

Study Title

Hydrolysis Reactions of Perfluorooctanoic Acid (PFOA)

Data Requirement:

Based on OPPTS: 835.2110

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Study Completion Date

March 30, 2001

Performing Laboratory

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Project Identification

3M Laboratory Report No: E00-1851

Total Number of Pages

99

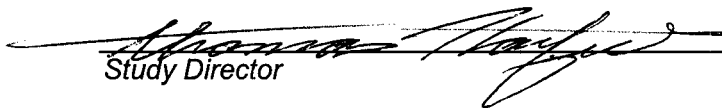
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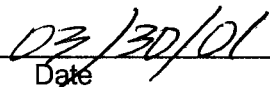
Statement of Non-Compliance

Study Title: Hydrolysis Reactions of Perfluorooctanoic Acid (PFOA)

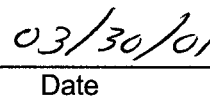
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This study does not fully comply with the requirements of the US EPA Good Laboratory Practices (GLP) Standards at 40 CFR Part 792 (TSCA). However, many GLP standards were used in the development of the analytical method (Appendix A), and many of the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


Study Director


Date


Sponsor Representative


Date

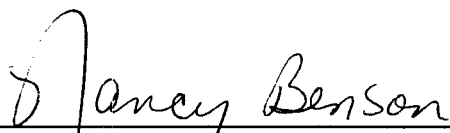
Quality Assurance Statement

Study Title: Hydrolysis Reactions of Perfluorooctanoic Acid (PFOA)

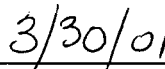
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The following table provides details of the audits performed by the 3M Environmental Laboratory Quality Assurance Unit (QAU).

Inspection Dates	Phase	Date Reported to	
		Management	Study Director
3/2/01	Data and Draft Report	3/2/01	3/2/01
3/26-30/01	Data and Draft Report	3/30/01	3/30/01



QAU Representative



Date

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Location of Archives

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

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Summary

We report here the results of our study of the hydrolysis of perfluorooctanoic acid (hereafter, PFOA). Our methods are described below and in Appendix A to this work; our results are based on the observed concentrations of PFOA in buffered aqueous solutions as a function of time. 3M's Environmental Laboratory staff developed the study procedures; they are based on EPA's OPPTS Guideline Document 835.2110¹ but do not fulfill all the requirements of the guideline.

Our methods are described below and in Appendix A to this work; our results are based on the observed concentrations of PFOA in buffered aqueous solutions as a function of time. The chosen analytical technique was high performance liquid chromatography with mass spectrometry detection (HPLC/MS). Table 1 summarizes 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 109 days, and our results indicate no dependence of the degradation rate of PFOA on the sample pH level. Our results based on the PFOA concentrations, pooled over three pH of the six levels (5.0, 7.0, and 9.0), are presented in Tables 1 and 2.

Table 1. Summary of Results Based on PFOA 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) Minimum Half Life at 25° C (years)
8.1×10^{-5}	8.1×10^{-6}	235	92

The mean value and precision of PFOA concentration measurements provide a second estimate of the PFOA half-life, presented in Table 2.

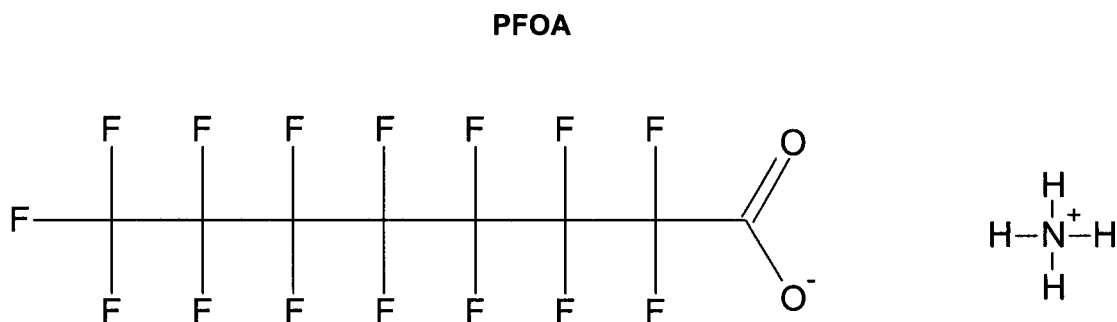
Table 2. Summary of Results Based on the Mean and Precision of PFOA Measurements		
Maximum Possible Rate at 50° C (day⁻¹)	Maximum Calculated Rate at 25° C (day⁻¹)	Calculated Half Life at 25° C (years)
2.0×10^{-4}	2.0×10^{-5}	≥ 97

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 PFOA (or, more generally, its degradation in the presence of H₂O) is addressed in this report. The structure of the ammonium salt of the PFOA is illustrated in Figure 1. This is the actual test material used in the study.

Figure 1. Structure of the Ammonium Salt of PFOA



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 PFOA.

The first estimate (see Table 1) is based on the observed degradation of the “parent” compound PFOA in dilute, appropriately buffered aqueous solutions. Equation 1 describes the estimated half-life $(\hat{T}_p^{1/2})_1$ in terms of the estimated total parent hydrolysis rate $\hat{k}_p$ (see Appendix B, Equation B10):

$$(\hat{T}_p^{1/2})_1 = \frac{\ln(2)}{\hat{k}_p} \quad \text{Eq. 1}$$

We determined the quantity $\hat{k}_p$ from the experimental data as described in Appendix B. To determine the relevant concentration ratios at each pH level (see Equations B8 and B9), we used either the data corresponding to “Day 0” ($t = 0$) or the earliest available data achieving the data quality objectives of the analytical method.

A second half-life estimate (see Equation B37) is available from the mean μ and standard deviation σ of the observed PFOA concentrations, assuming that they were essentially constant over the experimental portion of the study. This estimate is

$$(\hat{T}_p^{1/2})_2 \geq (\hat{T}_p^{1/2})_{\min} = \frac{\ln(2)}{(k_p)_{\max}} = \frac{\mu \Delta t \ln(2)}{2\sigma}, \quad \text{Eq. 2}$$

where Δt represents the sample incubation period.

All the samples used in this study were maintained at a reaction temperature of 50° ($\pm 3^\circ$) C. The quoted results, valid for the reaction temperature 25° C, were estimated from our experimental results according to methods described in Appendix B (Eqs. 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-8-212.0, "Preparation of Perfluorooctanoic Acid (PFOA) Hydrolysis Samples in Buffered Solutions 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 June 15 and November 12, 2000.

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 the related calculations involve only ratios of the compound concentrations (see Appendix B, Equations B8 and B36), the resulting rate and half-life estimates are largely independent of the material purity levels.

Table 3. Characterizations of Test and Reference Substances		
	PFOA (Ammonium Salt)	PFBS (Ammonium salt)
Source	3M ICP/PCP Division	3M Specialty Chemicals
Chemical Lot Number ^a	TCR-00930-30, Lot 332	TCR-99030-28, Lot 101
Physical Description	White powder	White powder
Molecular Weight (gm mole ⁻¹)	431	317

^aperfluorobutanesulfonate

Sample Preparation

We prepared four 5.0-mL aqueous buffer samples (a sample, a duplicate, a triplicate, 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, 28, 42, 64, 84 and 109 days). Buffered solutions containing 5154 ng/mL of the analyte PFOA 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 incubator/orbital shaker maintained at 50° (± 3°) C. One of the "Day 0" samples and a blank were spiked with the PFOA solution, diluted 7:1 with methanol containing the internal standard perfluorobutanesulfonate (PFBS) and frozen. The resulting PFBS concentration in all these samples was 158 ng/ml; the resulting PFOA concentration in the spiked samples was 143 ng/ml above the original sample concentration. After the appropriate incubation times, subsets of the sample vials were removed from the incubator and then spiked (as required), diluted, and frozen as

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

Nine calibration standards containing PFOA (25 to 999 ng/ml) and PFBS served as the quantitative basis of the study. All these standards were prepared in 1.5 mL of the appropriate pH buffer solution and 8.5 mL of methanol.

Sample Analysis

The equipment we used for the HPLC/MS analysis was a Hewlett Packard model 1100 equipped with a Dionex IonPac® NG-1 HPLC column (aqueous ammonium acetate/methanol solvent gradient) and an ALS Model G1322A degassing module. An ALS Model G1316A 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 10 µL sample injections. The detector was a Hewlett Packard MSD mass spectrometer, operated in negative-mode electrospray ionization mode; the anions of PFBS and PFOA were detected at the mass-to-charge (m/z) ratios 299 and 413, respectively. We processed the resulting data using the computer program *Target® NT Genie Integrator*. 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. All calibration and sample results that failed to meet the data quality objectives of the method (and were excluded from further analysis) are noted in the following sections.

Results and Discussion

Data Quality Objectives (DQO's)

We briefly describe the data quality objectives applied in this study below. Appendix A describes them in greater detail. 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 $\pm 25\%$ of the linear fit value; data outside this range are excluded and the linear fit is recalculated. Data for the high or low calibration standards may be rejected, though this results in a smaller effective calibration range. The average results of calibrations performed before and after the analytical procedures are used to calculate the analyte concentrations.
- **Continuing Calibration Verification (CCV).** Selected calibration samples are examined at the beginning of, during, and at the end of each analytical procedure. The results may not deviate by more than $\pm 25\%$ of the known values.
- **Sample Spikes.** The acceptable percent spike recovery range is 70% to 130%; recoveries outside this range % place the analysis out of control, and require intervention by the Team Leader or designee. Analyte specificity is demonstrated by acceptable analyte spike recoveries.
- **Identically Prepared Samples.** Triplicate sample results with relative standard deviations (RSDs) greater than 25% place the analysis out of control, and require intervention by the Team Leader or designee.
- **Solvent Blanks.** Concentration results for solvent blanks must 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

With the following exceptions, our analytical results met or exceeded the data quality objectives of Appendix A.

- **Spike Recoveries.** Results for five of the eight sample triplicates for pH = 3.0 failed to meet this data quality objective. Least squares determinations of a slope and offset from the three remaining data points at this pH would be highly undetermined, so we have excluded all the pH = 3.0 data from further consideration. Similarly, results for seven of the eight sample triplicates for pH = 11.0 failed to meet this data quality objective. No least squares determinations of a slope are possible in this case, so we have excluded all the pH = 11.0 data from further consideration. Finally, the "Day 14" samples at pH = 1.5 (061500-PFOA-075 through 061500-PFOA-078) failed to meet this spike recovery data quality objective, and we have excluded these data from the following rate and half-life estimates.

Statistical Methods and Calculations

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

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

Data Summary and Discussion

The LOQ is defined as the concentration of the lowest (accepted) standard in the calibration set for which the known concentration exceeds 400% of the indicated solvent blank level (see Appendix A). During this study, the LOQ for PFOA was 25 ng/mL.

Results for the internal standard (PFBS) were very consistent throughout the study. The percent relative standard deviations of the measured values, calculated for each of the four pH levels discussed here (1.5, 5.0, 7.0, and 9.0), ranged from 1 to 2%.

Figure 2 illustrates the concentration ratios for the four pH levels versus time. Table 4 presents the results of the 50°C rate determinations based on these data.

Figure 2. Observed PFOA Degradation for Various pH levels.

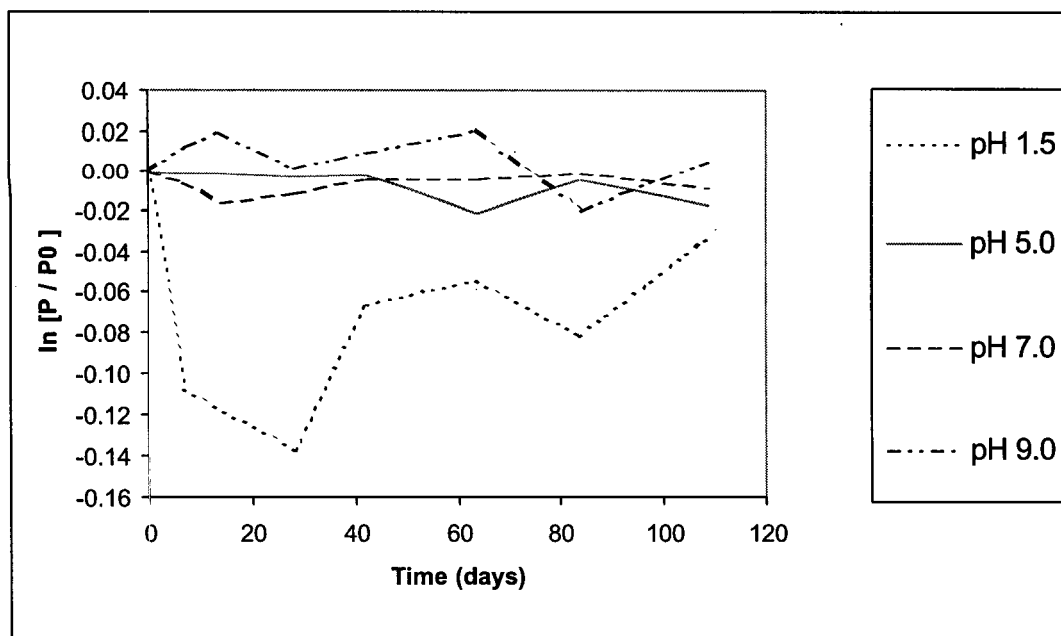
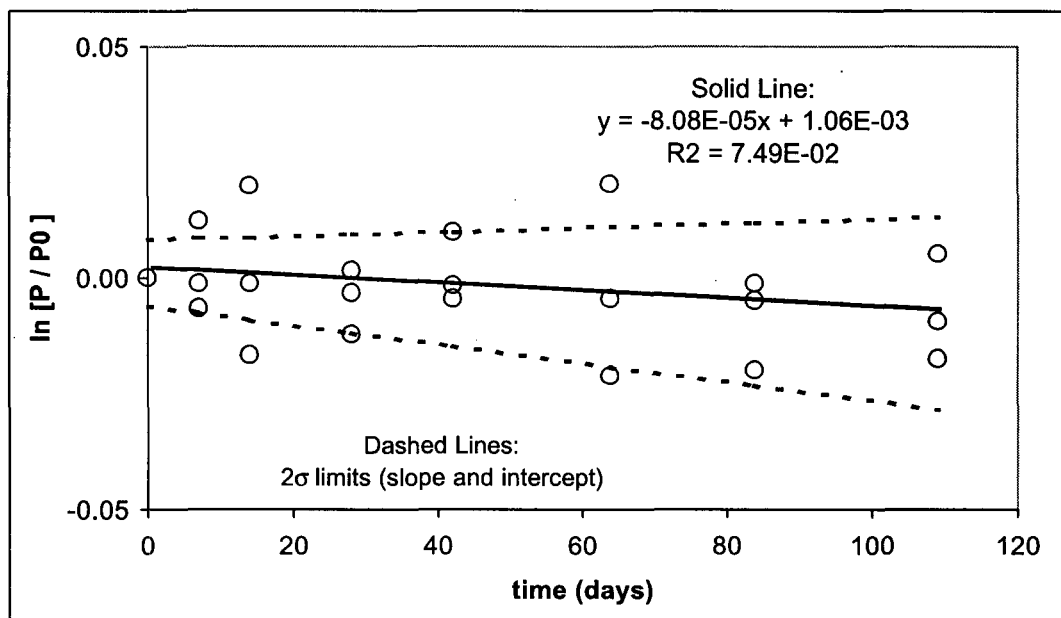


Table 4. Observed (50° C) Degradation Rates of PFOA in Aqueous Buffered Solutions and at Various pH Levels.		
PH	Observed Slope (day⁻¹)	Percent (2σ) Slope Uncertainty (day⁻¹)
1.5	1.7×10^{-4}	597
5.0	-1.5×10^{-4}	76
7.0	1.8×10^{-5}	664
9.0	-1.1×10^{-4}	244

The slopes in Table 4 are small in magnitude, of varying sign, and only poorly determined; their percent relative 2σ (95% confidence) uncertainties range from 76% to 664%. The data do not indicate degradation of PFOA at any of the four pH levels.

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. The mean concentrations (ng/ml) at the pH levels 1.5, 5.0, 7.0, and 9.0 are 464, 653, 649, and 657. The pH = 1.5 data are clearly not equivalent to the data at the other three pH levels; this is an effect of ion pairing at the lowest pH level. Under these circumstances, the only reliable data for a pooled estimate are the data at pH levels 5.0, 7.0, and 9.0. 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 PFOA in Aqueous Buffered Solutions Using Data Pooled Over pH Levels 5.0, 7.0, and 9.0				
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 Minimum at 25° C (years)
8.1×10^{-5}	150	8.1×10^{-6}	235	92

Figure 3. Pooled (pH = 5.0, 7.0, and 9.0) PFOA Data and Slope Regression.

The mean and standard deviation of the PFOA data provide an alternative estimate of its half-life. Details of the related calculations are presented below in Appendix B. The maximum degradation rate is given in Equation 3 (see Equation B36):

$$k_p \leq (k_p)_{\max} = \frac{2\sigma_p}{\mu_p \Delta t} \quad \text{Eq. 3}$$

and the minimum half-life is given in Equation 4 (see Equation B37):

$$T_p^{1/2} \geq (T_p^{1/2})_{\min} = \frac{\ln(2)}{(k_p)_{\max}} = \frac{\mu_p \Delta t \ln(2)}{2\sigma_p} \quad \text{Eq. 4}$$

In both Equations 3 and 4, the mean PFOS concentration (μ_p) and standard deviation (σ_p) can be either molar or mass quantities. Table 6 presents the results of the calculation.

Table 6. Degradation Rate and Half Life of PFOA in Aqueous Buffered Solutions Based on the Concentration Mean and Standard Deviation					
Δt (days)	μ_p (ng/ml)	σ_p (ng/ml)	Maximum Observed Rate at 50° C (day ⁻¹)	Maximum Calculated Rate at 25° C (day ⁻¹)	Calculated Half Life at 25° C (years)
109	653	7.0	2.0×10^{-4}	2.0×10^{-5}	≥ 97

Conclusions

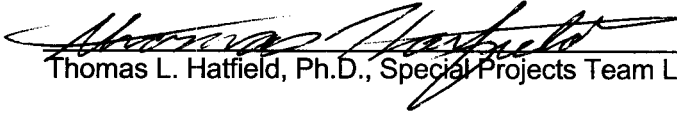
We have performed a study of the aqueous hydrolysis of perfluorooctanoic acid (PFOA) at 50°C and extrapolated the results to 25°C. We included six different pH levels in the study, though data from two of these pH levels (3.0 and 11) failed to meet the data quality objective for matrix spike recovery. We also rejected the data obtained for pH = 1.5 because ion pairing led to artificially low concentrations for all the incubation periods. Our results for the remaining pH levels (5.0, 7.0, and 9.0) indicate no clear dependence of the degradation rate of PFOA on pH. From the data pooled over these three pH levels, we estimate that the hydrolytic half-life of PFOA at 25°C is greater than 92 years, with the most likely value of 235 years. From the mean value and precision of PFOA concentrations, we estimate the hydrolytic half-life of PFOA to be greater than 97 years.

References

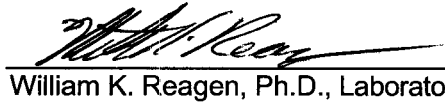
"Fate, Transport and Transformation Test Guidelines: 835.2110: Hydrolysis as a Function of pH," U.S. EPA Office of Prevention, Pesticides and Toxic Substances, publication number 712-C-98-057, January 1998.

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


William K. Reagen, Ph.D., Laboratory Management


Date

Appendix A: Analytical Method

Method Number: ETS-8-212.0, "Preparation of Perfluorooctanoic Acid (PFOA) Hydrolysis Samples in Buffered Solutions 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 PERFLUOROOCTANOIC ACID (PFOA) HYDROLYSIS SAMPLES IN BUFFERED SOLUTIONS AND ANALYSIS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH MASS SPECTROMETRY DETECTION

Method Number: ETS-8-212.0

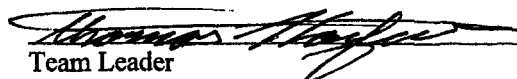
Adoption Date: 3/27/01

Effective Revision Date: NA

Approved By:


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03/27/01
Date


Team Leader

March 27, 01
Date

1.0 SCOPE AND APPLICATION

- 1.1** This method describes how to test for possible hydrolysis reactions of perfluorooctanoic acid (PFOA) in buffered solutions of pHs 1.5, 3.0, 5.0, 7.0, 9.0, and 11.0. The method is based on EPA OPPTS:835.2110 (Reference 18.1). Hydrolysis products are analyzed by high performance liquid chromatography (HPLC) with mass spectrometry (MS) detection and quantitation. PFOA anion is quantified using the anion of perfluorobutanesulfonate (PFBS) as an internal standard. Representative chemical structures are shown in Attachment A.
- 1.2** **Compatible analytes.** PFOA and PFBS samples may be prepared and analyzed by this method.
- 1.3** **Acceptable matrices.** Aqueous buffered solutions ranging from pH 1.5 to 11.0 are acceptable.
- 1.4** **Method Performance.** This method is defined as performance-based (see Section 14).

2.0 SUMMARY OF METHOD

- 2.1** This method is based on OPPTS:835.2110 and has been modified to give additional information. Specifically, multiple time points over far longer periods of time are used to provide additional information regarding the hydrolytic behavior. Additional pH's are tested to provide insight into acid/base catalysis. Because of the longer time points used, (e.g. 109 days vs. 7) and the multiple time points (7 or 8 vs. 2) the temperature requirement of $\pm 0.1^\circ\text{C}$ has been relaxed (it is impossible to hold this tight of temperature and open the incubator to take out samples.) Oxygen is to be excluded (according to the OPPTS methodology) to minimize bacterial growth. However, the 50°C temperature used in the present study is forbidding to most mesophilic organisms and the pH dependence for bacterial growth is different than that observed for most chemical reactions that occur in water. Aliquots of PFOA solution are added to sample vials that contain 5 mL of buffered solution of pH 1.5, 3.0, 5.0, 7.0, 9.0, or 11.0. Appropriate quality control samples are prepared. Sample vials are tightly capped and placed into an orbital incubator/shaker at $50 \pm 3^\circ\text{C}$ and 100 ± 50 RPM, except for Day 0 samples, which are immediately refrigerated at $4 \pm 3^\circ\text{C}$ or frozen at $-20 \pm 10^\circ\text{C}$. Sample vials are removed from the incubator/shaker at designated intervals and refrigerated or frozen. When analyzed, all samples are allowed to come to room temperature, diluted with approximately 30 mL of methanol containing an internal standard, spiked (for those samples receiving spikes) and mechanically shaken for approximately 15 minutes. Samples are aliquoted into separate autovials. Calibration and quality control samples are prepared. PFOA is separated on a reverse-phase Dionex IonPac® NG-1 HPLC column, using an $\text{H}_2\text{O}/\text{MeOH}$ solvent gradient containing ammonium acetate. Analytes are detected and quantified by electrospray ionization mass spectrometry in the negative-ion mode.

3.0 DEFINITIONS

- 3.1 Method blank.** The method blank determines if there is contamination of the matrix. It is prepared identically to other samples, but it does not contain analyte (see Section 12.1.1). It is used alone and in conjunction with a method spike to detect accidental contamination (see Section 14.3).
- 3.2 Method spike.** A method spike is a spiked method blank (see Section 12.1.1) used to establish that the sample preparation method and analytical method would quantitatively detect target analyte in the method blank. (see Section 14.6).
- 3.3 Solvent (methanol) blank.** To isolate instrument contamination, pure methanol is analyzed. If the instrument is contaminated, analyte will be detected in the solvent blank. (see section 14.4).
- 3.4 Sample triplicates.** Samples are prepared in triplicate for each time point and pH, and are analyzed identically (see Section 12.1.1). The averaged result represents the PFOA concentration for that time point and pH.
- 3.5 Sample spike.** A sample spike confirms that the method recovers analyte effectively from the sample matrix (see Section 14.6). A known amount of analyte is added to a sample after it is incubated (see Sections 12.1.1).
- 3.6 Internal standard (IS).** An internal standard is used to evaluate and control the precision (Section 3.7) and bias (Section 3.8) of the analytical process. An analytically similar compound is added to all samples and standards, creating a known, fixed concentration in all samples and standards through the entire measurement process.
- 3.7 Precision.** Precision is the degree of agreement between individual sample results. It is usually expressed as a standard deviation.
- 3.8 Bias.** Measurements are biased if they are consistently high or low (a systematic error), as compared to a known value.
- 3.9 Calibration standard.** A calibration standard is a solution containing a known concentration of analyte. A series of calibration standards is used to calibrate analytical instrument response, producing a calibration curve to determine analyte concentrations in samples.
- 3.10 Continuing calibration verification (CCV) or Check standard.** After the analytical instrument has been calibrated, a single calibration standard is analyzed at selected time intervals throughout the analytical run. The values are compared to the calibration curve to ensure that the calibration curve is valid for all samples over time, that the instrument has not drifted out of calibration.
- 3.11 Dilution.** A miscible solvent is added to all samples to prepare them for instrumental analysis.
- 3.12 Laboratory Water.** Water with a measured resistivity of 18.0 MΩ-cm (or greater).
- 3.13 Limit of quantitation (LOQ).** The limit of quantitation (LOQ) is equal to the concentration of the lowest standard in the calibration curve that gives a response of more than four times the response of the solvent blanks and/or MeOH blanks. Use the solvent blanks and/or MeOH blanks that give the highest analyte counts.
- 3.14 Residuals.** In this method, a residual is the absolute value of the difference between the known, prepared concentration of a calibration standard and its concentration predicted by the calibration curve, divided by the known, prepared concentration, all multiplied by

100. Residuals for linear calibration curves can be determined using Excel. For non-linear calibration curves, the following equation should be used to determine a residual.

$$\text{Residual} = \left| \frac{[\text{Analyte}]_{\text{known}} - [\text{Analyte}]_{\text{predicted by the calibration curve}}}{[\text{Analyte}]_{\text{known}}} \right| \times 100\%$$

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 appropriate gloves and proper eyewear at all times.
- 4.1.2 For sample preparation and whenever possible, use solvents in the hood.
- 4.1.3 For potential hazards of each chemical used, refer to material safety data sheets, packing materials, and 3M Environmental Laboratories Chemical Hazard Review.

4.2 Cautions

- 4.2.1 Rinse all glassware for standards preparation with MeOH and acetone and dry, to reduce the possibility of contamination.
- 4.2.2 Prepare enough fresh HPLC mobile phase to analyze all samples. Do not allow the HPLC pump to run dry.
- 4.2.3 Ensure that there is ample memory on the computer hard drive to save all run data.
- 4.2.4 Ensure that there is enough nitrogen in the supply tank to analyze all samples.

5.0 INTERFERENCES

- 5.1 Contaminants in solvents, reagents, glassware, and other sample preparation or analysis hardware may produce interferences. Routinely analyze method blanks to detect contamination from these sources (see Section 10.4).
- 5.2 Contaminants from columns, HPLC tubing, and detector components may cause interference at low detection levels. Routinely analyze solvent blanks (methanol) to detect contamination from these sources (see Section 10.5).

