

***Study Title***

Hydrolysis Reactions of Perfluorooctanesulfonamide (FOSA)

***Data Requirement:***

Based on OPPTS: 835.2110

***Author***

Thomas L. Hatfield, Ph.D.

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3M Environmental Laboratory  
Building 2-3E-09, 935 Bush Avenue  
St. Paul, MN 55106

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## Statement of Non-Compliance

Study Title: Hydrolysis Reactions of Perfluorooctanesulfonamide (FOSA)

Study Identification Number: EL1132

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

 4/03/01  
Study Director Date

 04/03/01  
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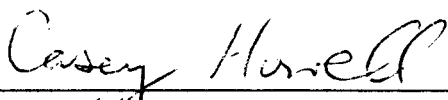
**Quality Assurance Statement**

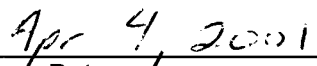
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The following table provides details of the audits performed by the 3M Environmental Laboratory Quality Assurance Unit (QAU).

| Inspection Dates | Phase         | Date Reported to |                |
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| 3/27/01          | Report        | 3/27/01          | 3/27/01        |

  
\_\_\_\_\_  
QAU Representative

  
\_\_\_\_\_  
Date

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## Study Personnel and Contributors

### Study Director

**Thomas L. Hatfield, Ph.D.**  
3M Environmental Laboratory  
Building 2-3E-09  
935 Bush Avenue  
St. Paul, MN 55106  
(651) 778-7863

### Sponsor

3M Corporation

### 3M Environmental Laboratory and Professional Services Contributing Personnel

Kuruppu Dharmasiri, Ph.D.  
Mark T. McCann  
Anthony E. Scales  
Joseph J. S. Tokos, Ph.D.  
(Pace Analytical Services, Inc., 1700 Elm St., Minneapolis, MN 55144)

Gregory Maisel  
Jill Maloney  
(Braun Intertec Corporation, 6875 Washington Ave. South, Minneapolis, MN 55439)

Grant M. Plummer, Ph.D.  
(Rho Squared, P.O. Box 61536, Durham, NC 27715)

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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:

3M Environmental Laboratory  
Building 2-3E-09  
935 Bush Avenue  
St. Paul, MN 55106

## Summary

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

During this study, we prepared and examined samples at six different pH levels from 1.5 to 11.0 over a period of 42 days. The slopes of the log-concentration ratios, calculated from observations of the FOSA concentrations pooled over the six observed pH levels, are presented in Table 1.

| <b>Table 1. Summary of Results Based on FOSA Concentrations.</b> |                                                                     |                                                                     |
|------------------------------------------------------------------|---------------------------------------------------------------------|---------------------------------------------------------------------|
| <b>Calculated slope<br/>(day<sup>-1</sup>)</b>                   | <b>Calculated slope<br/>upper limit (2σ)<br/>(day<sup>-1</sup>)</b> | <b>Calculated slope<br/>lower limit (2σ)<br/>(day<sup>-1</sup>)</b> |
| $-4.4 \times 10^{-6}$                                            | $8.6 \times 10^{-4}$                                                | $-8.7 \times 10^{-4}$                                               |

We also monitored the concentration of one of the potential hydrolysis products (perfluorooctane sulfonate, PFOS), but never observed this compound at levels above its limit of quantification (LOQ, equal to 52 ng/mL). Assuming PFOS to be the only hydrolysis product of FOSA, this LOQ provides a second estimate of the minimum FOSA half-life, presented in Table 2.

| <b>Table 2. Summary of Results Based on PFOS Limit of Quantification</b> |                                                                        |                                                      |
|--------------------------------------------------------------------------|------------------------------------------------------------------------|------------------------------------------------------|
| <b>Maximum<br/>Possible Rate at<br/>50° C<br/>(day<sup>-1</sup>)</b>     | <b>Maximum<br/>Calculated<br/>Rate at 25° C<br/>(day<sup>-1</sup>)</b> | <b>Calculated Half<br/>Life at 25° C<br/>(years)</b> |
| $2.3 \times 10^{-3}$                                                     | $2.3 \times 10^{-4}$                                                   | $\geq 8.3$                                           |



We also failed to detect PFOS above its limit of detection (LOD, with a value estimated to be below 13.0 ng/ml). This LOD value also provides an estimate of the minimum FOSA half-life, presented in Table 3.

| <b>Table 3. Summary of Results Based on the Estimated PFOS Limit of Detection</b> |                                                            |                                              |
|-----------------------------------------------------------------------------------|------------------------------------------------------------|----------------------------------------------|
| <b>Maximum Possible Rate at 50° C (day<sup>-1</sup>)</b>                          | <b>Maximum Calculated Rate at 25° C (day<sup>-1</sup>)</b> | <b>Calculated Half Life at 25° C (years)</b> |
| $5.7 \times 10^{-4}$                                                              | $5.7 \times 10^{-5}$                                       | $\geq 33$                                    |

According to the data available from this study, the half-life estimates of Tables 2 and 3 represent the minimum possible half-life of the compound FOSA under the assumption that it hydrolyzes to form *only* the compound PFOS.

A third half-life estimate (see Equations B 36 and B37) is available from the mean  $\mu$  and standard deviation  $\sigma$  of the observed FOSA concentrations, assuming that they were essentially constant over the experimental portion of the study. This estimate is

$$\left(T_p^{1/2}\right)_2 \geq \left(T_p^{1/2}\right)_{\min} = \frac{\ln(2)}{(k_p)_{\max}} = \frac{\mu \Delta t \ln(2)}{2\sigma}, \quad \text{Eq. 3}$$

where  $\Delta t$  represents the sample incubation period. Table 4 presents the results of the calculation.

| <b>Table 4. Summary of Results Based on the Mean and Precision of FOSA Measurements</b> |                                                            |                                              |
|-----------------------------------------------------------------------------------------|------------------------------------------------------------|----------------------------------------------|
| <b>Maximum Possible Rate at 50° C (day<sup>-1</sup>)</b>                                | <b>Maximum Calculated Rate at 25° C (day<sup>-1</sup>)</b> | <b>Calculated Half Life at 25° C (years)</b> |
| $1.80 \times 10^{-3}$                                                                   | $1.80 \times 10^{-4}$                                      | $\geq 11$                                    |

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## 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.

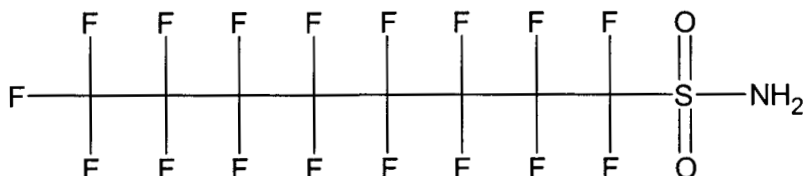
The hydrolysis of FOSA (or, more generally, its degradation in the presence of H<sub>2</sub>O) is addressed in this report. Structures of the "parent" compound FOSA and the potassium salt of its potential hydrolysis product PFOS are illustrated in Figure 1.

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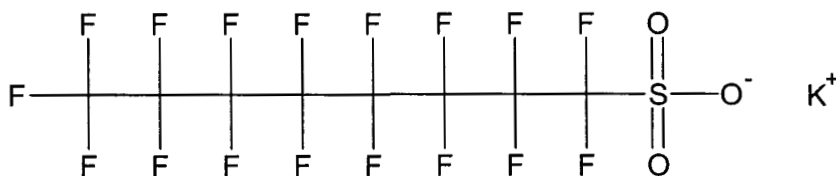
**Figure 1. Structures of FOSA and the Potassium Salt of PFOS**

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### FOSA



### Potassium Salt of PFOS



## Summary of Kinetics Model

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

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

The measured concentrations of the potential hydrolysis product PFOS (also obtained during the experiments described here) provide a second estimate (see Table 2) of the parent half-life. During the course of this study, we did not detect PFOS above its limit of quantitation (LOQ), and related studies<sup>1</sup> show that PFOS is itself hydrolytically stable. Assuming also that PFOS is the only hydrolytic product of the parent compound FOSA, these PFOS analyses provide the following estimate  $(T_p^{1/2})_2$  of the minimum FOSA half-life (see Appendix B, Equations B32 and B33):

$$(T_p^{1/2})_2 \geq \frac{\Delta t [P_0] \ln(2)}{A_{\text{PFOS}}^{\text{LOQ}}} \quad \text{Eq. 2}$$

where

$[P_0]$  = the initial FOSA molar concentration,

$\Delta t$  = the time interval over which the study was conducted (42 days), and

$A_{\text{PFOS}}^{\text{LOQ}}$  = the molar limit of quantitation for the compound PFOS.

Equation 2 implies that when the experimental data provide an estimate of the instrumental limit of detection (LOD) for the compound PFOS ( $A_{\text{PFOS}}^{\text{LOD}}$ ), a third estimate  $(T_p^{1/2})_3$  of the parent half-life is available. This estimate (Equation 3) is identical to that described in Equation 2, with the substitution  $A_{\text{PFOS}}^{\text{LOD}} = A_{\text{PFOS}}^{\text{LOQ}}$  (see Appendix B, Equations B38 and B39):

$$\left(T_p^{1/2}\right)_3 \geq \frac{\Delta t \left[P_0\right] \ln(2)}{A_{\text{PFOS}}^{\text{LOD}}} \quad \text{Eq. 3}$$

All the samples used in this study were maintained at a reaction temperature of 50° C. The quoted results, valid for the reaction temperature of 25° C, were approximated from our experimental results according to methods described in Appendix B (Eq. B40 and B41).

## Materials and Methods

Details of the characteristics of the test materials, sample preparation techniques, and analytical methods are presented in Appendix A (ETS-8-210.0, "Preparation of FOSA, MeFOSA, or EtFOSA Hydrolysis Samples and Analysis by High Performance Liquid Chromatography with Mass Spectrometry Detection.") A summary of these items is provided below, as well as a description the known deviations from the procedures of Appendix A. 3M prepared and analyzed the samples included in this study between March 13 and October 5, 1999.

### Chemical Characterizations

Table 5 describes the sources and properties of the materials used in this work. These materials were used to prepare both the samples and the quantitative standards used to quantify them. For this reason, and because Equations 3 and 6 involve only ratios of the parent and product concentrations, the resulting rate and half-life estimates are largely independent of the material purity levels.

| Table 5. Characterizations of Test and Reference Substances |                        |                        |                                        |
|-------------------------------------------------------------|------------------------|------------------------|----------------------------------------|
|                                                             | FOSA                   | Potassium Salt of PFOS | FBSA <sup>A</sup>                      |
| Source                                                      | 3M Specialty Chemicals | 3M Specialty Chemicals | 3M Specialty Chemicals<br>NB 107715-87 |
| Chemical Lot Number                                         | TNA-1886               | TNA-0844               | TCR-99030-026                          |
| Physical Description                                        | Off-white waxy solid   | Light colored powder   | White powder                           |
| Molecular Weight (gm mole <sup>-1</sup> )                   | 499                    | 538                    | 299                                    |

<sup>A</sup> perfluorobutanesulfonamide

### Sample Preparation

We prepared four 1.0-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 seven time intervals (0, 7, 14, 21, 28, 35, and 42 days). Buffered solutions containing 499 ng/mL of the analyte FOSA and 225 ng/mL of perfluorobutanesulfonamide (FBSA), the latter serving as the internal standard for the compounds FOSA and PFOS, formed the basis of all these samples. The chosen buffer solutions are described fully in Appendix A.

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

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

Five calibration standards containing FOSA (50 to 998 ng/ml) and six standards containing PFOS (13.0 to 1037 ng/ml) served as the fundamental quantitative basis of the study. All other calibration standards were prepared from these fundamental standards, at the appropriate pH levels, using the buffer solutions described above.

### **Sample Analysis**

The equipment we used for the HPLC/ITMS analysis was a Hewlett Packard model 1100 equipped with a Dionex IonPac® NG-1 HPLC column (aqueous ammonium acetate/methanol solvent gradient) and an ALS Model G1322A degassing module. An ALS Model G1315A column heater maintained the column temperature at 40 °C, a quaternary pump supplied a column flow rate of 0.3 mL/min, and an ALS Model G1313A auto-sampler provided 5 µL sample injections. The detector was a Hewlett Packard MSD mass spectrometer, operated in negative-mode electrospray ionization mode; anions of the acetate adduct of FOSA, PFOS, and FBSA were detected at the charge-to-mass ratios 498, 499, and 298, respectively. We processed the resulting data using the computer program *HP ChemStation for LC* (Rev.A.06.0). Further analytical details, including the gradient elution program, instrument and detector parameters, and performance specifications, are presented in Appendix A.

### **Deviations**

We failed to spike one blank sample at each of the pH levels 3.0, 5.0, 7.0, and 9.0. In each case, the results of additional blank samples performed immediately before the actual sample analyses indicate that the reported data meet the pertinent data quality objectives (see below).

Through either human or mechanical error, samples 80399PFOSA-094 through 096 (pH 11.0, "Day 21") were not analyzed, and they have been excluded from the following discussion.

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## Results and Discussion

### Data Quality Objectives (DQO's)

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

- **Calibrations.** The minimum acceptable coefficient of determination ( $r^2$ ) for linear fits to calibration data is 0.990. The acceptance criterion for individual calibration points is that their values fall within  $\pm 25\%$  of the linear fit value; data outside this range are excluded and the linear fit is recalculated. No more than two points may be rejected from a calibration data set. Data for the high or low calibration standards may be rejected, though this results in a smaller effective calibration range.
- **Continuing Calibration Verification (CCV).** Identical calibration samples are examined at the beginning and end of each sample run. Results of the second calibration run may not deviate by more than  $\pm 25\%$  of the first run for any analyte. The average results of the calibration runs are used to calculate the analyte concentrations.
- **Matrix Spikes.** The acceptable percent spike recovery range is 75% to 125%. Analyte specificity is demonstrated by acceptable analyte spike recoveries.
- **Sample Duplicates.** Duplicate pairs with relative percent deviation (RSD) greater than 25% may be accepted at the analyst's discretion, but must be noted.
- **Solvent Blanks.** Concentration results for solvent blanks may exceed neither 5% of the highest calibration standard nor 25% of the lowest calibration level.
- **System Suitability.** Suitability was demonstrated by either an abbreviated mass-to-charge ( $m/z$ ) check-tune or performance of a full auto-tune routine.

### Anomalous Analytical Results

- **Calibrations.** The PFOS calibration standards at 13.0 ng/ml failed to meet the relevant DQO, so we calculated all the reported concentrations according to the calibration results for the PFOS standard at 52 ng/ml. However, the responses to the 13.0 ng/ml were all non-zero, and were quite consistent, so the instrumental limit of detection (LOD) was clearly below this value. This fact is employed below in the "Data Summary and Discussion" section.
- **Spike Recoveries.** As discussed above, the samples 80399PFOSA-094 through 096 failed to meet the related DQO, so we have excluded the "Day 21" data for pH = 11.
- **Other Detected Peaks:** Some pH = 9.0 data indicate the presence of peaks at the value  $m/z = 616$ , corresponding to the compound 2-(*N*-ethylperfluorooctane-sulfonamido). No explanation for the appearance of these peaks is apparent. In addition, a very consistent response was noted at  $m/z = 499$  in all the standards and samples. This response is possibly due to various  $^{13}\text{C}$  forms of FOSA. Because the response is consistent, it does not compromise the reported results for the dominant  $^{12}\text{C}$  FOSA isotopomer at  $m/z = 498$ .

### Statistical Methods and Calculations

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

As described in Appendix B (Equations B40 and B41), 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.<sup>2</sup>

### Data Summary and Discussion

The LOQ is defined as the concentration of the lowest (accepted) standard in the calibration set for which the known concentration exceeds 400% of the indicated solvent blank level (see Appendix A). During this study, the LOQ's for FOSA and PFOS were 50 ng/mL and 52 ng/mL, respectively.

