0	8.3	0	0
MeOH Blank	EtFOS106.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0	8.3	0	0
L1 3.0, 155.40	EtFOS107.D	41	8.6	100153	31	(A)			6.2	14073	3.02	(A)	5.9	439145	403	8.3	790968	316
L2 3.0, 310.80	EtFOS108.D	42	8.6	957828	314	101%			6.2	46697	13.67	107%	5.9	444646	403	8.3	821694	316
L3 3.0, 466.20	EtFOS109.D	43	8.6	1450723	469	101%			6.2	83364	25.78	101%	5.9	443376	403	8.3	837999	316
L4 3.0, 621.60	EtFOS110.D	44	8.6	1815257	612	98%			6.2	119432	38.69	101%	5.9	432006	403	8.3	804635	316
L5 3.0, 777.00	EtFOS111.D	45	8.6	2282741	770	99%			6.2	199387	65.17	(A)	5.9	435449	403	8.3	805556	316
L6 3.0, 932.40	EtFOS112.D	46	8.6	2876504	967	104%			6.2	197627	63.60	99%	5.9	442026	403	8.3	808753	316
Method ID: 0830D00.M			Internal Standard quant: r2 = 0.998						Internal Standard quant: r2 = 0.999				mean	443808		mean	811667	
			Curve averaged, Linear,include origin.						Curve averaged, Linear,include origin.				S.D.	8328		S.D.	28775	
			Calibration range: 155-932 ng/ml						Calibration range: 12.8-64 ng/ml				%S.D.	1.9%		%S.D.	3.5%	

(A) Exluded on te basis of data quality objectives; see text.

BACK TO MAIN

EtFOSE Alcohol Hydrolysis Study
50C; pH 5
Reprocessed data on 8-31-00 using internal standard quantitation.

			N-EtFOSE Alcohol						PFOS				THPFOS			N-MeFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc. (ng/ml)	% of Standard or % RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc. (ng/ml)	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	EtFOS002.D	91	8.6	0	0				6.1	0	0.00		5.9	0	0.00	8.3	0	0
L1 5.0, 155.40	EtFOS003.D	1	8.6	403311	155	99%			6.1	9925	(A)		5.9	463166	402.60	8.3	762610	316
L2 5.0, 310.80	EtFOS004.D	2	8.6	835089	321	103%			6.2	40365	12.83	101%	5.9	459954	402.60	8.3	757407	316
L3 5.0, 466.20	EtFOS005.D	3	8.6	1242363	458	98%			6.2	85079	26.71	105%	5.9	464019	402.60	8.3	787573	316
L4 5.0, 621.60	EtFOS006.D	4	8.6	1685478	637	102%			6.2	119421	37.32	97%	5.9	465570	402.60	8.3	767532	316
L5 5.0, 777.00	EtFOS007.D	5	8.6	2022413	765	98%			6.2	162768	51.42	97%	5.9	460220	402.60	8.3	766958	316
L6 5.0, 932.40	EtFOS008.D	6	8.6	2447180	937	101%			6.2	198997	65.61	103%	5.9	440802	402.60	8.3	756833	316
MeOH Blank	EtFOS009.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0.00	8.3	0	0
MeOH Blank	EtFOS010.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0.00	8.3	0	0
EFA-007	EtFOS011.D	11	8.6	1134978	485	4%		Day 0	6.2	0	0.00		5.9	450402	402.60	8.3	678947	316
EFA-008	EtFOS012.D	12	8.6	1131090	468			Day 0	6.2	0	0.00		5.9	460158	402.60	8.3	702308	316
EFA-009	EtFOS013.D	13	8.6	1672237	700		103%	Day 0	6.2	0	0.00		5.9	455075	402.60	8.3	692884	316
EFA-025	EtFOS014.D	14	8.6	1147480	461	2%		Day 7	6.2	0	0.00		5.9	471566	402.60	8.3	722196	316
EFA-026	EtFOS015.D	15	8.6	1100973	451			Day 7	6.2	0	0.00		5.9	471165	402.60	8.3	709296	316
EFA-027	EtFOS016.D	16	8.6	1642451	680		103%	Day 7	6.2	0	0.00		5.9	463772	402.60	8.3	700710	316
EFA-043	EtFOS017.D	17	8.6	1098638	468	2%		Day 14	6.2	0	0.00		5.9	464470	402.60	8.3	681111	316
EFA-044	EtFOS018.D	18	8.6	1111964	476			Day 14	6.2	0	0.00		5.9	464484	402.60	8.3	677627	316
EFA-045	EtFOS019.D	19	8.6	1595846	659		86%	Day 14	6.2	0	0.00		5.9	457576	402.60	8.3	702137	316
EFA-061	EtFOS020.D	20	8.6	1057199	411	2%		Day 21	6.2	0	0.00		5.9	461557	402.60	8.3	746995	316
EFA-062	EtFOS021.D	21	8.6	1073533	421			Day 21	6.2	0	0.00		5.9	473678	402.60	8.3	741107	316
EFA-063	EtFOS022.D	22	8.6	1872384	633		100%	Day 21	6.2	0	0.00		5.9	516735	402.60	8.3	858002	316
EFA-079	EtFOS023.D	23	8.6	1057738	421	4%		Day 28	6.2	0	0.00		5.9	456157	402.60	8.3	729455	316
EFA-080	EtFOS024.D	24	8.6	1047642	438			Day 28	6.2	0	0.00		5.9	457475	402.60	8.3	695006	316
EFA-081	EtFOS025.D	25	8.6	1615792	643		98%	Day 28	6.2	0	0.00		5.9	454552	402.60	8.3	729303	316
MeOH Blank	EtFOS026.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0.00	8.3	0	0
L4 5.0, 621.60	EtFOS027.D	4	8.6	1595911	602	97%			6.2	122855	37.69	99%	5.9	474181	402.60	8.3	769377	316
MeOH Blank	EtFOS028.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0.00	8.3	0	0
EFA-097	EtFOS029.D	26	8.6	1053254	434	1%		Day 35	6.2	0	0.00		5.9	467618	402.60	8.3	704694	316
EFA-098	EtFOS030.D	27	8.6	1065586	429			Day 35	6.2	0	0.00		5.9	458821	402.60	8.3	720938	316
EFA-099	EtFOS031.D	28	8.6	1619591	638		95%	Day 35	6.2	0	0.00		5.9	455327	402.60	8.3	736712	316
EFA-115	EtFOS032.D	29	8.6	1060903	426	1%		Day 42	6.2	0	0.00		5.9	457873	402.60	8.3	722797	316
EFA-116	EtFOS033.D	30	8.6	1040839	429			Day 42	6.2	0	0.00		5.9	460032	402.60	8.3	704234	316
EFA-117	EtFOS034.D	31	8.6	1639828	651		103%	Day 42	6.2	0	0.00		5.9	464180	402.60	8.3	730256	316
EFA-133	EtFOS035.D	32	8.6	1001513	402	4%		Day 49	6.2	0	0.00		5.9	448743	402.60	8.3	722905	316
EFA-134	EtFOS036.D	33	8.6	1026623	418			Day 49	6.2	0	0.00		5.9	461024	402.60	8.3	712643	316
EFA-135	EtFOS037.D	34	8.6	1701880	680		124%	Day 49	6.2	0	0.00		5.9	445353	402.60	8.3	726449	316
MeOH Blank	EtFOS049.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0.00	8.3	0	0
MeOH Blank	EtFOS050.D	91	8.6	0	0				6.2	0	0.00		5.9	0	0.00	8.3	0	0
L1 5.0, 155.40	EtFOS051.D	1	8.6	368386	147	95%			6.2	12848	(A)		5.9	461447	402.60	8.4	733167	316
L2 5.0, 310.80	EtFOS052.D	2	8.6	763938	311	100%			6.2	43154	13.86	109%	5.9	455083	402.60	8.4	714671	316
L3 5.0, 466.20	EtFOS053.D	3	8.6	1174479	463	99%			6.2	76798	24.22	95%	5.9	462085	402.60	8.4	735880	316
L4 5.0, 621.60	EtFOS054.D	4	8.6	1571631	628	101%			6.2	116168	37.44	98%	5.9	451392	402.60	8.4	726280	316
L5 5.0, 777.00	EtFOS055.D	5	8.6	2004306	776	100%			6.2	153191	49.42	93%	5.9	450735	402.60	8.4	748759	316
L6 5.0, 932.40	EtFOS056.D	6	8.6	2360319	927	99%			6.2	184840	63.88	100%	5.9	420520	402.60	8.4	737916	316
Method ID: 0830A00.M			Internal Standard quant: r2 = 0.999						Internal Standard quant: r2 = 0.999				mean	460188		mean	730099	
			Curve averaged, Linear,include origin.						Curve averaged, Linear,include origin.				S.D.	13657		S.D.	34464	
			Calibration range: 155-932 ng/ml						Calibration range: 12.8-64 ng/ml				%S.D.	3.0%		%S.D.	4.7%	

(A) Exluded on te basis of data quality objectives; see text.

BACK TO MAIN

EtFOSE Alcohol Hydrolysis Study
50C; pH 7
Reprocessed data on 8-31-00 using internal standard quantitation.