6.0 EQUIPMENT

- 6.1 Analytical balance sensitive to 0.0001 g
- 6.2 Shaker/incubator capable of maintaining a temperature of 50 ± 3 °C
- 6.3 Hewlett-Packard (HP) 1100 HPLC System, or equivalent
 - 6.3.1 Quaternary pump, Model G1311A, or equivalent
 - 6.3.2 Solvent degasser, Model G1322A or equivalent
 - 6.3.3 Autosampler, ALS Model G1313A, variable injection volume capable, or equivalent
 - 6.3.4 Column heater, Model G1316A or equivalent
- 6.4 Dionex IonPac®NG-1 column, 35 mm x 4.0 mm, 10-µm diameter packing, or equivalent

- 6.5 Mass spectrometer. Hewlett-Packard LC/MSD, or equivalent, capable of operating in the selected-ion-monitoring mode
- 6.6 Digital clock. The same clock should be used during any one step of sample preparation, to ensure that procedures are performed correctly and documented accurately.
- 6.7 Corning Model 308 pH/Temperature Meter with 3-in-1 gel-filled combination electrode (pH/reference/temperature), or equivalent. The pH meter must be calibrated with suitable buffer standards (e.g. pH 4.0, 7.0 and 9.0 - Mallinckrodt or equivalent) as recommended by the manufacturer for this broad of a pH range.
- 6.8 Refrigerator, capable of maintaining 4 ± 3 °C, or a freezer capable of maintaining -20 ± 10 °C
- 6.9 Data system. A PC computer capable of simultaneously controlling the HPLC system and recording and processing signals from the detector
- 6.10 Data analysis software. Hewlett-Packard-ChemStation®, Version A 6.03 or higher, or Target Software.

7.0 SUPPLIES AND MATERIALS

- 7.1 Vials, 40-mL VOA (I-CHEM or equivalent)
- 7.2 Crimp-cap autovials, 1.5-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 $\pm 1\%$ of total volume), 10- μ L to 1000- μ L
- 7.7 Volumetric flasks, 10-, 25-, 100-, 1000-, and 4000-mL
- 7.8 Beakers, glass, various sizes
- 7.9 Automatic pipettor, capable of dispensing 10 to 5000 μ L
- 7.10 Miscellaneous equipment as needed

8.0 REAGENTS AND SOLUTIONS

8.1 Reagents

- 8.1.1 Methanol (MeOH). HPLC/SPEC/GC grade from EM Science, or equivalent
- 8.1.2 Laboratory Water (LW)
- 8.1.3 Hydrochloric acid (HCl), reagent grade
- 8.1.4 Sodium hydroxide (NaOH), reagent grade
- 8.1.5 Sodium borate decahydrate (borax), reagent grade
- 8.1.6 Sodium bicarbonate (NaHCO_3), reagent grade
- 8.1.7 Potassium dihydrogen phosphate (KH_2PO_4), reagent grade
- 8.1.8 Potassium acid phthalate (KHP), reagent grade
- 8.1.9 Ammonium acetate ($\text{CH}_3\text{CO}_2\text{-NH}_4$), reagent grade
- 8.1.10 Potassium salt of Perfluorooctanoic acid (PFOA)
- 8.1.11 Potassium salt of Perfluorobutanesulfonate (PFBS)

8.2 Stock Solutions

- 8.2.1 **Ammonium acetate stock solution.** Dissolve approximately 3.9 g $\text{CH}_3\text{CO}_2\cdot\text{NH}_4$ in MeOH to a total volume of 100 mL to give approximately 500 mM $\text{CH}_3\text{CO}_2\cdot\text{NH}_4$.
- 8.2.2 **PFOA stock solution.** Dissolve approximately 0.1000 g of PFOA in MeOH to a total volume of 10 mL to give approximately 10,000 $\mu\text{g/mL}$ PFOA.
- 8.2.3 **PFBS stock solution.** Dissolve approximately 0.0500 g of PFBS in MeOH to a total volume of 25 mL to give approximately 2,000 $\mu\text{g/mL}$ PFBS.
- 8.2.4 **Hydrochloric acid (HCl) stock solution.** Add approximately 8.6 mL of concentrated HCl to approximately 600 mL LW, dilute to a total volume of 1 L to give approximately 0.1 M HCl.
- 8.2.5 **Sodium hydroxide (NaOH) stock solution.** Dissolve approximately 4.0 g of solid NaOH in LW to a total volume of 1 L to give approximately 0.1 M NaOH.
- 8.2.6 **Potassium dihydrogen phosphate stock solution.** Dissolve approximately 13.6 g of KH_2PO_4 in LW to a total volume of 1 L to give approximately 0.1 M (KH_2PO_4).
- 8.2.7 **Borax stock solution.** Dissolve approximately 9.54 g of $\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$ in LW to a total volume of 1 L to give approximately 0.025 M $\text{Na}_2\text{B}_4\text{O}_7$.
- 8.2.8 **Sodium bicarbonate stock solution.** Dissolve approximately 4.2 g of NaHCO_3 in LW to a total volume of 1 L to give approximately 0.05 M NaHCO_3 .
- 8.3 **PFOA test analyte solution.** Dilute 2 mL of PFOA stock solution (Section 8.2.2) with MeOH to a total volume of 10 mL to give approximately 2,000 $\mu\text{g/mL}$ PFOA.
- 8.4 **PFOA spike solution.** Dilute 0.5 mL of PFOA stock solution (Section 8.2.2) with MeOH to a total volume of 10 mL to give approximately 500 $\mu\text{g/mL}$ PFOA.
- 8.5 **PFBS diluting solution.** Dilute 400 μL of PFBS stock solution (Section 8.2.3) with MeOH to a total volume of 4 L to give approximately 200 ng/mL PFBS.
- 8.6 **Chromatographic solvents**
 - 8.6.1 **Chromatographic solvent A.** Dilute 10 mL of ammonium acetate stock solution (Section 8.2.1) LW to a total volume of 1 L to give approximately 5 mM $\text{CH}_3\text{CO}_2\cdot\text{NH}_4$.
 - 8.6.2 **Chromatographic solvent B.** Dilute 10 mL of ammonium acetate stock solution (Section 8.2.1) with MeOH to a total volume of 1 L to give approximately 5 mM $\text{CH}_3\text{CO}_2\cdot\text{NH}_4$.
- 8.7 **Calibration buffers for pH meter.** Use commercially available pH meter calibration buffers of pH 4.0, 7.0, and 10.0 (Mallinckrodt or equivalent) as recommended by the manufacturer for this broad of a pH range.
- 8.8 **Hydrolysis buffers.** Buffer solutions should be made following EPA and CRC Handbook of Chemistry and Physics guidelines (References 18.1 and 18.2)
 - 8.8.1 **Hydrolysis buffer pH 1.5**
 - a) Add 207 mL 0.1 N HCl, 250 mL 0.1 M KCl to a 1 L volumetric flask.
 - b) Adjust pH to 1.5 with HCl stock solution
 - c) Dilute to mark with LW
 - 8.8.2 **Hydrolysis buffer pH 3.0**
 - a) Dissolve approximately 10.2 g of potassium acid phthalate (KHP) with approximately 600 mL of LW water in a 1 L volumetric flask.
 - b) Add approximately 223 mL of HCl stock solution (Section 8.2.4).

- c) Adjust the pH to 3.0 with either HCl stock solution (Section 8.2.4) or NaOH stock solution (Section 8.2.5).
- d) Bring to a final volume of 1 L with LW.

8.8.3 Hydrolysis buffer pH 5.0

- a) Dissolve approximately 10.2 g of potassium acid phthalate (KHP) with approximately 600 mL of LW in a 1 L volumetric flask.
- b) Add approximately 226 mL of NaOH stock solution (Section 8.2.5).
- c) Adjust the pH to 5.0 with either HCl stock solution (Section 8.2.4) or NaOH stock solution (Section 8.2.5).
- d) Bring to a final volume of 1 L with LW.

8.8.4 Hydrolysis buffer pH 7.0

- a) Add approximately 500 mL of potassium dihydrogen phosphate stock solution (Section 8.2.6) to a 1 L volumetric flask.
- b) Add approximately 291 mL of NaOH stock solution (Section 8.2.5).
- c) Adjust the pH to 7.0 with either HCl stock solution (Section 8.2.4) or NaOH stock solution (Section 8.2.5).
- d) Bring to a final volume of 1 L with LW.

8.8.5 Hydrolysis buffer pH 9.0

- a) Add approximately 500 mL of borax stock solution (Section 8.2.7) to a 1 L volumetric flask.
- b) Add approximately 46 mL of HCl stock solution (Section 8.2.4).
- c) Add LW to a total volume of approximately 950 mL.
- d) Adjust the pH to 9.0 with either HCl stock solution (Section 8.2.4) or NaOH stock solution (Section 8.2.5).
- e) Bring to a final volume of 1 L with LW.

8.8.6 Hydrolysis buffer pH 11.0

- a) Add approximately 500 mL of sodium bicarbonate stock solution (Section 8.2.8) to a 1 L volumetric flask.
- b) Add approximately 227 mL of NaOH stock solution (Section 8.2.5).
- c) Add LW to a total volume of approximately 950 mL.
- d) Adjust the pH to 11.0 with NaOH stock solution (Section 8.2.5).
- e) Bring to a final volume of 1 L with LW.

9.0 SAMPLE HANDLING

- 9.1** Record the times that samples were initially prepared, removed from the incubator, placed in the freezer (when applicable), removed from the freezer (when applicable) and subsequently diluted, using the fluorochemical degradation (hydrolysis) analysis sample preparation sheet (Attachment B). All samples for the same pH group should be analyzed together. Refrigerate or freeze them if necessary, and dilute them immediately preceding the analysis.

10.0 QUALITY CONTROL

- 10.1** See Section 12 for directions on preparing quality control samples.

- 10.2 Calibration standards.** Analyze a complete series of calibration standards before and after each analytical run for use as calibration curve. At the discretion of the analyst, this complete series may be run more often in the analytical run.
- 10.3 Continuing calibration verification (CCV).** A CCV sample should be analyzed after no more than 20 sample injections. A single calibration standard or a complete series of such standards (for use as a calibration curve) will satisfy this requirement.
- 10.4 Method blank.** Analyze a minimum of one method blank per time point per pH (e.g., Day 42, pH 7.0).
- 10.5 Solvent blank.** Analyze one solvent blank before and after every calibration curve as well as before and after every CCV.
- 10.6 Sample triplicates.** Prepare and analyze all hydrolysis samples in triplicate to determine analysis precision.
- 10.7 Sample spikes.** Prepare a sample spike for each pH and time point used in the study. Final (diluted) spike concentrations should approximate a mid-range calibration standard. The sample spike sample should be analyzed immediately following the sample triplicates to which it corresponds.
- 10.8 Internal standards.** Aliquots of PFBS stock solution (Section 8.2.3) are added to calibration standards to give a final internal standard concentration of approximately 158 ng/mL PFBS. Samples should also contain approximately the same concentration of PFBS.
- 11.0 CALIBRATION AND STANDARDIZATION**
-
- 11.1 Standard preparation.** Prepare at least five calibration standards of approximately 25 to 1000 ng/mL PFOA, containing approximately 158 ng/mL PFBS internal standard.
- 11.2 Standard curve.** Relate the ratio of the analyte peak area of the calibration standards to the internal standard peak area, using linear regression software. If calibration ratios are inconsistent, external standard calibration may be used. Consult the Team Leader or designated supervisor for recommendations before using external calibration.
- 12.0 PROCEDURES**
-

12.1 Sample preparation and hydrolysis

12.1.1 The following table summarizes sample preparation.

	Analyte	Buffer	Diluting solution containing internal standard	Spike	Methanol only	Incubate /shake
Calibration standards	X	X	X	--	--	--
Continued Calibration verification (CCV)	X	X	X	--	--	--
Method blank ^a	--	X	X	--	--	X
Solvent blank	--	--	--	--	X	--
Sample	X	X	X	--	--	X
Duplicate	X	X	X	--	--	X
Triplicate	X	X	X	--	--	X
Sample spike ^b	X	X	X	X	--	X
Method spike ^c	--	X	X	X	--	X

^a Also called Blank

^b Also called Spike, Matrix spike, and Replicate spike

^c Also called Blank spike

12.1.2 Determine the number of time points and corresponding samples to be analyzed. Each time point should have six vials per pH: *sample, duplicate, triplicate, sample spike, method spike, and method blank*.

12.1.3 Obtain the appropriate number of 40-mL VOA vials with caps, and the partitioned cardboard box they were shipped in. It will be helpful to use a separate box to hold the vials for each time point.

12.1.4 Prepare sample preparation worksheets (Attachment B), and label the vials with the sample number, pH, time point, and initials of the analyst. Record the pH and buffer ID of each hydrolysis buffer solution.

12.1.5 Add 5 mL of the appropriate buffer solution to all of the labeled vials. Always recap each vial immediately to minimize solvent evaporation.

12.1.6 Using a 10- μ L Gastight[®] syringe, add 15 μ L of PFOA test analyte solution (Section 8.3) to the following sample types for each time point and pH: *sample, duplicate, triplicate, and sample spike* (see Section 12.1.1).

- 12.1.7 Make sure all vials are well-sealed, and place them into a partitioned cardboard case (with cover) to exclude light. It will be helpful to use a separate box to hold the vials for each time point.
- 12.1.8 Refrigerate Day 0 samples at 4 ± 3 °C, or freeze them at -20 ± 10 °C.
- 12.1.9 Place the remaining samples into a pre-warmed incubator/shaker for the appropriate time. Record the time, temperature, and shaking rate (RPM). Continue to manually monitor the incubator temperature daily during the entire incubation. Record the temperature on the sample preparation sheet (Attachment B).
- 12.1.10 Remove each case from the incubator at the designated time. Refrigerate them at 4 ± 3 °C, or freeze them at -20 ± 10 °C, until all samples from all time points can be analyzed together.
- 12.1.11 Before analysis, allow the incubated vials to come to room temperature.
- 12.1.12 Add 30 mL of PFBS diluting solution (Section 8.5) to all vials.
- 12.1.13 Using a 10- μ L Gastight® syringe, add 10 μ L of PFOA spike solution (Section 8.4) to the *method spike* and *sample spike* vials (see Section 12.1.1). Recap the vials tightly, and invert them several times to mix.
- 12.1.14 Aliquot approximately 1 mL of each sample into the appropriately labeled autovial. Cap the vials.
- 12.1.15 Transfer the vials to the HPLC autosampler.
- 12.2 **HPLC set-up**
 - 12.2.1 Review instrument method 1101 PFOA.M. *Note that the following instrumentation set-up procedures apply to Hewlett-Packard/Agilent HP1100 equipment only.*
 - 12.2.2 For each analysis, ensure the appropriate HPLC column is in the instrument (Dionex IonPac® NG-1, 35 mm x 4.0 mm, 10- μ m diameter packing, or equivalent).
 - 12.2.3 Ensure the correct type and amount of eluent is loaded in the instrument. Be sure there is enough loaded to complete the analytical sequence.
 - 12.2.4 Place the samples in the autosampler tray and construct a sequence table with calibration standards, calibration check standards, and solvent blanks.
 - 12.2.5 Verify that all samples and standards are positioned correctly. Enter sequence information: sample or standard ID, method name, and one injection per sample.
 - 12.2.6 Save sequence as analysis date (e.g., on September 14, 2000 save sequence as 091400.s). Save data to a subdirectory labeled with analysis date (e.g., 091400).
 - 12.2.7 Set the post-sequence command macro to shut the system down ("STANDBY" on HP systems).
 - 12.2.8 Use the following solvents (see Section 8.6), gradient, and instrument settings (or equivalent - note that the following instrumentation set-up procedures apply to Hewlett-Packard/Agilent HP1100 MSD equipment only).
 - 12.2.8.1 Chromatographic solvent A (Section 8.6.1).
 - 12.2.8.2 Chromatographic solvent B (Section 8.6.2).
 - 12.2.8.3 Solvent Gradient

Time (min)	Percent A	Percent B	Flow Rate (mL/min)
0.0	95	5	0.3
1.0	95	5	0.3
8.0	0	100	0.3
15.0	0	100	0.3

12.2.8.4 Instrument settings

Injection volume (μL)	Back pressure (bar)	Column compartment temperature (°C)	Stop time (min)	Post time (min)
10.0	6-12	40	12.50	5.00

12.3 Mass spectrometer set-up

12.3.1 Use the following tables (or equivalent) to set up the mass spectrometer. (Note that the following instrumentation set-up procedures apply to Hewlett-Packard/Agilent HP1100 MSD equipment only.)

Acquisition Mode	Ionization Mode	Peak width (min)	Polarity	Drying Gas Flow (L/min)	Nebulizer Pressure (psig)	Drying Gas Temp (°C)	Capillary Voltage (V)
Selected Ion (SIM)	API-ES	0.25	Negative	8	30	350	3500

Analyte	HPLC Column Retention Time (min)	Selected Ion (amu)	Gain (EMV)	Fragmentor (V)	Dwell Time (msec)
PFBS	8.1	299 (M-H ⁺)*	4.0	Var.	739
PFOA	9.4	413 (M-H ⁺)*	4.0	Var.	739

* M-H⁺ is the molecular ion with a loss of proton. For example, the molecular weight of PFBS is 300, when it loses a proton, it assumes a negative charge and has a mass of 299.

12.4 Autosampler set-up

12.4.1 Use the following parameters (or equivalent) to set up the autosampler.

Autosampler	Autosampler Program	Injection volume (μL)
ALS Model G1313A	None	10.0

12.5 Sample analysis

12.5.1 After setting-up the HPLC, the mass spectrometer, and the autosampler, analyze a CCV after every 20 sample injections, and the calibration standards at the end of the sequence. To check for analyte carry-over, run solvent blanks after the highest calibration standard as well as before and after the CCV (see Section 10.0).

12.5.2 Identify the electronic acquisition files with an appropriate prefix. Do not exceed five characters if the sequence contains more than 99 lines.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Peak evaluation. Analyte and internal standard peaks must be symmetrical - if peak tailing is observed, consult team leader or designee for direction. They must be identified by measuring their retention times and by the compound-specific ions. When generating a calibration curve, analyte peak heights must be greater than four times the baseline noise and/or solvent blank for that region of the chromatogram. Peak areas are integrated manually or automatically from baseline to baseline through the peak. If present, analyte isomers appearing as either a shoulder or a discrete second peak in the chromatogram should be integrated with the analyte peak, unless otherwise indicated. Quantitation data are calculated using PFBS as the internal standard. However, external standard calibration may be acceptable. Consult the Team Leader or designated supervisor before using external calibration methodology.

13.2 Calculations involving analyte purity. In calculations where analyte purity ratios cancel each other (e.g., calculation of the hydrolysis rate constant, k based on loss of parent material), purity does not need to be considered.

13.3 Percent recovery. Calculate the percent of PFOA recovered from each of the sample spikes using the following equation.

$$\% \text{ Recovery} = \frac{[\text{PFOA}]_{\text{detected in sample spike}} - [\text{PFOA}]_{\text{detected in corresponding sample}}}{[\text{PFOA}]_{\text{in sample spike, added via spike}}} \times 100\%$$

13.4 Sample triplicates. Use the following equation to calculate the relative standard deviation (RSD) for the triplicate samples.

$$\text{RSD} = \frac{\text{Standard Deviation}}{\text{Mean Concentration}} \times 100\%$$

The RSD is also known as the coefficient of variation, a measure of the precision.

13.5 Calculation of k . Calculate the PFOA concentrations in each of the pH matrices using the calibration and internal (or external) standard curves. Assuming first-order kinetics a rate constant (k) can be determined by plotting

$$\ln \left(\frac{[PFOA]_t}{[PFOA]_0} \right) \text{ versus negative elapsed time } (-t),$$

where the subscripts t and 0 refer to analyte concentrations at time t and at $t = 0$, respectively. The slope of the resulting line is k .

14.0 METHOD PERFORMANCE

- 14.1 Calibration curves.** An acceptable coefficient of determination (R^2) for linear curves is 0.990 or greater. Accuracy should be verified, particularly at upper and lower calibration limits by calculating the residuals -residuals generated in curve-fitting must be between 0 and 25%. Alternative methods of curve-fitting (e.g., quadratic) require a coefficient of determination (r^2) of 0.990 or greater. Record the reason(s) for using quadratic curve-fitting in the raw data. If the quality control parameters are not met, consult team leader or designee for direction.
- 14.2 Continuing calibration verification (CCV).** Analyte concentration must not differ by more than 25% of its expected value, relative to the initial calibration curve. Accept only those samples analyzed before the last accepted calibration verification. Reanalyze remaining samples with a new calibration curve.
- 14.3 Method blank.** If the contamination levels are greater than 5% for any particular target, consult the Team Leader or designated supervisor for recommendations. For example, if PFOA were targeted at 500 ppb, contamination should be less than 25 ppb. In those instances where the 5% level would be below the lowest point of the calibration curve, a 25% value is used rather than the 5% (as 5% cannot be quantified and the 25% level brings this in line with the LOQ definition, see section 14.8).
- 14.4 Solvent blanks.** Solvent blanks should show no more than a 5% carry-over from a high standard or CCV. If they do, two solvent blanks should be analyzed to rule out instrument contamination. If solvent blanks are still showing more than 5% carry-over, or are adversely affecting the LOQ (see Section 14.8), the run should be stopped. This indicates that the instrument is contaminated and should be thoroughly cleaned. Pay particular attention to the electrospray source. The column and tubing may need to be replaced. When the solvent blanks are improved, reanalyze the sequence beginning at the last acceptable CCV or calibration curve, starting with a new calibration curve.
- 14.5 Sample triplicates.** If RSD precision values are 25% or greater, consult the Team Leader or designated supervisor for recommendations.
- 14.6 Sample spikes.** If sample spike recoveries are less than 70% or greater than 130%, consult the Team Leader or designated supervisor for recommendations.
- 14.7 Residuals.** The acceptance criterion for the residuals is less than or equal to $\pm 25\%$. If the residuals are higher than this, consult team leader or designee for direction. No calibration curve will be accepted with residuals outside this range.
- 14.8 Limit of Quantitation.** The limit of quantitation (LOQ) is equal to the lowest standard in the calibration curve that gives a response of more than four times that of the test analyte in the solvent blanks.
- 14.9 Method Spike.** If method spike recoveries are less than 70% or greater than 130%, consult the Team Leader or designated supervisor for recommendations.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 15.1** Dispose of sample waste by placing it 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. When the satellite can is full, empty it into the flammable storage drum in the hazardous waste collection area on the second floor.

16.0 RECORDS

- 16.1** Print out hard copies of all graphics and data analysis summaries for archiving.
- 16.2** Sign and date all graphics, and label with the instrument ID.
- 16.3** Immediately fill out the hydrolysis sample preparation worksheet completely, including all initials and dates.
- 16.4** Print chromatograms and internal standard reports for all analyses.
- 16.5** Print calibration tables and curve information, and store them in the raw data file.
- 16.6** Store hydrolysis sample preparation worksheets in the raw data file.
- 16.7** 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.
- 16.8** Archive electronic data to appropriate media when necessary.

17.0 ATTACHMENTS

- 17.1** Attachment A. Representative Chemical Structures
- 17.2** Attachment B. Hydrolysis Sample Log sheet

18.0 REFERENCES

- 18.1** *Fate, Transport and Transformation Test Guidelines OPPTS 835.2110: Hydrolysis as a Function of pH*; EPA 712-C-98-057; U.S. Environmental Protection Agency, Office of Prevention, Pesticides and Toxic Substances, U.S. Government Printing Office: Washington, DC, 1998.
- 18.2** *CRC Handbook of Chemistry and Physics*, 1st Student Edition; Weast, R. C., Ed., CRC Press: Cleveland, OH, 1988; p. D-87.

19.0 AFFECTED DOCUMENTS

None.

20.0 REVISIONS

Revision Number

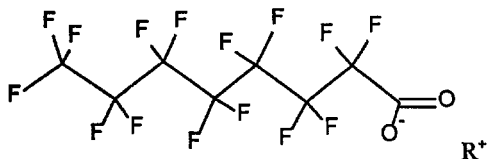
Reason for Revision

Attachment A. Representative Chemical Structures

$R^+ = NH_4^+$ in this study, but may also be Li^+ , Na^+ , K^+ , H^+ .

1. PFOA (Perfluorooctanoic Acid)

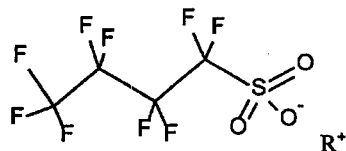
MW (anion) = 413



$R^+ = NH_4^+$

2. PFBS (Perfluorobutanesulfonate)

MW (anion) = 299



$R^+ = K^+$

Attachment B. Sample Preparation Sheet

Fluorochemical Degradation (Hydrolysis) Analysis

TEST ANALYTE: _____ Incubator ID: _____ Freezer ID: _____
 Lab Request Number: _____ Start Date: _____ Time: _____
 Nominal Interval: _____ Stop Date: _____ Time: _____
 Nominal Temperature: _____ °C Incubation Interval: _____ days _____ hours _____ min

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

Comments: _____

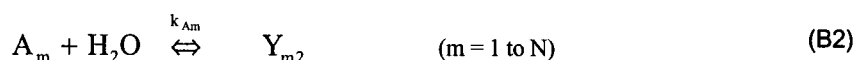
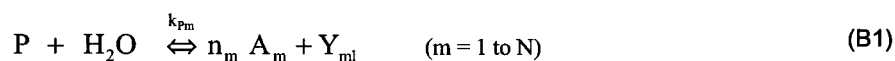
Appendix B: Kinetics Model

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).