Results for the internal standard compound (PFBS) were reasonably consistent throughout the study. The percent relative standard deviations of the measured values, calculated for each pH level, ranged from 1.8% to 17%.

Table 6 presents the results of the slope determinations (see Appendix B, Equation B8) at six pH levels and 50°C.

| <b>Table 6. Observed (50° C) Degradation Slopes for FOSA in Aqueous Buffered Solutions and at Various pH Levels.</b> |                                          |                                                          |
|----------------------------------------------------------------------------------------------------------------------|------------------------------------------|----------------------------------------------------------|
| <b>PH</b>                                                                                                            | <b>Observed Slope (day<sup>-1</sup>)</b> | <b>Percent (2σ) Slope Uncertainty (day<sup>-1</sup>)</b> |
| 1.5                                                                                                                  | -0.0013                                  | 98                                                       |
| 3.0                                                                                                                  | -0.0009                                  | 261                                                      |
| 5.0                                                                                                                  | -0.0003                                  | 622                                                      |
| 7.0                                                                                                                  | +0.0001                                  | 1145                                                     |
| 9.0                                                                                                                  | +0.0012                                  | 71                                                       |
| 11                                                                                                                   | +0.0011                                  | 115                                                      |

These slopes are generally only poorly determined; their percent relative 2σ (95% confidence) uncertainties range from 71% to 1145%. The data do not indicate any degradation of FOSA. A regression of the slopes vs. pH yields a line with well-determined slope; however, considering the near-zero values of the slopes, their generally large uncertainties, and the lack of physical meaning of the positive slopes, this trend is not meaningful. We conclude that the data indicate no relationship between the degradation of FOSA and the sample pH level.

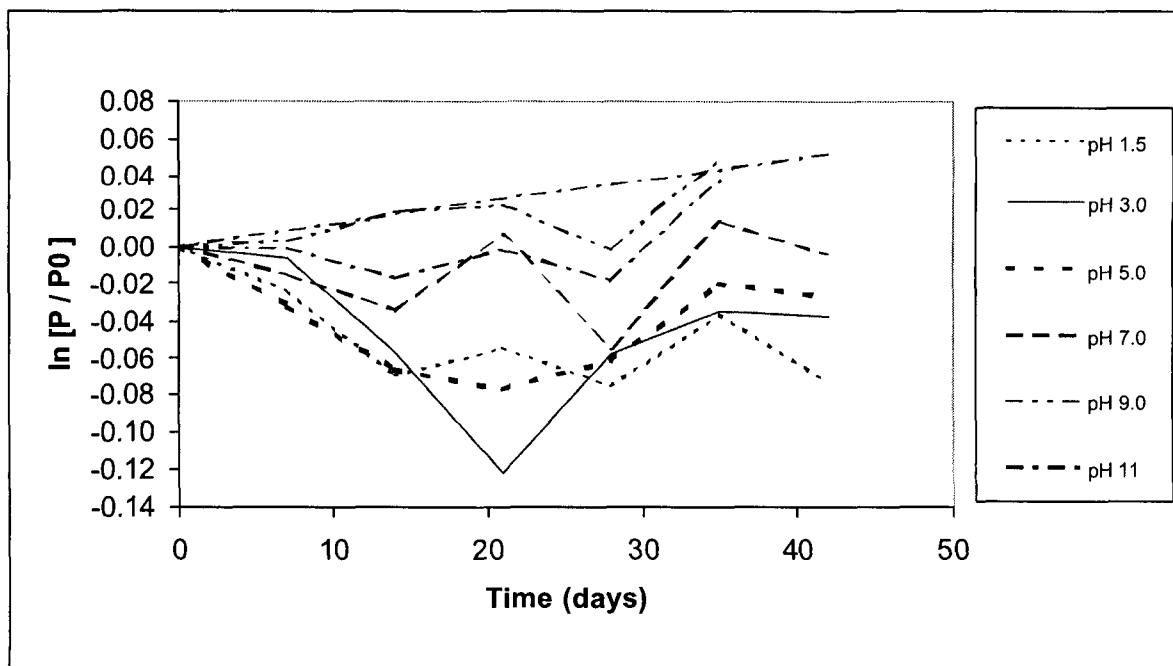


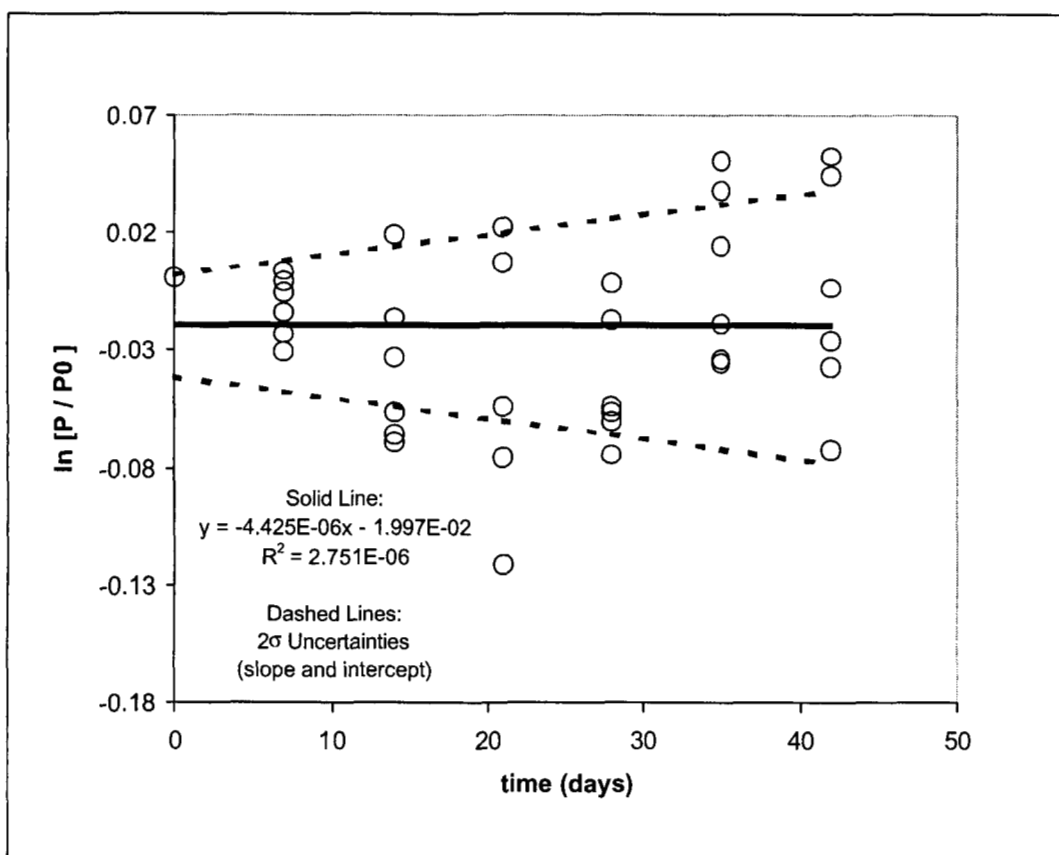
In the absence of a clear relationship between the FOSA degradation rate and the sample pH, it is appropriate to "pool" the data over pH level and determine the degradation rate using the entire data set. Figure 3 illustrates the results of this pooled analysis according to Equation 1, and Table 7 summarizes the results of the analysis.

| <b>Table 7. Degradation Rate Constant of FOSA in Aqueous Buffered Solutions Using Data Pooled Over pH Levels.</b> |                                                                           |
|-------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------|
| <b>Observed Rate Constant at 50° C (day<sup>-1</sup>)</b>                                                         | <b>Percent (2σ) Rate Constant Uncertainty at 50° C (day<sup>-1</sup>)</b> |
| $4.43 \times 10^{-6}$                                                                                             | 19000                                                                     |

The uncertainty in the rate constant presented in Table 7, which is based on direct observations of the compound's concentration in aqueous solution, indicates that FOSA is hydrolytically stable to within the experimental limitations of this study.

**Figure 2. Observed FOSA Degradation for Various pH levels.**



**Figure 3. Pooled FOSA Data and Slope Regression.**

We also monitored the concentration of the potential hydrolysis product (PFOS), but never observed this compound at levels above its limit of quantification (LOQ, equal to 52 ng/mL). The initial FOSA concentration (499 ng/mL) and the PFOS LOQ provide an estimate of the FOSA half-life (see in Appendix B, Equations B32 and B33). The maximum degradation rate is given by Equation 3:

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

and the minimum half-life is given by Equation 4

$$(T_{p/2}) \geq \frac{\Delta t [P_0] \ln(2)}{A_{\text{PFOS}}^{\text{LOQ}}} \quad \text{Eq. 4}$$

We note that in both Equations 3 and 4, the initial FOSA concentration ( $P_0$ ) and the PFOS LOQ ( $A_{\text{PFOS}}^{\text{LOQ}}$ ) are molar quantities. Table 8 presents the results of the related calculations.

| <b>Table 8. Degradation Rate and Half Life of FOSA in Aqueous Buffered Solutions Based on PFOS Limit of Quantification</b> |                    |                                           |                                                              |                                                                |                                                |
|----------------------------------------------------------------------------------------------------------------------------|--------------------|-------------------------------------------|--------------------------------------------------------------|----------------------------------------------------------------|------------------------------------------------|
| $\Delta t$<br>(days)                                                                                                       | $[P_0]$<br>(nm/ml) | $A_{\text{PFOS}}^{\text{LOQ}}$<br>(nm/ml) | Maximum<br>Observed Rate at<br>50° C<br>(day <sup>-1</sup> ) | Maximum<br>Calculated<br>Rate at 25° C<br>(day <sup>-1</sup> ) | Calculated<br>Half Life<br>at 25° C<br>(years) |
| 42                                                                                                                         | 1.0                | 0.097                                     | $2.3 \times 10^{-3}$                                         | $2.3 \times 10^{-4}$                                           | $\geq 8.3$                                     |

Appendix C lists the analytical results for a number of calibration standards containing PFOS at 13.0 ng/ml (e.g. sample 99039-135-02, analyzed just before the initial calibration standard set for pH 1.5). These results failed to meet the relevant data quality objectives for calibration standards, so the test analyte results presented in this report are based on the results for the PFOS standards at 51.9 ng/ml; these latter standards establish the LOQ of 52 ng/ml for PFOS.

Using the accepted calibration regressions for PFOS, the instrumental responses to the 13.0 ng/ml standards had an average value of 16 ng/ml and a standard deviation of 4.4 ng/ml. Although these results did fail to meet the relevant data quality objectives for calibration standards, they do firmly establish the fact that the instrumental LOD for PFOS ( $A_{\text{PFOS}}^{\text{LOD}}$ ) was below 13 ng/ml. The initial FOSA concentration (499 ng/ml) and the PFOS LOD provide an estimate of the FOSA half-life (see in Appendix B, Equations B38 and B39). The maximum degradation rate is given by Equation 5:

$$k_p \leq (k_p)_{\text{max}} = \frac{1}{P_0 \Delta t} \sum_{m=1}^N A_m^{\text{LOD}} \quad \text{Eq. 5}$$

and the minimum half-life is given by Equation 6

$$(T_{1/2})_p \geq \frac{\Delta t [P_0] \ln(2)}{A_{\text{PFOS}}^{\text{LOD}}} \quad \text{Eq. 6}$$

We note that in both Equations 5 and 6, the initial FOSA concentration ( $P_0$ ) and the PFOS LOD ( $A_{\text{PFOS}}^{\text{LOD}}$ ) are molar quantities. Table 9 presents the results of the related calculations.

| <b>Table 9. Degradation Rate and Half Life of FOSA in Aqueous Buffered Solutions Based on the Estimated PFOS Limit of Detection</b> |                    |                                           |                                                              |                                                                |                                                |
|-------------------------------------------------------------------------------------------------------------------------------------|--------------------|-------------------------------------------|--------------------------------------------------------------|----------------------------------------------------------------|------------------------------------------------|
| $\Delta t$<br>(days)                                                                                                                | $[P_0]$<br>(nm/ml) | $A_{\text{PFOS}}^{\text{LOD}}$<br>(nm/ml) | Maximum<br>Observed Rate at<br>50° C<br>(day <sup>-1</sup> ) | Maximum<br>Calculated<br>Rate at 25° C<br>(day <sup>-1</sup> ) | Calculated<br>Half Life<br>at 25° C<br>(years) |
| 42                                                                                                                                  | 1.0                | 0.024                                     | $5.7 \times 10^{-4}$                                         | $5.7 \times 10^{-5}$                                           | $\geq 33$                                      |

The mean and standard deviation of the FOSA concentrations also provide a useful estimate of its half-life. Details of the related calculations are presented in below (see Appendix B, Equations B36 and B37). The maximum degradation rate is given in Equation 7:

$$k_p \leq (k_p)_{\max} = \frac{2\sigma_p}{\mu_p \Delta t} \quad \text{Eq. 7}$$

and the minimum half-life is given in Equation 4

$$T_p^{1/2} \geq (T_p^{1/2})_{\min} = \frac{\ln(2)}{(k_p)_{\max}} = \frac{\mu_p \Delta t \ln(2)}{2\sigma_p} \quad \text{Eq. 8}$$

We note that in both Equations 7 and 8, the mean FOSA concentration ( $\mu_p$ ) and standard deviation ( $\sigma_p$ ) can be either molar or mass quantities. Table 10 presents the results of the calculation.

| <b>Table 10. Degradation Rate and Half Life of FOSA in Aqueous Buffered Solutions Based on the Concentration Mean and Standard Deviation</b> |                    |                       |                                                              |                                                                |                                                |
|----------------------------------------------------------------------------------------------------------------------------------------------|--------------------|-----------------------|--------------------------------------------------------------|----------------------------------------------------------------|------------------------------------------------|
| $\Delta t$<br>(days)                                                                                                                         | $\mu_p$<br>(ng/ml) | $\sigma_p$<br>(ng/ml) | Maximum<br>Observed Rate at<br>50° C<br>(day <sup>-1</sup> ) | Maximum<br>Calculated<br>Rate at 25° C<br>(day <sup>-1</sup> ) | Calculated<br>Half Life<br>at 25° C<br>(years) |
| 42                                                                                                                                           | 455                | 17.2                  | $1.80 \times 10^{-3}$                                        | $1.80 \times 10^{-4}$                                          | $\geq 11$                                      |

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## Conclusions

We have performed a study of the aqueous hydrolytic degradation of perfluorooctane-sulfonamide (FOSA). Six different pH levels were included in the study, which were carried out at 50°C and extrapolated to 25°C. Our results, based on direct observations of the FOSA concentration and a model of the related first-order kinetics, indicate no hydrolytic degradation of FOSA.

We also monitored the concentration of the compound PFOS, a potential hydrolytic product of FOSA. Throughout the study, the PFOS concentration remained below both our limit of quantification (52 ng/mL) and our limit of detection (13.0 ng/ml). Using the LOQ for PFOS and the initial FOSA concentration (499 ng/ml), and assuming PFOS is the only hydrolytic product of FOSA, we estimate that the hydrolytic half-life of FOSA at 25°C is greater than or equal to 8.3 years. Using the LOD for PFOS and the same initial FOSA concentration, and assuming PFOS is the only hydrolytic product of FOSA, we estimate that the hydrolytic half-life of FOSA at 25°C is greater than or equal to 33 years. Finally, using the mean and standard deviation of the observed FOSA concentrations, we estimate that the hydrolytic half-life of FOSA at 25°C is greater than or equal to 11 years.