			N-EtFOSE Alcohol						PFOS				THPFOS			N-MeFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc. (ng/ml)	% of Standard or % RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc. (ng/ml)	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	EtFOS058.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.4	0	0
L1 7.0, 155.40	EtFOS059.D	41	8.6	480449	173	111%			6.2	11467	4	(A)	5.9	451663	403	8.4	706921	316
L2 7.0, 310.80	EtFOS060.D	42	8.6	820775	308	99%			6.2	38240	12	98%	5.9	453588	403	8.4	725999	316
L3 7.0, 466.20	EtFOS061.D	43	8.6	1195458	474	102%			6.2	111921	36	(A)	5.9	461481	403	8.4	709796	316
L4 7.0, 621.60	EtFOS062.D	44	8.6	1158453	457	(A)			6.2	117460	39	101%	5.9	454013	403	8.4	711675	316
L5 7.0, 777.00	EtFOS063.D	45	8.6	1970492	739	95%			6.2	157029	51	96%	5.9	463358	403	8.4	767139	316
L6 7.0, 932.40	EtFOS064.D	46	8.6	2451486	951	102%			6.2	191758	63	99%	5.9	454355	403	8.4	748395	316
MeOH Blank	EtFOS065.D	91	8.6	0	0				6.2	7938	0		5.9	0	0	8.3	0	0
MeOH Blank	EtFOS066.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
EFA-010	EtFOS067.D	51	8.6	1152415	462	3%		Day 0	6.2	0	0		5.9	457166	403	8.3	701483	316
EFA-011	EtFOS068.D	52	8.6	1143301	474			Day 0	6.2	0	0		5.9	457511	403	8.4	679580	316
EFA-012	EtFOS069.D	53	8.6	1745528	700		107%	Day 0	6.2	0	0		5.9	471437	403	8.3	716153	316
EFA-028	EtFOS070.D	54	8.6	1107418	452	1%		Day 7	6.2	0	0		5.9	457174	403	8.3	687125	316
EFA-029	EtFOS071.D	55	8.6	1098715	459			Day 7	6.2	0	0		5.9	460597	403	8.3	672434	316
EFA-030	EtFOS072.D	56	8.6	1678444	681		104%	Day 7	6.2	0	0		5.9	465446	403	8.3	706387	316
EFA-046	EtFOS073.D	57	8.6	1100624	450	2%		Day 14	6.2	0	0		5.9	458934	403	8.3	685975	316
EFA-047	EtFOS074.D	58	8.6	1116212	458			Day 14	6.2	9782	3		5.9	468514	403	8.3	684580	316
EFA-048	EtFOS075.D	59	8.6	1622694	692		110%	Day 14	6.2	10000	3		5.9	461270	403	8.3	672527	316
EFA-064	EtFOS076.D	60	8.6	1085190	430	4%		Day 21	6.2	9278	3		5.9	453289	403	8.3	705473	316
EFA-065	EtFOS077.D	61	8.6	1055654	412			Day 21	6.2	0	0		5.9	446052	403	8.3	714354	316
EFA-066	EtFOS078.D	62	8.6	1643724	636		99%	Day 21	6.2	0	0		5.9	455047	403	8.3	738802	316
EFA-082	EtFOS079.D	63	8.6	1056770	422	0%		Day 28	6.2	0	0		5.9	450095	403	8.3	699469	316
EFA-083	EtFOS080.D	64	8.6	1074530	420			Day 28	6.2	0	0		5.9	455593	403	8.3	714231	316
EFA-084	EtFOS081.D	65	8.6	1619857	660		110%	Day 28	6.2	0	0		5.9	451026	403	8.3	702764	316
MeOH Blank	EtFOS082.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
L4 7.0, 621.60	EtFOS081.D	44	8.6	1153406	467	(A)			6.2	113680	38	100%	5.9	447307	403	8.3	695268	316
MeOH Blank	EtFOS084.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
EFA-100	EtFOS085.D	66	8.6	1054195	418	2%		Day 35	6.2	0	0		5.9	449888	403	8.3	703676	316
EFA-101	EtFOS086.D	67	8.6	1057309	425			Day 35	6.2	0	0		5.9	447436	403	8.3	695096	316
EFA-102	EtFOS087.D	68	8.6	1578463	648		104%	Day 35	6.2	0	0		5.9	446563	403	8.3	697321	316
EFA-118	EtFOS088.D	69	8.6	1054100	420	3%		Day 42	6.2	0	0		5.9	451243	403	8.3	700866	316
EFA-119	EtFOS089.D	70	8.6	1061878	408			Day 42	6.2	0	0		5.9	453084	403	8.3	725550	316
EFA-120	EtFOS090.D	71	8.6	1629040	629		99%	Day 42	6.2	0	0		5.9	470642	403	8.3	739763	316
EFA-136	EtFOS091.D	72	8.6	1043657	408	1%		Day 49	6.2	0	0		5.9	441948	403	8.3	712663	316
EFA-137	EtFOS092.D	73	8.6	1041709	412			Day 49	6.2	0	0		5.9	451166	403	8.3	705068	316
EFA-138	EtFOS093.D	74	8.6	1729685	729		(A)	Day 49	6.2	0	0		5.9	445912	403	8.3	682117	316
MeOH Blank	EtFOS105.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
MeOH Blank	EtFOS106.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
L1 7.0, 155.40	EtFOS107.D	41	8.6	486234	177	114%			6.2	6766	(A)		5.9	438170	403	8.3	701583	316
L2 7.0, 310.80	EtFOS108.D	42	8.6	810866	313	101%			6.2	37598	12	97%	5.9	448318	403	8.3	707107	316
L3 7.0, 466.20	EtFOS109.D	43	8.6	1178478	468	100%			6.2	119963	40	157%	5.9	449708	403	8.3	708259	316
L4 7.0, 621.60	EtFOS110.D	44	8.6	1140569	448	(A)			6.2	117083	40	104%	5.9	442333	403	8.3	714457	316
L5 7.0, 777.00	EtFOS111.D	45	8.6	1936040	769	99%			6.2	152954	51	97%	5.9	446823	403	8.3	725954	316
L6 7.0, 932.40	EtFOS112.D	46	8.6	2364608	941	101%			6.2	184423	63	99%	5.9	438757	403	8.3	729327	316
Method ID: 0830C00.M			Internal Standard quant: r2 = 0.998						Internal Standard quant: r2 = 0.999				mean	453655		mean	708376	
			Curve averaged, Linear,include origin.						Curve averaged, Linear,include origin.				S.D.	8350		S.D.	20821	
			Calibration range: 155-932 ng/ml						Calibration range: 12.8-64 ng/ml				%S.D.	1.8%		%S.D.	2.9%	

(A) Exluded on te basis of data quality objectives; see text.

BACK TO MAIN

EtFOSE Alcohol Hydrolysis Study
50C; pH 9
Reprocessed data on 8-29-00

			N-EtFOSE Alcohol						PFOS				THPFOS			N-MeFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc. (ng/ml)	% of Standard or % RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc. (ng/ml)	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	EtFOS002.D	91	8.5	0	0.0				6.1	0	0.0		5.86	0	0.0	8.3	0	0.0
L1 9.0, 155.40	EtFOS003.D	1	8.5	409224	144	93%			6.1	7965	1.4	110%	5.9	303076	402.6	8.3	714775	316
L2 9.0, 310.80	EtFOS004.D	2	8.5	892538	324	104%			6.2	39881	14.1	111%	5.9	338869	402.6	8.3	720711	316
L3 9.0, 466.20	EtFOS005.D	3	8.6	1348314	482	103%			6.2	74208	28.3	111%	5.9	338620	402.6	8.3	739101	316
L4 9.0, 621.60	EtFOS006.D	4	8.5	1769188	631	102%			6.2	112382	41.3	108%	5.9	358974	402.6	8.3	744916	316
L5 9.0, 777.00	EtFOS007.D	5	8.5	2119590	763	98%			6.2	147683	54.4	107%	5.9	362649	402.6	8.3	739652	316
L6 9.0, 932.40	EtFOS008.D	6	8.5	2618151	949	102%			6.2	181333	65.1	102%	5.9	374889	402.6	8.3	736444	316
MeOH Blank	EtFOS009.D	91	8.5	0	0				6.2	0	0.0		5.9	0	0.0	8.3	0	0
MeOH Blank	EtFOS010.D	91	8.5	0	0				6.2	0	0.0		5.9	0	0.0	8.3	0	0
EFA-013	EtFOS011.D	11	8.5	1143422	435	1%		Day 0	6.2	0	0.0		5.9	392100	402.6	8.3	692760	316
EFA-014	EtFOS012.D	12	8.5	1142252	441			Day 0	6.2	0	0.0		5.9	388527	402.6	8.3	683190	316
EFA-015	EtFOS013.D	13	8.5	1685734	640		93%	Day 0	6.2	0	0.0		5.9	401788	402.6	8.3	699902	316
EFA-031[A]	EtFOS014.D	14	8.5	1134663	426	33%		Day 7	6.2	0	0.0		5.9	398631	402.6	8.3	701801	316
EFA-032	EtFOS015.D	15	8.5	809137	304			Day 7	6.2	0	0.0		5.9	304032	402.6	8.3	694599	316
EFA-033	EtFOS016.D	16	8.5	1640714	632		(A)	Day 7	6.2	0	0.0		5.9	398182	402.6	8.3	689954	316
EFA-049	EtFOS017.D	17	8.5	1119730	438	0%		Day 14	6.2	0	0.0		5.9	407818	402.6	8.3	674255	316
EFA-050	EtFOS018.D	18	8.5	1118496	437			Day 14	6.2	0	0.0		5.9	400267	402.6	8.3	675759	316
EFA-051	EtFOS019.D	19	8.5	1650519	642		94%	Day 14	6.2	0	0.0		5.9	402785	402.6	8.3	683372	316
EFA-067	EtFOS020.D	20	8.5	1099106	394	0%		Day 21	6.2	0	0.0		5.9	401387	402.6	8.3	733563	316
EFA-068	EtFOS021.D	21	8.5	1102692	394			Day 21	6.2	0	0.0		5.9	403133	402.6	8.3	736476	316
EFA-069	EtFOS022.D	22	8.5	1658623	602		96%	Day 21	6.2	0	0.0		5.9	402836	402.6	8.3	731089	316
EFA-085	EtFOS023.D	23	8.5	1092118	398	4%		Day 28	6.2	0	0.0		5.9	402632	402.6	8.3	722324	316
EFA-086	EtFOS024.D	24	8.5	1088786	413			Day 28	6.2	0	0.0		5.9	401613	402.6	8.3	694410	316
EFA-087	EtFOS025.D	25	8.5	1619682	621		99%	Day 28	6.2	0	0.0		5.9	406718	402.6	8.3	692520	316
MeOH Blank	EtFOS026.D	91	8.5	0	0				6.2	0	0.0		5.9	0	0.0	8.3	0	0
L4 9.0, 621.60	EtFOS027.D	4	8.5	1678694	638	103%			6.2	110233	35.1	94%	5.9	400498	402.6	8.3	699217	316
MeOH Blank	EtFOS028.D	91	8.5	0	0				6.2	0	0.0		5.9	0	0.0	8.3	0	0
EFA-103	EtFOS029.D	26	8.5	1079749	404	3%		Day 35	6.2	0	0.0		5.9	414779	402.6	8.3	704303	316
EFA-104	EtFOS030.D	27	8.5	1067069	393			Day 35	6.2	0	0.0		5.9	402920	402.6	8.3	713838	316
EFA-105	EtFOS031.D	28	8.5	1655168	611		97%	Day 35	6.2	0	0.0		5.9	416468	402.6	8.3	719832	316
EFA-121	EtFOS032.D	29	8.5	1095265	421	7%		Day 42	6.2	0	0.0		5.9	400033	402.6	8.3	685534	316
EFA-122	EtFOS033.D	30	8.5	1026620	395			Day 42	6.2	0	0.0		5.9	400713	402.6	8.3	684709	316
EFA-123	EtFOS034.D	31	8.5	1593121	577		78%	Day 42	6.2	0	0.0		5.9	404666	402.6	8.3	732430	316
EFA-139	EtFOS035.D	32	8.5	1058111	390	1%		Day 49	6.2	0	0.0		5.9	401817	402.6	8.3	713040	316
EFA-140	EtFOS036.D	33	8.5	1080272	394			Day 49	6.2	0	0.0		5.9	409040	402.6	8.3	720879	316
EFA-141	EtFOS037.D	34	8.5	1114214	424		(A,B)	Day 49	6.2	0	0.0		5.9	(B)	402.6	8.3	693569	316
MeOH Blank	EtFOS049.D	91	8.5	0	0				6.2	0	0.0		5.9	0	0.0	8.3	0	0
MeOH Blank	EtFOS050.D	91	8.5	0	0				6.2	0	0.0		5.9	0	0.0	8.3	0	0
L1 9.0, 155.40	EtFOS051.D	1	8.5	406650	148	95%			6.2	9371	0.9	(A)	5.9	414987	402.6	8.3	694298	316
L2 9.0, 310.80	EtFOS052.D	2	8.5	832823	320	103%			6.2	42306	12.2	96%	5.9	407028	402.6	8.3	680556	316
L3 9.0, 466.20	EtFOS053.D	3	8.6	1278664	475	102%			6.2	78648	24.6	96%	5.9	407820	402.6	8.3	712130	316
L4 9.0, 621.60	EtFOS054.D	4	8.6	1637356	623	100%			6.2	112614	37.0	97%	5.9	399211	402.6	8.3	697612	316
L5 9.0, 777.00	EtFOS055.D	5	8.5	2035025	756	97%			6.2	147384	47.8	94%	5.9	410184	402.6	8.3	717132	316
L6 9.0, 932.40	EtFOS056.D	6	8.5	2422983	920	99%			6.2	180489	60.4	95%	5.9	401194	402.6	8.3	703103	316
														Mean	391136		Mean	702521
														S.D.	28476		S.D.	17720
														%S.D.	7.3%		%S.D.	2.5%

Method ID: 08289is.m

Internal Standard quant: $r^2 = 0.999$
Curve averaged, linear, include origin
Calibration range : 155-932 ng/ml

Internal Standard quant: $r^2 = 0.995$
Curve averaged, linear, include origin
Calibration range : 12.8-64 ng/ml

(A) Exluded on te basis of data quality objectives; see text.