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

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

which is equivalent to the separable differential equation

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

Equation B4 may be directly integrated to obtain the general solution

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

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

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

using the additional definition of the total parent hydrolysis rate

$$k_p \equiv \sum_{m=1}^N n_m k_{pm} \quad (B7)$$

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

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

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 $T_p^{1/2}$ (the parent hydrolysis half-life) the parent concentration P is reduced through hydrolysis by a factor of two, where

$$T_p^{1/2} = \frac{\ln(2)}{k_p} \quad (B9)$$

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

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

B3. Product Compound Concentrations

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

$$dA_m = (n_m k_{pm} P - k_{Am} A_m) dt = (n_m k_{pm} P_0 e^{-k_p t} - k_{Am} A_m) dt \quad (B11)$$

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

$$\frac{dA_m}{dt} + k_{Am} A_m = n_m k_{pm} P_0 e^{-k_p t} \quad (B12)$$

The "standard form" of Equation B12 is

$$A_m' + S(t) A_m = Q(t) \quad (B13)$$

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

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

and

$$Q(t) = n_m k_{Pm} P_0 e^{-k_p t} \quad (B15)$$

The general solution A_m to Equation B12 is contained in

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

where

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

and

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

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

$$\text{(for } k_{Am} = k_p \text{)} \quad (B19)$$

$$A_m e^{k_p t} = n_m k_{Pm} P_0 t + C$$

and, using the initial condition $A_m(t=0) = A_{m0}$, the specific solution to Equation 18 is

$$\text{(for } k_{Am} = k_p \text{)} \quad (B20)$$

$$A_m = (n_m k_{Pm} P_0 t + A_{m0}) e^{-k_p t}.$$

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

$$A_m = A_{m0} \quad (B21)$$

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

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

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

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

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)

$$A_m = A_{m0} + \frac{n_m k_{Pm} P_0}{k_p} (1 - e^{-k_p t}) \quad (B24)$$

B4. Relationships Between the Parent and Compound Concentrations

Equations B7 and B24 can be combined to obtain

(for hydrolytically stable products)

$$k_p = \sum_{m=1}^N n_m k_{Pm} = \frac{k_p}{(1 - e^{-k_p t})} \sum_{m=1}^N \frac{(A_m - A_{m0})}{P_0} \quad (B25)$$

so that

(for hydrolytically stable products)

$$(1 - e^{-k_p t}) = \sum_{m=1}^N \frac{(A_m - A_{m0})}{P_0} \quad (\text{B26})$$

or

(for hydrolytically stable products)

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

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

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

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

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

and Equation B23 becomes

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

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

or

$$\begin{aligned} & \text{(for hydrolytically stable products and } \left| \sum_m A_m - A_{m0} \right| \ll P_0) \\ & k_p t \equiv \sum_{m=1}^N \frac{A_m - A_{m0}}{P_0} \end{aligned} \quad (B31)$$

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 $A_m = A_m^{\text{LOQ}}$. With these assumptions, the experimental data indicate that the reaction rate k_p is less than some maximum value $(k_p)_{\text{max}}$ as follows:

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

$$k_p \leq (k_p)_{\text{max}} = \frac{1}{P_0 \Delta t} \sum_{m=1}^N A_m^{\text{LOQ}}. \quad (B32)$$

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 $(T_p^{1/2})_{\text{min}}$ as follows:

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

$$T_p^{1/2} \geq (T_p^{1/2})_{\text{min}} = \frac{\ln(2)}{(k_p)_{\text{max}}} = \Delta t P_0 \ln(2) \left[\sum_{m=1}^N A_m^{\text{LOQ}} \right]^{-1} \quad (B33)$$

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 $(k_p)_{\text{max}}$ 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 $(T_p^{1/2})_{\text{min}}$ 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)

$$k_p \leq (k_p)_{\max} = \frac{1}{P_0 \Delta t} \left[\sum_{\text{Below LOQ}} A_m^{\text{LOQ}} + \sum_{\text{Constan t}} 2\sigma_m \right]. \quad (\text{B34})$$

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)

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

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.

B7. 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 $t = \Delta t$. This reasoning suggests that the following estimate of the maximum parent hydrolysis rate is appropriate:

(for essentially constant parent concentrations with mean value μ_p and standard deviation σ_p)

$$k_p \leq (k_p)_{\max} = \frac{2\sigma_p}{\mu_p \Delta t} \quad (\text{B36})$$

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 essentially constant parent concentrations with mean value μ_p and standard deviation σ_p)

$$T_p^{1/2} \geq (T_p^{1/2})_{\min} = \frac{\ln(2)}{(k_p)_{\max}} = \frac{\mu_p \Delta t \ln(2)}{2\sigma_p} \quad (\text{B37})$$

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 $50^\circ\text{C} = 323 \text{ K}$. Of greater interest are the corresponding results for the environmentally important temperature $25^\circ\text{C} = 298 \text{ K}$.

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):

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

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

$$\frac{k_1}{k_2} = \exp\left\{\frac{18}{1.99 \times 10^{-3}} \left[\frac{1}{323} - \frac{1}{298}\right]\right\} = \exp(-2.35) = 0.095 \quad (\text{B39})$$

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).

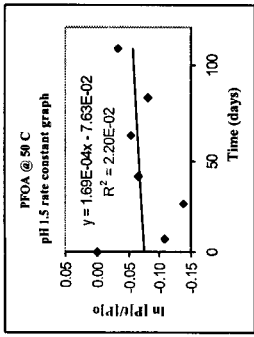
Appendix C: Selected Analytical and Kinetics Results

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

PFOA Buffer Hydrolysis Study at 50° C.

All concentrations in ng/ml.

pH	Time (Days)	Conc. (A)	$\ln(P)/[P]_0$
7.5	14	378	0.000
1.5	0	496	-0.107
1.5	7	445	-0.137
1.5	28	432	-0.066
1.5	42	464	-0.054
1.5	64	470	-0.081
1.5	84	457	-0.032
1.5	109	480	-0.032



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.14822
R Square	0.02197
Adjusted R Square	-0.17364
Standard Error	0.04960
Observations	7

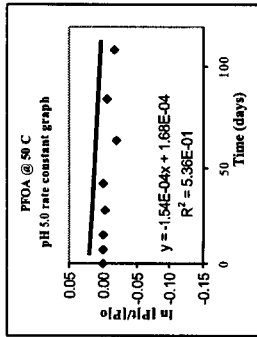
ANOVA

	df	SS
Regression	1	0.00028
Residual	5	0.01230
Total	6	0.01257

Coefficients		Standard Error
Intercept	-0.07628	0.03048
X Variable 1	0.00017	0.00050

% 2s Slope Uncertainty
587%

pH	Time (Days)	Conc.	$\ln(P)/[P]_0$
5.0	0	657	0.000
5.0	7	656	-0.002
5.0	14	656	-0.002
5.0	28	655	-0.003
5.0	42	656	-0.002
5.0	64	643	-0.021
5.0	84	654	-0.005
5.0	109	645	-0.018



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.73221
R Square	0.53613
Adjusted R Square	0.45882
Standard Error	0.00605
Observations	8

ANOVA

	df	SS
Regression	1	0.00025
Residual	6	0.00022
Total	7	0.00047

Coefficients		Standard Error
Intercept	0.00017	0.00333
X Variable 1	-0.00015	0.00006

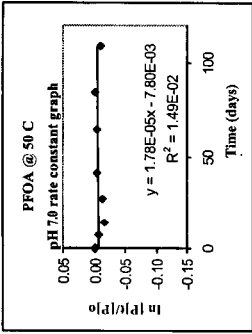
% 2s Slope Uncertainty
76%

(A) Data in *italics* have been excluded from the fits on the basis of the data quality objectives (see text).

PFOA Buffer Hydrolysis Study at 50° C.

All concentrations in ng/ml.

pH	Time (Days)	Conc.	ln([P]/[P] ₀)
7.0	0	653	0.000
7.0	7	649	-0.007
7.0	14	642	-0.017
7.0	28	645	-0.012
7.0	42	650	-0.005
7.0	64	650	-0.005
7.0	84	652	-0.001
7.0	109	647	-0.009



SUMMARY OUTPUT

Regression Statistics		
Multiple R		0.12196
R Square		0.01487
Adjusted R Square		-0.14931
Standard Error		0.00611
Observations		8

ANOVA

	df	SS
Regression	1	0.00000
Residual	6	0.00022
Total	7	0.00023

Coefficients			Standard Error
Intercept		-0.00780	0.00336
X Variable 1		0.00002	0.00006

% 2σ Slope Uncertainty
664%

SUMMARY OUTPUT

Regression Statistics		
Multiple R		0.31722
R Square		0.10063
Adjusted R Square		-0.04927
Standard Error		0.01338
Observations		8

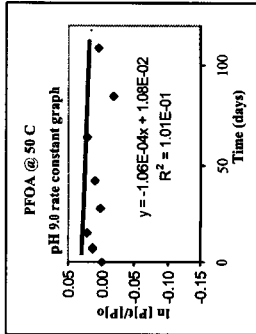
ANOVA

	df	SS
Regression	1	0.00012
Residual	6	0.00107
Total	7	0.00119

Coefficients			Standard Error
Intercept		0.01081	0.00735
X Variable 1		-0.00011	0.00013

% 2σ Slope Uncertainty
244%

pH	Time (Days)	Conc.	ln([P]/[P] ₀)
9.0	0	653	0.000
9.0	7	661	0.012
9.0	14	666	0.020
9.0	28	654	0.001
9.0	42	660	0.010
9.0	64	667	0.020
9.0	84	640	-0.020
9.0	109	657	0.005



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

Page 1 of 3

PFDA				PFBS			
Sample ID	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	RT
MeOH	9.4	0	0				8.1
00028-94-00pH1.5	9.4	0	0				8.1
HILL0012.D	9.4	0	0				8.1
00028-94-01pH1.5	9.4	87108	25	101%			8.1
HILL0014.D	9.4	269160	74	98%			8.1
00028-94-02pH1.5	9.4	567497	155	103%			8.1
HILL0015.D	9.4	1088824	305	102%			8.1
00028-94-03pH1.5	9.4	1544196	446	99%			8.1
HILL0016.D	9.4	2074680	627	105%			8.1
00028-94-04pH1.5	9.4	2396263	749	100%			8.1
HILL0017.D	9.4	2742512	886	99%			8.1
00028-94-05pH1.5	9.4	3048151	1040	104%			8.1
HILL0018.D	9.4	1536260	445		99%		8.1
00028-94-06pH1.5	9.4	0	0				8.1
HILL0019.D	9.4	0	0				8.1
00028-94-07pH1.5	9.4	0	0				8.1
HILL0020.D	9.4	0	0				8.1
00028-94-08pH1.5	9.4	0	0				8.1
HILL0021.D	9.4	0	0				8.1
00028-94-09pH1.5	9.4	0	0				8.1
HILL0022.D	9.4	0	0				8.1
MeOH	9.4	0	0				8.1
00028-94-05pH1.5	9.4	0	0				8.1
HILL0023.D	9.4	0	0				8.1
061500-PFOA-001	9.4	620229	171		119%	Day 0	8.1
HILL0024.D	9.4	1618886	469			Day 0	8.1
061500-PFOA-002	9.4	1736393	511			Day 0	8.1
HILL0025.D	9.4	1724702	508			Day 0	8.1
061500-PFOA-003	9.4	2128004	655	5%	111%	Day 0	8.1
HILL0026.D	9.4	0	0				8.1
061500-PFOA-004	9.4	615225	169		118%	Day 7	8.1
HILL0027.D	9.4	1550782	449			Day 7	8.1
061500-PFOA-005	9.4	1538986	445			Day 7	8.1
HILL0028.D	9.4	1535116	443			Day 7	8.1
061500-PFOA-006	9.4	1989327	588	1%	99%	Day 7	8.1
HILL0029.D	9.4	0	0				8.1
061500-PFOA-007	9.4	615864	167		117%	(A)	8.1
HILL0030.D	9.4	1346582	380			(A)	8.1
061500-PFOA-008	9.4	1424910	402			(A)	8.1
HILL0031.D	9.4	1254577	350			(A)	8.1
061500-PFOA-009	9.4	2049553	609	7%	162%	(A)	8.1
HILL0032.D	9.4	0	0				8.1
061500-PFOA-010	9.4	0	0				8.1
HILL0033.D	9.4	88571	25	101%			8.1
061500-PFOA-011	9.4	268237	72	96%			8.1
HILL0034.D	9.4	555918	150	100%			8.1
061500-PFOA-012	9.4	1074690	296	99%			8.1
HILL0035.D	9.4	1528049	433	96%			8.1
061500-PFOA-013	9.4	2065254	618	103%			8.1
HILL0036.D	9.4	2395045	734	98%			8.1
061500-PFOA-014	9.4	2735814	862	96%			8.1
HILL0037.D	9.4	3013737	997	100%			8.1
061500-PFOA-015	9.4	0	0				8.1
HILL0038.D	9.4	1518810	431		96%		8.1
061500-PFOA-016	9.4	0	0				8.1
HILL0039.D	9.4	0	0				8.1
061500-PFOA-017	9.4	0	0				8.1
HILL0040.D	9.4	0	0				8.1
061500-PFOA-018	9.4	0	0				8.1
HILL0041.D	9.4	0	0				8.1
061500-PFOA-019	9.4	0	0				8.1
HILL0042.D	9.4	0	0				8.1
061500-PFOA-020	9.4	0	0				8.1
HILL0043.D	9.4	0	0				8.1
061500-PFOA-021	9.4	0	0				8.1
HILL0044.D	9.4	0	0				8.1
061500-PFOA-022	9.4	0	0				8.1
HILL0045.D	9.4	0	0				8.1
061500-PFOA-023	9.4	0	0				8.1
HILL0046.D	9.4	0	0				8.1
061500-PFOA-024	9.4	0	0				8.1
HILL0047.D	9.4	0	0				8.1
061500-PFOA-025	9.4	0	0				8.1
HILL0048.D	9.4	0	0				8.1
061500-PFOA-026	9.4	0	0				8.1
HILL0049.D	9.4	0	0				8.1
061500-PFOA-027	9.4	0	0				8.1
HILL0050.D	9.4	0	0				8.1
061500-PFOA-028	9.4	0	0				8.1
HILL0051.D	9.4	0	0				8.1
061500-PFOA-029	9.4	0	0				8.1
HILL0052.D	9.4	0	0				8.1
061500-PFOA-030	9.4	0	0				8.1
HILL0053.D	9.4	0	0				8.1
061500-PFOA-031	9.4	0	0				8.1
HILL0054.D	9.4	0	0				8.1
061500-PFOA-032	9.4	0	0				8.1
HILL0055.D	9.4	0	0				8.1
061500-PFOA-033	9.4	0	0				8.1
HILL0056.D	9.4	0	0				8.1
061500-PFOA-034	9.4	0	0				8.1
HILL0057.D	9.4	0	0				8.1

Method ID: Internal Standard Quant (2=)
Curve averaged: Linear. Include origin
Calibration range: 1%
(A) Data excluded on the basis of data quality objectives; see text.

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

Page 2 of 3

PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	%Spike Recovery	Time Point
MeOH	HILL0004.D	9.4	0	0			
00028-94-00pH1.5	HILL0005.D	9.4	86727	25	99%		
00028-94-01pH1.5	HILL0006.D	9.4	263618	72	96%		
00028-94-02pH1.5	HILL0007.D	9.4	566304	155	104%		
00028-94-03pH1.5	HILL0008.D	9.4	1086829	303	101%		
00028-94-04pH1.5	HILL0009.D	9.4	1527863	439	97%		
00028-94-05pH1.5	HILL0010.D	9.4	2057013	620	104%		
00028-94-06pH1.5	HILL0011.D	9.4	2397916	741	99%		
00028-94-07pH1.5	HILL0012.D	9.4	2723201	877	98%		
00028-94-08pH1.5	HILL0013.D	9.4	3008216	1015	102%		
MeOH	HILL0014.D	9.4	0	0			
00028-94-09pH1.5	HILL0015.D	9.4	1530134	442	98%		
MeOH	HILL0016.D	9.4	0	0			
061500-PFOA-109	HILL0017.D	9.4	0	0			
061500-PFOA-110	HILL0018.D	9.4	611006	167	117%		Day 28
061500-PFOA-111	HILL0019.D	9.4	1566907	455			Day 28
061500-PFOA-112	HILL0020.D	9.4	1449273	418			Day 28
061500-PFOA-113	HILL0021.D	9.4	1470566	424			Day 28
061500-PFOA-114	HILL0022.D	9.4	1896744	566	5%		Day 28
061500-PFOA-145	HILL0023.D	9.4	0	0			
061500-PFOA-146	HILL0024.D	9.4	612576	168	117%		Day 42
061500-PFOA-147	HILL0025.D	9.4	1539839	453			Day 42
061500-PFOA-148	HILL0026.D	9.4	1645514	488			Day 42
061500-PFOA-149	HILL0027.D	9.4	1539497	452			Day 42
061500-PFOA-150	HILL0028.D	9.4	2004080	612	103%		Day 42
061500-PFOA-181	HILL0030.D	9.4	0	0			
061500-PFOA-182	HILL0031.D	9.4	614207	170	119%		Day 64
061500-PFOA-183	HILL0032.D	9.4	1586941	470			Day 64
061500-PFOA-184	HILL0033.D	9.4	1588535	471			Day 64
061500-PFOA-185	HILL0034.D	9.4	1593078	468			Day 64
061500-PFOA-186	HILL0035.D	9.4	2031468	615	0%		Day 64
MeOH	HILL0036.D	9.4	0	0			
00028-94-00pH1.5	HILL0037.D	9.4	0	0			
00028-94-01pH1.5	HILL0038.D	9.4	85229	25	98%		
00028-94-02pH1.5	HILL0039.D	9.4	263990	73	97%		
00028-94-03pH1.5	HILL0040.D	9.4	547560	152	101%		
00028-94-04pH1.5	HILL0041.D	9.4	1055076	299	100%		
00028-94-05pH1.5	HILL0042.D	9.4	1507704	441	98%		
00028-94-06pH1.5	HILL0043.D	9.4	2029865	619	103%		
00028-94-07pH1.5	HILL0044.D	9.4	2358294	744	99%		
00028-94-08pH1.5	HILL0045.D	9.4	2709145	877	98%		
00028-94-09pH1.5	HILL0046.D	9.4	2974918	1016	102%		
MeOH	HILL0047.D	9.4	0	0			
00028-94-05pH1.5	HILL0048.D	9.4	1490907	438	97%		
MeOH	HILL0049.D	9.4	0	0			
Method ID:				Internal Standard Quant r2=			
				Curve averaged: Linear: Include origin			
				Calibration range:			
				Mean			
				S.D.			
				%RSD			

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

Page 3 of 3

Method ID:

PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point
MeOH	HILL0036.D	9.4	0	0			
00028-94-00pH1.5	HILL0037.D	9.4	0	0			
00028-94-01pH1.5	HILL0038.D	9.4	85229	25	101%		
00028-94-02pH1.5	HILL0039.D	9.4	263990	74	98%		
00028-94-03pH1.5	HILL0040.D	9.4	547960	154	103%		
00028-94-04pH1.5	HILL0041.D	9.4	1055076	303	101%		
00028-94-05pH1.5	HILL0042.D	9.4	1507704	447	99%		
00028-94-06pH1.5	HILL0043.D	9.4	2029865	627	105%		
00028-94-07pH1.5	HILL0044.D	9.4	2358294	755	101%		
00028-94-08pH1.5	HILL0045.D	9.4	2709145	890	99%		
00028-94-09pH1.5	HILL0046.D	9.4	2974918	1031	103%		
MeOH	HILL0047.D	9.4	0	0			
00028-94-05pH1.5	HILL0048.D	9.4	1490907	444	99%		
MeOH	HILL0049.D	9.4	0	0			
061500-PFOA-217	HILL0050.D	9.4	0	0			
061500-PFOA-218	HILL0051.D	9.4	604624	172	120%		Day 84
061500-PFOA-219	HILL0052.D	9.4	1517361	456			Day 84
061500-PFOA-220	HILL0053.D	9.4	1510944	451			Day 84
061500-PFOA-221	HILL0054.D	9.4	1539754	465			Day 84
061500-PFOA-222	HILL0055.D	9.4	1960046	611	2%	108%	Day 84
061500-PFOA-253	HILL0056.D	9.4	0	0			
061500-PFOA-254	HILL0057.D	9.4	560356	162		113%	Day 109
061500-PFOA-255	HILL0058.D	9.4	1595422	492			Day 109
061500-PFOA-256	HILL0059.D	9.4	1537794	474			Day 109
061500-PFOA-257	HILL0060.D	9.4	1535979	474			Day 109
061500-PFOA-258	HILL0061.D	9.4	1990160	641	2%	112%	Day 109
MeOH	HILL0062.D	9.4	0	0			
00028-94-00pH1.5	HILL0063.D	9.4	0	0			
00028-94-01pH1.5	HILL0064.D	9.4	79881	25	99%		
00028-94-02pH1.5	HILL0065.D	9.4	247801	72	96%		
00028-94-03pH1.5	HILL0066.D	9.4	514837	151	100%		
00028-94-04pH1.5	HILL0067.D	9.4	1001851	289	100%		
00028-94-05pH1.5	HILL0068.D	9.4	1402353	433	96%		
00028-94-06pH1.5	HILL0069.D	9.4	1903303	613	102%		
00028-94-07pH1.5	HILL0070.D	9.4	2201968	731	98%		
00028-94-08pH1.5	HILL0071.D	9.4	2518146	863	96%		
00028-94-09pH1.5	HILL0072.D	9.4	2781745	1001	100%		
MeOH	HILL0073.D	9.4	0	0			
00028-94-05pH1.5	HILL0074.D	9.4	1402132	433	96%		
MeOH	HILL0075.D	9.4	0	0			
Internal Standard Quant r2=				Mean			
Curve averaged: Linear: Include origin				S.D.			
Calibration range:				17947			
				2%			

PFOA Hydrolysis Study
 50C pH 3.0 Page 1 of 3
 Retention times (RT) in minutes
 All concentrations in ng/ml

PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	%Spike Recovery	Time Point
MeOH		9.4	0	0			
00028-95-00 pH3	HILL0012.D	9.4	0	0			
00028-95-01 pH3	HILL0013.D	9.4	82288	27	107%		
00028-95-02 pH3	HILL0014.D	9.5	269055	73	98%		
00028-95-03 pH3	HILL0015.D	9.5	579192	150	100%		
00028-95-04 pH3	HILL0016.D	9.5	1147139	302	101%		
00028-95-05 pH3	HILL0017.D	9.5	1655072	443	98%		
00028-95-06 pH3	HILL0018.D	9.5	2202783	611	102%		
00028-95-07 pH3	HILL0019.D	9.5	2587642	745	99%		
00028-95-08 pH3	HILL0020.D	9.5	3017472	901	100%		
00028-95-09 pH3	HILL0021.D	9.5	3283065	1007	101%		
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0022.D	9.5	1650531	443		98%	
MeOH		9.4	0	0			
061500-PFOA-007	HILL0023.D	9.5	0	0			
061500-PFOA-008	HILL0024.D	9.4	0	0			
061500-PFOA-009	HILL0025.D	9.4	614033	161		113%	(A)
061500-PFOA-010	HILL0026.D	9.5	2150355	601			(A)
061500-PFOA-011	HILL0027.D	9.5	1896072	519			(A)
061500-PFOA-012	HILL0028.D	9.5	2258658	634			(A)
061500-PFOA-043	HILL0029.D	9.5	2755176	802	10%	152%	(A)
061500-PFOA-044	HILL0030.D	9.4	0	0			
061500-PFOA-045	HILL0031.D	9.5	611332	163		114%	(A)
061500-PFOA-046	HILL0032.D	9.5	1905798	527			(A)
061500-PFOA-047	HILL0033.D	9.5	2206679	620			(A)
061500-PFOA-048	HILL0034.D	9.5	2221831	628			(A)
061500-PFOA-079	HILL0035.D	9.5	2752943	798	10%	144%	(A)
061500-PFOA-080	HILL0036.D	9.4	0	0			
061500-PFOA-081	HILL0037.D	9.5	604571	161		113%	(A)
061500-PFOA-082	HILL0038.D	9.5	1896593	526			(A)
061500-PFOA-083	HILL0039.D	9.5	2232596	630			(A)
061500-PFOA-084	HILL0040.D	9.5	2237534	630			(A)
MeOH		9.4	0	0			
00028-95-00 pH3	HILL0041.D	9.5	2702174	796	10%	140%	(A)
00028-95-01 pH3	HILL0042.D	9.4	0	0			
00028-95-02 pH3	HILL0043.D	9.5	81670	27	108%		
00028-95-03 pH3	HILL0044.D	9.5	266593	72	96%		
00028-95-04 pH3	HILL0045.D	9.5	568629	150	100%		
00028-95-05 pH3	HILL0046.D	9.5	1138030	301	100%		
00028-95-06 pH3	HILL0047.D	9.5	1633153	444	99%		
00028-95-07 pH3	HILL0048.D	9.5	2188592	611	102%		
00028-95-08 pH3	HILL0049.D	9.5	2570321	742	99%		
00028-95-09 pH3	HILL0050.D	9.5	3007296	890	99%		
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0051.D	9.5	3275842	997	100%	99%	
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0052.D	9.5	1631027	445			
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0053.D	9.5	0	0			
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0054.D	9.5	0	0			
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0055.D	9.5	0	0			
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0056.D	9.5	0	0			
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0057.D	9.5	0	0			
Method ID:				Internal Standard Quant (2=			
				Curve averaged: Linear. Include origin			
				Calibration range:			
				(A) Data excluded on the basis of data quality objectives; see text.			
				Mean			
				S.D.			
				%RSD			
				1%			

PFOA Hydrolysis Study Page 2 of 3
 50C pH 3.0 Retention times (RT) in minutes
 All concentrations in ng/ml

PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point
MeOH		9.4	0	0			
00028-95-00 pH3	HILL0044.D	9.4	0	0			
00028-95-01 pH3	HILL0045.D	9.4	0	0			
00028-95-02 pH3	HILL0046.D	9.5	81670	27	109%		
00028-95-03 pH3	HILL0047.D	9.5	266593	72	96%		
00028-95-04 pH3	HILL0048.D	9.5	566629	149	100%		
00028-95-05 pH3	HILL0049.D	9.5	1138030	300	100%		
00028-95-06 pH3	HILL0050.D	9.5	1633153	443	98%		
00028-95-07 pH3	HILL0051.D	9.5	2186592	610	102%		
00028-95-08 pH3	HILL0052.D	9.5	2570321	741	99%		
00028-95-09 pH3	HILL0053.D	9.5	3007296	889	99%		
00028-95-05 pH3	HILL0054.D	9.5	3275642	997	100%		
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0055.D	9.4	1631027	444		99%	
MeOH		9.4	0	0			
061500-PFOA-115	HILL0056.D	9.4	0	0			
061500-PFOA-116	HILL0058.D	9.4	0	0			
061500-PFOA-117	HILL0059.D	9.5	613665	164		115%	Day 28
061500-PFOA-118	HILL0060.D	9.5	2267521	642			Day 28
061500-PFOA-119	HILL0061.D	9.5	2205319	625			Day 28
061500-PFOA-120	HILL0062.D	9.5	2248453	635			Day 28
061500-PFOA-151	HILL0063.D	9.5	2696754	787	1%	107%	Day 28
061500-PFOA-152	HILL0064.D	9.4	0	0			
061500-PFOA-153	HILL0065.D	9.5	306384	84		59%	Day 42
061500-PFOA-154	HILL0066.D	9.5	2229287	642			Day 42
061500-PFOA-155	HILL0067.D	9.5	2291761	654			Day 42
061500-PFOA-156	HILL0068.D	9.5	2222262	632			Day 42
061500-PFOA-187	HILL0069.D	9.5	2728089	801	2%	118%	Day 42
061500-PFOA-188	HILL0070.D	9.4	0	0			
061500-PFOA-189	HILL0071.D	9.5	597768	158		111%	(A)
061500-PFOA-190	HILL0072.D	9.5	2230834	636			(A)
061500-PFOA-191	HILL0073.D	9.5	2220767	624			(A)
061500-PFOA-192	HILL0074.D	9.5	2175592	617			(A)
MeOH		9.5	2218869	634	2%	6%	(A)
00028-95-00 pH3	HILL0075.D	9.4	0	0			
00028-95-01 pH3	HILL0076.D	9.4	0	0			
00028-95-02 pH3	HILL0077.D	9.4	0	0			
00028-95-03 pH3	HILL0078.D	9.5	81990	27	109%		
00028-95-04 pH3	HILL0079.D	9.5	263046	72	96%		
00028-95-05 pH3	HILL0080.D	9.5	561799	149	100%		
00028-95-06 pH3	HILL0081.D	9.5	1126245	303	101%		
00028-95-07 pH3	HILL0082.D	9.5	1628681	450	100%		
00028-95-08 pH3	HILL0083.D	9.5	2163770	605	101%		
00028-95-09 pH3	HILL0084.D	9.5	2571272	749	100%		
00028-95-05 pH3	HILL0085.D	9.5	3008963	899	100%		
00028-95-06 pH3	HILL0086.D	9.5	3281436	1009	101%		
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0087.D	9.4	1627096	448		99%	
MeOH		9.5	0	0			
00028-95-05 pH3	HILL0088.D	9.5	0	0			
00028-95-05 pH3	HILL0089.D	9.4	0	0			
Method ID:							
Internal Standard Quant r2=				Mean	896055		
Curve averaged: Linear: Include origin				S.D.	8041		
Calibration range:				%RSD	1%		