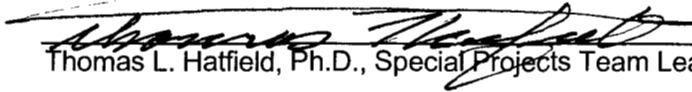
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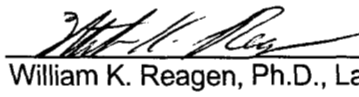
## References

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- <sup>1</sup> "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.
  - <sup>2</sup> "Experimental Physical Chemistry", F. Daniels, et al., McGraw Hill Book Co. (New York), p. 131, 1962.

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**Signatures**

   
Thomas L. Hatfield, Ph.D., Special Projects Team Leader Date

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

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## **Appendix A: Analytical Method**

ETS - 8-210.0, "Preparation of FOSA, MeFOSA, or EtFOSA Hydrolysis Samples and Analysis by High Performance Liquid Chromatography with Mass Spectrometry Detection."

This Appendix presents the analytical method employed in this study.



## 3M ENVIRONMENTAL LABORATORY

### METHOD

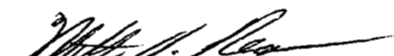
#### PREPARATION OF FOSA, MeFOSA, OR EtFOSA HYDROLYSIS SAMPLES AND ANALYSIS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH MASS SPECTROMETRY DETECTION

Method Number: ETS-8-210.0

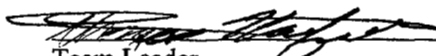
Adoption Date:

Revision Effective Date:

Approved by:

  
Laboratory Manager

03/30/01  
Date

  
Team Leader

03/30/01  
Date

## 1.0 SCOPE AND APPLICATION

- 1.1** This procedure defines common steps for sample preparation, hydrolysis, and analysis of perfluorooctanesulfonamide (FOSA,  $C_8F_{17}SO_2NH_2$ , sometimes referred to as PFOSA), N-methylperfluorooctanesulfonamide (MeFOSA,  $C_8F_{17}SO_2NHCH_3$ , sometimes referred to as FOSMA or PFOSMA), or N-ethylperfluorooctanesulfonamide (EtFOSA,  $C_8F_{17}SO_2NHC_2H_5$ , sometimes referred to as FOSEA or PFOSEA). Hydrolysis products are identified and quantified by high performance liquid chromatography (HPLC) with mass spectrometry (MS) detection. Each test analyte (FOSA, MeFOSA, or EtFOSA) is studied separately using common, multi-component spike and internal standard solutions. The method is based on EPA OPPTS: 835.2110 (Reference 18.1). FOSA is quantified using FBSA (perfluorobutanesulfonamide,  $C_4F_9SO_2NH_2$ , sometimes called PFBSA) as an internal standard. MeFOSA and EtFOSA may be quantified using either N-methylperfluorobutanesulfonamide (N-MeFBSA,  $C_4F_9SO_2NHCH_3$ , sometimes called PFBSMA or MePFBSA) or THPFOS (the anion of 3, 3, 4, 4, 5, 5, 6, 6, 7, 7, 8, 8, 8-tridecafluorooctane sulfonic acid) as internal standard. Perfluorooctanesulfonate (PFOS anion, a potential hydrolysis product) may be quantified using either FBSA or THPFOS as internal standard. External standard quantification is permitted for all chemical species with appropriate technical justification. Representative structures are shown in Attachment A.
- 1.2** **Compatible analytes.** FOSA, MeFOSA, EtFOSA, FBSA, N-MeFBSA, THPFOS, and PFOS.
- 1.3** **Acceptable matrices for analysis.** Aqueous solutions at various buffered pH levels.
- 1.4** This is a performance-based method. Refer to Section 10 for the quality control parameters to be analyzed by this method. Refer to Section 14 for the quality assurance evaluation criteria for this method.

## 2.0 SUMMARY OF METHOD

- 2.1** Aliquots of FOSA, MeFOSA, or EtFOSA test-analyte stock solution are added to vials that contain buffers at pH levels 1.5, 3.0, 5.0, 7.0, 9.0, and 11.0. The vials are then placed in an orbital incubator/shaker set at  $50 \pm 3^\circ C$ . Sets of vials (sample, duplicate, triplicate, spike, and blank) are removed at designated intervals and the date and time recorded. An aliquot of mixed spike solution (containing PFOS, FOSA, MeFOSA, and EtFOSA) is added to the "spike" samples, and all vials are diluted 1:10 with methanol (MeOH) containing an internal standard mixture (e.g. THPFOS, FBSA, and N-MeFBSA). Samples are separated on a reverse phase Dionex IonPac® NG-1 HPLC column using an ammonium acetate/MeOH solvent gradient, with detection by electrospray ionization mass spectrometry in the negative mode.

## 3.0 DEFINITIONS

- 3.1** **Solvent Blank.** A sample of analyte-free medium (e.g. MeOH) that is not taken through the sample preparation process. This blank is used to evaluate instrument contamination.

- 3.2 Buffer Blank.** A sample of each buffer used in the hydrolysis incubations that is not taken through the sample preparation process.
- 3.3 Laboratory Control Sample (LCS).** In this method, a sample of each buffer that is spiked with spiking solution and internal standard solution. This sample does not undergo hydrolysis and is prepared prior to analysis.
- 3.4 Continuing Calibration Blank (CCB).** A sample of analyte-free medium that matches the matrix of the standards used to calibrate the instrument and is run in tandem with the Continuing Calibration Verification (CCV). The CCB is used to evaluate carryover from the standards and instrument baseline, and is run periodically throughout the analytical run (see Section 10 for frequency).
- 3.5 Calibration Standard.** A stock, intermediate or purchased standard diluted appropriately to achieve standard solutions of test analyte in a concentration range of interest.
- 3.6 Continuing Calibration Verification (CCV).** Standards analyzed during an analytical run to verify the continued accuracy of the calibration curve. This solution may be prepared from a different source or lot number than the calibration curve standards.
- 3.7 Internal Standard:** A known amount of a compound similar in analytical behavior to the compound(s) of interest, added to all samples and standards, and carried through the entire measurement process. It provides a reference for evaluating and controlling the precision and bias of the applied analytical method.
- 3.8 Matrix Spike (MS).** Prepared by adding a known mass of target analyte to a specified amount of a sample matrix exposed to hydrolytic conditions for which an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the matrix on the method's recovery efficiency.
- 3.9 Sample Triplicates.** Three samples taken from and representative of the same sample source and carried through all steps of the sampling, treatment and analytical procedures in an identical manner. Sample triplicates are used to assess variance of the total method, including sampling, treatment and analysis.
- 3.10 Dilution.** A step in the hydrolysis study procedure in which a solvent (e.g. MeOH) is added to the test analyte/buffer solution to prepare it for instrumental analysis. This step occurs after the vials are removed from incubation and before the samples are analyzed. IS(s) may be incorporated into the diluting solvent, if desired.
- 3.11 Limit of Quantitation (LOQ).** The lowest concentration that can be reliably measured within specified limits of accuracy during routine laboratory operating conditions. The LOQ is generally 5 to 10 times the minimum concentration with a 99% confidence limit that the concentration is greater than zero. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes, the LOQ is selected as the lowest non-zero standard in the calibration curve that is greater than 4 times the level of the solvent blanks. Sample LOQs are highly matrix-dependent.

#### **4.0 WARNINGS AND CAUTIONS**

##### **4.1 Health and safety warnings**

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- 4.1.1 Wear the proper lab attire for all parts of this procedure. Wear gloves and proper eyewear at all times.
- 4.1.2 Handle all solvents in a hood for all parts of the described sample preparation procedure.
- 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 All glassware in which standards are prepared should be triple-rinsed with 1:1 acetone/MeOH to reduce the possibility of accidental contamination.
- 4.2.2 Ensure that the mobile phase eluents are freshly prepared and that there is sufficient quantity to complete the run. Do not allow the pump to run dry.
- 4.2.3 Ensure that before starting the run sequence there is ample hard disk space on the computer to save all run data.
- 4.2.4 Ensure that there is enough nitrogen in the supply tank to complete sequence runs.

#### 5.0 INTERFERENCE

- 5.1 Contaminants in solvents, reagents, glassware, and other sample processing or analysis hardware may cause interference. Use the routine analysis of laboratory method blanks to demonstrate that there is no such interference.
- 5.2 Contamination from columns, HPLC tubing, and detector components may cause interference at low detection levels. The routine analysis of solvent blanks must be used to demonstrate that there is no such interference.

#### 6.0 EQUIPMENT

- 6.1 Analytical balance sensitive to 0.1 mg
- 6.2 Shaker, incubator capable of maintaining temperature at  $50 \pm 3$  °C
- 6.3 Hewlett-Packard™ (HP) 1100 HPLC system, or equivalent
  - 6.3.1 Pump, quaternary, Model G1311A, or equivalent
  - 6.3.2 Solvent degasser, Model G1322A or equivalent
  - 6.3.3 Autosampler, ALS Model G1313A, variable injection volume capable
  - 6.3.4 Column heater, Model G1316A
- 6.4 Dionex IonPac®NG-1, 35 mm x 4.0 mm, 10 µm packing, or equivalent
- 6.5 Mass spectrometer, Hewlett-Packard LC/MSD, or equivalent, capable of operating in the selected-ion-monitoring mode
- 6.6 Clock, digital
- 6.7 Centrifuge capable of maintaining 3000 rpm for 5 minutes
- 6.8 pH meter, Corning™ Model 308 pH/Temperature Meter with 3-in-1 gel-filled combination electrode (pH/reference/temperature), or equivalent
- 6.9 Refrigerator, capable of maintaining  $4 \pm 3$  °C

- 6.10 Data system, a personal computer capable of controlling the HPLC system as well as recording and processing signals from the detector
- 6.11 Data analysis software: Hewlett-Packard - ChemStation®, Version A.6.03 or later

## 7.0 SUPPLIES AND MATERIALS

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- 7.1 VOA (volatile organic analysis) vials, 40 mL, I-CHEM or equivalent
- 7.2 Crimp cap autovials, 1.5 mL
- 7.3 Labels
- 7.4 Graduated pipets, glass, disposable, 1 mL–10 mL
- 7.5 Pasteur pipets, glass, disposable
- 7.6 Hamilton Gastight® syringes (precision  $\pm 1\%$  of total volume), 10  $\mu\text{L}$ –1000  $\mu\text{L}$
- 7.7 Volumetric flasks, various sizes
- 7.8 Beakers, glass, various sizes
- 7.9 Automatic pipettor, capable of dispensing 10–5000  $\mu\text{L}$

## 8.0 REAGENTS AND STANDARDS

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- 8.1 **Methanol (MeOH):** HPLC/SPEC/GC grade from EM Science, or equivalent
- 8.2 **Acetone:** HPLC/SPEC/GC grade from EM Science, or equivalent
- 8.3 **18.0 M $\Omega$  water.** Water with resistance < 18.0 M $\Omega$  must not be used.
- 8.4 **Calibration and standard stock solutions**
- 8.5 All weights should be recorded to the nearest 0.0001 g
  - 8.5.1 FOSA prepared in MeOH. (Example: An FOSA stock solution is prepared at a concentration of approximately 14,350  $\mu\text{g/mL}$  by weighing approximately 0.1435 g of FOSA in a 10-mL volumetric flask and bringing to the mark with MeOH. This solution is diluted to make additional, appropriate standards.)
  - 8.5.2 MeFOSA prepared in MeOH. (Example: A MeFOSA stock solution is prepared at a concentration of approximately 19,900  $\mu\text{g/mL}$  by weighing approximately 0.1990 g of MeFOSA in a 10-mL volumetric flask and bringing to the mark with MeOH. This solution is diluted to make additional, appropriate standards.)
  - 8.5.3 EtFOSA prepared in MeOH. (Example: An EtFOSA stock solution is prepared at a concentration of approximately 56,580  $\mu\text{g/mL}$  by weighing approximately 0.5658 g of EtFOSA in a 10-mL volumetric flask and bringing to the mark with MeOH. This solution is diluted to make additional, appropriate standards.)
  - 8.5.4 PFOS prepared in MeOH. (Example: A PFOS stock solution is prepared at a concentration of approximately 30,520  $\mu\text{g/mL}$  by weighing approximately 0.3052 g of PFOS in a 10-mL volumetric flask and bringing to the mark with acetone. This solution is diluted to make additional, appropriate standards.)
  - 8.5.5 Ammonium acetate buffer, approximately 2 mM. This is chromatographic eluent A. See Section 12.3.1. (Example: An acceptable buffer solution is made by weighing out approximately 0.16 g of ammonium acetate into a weigh boat and then quantitatively transferring to a 1-L volumetric flask. Add

approximately 500 mL of 18.0 MΩ water and 10 mL of MeOH as a preservative. Dilute to the mark with 18.0 MΩ water and mix thoroughly.)

## 8.6 Internal standards

- 8.6.1** THPFOS prepared in MeOH. (Example: A THPFOS stock solution is prepared at a concentration of approximately 2,000 µg/mL by weighing approximately 0.0200 g of THPFOS in a 10-mL volumetric flask and bringing to the mark with MeOH. This solution is diluted to make additional, appropriate standards.)
- 8.6.2** FBSA prepared in MeOH. (Example: An FBSA stock solution is prepared at a concentration of approximately 2,000 µg/mL by weighing approximately 0.0200 g of FBSA in a 10-mL volumetric flask and bringing to the mark with MeOH. This solution is diluted to make additional, appropriate standards.)
- 8.6.3** N-MeFBSA prepared in MeOH. (Example: An N-MEFBSA stock solution is prepared at a concentration of approximately 2,000 µg/mL by weighing approximately 0.0200 g of N-MEFBSA in a 10-mL volumetric flask and bringing to the mark with MeOH. This solution is diluted to make additional, appropriate standards.)

## 8.7 Buffers for calibration of pH meter

Purchased pH calibration standards of pH 4.0, 7.0, and 10.0 (suppliers vary).

## 8.8 Buffer solutions for hydrolysis study

Prepare pH 5.0 buffer solution using guidelines from *Fate, Transport and Transformation Test Guidelines* (Reference 18.1). Prepare buffer solutions of pH = 1.5, 3.0, 7.0, 9.0, and 11.0 at ambient room temperature using guidelines from the *CRC Handbook of Chemistry and Physics* (Reference 18.2). Prepare the buffer solutions in 1-L quantities. Calibrate a portable pH/temperature meter using purchased pH calibration standards of pH 4.0, 7.0, and 10.0, and measure the pH of all buffer solutions. The concentrations are presented below. Record final pH measurements of all buffers. Store buffers in sealed glass containers.