(B) Excluded - no internal standard added.

BACK TO MAIN

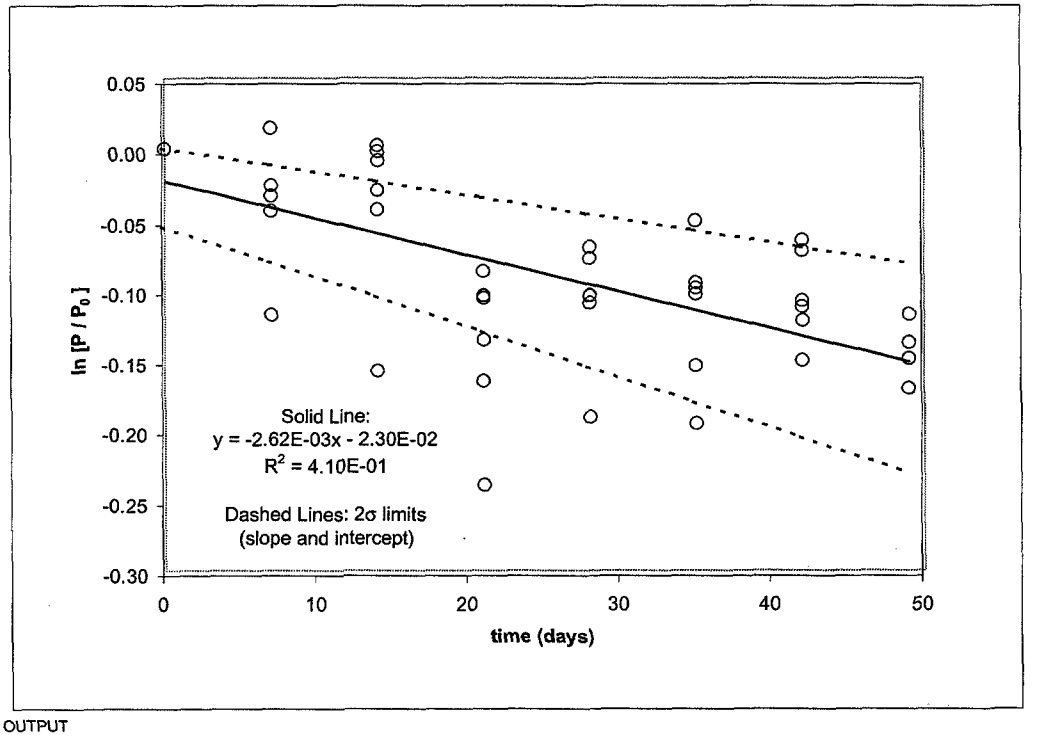
EtFOSE Alcohol Hydrolysis Study
50C; pH 11
Reprocessed data on 8-31-00 using internal standard quantitation.

			N-EtFOSE Alcohol						PFOS				THPFOS			N-MeFOSE Alcohol		
Sample I.D.	Data File	Vial #	Ret Time	Area	Conc. (ng/ml)	% of Standard or % RSD	% Spike Recovery	Time Point	Ret Time	Area	Conc. (ng/ml)	%Standard	Ret Time	Area	Conc.	Ret Time	Area	Conc.
MeOH Blank	EtFOS058.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
L1 11.0, 155.40	EtFOS059.D	41	8.6	446561	145	93%			6.2	17657	2	(A)	5.9	430913	403	8.3	705152	316
L2 11.0, 310.80	EtFOS060.D	42	8.6	926265	347	112%			6.2	48588	13	100%	5.9	446510	403	8.3	665610	316
L3 11.0, 466.20	EtFOS061.D	43	8.6	1283574	467	100%			6.2	87017	27	105%	5.9	433489	403	8.3	697751	316
L4 11.0, 621.60	EtFOS062.D	44	8.6	1726739	631	101%			6.2	116706	37	97%	5.9	438329	403	8.3	704137	316
L5 11.0, 777.00	EtFOS063.D	45	8.6	2146734	774	100%			6.2	149347	48	90%	5.9	442757	403	8.3	717974	316
L6 11.0, 932.40	EtFOS064.D	46	8.6	2634375	911	98%			6.2	194601	63	98%	5.9	448236	403	8.3	751856	316
MeOH Blank	EtFOS065.D	91	8.6	0	0				6.2	11457	0		5.9	0	0	8.3	0	0
MeOH Blank	EtFOS066.D	91	8.6	0	0				6.2	0	0		5.9	0	0	8.3	0	0
EFA-016	EtFOS067.D	51	8.6	1101922	399	6%		Day 0	6.2	16162	2		5.9	440185	403	8.3	694734	316
EFA-017	EtFOS068.D	52	8.6	1115067	426			Day 0	0.0	0	0		5.9	452518	403	8.3	661592	316
EFA-018	EtFOS069.D	53	8.6	1618557	624		97%	Day 0	6.2	13427	1		5.9	442063	403	8.3	666969	316
EFA-034	EtFOS070.D	54	8.6	1097421	416	1%		Day 7	6.2	13137	0		5.9	458756	403	8.3	666079	316
EFA-035	EtFOS071.D	55	8.6	1135383	421			Day 7	6.2	8862	0		5.9	473828	403	8.3	680123	316
EFA-036	EtFOS072.D	56	8.6	1607415	630		97%	Day 7	6.2	8220	0		5.9	445182	403	8.3	656395	316
EFA-052	EtFOS073.D	57	8.6	1103015	413	0%		Day 14	6.2	12032	0		5.9	461084	403	8.3	672829	316
EFA-053	EtFOS074.D	58	8.6	1082138	413			Day 14	6.2	11113	0		5.9	449074	403	8.3	660257	316
EFA-054	EtFOS075.D	59	8.6	1675149	632		100%	Day 14	6.2	16343	1		5.9	473317	403	8.3	681695	316
EFA-070	EtFOS076.D	60	8.6	1079072	378	0%		Day 21	6.2	13652	1		5.9	458391	403	8.3	715527	316
EFA-071	EtFOS077.D	61	8.6	1104263	378			Day 21	0.0	0	0		5.9	460771	403	8.3	733086	316
EFA-072	EtFOS078.D	62	8.6	1611497	579		92%	Day 21	6.2	16288	1		5.9	459247	403	8.3	712998	316
EFA-088	EtFOS079.D	63	8.6	1046583	392	3%		Day 28	6.2	8544	0		5.9	456132	403	8.3	671926	316
EFA-089	EtFOS080.D	64	8.6	1064303	378			Day 28	6.2	13507	1		5.9	458997	403	8.3	706016	316
EFA-090	EtFOS081.D	65	8.5	1652545	613		105%	Day 28	6.2	13263	0		5.9	464442	403	8.3	692615	316
MeOH Blank	EtFOS082.D	91	8.5	0	0				6.2	0	0		5.9	0	0	8.3	0	0
L4 9.0, 621.60	EtFOS083.D	44	8.5	1748022	636	102%			6.2	113172	35	91%	5.9	450159	403	8.3	705958	316
MeOH Blank	EtFOS084.D	91	8.5	0	0				6.2	14915	0		5.9	0	0	8.3	0	0
EFA-106	EtFOS085.D	66	8.5	1049049	392	0%		Day 35	6.2	16449	2		5.9	451843	403	8.3	672663	316
EFA-107	EtFOS086.D	67	8.5	1083945	392			Day 35	6.2	16564	2		5.9	462459	403	8.3	695241	316
EFA-108	EtFOS087.D	68	8.5	1604358	584		88%	Day 35	6.2	12770	0		5.9	460398	403	8.3	704039	316
EFA-124	EtFOS088.D	69	8.5	1087655	391	2%		Day 42	6.2	9498	0		5.9	467646	403	8.3	700030	316
EFA-125	EtFOS089.D	70	8.5	1046304	383			Day 42	6.2	14181	1		5.9	453505	403	8.3	685689	316
EFA-126	EtFOS090.D	71	8.5	1605607	586		92%	Day 42	6.2	13628	1		5.9	453474	403	8.3	701982	316
EFA-142	EtFOS091.D	72	8.5	1042824	364	1%		Day 49	6.2	17403	2		5.9	449375	403	8.3	716622	316
EFA-143	EtFOS092.D	73	8.5	1019110	369			Day 49	6.2	18320	2		5.9	437907	403	8.3	691579	316
EFA-144	EtFOS093.D	74	8.5	1655321	597		106%	Day 49	6.2	15319	1		5.9	436689	403	8.3	711998	316
MeOH Blank	EtFOS105.D	91	8.5	0	0				6.2	15463	0		5.9	0	0	8.3	0	0
MeOH Blank	EtFOS106.D	91	8.5	0	0				6.2	16895	0		5.9	0	0	8.3	0	0
L1 11.0, 155.40	EtFOS107.D	41	8.5	453167	148	95%			6.2	22157	4	(A)	5.9	439507	403	8.3	702584	316
L2 11.0, 310.80	EtFOS108.D	42	8.5	899458	335	108%			6.2	55912	15	118%	5.9	451746	403	8.3	667702	316
L3 11.0, 466.20	EtFOS109.D	43	8.5	1289183	460	99%			6.2	92925	28	111%	5.9	441567	403	8.3	710837	316
L4 11.0, 621.60	EtFOS110.D	44	8.5	1730293	625	101%			6.2	126498	39	103%	5.9	447483	403	8.3	711341	316
L5 11.0, 777.00	EtFOS111.D	45	8.5	2150602	767	99%			6.2	162546	52	99%	5.9	443873	403	8.3	726151	316
L6 11.0, 932.40	EtFOS112.D	46	8.5	2594308	941	101%			6.2	196851	64	100%	5.9	443604	403	8.3	717471	316
Method ID: 0830A00.M			Internal Standard quant: r2 = 0.998 Curve averaged, Linear,include origin. Calibration range: 155-932 ng/ml						Internal Standard quant: r2 = 0.995 Curve averaged, Linear,include origin. Calibration range: 12.8-64 ng/ml				Mean S.D. %S.D.	450958 10675 2.4%		Mean S.D. %S.D.	695600 22615 3.3%	

(A) Exluded on te basis of data quality objectives; see text.