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

PFOA Hydrolysis Study

Page 3 of 3

50C pH 3.0

Retention times (RT) in minutes

All concentrations in ng/ml

PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point
MeOH		9.5	0	0			
00028-95-00 pH3	HILL0036.D	9.5	0	0			
00028-95-01 pH3	HILL0037.D	9.5	81990	72	108%		
00028-95-02 pH3	HILL0038.D	9.5	263046	27	96%		
00028-95-03 pH3	HILL0039.D	9.5	561799	149	99%		
00028-95-04 pH3	HILL0040.D	9.5	1126245	302	101%		
00028-95-05 pH3	HILL0041.D	9.5	1628681	448	100%		
00028-95-06 pH3	HILL0042.D	9.5	2163770	601	100%		
00028-95-07 pH3	HILL0043.D	9.5	2571272	744	99%		
00028-95-08 pH3	HILL0044.D	9.5	3008963	892	99%		
00028-95-09 pH3	HILL0045.D	9.5	3281436	1000	100%		
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0047.D	9.4	1627096	445		99%	
MeOH		9.5	0	0			
061500-PFOA-223	HILL0048.D	9.4	0	0			
061500-PFOA-224	HILL0050.D	9.4	612098	163		114%	(A)
061500-PFOA-225	HILL0051.D	9.5	2228621	626			(A)
061500-PFOA-226	HILL0052.D	9.5	2239448	636			(A)
061500-PFOA-227	HILL0053.D	9.5	2240728	622			(A)
061500-PFOA-228	HILL0054.D	9.5	2760460	815	1%	131%	(A)
061500-PFOA-259	HILL0055.D	9.5	0	0			
061500-PFOA-260	HILL0056.D	9.4	616878	163		114%	Day 109
061500-PFOA-261	HILL0057.D	9.5	2250975	633			Day 109
061500-PFOA-262	HILL0058.D	9.5	2240681	627			Day 109
061500-PFOA-263	HILL0059.D	9.5	2271686	645			Day 109
061500-PFOA-264	HILL0060.D	9.5	2756815	808	1%	121%	Day 109
MeOH		9.4	0	0			
00028-95-00 pH3	HILL0062.D	9.4	0	0			
00028-95-01 pH3	HILL0063.D	9.4	82634	27	105%		
00028-95-02 pH3	HILL0064.D	9.5	263401	72	96%		
00028-95-03 pH3	HILL0065.D	9.5	566621	150	100%		
00028-95-04 pH3	HILL0066.D	9.5	1133921	302	101%		
00028-95-05 pH3	HILL0067.D	9.5	1637254	445	99%		
00028-95-06 pH3	HILL0068.D	9.5	2208136	619	103%		
00028-95-07 pH3	HILL0069.D	9.5	2589080	740	99%		
00028-95-08 pH3	HILL0070.D	9.5	3039283	895	100%		
00028-95-09 pH3	HILL0071.D	9.5	3335024	1010	101%		
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0072.D	9.4	1640712	446		99%	
MeOH		9.4	0	0			
00028-95-05 pH3	HILL0073.D	9.4	0	0			
00028-95-05 pH3	HILL0074.D	9.4	0	0			
Method ID:							
Internal Standard Quant (2=				Mean	895323		
Curve averaged: Linear: Include origin				S.D.	5879		
Calibration range:				%RSD	1%		

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

PFOA Hydrolysis Study
50C pH 5.0 Page 1 of 3
Retention times (RT) in minutes
All concentrations in ng/ml

		PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	RT	Area
00028-96-00 pH5	HILL0013.D	9.4	0	0				8.1	0
00028-96-01 pH5	HILL0014.D	9.4	82475	22	89%			8.1	921389
00028-96-02 pH5	HILL0015.D	9.5	255658	70	93%			8.2	915385
00028-96-03 pH5	HILL0016.D	9.5	562034	157	104%			8.2	924155
00028-96-04 pH5	HILL0017.D	9.5	1058309	305	102%			8.2	923989
00028-96-05 pH5	HILL0018.D	9.5	1526761	455	101%			8.2	917918
00028-96-06 pH5	HILL0019.D	9.5	2024353	620	104%			8.2	912758
00028-96-07 pH5	HILL0020.D	9.5	2321582	718	96%			8.2	916325
00028-96-08 pH5	HILL0021.D	9.5	2786856	903	100%			8.2	924386
00028-96-09 pH5	HILL0022.D	9.5	3056755	1021	102%			8.2	915970
00028-96-05 pH5	HILL0023.D	9.4	0	0				8.2	909916
00028-96-05 pH5	HILL0024.D	9.5	1515645	449		100%		8.2	0
00028-96-05 pH5	HILL0025.D	9.5	0	0				8.1	917581
00028-96-05 pH5	HILL0026.D	9.4	0	0				8.2	0
061500-PFOA-013	HILL0027.D	9.4	0	0				8.1	920231
061500-PFOA-014	HILL0028.D	9.5	574463	161		113%	Day 0	8.2	919417
061500-PFOA-015	HILL0029.D	9.5	2136301	655			Day 0	8.2	921753
061500-PFOA-016	HILL0030.D	9.5	2146831	659			Day 0	8.2	921718
061500-PFOA-017	HILL0031.D	9.5	2133454	657			Day 0	8.2	918250
061500-PFOA-018	HILL0032.D	9.5	2608029	822	0%	115%	Day 0	8.2	925469
061500-PFOA-049	HILL0033.D	9.4	0	0				8.2	922553
061500-PFOA-050	HILL0034.D	9.5	606301	169		118%	Day 7	8.2	927505
061500-PFOA-051	HILL0035.D	9.5	2158146	654			Day 7	8.2	932092
061500-PFOA-052	HILL0036.D	9.5	2167566	657			Day 7	8.2	932872
061500-PFOA-053	HILL0037.D	9.5	2181107	657			Day 7	8.2	938261
061500-PFOA-054	HILL0038.D	9.5	2647149	828	0%	120%	Day 7	8.2	934544
061500-PFOA-085	HILL0039.D	9.4	0	0				8.2	940621
061500-PFOA-086	HILL0040.D	9.5	607581	168		117%	Day 14	8.2	933688
061500-PFOA-087	HILL0041.D	9.5	2184729	654			Day 14	8.2	944180
061500-PFOA-088	HILL0042.D	9.5	2158052	657			Day 14	8.2	928637
061500-PFOA-089	HILL0043.D	9.5	2152055	657			Day 14	8.2	925961
061500-PFOA-090	HILL0044.D	9.5	2605276	819	0%	114%	Day 14	8.2	925366
00028-96-00 pH5	HILL0045.D	9.4	0	0				8.2	0
00028-96-01 pH5	HILL0046.D	9.4	0	0				8.1	940846
00028-96-02 pH5	HILL0047.D	9.5	84676	22	89%			8.2	941774
00028-96-03 pH5	HILL0048.D	9.5	260410	69	92%			8.2	951765
00028-96-04 pH5	HILL0049.D	9.5	571088	155	103%			8.2	950664
00028-96-05 pH5	HILL0050.D	9.5	1096775	306	102%			8.2	949249
00028-96-06 pH5	HILL0051.D	9.5	1553008	449	100%			8.2	939795
00028-96-07 pH5	HILL0052.D	9.5	2059026	610	102%			8.2	945478
00028-96-08 pH5	HILL0053.D	9.5	2349365	709	95%			8.2	945728
00028-96-09 pH5	HILL0054.D	9.5	2880656	901	100%			8.2	948113
00028-96-09 pH5	HILL0055.D	9.5	3135872	1001	100%			8.2	948233
00028-96-05 pH5	HILL0056.D	9.4	0	0				8.2	0
00028-96-05 pH5	HILL0057.D	9.5	1562785	453		101%		8.2	947886
00028-96-05 pH5	HILL0058.D	9.4	0	0				8.2	0

Method ID: MeOH
Internal Standard Quant r2= 0.9999
Curve averaged: Linear. Include origin
Calibration range: 0.000000 to 1000000.000000

Mean
S.D.
%RSD

PFOA Hydrolysis Study
50C pH 5.0 Page 2 of 3
Retention times (RT) in minutes
All concentrations in ng/ml

PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point
MeOH		9.5	0	0			
00028-96-00 pH5	HILL0045.D	9.5	0	0			8.1
00028-96-01 pH5	HILL0046.D	9.5	84676	23	91%		8.2
00028-96-02 pH5	HILL0047.D	9.5	260410	70	94%		8.2
00028-96-03 pH5	HILL0048.D	9.5	571088	156	104%		8.2
00028-96-04 pH5	HILL0049.D	9.5	1096775	302	101%		8.2
00028-96-05 pH5	HILL0050.D	9.5	1553008	446	99%		8.2
00028-96-06 pH5	HILL0051.D	9.5	2059026	612	102%		8.2
00028-96-07 pH5	HILL0052.D	9.5	2349365	713	95%		8.2
00028-96-08 pH5	HILL0053.D	9.5	2880656	898	100%		8.2
00028-96-09 pH5	HILL0054.D	9.5	3135872	990	99%		8.2
MeOH		9.5	0	0			8.1
00028-96-05 pH5	HILL0055.D	9.5	1562785	452	100%		8.2
MeOH		9.5	0	0			8.1
061500-PFOA-121	HILL0056.D	9.5	0	0			8.2
061500-PFOA-122	HILL0057.D	9.5	605920	166	116%		8.2
061500-PFOA-123	HILL0058.D	9.5	2183595	656			8.2
061500-PFOA-124	HILL0059.D	9.5	2158582	652			8.2
061500-PFOA-125	HILL0060.D	9.5	2189482	656			8.2
061500-PFOA-126	HILL0061.D	9.5	2631516	823	0%		8.2
061500-PFOA-157	HILL0062.D	9.5	0	0			8.2
061500-PFOA-158	HILL0063.D	9.5	614384	168	118%		8.2
061500-PFOA-159	HILL0064.D	9.5	2177138	652			8.2
061500-PFOA-160	HILL0065.D	9.5	2203841	664			8.2
061500-PFOA-161	HILL0066.D	9.5	0	0			8.2
061500-PFOA-162	HILL0067.D	9.5	605707	166	116%		8.2
061500-PFOA-193	HILL0070.D	9.5	2166222	651			8.2
061500-PFOA-194	HILL0071.D	9.5	2631811	816	1%		8.2
061500-PFOA-195	HILL0072.D	9.5	2170827	651			8.2
061500-PFOA-196	HILL0073.D	9.5	2143552	643			8.2
061500-PFOA-197	HILL0074.D	9.5	2135796	635			8.2
061500-PFOA-198	HILL0075.D	9.5	2616028	810	1%		8.2
MeOH		9.5	0	0			8.1
00028-96-00 pH5	HILL0076.D	9.5	0	0			8.2
00028-96-01 pH5	HILL0077.D	9.5	86643	23	91%		8.2
00028-96-02 pH5	HILL0078.D	9.5	262432	70	93%		8.2
00028-96-03 pH5	HILL0079.D	9.5	570398	155	104%		8.2
00028-96-04 pH5	HILL0080.D	9.5	1088630	308	103%		8.2
00028-96-05 pH5	HILL0081.D	9.5	1546530	453	101%		8.2
00028-96-06 pH5	HILL0082.D	9.5	2054021	617	103%		8.2
00028-96-07 pH5	HILL0083.D	9.5	2310100	718	96%		8.2
00028-96-08 pH5	HILL0084.D	9.5	2796128	917	102%		8.2
00028-96-09 pH5	HILL0085.D	9.5	3056737	1021	102%		8.2
MeOH		9.5	0	0			8.1
00028-96-05 pH5	HILL0086.D	9.5	1542658	442	98%		8.2
MeOH		9.5	0	0			8.1
061500-PFOA-199	HILL0087.D	9.5	0	0			8.2
061500-PFOA-200	HILL0088.D	9.5	0	0			8.2
061500-PFOA-201	HILL0089.D	9.5	0	0			8.2
061500-PFOA-202	HILL0090.D	9.5	0	0			8.2
Method ID:				Mean			
				S.D.			
				%RSD			
				948384			
				5145			
				1%			
				Calibration range:			
				Internal Standard Quant r2=			
				Curve averaged: Linear: Include origin			

PFOA Hydrolysis Study
50C pH 5.0 Page 3 of 3
Retention times (RT) in minutes
All concentrations in ng/ml

PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point
MeOH	HILL0077.D	9.5	0	0			
00028-96-00 pH5	HILL0078.D	9.5	0	0			
00028-96-01 pH5	HILL0079.D	9.5	86643	22	89%		
00028-96-02 pH5	HILL0080.D	9.5	262432	70	93%		
00028-96-03 pH5	HILL0081.D	9.5	570398	155	103%		
00028-96-04 pH5	HILL0082.D	9.5	1088630	301	100%		
00028-96-05 pH5	HILL0083.D	9.5	1546530	446	99%		
00028-96-06 pH5	HILL0084.D	9.5	2054021	613	102%		
00028-96-07 pH5	HILL0085.D	9.5	2310100	715	95%		
00028-96-08 pH5	HILL0086.D	9.5	2796128	902	100%		
00028-96-09 pH5	HILL0087.D	9.5	3056737	996	100%		
MeOH	HILL0088.D	9.4	0	0			
00028-96-05 pH5	HILL0089.D	9.5	1542658	442	98%		
MeOH	HILL0090.D	9.4	0	0			
061500-PFOA-229	HILL0091.D	9.4	0	0			
061500-PFOA-230	HILL0092.D	9.5	608076	163	114%		Day 84
061500-PFOA-231	HILL0093.D	9.5	2176525	659			Day 84
061500-PFOA-232	HILL0094.D	9.5	2174338	659			Day 84
061500-PFOA-233	HILL0095.D	9.5	2188043	652			Day 84
061500-PFOA-234	HILL0096.D	9.5	2641765	818	1%		Day 84
061500-PFOA-265	HILL0097.D	9.4	0	0			
061500-PFOA-266	HILL0098.D	9.5	614037	165	115%		Day 109
061500-PFOA-267	HILL0099.D	9.5	2165828	646			Day 109
061500-PFOA-268	HILL0100.D	9.5	2158127	645			Day 109
061500-PFOA-269	HILL0101.D	9.5	2158778	646			Day 109
061500-PFOA-270	HILL0102.D	9.5	2614502	815	0%		Day 109
MeOH	HILL0103.D	9.4	0	0			
00028-96-00 pH5	HILL0104.D	9.4	0	0			
00028-96-01 pH5	HILL0105.D	9.5	85429	22	87%		
00028-96-02 pH5	HILL0106.D	9.5	272871	72	95%		
00028-96-03 pH5	HILL0107.D	9.5	591035	158	106%		
00028-96-04 pH5	HILL0108.D	9.5	1112890	309	103%		
00028-96-05 pH5	HILL0109.D	9.5	1573750	450	100%		
00028-96-06 pH5	HILL0110.D	9.5	2082125	617	103%		
00028-96-07 pH5	HILL0111.D	9.5	2358719	719	96%		
00028-96-08 pH5	HILL0112.D	9.5	2859084	914	102%		
00028-96-09 pH5	HILL0113.D	9.5	3126850	1013	101%		
MeOH	HILL0114.D	9.4	0	0			
00028-96-05 pH5	HILL0115.D	9.5	1586479	453		101%	
MeOH	HILL0116.D	9.4	0	0			
Internal Standard Quant (2=				Mean	953207		
Curve averaged: Linear: Include origin				S.D.	6605		
Calibration range:				%RSD	1%		

PFOA Hydrolysis Study
50C pH 7.0 Page 1 of 3
Retention times (RT) in minutes
All concentrations in ng/ml

PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point
MeOH		9.5	0	0			
00028-97-00 pH7	HILL0013.D	9.5	0	0			
00028-97-01 pH7	HILL0015.D	9.5	89549	27	110%		
00028-97-02 pH7	HILL0016.D	9.5	273000	74	98%		
00028-97-03 pH7	HILL0017.D	9.5	564682	149	99%		
00028-97-04 pH7	HILL0018.D	9.5	1118278	300	100%		
00028-97-05 pH7	HILL0019.D	9.5	1584297	436	97%		
00028-97-06 pH7	HILL0020.D	9.5	2153006	616	103%		
00028-97-07 pH7	HILL0021.D	9.5	2550401	749	100%		
00028-97-08 pH7	HILL0022.D	9.5	2901395	880	98%		
00028-97-09 pH7	HILL0023.D	9.5	3194702	1004	100%		
MeOH		9.5	0	0			
00028-97-05 pH7	HILL0024.D	9.5	1599875	440		98%	
MeOH		9.5	0	0			
061500-PFOA-019	HILL0027.D	9.5	0	0			
061500-PFOA-020	HILL0028.D	9.5	623804	165		116%	Day 0
061500-PFOA-021	HILL0029.D	9.5	2274515	662			Day 0
061500-PFOA-022	HILL0030.D	9.5	2249080	645			Day 0
061500-PFOA-023	HILL0031.D	9.5	2269461	652			Day 0
061500-PFOA-024	HILL0032.D	9.5	2742883	818	1%	116%	Day 0
061500-PFOA-055	HILL0033.D	9.5	0	0			
061500-PFOA-056	HILL0034.D	9.5	619990	166		116%	Day 7
061500-PFOA-057	HILL0035.D	9.5	2255587	648			Day 7
061500-PFOA-058	HILL0036.D	9.5	2246779	648			Day 7
061500-PFOA-059	HILL0037.D	9.5	2234292	650			Day 7
061500-PFOA-060	HILL0038.D	9.5	2714705	816	0%	117%	Day 7
061500-PFOA-091	HILL0039.D	9.5	0	0			
061500-PFOA-092	HILL0040.D	9.5	621311	166		116%	Day 14
061500-PFOA-093	HILL0041.D	9.5	2242645	653			Day 14
061500-PFOA-094	HILL0042.D	9.5	2192603	633			Day 14
061500-PFOA-095	HILL0043.D	9.5	2182058	641			Day 14
061500-PFOA-096	HILL0044.D	9.5	2717288	822	2%	125%	Day 14
MeOH		9.5	0	0			
00028-97-00 pH7	HILL0045.D	9.5	0	0			
00028-97-01 pH7	HILL0046.D	9.5	87291	27	108%		
00028-97-02 pH7	HILL0047.D	9.5	271707	74	98%		
00028-97-03 pH7	HILL0048.D	9.5	556644	150	100%		
00028-97-04 pH7	HILL0049.D	9.5	1104538	301	100%		
00028-97-05 pH7	HILL0051.D	9.5	1574928	443	99%		
00028-97-06 pH7	HILL0052.D	9.5	2120131	617	103%		
00028-97-07 pH7	HILL0053.D	9.5	2501752	746	100%		
00028-97-08 pH7	HILL0054.D	9.5	2834850	890	99%		
00028-97-09 pH7	HILL0055.D	9.5	3142471	1010	101%		
MeOH		9.5	0	0			
00028-97-05 pH7	HILL0056.D	9.5	1571464	437		97%	
MeOH		9.5	0	0			
00028-97-05 pH7	HILL0058.D	9.5	0	0			

Method ID: Internal Standard Quant (I2=)
Curve averaged: Linear. Include origin
Calibration range:
(A) Data excluded on the basis of data quality objectives; see text.

Mean
S.D.
%RSD

965889
11537
1%

PFOA										PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	RT	Area	Conc.			
00028-97-00 pH7 MeOH	HILL0045.D	9.5	0	0				8.1	0	0			
00028-97-01 pH7	HILL0046.D	9.5	0	0				8.2	995126	158			
00028-97-02 pH7	HILL0047.D	9.5	87291	28	110%			8.2	970814	158			
00028-97-03 pH7	HILL0048.D	9.5	271707	74	99%			8.2	966617	158			
00028-97-04 pH7	HILL0049.D	9.5	556844	150	100%			8.2	953431	158			
00028-97-05 pH7	HILL0050.D	9.5	1174528	300	100%			8.2	955535	158			
00028-97-05 pH7	HILL0051.D	9.5	1574938	443	98%			8.2	949127	158			
00028-97-06 pH7	HILL0052.D	9.5	2120131	618	103%			8.2	952380	158			
00028-97-07 pH7	HILL0053.D	9.5	2501752	748	100%			8.2	957556	158			
00028-97-08 pH7	HILL0054.D	9.5	2834850	895	100%			8.2	941774	158			
00028-97-09 pH7	HILL0055.D	9.5	3142471	1018	102%			8.2	948270	158			
MeOH	HILL0056.D	9.5	0	0				8.1	0	0			
00028-97-05 pH7	HILL0057.D	9.5	1571464	437	97%			8.2	958489	158			
MeOH	HILL0058.D	9.5	0	0				8.1	0	0			
061500-PFOA-127	HILL0059.D	9.5	0	0				8.2	946761	158			
061500-PFOA-128	HILL0060.D	9.5	605620	163		114%	Day 28	8.2	952433	158			
061500-PFOA-129	HILL0061.D	9.5	2204642	649			Day 28	8.2	949777	158			
061500-PFOA-130	HILL0062.D	9.5	2192955	642			Day 28	8.2	953745	158			
061500-PFOA-131	HILL0063.D	9.5	2206098	645			Day 28	8.2	955727	158			
061500-PFOA-132	HILL0064.D	9.5	2699217	828	1%	128%	Day 28	8.2	952217	158			
061500-PFOA-163	HILL0065.D	9.5	0	0				8.2	949483	158			
061500-PFOA-164	HILL0066.D	9.5	602566	163		114%	Day 42	8.2	946443	158			
061500-PFOA-165	HILL0067.D	9.5	2185187	654			Day 42	8.2	935651	158			
061500-PFOA-166	HILL0068.D	9.5	2190101	650			Day 42	8.2	942456	158			
061500-PFOA-167	HILL0069.D	9.5	2194629	647			Day 42	8.2	947731	158			
061500-PFOA-168	HILL0070.D	9.5	2644173	814	1%	115%	Day 42	8.2	945474	158			
061500-PFOA-199	HILL0071.D	9.5	0	0				8.2	947388	158			
061500-PFOA-200	HILL0072.D	9.5	605657	164		114%	Day 64	8.2	947990	158			
061500-PFOA-201	HILL0073.D	9.5	2167144	652			Day 64	8.2	929447	158			
061500-PFOA-202	HILL0074.D	9.5	2213132	655			Day 64	8.2	946316	158			
061500-PFOA-203	HILL0075.D	9.5	2214971	643			Day 64	8.3	961798	158			
061500-PFOA-204	HILL0076.D	9.5	2656837	820	1%	119%	Day 64	8.2	944627	158			
MeOH	HILL0077.D	9.5	0	0				8.1	0	0			
00028-97-00 pH7	HILL0078.D	9.4	0	0				8.2	976974	158			
00028-97-01 pH7	HILL0079.D	9.5	85517	27	110%			8.2	956841	158			
00028-97-02 pH7	HILL0080.D	9.5	266273	74	98%			8.2	950814	158			
00028-97-03 pH7	HILL0081.D	9.5	546536	149	100%			8.2	938192	158			
00028-97-04 pH7	HILL0082.D	9.5	1086341	299	100%			8.3	943983	158			
00028-97-05 pH7	HILL0083.D	9.5	1541845	436	97%			8.2	943263	158			
00028-97-06 pH7	HILL0084.D	9.5	2069653	613	102%			8.3	935696	158			
00028-97-07 pH7	HILL0085.D	9.5	2447183	751	100%			8.3	933778	158			
00028-97-08 pH7	HILL0086.D	9.5	2719965	880	98%			8.2	935659	158			
00028-97-09 pH7	HILL0087.D	9.5	3006154	991	99%			8.3	925184	158			
MeOH	HILL0088.D	9.5	0	0				8.1	0	0			
00028-97-05 pH7	HILL0089.D	9.5	1524429	436	97%			8.3	930947	158			
MeOH	HILL0090.D	9.5	0	0				8.1	0	0			
Method ID:										Mean			
Internal Standard Quant Z=										949398			
Curve averaged: Linear. Include origin										S.D. 13021			
Calibration range:										%RSD 1%			

PFOA Hydrolysis Study
50C pH
Retention times (RT) in minutes
All concentrations in ng/ml