### 8.8.1 pH 1.5

- 8.8.1.1** 207 mL of 0.2 M HCl
- 8.8.1.2** 250 mL of 0.2 M KCl
- 8.8.1.3** Adjust pH to 1.5 with additional 1 N HCl
- 8.8.1.4** Bring to a final volume of 1 L with 18.0 MΩ water

### 8.8.2 pH 3.0

- 8.8.2.1** 500 mL of 0.1 M Potassium Hydrogen Phthalate
- 8.8.2.2** 223 mL of 0.1 M HCL

### 8.8.3 pH 5.0

- 8.8.3.1** 3.8777 g ammonium acetate added to 250 mL 18.0 MΩ water
- 8.8.3.2** Add 250 mL 0.052 M acetic acid
- 8.8.3.3** Add 18.0 MΩ water to approximately 900 mL
- 8.8.3.4** Adjust to pH 5.0 with glacial acetic acid (approximately 0.5 mL)
- 8.8.3.5** Bring to a final volume of 1 L with 18.0 MΩ water

### 8.8.4 pH 7.0

- 8.8.4.1** 500 mL 0.1 M  $\text{KH}_2\text{PO}_4$  buffer
- 8.8.4.2** 291 mL 0.1 N NaOH

- 8.8.4.3 Adjust pH to 7.0 with either 1 N HCl or 1 N NaOH
- 8.8.4.4 Bring to a final volume of 1 L with 18.0 MΩ water.
- 8.8.5 **pH 9.0**
  - 8.8.5.1 46 mL of 0.1 N HCl
  - 8.8.5.2 500 mL of 0.1 M borax ( $\text{Na}_2\text{BO}_4 \cdot \text{H}_2\text{O}$ )
  - 8.8.5.3 Adjust pH to 9.0 with either 1 N HCl or 1 N NaOH
  - 8.8.5.4 Bring to a final volume of 1 L with 18.0 MΩ water.
- 8.8.6 **pH 11.0**
  - 8.8.6.1 500 mL 0.05M  $\text{NaHCO}_2$
  - 8.8.6.2 227 mL 0.1N NaOH
  - 8.8.6.3 Add water to 950 mL
  - 8.8.6.4 Adjust pH to 11.0 with 1 N NaOH
  - 8.8.6.5 Bring to a final volume of 1 L with 18.0 MΩ water
- 8.9 Test Analyte, Spike, and Diluting solutions:
  - 8.9.1 **FOSA test analyte solution.** [Example: Approximately 150 µL of solution 8.5.1 is diluted in 10 mL MeOH, yielding a concentration of approximately 215 µg/mL FOSA. A 10-µL aliquot of this solution is then added to 1.0 mL of buffer before hydrolysis testing. After hydrolysis, dilution with 9.0 mL MeOH containing internal standards (step performed in Section 12.1.12) results in concentrations before analysis of approximately 215 ng/mL FOSA.
  - 8.9.2 **MeFOSA test analyte solution.** Follow the procedures in Section 8.9.1, but use the MeFOSA test analyte solution prepared in Section 8.5.2.
  - 8.9.3 **EtFOSA test analyte solution.** Follow the procedures in Section 8.9.1, but use the EtFOSA test analyte solution prepared in Section 8.5.3.
  - 8.9.4 **Spiking solution.** [Example: A common post-hydrolysis spiking solution is used for the analysis of FOSA, MeFOSA or EtFOSA hydrolysis samples. This solution contains approximately 50 µg/mL FOSA, 50 µg/mL MeFOSA, 50 µg/mL EtFOSA, and 50 µg/mL PFOS in MeOH. It is made by first preparing intermediate solutions of approximately 500 µg/mL of each compound by MeOH dilution of the solutions prepared in Sections 8.5.1, 8.5.2, 8.5.3, and 8.5.4, respectively. The combined 50 µg/mL FOSA, 50 µg/mL MeFOSA, 50 µg/mL EtFOSA, and 50 µg/mL PFOS solution is made by adding 1.0-mL aliquots of each of the intermediate solutions to a 10-mL volumetric flask and diluted to the mark with MeOH. After the hydrolysis period is complete, 50 µL of the mixed solution is added to each of the "spike" samples. After dilution with 9.0 mL MeOH solution containing internal standard (step performed in Section 12.1.12), the final concentrations of the added spikes in the sample are approximately 250 ng/mL FOSA, MeFOSA, EtFOSA and PFOS].
  - 8.9.5 **Diluting Solution Containing THPFOS, FBSA, and N-MeFBSA Internal Standards.** A common, mixed-stock solution is first prepared in MeOH. This solution is used for the analysis of FOSA, MeFOSA, or EtFOSA hydrolysis samples. [Example: A solution containing approximately 250 µg/mL each THPFOS, FBSA, and N-MeFBSA is prepared by adding appropriate volumes of THPFOS stock (Section 8.6.1), FBSA stock (Section 8.6.2), and MeFBSA stock

(Section 8.6.3) to a 10-mL volumetric flask and diluting to the mark with MeOH. A working dilution solution is prepared by adding 2 mL of the 250 µg/mL mixed THPFOS, FBSA, and N-MeFBSA stock solution to a 2 L volumetric and diluting to the mark with MeOH (final concentration of 250 ng/mL of each compound). After the hydrolysis period is complete, 9 mL of the working solution is added to the sample. The final concentrations of the added internal standards in the sample are approximately 225 ng/mL THPFOS, 225 ng/mL FBSA, and 225 ng/mL N-MeFBSA].

## 9.0 SAMPLE HANDLING

- 9.1 Handle samples and standards in a well-ventilated area. Wear gloves and eyewear whenever handling any solutions.
- 9.2 Record times of initial preparation and quenching additions on the fluorochemical degradation (hydrolysis) Analysis Sample Preparation Data Sheet (Attachment B).
- 9.3 Once the 9.0 mL of final diluting solvent has been added, analyze the samples. Alternatively, aliquots of the methanol-diluted samples should be refrigerated at  $4 \pm 3$  °C or frozen until analysis can be performed.

## 10.0 QUALITY CONTROL (FREQUENCY OF PERFORMANCE)

- 10.1 **Solvent Blank.** Solvent blanks should be run before and after every calibration curve, before and after every CCV (where it is called a CCB), and after no more than 20 sample injections.
- 10.2 **Buffer Blank.** A buffer blank should be analyzed once per run (30 samples or less).
- 10.3 **Laboratory Control Sample (LCS).** An LCS should be analyzed once per run (30 samples or less).
- 10.4 **Continuing Calibration Blank (CCB).** A CCB (solvent blank) is run before and after every CCV.
- 10.5 **Continuing Calibration Verification (CCV).** A mid-range CCV standard, flanked by solvent blanks (CCBs), is run after every 20 samples (or less) to verify the calibration stability. This solution may be prepared from a different source or lot number than the calibration curve standards.
- 10.6 **Internal Standards.** IS(s) are added in a constant concentration to all standards, samples, and matrix spikes. In this method, internal standards are added to the diluting solvent (Section 8.9.5).
- 10.7 **Matrix Spikes.** Prepare a post-hydrolysis matrix spike solution along with each set of triplicate samples at each of the pH levels used in the study (refer to Section 8.9.4). Concentrations of the spike should be approximately equal to the mid-range calibration standard.
- 10.8 **Triplicate Samples.** All samples are prepared in triplicate incubation mixtures for each pH and time point.

## 11.0 CALIBRATION AND STANDARDIZATION



- 11.1 Standard preparation.** Prepare six calibration standards of FOSA, MeFOSA or EtFOSA in MeOH, depending on the choice of test analyte for the analysis. Standards from approximately 10–1000 ng/mL are suggested. This solution should also contain appropriate concentrations of the internal standard.
- 11.2 Calibration standards.** Analyze the calibration standards at the beginning and end of the run. Use the data reduction software program for linear regression calculations to relate the analyte peak area ratio versus the amount ratio from the internal standard. External standard calibration may be used if data review shows a problem with the internal standard analysis. Consult with the group leader for direction prior to performing the external calibration methodology. Note on the standard curve and in the report why a decision was made to perform external standard calibration on the raw data.

## 12.0 PROCEDURES

### 12.1 Sample and spike preparation

- 12.1.1** Before spiking with any of the stock standards, transfer approximately 1 mL of the solution to an autovial and cap it. Use this smaller volume for spiking to minimize the effects of evaporation from stock solutions.
- 12.1.2** Determine the number of time points that will be analyzed. Each time point will have four vials for each pH, multiplied by the number of pH levels analyzed. One vial at each level will be labeled as “sample”, “duplicate”, “triplicate”, or “spike.”
- 12.1.3** Obtain the appropriate number of 40-mL I-CHEM vials with caps and cardboard boxes. Prepare appropriate sample preparation worksheets and create labels and affix to the vials. The labels should include the sample number, pH, time point, and initials of the analyst. Record the pH of each buffer solution.
- 12.1.4** Remove the cap of the I-Chem vial and add 1 mL of the appropriate buffer solution to all of the prelabeled vials. Always replace the cap immediately after any addition to minimize evaporation.
- 12.1.5** To all of the vials, add 10 µL of the appropriate FOSA, MeFOSA, or EtFOSA test analyte solution (prepared in Section 8.9.1, 8.9.2, or 8.9.3) with a 10-µL Gastight® syringe. Record the time of addition for each vial.
- 12.1.6** **For “Time Zero” samples only, proceed to section 12.1.11. For all other samples, continue on to section 12.1.7.**
- 12.1.7** Make sure that the cap has been firmly tightened and place the samples back in the cardboard case.
- 12.1.8** Place the case into a pre-warmed incubator/shaker for the appropriate time. Record the time, temperature, and rate of shaking. The temperature is determined by the conditions of the experiment. Continue to monitor the incubator temperature daily during the entire incubation. Record the temperature on the sample preparation sheet.
- 12.1.9** Remove the case from the incubator at the designated preset time.

- 12.1.10** Remove the vials from the case and place in racks. Allow the vials to cool for 15 minutes to room temperature. Alternatively, freeze the vials if solutions are to be diluted and analyzed at a later date.
- 12.1.11** Using a 10- $\mu$ L gas tight syringe, add 10  $\mu$ L of mixed-spike solution (Section 8.9.4) to the "spike" vials. Invert each vial several times to mix the contents.
- 12.1.12** Using a 10-mL graduated pipet, add 9.0 mL of the MeOH diluting solution prepared in Section 8.9.5 to each vial. Invert each vial several times to mix the contents.
- 12.1.13** Aliquot approximately 1 mL of each sample to the appropriately labeled autovial. Cap the vials and mark the bottom of the meniscus.
- 12.1.14** Place the vials in the HPLC autosampler.
- 12.2 Instrument setup**
- 12.2.1** Check that the appropriate HPLC column is in the instrument for each analysis.
- 12.2.2** Check that the correct eluent solutions are in bottles to be used and that enough is available to complete the sequence run.
- 12.2.3** Place the samples in the autosampler tray and construct a sequence table with appropriate calibration standards, calibration check standards and solvent blanks.
- 12.2.4** Verify that all samples and standards are positioned correctly. Enter sequence information: (sample or standard ID, method name) use one injection per sample.
- 12.2.5** Save sequence as analysis date (e.g. on March 14, 1999, save sequence table as 031499.s). Save all data to a subdirectory labeled with analysis date (e.g., 031499).
- 12.2.6** Set post-sequence command macro to shut down system (Example: "STANDBY" on HP systems).
- 12.3 HPLC set up:**
- 12.3.1** Analysis of FOSA, MeFOSA, or EtFOSA samples from pH levels 1.5, 3.0, 5.0, 7.0, 9.0, and 11.0:

Install the column: Dionex IonPac®NG-1, 4.0 x 35 mm, 10- $\mu$ m packing, or equivalent.

Solvent A: 2 mM ammonium acetate including 1% MeOH

Solvent B: MeOH

**Solvent Gradient (may vary slightly from study to study:**

| TIME (MIN) | %A | %B | FLOW RATE  |
|------------|----|----|------------|
| 0.0        | 60 | 40 | 0.5 mL/min |
| 0.5        | 60 | 40 | 0.5 mL/min |
| 2.5        | 5  | 95 | 0.5 mL/min |
| 6.0 - 7.0  | 5  | 95 | 0.5 mL/min |

Post time: 2.0 – 3.0 minutes; column temperature: 40°C.

## 12.4 Recommended Mass Spectrometer set up:

### 12.4.1 Analysis of FOSA, MeFOSA, or EtFOSA at pH 1.5, 3.0, 5.0, 7.0, 9.0, and 11.0:

|                           |                    |
|---------------------------|--------------------|
| <b>Acquisition Mode</b>   | Selected Ion (SIM) |
| <b>Ionization Mode</b>    | Electrospray       |
| <b>Polarity</b>           | Negative           |
| <b>Drying Gas Flow</b>    | 8 L/min            |
| <b>Nebulizer Pressure</b> | 30 psig            |
| <b>Drying Gas Temp</b>    | 300° C             |
| <b>Capillary Voltage</b>  | 3500 V             |

\*Example conditions are applicable to Hewlett Packard HP1100 equipment only.

### Identifiable ions:

| NAME                            | SIM IONS | GAIN (EMV) | FRAGMENTOR (V) | DWELL (MSEC) |
|---------------------------------|----------|------------|----------------|--------------|
| FBSA (M-H <sup>+</sup> )        | 298.00   | 2.0        | 70             | 97           |
| PFBS (M-H <sup>+</sup> )        | 299.00   | 2.0        | 70             | 97           |
| N-MeFBSA (M-H <sup>+</sup> )    | 312.00   | 2.0        | 70             | 97           |
| THPFOS (M-H <sup>+</sup> )      | 427.00   | 2.0        | 70             | 97           |
| *FOSA (M-H <sup>+</sup> )       | 498.00   | 2.0        | 70             | 97           |
| **PFOS (M-H <sup>+</sup> )      | 499.00   | 2.0        | 70             | 97           |
| MeFOSA (M-H <sup>+</sup> )      | 512.00   | 2.0        | 70             | 97           |
| EtFOSA (M-H <sup>+</sup> )      | 526.00   | 2.0        | 70             | 97           |
| N-MeFOSE-OH (M-H <sup>+</sup> ) | 616.00   | 2.0        | 70             | 97           |
| N-EtFOSE-OH (M-H <sup>+</sup> ) | 630.00   | 2.0        | 70             | 97           |

\*"Quant" ion for FOSA

\*\* "Quant" ion for PFOS

## 12.5 Autosampler set-up\*

|                             |                  |
|-----------------------------|------------------|
| <b>AUTOSAMPLER:</b>         | ALS Model G1313A |
| <b>AUTOSAMPLER PROGRAM:</b> | None             |
| <b>INJECTION VOLUME:</b>    | 5.0 µL           |

\*Example conditions are applicable to Hewlett Packard HP1100 equipment only.

## 12.6 Sample analysis

**12.6.1** Enter the standard, sample and QC information into the sequence table. Analyze solvent blanks and calibration standards first, then up to 20 sample injections, followed by solvent blanks and calibration standards. If more than 20 sample

injections are to be run, analyze a continuing calibration standard (CCV) after every 20 and run the calibration standards again at the end of the sequence. Run solvent blanks before and after each standard curve, before and after the CCV, and after the set of samples to check for any analyte carryover.

- 12.6.2 Identify the electronic acquisition files with an appropriate prefix. Do not exceed five characters if the sequence contains more than 99 lines.
- 12.6.3 Place the standards, samples, and QC (matrix spikes and sample blanks) into the autosampler tray according to the order they are listed in the sequence.
- 12.6.4 Save the sequence table with a name corresponding to today's date. (e.g. if today is December 1, 1998, save the sequence as 120198.S)
- 12.6.5 Start the sequence.