BACK TO MAIN

Pooled N-EtFOSE Alcohol Data and Slope Regression



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.640148735
R Square	0.409790403
Adjusted R Sq	0.396064598
Standard Erro	0.049834563
Observations	45

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	0.074145584	0.074145584	29.85547405	2.18808E-06
Residual	43	0.1067898	0.002483484		
Total	44	0.180935383			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-0.023015945	0.013609243	-1.691199513	0.098035439	-0.050461581	0.00442969	-0.050461581	0.00442969
X Variable 1	-0.002617852	0.000479108	-5.464016293	2.18808E-06	-0.003584064	-0.00165164	-0.003584064	-0.00165164

% 2σ Slope Uncertainty
37%

Appendix D: Selected Chromatograms

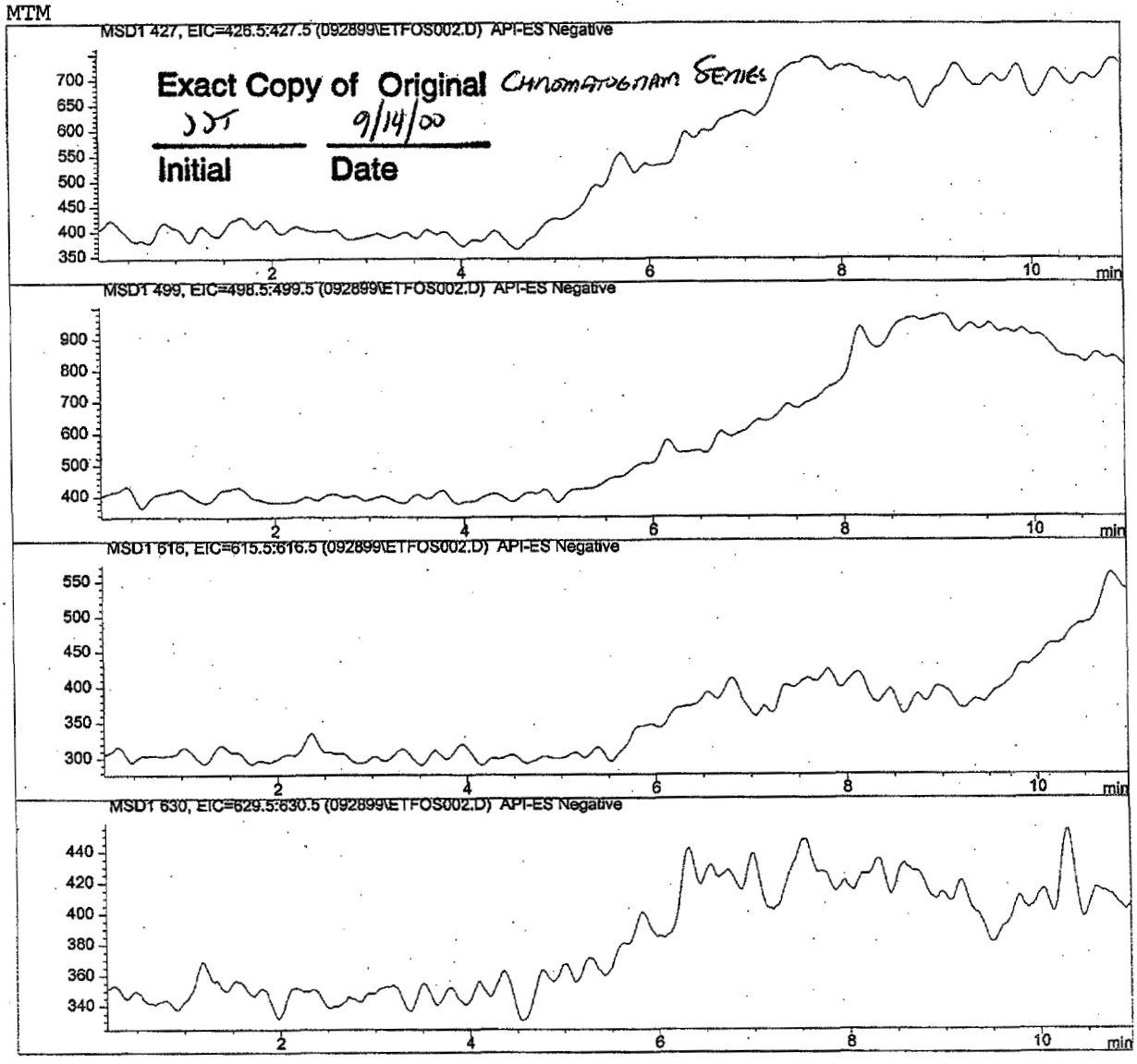
A representative set of chromatograms from the present study is included in this Appendix.

Batch Run # 2 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS002.D Sample Name: MeOH Blank

==========

Injection Date : 9/28/99 3:10:02 PM Seq. Line : 2
Sample Name : MeOH Blank Vial : 91
Acq. Operator : MTM Inj : 1
Acq. Instrument : Instrument 1
Acq. Method : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed : 9/5/00 8:28:58 AM by kej
(Results are from a previously saved Batch)
SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using 4mmx35mm Dionex IonPac NG1 column, S/N 12879.

MTM



Batch Run # 2 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS002.D Sample Name: MeOH Blank

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

DDT 9/14/20

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.940	1		-	-	-		THPFOS
6.221	2		-	-	-		PFOS
8.355	3		-	-	-		MeFOSE-OH
8.586	4		-	-	-		EtFOSE-OH

Totals without ISTD(s) : 0.00000

1 Warnings or Errors :

Warning : ISTD compound(s) not found

Area Percent Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000

Peak #	RetTime [min]	Sig	Type	Area	Area %	Name
1	5.940	1	I	0.00000	0.0000	THPFOS
2	6.221	2		0.00000	0.0000	PFOS
3	8.355	3	I	0.00000	0.0000	MeFOSE-OH
4	8.586	4		0.00000	0.0000	EtFOSE-OH

Totals : 0.00000

1 Warnings or Errors :

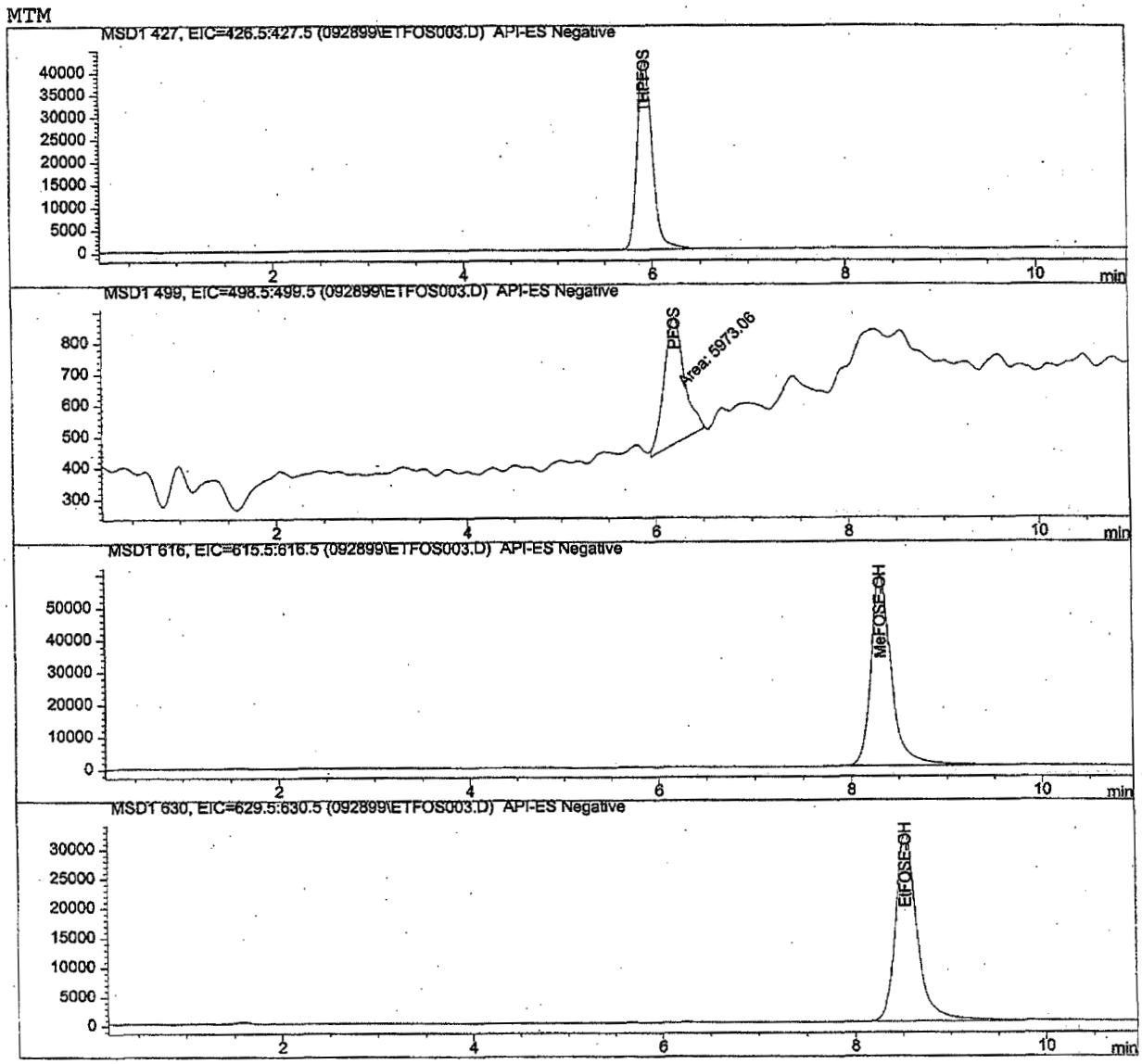
Batch Run # 3 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS003.D Sample Name: L1 1.5, 155.40

Injection Date : 9/28/99 3:28:12 PM Seq. Line : 3
Sample Name : L1 1.5, 155.40 Vial : 1
Acq. Operator : MTM Inj : 1
Acq. Instrument : Instrument 1
Acq. Method : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed : 9/5/00 8:28:58 AM by kej
(Results are from a previously saved Batch)

DST 9/14/00

SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using 4mmx35mm Dionex IonPac NG1 column, S/N 12879.