Page 3 of 3

PFOA				PFBS			
Sample ID	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	RT
MeOH	9.5	0	0				8.2
00028-97-00 pH7	9.5	0	0				8.2
00028-97-01 pH7	9.5	85517	27	110%			8.2
00028-97-02 pH7	9.5	266273	74	99%			8.2
00028-97-03 pH7	9.5	546536	150	100%			8.2
00028-97-04 pH7	9.5	1086341	300	100%			8.3
00028-97-05 pH7	9.5	1541845	437	97%			8.3
00028-97-06 pH7	9.5	2069653	616	103%			8.3
00028-97-07 pH7	9.5	2447183	734	101%			8.3
00028-97-08 pH7	9.5	2779965	884	98%			8.2
00028-97-09 pH7	9.5	3006154	996	100%			8.3
MeOH	9.5	0	0				8.3
00028-97-05 pH7	9.5	1524429	438	97%			8.3
MeOH	9.5	0	0				8.3
061500-PFOA-235	9.5	595311	166	116%		Day 84	8.3
061500-PFOA-236	9.5	2174118	648			Day 84	8.3
061500-PFOA-237	9.5	2168837	652			Day 84	8.3
061500-PFOA-238	9.5	2176750	657			Day 84	8.2
061500-PFOA-239	9.5	2587928	821	1%		Day 84	8.2
061500-PFOA-240	9.5	0	0				8.3
061500-PFOA-271	9.5	602602	167			Day 109	8.3
061500-PFOA-272	9.5	2146703	651	117%		Day 109	8.3
061500-PFOA-273	9.5	2137239	645			Day 109	8.3
061500-PFOA-274	9.5	2138359	645			Day 109	8.3
061500-PFOA-275	9.5	2596175	818	1%		Day 109	8.2
061500-PFOA-276	9.5	0	0				8.2
MeOH	9.5	0	0				8.2
00028-97-00 pH7	9.5	0	0				8.3
00028-97-01 pH7	9.5	84002	28	110%			8.3
00028-97-02 pH7	9.5	259555	73	98%			8.2
00028-97-03 pH7	9.5	541262	149	99%			8.3
00028-97-04 pH7	9.5	1067145	300	100%			8.2
00028-97-05 pH7	9.5	1526624	439	98%			8.2
00028-97-06 pH7	9.5	2052935	613	102%			8.3
00028-97-07 pH7	9.5	2429369	749	100%			8.2
00028-97-08 pH7	9.5	2778668	890	99%			8.3
00028-97-09 pH7	9.5	3050552	1012	101%			8.2
MeOH	9.5	0	0				8.2
00028-97-05 pH7	9.5	1519453	443	98%			8.2
MeOH	9.5	0	0				8.2
061500-PFOA-235	9.5	595311	166	116%		Day 84	8.3
061500-PFOA-236	9.5	2174118	648			Day 84	8.3
061500-PFOA-237	9.5	2168837	652			Day 84	8.3
061500-PFOA-238	9.5	2176750	657			Day 84	8.2
061500-PFOA-239	9.5	2587928	821	1%		Day 84	8.2
061500-PFOA-240	9.5	0	0				8.3
061500-PFOA-271	9.5	602602	167			Day 109	8.3
061500-PFOA-272	9.5	2146703	651	117%		Day 109	8.3
061500-PFOA-273	9.5	2137239	645			Day 109	8.3
061500-PFOA-274	9.5	2138359	645			Day 109	8.3
061500-PFOA-275	9.5	2596175	818	1%		Day 109	8.2
061500-PFOA-276	9.5	0	0				8.2
MeOH	9.5	0	0				8.2
00028-97-00 pH7	9.5	0	0				8.3
00028-97-01 pH7	9.5	84002	28	110%			8.3
00028-97-02 pH7	9.5	259555	73	98%			8.2
00028-97-03 pH7	9.5	541262	149	99%			8.3
00028-97-04 pH7	9.5	1067145	300	100%			8.2
00028-97-05 pH7	9.5	1526624	439	98%			8.2
00028-97-06 pH7	9.5	2052935	613	102%			8.3
00028-97-07 pH7	9.5	2429369	749	100%			8.2
00028-97-08 pH7	9.5	2778668	890	99%			8.3
00028-97-09 pH7	9.5	3050552	1012	101%			8.2
MeOH	9.5	0	0				8.2
00028-97-05 pH7	9.5	1519453	443	98%			8.2
MeOH	9.5	0	0				8.2
061500-PFOA-235	9.5	595311	166	116%		Day 84	8.3
061500-PFOA-236	9.5	2174118	648			Day 84	8.3
061500-PFOA-237	9.5	2168837	652			Day 84	8.3
061500-PFOA-238	9.5	2176750	657			Day 84	8.2
061500-PFOA-239	9.5	2587928	821	1%		Day 84	8.2
061500-PFOA-240	9.5	0	0				8.3
061500-PFOA-271	9.5	602602	167			Day 109	8.3
061500-PFOA-272	9.5	2146703	651	117%		Day 109	8.3
061500-PFOA-273	9.5	2137239	645			Day 109	8.3
061500-PFOA-274	9.5	2138359	645			Day 109	8.3
061500-PFOA-275	9.5	2596175	818	1%		Day 109	8.2
061500-PFOA-276	9.5	0	0				8.2
MeOH	9.5	0	0				8.2
00028-97-00 pH7	9.5	0	0				8.3
00028-97-01 pH7	9.5	84002	28	110%			8.3
00028-97-02 pH7	9.5	259555	73	98%			8.2
00028-97-03 pH7	9.5	541262	149	99%			8.3
00028-97-04 pH7	9.5	1067145	300	100%			8.2
00028-97-05 pH7	9.5	1526624	439	98%			8.2
00028-97-06 pH7	9.5	2052935	613	102%			8.3
00028-97-07 pH7	9.5	2429369	749	100%			8.2
00028-97-08 pH7	9.5	2778668	890	99%			8.3
00028-97-09 pH7	9.5	3050552	1012	101%			8.2
MeOH	9.5	0	0				8.2
00028-97-05 pH7	9.5	1519453	443	98%			8.2
MeOH	9.5	0	0				8.2
061500-PFOA-235	9.5	595311	166	116%		Day 84	8.3
061500-PFOA-236	9.5	2174118	648			Day 84	8.3
061500-PFOA-237	9.5	2168837	652			Day 84	8.3
061500-PFOA-238	9.5	2176750	657			Day 84	8.2
061500-PFOA-239	9.5	2587928	821	1%		Day 84	8.2
061500-PFOA-240	9.5	0	0				8.3
061500-PFOA-271	9.5	602602	167			Day 109	8.3
061500-PFOA-272	9.5	2146703	651	117%		Day 109	8.3
061500-PFOA-273	9.5	2137239	645			Day 109	8.3
061500-PFOA-274	9.5	2138359	645			Day 109	8.3
061500-PFOA-275	9.5	2596175	818	1%		Day 109	8.2
061500-PFOA-276	9.5	0	0				8.2
MeOH	9.5	0	0				8.2
00028-97-00 pH7	9.5	0	0				8.3
00028-97-01 pH7	9.5	84002	28	110%			8.3
00028-97-02 pH7	9.5	259555	73	98%			8.2
00028-97-03 pH7	9.5	541262	149	99%			8.3
00028-97-04 pH7	9.5	1067145	300	100%			8.2
00028-97-05 pH7	9.5	1526624	439	98%			8.2
00028-97-06 pH7	9.5	2052935	613	102%			8.3
00028-97-07 pH7	9.5	2429369	749	100%			8.2
00028-97-08 pH7	9.5	2778668	890	99%			8.3
00028-97-09 pH7	9.5	3050552	1012	101%			8.2
MeOH	9.5	0	0				8.2
00028-97-05 pH7	9.5	1519453	443	98%			8.2
MeOH	9.5	0	0				8.2
061500-PFOA-235	9.5	595311	166	116%		Day 84	8.3
061500-PFOA-236	9.5	2174118	648			Day 84	8.3
061500-PFOA-237	9.5	2168837	652			Day 84	8.3
061500-PFOA-238	9.5	2176750	657			Day 84	8.2
061500-PFOA-239	9.5	2587928	821	1%		Day 84	8.2
061500-PFOA-240	9.5	0	0				8.3
061500-PFOA-271	9.5	602602	167			Day 109	8.3
061500-PFOA-272	9.5	2146703	651	117%		Day 109	8.3
061500-PFOA-273	9.5	2137239	645			Day 109	8.3
061500-PFOA-274	9.5	2138359	645			Day 109	8.3
061500-PFOA-275	9.5	2596175	818	1%		Day 109	8.2
061500-PFOA-276	9.5	0	0				8.2
MeOH	9.5	0	0				8.2
00028-97-00 pH7	9.5	0	0				8.3
00028-97-01 pH7	9.5	84002	28	110%			8.3
00028-97-02 pH7	9.5	259555	73	98%			8.2
00028-97-03 pH7	9.5	541262	149	99%			8.3
00028-97-04 pH7	9.5	1067145	300	100%			8.2
00028-97-05 pH7	9.5	1526624	439	98%			8.2
00028-97-06 pH7	9.5	2052935	613	102%			8.3
00028-97-07 pH7	9.5	2429369	749	100%			8.2
00028-97-08 pH7	9.5	2778668	890	99%			8.3
00028-97-09 pH7	9.5	3050552	1012	101%			8.2
MeOH	9.5	0	0				8.2
00028-97-05 pH7	9.5	1519453	443	98%			8.2
MeOH	9.5	0	0				8.2
061500-PFOA-235	9.5	595311	166	116%		Day 84	8.3
061500-PFOA-236	9.5	2174118	648			Day 84	8.3
061500-PFOA-237	9.5	2168837	652			Day 84	8.3
061500-PFOA-238	9.5	2176750	657			Day 84	8.2
061500-PFOA-239	9.5	2587928	821	1%		Day 84	8.2
061500-PFOA-240	9.5	0	0				8.3
061500-PFOA-271	9.5	602602	167			Day 109	8.3
061500-PFOA-272	9.5	2146703	651	117%		Day 109	8.3
061500-PFOA-273	9.5	2137239	645			Day 109	8.3
061500-PFOA-274	9.5	2138359	645			Day 109	8.3
061500-PFOA-275	9.5	2596175	818	1%		Day 109	8.2
061500-PFOA-276	9.5	0	0				8.2
MeOH	9.5	0	0				8.2
00028-97-00 pH7	9.5	0	0				8.3
00028-97-01 pH7	9.5	84002	28	110%			8.3
00028-97-02 pH7	9.5	259555	73	98%			8.2
00028-97-03 pH7	9.5	541262	149	99%			8.3
00028-97-04 pH7	9.5	1067145	300	100%			8.2
00028-97-05 pH7	9.5	1526624	439	98%			8.2
00028-97-06 pH7	9.5	2052935	613	102%			8.3
00028-97-07 pH7	9.5	2429369	749	100%			8.2
00028-97-08 pH7	9.5	2778668	890	99%			8.3
00028-97-09 pH7	9.5						

PFOA Hydrolysis Study
 50C pH 9.0
 Retention times (RT) in minutes
 All concentrations in ng/ml

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PFOA				PFBS						
Sample I.D.	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	RT	Area	Conc.
MeOH	HILL0002.D	9.5	0	0				8.2	0	0.0
00028-98-00 pH9	HILL0003.D	9.5	0	0				8.2	919569	158
00028-98-01 pH9	HILL0004.D	9.5	81677	25	98%			8.2	922127	158
00028-98-02 pH9	HILL0005.D	9.5	263406	73	97%			8.2	913873	158
00028-98-03 pH9	HILL0006.D	9.5	547426	148	99%			8.2	919337	158
00028-98-04 pH9	HILL0007.D	9.5	1080831	298	99%			8.3	912713	158
00028-98-05 pH9	HILL0008.D	9.5	1551837	434	96%			8.2	919513	158
00028-98-06 pH9	HILL0009.D	9.5	2097408	603	101%			8.2	919055	158
00028-98-07 pH9	HILL0010.D	9.5	2492449	740	99%			8.2	911116	158
00028-98-08 pH9	HILL0011.D	9.5	2883027	879	98%			8.2	909972	158
00028-98-09 pH9	HILL0012.D	9.5	3151926	991	99%			8.2	901274	158
MeOH	HILL0013.D	9.5	0	0				8.2	0	0
00028-98-05 pH9	HILL0014.D	9.5	1545140	435	97%			8.2	913746	158
MeOH	HILL0015.D	9.5	0	0				8.2	0	0
061500-PFOA-025	HILL0016.D	9.5	0	0				8.2	893446	158
061500-PFOA-026	HILL0017.D	9.5	611497	168		117%	Day 0	8.2	906490	158
061500-PFOA-027	HILL0018.D	9.5	2234987	661			Day 0	8.2	902721	158
061500-PFOA-028	HILL0019.D	9.5	2204470	643			Day 0	8.2	912251	158
061500-PFOA-029	HILL0020.D	9.5	2240142	656			Day 0	8.2	910419	158
061500-PFOA-030	HILL0021.D	9.5	2657993	806	1%	107%	Day 0	8.2	903282	158
061500-PFOA-061	HILL0022.D	9.5	0	0				8.2	906146	158
061500-PFOA-062	HILL0023.D	9.5	623086	171		119%	Day 7	8.3	908411	158
061500-PFOA-063	HILL0024.D	9.5	2248154	663			Day 7	8.2	905087	158
061500-PFOA-064	HILL0025.D	9.5	2237255	662			Day 7	8.2	901785	158
061500-PFOA-065	HILL0026.D	9.5	2250744	659			Day 7	8.2	911263	158
061500-PFOA-066	HILL0027.D	9.5	2715890	807	0%	102%	Day 7	8.2	921345	158
061500-PFOA-097	HILL0028.D	9.5	0	0				8.2	915788	158
061500-PFOA-098	HILL0029.D	9.5	627431	171		120%	Day 14	8.2	910482	158
061500-PFOA-099	HILL0030.D	9.5	2266363	669			Day 14	8.2	904859	158
061500-PFOA-100	HILL0031.D	9.5	2264539	668			Day 14	8.2	905811	158
061500-PFOA-101	HILL0032.D	9.5	2246880	662			Day 14	8.2	905698	158
061500-PFOA-102	HILL0033.D	9.5	2735222	824	1%	110%	Day 14	8.2	912348	158
MeOH	HILL0034.D	9.5	0	0				8.2	0	0
00028-98-00 pH9	HILL0035.D	9.5	0	0				8.2	911956	158
00028-98-01 pH9	HILL0036.D	9.5	84910	26	103%			8.2	906017	158
00028-98-02 pH9	HILL0037.D	9.5	273512	75	100%			8.2	920039	158
00028-98-03 pH9	HILL0038.D	9.5	569596	155	103%			8.2	916507	158
00028-98-04 pH9	HILL0039.D	9.5	1116548	309	103%			8.2	912425	158
00028-98-05 pH9	HILL0040.D	9.5	1561236	445	99%			8.2	903511	158
00028-98-06 pH9	HILL0041.D	9.5	2104613	614	102%			8.2	907628	158
00028-98-07 pH9	HILL0042.D	9.5	2519089	761	102%			8.2	899479	158
00028-98-08 pH9	HILL0043.D	9.5	2937090	904	101%			8.2	906115	158
00028-98-09 pH9	HILL0044.D	9.5	3225468	1015	102%			8.2	904792	158
MeOH	HILL0045.D	9.5	0	0				8.2	0	0
00028-98-05 pH9	HILL0046.D	9.5	1562031	442	98%			8.2	908839	158
MeOH	HILL0047.D	9.5	0	0				8.2	0	0

Method ID: Internal Standard Quant (2=)
 Curve averaged: Linear. Include origin
 Calibration range:

Mean 909831
 S.D. 6626
 %RSD 1%

PFOA Hydrolysis Study

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50C pH 9.0

Retention times (RT) in minutes

All concentrations in ng/ml

PFOA				PFBS			
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point
MeOH		9.5	0	0			
00028-98-00 pH9	HILL0034.D	9.5	0	0			
00028-98-01 pH9	HILL0035.D	9.5	84910	24	98%		
00028-98-02 pH9	HILL0036.D	9.5	273512	73	97%		
00028-98-03 pH9	HILL0037.D	9.5	569596	152	101%		
00028-98-04 pH9	HILL0038.D	9.5	1116548	305	102%		
00028-98-05 pH9	HILL0039.D	9.5	1561236	439	98%		
00028-98-06 pH9	HILL0040.D	9.5	2104613	607	101%		
00028-98-07 pH9	HILL0041.D	9.5	2519089	753	101%		
00028-98-08 pH9	HILL0042.D	9.5	2937090	895	100%		
00028-98-09 pH9	HILL0043.D	9.5	3225468	1006	101%		
MeOH		9.5	0	0			
00028-98-05 pH9	HILL0045.D	9.5	1562031	437		97%	
MeOH		9.5	0	0			
061500-PFOA-133	HILL0046.D	9.5	0	0			
061500-PFOA-134	HILL0047.D	9.5	603971	162		114%	Day 28
061500-PFOA-135	HILL0048.D	9.5	2212354	645			Day 28
061500-PFOA-136	HILL0050.D	9.5	2275081	661			Day 28
061500-PFOA-137	HILL0051.D	9.5	2257809	656			Day 28
061500-PFOA-138	HILL0052.D	9.5	2725058	819	1%	115%	Day 28
061500-PFOA-169	HILL0053.D	9.5	0	0			
061500-PFOA-170	HILL0054.D	9.5	618961	170		119%	Day 42
061500-PFOA-171	HILL0055.D	9.5	2247447	662			Day 42
061500-PFOA-172	HILL0056.D	9.5	2248403	659			Day 42
061500-PFOA-173	HILL0057.D	9.5	2249101	659			Day 42
061500-PFOA-174	HILL0058.D	9.5	2731098	827	0%	117%	Day 42
061500-PFOA-205	HILL0059.D	9.5	0	0			
061500-PFOA-206	HILL0060.D	9.5	614187	166		116%	Day 64
061500-PFOA-207	HILL0061.D	9.5	2240109	663			Day 64
061500-PFOA-208	HILL0062.D	9.5	2275575	663			Day 64
061500-PFOA-209	HILL0063.D	9.5	2293097	674			Day 64
061500-PFOA-210	HILL0064.D	9.5	2749459	829	1%	113%	Day 64
MeOH		9.5	0	0			
00028-98-00 pH9	HILL0065.D	9.5	0	0			
00028-98-01 pH9	HILL0066.D	9.5	0	0			
00028-98-02 pH9	HILL0067.D	9.5	84937	24	98%		
00028-98-03 pH9	HILL0068.D	9.5	270230	73	98%		
00028-98-04 pH9	HILL0069.D	9.5	562673	153	102%		
00028-98-05 pH9	HILL0070.D	9.5	1110329	307	102%		
00028-98-06 pH9	HILL0071.D	9.5	1547236	438	97%		
00028-98-07 pH9	HILL0072.D	9.5	2086611	606	101%		
00028-98-08 pH9	HILL0073.D	9.5	2497789	746	100%		
00028-98-09 pH9	HILL0074.D	9.5	2900192	892	99%		
MeOH		9.5	0	0			
00028-98-05 pH9	HILL0075.D	9.5	3160795	999	100%	96%	
MeOH		9.5	0	0			
00028-97-05 pH9	HILL0076.D	9.5	1537078	433			
MeOH		9.5	0	0			
00028-97-09 pH9	HILL0077.D	9.5	0	0			
00028-97-10 pH9	HILL0078.D	9.5	0	0			
00028-97-11 pH9	HILL0079.D	9.5	0	0			
Method ID:				Internal Standard Quant r2=			
				Curve averaged: Linear. Include origin			
				Calibration range:			
				Mean			
				S.D.			
				%RSD			
				8.1			
				903488			
				6096			
				1%			

PFOA Hydrolysis Study									
50C pH 9.0									
Retention times (RT) in minutes									
All concentrations in ng/ml									
PFOA					PFBS				
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	RT	Area
MeOH		9.5	0	0				8.2	0
00028-98-00 pH9	HILL0066.D	9.5	0	0				8.2	898777
00028-98-01 pH9	HILL0068.D	9.5	84937	26	103%			8.2	907357
00028-98-02 pH9	HILL0069.D	9.5	270230	74	99%			8.2	905164
00028-98-03 pH9	HILL0070.D	9.5	562673	153	102%			8.2	899013
00028-98-04 pH9	HILL0071.D	9.5	1110329	305	97%			8.2	901618
00028-98-05 pH9	HILL0072.D	9.5	1547236	434	100%			8.2	898747
00028-98-06 pH9	HILL0073.D	9.5	2088611	601	99%			8.2	902197
00028-98-07 pH9	HILL0074.D	9.5	2497789	740	98%			8.2	898658
00028-98-08 pH9	HILL0075.D	9.5	2900192	884	99%			8.2	897407
00028-98-09 pH9	HILL0076.D	9.5	3160795	990	99%			8.2	891718
MeOH		9.5	0	0				8.2	0
00028-97-05 pH9	HILL0078.D	9.5	1537078	429	95%			8.2	902240
MeOH		9.5	0	0				8.2	0
061500-PFOA-241	HILL0080.D	9.5	0	0				8.2	890731
061500-PFOA-242	HILL0081.D	9.5	614030	166	116%		Day 84	8.2	903188
061500-PFOA-243	HILL0082.D	9.5	2168895	633			Day 84	8.2	894803
061500-PFOA-244	HILL0083.D	9.5	2233715	644			Day 84	8.2	906955
061500-PFOA-245	HILL0084.D	9.5	2214363	644	1%		Day 84	8.2	898578
061500-PFOA-246	HILL0085.D	9.5	2685763	812			Day 84	8.2	891932
061500-PFOA-277	HILL0086.D	9.5	0	0				8.2	895005
061500-PFOA-278	HILL0087.D	9.5	605776	166	116%		Day 109	8.2	893945
061500-PFOA-279	HILL0088.D	9.5	2237888	660			Day 109	8.2	889480
061500-PFOA-280	HILL0089.D	9.5	2215935	653			Day 109	8.2	889193
061500-PFOA-281	HILL0090.D	9.5	2221268	657			Day 109	8.2	885882
061500-PFOA-282	HILL0091.D	9.5	2705639	827	119%		Day 109	8.2	885088
MeOH		9.5	0	0				8.2	0
00028-98-00 pH9	HILL0092.D	9.5	0	0				8.2	887837
00028-98-01 pH9	HILL0093.D	9.5	81580	25	101%			8.2	892635
00028-98-02 pH9	HILL0094.D	9.5	261882	73	98%			8.2	887309
00028-98-03 pH9	HILL0095.D	9.5	546205	150	100%			8.2	887505
00028-98-04 pH9	HILL0097.D	9.5	1083979	302	101%			8.2	888627
00028-98-05 pH9	HILL0098.D	9.5	1535318	440	98%			8.2	881276
00028-98-06 pH9	HILL0099.D	9.5	2087627	616	103%			8.2	881348
00028-98-07 pH9	HILL0100.D	9.5	2478930	764	102%			8.2	867135
00028-98-08 pH9	HILL0101.D	9.5	2891451	907	101%			8.2	875807
00028-98-09 pH9	HILL0102.D	9.5	3142079	1009	101%			8.2	872831
MeOH		9.5	0	0				8.2	0
00028-98-05 pH9	HILL0104.D	9.5	1538828	439	98%			8.2	884407
MeOH		9.5	0	0				8.2	0
Method ID:								Mean	892188
								S.D.	9602
								%RSD	1%
Internal Standard Quant r2=					Curve averaged: Linear: Include origin				
Calibration range:									

PFOA Hydrolysis Study

50C pH 11

Retention times (RT) in minutes

All concentrations in ng/ml

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Method ID:

Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	%Spike Recovery	Time Point	RT	Area	Conc.
MeOH	HILL0002.D	9.5	0	0				8.2	0	0.0
00028-99-00 pH11	HILL0003.D	9.5	0	0				8.2	836994	158
00028-99-01 pH11	HILL0004.D	9.5	77656	25	99%			8.2	842003	158
00028-99-02 pH11	HILL0005.D	9.5	249935	72	96%			8.2	838243	158
00028-99-03 pH11	HILL0006.D	9.5	537047	152	101%			8.2	842722	158
00028-99-04 pH11	HILL0007.D	9.5	1042553	303	101%			8.2	832105	158
00028-99-05 pH11	HILL0008.D	9.5	1500587	445	99%			8.2	837049	158
00028-99-06 pH11	HILL0009.D	9.5	1977142	610	102%			8.2	832100	158
00028-99-07 pH11	HILL0010.D	9.5	2325011	744	99%			8.2	826734	158
00028-99-08 pH11	HILL0011.D	9.5	2707083	900	100%			8.2	824144	158
00028-99-09 pH11	HILL0012.D	9.5	2985254	1015	102%			8.2	827886	158
MeOH	HILL0013.D	9.5	0	0				8.2	0	0
00028-99-05 pH11	HILL0014.D	9.5	1506903	449	100%			8.2	833679	158
MeOH	HILL0015.D	9.5	0	0				8.2	0	0
061500-PFOA-031	HILL0016.D	9.5	0	0				8.2	827649	158
061500-PFOA-032	HILL0017.D	9.5	574651	166	116%		(A)	8.2	824926	158
061500-PFOA-033	HILL0018.D	9.5	2088515	654			(A)	8.2	827776	158
061500-PFOA-034	HILL0019.D	9.5	2097206	653			(A)	8.2	832611	158
061500-PFOA-035	HILL0020.D	9.5	2169713	683			(A)	8.2	829560	158
061500-PFOA-036	HILL0021.D	9.5	2666925	879	3%	151%	(A)	8.2	827032	158
061500-PFOA-067	HILL0022.D	9.5	0	0				8.2	827614	158
061500-PFOA-068	HILL0023.D	9.5	565130	163	114%		(A)	8.2	825475	158
061500-PFOA-069	HILL0024.D	9.5	2078787	654			(A)	8.2	824330	158
061500-PFOA-070	HILL0025.D	9.5	2115304	665			(A)	8.2	826822	158
061500-PFOA-071	HILL0026.D	9.5	1528187	461			(A)	8.2	826508	158
061500-PFOA-072	HILL0027.D	9.5	2655140	870	19%	194%	(A)	8.2	830218	158
061500-PFOA-103	HILL0028.D	9.5	0	0				8.2	827682	158
061500-PFOA-104	HILL0029.D	9.5	584023	166	116%		(A)	8.2	835459	158
061500-PFOA-105	HILL0030.D	9.5	2084579	650			(A)	8.2	830844	158
061500-PFOA-106	HILL0031.D	9.5	2152418	688			(A)	8.2	838043	158
061500-PFOA-107	HILL0032.D	9.5	2076572	646			(A)	8.2	832314	158
061500-PFOA-108	HILL0033.D	9.5	2704060	884	2%	160%	(A)	8.2	835109	158
MeOH	HILL0034.D	9.5	0	0				8.2	0	0
00028-99-00 pH11	HILL0035.D	9.5	0	0				8.2	847430	158
00028-99-01 pH11	HILL0036.D	9.5	78888	25	100%			8.2	846510	158
00028-99-02 pH11	HILL0037.D	9.5	250493	71	95%			8.2	848213	158
00028-99-03 pH11	HILL0038.D	9.5	546767	153	102%			8.2	850916	158
00028-99-04 pH11	HILL0039.D	9.5	1065900	304	101%			8.2	847577	158
00028-99-05 pH11	HILL0040.D	9.5	1513897	444	99%			8.2	847024	158
00028-99-06 pH11	HILL0041.D	9.5	2026496	613	102%			8.2	849662	158
00028-99-07 pH11	HILL0042.D	9.5	2360153	732	98%			8.2	850765	158
00028-99-08 pH11	HILL0043.D	9.5	2751023	883	98%			8.2	850709	158
00028-99-09 pH11	HILL0044.D	9.5	3029716	1001	100%			8.2	849247	158
MeOH	HILL0045.D	9.5	0	0				8.2	0	0
00028-99-05 pH11	HILL0046.D	9.5	1546259	445	99%			8.2	862345	158
MeOH	HILL0047.D	9.5	0	0				8.2	0	0