### 13.0 DATA ANALYSIS AND CALCULATIONS

- 13.1 **Peak evaluation:** Peaks must be symmetric in shape and identified by extracting compound specific ions. Peaks considered for calibration must have peak heights greater than 5 (five) times the baseline noise for that region of the chromatogram. Peak integration is from baseline to baseline through a peak using automatic or manual integration. Compounds with isomers present as a shoulder or as a discrete second peak should be integrated with the parent compound unless otherwise noted. Quantitation data are calculated using THPFOS, FBSA, or N-MEFBSA as the internal standards, as appropriate. However, external standard calibration may be acceptable when sample integrity is intact. Consult with the team leader for direction prior to performing the external calibration methodology. Document change in raw data and final report.
- 13.2 **Calculation of k:** Calculate the FOSA, MeFOSA, or EtFOSA concentrations in each of the pH matrices using the curves obtained from the calibrations and the internal standard. Assuming first-order kinetics, a rate constant (k) can be determined by plotting the natural logarithm of the ratio (concentration of FOSA at any given time / initial concentration of FOSA) versus the time (-t). The slope of the resultant line is k. Half-life of the test analyte ( $t_{1/2}$ , in the units of t) is obtained from the relation:  $\ln 2.0/k = 0.693/k$ .
- 13.3 **Matrix spikes.** Calculate the percent recovery for each of the matrix spikes. Using the observed matrix spike recoveries, calculate the average spike recovery. Calculate the matrix spike percent recoveries using the following equation:

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

- 13.4 **Sample triplicates.** Calculate the relative standard deviation (RSD) for the triplicate samples.

$$\frac{\text{STDEV } \{A, B, C\}}{\text{AVERAGE } \{A, B, C\}} \times 100 \%$$

Where: A = the concentration measured in the sample, and  
 B = the concentration measured in the duplicate, and  
 C = the concentration measured in the triplicate

#### 14.0 METHOD PERFORMANCE

- 14.1 Solvent Blanks, Buffer Blanks, and Continuing Calibration Blanks.** The measured value for these blanks should be less than 25% of the limit of quantitation (LOQ) of the method. Solvent blanks should show no more than a 5% carry over from a high standard or calibration check standard. If so, two solvent blanks may be necessary to rule out instrumental contamination. If peaks with greater than 50% of the peak area of a low standard value are observed in sequential solvent blanks, the run should be stopped. This is indicative of instrument contamination. The instrument shall be brought into compliance by thoroughly cleaning the electrospray source, and replacing or cleaning columns, tubing, etc.
- 14.2 Coefficient of Determination ( $r^2$ ).** The coefficient of determination ( $r^2$ ) for linear calibration curves should be 0.990 or greater. The curves should be examined closely for linearity and intercept, particularly for accuracy of quantitation at the low and high ends of the curve. On occasion it may be necessary to utilize exponential or quadratic fits of the data, usually when broad range curves (greater than 3 orders of magnitude between the low and high concentration standards) are used. Record in the raw data the reasons for using quadratic equations, and get approval from QAU. If a point is  $\pm 20\%$  of the nominal value the point is used for quantitation. If the value exceeds  $\pm 20\%$ , the curve is out of range and the curve is rerun until all points pass the criteria.
- 14.3 Continuing Calibration Verification (CCV).** If the relative percent difference for the amount of measured analyte is  $> 15\%$  of the true value, relative to the initial standard curve, stop the run. Only those samples analyzed before the last acceptable CCV will be used. Appropriate steps must be taken to correct the problem before analysis is allowed to proceed (consult the project lead for guidance). Reanalyze the remaining samples with a new calibration curve.
- 14.4 Internal Standards.** Peak-area ratios of samples (relative to the IS) are used for quantification. The %RSD of the internal standards in calibration curves should be  $< 10\%$ , or the project lead should be consulted. The area response of an IS in a sample must be between 50% and 150% of the mean area response of the IS in an acceptable calibration curve. Samples having area response outside this range should be reanalyzed or prepared again, at the discretion of the project lead.
- 14.5 Matrix Spikes.** The analyst shall accept percent spike recovery values of  $100 \pm 25\%$ . Spike recoveries of 70 - 75% or 125 - 130% of nominal may be used at the discretion of the project lead. Spike recoveries  $< 70\%$  or  $> 130\%$  place the analysis out of control. Appropriate steps (e.g. cleaning, blank tests, install a new column) must be taken to correct the problem before the analysis is allowed to proceed.
- 14.6 Sample Triplicates.** The analyst shall accept RPD values of  $< 25\%$ . RPD values  $> 25\%$  place the analysis out of control. Appropriate steps must be taken to correct the problem before analysis is allowed to proceed; consult the group leader for direction.

- 14.7 Limit of Quantitation (LOQ).** The limit of quantitation is equal to the lowest standard in the calibration curve that is  $< 5$  times the blank level or the S/N, whichever is greater.
- 14.8 System Suitability.** System suitability is demonstrated by acceptable instrumental checks (e.g. abbreviated m/z check-tune, or full auto-tune routines). Acceptable checks should be documented in instrument run logs.

#### **15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT**

- 15.1** Dispose of sample waste by placing in high or low BTU containers as appropriate. Use broken glass containers to dispose of glass pipettes.
- 15.2** Collect HPLC solvent waste in the satellite accumulation can. Empty into the flammable storage drum in the hazardous waste collection area on the 2nd floor.
- 15.3** Use smaller bore columns when possible to minimize waste generation.

#### **16.0 RECORDS**

- 16.1** Print out hard copies of all graphics and data analysis summaries for archiving.
- 16.2** Sign and date all graphics and label with instrument ID.
- 16.3** Fill out the hydrolysis sample preparation worksheet completely, making sure to include all initials and dates.
- 16.4** Print out the sample sequence table, photocopy a reduced-size duplicate, and tape the photocopy to the instrument log. Keep the original copy for the raw data files package.
- 16.5** Print chromatograms and internal standard reports for all analyses.
- 16.6** Print calibration tables and curve information and store in the raw data file.
- 16.7** Store hydrolysis sample preparation worksheets in the raw data file.
- 16.8** Enter all standard preparation information in the standards preparation logbook. Make a photocopy of the logbook page and include the copy in the raw data file.
- 16.9** Archive electronic data to appropriate media when necessary.

#### **17.0 ATTACHMENTS**

- 17.1** Attachment A: Representative Chemical Structures
- 17.2** Attachment B: Hydrolysis Sample Logsheet

#### **18.0 BIBLIOGRAPHY**

- 18.1** Fate, Transport and Transformation Test Guidelines Office of Prevention, Pesticides and Toxic Substances (OPPTS) 835.2110 Hydrolysis as a Function of pH, EPA 712-C-98-057, January 1998.
- 18.2** *CRC Handbook of Chemistry and Physics, 1<sup>st</sup> Student Edition*, "Buffer Solutions Operational Definitions of pH," Robert C. Weast, Ph.D., 1988, p. D-87.

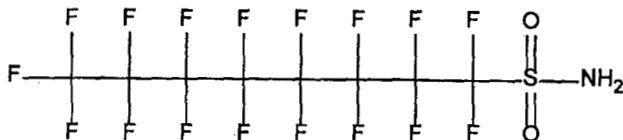
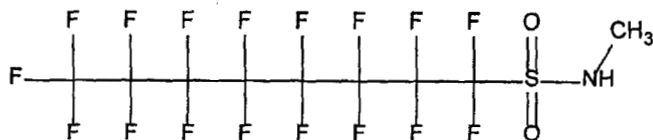
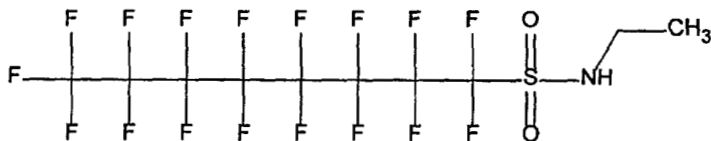
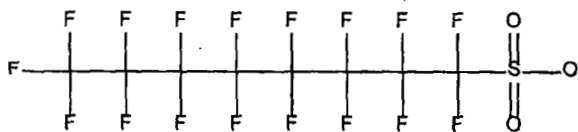
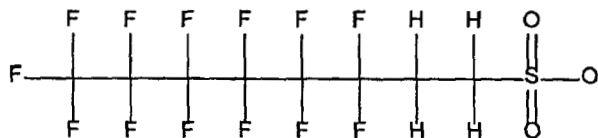
**19.0 AFFECTED DOCUMENTS**

None.

**20.0 REVISIONS**

| <u>Revision<br/>Number</u> | <u>Reason for revision</u> | <u>Date</u> |
|----------------------------|----------------------------|-------------|
|----------------------------|----------------------------|-------------|

## Attachment A: Representative Chemical Structures

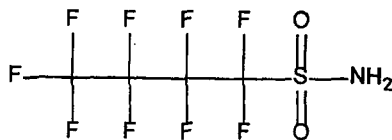
FOSA (MW = 499)MeFOSA (MW = 513)EtFOSA (MW = 527)PFOS (MW of anion = 499)THPFOS (MW of anion = 427)

ETS-8-210.0

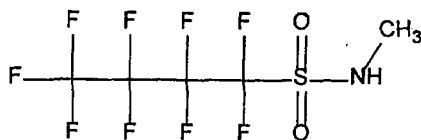
Prep. of FOSA, MeFOSA, or EtFOSA Hydrolysis Samples and Analysis by HPLC/MS Page 16 of 18



FBSA (MW = 299)



N-MeFBSA (MW = 313)



ETS-8-210.0

Prep. of FOSA, MeFOSA, or EtFOSA Hydrolysis Samples and Analysis by HPLC/MS Page 17 of 18

## Attachment B: Hydrolysis Sample Logsheet

## Fluorochemical Degradation (Hydrolysis) Analysis

TEST ANALYTE:

HOURS:

| Sample No. | Description | Time of Initial Prep           | Buffer pH | Buffer Volume (mL) | Test Analyte Solution (μL) | ISTD Solution (μL) | Spike Solution (μL) | Time of Quenching | Quenching Solvent (mL) |
|------------|-------------|--------------------------------|-----------|--------------------|----------------------------|--------------------|---------------------|-------------------|------------------------|
| -          | -           | Sample                         | 1.5       | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Duplicate                      | 1.5       | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Spike <input type="checkbox"/> | 1.5       | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Sample                         | 5         | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Duplicate                      | 5         | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Spike <input type="checkbox"/> | 5         | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Sample                         | 7         | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Duplicate                      | 7         | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Spike <input type="checkbox"/> | 7         | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Sample                         | 9         | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Duplicate                      | 9         | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Spike <input type="checkbox"/> | 9         | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Sample                         | 11        | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Duplicate                      | 11        | 1.0                |                            |                    |                     |                   |                        |
| -          | -           | Spike <input type="checkbox"/> | 11        | 1.0                |                            |                    |                     |                   |                        |

Date of Initial Prep: \_\_\_\_\_

Date of Quenching: \_\_\_\_\_

| Test Analyte Solution           | ISTD Solution | Spike Solution | Quenching Solution |
|---------------------------------|---------------|----------------|--------------------|
| Standard/Traceability No. _____ |               |                |                    |
| Component _____                 |               |                |                    |
| Concentration (μg/mL) _____ NA  |               |                |                    |

Temperature of Incubator (°C): \_\_\_\_\_  
 Incubation Start (Date and Time): \_\_\_\_\_  
 Incubation Stop (Date and Time): \_\_\_\_\_  
 Total Incubation Time: \_\_\_\_\_

Buffer Addition by: \_\_\_\_\_ Centrifugation Yes / No \_\_\_\_\_  
 ISTD Addition by: \_\_\_\_\_ Centrifuge: \_\_\_\_\_  
 Test Analyte Addition by: \_\_\_\_\_ RPM: \_\_\_\_\_  
 Quenching by: \_\_\_\_\_ Time: \_\_\_\_\_ min  
 Spike Addition by: \_\_\_\_\_ By: \_\_\_\_\_  
 Autovial Aliquoting by: \_\_\_\_\_

Filtration: Yes / No \_\_\_\_\_  
 By: \_\_\_\_\_  
 Pore Size: \_\_\_\_\_ μm, Brand: \_\_\_\_\_  
 Reviewed by: \_\_\_\_\_

ETS-8-210.0

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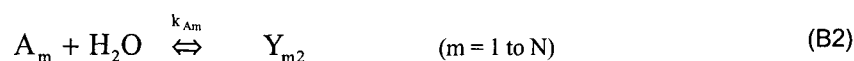
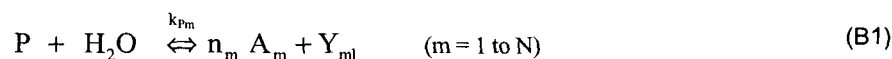
## **Appendix B: Kinetics Model**

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

## **Kinetics Model**

### **B1. Reaction Components and Rates**

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



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

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

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  $m^{\text{th}}$  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 B18 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  $m^{\text{th}}$  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)

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

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

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

### 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  $\Delta t$  all the product concentrations  $A_m$  remain below their limits of quantitation, then the maximum possible value of the rate  $k_p$  is obtained by assuming (for all the products) that 1)  $A_{m0} = 0$  and 2) at time  $t = \Delta t$ , the product concentrations have increased to the values  $A_m = A_m^{LOQ}$ . With these assumptions, the experimental data indicate that the reaction rate  $k_p$  is less than some maximum value  $(k_p)_{max}$  as follows:

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

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

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

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

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

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  $(k_p)_{max}$  in Equation B32 may not actually represent the maximum possible value of the rate constant  $k_p$ , and the related result in Equation B33 for  $(T_p^{1/2})_{min}$  is also questionable.

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

In certain experiments, some hydrolysis products are present at quantifiable but essentially constant concentrations over the time ( $\Delta t$ ) of the experiment. In this case, it is the experimental precision of the measured product concentrations, rather than the limits of quantitation, which contribute to the estimate of the maximum value of the parent hydrolysis rate  $k_p$ . If the set of concentrations measured for the  $m^{\text{th}}$  product have the mean value  $\mu_m$  and standard deviation  $\sigma_m$ , 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  $\sigma_m$  or 2) concentrations below the limits of quantitation)

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

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  $\sigma_m$  or 2) concentrations below the limits of quantitation)

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

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.

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

In certain experiments, the hydrolytic parent remains at an essentially constant concentration over the time ( $\Delta t$ ) of the experiment. In this case, it is the experimental precision of the measured parent concentrations that determines the maximum value of the parent hydrolysis rate  $k_p$ . If the set of concentrations measured for the parent have the mean value  $\mu_p$  and standard deviation  $\sigma_p$ , 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  $\mu_p$  and standard deviation  $\sigma_p$ )

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

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  $\mu_p$  and standard deviation  $\sigma_p$ )

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

#### **B5. Parent Half-Life Estimates Based on Limits of Detection of the Products**

In every experimental determination of  $k_p$ , there is some set of values  $A_m^{\text{LOD}}$  (the "limits of detection") below which the product concentrations  $A_m$  cannot be reliably detected. If during an experiment carried out over the period of time  $\Delta t$  all the product concentrations  $A_m$  remain below their limits of detection, 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  $A_m = A_m^{\text{LOD}}$ . With these assumptions, the experimental data indicate that the reaction rate  $k_p$  is less than some maximum value  $(k_p)_{\max}$  as follows:

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

$$k_p \leq (k_p)_{\max} = \frac{1}{P_0 \Delta t} \sum_{m=1}^N A_m^{\text{LOD}}. \quad (\text{B38})$$

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

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

$$T_p^{1/2} \geq (T_p^{1/2})_{\min} = \frac{\ln(2)}{(k_p)_{\max}} = \Delta t \ P_0 \ \ln(2) \left[ \sum_{m=1}^N A_m^{\text{LOD}} \right]^{-1} \quad (\text{B39})$$

The reader should note that Equations B38 and B39 are valid only when both 1) the products are hydrolytically stable and 2) the concentrations of *all* the potential products are measured. Otherwise, the quantity  $(k_p)_{\max}$  in Equation B38 may not actually represent the maximum possible value of the rate constant  $k_p$ , and the related result in Equation B39 for  $(T_p^{1/2})_{\min}$  is also questionable.