MTM



Batch Run # 3 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS003.D Sample Name: L1 1.5, 155.40

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000

Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

JJS 9/14/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type		Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.925	1	PB	I	4.65165e5	1.00000	402.60000		THPFOS
6.207	2	MM		5973.06250	3.07266e-1	1.58847		PFOS
8.313	3	PB	I	8.80788e5	1.00000	315.60000		MeFOSE-OH
8.538	4	PB		5.05275e5	8.83244e-1	159.90937		EtFOSE-OH

Totals without ISTD(s) : 161.49784

*** End of Report ***

Batch Run # 4 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS004.D Sample Name: L2 1.5, 310.80

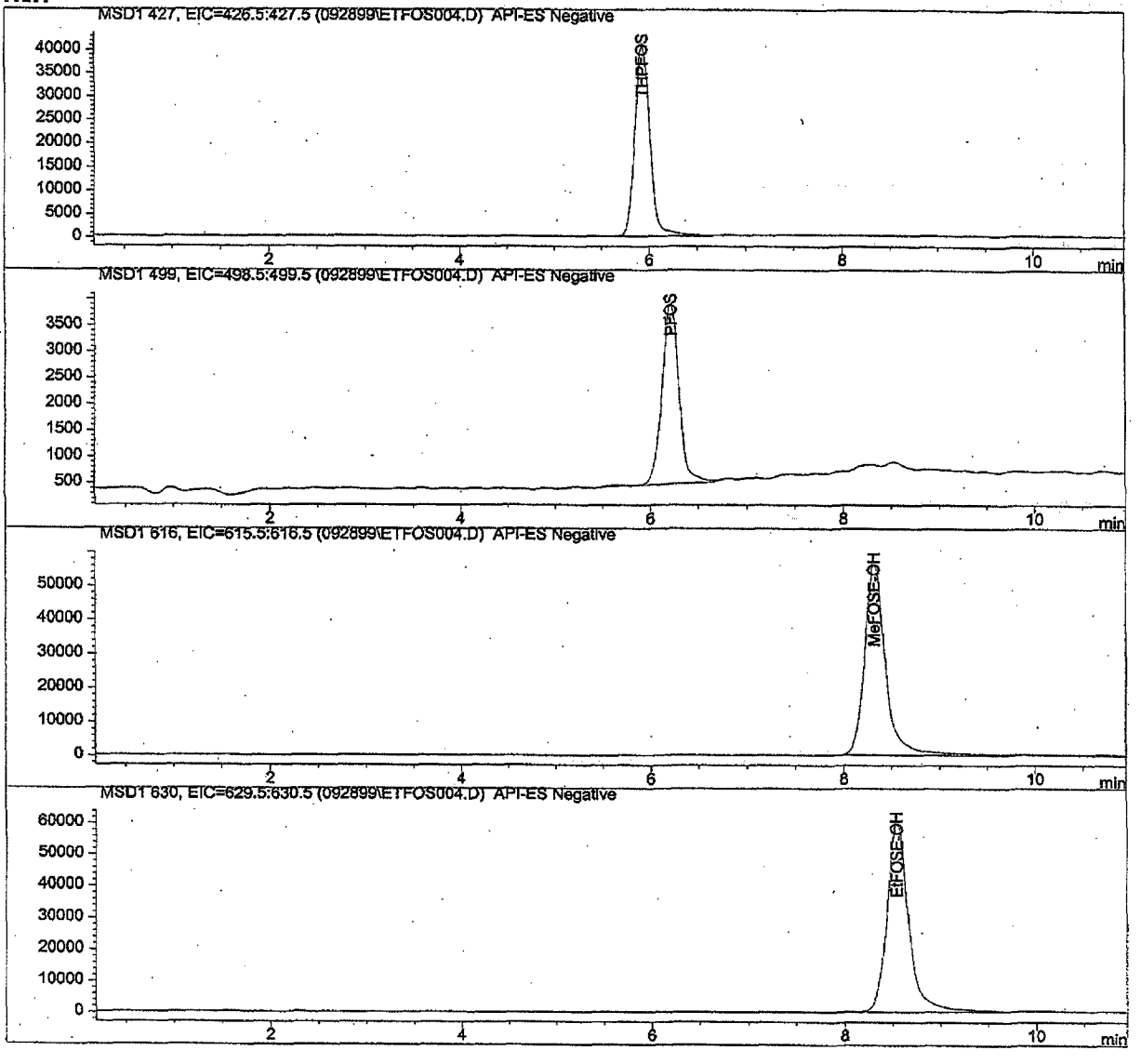
==========

Injection Date	: 9/28/99 3:46:24 PM	Seq. Line	: 4
Sample Name	: L2 1.5, 310.80	Vial	: 2
Acq. Operator	: MTM	Inj	: 1
Acq. Instrument	: Instrument 1		
Acq. Method	: C:\HPCHEM\1\METHODS\FOSESIM.M		
Last changed	: 9/28/99 2:49:57 PM by MTM		
Analysis Method	: C:\HPCHEM\1\METHODS\0824_1IS.M		
Last changed	: 9/5/00 8:28:58 AM by kej		

DDT 9/14/00

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

MTM



Batch Run # 4 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS004.D Sample Name: L2 1.5, 310.80

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

JJF 9/14/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type		Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.927	1	BV	I	4.56303e5	1.00000	402.60000		THPFOS
6.218	2	PB		4.24807e4	3.52850e-1	13.22520		PFOS
8.320	3	BB	I	8.73131e5	1.00000	315.60000		MeFOSE-OH
8.545	4	PB		9.57739e5	9.01077e-1	311.93664		EtFOSE-OH

Totals without ISTD(s) : 325.16184

*** End of Report ***

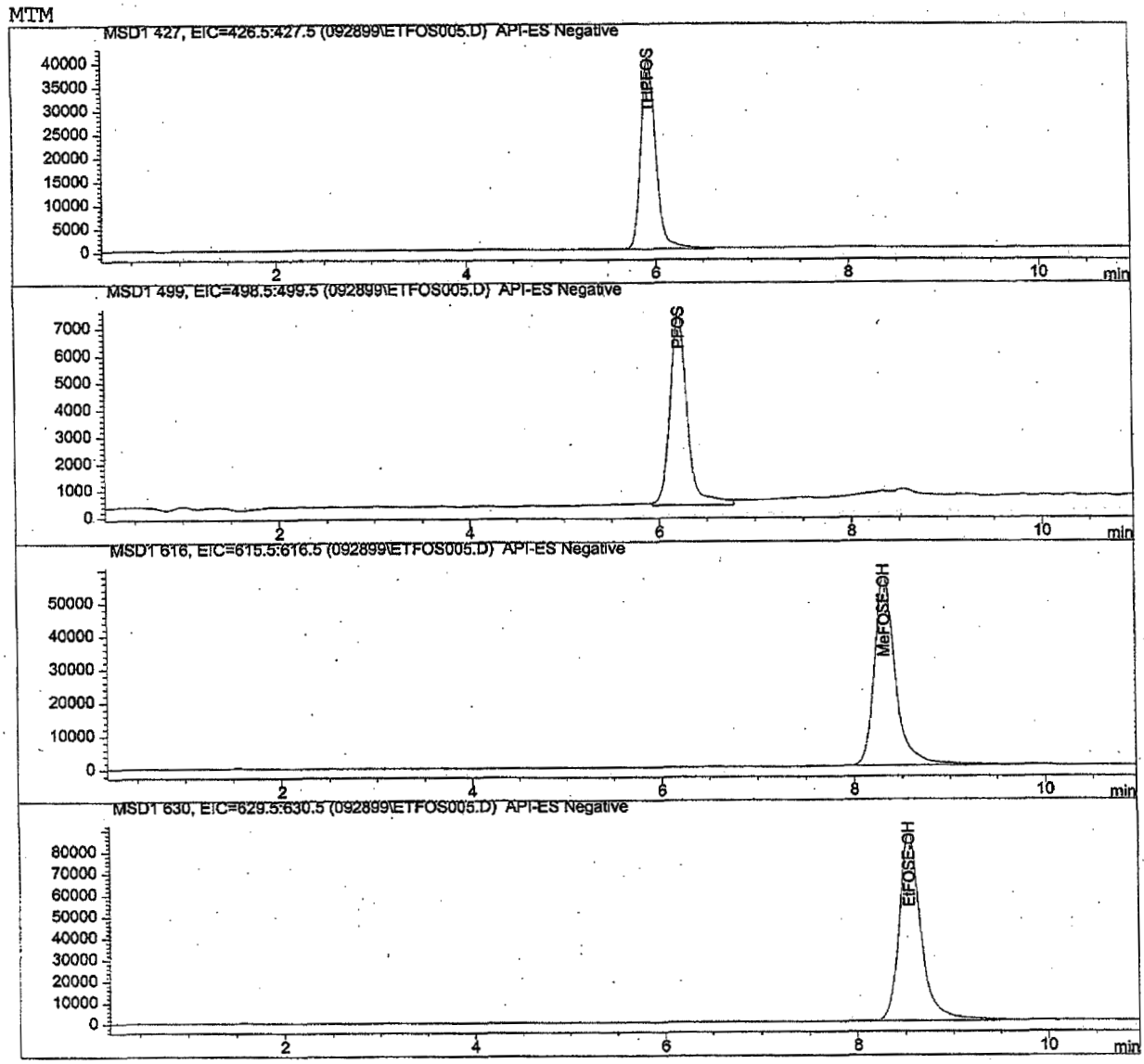
Batch Run # 5 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS005.D Sample Name: L3 1.5, 466.20

Injection Date : 9/28/99 4:04:35 PM Seq. Line : 5
Sample Name : L3 1.5, 466.20 Vial : 3
Acq. Operator : MTM Inj : 1
Acq. Instrument : Instrument 1
Acq. Method : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed : 9/5/00 8:28:58 AM by kej
(Results are from a previously saved Batch)

JJT 9/14/00

SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using 4mmx35mm Dionex IonPac NG1 column, S/N 12879.

MTM



Batch Run # 5 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS005.D Sample Name: L3 1.5, 466.20

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

DDT 9/24/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type		Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.928	1	BB	I	4.47867e5	1.00000	402.60000		THPFOS
6.217	2	BB		8.87226e4	3.56716e-1	28.44993		PFOS
8.322	3	PB	I	8.68287e5	1.00000	315.60000		MeFOSE-OH
8.546	4	PV		1.39693e6	9.07298e-1	460.67730		EtFOSE-OH

Totals without ISTD(s) : 489.12723

*** End of Report ***

Batch Run # 6 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS006.D Sample Name: L4 1.5, 621.60