PFOA Hydrolysis Study

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50C pH 11

Retention times (RT) in minutes

All concentrations in ng/ml

PFOA				PFBS						
Sample ID	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	RT	Area	Conc.
MeOH	HILL0034.D	9.5	0	0				8.1	0	0
00028-99-00 pH11	HILL0035.D	9.5	0	0				8.2	847430	158
00028-99-01 pH11	HILL0036.D	9.5	78888	25	100%			8.2	846510	158
00028-99-02 pH11	HILL0037.D	9.5	250493	71	95%			8.2	846213	158
00028-99-03 pH11	HILL0038.D	9.5	546767	153	102%			8.2	850916	158
00028-99-04 pH11	HILL0039.D	9.5	1065800	305	102%			8.2	847577	158
00028-99-05 pH11	HILL0040.D	9.5	1513897	444	99%			8.2	847024	158
00028-99-06 pH11	HILL0041.D	9.5	2026496	611	102%			8.2	849662	158
00028-99-07 pH11	HILL0042.D	9.5	2360153	729	97%			8.2	850709	158
00028-99-08 pH11	HILL0043.D	9.5	2751023	877	98%			8.2	850709	158
00028-99-09 pH11	HILL0044.D	9.5	3029716	993	99%			8.2	849247	158
MeOH	HILL0045.D	9.5	0	0				8.1	0	0
00028-99-05 pH11	HILL0046.D	9.5	1546259	445		99%		8.2	862345	158
MeOH	HILL0047.D	9.5	0	0				8.1	0	0
061500-PFOA-139	HILL0048.D	9.5	0	0				8.2	859034	158
061500-PFOA-140	HILL0049.D	9.5	575939	161		113%	(A)	8.2	851490	158
061500-PFOA-141	HILL0050.D	9.5	2115608	636			(A)	8.2	857366	158
061500-PFOA-142	HILL0051.D	9.5	2188859	664			(A)	8.2	854875	158
061500-PFOA-143	HILL0052.D	9.5	2259548	678			(A)	8.2	865743	158
061500-PFOA-144	HILL0053.D	9.5	2714384	850	3%	133%	(A)	8.2	860627	158
061500-PFOA-175	HILL0054.D	9.5	0	0				8.2	862052	158
061500-PFOA-176	HILL0055.D	9.5	1152389	327		229%	(A)	8.2	857129	158
061500-PFOA-177	HILL0056.D	9.5	2065839	616			(A)	8.2	860411	158
061500-PFOA-178	HILL0057.D	9.5	2007513	675			(A)	8.2	772681	158
061500-PFOA-179	HILL0058.D	9.5	1866921	708		161%	(A)	8.2	689691	158
061500-PFOA-180	HILL0059.D	9.5	2269428	897	7%		(A)	8.2	688888	158
061500-PFOA-211	HILL0060.D	9.5	0	0				0.0	0	0
061500-PFOA-212	HILL0061.D	0.0	0	0		0%	Day 64	0.0	0	0
061500-PFOA-213	HILL0062.D	9.5	2151980	647			Day 64	8.2	859405	158
061500-PFOA-214	HILL0063.D	9.5	2245485	683			Day 64	8.2	855708	158
061500-PFOA-215	HILL0064.D	9.5	2272096	683			Day 64	8.2	865487	158
061500-PFOA-216	HILL0065.D	9.5	2747811	845	3%	122%	Day 64	8.2	875709	158
MeOH	HILL0066.D	9.5	0	0				8.1	0	0
00028-99-00 pH11	HILL0067.D	9.4	0	0				8.2	882190	158
00028-99-01 pH11	HILL0068.D	9.5	80241	24	97%			8.2	882930	158
00028-99-02 pH11	HILL0069.D	9.5	262214	71	95%			8.2	893562	158
00028-99-03 pH11	HILL0070.D	9.5	565808	152	101%			8.2	885381	158
00028-99-04 pH11	HILL0071.D	9.5	1105592	304	101%			8.2	882529	158
00028-99-05 pH11	HILL0072.D	9.5	1596046	448	100%			8.2	885743	158
00028-99-06 pH11	HILL0073.D	9.5	2122804	616	103%			8.2	884851	158
00028-99-07 pH11	HILL0074.D	9.5	2492123	740	99%			8.2	887106	158
00028-99-08 pH11	HILL0075.D	9.5	2912475	900	100%			8.2	882114	158
00028-99-09 pH11	HILL0076.D	9.5	3188896	1032	103%			8.2	867461	158
MeOH	HILL0077.D	9.5	0	0				8.1	0	0
00028-99-05 pH11	HILL0078.D	9.5	1590283	448		99%		8.2	882607	158
MeOH	HILL0079.D	9.5	0	0				8.1	0	0
Method ID:								Mean		
								S.D.		
								%RSD		
Internal Standard Quant (Z=										
Curve averaged: Linear: Include origin										
Calibration range:										
A) Data excluded on the basis of data quality objectives; see text.										

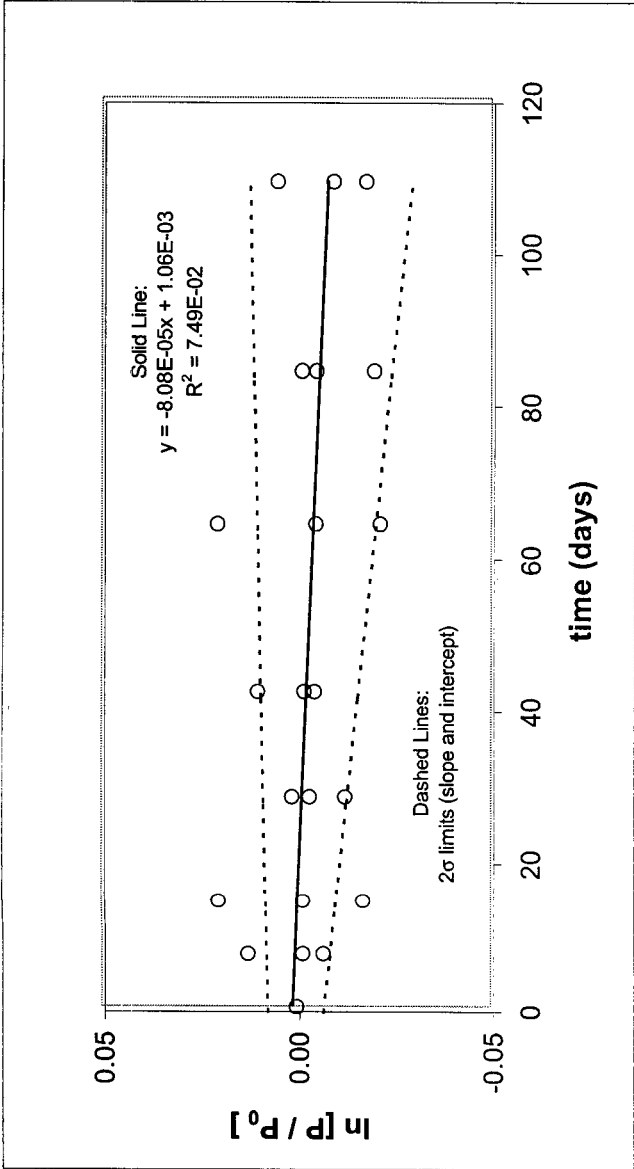
PFOA Hydrolysis Study Page 3 of 3
 50C pH 11 Retention times (RT) in minutes
 All concentrations in ng/ml

PFOA										PFBS											
Sample I.D.	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	RT	Area	Conc.	Sample I.D.	Data File	RT	Area	Conc.	%Standard or RSD	% Spike Recovery	Time Point	RT	Area	Conc.
MeOH	HILL0066.D	9.5	0	0				8.2	0	0	00028-99-00 pH11	HILL0066.D	9.5	0	0				8.2	0	0
00028-99-00 pH11	HILL0067.D	9.5	0	0				8.2	882189	158	00028-99-01 pH11	HILL0068.D	9.5	80241	26	102%			8.2	882932	158
00028-99-01 pH11	HILL0068.D	9.5	80241	26	102%			8.2	882932	158	00028-99-02 pH11	HILL0069.D	9.5	262214	72	95%			8.2	893562	158
00028-99-02 pH11	HILL0069.D	9.5	262214	72	95%			8.2	893562	158	00028-99-03 pH11	HILL0070.D	9.5	565808	152	101%			8.2	885381	158
00028-99-03 pH11	HILL0070.D	9.5	565808	152	101%			8.2	885381	158	00028-99-04 pH11	HILL0071.D	9.5	1105592	302	101%			8.2	882529	158
00028-99-04 pH11	HILL0071.D	9.5	1105592	302	101%			8.2	882529	158	00028-99-05 pH11	HILL0072.D	9.5	1598046	444	99%			8.2	885743	158
00028-99-05 pH11	HILL0072.D	9.5	1598046	444	99%			8.2	885743	158	00028-99-06 pH11	HILL0073.D	9.5	2122804	609	102%			8.2	884851	158
00028-99-06 pH11	HILL0073.D	9.5	2122804	609	102%			8.2	884851	158	00028-99-07 pH11	HILL0074.D	9.5	2492123	731	98%			8.2	887106	158
00028-99-07 pH11	HILL0074.D	9.5	2492123	731	98%			8.2	887106	158	00028-99-08 pH11	HILL0075.D	9.5	2912475	888	99%			8.2	882114	158
00028-99-08 pH11	HILL0075.D	9.5	2912475	888	99%			8.2	882114	158	00028-99-09 pH11	HILL0076.D	9.5	3188696	1016	102%			8.2	867461	158
00028-99-09 pH11	HILL0076.D	9.5	3188696	1016	102%			8.2	867461	158	MeOH	HILL0077.D	9.5	0	0				8.2	0	0
MeOH	HILL0077.D	9.5	0	0		99%		8.2	0	0	00028-99-05 pH11	HILL0078.D	9.5	1590283	444				8.2	882607	158
00028-99-05 pH11	HILL0078.D	9.5	1590283	444				8.2	882607	158	MeOH	HILL0079.D	9.5	0	0				8.2	0	0
MeOH	HILL0079.D	9.5	0	0				8.2	0	0	061500-PFOA-247	HILL0080.D	9.5	0	0				8.2	0	0
061500-PFOA-247	HILL0080.D	9.5	0	0				8.2	0	0	061500-PFOA-248	HILL0081.D	9.5	609981	166		116%	(A)	8.2	871661	158
061500-PFOA-248	HILL0081.D	9.5	609981	166		116%	(A)	8.2	871661	158	061500-PFOA-249	HILL0082.D	9.5	2202106	637			(A)	8.2	882950	158
061500-PFOA-249	HILL0082.D	9.5	2202106	637			(A)	8.2	882950	158	061500-PFOA-250	HILL0083.D	9.5	2350972	694			(A)	8.2	874463	158
061500-PFOA-250	HILL0083.D	9.5	2350972	694			(A)	8.2	874463	158	061500-PFOA-251	HILL0084.D	9.5	2826619	858	6%		(A)	8.2	880217	158
061500-PFOA-251	HILL0084.D	9.5	2826619	858	6%		(A)	8.2	880217	158	061500-PFOA-252	HILL0085.D	9.5	0	0				8.2	0	0
061500-PFOA-252	HILL0085.D	9.5	0	0				8.2	0	0	061500-PFOA-283	HILL0086.D	9.5	0	0				8.2	883613	158
061500-PFOA-283	HILL0086.D	9.5	0	0				8.2	883613	158	061500-PFOA-284	HILL0087.D	9.5	604634	162		113%	(A)	8.2	887173	158
061500-PFOA-284	HILL0087.D	9.5	604634	162		113%	(A)	8.2	887173	158	061500-PFOA-285	HILL0088.D	9.5	2263208	652			(A)	8.2	888570	158
061500-PFOA-285	HILL0088.D	9.5	2263208	652			(A)	8.2	888570	158	061500-PFOA-286	HILL0089.D	9.5	2338785	680			(A)	8.2	885977	158
061500-PFOA-286	HILL0089.D	9.5	2338785	680			(A)	8.2	885977	158	061500-PFOA-287	HILL0090.D	9.5	1363306	349			(A)	8.2	946290	158
061500-PFOA-287	HILL0090.D	9.5	1363306	349			(A)	8.2	946290	158	061500-PFOA-288	HILL0091.D	9.5	2649738	781	33%	154%	(A)	8.2	891813	158
061500-PFOA-288	HILL0091.D	9.5	2649738	781	33%	154%	(A)	8.2	891813	158	MeOH	HILL0092.D	9.5	0	0				8.2	0	0
MeOH	HILL0092.D	9.5	0	0				8.2	0	0	00028-99-00 pH11	HILL0093.D	9.5	0	0				8.2	900429	158
00028-99-00 pH11	HILL0093.D	9.5	0	0				8.2	900429	158	00028-99-01 pH11	HILL0094.D	9.5	81748	25	102%			8.2	904090	158
00028-99-01 pH11	HILL0094.D	9.5	81748	25	102%			8.2	904090	158	00028-99-02 pH11	HILL0095.D	9.5	267734	72	96%			8.2	903277	158
00028-99-02 pH11	HILL0095.D	9.5	267734	72	96%			8.2	903277	158	00028-99-03 pH11	HILL0096.D	9.5	575392	152	101%			8.2	901713	158
00028-99-03 pH11	HILL0096.D	9.5	575392	152	101%			8.2	901713	158	00028-99-04 pH11	HILL0097.D	9.5	1128511	302	101%			8.2	900386	158
00028-99-04 pH11	HILL0097.D	9.5	1128511	302	101%			8.2	900386	158	00028-99-05 pH11	HILL0098.D	9.5	1640031	447	99%			8.2	904513	158
00028-99-05 pH11	HILL0098.D	9.5	1640031	447	99%			8.2	904513	158	00028-99-06 pH11	HILL0099.D	9.5	2183352	617	103%			8.2	900220	158
00028-99-06 pH11	HILL0099.D	9.5	2183352	617	103%			8.2	900220	158	00028-99-07 pH11	HILL0100.D	9.5	2558520	746	100%			8.2	894872	158
00028-99-07 pH11	HILL0100.D	9.5	2558520	746	100%			8.2	894872	158	00028-99-08 pH11	HILL0101.D	9.5	2974153	891	99%			8.2	897865	158
00028-99-08 pH11	HILL0101.D	9.5	2974153	891	99%			8.2	897865	158	00028-99-09 pH11	HILL0102.D	9.5	3267410	1002	100%			8.2	898862	158
00028-99-09 pH11	HILL0102.D	9.5	3267410	1002	100%			8.2	898862	158	MeOH	HILL0103.D	9.5	0	0				8.2	0	0
MeOH	HILL0103.D	9.5	0	0				8.2	0	0	00028-99-05 pH11	HILL0104.D	9.5	1651887	450		100%		8.2	904102	158
00028-99-05 pH11	HILL0104.D	9.5	1651887	450		100%		8.2	904102	158	MeOH	HILL0105.D	9.5	0	0				8.2	0	0
MeOH	HILL0105.D	9.5	0	0				8.2	0	0	Internal Standard Quant 12=										
Method ID:										Mean	838809	Curve averaged: Linear. Include origin									
										S.D.	213297	Calibration range:									
										%RSD	25%										

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

Internal Standard Quant r2= Curve averaged: Linear: Include origin Calibration range:

Figure 3. Pooled PFOA Data and Slope Regression (pH = 5.0, 7.0, and 9.0 only)



SUMMARY OUTPUT

Regression Statistics			
Multiple R	0.273674341		
R Square	0.074897645		
Adjusted R Square	0.032847538		
Standard Error	0.010843571		
Observations	24		

ANOVA					
	df	SS	MS	F	Significance F
Regression	1	0.000209433	0.000209433	1.781152306	0.195657077
Residual	22	0.002586827	0.000117583		
Total	23	0.00279626			

	Coefficients	Standard Error	t Stat	P-value	Lower 95%	Upper 95%
Intercept	0.001060684	0.003440702	0.308275508	0.76077268	-0.006074903	0.008196271
X Variable 1	-8.08191E-05	6.05569E-05	-1.334598181	0.195657077	-0.000206407	4.47683E-05

EE1851I.XLS 3/30/2001 3:05 PM

Rate and T_{1/2} Calculations**Author: GMP****Date: 3/30/01**

Incubation time (days)	1.090E+02
-------------------------------	-----------

Parent PFOA	Parent Mass (gm/mole) N/A	Parent mean μ (ng/ml) 6.528E+02	Parent stdev σ (nm/ml) 6.963E+00
-----------------------	-------------------------------------	--	--

Rates and Half-Lives from μ and σ (Eq's B37 and B41)	Max Rate @50C (day-1) 1.957E-04	Max Rate @25C (day-1) 1.957E-05	Min Half-Life @25C (year) 97.0
---	---	---	--

Pooled Data	Slopes @50C (day-1)	Slopes @25C (day-1)	
Regression Slope 2 σ Minimum (day-1)	-2.064E-04	-2.064E-05	
Regression Slope Value (day-1)	-8.082E-05	-8.082E-06	
Regression Slope 2 σ Maximum (day-1)	4.477E-05	4.477E-06	
Max. H-Life @25C (year) (Eq's B8, B9, and B41)	Max. H-Life @25C (year) N/A - Slope positive	Calc. H-Life @25C (year) 235	Min. Half-Life @25C (year) 92

Appendix D: Selected Chromatograms

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

Data File: \\Etshillary\D\Data\H110300\H110300a\HILL0045.D
Report Date: 08-Nov-2000 14:14

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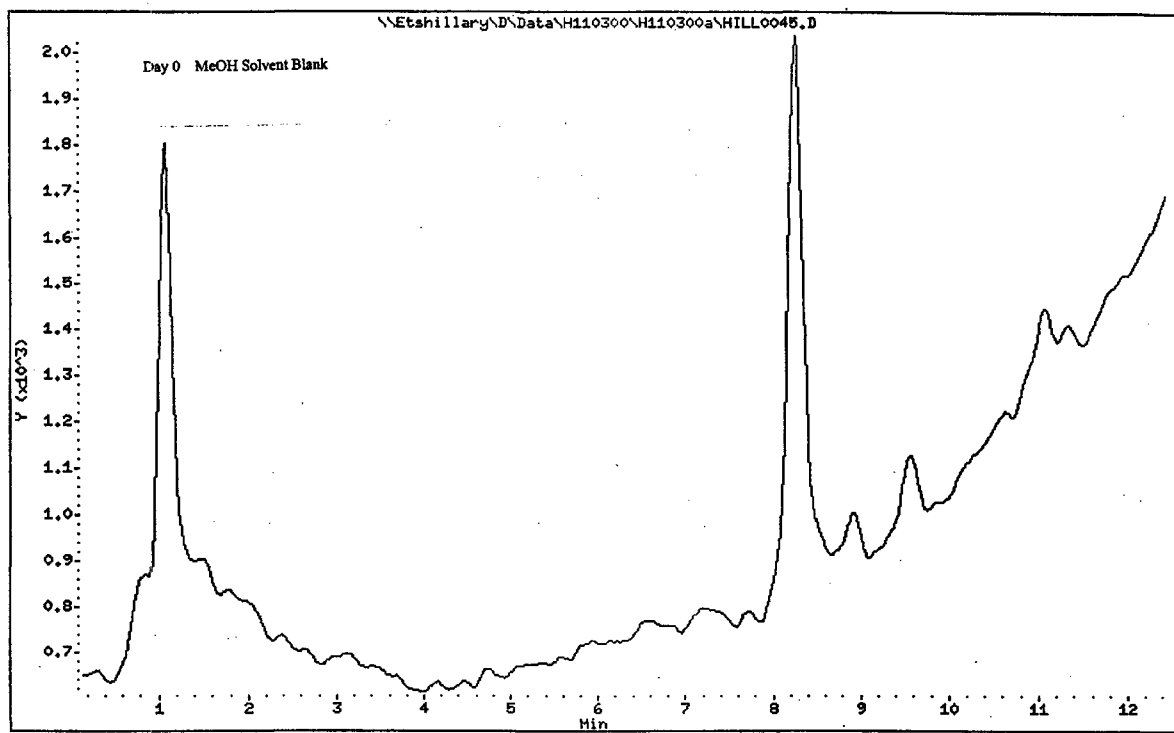
Data file : \\Etshillary\D\Data\H110300\H110300a\HILL0045.D
Lab Smp Id:
Inj Date : 04-NOV-2000 00:37
Operator : DDW
Smp Info : MeOH
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300a\PFOA1103.m
Meth Date : 08-Nov-2000 14:12 wright
Cal Date : 03-NOV-2000 15:20
Als bottle: 100
Dil Factor: 1.00000
Integrator: HP Genie
Target Version: 4.04
Processing Host: WW19507

Inst ID: hillary.i
Quant Type: ISTD
Cal File: HILL0015.D
Compound Sublist: all.sub

Compounds	QUANT SIG	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS	
						ON-COLUMN	FINAL
	MASS					(ng/mL)	(ng/mL)
-----	----	--	-----	-----	-----	-----	-----
* 1 PFBS	299				Compound Not Detected.		
75 PFOA	413				Compound Not Detected.		

Data File: \\Etshillary\D\Data\H110300\H110300a\HILL0045.D

Page 2



Data File: \\Etshillary\D\Data\H110300\H110300a\HILL0046.D
Report Date: 08-Nov-2000 14:14

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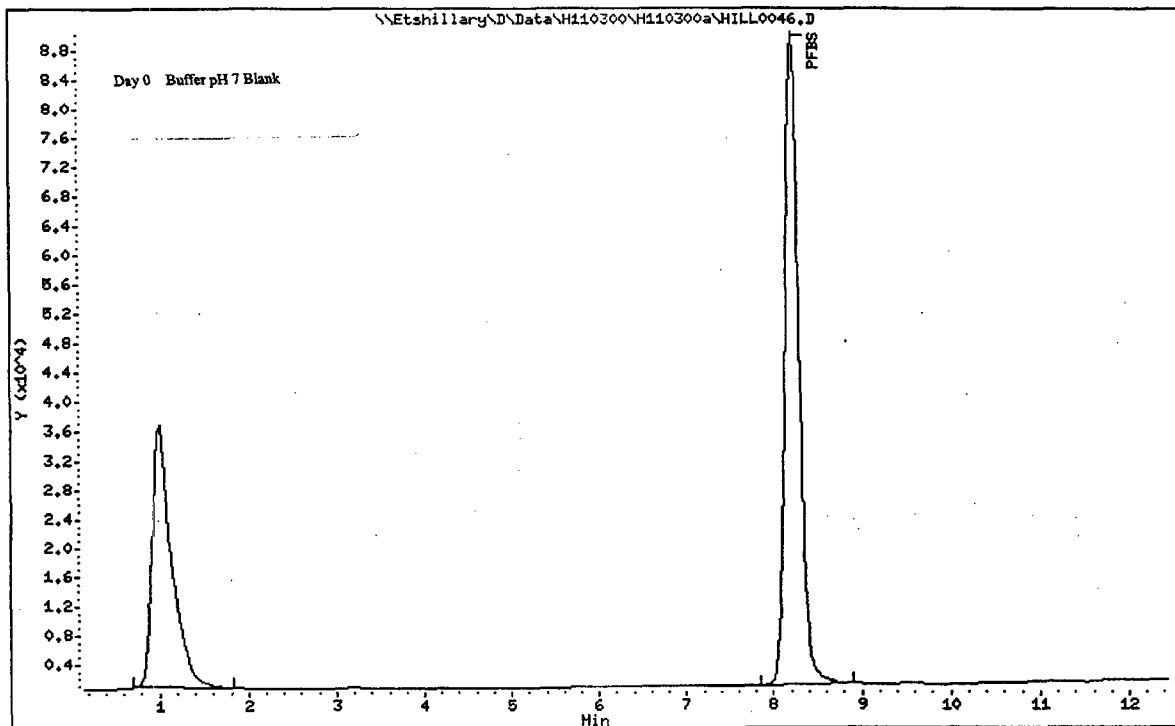
3M Environmental Lab

Data file : \\Etshillary\D\Data\H110300\H110300a\HILL0046.D
Lab Smp Id:
Inj Date : 04-NOV-2000 00:55
Operator : DDW Inst ID: hillary.i
Smp Info : 00028-97-00 pH7
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300a\PFOA1103.m
Meth Date : 08-Nov-2000 14:12 wright Quant Type: ISTD
Cal Date : 03-NOV-2000 15:20 Cal File: HILL0015.D
Als bottle: 1
Dil Factor: 1.00000
Integrator: HP Genie Compound Sublist: all.sub
Target Version: 4.04
Processing Host: WW19507

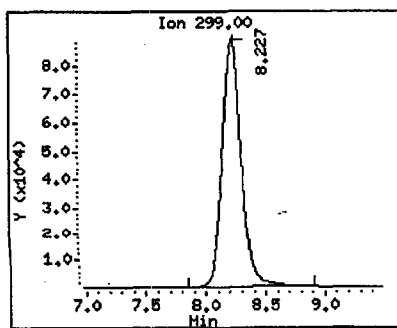
Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS	
						ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 PFBS	299	8.226	8.226	(1.000)	995123	158.000	
75 PFOA	413				Compound Not Detected.		