### **B9. 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  $\Delta H_a$ , Equation B38<sup>B1</sup> provides the following relationship between the hydrolysis rates ( $k_1$  and  $k_2$ ) for that reaction at two different absolute temperatures ( $T_1$  and  $T_2$ ):

$$\frac{k_1}{k_2} = \exp \left\{ \frac{\Delta H_a}{R} \left[ \frac{1}{T_2} - \frac{1}{T_1} \right] \right\} \quad (\text{B40})$$

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

$$\frac{k_1}{k_2} = \exp \left\{ \frac{18}{1.99 \times 10^{-3}} \left[ \frac{1}{323} - \frac{1}{298} \right] \right\} = \exp(-2.35) = 0.095 \quad (\text{B41})$$

Equation B39 indicates that the hydrolysis reactions of interest proceed approximately ten times more slowly at  $25^\circ\text{C}$  than at the chosen experimental temperature of  $50^\circ\text{C}$ . Accordingly, the rate reactions reported here for the temperature  $25^\circ\text{C}$  are ten times lower than those measured at  $50^\circ\text{C}$ , 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:**

<sup>B1</sup> I. N Levine, "Physical Chemistry," McGraw-Hill (New York), pp. 498-501 (1978).

<sup>B2</sup> F. Daniels, et al., "Experimental Physical Chemistry", McGraw Hill (New York), p.131 (1962).

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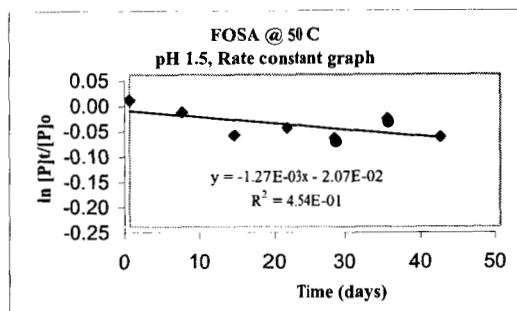
## **Appendix C: Selected Analytical and Kinetics Results**

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

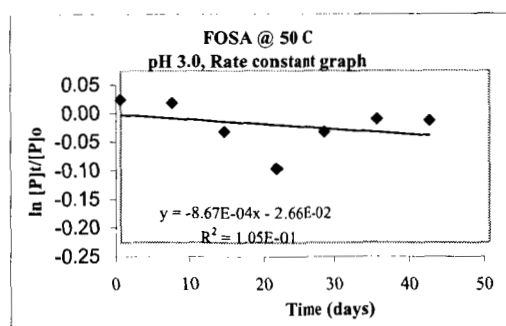
## FOSA Buffer Hydrolysis Study at 50° C.

All concentrations in ng/ml.

| pH  | Time (Days) | Conc. | $\ln([P]_t/[P]_0)$ |
|-----|-------------|-------|--------------------|
| 1.5 | 0           | 475   | 0.000              |
| 1.5 | 7           | 464   | -0.024             |
| 1.5 | 14          | 443   | -0.069             |
| 1.5 | 21          | 450   | -0.054             |
| 1.5 | 28          | 441   | -0.075             |
| 1.5 | 35          | 458   | -0.036             |
| 1.5 | 42          | 441   | -0.073             |



| pH  | Time (Days) | Conc. | $\ln([P]_t/[P]_0)$ |
|-----|-------------|-------|--------------------|
| 3.0 | 0           | 484   | 0.000              |
| 3.0 | 7           | 481   | -0.006             |
| 3.0 | 14          | 457   | -0.057             |
| 3.0 | 21          | 429   | -0.121             |
| 3.0 | 28          | 457   | -0.057             |
| 3.0 | 35          | 467   | -0.035             |
| 3.0 | 42          | 466   | -0.037             |



## SUMMARY OUTPUT

| Regression Statistics |         |
|-----------------------|---------|
| Multiple R            | 0.67379 |
| R Square              | 0.45399 |
| Adjusted R Square     | 0.34479 |
| Standard Error        | 0.02299 |
| Observations          | 7       |

## ANOVA

|            | df | SS      |
|------------|----|---------|
| Regression | 1  | 0.00220 |
| Residual   | 5  | 0.00264 |
| Total      | 6  | 0.00484 |

|              | Coefficients | Standard Error |
|--------------|--------------|----------------|
| Intercept    | -0.02072     | 0.01567        |
| X Variable 1 | -0.00127     | 0.00062        |

% 2σ Slope Uncertainty  
98%

## SUMMARY OUTPUT

| Regression Statistics |          |
|-----------------------|----------|
| Multiple R            | 0.32433  |
| R Square              | 0.10519  |
| Adjusted R Square     | -0.07377 |
| Standard Error        | 0.04187  |
| Observations          | 7        |

## ANOVA

|            | df | SS      |
|------------|----|---------|
| Regression | 1  | 0.00103 |
| Residual   | 5  | 0.00877 |
| Total      | 6  | 0.00980 |

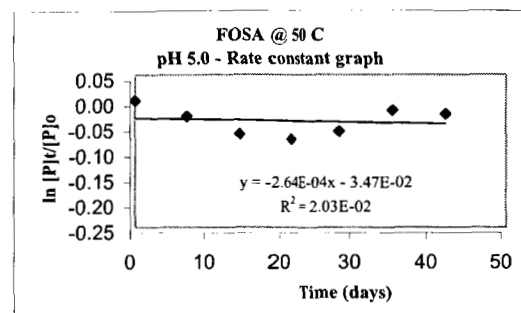
|              | Coefficients | Standard Error |
|--------------|--------------|----------------|
| Intercept    | -0.02664     | 0.02853        |
| X Variable 1 | -0.00087     | 0.00113        |

% 2σ Slope Uncertainty  
261%

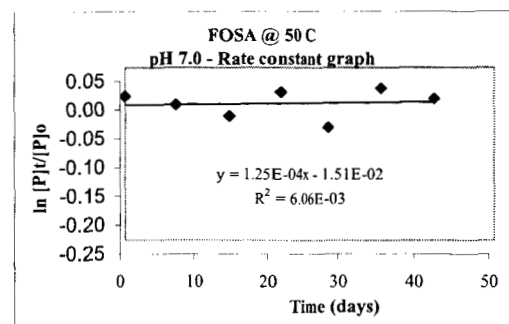
## FOSA Buffer Hydrolysis Study at 50° C.

All concentrations in ng/ml.

| pH  | Time (Days) | Conc. | ln([P] <sub>t</sub> /[P] <sub>o</sub> ) |
|-----|-------------|-------|-----------------------------------------|
| 5.0 | 0           | 470   | 0.000                                   |
| 5.0 | 7           | 455   | -0.032                                  |
| 5.0 | 14          | 440   | -0.066                                  |
| 5.0 | 21          | 435   | -0.076                                  |
| 5.0 | 28          | 442   | -0.061                                  |
| 5.0 | 35          | 461   | -0.019                                  |
| 5.0 | 42          | 457   | -0.027                                  |



| pH  | Time (Days) | Conc. | ln([P] <sub>t</sub> /[P] <sub>o</sub> ) |
|-----|-------------|-------|-----------------------------------------|
| 7.0 | 0           | 481   | 0.000                                   |
| 7.0 | 7           | 474   | -0.015                                  |
| 7.0 | 14          | 465   | -0.034                                  |
| 7.0 | 21          | 484   | 0.007                                   |
| 7.0 | 28          | 456   | -0.055                                  |
| 7.0 | 35          | 488   | 0.014                                   |
| 7.0 | 42          | 479   | -0.004                                  |



## SUMMARY OUTPUT

| Regression Statistics |          |
|-----------------------|----------|
| Multiple R            | 0.14235  |
| R Square              | 0.02026  |
| Adjusted R Square     | -0.17568 |
| Standard Error        | 0.03036  |
| Observations          | 7        |

## ANOVA

|            | df | SS      |
|------------|----|---------|
| Regression | 1  | 0.00010 |
| Residual   | 5  | 0.00461 |
| Total      | 6  | 0.00470 |

|              | Coefficients | Standard Error |
|--------------|--------------|----------------|
| Intercept    | -0.03470     | 0.02069        |
| X Variable 1 | -0.00026     | 0.00082        |

% 2σ Slope Uncertainty  
622%

## SUMMARY OUTPUT

| Regression Statistics |          |
|-----------------------|----------|
| Multiple R            | 0.07787  |
| R Square              | 0.00606  |
| Adjusted R Square     | -0.19272 |
| Standard Error        | 0.02653  |
| Observations          | 7        |

## ANOVA

|            | df | SS      |
|------------|----|---------|
| Regression | 1  | 0.00002 |
| Residual   | 5  | 0.00352 |
| Total      | 6  | 0.00354 |

|              | Coefficients | Standard Error |
|--------------|--------------|----------------|
| Intercept    | -0.01505     | 0.01807        |
| X Variable 1 | 0.00013      | 0.00072        |

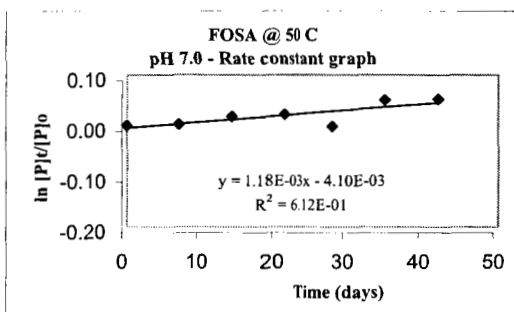
% 2σ Slope Uncertainty  
1145%



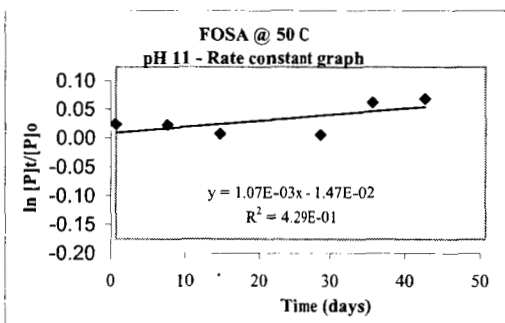
## FOSA Buffer Hydrolysis Study at 50° C.

All concentrations in ng/ml.

| pH  | Time (Days) | Conc. | $\ln([P]_t/[P]_0)$ |
|-----|-------------|-------|--------------------|
| 9.0 | 0           | 429   | 0.000              |
| 9.0 | 7           | 430   | 0.003              |
| 9.0 | 14          | 437   | 0.018              |
| 9.0 | 21          | 438   | 0.022              |
| 9.0 | 28          | 428   | -0.002             |
| 9.0 | 35          | 451   | 0.050              |
| 9.0 | 42          | 452   | 0.052              |



| pH          | Time (Days) | Conc.      | $\ln([P]_t/[P]_0)$ |
|-------------|-------------|------------|--------------------|
| <i>11.0</i> | <i>21</i>   | <i>445</i> |                    |
| 11.0        | 0           | 446        | 0.000              |
| 11.0        | 7           | 445        | -0.001             |
| 11.0        | 14          | 439        | -0.017             |
| 11.0        | 28          | 438        | -0.018             |
| 11.0        | 35          | 463        | 0.038              |
| 11.0        | 42          | 466        | 0.044              |

Data in *italics* excluded on the basis of data quality objectives; see text.

## SUMMARY OUTPUT

| Regression Statistics |         |
|-----------------------|---------|
| Multiple R            | 0.78210 |
| R Square              | 0.61167 |
| Adjusted R Square     | 0.53401 |
| Standard Error        | 0.01555 |
| Observations          | 7       |

## ANOVA

|            | df | SS      |
|------------|----|---------|
| Regression | 1  | 0.00190 |
| Residual   | 5  | 0.00121 |
| Total      | 6  | 0.00311 |

|              | Coefficients | Standard Error |
|--------------|--------------|----------------|
| Intercept    | -0.00410     | 0.01060        |
| X Variable 1 | 0.00118      | 0.00042        |

% 2σ Slope Uncertainty

71%

## SUMMARY OUTPUT

| Regression Statistics |         |
|-----------------------|---------|
| Multiple R            | 0.65519 |
| R Square              | 0.42927 |
| Adjusted R Square     | 0.28659 |
| Standard Error        | 0.02276 |
| Observations          | 6       |

## ANOVA

|            | df | SS      |
|------------|----|---------|
| Regression | 1  | 0.00156 |
| Residual   | 4  | 0.00207 |
| Total      | 5  | 0.00363 |

|              | Coefficients | Standard Error |
|--------------|--------------|----------------|
| Intercept    | -0.01467     | 0.01590        |
| X Variable 1 | 0.00107      | 0.00061        |

% 2σ Slope Uncertainty

115%

FOSA (Perfluorooctanesulfonamide) Hydrolysis Study  
50C; pH 1.5  
All concentrations in ng/ml. All areas x 10<sup>-3</sup>