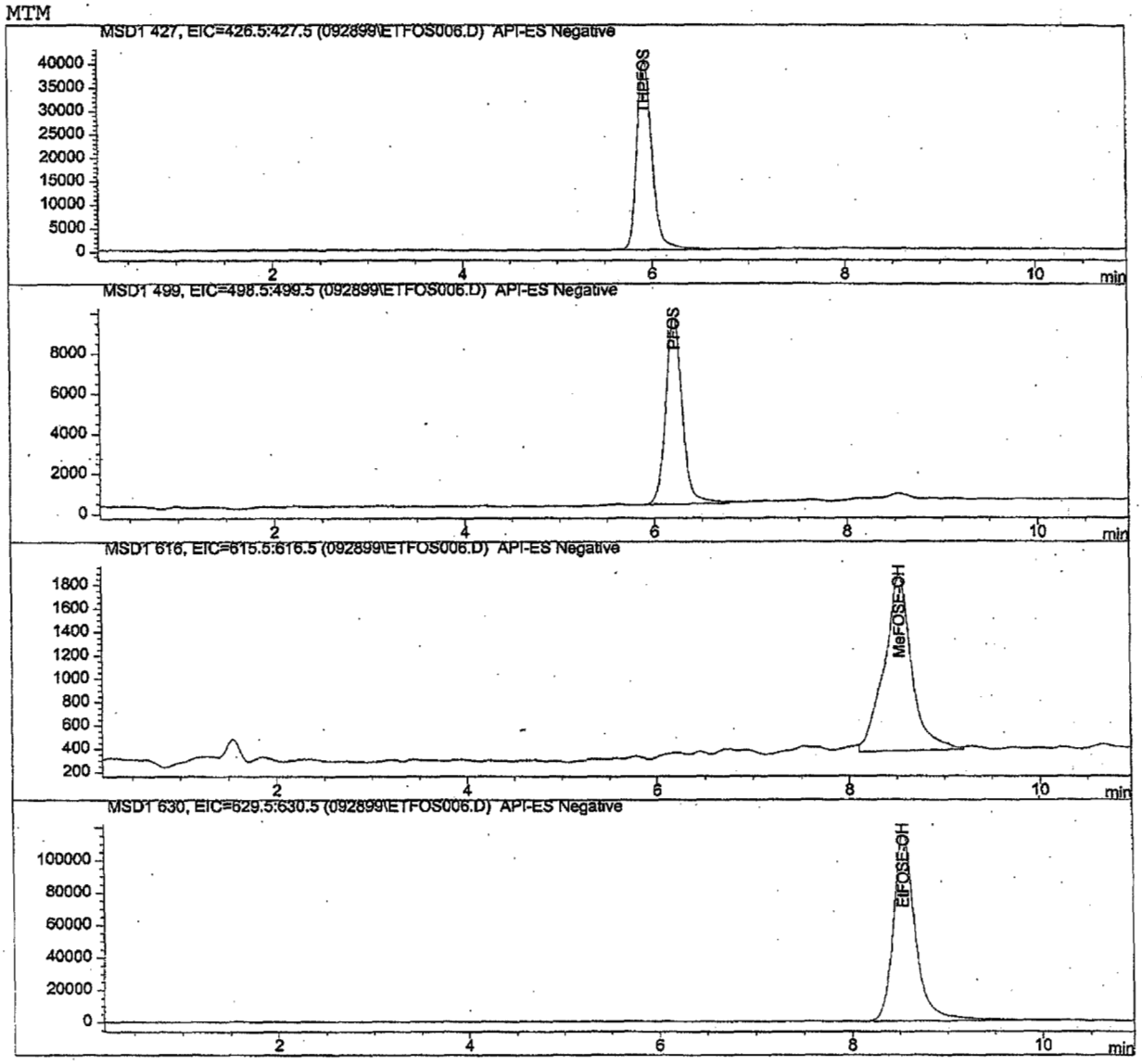
==========

Injection Date : 9/28/99 4:22:47 PM Seq. Line : 6
Sample Name : L4 1.5, 621.60 Vial : 4
Acq. Operator : MTM Inj : 1
Acq. Instrument : Instrument 1
Acq. Method : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed : 9/5/00 8:28:58 AM by kej
(Results are from a previously saved Batch)

JJK 9/14/00

SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using 4mmx35mm Dionex IonPac NG1 column, S/N 12879.

MTM



Batch Run # 6 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS006.D Sample Name: L4 1.5, 621.60

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

JDF 9/14/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type		Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.926	1	BB	I	4.51811e5	1.00000	402.60000		THPFOS
6.215	2	VB		1.12072e5	3.57406e-1	35.69254		PFOS
8.534	3	BB	I	3.00592e4	1.00000	315.60000		MeFOSE-OH
8.546	4	PP		1.86971e6	9.20283e-1	1.80657e4		EtFOSE-OH

Totals without ISTD(s) : 1.81014e4

*** End of Report ***

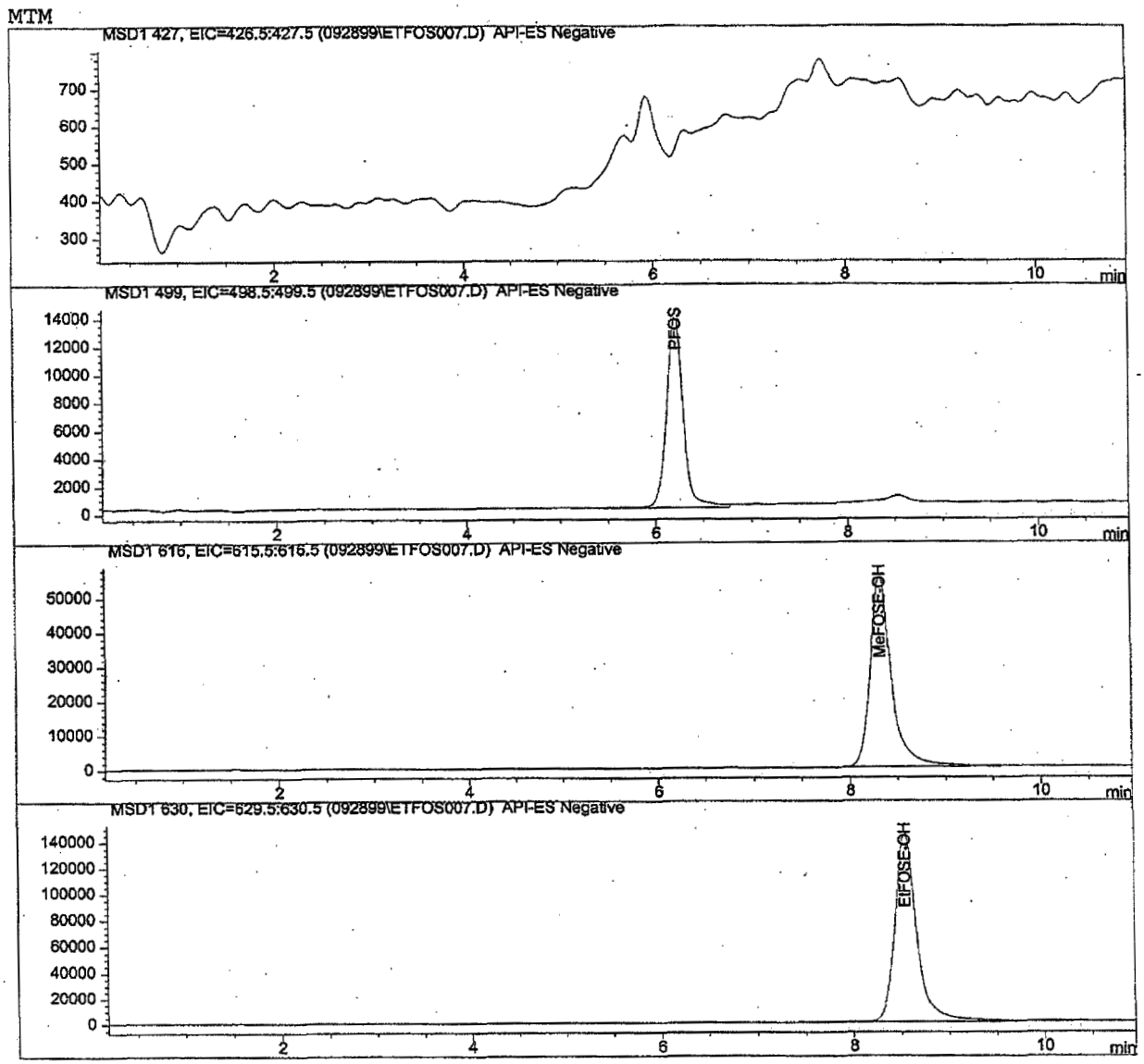
Batch Run # 7 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS007.D Sample Name: L5 1.5, 777.00

==========

Injection Date : 9/28/99 4:41:01 PM Seq. Line : 7
Sample Name : L5 1.5, 777.00 Vial : 5
Acq. Operator : MTM Inj : 1
Acq. Instrument : Instrument 1
Acq. Method : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed : 9/5/00 8:28:58 AM by kej
(Results are from a previously saved Batch)

SDT 9/14/00

SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using 4mmx35mm Dionex IonPac NG1 column, S/N 12879.



Batch Run # 7 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS007.D Sample Name: L5 1.5, 777.00

==========

Internal Standard Report

==========

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

JJT 9/14/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type		Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.940	1			-	-	-		THPFOS
6.213	2	BB		1.67565e5	0.00000	0.00000		PFOS
8.318	3	PB	I	8.56724e5	1.00000	315.60000		MeFOSE-OH
8.544	4	PB		2.34711e6	9.12800e-1	789.23438		EtFOSE-OH

Totals without ISTD(s) : 789.23438

2 Warnings or Errors :

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

==========

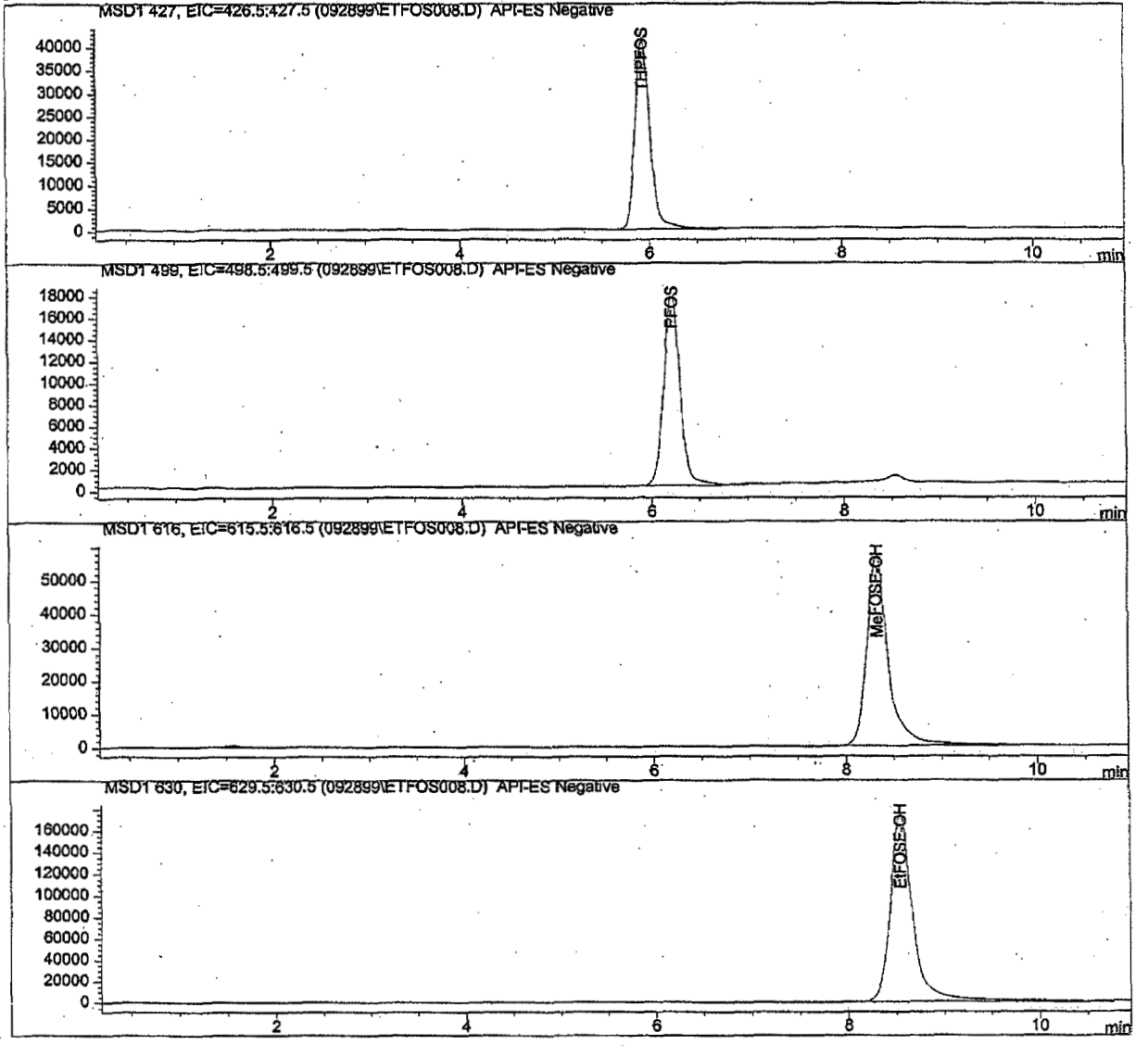
*** End of Report ***

Batch Run # 8 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS008.D Sample Name: L6 1.5, 932.40

Injection Date : 9/28/99 4:59:12 PM Seq. Line : 8
Sample Name : L6 1.5, 932.40 Vial : 6
Acq. Operator : MTM Inj : 1
Acq. Instrument : Instrument 1
Acq. Method : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed : 9/5/00 8:28:58 AM by kej
(Results are from a previously saved Batch)
SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using 4mmx35mm Dionex IonPac NG1 column, S/N 12879.