Data File: \\Etshillary\D\Data\H110300\H110300a\HILL0046.D

Page 2



* 1 PFBS



Data File: \\Etshillary\D\Data\H110300\H110300a\HILL0047.D
Report Date: 08-Nov-2000 14:14

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3M Environmental Lab

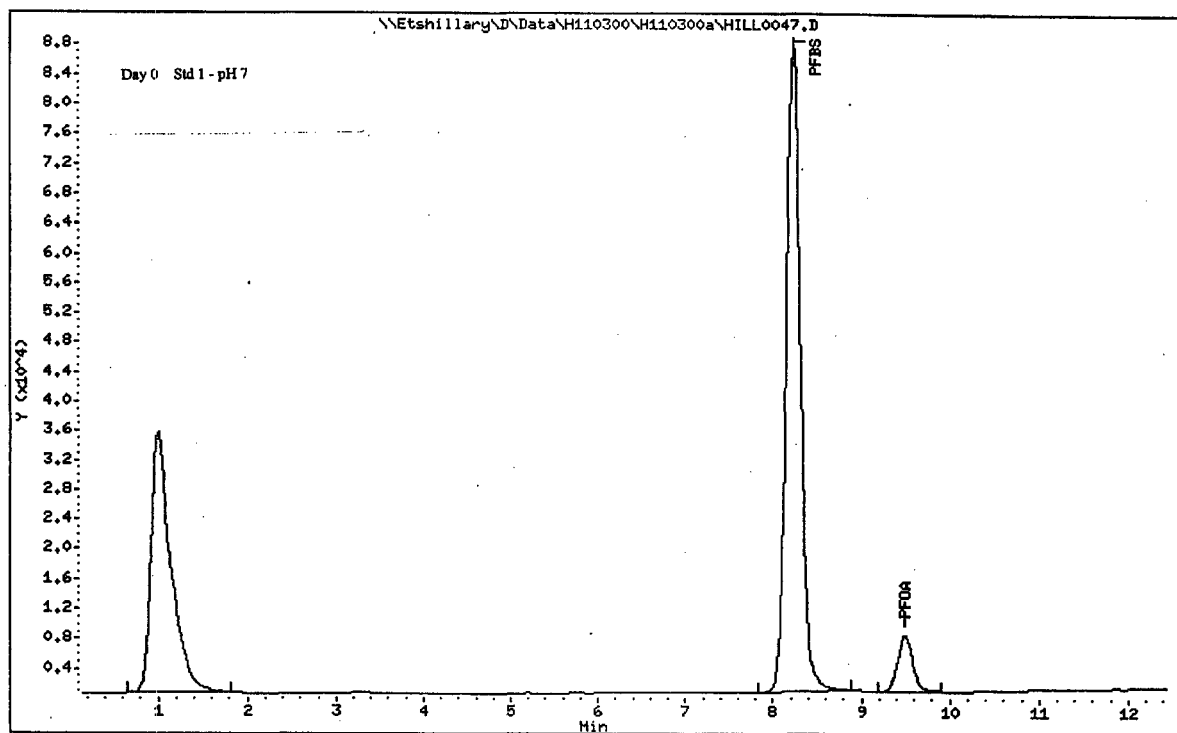
Data file : \\Etshillary\D\Data\H110300\H110300a\HILL0047.D
Lab Smp Id:
Inj Date : 04-NOV-2000 01:14
Operator : DDW
Smp Info : 00028-97-01 pH7
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300a\PFOA1103.m
Meth Date : 08-Nov-2000 14:14 wright
Cal Date : 03-NOV-2000 15:20
Als bottle: 2
Dil Factor: 1.00000
Integrator: HP Genie
Target Version: 4.04
Processing Host: WW19507

Inst ID: hillary.i
Quant Type: ISTD
Cal File: HILL0015.D
Calibration Sample, Level: 11
Compound Sublist: all.sub

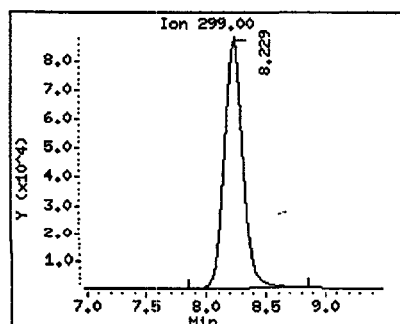
Compounds	QUANT SIG						AMOUNTS	
	MASS	RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ng/mL)	ON-COL (ng/mL)	
-----	----	--	-----	-----	-----	-----	-----	
* 1 PFBS	299	8.229	8.229	(1.000)	970814	158.000		
75 PFOA	413	9.502	9.502	(1.155)	87291	25.0000	27.02	

Data File: \\Etshillary\J\Data\H110300\H110300a\HILL0047.D

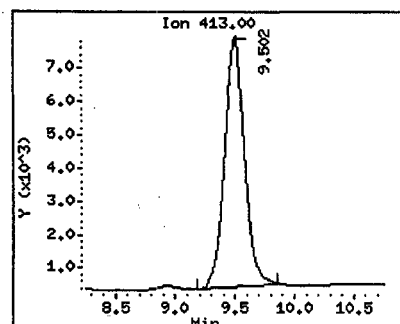
Page 2



1 PFBS



75 PFOA



Data File: \\Etshillary\D\Data\H110300\H110300a\HILL0055.D
Report Date: 08-Nov-2000 14:15

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3M Environmental Lab

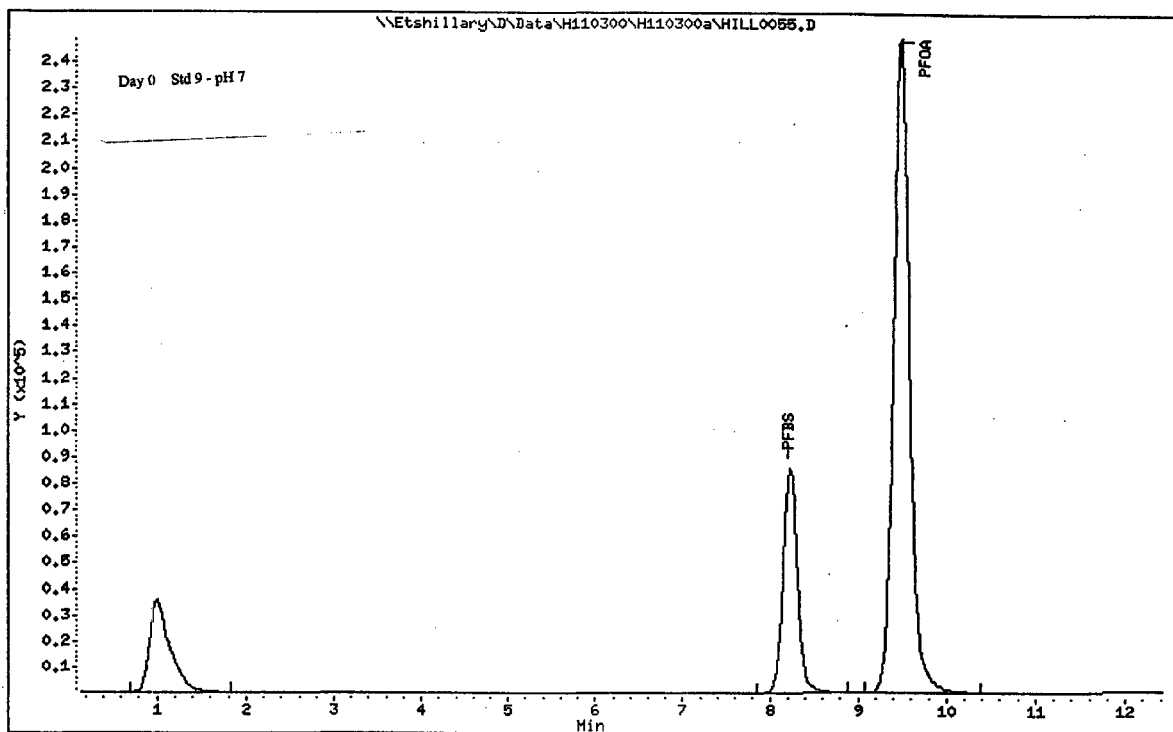
Data file : \\Etshillary\D\Data\H110300\H110300a\HILL0055.D
Lab Smp Id:
Inj Date : 04-NOV-2000 03:42
Operator : DDW
Smp Info : 00028-97-09 pH7
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300a\PFOA1103.m
Meth Date : 08-Nov-2000 14:15 wright
Cal Date : 04-NOV-2000 03:42
Als bottle: 10
Dil Factor: 1.00000
Integrator: HP Genie
Target Version: 4.04
Processing Host: WW19507

Inst ID: hillary.i
Quant Type: ISTD
Cal File: HILL0055.D
Calibration Sample, Level: 19
Compound Sublist: all.sub

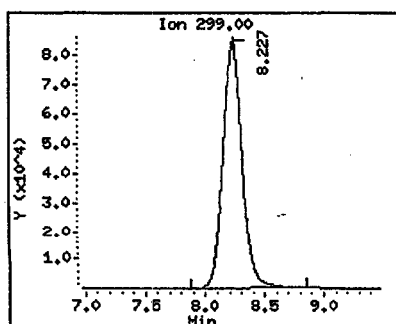
Compounds	QUANT SIG						AMOUNTS	
	MASS	RT	EXP RT	REL RT	RESPONSE		CAL-AMT	ON-COL
							(ng/mL)	(ng/mL)
*****	----	--	-----	-----	-----		-----	-----
* 1 PFBS	299	8.226	8.226	(1.000)	948270		158.000	
75 PFOA	413	9.498	9.498	(1.155)	3142471		999.000	1010

Data File: \\Etshillary\N\Data\H110300\H110300a\HILL0055.D

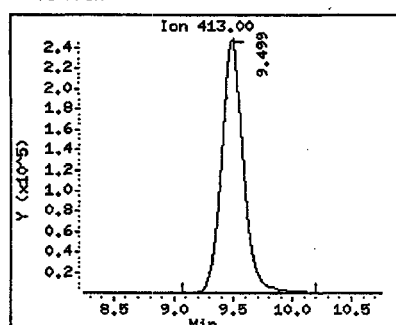
Page 2



M 1 PFBS



75 PFOA



Data File: \\Etshillary\D\Data\H110300\H110300a\HILL0027.D
Report Date: 08-Nov-2000 14:13

Page 1

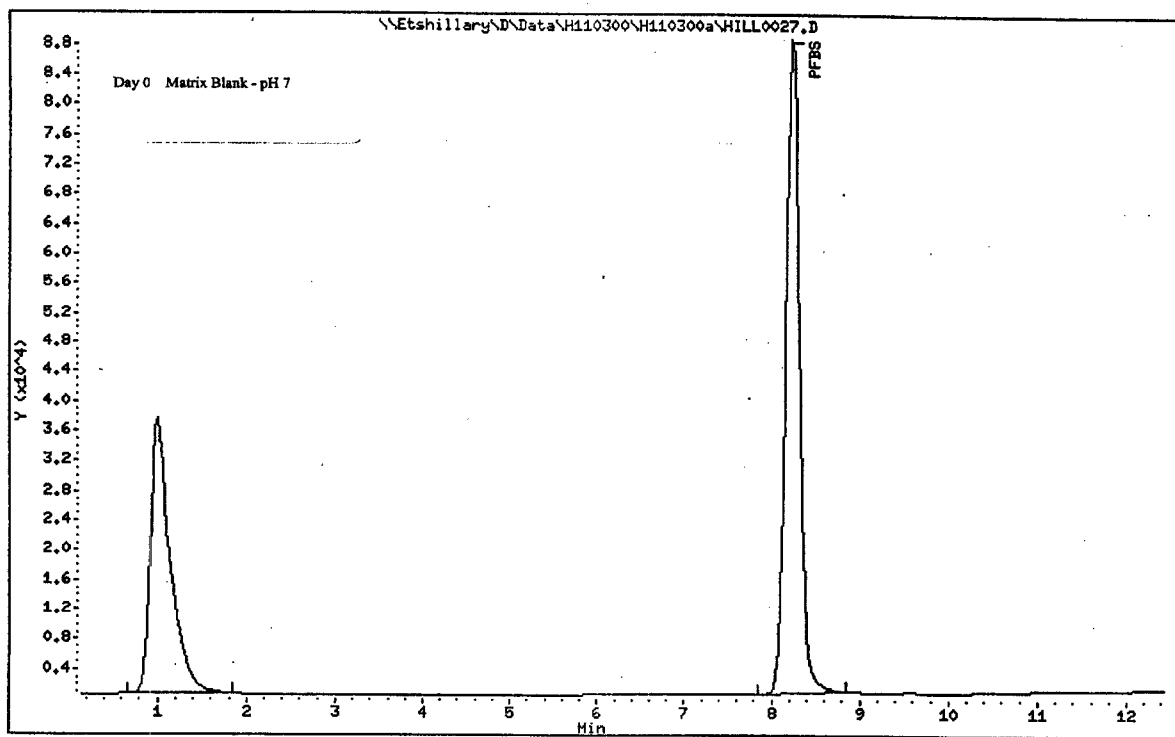
3M Environmental Lab

Data file : \\Etshillary\D\Data\H110300\H110300a\HILL0027.D
Lab Smp Id:
Inj Date : 03-NOV-2000 19:03
Operator : DDW Inst ID: hillary.i
Smp Info : 061500-PFOA-019
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300a\PFOA1103.m
Meth Date : 08-Nov-2000 14:12 wright Quant Type: ISTD
Cal Date : 03-NOV-2000 15:20 Cal File: HILL0015.D
Als bottle: 11
Dil Factor: 1.00000
Integrator: HP Genie Compound Sublist: all.sub
Target Version: 4.04
Processing Host: WW19507

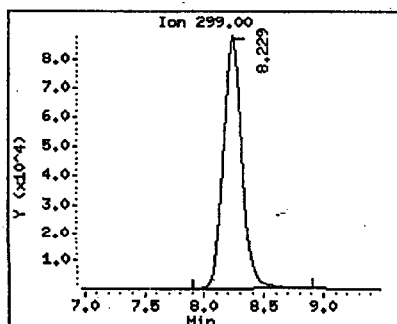
Compounds	QUANT SIG	MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS	
							ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 PFBS	299	8.228	8.226	(1.000)	968331	158.000		
75 PFOA	413				Compound Not Detected.			

Data File: \\Etshillary\N\Data\H110300\H110300a\HILL0027.D

Page 2



* 1 PFBS



Data File: \\Etshillary\D\Data\H110300\H110300a\HILL0028.D
Report Date: 08-Nov-2000 14:13

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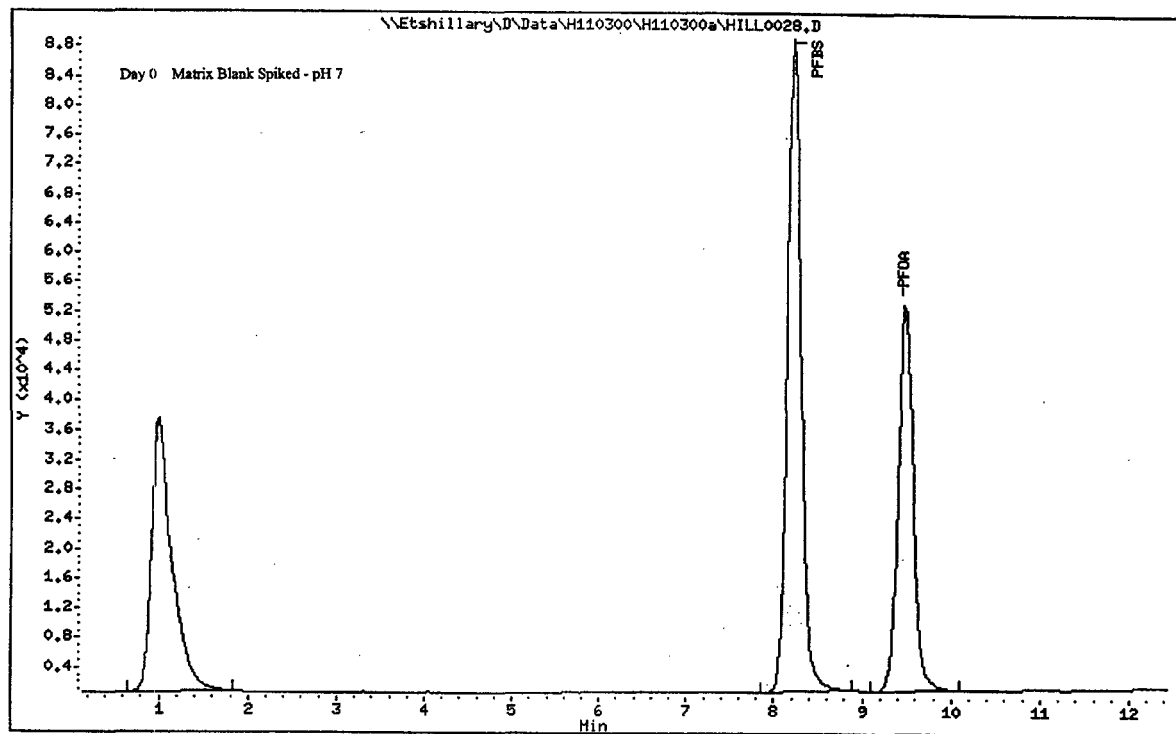
3M Environmental Lab

Data file : \\Etshillary\D\Data\H110300\H110300a\HILL0028.D
Lab Smp Id:
Inj Date : 03-NOV-2000 19:22
Operator : DDW Inst ID: hillary.i
Smp Info : 061500-PFOA-020
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300a\PFOA1103.m
Meth Date : 08-Nov-2000 14:12 wright Quant Type: ISTD
Cal Date : 03-NOV-2000 15:20 Cal File: HILL0015.D
Als bottle: 12
Dil Factor: 1.00000
Integrator: HP Genie Compound Sublist: all.sub
Target Version: 4.04
Processing Host: WW19507

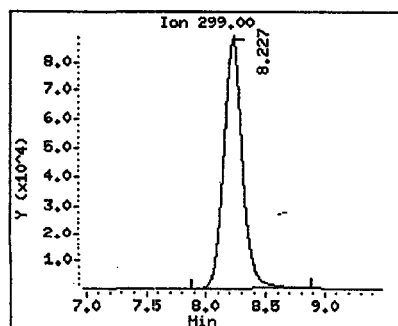
Compounds	QUANT SIG						CONCENTRATIONS	
	MASS	RT	EXP RT	REL RT	RESPONSE		ON-COLUMN	FINAL
							(ng/mL)	(ng/mL)
* 1 PFBS	299	8.226	8.226	(1.000)	968193		158.000	
75 PFOA	413	9.479	9.498	(1.152)	623804		165.237	165.2

Data File: \\Etshillary\B\Data\H110300\H110300a\HILL0028.D

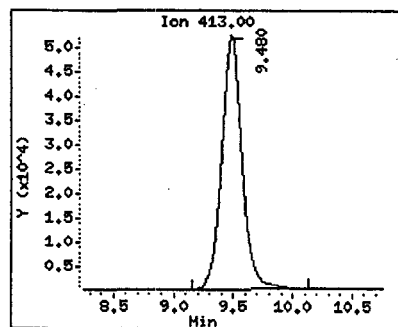
Page 2



* 1 PFBS



75 PFOA



Data File: \\Etshillary\D\Data\H110300\H110300a\HILL0029.D
Report Date: 08-Nov-2000 14:13

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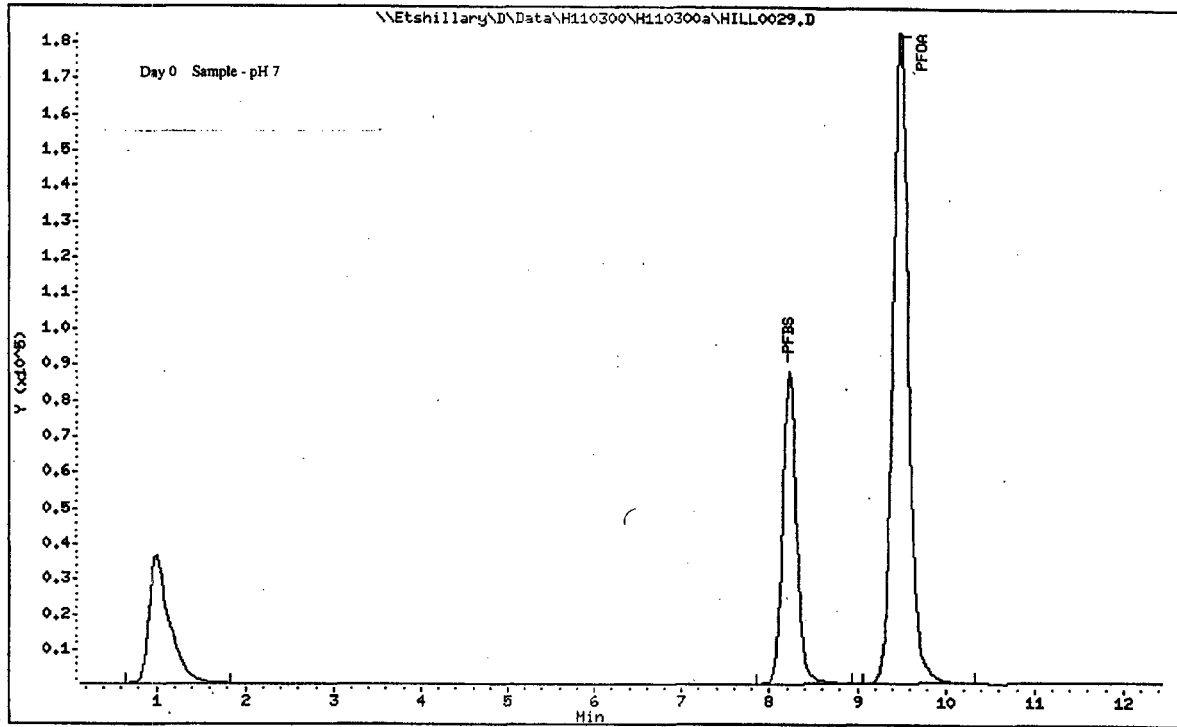
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Lab Smp Id:
Inj Date : 03-NOV-2000 19:40
Operator : DDW
Smp Info : 061500-PFOA-021
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300a\PFOA1103.m
Meth Date : 08-Nov-2000 14:12 wright
Cal Date : 03-NOV-2000 15:20
Als bottle: 13
Dil Factor: 1.00000
Integrator: HP Genie
Target Version: 4.04
Processing Host: WW19507

Inst ID: hillary.i
Quant Type: ISTD
Cal File: HILL0015.D
Compound Sublist: all.sub

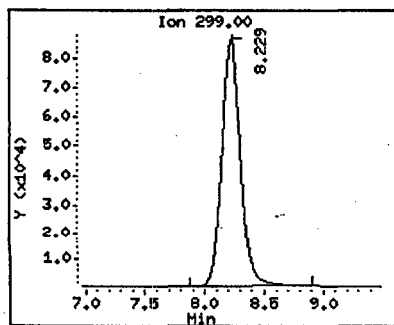
Compounds	QUANT SIG						CONCENTRATIONS	
		MASS	RT	EXP RT	REL RT	RESPONSE	ON-COLUMN (ng/mL)	FINAL (ng/mL)
-----	----	----	----	-----	-----	-----	-----	-----
* 1 PFBS		299	8.228	8.226	(1.000)	962126	158.000	
75 PFOA		413	9.482	9.498	(1.152)	2274515	662.113	662.1

Data File: \\Etshillary\N\Data\H110300\H110300a\HILL0029.D

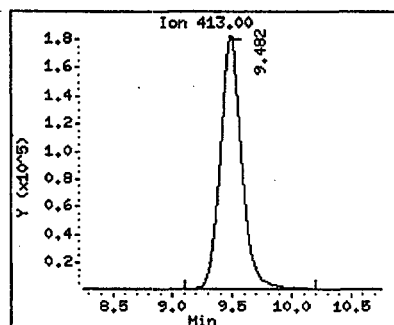
Page 2



1 PFBS



75 PFDA



Data File: \\Etshillary\D\Data\H110300\H110300c\HILL0103.D
Report Date: 08-Nov-2000 13:56

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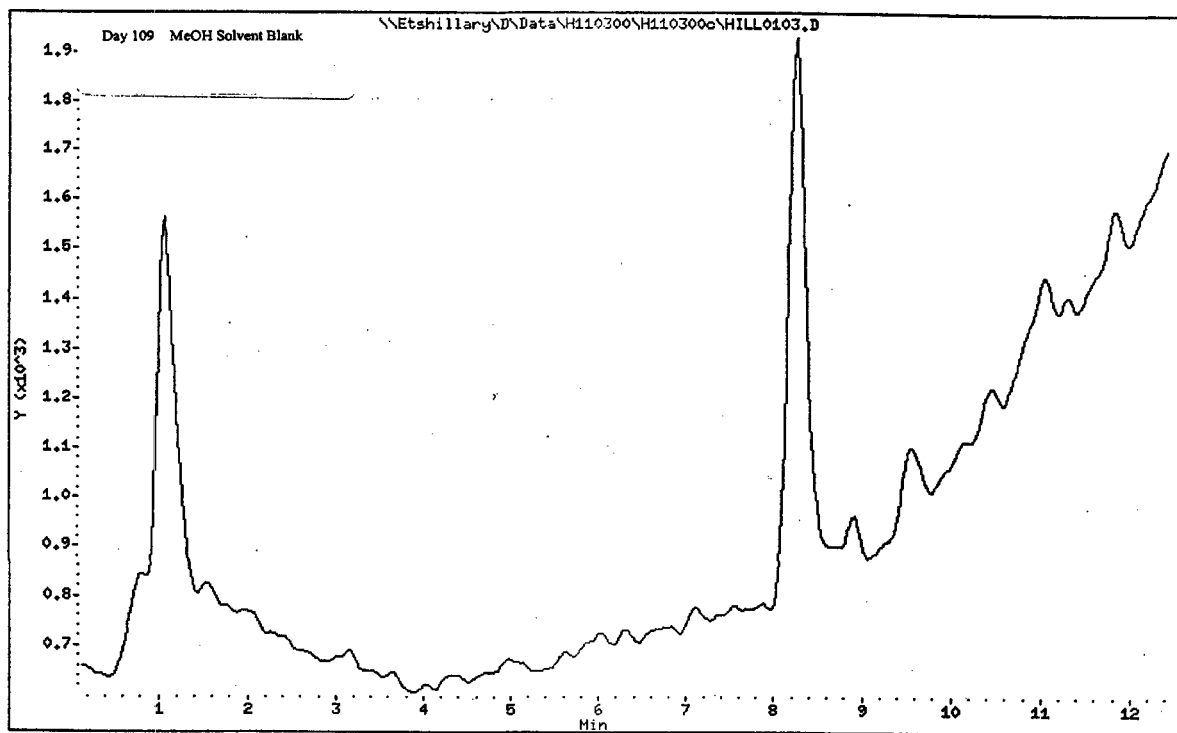
Data file : \\Etshillary\D\Data\H110300\H110300c\HILL0103.D
Lab Smp Id:
Inj Date : 04-NOV-2000 18:33
Operator : DDW
Smp Info : MeOH
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300c\PFOA1103.m
Meth Date : 08-Nov-2000 13:54 wright
Cal Date : 04-NOV-2000 11:08
Als bottle: 100
Dil Factor: 1.00000
Integrator: HP Genie
Target Version: 4.04
Processing Host: WW19507

Inst ID: hillary.i
Quant Type: ISTD
Cal File: HILL0079.D
Compound Sublist: all.sub

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS	
						ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 PFBS	299				Compound Not Detected.		
75 PFOA	413				Compound Not Detected.		