| All concentrations in ng/mL. All areas x 10 |            |            | FOSA                                                                                                                                                                             |      |        |                   |                  | PFOS                                                                                                                                                      |      |        |                   |                  | FBSA                                            |      |  |
|---------------------------------------------|------------|------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|-------------------------------------------------|------|--|
| Sample I.D.                                 | Data File  | Time Point | Ret Time                                                                                                                                                                         | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                                                                                                                                                  | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                                        | Area |  |
| MeOH Blank                                  | PFOSA003.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                                             | 0    |  |
| MeOH Blank                                  | PFOSA001.D |            |                                                                                                                                                                                  |      |        |                   |                  | 3.7                                                                                                                                                       | 25   | 15     |                   |                  |                                                 |      |  |
| 99039-135-02                                | PFOSA005.D |            | 4.5                                                                                                                                                                              | 200  | 53     | 106%              |                  | 3.7                                                                                                                                                       | 119  | 54     | 104%              |                  | 3.8                                             | 1008 |  |
| 99039-135-03                                | PFOSA006.D |            | 4.5                                                                                                                                                                              | 412  | 101    | 102%              |                  | 3.7                                                                                                                                                       | 238  | 104    | 100%              |                  | 3.8                                             | 1012 |  |
| 99039-135-04                                | PFOSA007.D |            | 4.5                                                                                                                                                                              | 1060 | 252    | 101%              |                  | 3.7                                                                                                                                                       | 611  | 264    | 102%              |                  | 3.8                                             | 1022 |  |
| 99039-135-05                                | PFOSA008.D |            | 4.5                                                                                                                                                                              | 2091 | 513    | 103%              |                  | 3.7                                                                                                                                                       | 1173 | 531    | 102%              |                  | 3.8                                             | 1012 |  |
| 99039-135-06                                | PFOSA009.D |            | 4.5                                                                                                                                                                              | 3951 | 1018   | 102%              |                  | 3.7                                                                                                                                                       | 2154 | 1050   | 101%              |                  | 3.8                                             | 1029 |  |
| MeOH Blank                                  | PFOSA014.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  |                                                 |      |  |
| MeOH Blank                                  | PFOSA015.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  |                                                 |      |  |
| 80399PFOSA-001                              | PFOSA016.D | Day 0      | 4.5                                                                                                                                                                              | 1958 | 474    | 1%                | 86%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 98%              | 3.8                                             | 1021 |  |
| 80399PFOSA-002                              | PFOSA017.D | Day 0      | 4.5                                                                                                                                                                              | 2005 | 481    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1031 |  |
| 80399PFOSA-003                              | PFOSA018.D | Day 0      | 4.5                                                                                                                                                                              | 1949 | 469    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1027 |  |
| 80399PFOSA-004                              | PFOSA019.D | Day 0      | 4.5                                                                                                                                                                              | 2789 | 689    |                   |                  | 3.7                                                                                                                                                       | 593  | 255    |                   |                  | 3.8                                             | 1026 |  |
| 80399PFOSA-025                              | PFOSA020.D | Day 7      | 4.5                                                                                                                                                                              | 1915 | 459    | 1%                | 96%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 96%              | 3.8                                             | 1029 |  |
| 80399PFOSA-026                              | PFOSA021.D | Day 7      | 4.5                                                                                                                                                                              | 1940 | 465    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1032 |  |
| 80399PFOSA-027                              | PFOSA022.D | Day 7      | 4.5                                                                                                                                                                              | 1945 | 467    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1030 |  |
| 80399PFOSA-028                              | PFOSA023.D | Day 7      | 4.5                                                                                                                                                                              | 2911 | 703    |                   |                  | 3.7                                                                                                                                                       | 596  | 250    |                   |                  | 3.8                                             | 1051 |  |
| 80399PFOSA-049                              | PFOSA024.D | Day 14     | 4.5                                                                                                                                                                              | 1885 | 447    | 1%                | 97%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 95%              | 3.8                                             | 1039 |  |
| 80399PFOSA-050                              | PFOSA025.D | Day 14     | 4.5                                                                                                                                                                              | 1881 | 438    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1058 |  |
| 80399PFOSA-051                              | PFOSA026.D | Day 14     | 4.5                                                                                                                                                                              | 1957 | 443    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1088 |  |
| 80399PFOSA-052                              | PFOSA027.D | Day 14     | 4.5                                                                                                                                                                              | 2850 | 684    |                   |                  | 3.7                                                                                                                                                       | 590  | 246    |                   |                  | 3.8                                             | 1056 |  |
| 80399PFOSA-073                              | PFOSA028.D | Day 21     | 4.5                                                                                                                                                                              | 1864 | 439    | 3%                | 81%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 97%              | 3.8                                             | 1044 |  |
| 80399PFOSA-074                              | PFOSA029.D | Day 21     | 4.5                                                                                                                                                                              | 1931 | 447    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1065 |  |
| 80399PFOSA-075                              | PFOSA030.D | Day 21     | 4.5                                                                                                                                                                              | 1976 | 463    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1055 |  |
| 80399PFOSA-076                              | PFOSA031.D | Day 21     | 4.5                                                                                                                                                                              | 2810 | 652    |                   |                  | 3.7                                                                                                                                                       | 597  | 242    |                   |                  | 3.8                                             | 1088 |  |
| MeOH Blank                                  | PFOSA035.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  |                                                 |      |  |
| MeOH Blank                                  | PFOSA036.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  |                                                 |      |  |
| 80399PFOSA-097                              | PFOSA037.D | Day 28     | 4.5                                                                                                                                                                              | 1850 | 437    | 1%                | 104%             | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 95%              | 3.8                                             | 1043 |  |
| 80399PFOSA-098                              | PFOSA038.D | Day 28     | 4.5                                                                                                                                                                              | 1840 | 441    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1028 |  |
| 80399PFOSA-099                              | PFOSA039.D | Day 28     | 4.5                                                                                                                                                                              | 1914 | 444    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1063 |  |
| 80399PFOSA-100                              | PFOSA040.D | Day 28     | 4.5                                                                                                                                                                              | 2905 | 699    |                   |                  | 3.7                                                                                                                                                       | 589  | 246    |                   |                  | 3.8                                             | 1054 |  |
| 80399PFOSA-121                              | PFOSA041.D | Day 35     | 4.5                                                                                                                                                                              | 1898 | 448    | 2%                | 77%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 96%              | 3.8                                             | 1044 |  |
| 80399PFOSA-122                              | PFOSA042.D | Day 35     | 4.5                                                                                                                                                                              | 1896 | 455    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1027 |  |
| 80399PFOSA-123                              | PFOSA043.D | Day 35     | 4.5                                                                                                                                                                              | 1974 | 470    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1038 |  |
| 80399PFOSA-124                              | PFOSA044.D | Day 35     | 4.5                                                                                                                                                                              | 2682 | 651    |                   |                  | 3.7                                                                                                                                                       | 587  | 249    |                   |                  | 3.8                                             | 1039 |  |
| 80399PFOSA-145                              | PFOSA045.D | Day 42     | 4.5                                                                                                                                                                              | 1927 | 451    | 2%                | 103%             | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 96%              | 3.8                                             | 1055 |  |
| 80399PFOSA-146                              | PFOSA046.D | Day 42     | 4.5                                                                                                                                                                              | 1871 | 434    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1060 |  |
| 80399PFOSA-147                              | PFOSA047.D | Day 42     | 4.5                                                                                                                                                                              | 1854 | 439    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1040 |  |
| 80399PFOSA-148                              | PFOSA048.D | Day 42     | 4.5                                                                                                                                                                              | 2898 | 700    |                   |                  | 3.7                                                                                                                                                       | 592  | 248    |                   |                  | 3.8                                             | 1051 |  |
| MeOH Blank                                  | PFOSA049.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  |                                                 |      |  |
| MeOH Blank                                  | PFOSA050.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  |                                                 |      |  |
| 99039-135-01                                | PFOSA051.D |            |                                                                                                                                                                                  |      |        |                   |                  | 3.7                                                                                                                                                       | 28   | 16     |                   |                  |                                                 |      |  |
| 99039-135-02                                | PFOSA052.D |            | 4.5                                                                                                                                                                              | 195  | 50     | 101%              |                  | 3.7                                                                                                                                                       | 115  | 51     | 98%               |                  | 3.8                                             | 1038 |  |
| 99039-135-03                                | PFOSA053.D |            | 4.5                                                                                                                                                                              | 405  | 96     | 96%               |                  | 3.7                                                                                                                                                       | 240  | 101    | 97%               |                  | 3.8                                             | 1053 |  |
| 99039-135-04                                | PFOSA054.D |            | 4.5                                                                                                                                                                              | 1037 | 243    | 97%               |                  | 3.7                                                                                                                                                       | 604  | 257    | 99%               |                  | 3.8                                             | 1036 |  |
| 99039-135-05                                | PFOSA055.D |            | 4.5                                                                                                                                                                              | 2067 | 489    | 98%               |                  | 3.7                                                                                                                                                       | 1160 | 506    | 98%               |                  | 3.8                                             | 1046 |  |
| 99039-135-06                                | PFOSA056.D |            | 4.5                                                                                                                                                                              | 3935 | 979    | 98%               |                  | 3.7                                                                                                                                                       | 2177 | 1026   | 99%               |                  | 3.8                                             | 1059 |  |
| Method ID: 0309_15A.M                       |            |            | Internal Standard quant: r <sup>2</sup> =0.999<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 49.9 998<br>Original conc.= 499.4<br>Spike conc.= 249.7 |      |        |                   |                  | Internal Standard quant: r <sup>2</sup> =0.999<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 51.9 1038<br>Spike conc.= 259.40 |      |        |                   |                  | Conc. 225<br>mean 1042<br>S.D. 18<br>%S.D. 1.8% |      |  |

(A) The relative standard deviation (RSD) is not defined when the mean concentration is zero.

FOSA (Perfluorooctanesulfonamide) Hydrolysis Study  
50C; pH 3.0All concentrations in ng/ml. All areas x 10<sup>-3</sup>

| FOSA           |            |            |                                                                                                                                                                                  |         |        |                   | PFOS             |                                                                                                                                                           |      |        |                   | FBSA             |                                |                             |
|----------------|------------|------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------|--------|-------------------|------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|--------------------------------|-----------------------------|
| Sample ID      | Data File  | Time Point | Ret Time                                                                                                                                                                         | Area    | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                                                                                                                                                  | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                       | Area                        |
| MeOH Blank     | PFOSA058.D |            | 4.5                                                                                                                                                                              |         | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                            | 0                           |
| 99039-136-01   | PFOSA059.D |            |                                                                                                                                                                                  |         |        |                   |                  | 3.7                                                                                                                                                       | 25   | 11     |                   |                  |                                | 0                           |
| 99039-136-02   | PFOSA060.D |            | 4.5                                                                                                                                                                              | 195     | 48     | 95%               |                  | 3.7                                                                                                                                                       | 117  | 51     | 98%               |                  | 3.8                            | 1041                        |
| 99039-136-03   | PFOSA061.D |            | 4.5                                                                                                                                                                              | 403     | 99     | 99%               |                  | 3.7                                                                                                                                                       | 232  | 101    | 98%               |                  | 3.8                            | 1041                        |
| 99039-136-04   | PFOSA062.D |            | 4.5                                                                                                                                                                              | 1059    | 262    | 105%              |                  | 3.7                                                                                                                                                       | 614  | 273    | 105%              |                  | 3.8                            | 1044                        |
| 99039-136-05   | PFOSA063.D |            | 4.5                                                                                                                                                                              | 2041    | 516    | 103%              |                  | 3.7                                                                                                                                                       | 1171 | 540    | 104%              |                  | 3.8                            | 1045                        |
| 99039-136-06   | PFOSA064.D |            | 4.5                                                                                                                                                                              | 3909    | 1022   | 102%              |                  | 3.7                                                                                                                                                       | 2160 | 1064   | 103%              |                  | 3.8                            | 1054                        |
| MeOH Blank     | PFOSA069.D |            | 4.5                                                                                                                                                                              | 0       | < 12.5 |                   |                  | 3.7                                                                                                                                                       | 0    | < 13.0 |                   |                  | 3.8                            | 0                           |
| MeOH Blank     | PFOSA070.D |            | 4.5                                                                                                                                                                              | 0       | < 12.5 |                   |                  | 3.7                                                                                                                                                       | 0    | < 13.0 |                   |                  | 3.8                            | 0                           |
| 80399PFOSA-005 | PFOSA071.D | Day 0      | 4.5                                                                                                                                                                              | 1972    | 484    | 1%                | 100%             | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 99%              | 3.8                            | 1073                        |
| 80399PFOSA-006 | PFOSA072.D | Day 0      | 4.5                                                                                                                                                                              | 1971    | 481    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1080                        |
| 80399PFOSA-007 | PFOSA073.D | Day 0      | 4.5                                                                                                                                                                              | 1984    | 486    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1076                        |
| 80399PFOSA-008 | PFOSA074.D | Day 0      | 4.5                                                                                                                                                                              | 2931    | 734    |                   |                  | 3.7                                                                                                                                                       | 593  | 256    |                   |                  | 3.8                            | 1075                        |
| 80399PFOSA-029 | PFOSA075.D | Day 7      | 4.5                                                                                                                                                                              | 1919    | 484    | 1%                | 100%             | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 98%              | 3.8                            | 1045                        |
| 80399PFOSA-030 | PFOSA076.D | Day 7      | 4.5                                                                                                                                                                              | 1917    | 476    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1060                        |
| 80399PFOSA-031 | PFOSA077.D | Day 7      | 4.5                                                                                                                                                                              | 1981    | 482    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1082                        |
| 80399PFOSA-032 | PFOSA078.D | Day 7      | 4.5                                                                                                                                                                              | 2953    | 732    |                   |                  | 3.7                                                                                                                                                       | 593  | 253    |                   |                  | 3.8                            | 1086                        |
| 80399PFOSA-053 | PFOSA079.D | Day 14     | 4.5                                                                                                                                                                              | 1908    | 448    | 2%                | 106%             | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 98%              | 3.8                            | 1118                        |
| 80399PFOSA-054 | PFOSA080.D | Day 14     | 4.5                                                                                                                                                                              | 1952    | 468    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1098                        |
| 80399PFOSA-055 | PFOSA081.D | Day 14     | 4.5                                                                                                                                                                              | 1890    | 455    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1092                        |
| 80399PFOSA-056 | PFOSA082.D | Day 14     | 4.5                                                                                                                                                                              | 3050    | 721    |                   |                  | 3.7                                                                                                                                                       | 621  | 253    |                   |                  | 3.8                            | 1137                        |
| 80399PFOSA-077 | PFOSA083.D | Day 21     | 4.5                                                                                                                                                                              | 3565    | 421    | 3%                | 105%             | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 97%              | 3.8                            | 2222                        |
| 80399PFOSA-078 | PFOSA084.D | Day 21     | 4.5                                                                                                                                                                              | 1880    | 446    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1108                        |
| 80399PFOSA-079 | PFOSA085.D | Day 21     | 4.5                                                                                                                                                                              | 1805    | 419    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1128                        |
| 80399PFOSA-080 | PFOSA086.D | Day 21     | 4.5                                                                                                                                                                              | 2874    | 690    |                   |                  | 3.7                                                                                                                                                       | 585  | 242    |                   |                  | 3.8                            | 1116                        |
| MeOH Blank     | PFOSA090.D |            | 4.5                                                                                                                                                                              |         | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                            | 0                           |
| MeOH Blank     | PFOSA091.D |            | 4.5                                                                                                                                                                              |         | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                            | 0                           |
| 80399PFOSA-101 | PFOSA092.D | Day 28     | 4.5                                                                                                                                                                              | 1871    | 458    | 0%                | 98%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 96%              | 3.8                            | 1074                        |
| 80399PFOSA-102 | PFOSA093.D | Day 28     | 4.5                                                                                                                                                                              | 1857    | 455    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1074                        |
| 80399PFOSA-103 | PFOSA094.D | Day 28     | 4.5                                                                                                                                                                              | 1883    | 458    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1081                        |
| 80399PFOSA-104 | PFOSA095.D | Day 28     | 4.5                                                                                                                                                                              | 2873    | 700    |                   |                  | 3.7                                                                                                                                                       | 589  | 248    |                   |                  | 3.8                            | 1101                        |
| 80399PFOSA-125 | PFOSA096.D | Day 35     | 4.5                                                                                                                                                                              | 1933    | 467    | 1%                | 105%             | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 98%              | 3.8                            | 1089                        |
| 80399PFOSA-126 | PFOSA097.D | Day 35     | 4.5                                                                                                                                                                              | 1953    | 471    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1091                        |
| 80399PFOSA-127 | PFOSA098.D | Day 35     | 4.5                                                                                                                                                                              | 1898    | 464    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1077                        |
| 80399PFOSA-128 | PFOSA099.D | Day 35     | 4.5                                                                                                                                                                              | 2962    | 730    |                   |                  | 3.7                                                                                                                                                       | 601  | 255    |                   |                  | 3.8                            | 1091                        |
| 80399PFOSA-149 | PFOSA100.D | Day 42     | 4.5                                                                                                                                                                              | 1958    | 471    | 3%                | 92%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 95%              | 3.8                            | 1095                        |
| 80399PFOSA-150 | PFOSA101.D | Day 42     | 4.5                                                                                                                                                                              | 1984    | 479    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1091                        |
| 80399PFOSA-151 | PFOSA102.D | Day 42     | 4.5                                                                                                                                                                              | 1867    | 448    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                            | 1094                        |
| 80399PFOSA-152 | PFOSA103.D | Day 42     | 4.5                                                                                                                                                                              | 2911900 | 695    |                   |                  | 3.7                                                                                                                                                       | 598  | 246    |                   |                  | 3.8                            | 1123                        |
| MeOH Blank     | PFOSA104.D |            | 4.5                                                                                                                                                                              |         | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                            | 0                           |
| MeOH Blank     | PFOSA105.D |            | 4.5                                                                                                                                                                              |         | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                            | 0                           |
| 99039-136-01   | PFOSA106.D |            |                                                                                                                                                                                  |         |        |                   |                  | 0.0                                                                                                                                                       | 27   | 12     |                   |                  |                                |                             |
| 99039-136-02   | PFOSA107.D |            | 4.5                                                                                                                                                                              | 201     | 48     | 96%               |                  | 3.7                                                                                                                                                       | 115  | 49     | 94%               |                  | 3.8                            | 1067                        |
| 99039-136-03   | PFOSA108.D |            | 4.5                                                                                                                                                                              | 411     | 98     | 98%               |                  | 3.7                                                                                                                                                       | 239  | 101    | 98%               |                  | 3.8                            | 1072                        |
| 99039-136-04   | PFOSA109.D |            | 4.5                                                                                                                                                                              | 1056    | 254    | 102%              |                  | 3.7                                                                                                                                                       | 615  | 265    | 102%              |                  | 3.8                            | 1076                        |
| 99039-136-05   | PFOSA110.D |            | 4.5                                                                                                                                                                              | 2108    | 472    | 94%               |                  | 3.7                                                                                                                                                       | 1191 | 484    | 93%               |                  | 3.8                            | 1176                        |
| 99039-136-06   | PFOSA111.D |            | 4.5                                                                                                                                                                              | 3986    | 977    | 98%               |                  | 3.7                                                                                                                                                       | 2205 | 1014   | 98%               |                  | 3.8                            | 1120                        |
| Method ID:     | 0309_15A.M |            | Internal Standard quant: r <sup>2</sup> =0.999<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 49.9 998<br>Original conc.= 499.4<br>Spike conc.= 249.7 |         |        |                   |                  | Internal Standard quant: r <sup>2</sup> =0.999<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 51.9 1038<br>Spike conc.= 259.40 |      |        |                   |                  | Conc.<br>mean<br>S.D.<br>%S.D. | 225<br>1116<br>186<br>16.7% |

(A) The relative percent deviation (RPD) is not defined when the mean concentration is zero.