JJY 9/14/00

MTM



Batch Run # 8 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS008.D Sample Name: L6 1.5, 932.40

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

JJK 9/14/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type		Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.927	1	BB	I	4.60976e5	1.00000	402.60000		THPFOS
6.215	2	VB		2.10524e5	3.58656e-1	65.94394		PFOS
8.323	3	PB	I	8.87024e5	1.00000	315.60000		MeFOSE-OH
8.546	4	BV		2.91155e6	9.14094e-1	946.92850		EtFOSE-OH

Totals without ISTD(s) : 1012.87244

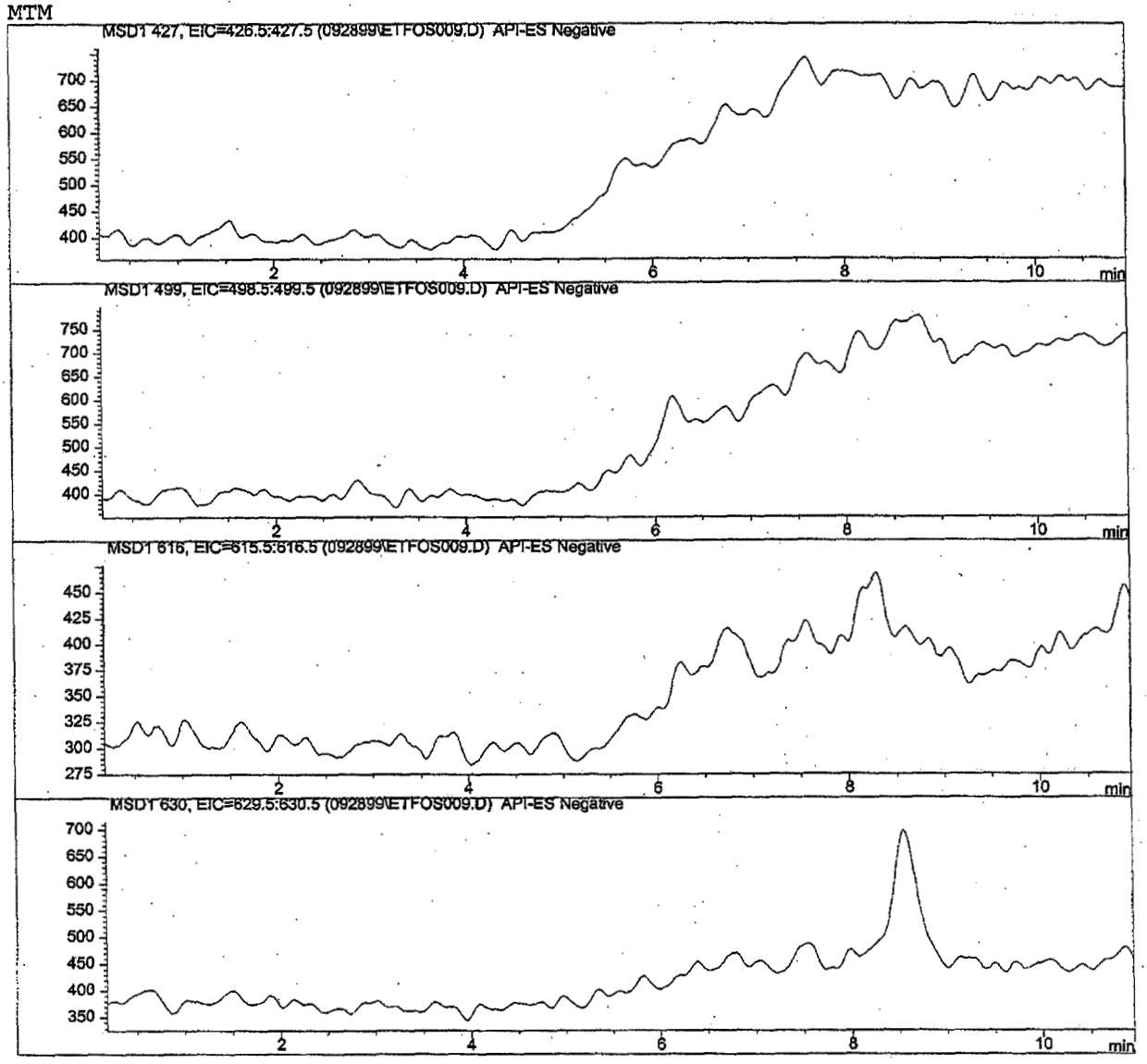
*** End of Report ***

Batch Run # 9 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS009.D Sample Name: MeOH Blank

```
==========================================================================
Injection Date  : 9/28/99 5:17:26 PM          Seq. Line :   9
Sample Name     : MeOH Blank                      Vial :  91
Acq. Operator   : MTM                             Inj :   1
Acq. Instrument : Instrument 1
Acq. Method     : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed    : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed    : 9/5/00 8:28:58 AM by kej
                  (Results are from a previously saved Batch)
SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using
4mmx35mm Dionex IonPac NG1 column, S/N 12879.
```

DJF 9/14/00

MTM



Batch Run # 9 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS009.D Sample Name: MeOH Blank

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

DDT 9/14/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.940	1		-	-	-		THPFOS
6.221	2		-	-	-		PFOS
8.355	3		-	-	-		MeFOSE-OH
8.586	4		-	-	-		EtFOSE-OH

Totals without ISTD(s) : 0.00000

1 Warnings or Errors :

Warning : ISTD compound(s) not found

Area Percent Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000

Peak #	RetTime [min]	Sig	Type	Area	Area %	Name
1	5.940	1	I	0.00000	0.0000	THPFOS
2	6.221	2		0.00000	0.0000	PFOS
3	8.355	3	I	0.00000	0.0000	MeFOSE-OH
4	8.586	4		0.00000	0.0000	EtFOSE-OH

Totals : 0.00000

1 Warnings or Errors :

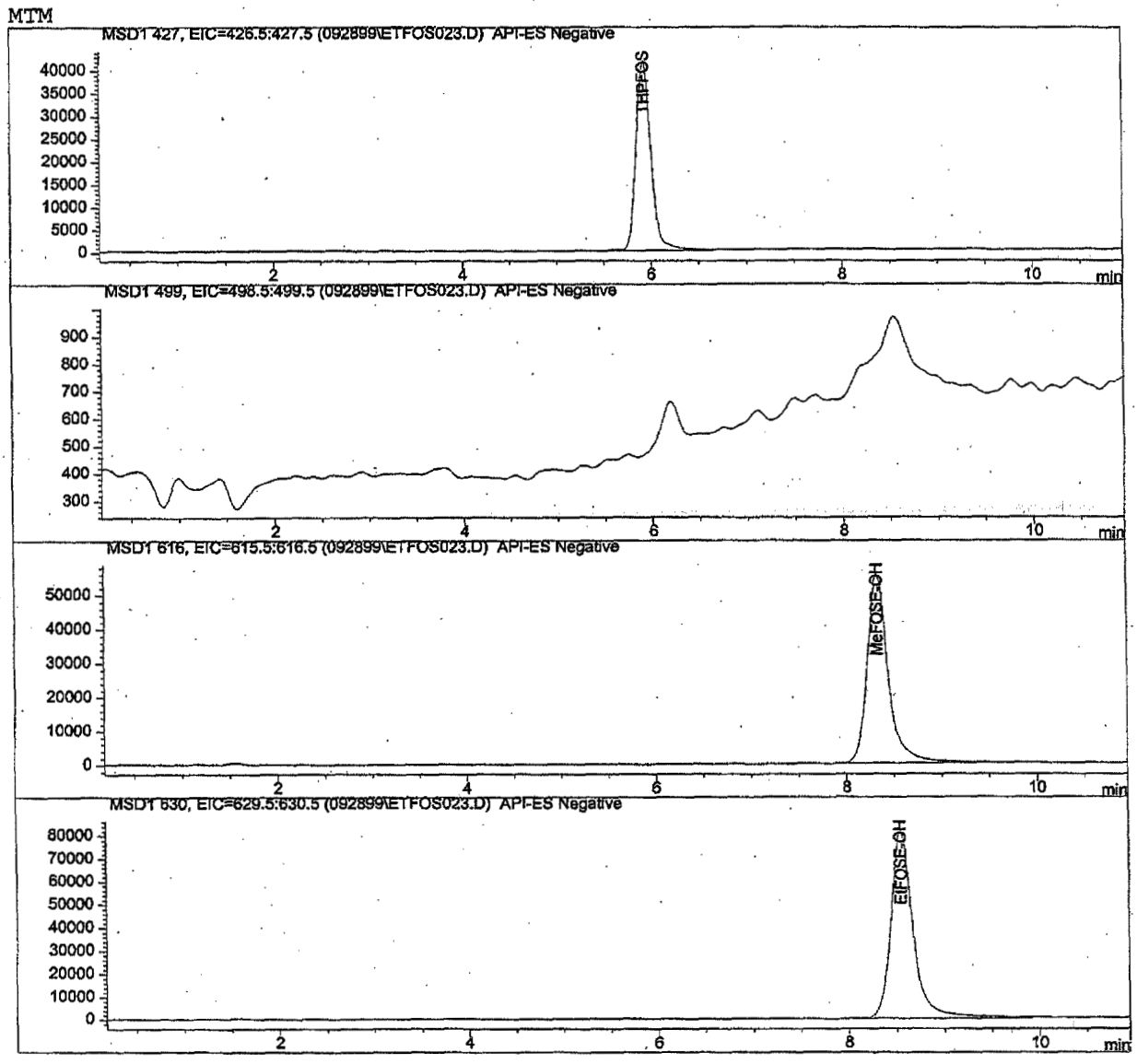
Batch Run # 23 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS023.D Sample Name: EFA-073

==========

Injection Date : 9/28/99 9:32:40 PM Seq. Line : 23
Sample Name : EFA-073 Vial : 23
Acq. Operator : MTM Inj : 1
Acq. Instrument : Instrument 1
Acq. Method : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed : 9/5/00 8:28:58 AM by kej
(Results are from a previously saved Batch)
SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using 4mmx35mm Dionex IonPac NG1 column, S/N 12879.