Data File: \\Etshillary\N\Data\H110300\H110300c\HILL0103.D

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Data File: \\Etshillary\D\Data\H110300\H110300c\HILL0104.D
Report Date: 08-Nov-2000 13:56

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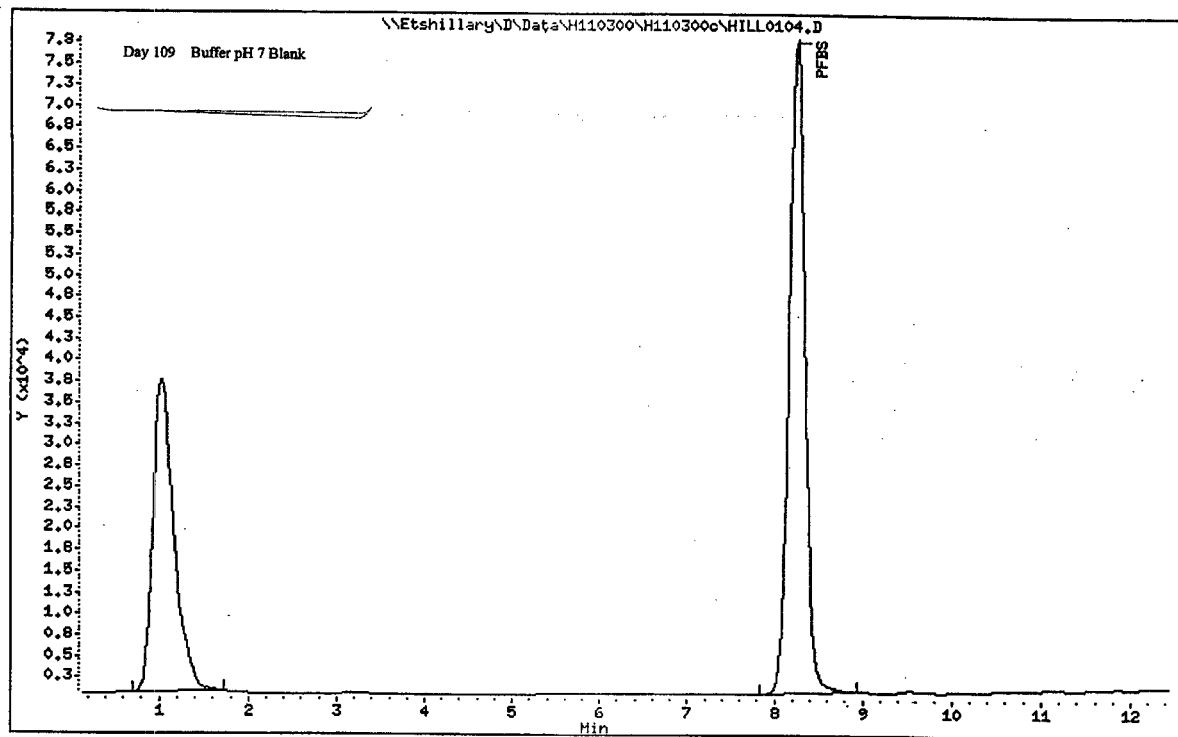
3M Environmental Lab

Data file : \\Etshillary\D\Data\H110300\H110300c\HILL0104.D
Lab Smp Id:
Inj Date : 04-NOV-2000 18:51
Operator : DDW Inst ID: hillary.i
Smp Info : 00028-97-00 pH7
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300c\PFOA1103.m
Meth Date : 08-Nov-2000 13:54 wright Quant Type: ISTD
Cal Date : 04-NOV-2000 11:08 Cal File: HILL0079.D
Als bottle: 1
Dil Factor: 1.00000
Integrator: HP Genie Compound Sublist: all.sub
Target Version: 4.04
Processing Host: WW19507

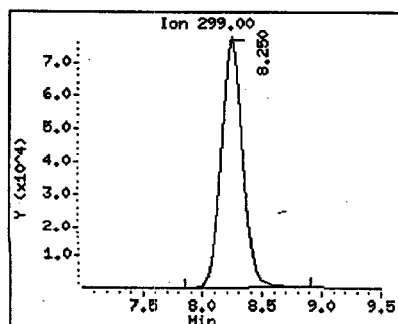
Compounds	QUANT SIG						CONCENTRATIONS	
		MASS	RT	EXP RT	REL RT	RESPONSE	ON-COLUMN (ng/mL)	FINAL (ng/mL)
*****	----	---	--	-----	-----	-----	-----	-----
* 1 PFBS	299	8.250	8.249	(1.000)	959979	158.000		
75 PFOA	413				Compound Not Detected.			

Data File: \\Etshillary\N\Data\H110300\H110300c\HILL0104.D

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1 PFBS



Data File: \\Etshillary\D\Data\H110300\H110300c\HILL0105.D
Report Date: 08-Nov-2000 13:56

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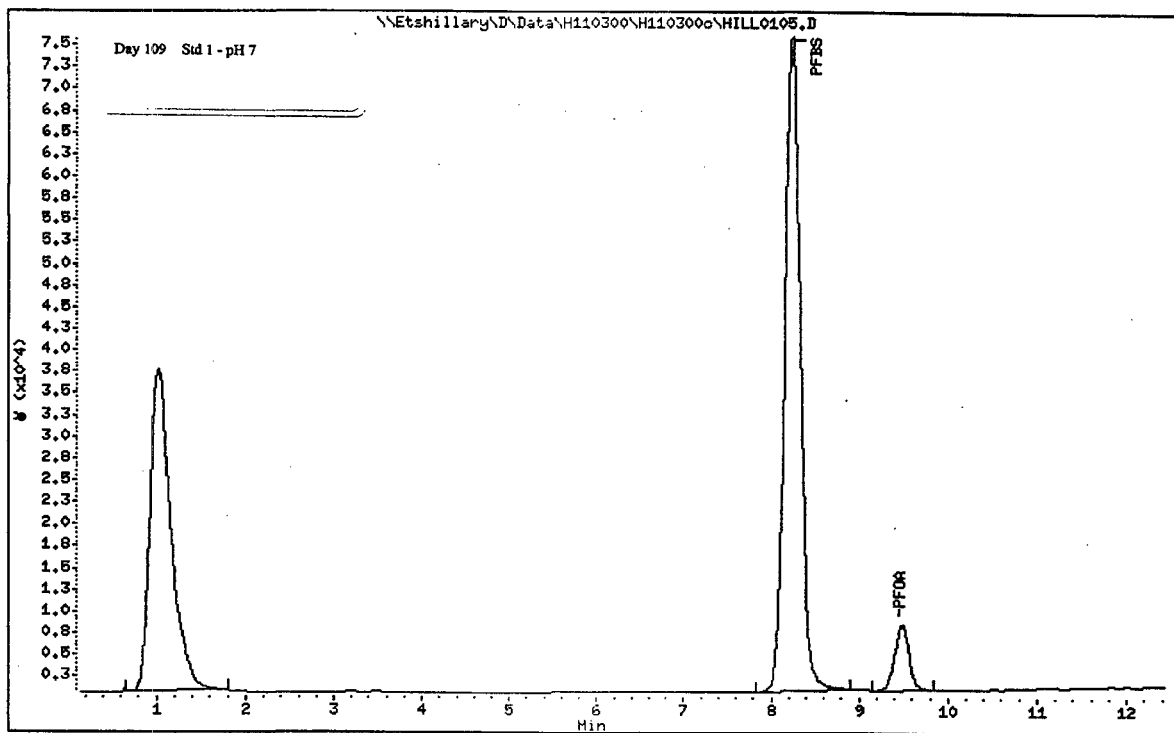
3M Environmental Lab

Data file : \\Etshillary\D\Data\H110300\H110300c\HILL0105.D
Lab Smp Id:
Inj Date : 04-NOV-2000 19:10
Operator : DDW Inst ID: hillary.i
Smp Info : 00028-97-01 pH7
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300c\PFOA1103.m
Meth Date : 08-Nov-2000 13:56 wright Quant Type: ISTD
Cal Date : 04-NOV-2000 11:08 Cal File: HILL0079.D
Als bottle: 2 Calibration Sample, Level: 11
Dil Factor: 1.00000
Integrator: HP Genie Compound Sublist: all.sub
Target Version: 4.04
Processing Host: WW19507

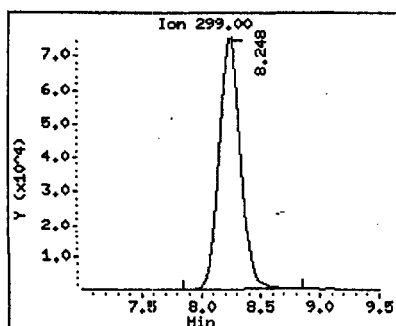
Compounds	QUANT SIG	AMOUNTS					
		RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ng/mL)	ON-COL (ng/mL)
*****	----	==	=====	=====	=====	=====	=====
* 1 PFBS	299	8.248	8.248	(1.000)	933019	158.000	
75 PFOA	413	9.479	9.479	(1.149)	84002	25.0000	27.59

Data File: \\Etshillary\N\Data\H110300\H110300c\HILL0105.D

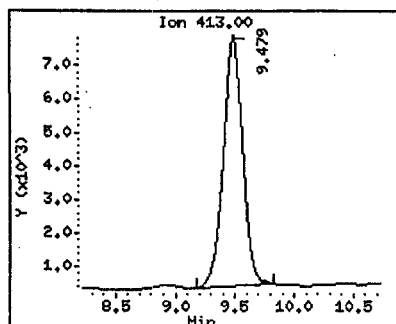
Page 2



* 1 PFBS



75 PFOA



Data File: \\Etshillary\D\Data\H110300\H110300c\HILL0113.D
Report Date: 08-Nov-2000 13:57

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3M Environmental Lab

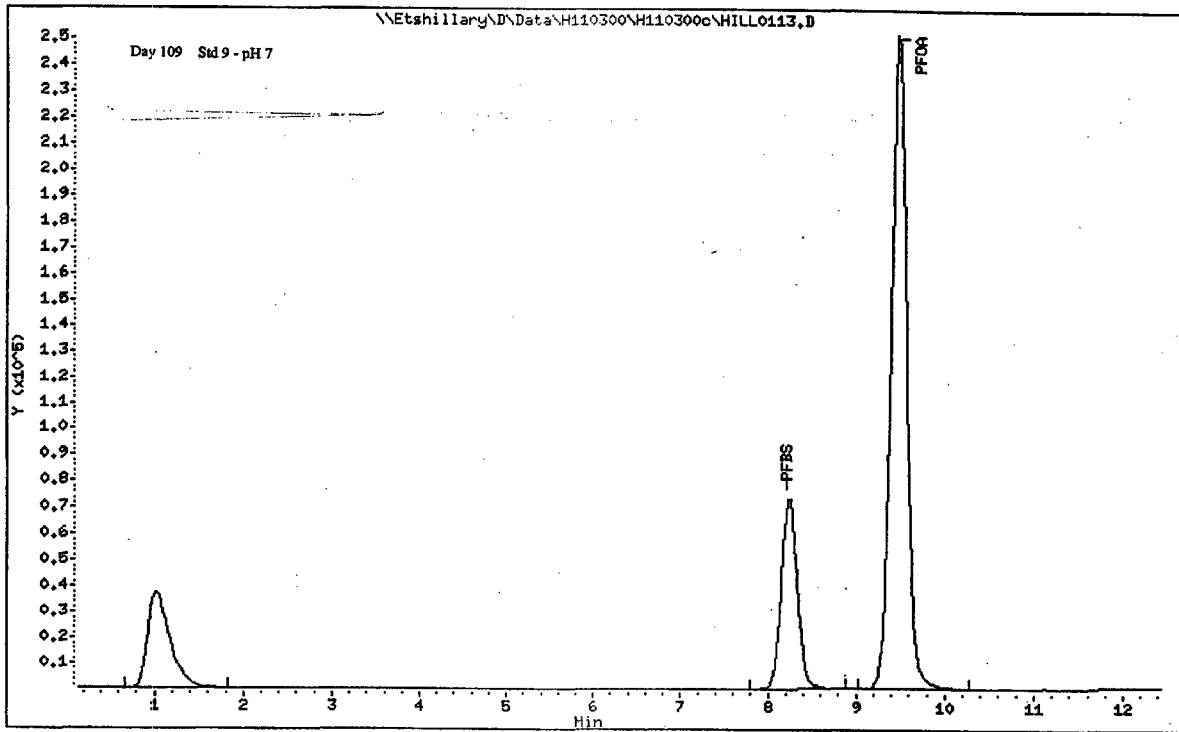
Data file : \\Etshillary\D\Data\H110300\H110300c\HILL0113.D
Lab Smp Id:
Inj Date : 04-NOV-2000 21:38
Operator : DDW
Smp Info : 00028-97-09 pH7
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300c\PFOA1103.m
Meth Date : 08-Nov-2000 13:57 wright
Cal Date : 04-NOV-2000 21:38
Als bottle: 10
Dil Factor: 1.00000
Integrator: HP Genie
Target Version: 4.04
Processing Host: WW19507

Inst ID: hillary.i
Quant Type: ISTD
Cal File: HILL0113.D
Calibration Sample, Level: 19
Compound Sublist: all.sub

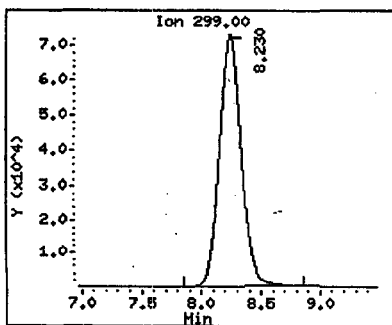
Compounds	QUANT SIG						AMOUNTS	
	MASS	RT	EXP RT	REL RT	RESPONSE		CAL-AMT	ON-COL
							(ng/mL)	(ng/mL)
• 1 PFBS	299	8.230	8.230	(1.000)	927764		158.000	
75 PFOA	413	9.479	9.479	(1.152)	3050552		999.000	1012

Data File: \\Etshillary\N\Data\H110300\H110300c\HILL0113.D

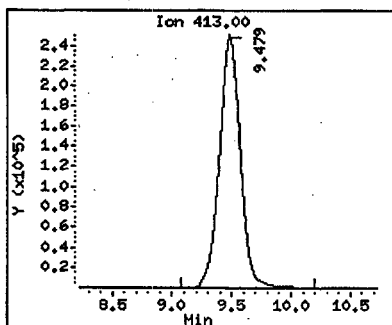
Page 2



M 1 PFBS



75 PFOA



Data File: \\Etshillary\D\Data\H110300\H110300c\HILL0097.D
Report Date: 08-Nov-2000 13:55

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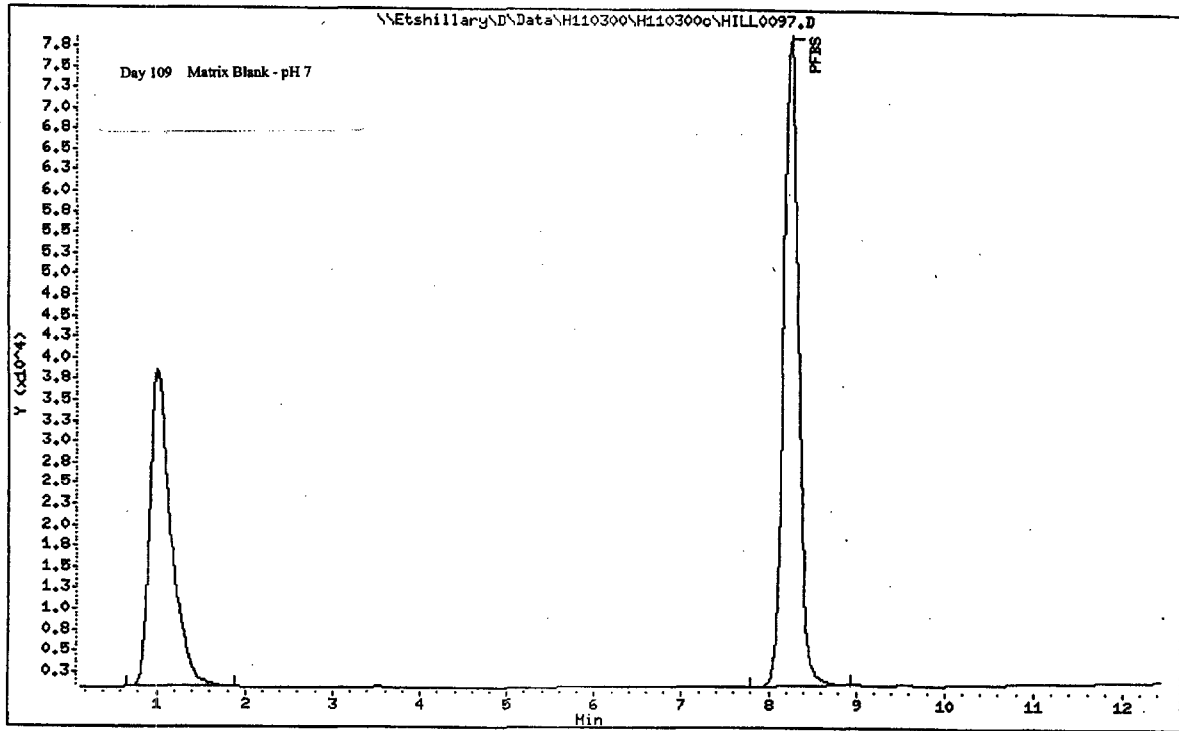
Data file : \\Etshillary\D\Data\H110300\H110300c\HILL0097.D
Lab Smp Id:
Inj Date : 04-NOV-2000 16:41
Operator : DDW
Smp Info : 061500-PFOA-271
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300c\PFOA1103.m
Meth Date : 08-Nov-2000 13:54 wright
Cal Date : 04-NOV-2000 11:08
Als bottle: 53
Dil Factor: 1.00000
Integrator: HP Genie
Target Version: 4.04
Processing Host: WW19507

Inst ID: hillary.i
Quant Type: ISTD
Cal File: HILL0079.D
Compound Sublist: all.sub

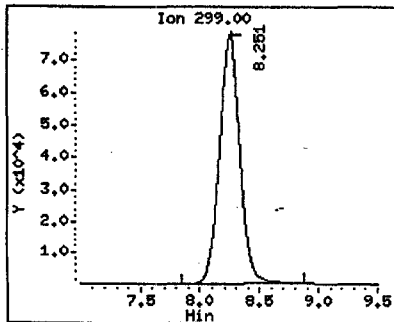
Compounds	QUANT SIG	RT	EXP RT	REL RT	RESPONSE	CONCENTRATIONS	
						ON-COLUMN (ng/mL)	FINAL (ng/mL)
* 1 PFBS	299	8.250	8.249	(1.000)	927163	158.000	
75 PFOA	413	Compound Not Detected.					

Data File: \\Etshillary\DATA\H110300\H110300c\HILL0097.D

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1 PFBS



Data File: \\Etshillary\D\Data\H110300\H110300c\HILL0098.D
Report Date: 08-Nov-2000 13:55

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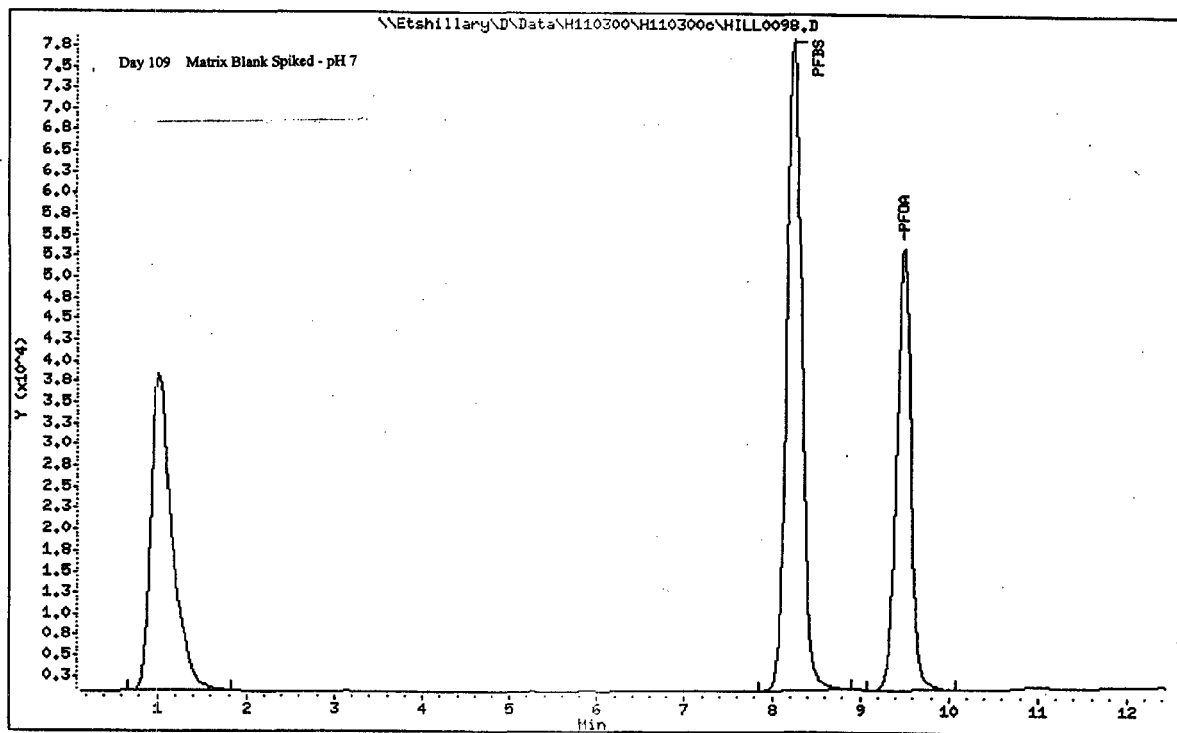
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Lab Smp Id:
Inj Date : 04-NOV-2000 17:00
Operator : DDW
Smp Info : 061500-PFOA-272
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300c\PFOA1103.m
Meth Date : 08-Nov-2000 13:54 wright
Cal Date : 04-NOV-2000 11:08
Als bottle: 54
Dil Factor: 1.00000
Integrator: HP Genie
Target Version: 4.04
Processing Host: WW19507

Inst ID: hillary.i
Quant Type: ISTD
Cal File: HILL0079.D
Compound Sublist: all.sub

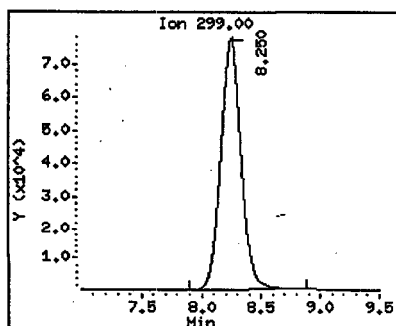
Compounds	QUANT SIG						CONCENTRATIONS	
	MASS	RT	EXP RT	REL RT	RESPONSE		ON-COLUMN	FINAL
							(ng/mL)	(ng/mL)
* 1 PFBS	299	8.249	8.249	(1.000)	924895		158.000	
75 PFOA	413	9.499	9.499	(1.151)	602602		167.470	167.5

Data File: \\Etshillary\N\Data\H110300\H110300c\HILL0098.D

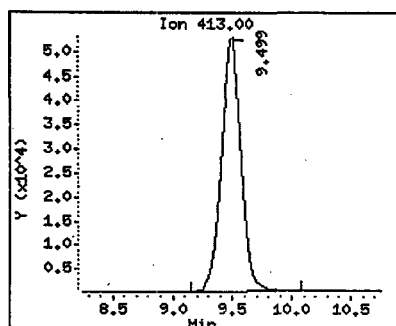
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* 1 PFBS



75 PFDA



Data File: \\Etshillary\D\Data\H110300\H110300c\HILL0099.D
Report Date: 08-Nov-2000 13:56

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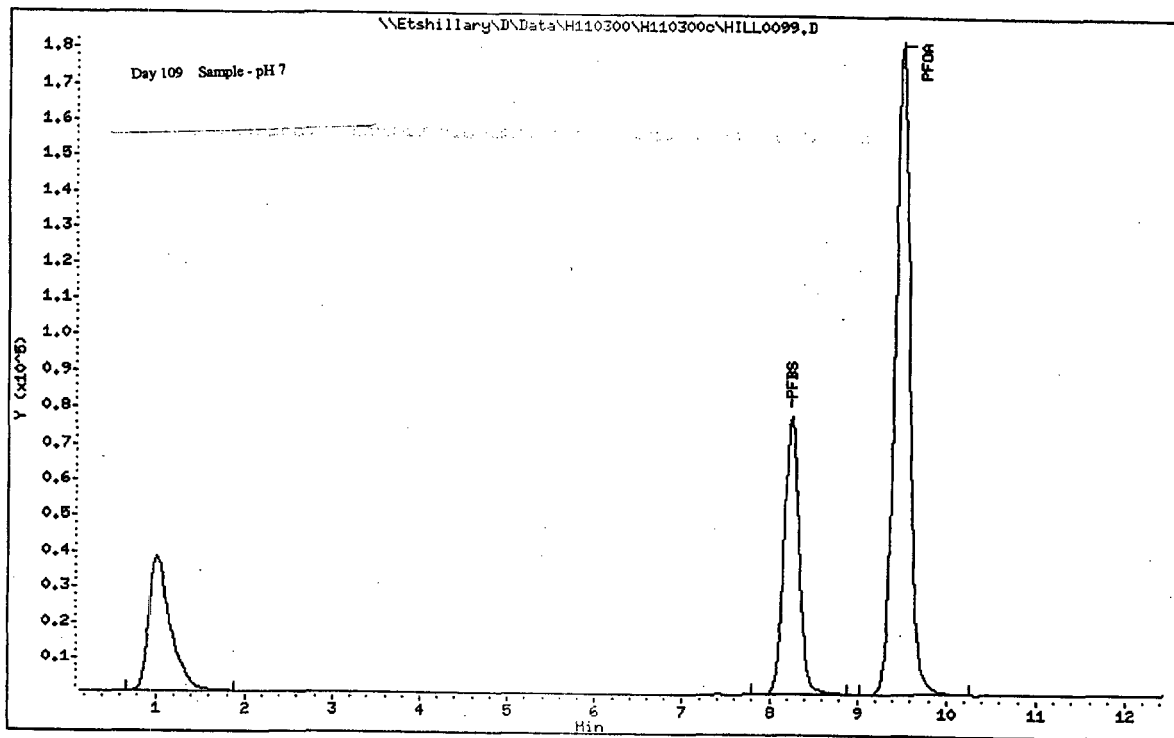
Data file : \\Etshillary\D\Data\H110300\H110300c\HILL0099.D
Lab Smp Id:
Inj Date : 04-NOV-2000 17:19
Operator : DDW
Smp Info : 061500-PFOA-273
Misc Info :
Comment :
Method : \\Etshillary\D\Data\H110300\H110300c\PFOA1103.m
Meth Date : 08-Nov-2000 13:54 wright
Cal Date : 04-NOV-2000 11:08
Als bottle: 55
Dil Factor: 1.00000
Integrator: HP Genie
Target Version: 4.04
Processing Host: WW19507

Inst ID: hillary.i
Quant Type: ISTD
Cal File: HILL0079.D
Compound Sublist: all.sub

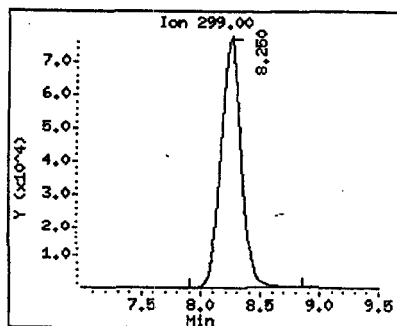
Compounds	QUANT SIG						CONCENTRATIONS	
		RT	EXP RT	REL RT	RESPONSE		ON-COLUMN	FINAL
	MASS						(ng/mL)	(ng/mL)
-----	----	--	-----	-----	-----		-----	-----
* 1 PFBS	299	8.250	8.249	(1.000)	925297		158.000	
75 PFOA	413	9.500	9.499	(1.152)	2146703		651.301	651.3

Data File: \\Etshillary\NData\H110300\H110300c\HILL0099.D

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* 1 PFBS



75 PFOA