DJH 9/14/00

MTM



Batch Run # 23 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS023.D Sample Name: EFA-073

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

DDT 9/14/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type		Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.926	1	BB	I	4.64774e5	1.00000	402.60000		THPFOS
6.221	2			-	-	-		PFOS
8.326	3	VB	I	8.48397e5	1.00000	315.60000		MeFOSE-OH
8.550	4	BB		1.33227e6	9.06971e-1	449.49185		EtFOSE-OH

Totals without ISTD(s) : 449.49185

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

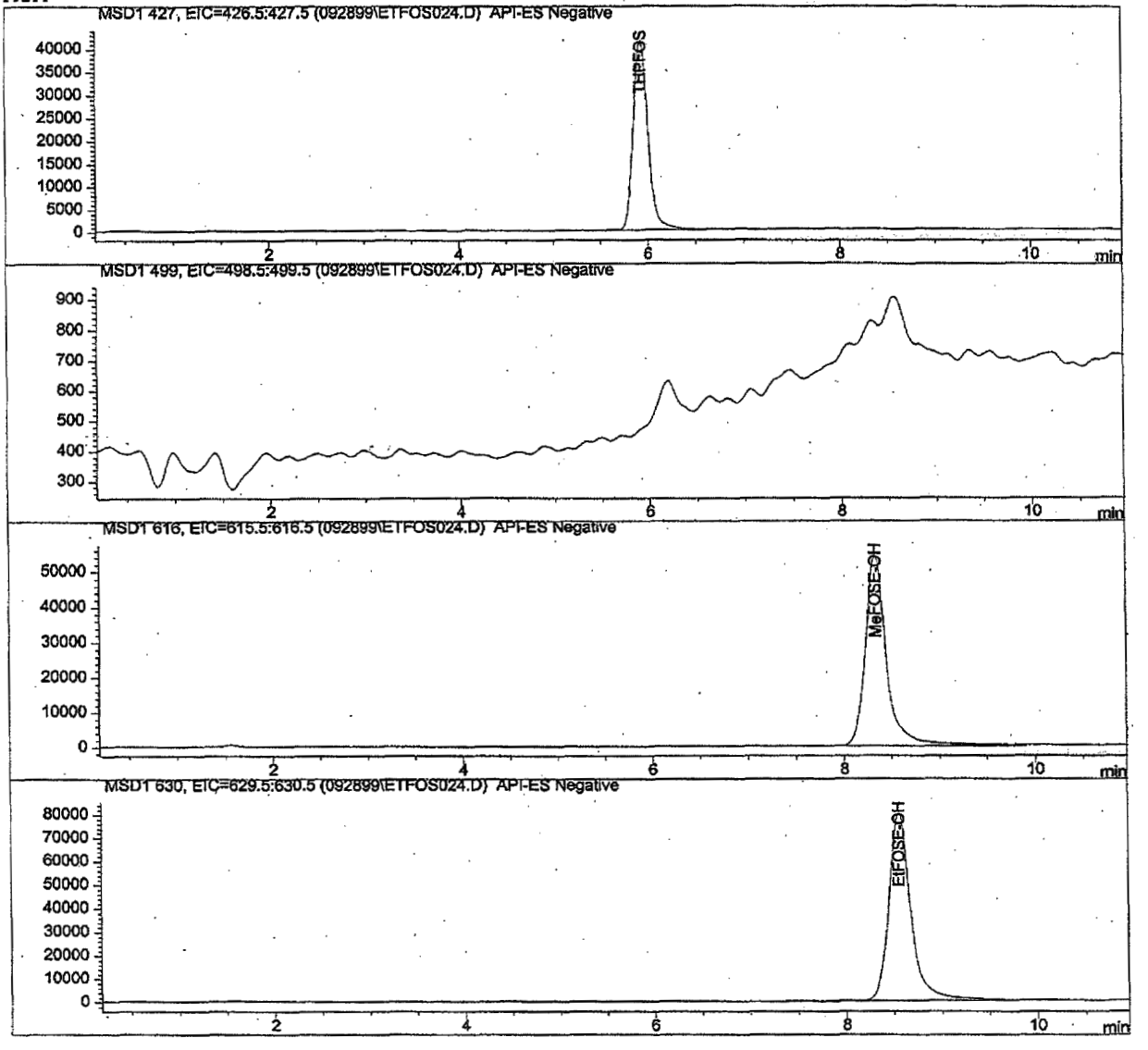
*** End of Report ***

Batch Run # 24 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS024.D Sample Name: EFA-074

```
==========================================================================
Injection Date  : 9/28/99 9:50:54 PM          Seq. Line :  24
Sample Name     : EFA-074                        Vial :  24
Acq. Operator   : MTM                             Inj :   1
Acq. Instrument : Instrument 1
Acq. Method     : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed    : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed    : 9/5/00 8:28:58 AM by kej
                  (Results are from a previously saved Batch)
SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using
4mmx35mm Dionex IonPac NG1 column, S/N 12879.
```

SDT 9/14/00

MTM



Batch Run # 24 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS024.D Sample Name: EFA-074

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

DST 9/14/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type	Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.925	1	BB I	4.56350e5	1.00000	402.60000		THPFOS
6.221	2		-	-	-		PFOS
8.329	3	BB I	8.55992e5	1.00000	315.60000		MeFOSE-OH
8.555	4	PB	1.28265e6	9.06316e-1	428.60235		EtFOSE-OH

Totals without ISTD(s) : 428.60235

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

*** End of Report ***

Batch Run # 25 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS025.D Sample Name: EFA-075

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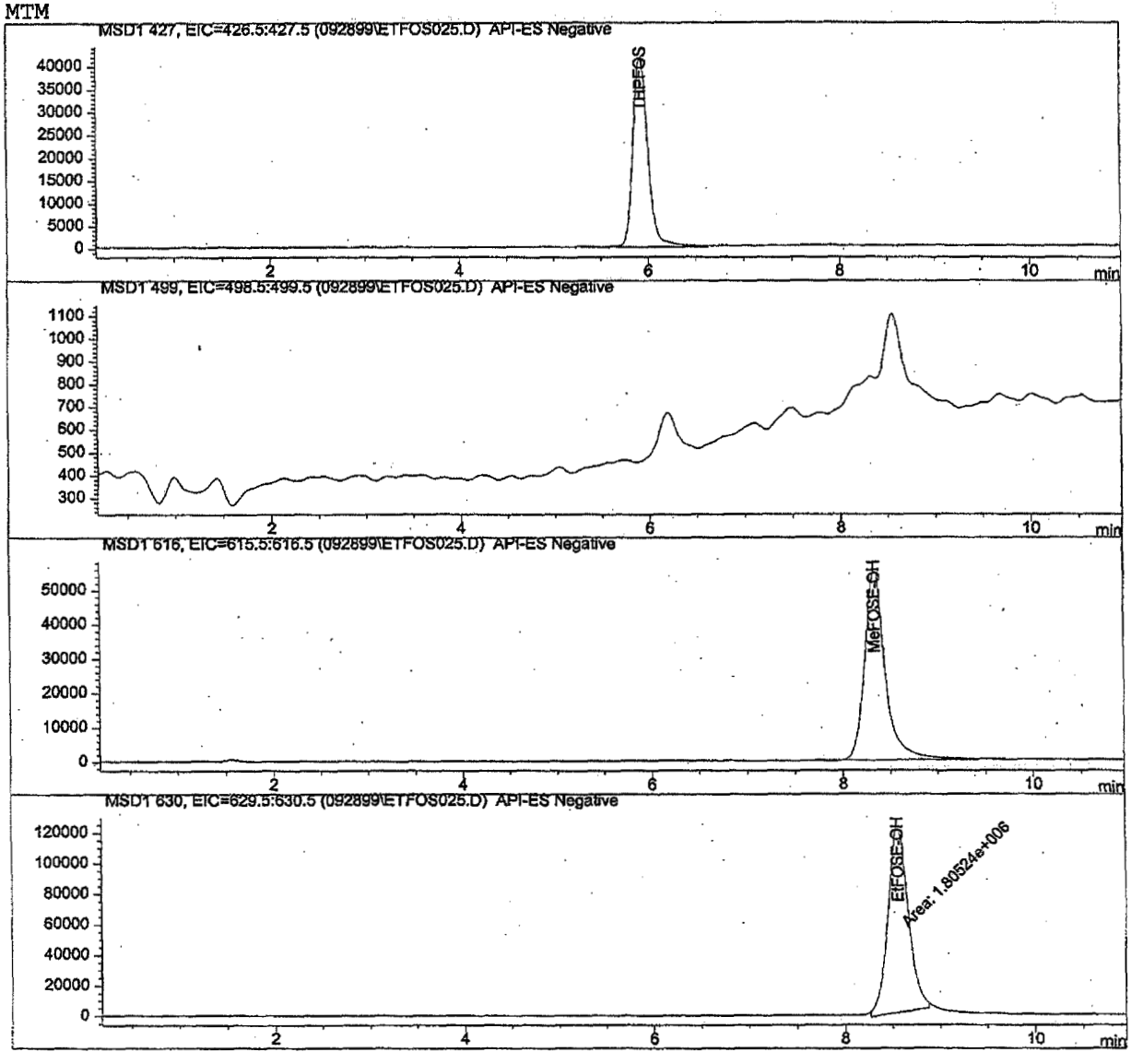
Injection Date : 9/28/99 10:09:06 PM Seq. Line : 25
Sample Name : EFA-075 Vial : 25
Acq. Operator : MTM Inj : 1
Acq. Instrument : Instrument 1
Acq. Method : C:\HPCHEM\1\METHODS\FOSESIM.M
Last changed : 9/28/99 2:49:57 PM by MTM
Analysis Method : C:\HPCHEM\1\METHODS\0824_1IS.M
Last changed : 9/5/00 8:28:58 AM by kej
(Results are from a previously saved Batch)

SIM Analysis (ES-) for Et-FOSE-OH, MeFOSE-OH, THPFOS, and PFOS using 4mmx35mm Dionex IonPac NG1 column, S/N 12879.

DJS 9/14/20

MTM



Batch Run # 25 of 56
Data File C:\HPCHEM\1\DATA\092899\EtFOS025.D Sample Name: EFA-075

Internal Standard Report

Sorted By : Retention Time
Calib. Data Modified : Tuesday, September 05, 2000 8:28:56 AM
Multiplier : 1.0000
Dilution : 1.0000
Sample ISTD Information:

ISTD #	ISTD Amount ppb (ng/ml	Name
1	402.60000	THPFOS
2	315.60000	MeFOSE-OH

JDT 9/14/00

Signal 1: MSD1 427, EIC=426.5:427.5
Signal 2: MSD1 499, EIC=498.5:499.5
Signal 3: MSD1 616, EIC=615.5:616.5
Signal 4: MSD1 630, EIC=629.5:630.5

RetTime [min]	Sig	Type		Area	Amt/Area ratio	Amount ppb (ng/ml	Grp	Name
5.923	1	BB	I	4.64731e5	1.00000	402.60000		THPFOS
6.221	2			-	-	-		PFOS
8.332	3	PB	I	8.46072e5	1.00000	315.60000		MeFOSE-OH
8.559	4	MM		1.80524e6	9.10577e-1	613.16914		EtFOSE-OH

Totals without ISTD(s) : 613.16914

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

*** End of Report ***