FOSA (Perfluorooctanesulfonamide) Hydrolysis Study  
 50C; pH 5.0  
 All concentrations in ng/ml. All areas x 10<sup>-3</sup>

|                |            |            | FOSA                                                                                                                                                                             |      |        |                   |                  | PFOS                                                                                                                                                      |      |        |                   |                  | FBSA                                            |      |  |
|----------------|------------|------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|-------------------------------------------------|------|--|
| Sample I.D.    | Data File  | Time Point | Ret Time                                                                                                                                                                         | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                                                                                                                                                  | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                                        | Area |  |
| MeOH Blank     | PFOSA003.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                                             | 0    |  |
| 99039-137-01   | PFOSA004.D |            |                                                                                                                                                                                  |      |        |                   |                  | 3.7                                                                                                                                                       | 26   | 15     |                   |                  |                                                 |      |  |
| 99039-137-02   | PFOSA005.D |            | 4.5                                                                                                                                                                              | 194  | 51     | 102%              |                  | 3.7                                                                                                                                                       | 118  | 54     | 104%              |                  | 3.8                                             | 1054 |  |
| 99039-137-03   | PFOSA006.D |            | 4.5                                                                                                                                                                              | 419  | 101    | 101%              |                  | 3.7                                                                                                                                                       | 236  | 103    | 99%               |                  | 3.8                                             | 1075 |  |
| 99039-137-04   | PFOSA007.D |            | 4.5                                                                                                                                                                              | 1026 | 246    | 98%               |                  | 3.7                                                                                                                                                       | 579  | 254    | 98%               |                  | 3.8                                             | 1062 |  |
| 99039-137-05   | PFOSA008.D |            | 4.5                                                                                                                                                                              | 2059 | 495    | 99%               |                  | 3.7                                                                                                                                                       | 1171 | 523    | 101%              |                  | 3.8                                             | 1070 |  |
| 99039-137-06   | PFOSA009.D |            | 4.5                                                                                                                                                                              | 4043 | 986    | 99%               |                  | 3.7                                                                                                                                                       | 2218 | 1028   | 99%               |                  | 3.8                                             | 1099 |  |
| MeOH Blank     | PFOSA014.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       | 0    | < 13.0 |                   |                  | 3.8                                             | 0    |  |
| MeOH Blank     | PFOSA015.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       | 0    | < 13.0 |                   |                  | 3.8                                             | 0    |  |
| 80399PFOSA-009 | PFOSA016.D | Day 0      | 4.5                                                                                                                                                                              | 2010 | 472    | 0%                | 94%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 96%              | 3.8                                             | 1092 |  |
| 80399PFOSA-010 | PFOSA017.D | Day 0      | 4.5                                                                                                                                                                              | 2039 | 470    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1113 |  |
| 80399PFOSA-011 | PFOSA018.D | Day 0      | 4.5                                                                                                                                                                              | 2003 | 468    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1098 |  |
| 80399PFOSA-012 | PFOSA019.D | Day 0      | 4.5                                                                                                                                                                              | 3010 | 705    |                   |                  | 3.7                                                                                                                                                       | 596  | 249    |                   |                  | 3.8                                             | 1115 |  |
| 80399PFOSA-033 | PFOSA020.D | Day 7      | 4.5                                                                                                                                                                              | 1934 | 446    | 2%                | 96%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 97%              | 3.8                                             | 1110 |  |
| 80399PFOSA-034 | PFOSA021.D | Day 7      | 4.5                                                                                                                                                                              | 1956 | 461    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1087 |  |
| 80399PFOSA-035 | PFOSA022.D | Day 7      | 4.5                                                                                                                                                                              | 1935 | 458    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1082 |  |
| 80399PFOSA-036 | PFOSA023.D | Day 7      | 4.5                                                                                                                                                                              | 3030 | 716    |                   |                  | 3.7                                                                                                                                                       | 598  | 252    |                   |                  | 3.8                                             | 1106 |  |
| 80399PFOSA-057 | PFOSA024.D | Day 14     | 4.5                                                                                                                                                                              | 2285 | 436    | 1%                | 97%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 98%              | 3.8                                             | 1341 |  |
| 80399PFOSA-058 | PFOSA025.D | Day 14     | 4.5                                                                                                                                                                              | 1964 | 441    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1141 |  |
| 80399PFOSA-059 | PFOSA026.D | Day 14     | 4.5                                                                                                                                                                              | 1942 | 443    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1124 |  |
| 80399PFOSA-060 | PFOSA027.D | Day 14     | 4.5                                                                                                                                                                              | 3055 | 684    |                   |                  | 3.7                                                                                                                                                       | 634  | 253    |                   |                  | 3.8                                             | 1165 |  |
| 80399PFOSA-081 | PFOSA028.D | Day 21     | 4.5                                                                                                                                                                              | 1966 | 443    | 5%                | 81%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 102%             | 3.8                                             | 1137 |  |
| 80399PFOSA-082 | PFOSA029.D | Day 21     | 4.5                                                                                                                                                                              | 1980 | 450    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1127 |  |
| 80399PFOSA-083 | PFOSA030.D | Day 21     | 4.5                                                                                                                                                                              | 1853 | 413    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1147 |  |
| 80399PFOSA-084 | PFOSA031.D | Day 21     | 4.5                                                                                                                                                                              | 2945 | 703    |                   |                  | 3.7                                                                                                                                                       | 598  | 254    |                   |                  | 3.8                                             | 1094 |  |
| MeOH Blank     | PFOSA035.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                                             | 0    |  |
| MeOH Blank     | PFOSA036.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                                             | 0    |  |
| 80399PFOSA-105 | PFOSA037.D | Day 28     | 4.5                                                                                                                                                                              | 1873 | 444    | 1%                | 104%             | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 95%              | 3.8                                             | 1081 |  |
| 80399PFOSA-106 | PFOSA038.D | Day 28     | 4.5                                                                                                                                                                              | 1888 | 446    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1086 |  |
| 80399PFOSA-107 | PFOSA039.D | Day 28     | 4.5                                                                                                                                                                              | 1869 | 437    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1095 |  |
| 80399PFOSA-108 | PFOSA040.D | Day 28     | 4.5                                                                                                                                                                              | 2863 | 692    |                   |                  | 3.7                                                                                                                                                       | 575  | 248    |                   |                  | 3.8                                             | 1081 |  |
| 80399PFOSA-129 | PFOSA041.D | Day 35     | 4.5                                                                                                                                                                              | 1949 | 461    | 2%                | 77%              | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 94%              | 3.8                                             | 1083 |  |
| 80399PFOSA-130 | PFOSA042.D | Day 35     | 4.5                                                                                                                                                                              | 1926 | 453    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1088 |  |
| 80399PFOSA-131 | PFOSA043.D | Day 35     | 4.5                                                                                                                                                                              | 2007 | 468    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1100 |  |
| 80399PFOSA-132 | PFOSA044.D | Day 35     | 4.5                                                                                                                                                                              | 2957 | 705    |                   |                  | 3.7                                                                                                                                                       | 571  | 243    |                   |                  | 3.8                                             | 1095 |  |
| 80399PFOSA-153 | PFOSA045.D | Day 42     | 4.5                                                                                                                                                                              | 1926 | 459    | 1%                | 103%             | 3.7                                                                                                                                                       | 0    | 0      | (A)               | 97%              | 3.8                                             | 1076 |  |
| 80399PFOSA-154 | PFOSA046.D | Day 42     | 4.5                                                                                                                                                                              | 1947 | 461    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1082 |  |
| 80399PFOSA-155 | PFOSA047.D | Day 42     | 4.5                                                                                                                                                                              | 1887 | 452    |                   |                  | 3.7                                                                                                                                                       | 0    | 0      |                   |                  | 3.8                                             | 1070 |  |
| 80399PFOSA-156 | PFOSA048.D | Day 42     | 4.5                                                                                                                                                                              | 2963 | 705    |                   |                  | 3.7                                                                                                                                                       | 592  | 251    |                   |                  | 3.8                                             | 1098 |  |
| MeOH Blank     | PFOSA049.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                                             | 0    |  |
| MeOH Blank     | PFOSA050.D |            | 4.5                                                                                                                                                                              |      | < 12.5 |                   |                  | 3.7                                                                                                                                                       |      | < 13.0 |                   |                  | 3.8                                             | 0    |  |
| 99039-137-01   | PFOSA051.D |            |                                                                                                                                                                                  |      |        |                   |                  | 3.7                                                                                                                                                       | 26   | 15     |                   |                  |                                                 |      |  |
| 99039-137-02   | PFOSA052.D |            | 4.5                                                                                                                                                                              | 197  | 52     | 103%              |                  | 3.7                                                                                                                                                       | 118  | 54     | 104%              |                  | 3.8                                             | 1050 |  |
| 99039-137-03   | PFOSA053.D |            | 4.5                                                                                                                                                                              | 404  | 100    | 100%              |                  | 3.7                                                                                                                                                       | 236  | 105    | 101%              |                  | 3.8                                             | 1054 |  |
| 99039-137-04   | PFOSA054.D |            | 4.5                                                                                                                                                                              | 1015 | 246    | 98%               |                  | 3.7                                                                                                                                                       | 576  | 255    | 98%               |                  | 3.8                                             | 1051 |  |
| 99039-137-05   | PFOSA055.D |            | 4.5                                                                                                                                                                              | 2083 | 510    | 102%              |                  | 3.7                                                                                                                                                       | 1147 | 521    | 100%              |                  | 3.8                                             | 1051 |  |
| 99039-137-06   | PFOSA056.D |            | 4.5                                                                                                                                                                              | 4003 | 1011   | 101%              |                  | 3.7                                                                                                                                                       | 2179 | 1046   | 101%              |                  | 3.8                                             | 1063 |  |
| Method ID:     | 0309_15B.M |            | Internal Standard quant: r <sup>2</sup> =0.999<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 49.9 998<br>Original conc.= 499.4<br>Spike conc.= 249.7 |      |        |                   |                  | Internal Standard quant: r <sup>2</sup> =0.999<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 51.9 1038<br>Spike conc.= 259.40 |      |        |                   |                  | Conc. 225<br>mean 1099<br>S.D. 49<br>%S.D. 4.5% |      |  |

(A) The relative percent deviation (RPD) is not defined when the mean concentration is zero.

FOSA (Perfluorooctanesulfonamide) Hydrolysis Study  
 50C; pH 7.0  
 All concentrations in ng/ml. All areas x 10<sup>3</sup>

|                |            |            | FOSA                                                                                                                                                                   |      |        |                   |                  | PFOS                                                                                                                                            |      |        |                   |                  | FBSA                           |                           |
|----------------|------------|------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|-------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|--------------------------------|---------------------------|
| Sample ID      | Data File  | Time Point | Ret Time                                                                                                                                                               | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                                                                                                                                        | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                       | Area                      |
| MeOH Blank     | PFOSA057.D |            | 4.5                                                                                                                                                                    |      | < 12.5 |                   |                  | 3.7                                                                                                                                             |      | < 13.0 |                   |                  | 3.8                            | 0                         |
| 99039-138-01   | PFOSA059.D |            |                                                                                                                                                                        |      |        |                   |                  | 3.7                                                                                                                                             | 26   | 11     |                   |                  | 3.8                            | 0                         |
| 99039-138-02   | PFOSA060.D |            | 4.5                                                                                                                                                                    | 203  | 48     | 97%               |                  | 3.7                                                                                                                                             | 122  | 52     | 101%              |                  | 3.8                            | 1043                      |
| 99039-138-03   | PFOSA061.D |            | 4.5                                                                                                                                                                    | 412  | 97     | 97%               |                  | 3.7                                                                                                                                             | 238  | 101    | 97%               |                  | 3.8                            | 1060                      |
| 99039-138-04   | PFOSA062.D |            | 4.5                                                                                                                                                                    | 1050 | 258    | 103%              |                  | 3.7                                                                                                                                             | 597  | 264    | 102%              |                  | 3.8                            | 1030                      |
| 99039-138-05   | PFOSA063.D |            | 4.5                                                                                                                                                                    | 2035 | 496    | 99%               |                  | 3.7                                                                                                                                             | 1155 | 516    | 100%              |                  | 3.8                            | 1050                      |
| 99039-138-06   | PFOSA064.D |            | 4.5                                                                                                                                                                    | 3978 | 1003   | 100%              |                  | 3.7                                                                                                                                             | 2162 | 1044   | 101%              |                  | 3.8                            | 1040                      |
| MeOH Blank     | PFOSA069.D |            | 4.5                                                                                                                                                                    |      | < 12.5 |                   |                  | 3.7                                                                                                                                             |      | < 13.0 |                   |                  | 3.8                            | 0                         |
| MeOH Blank     | PFOSA070.D |            | 4.5                                                                                                                                                                    |      | < 12.5 |                   |                  | 3.7                                                                                                                                             |      | < 13.0 |                   |                  | 3.8                            | 0                         |
| 80399PFOSA-013 | PFOSA071.D | Day 0      | 4.5                                                                                                                                                                    | 1974 | 486    | 1%                | 101%             | 3.7                                                                                                                                             | 0    | 0      | (A)               | 97%              | 3.8                            | 1039                      |
| 80399PFOSA-014 | PFOSA072.D | Day 0      | 4.5                                                                                                                                                                    | 1992 | 476    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1070                      |
| 80399PFOSA-015 | PFOSA073.D | Day 0      | 4.5                                                                                                                                                                    | 1977 | 482    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1048                      |
| 80399PFOSA-016 | PFOSA074.D | Day 0      | 4.5                                                                                                                                                                    | 2979 | 733    |                   |                  | 3.7                                                                                                                                             | 583  | 252    |                   |                  | 3.8                            | 1051                      |
| 80399PFOSA-037 | PFOSA075.D | Day 7      | 4.5                                                                                                                                                                    | 1936 | 469    | 1%                | 105%             | 3.7                                                                                                                                             | 0    | 0      | (A)               | 98%              | 3.8                            | 1055                      |
| 80399PFOSA-038 | PFOSA076.D | Day 7      | 4.5                                                                                                                                                                    | 1899 | 474    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1025                      |
| 80399PFOSA-039 | PFOSA077.D | Day 7      | 4.5                                                                                                                                                                    | 1968 | 480    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1048                      |
| 80399PFOSA-040 | PFOSA078.D | Day 7      | 4.5                                                                                                                                                                    | 3004 | 737    |                   |                  | 3.7                                                                                                                                             | 593  | 256    |                   |                  | 3.8                            | 1056                      |
| 80399PFOSA-061 | PFOSA079.D | Day 14     | 4.5                                                                                                                                                                    | 2006 | 457    | 2%                | 98%              | 3.7                                                                                                                                             | 0    | 0      | (A)               | 98%              | 3.8                            | 1121                      |
| 80399PFOSA-062 | PFOSA080.D | Day 14     | 4.5                                                                                                                                                                    | 1921 | 469    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1046                      |
| 80399PFOSA-063 | PFOSA081.D | Day 14     | 4.5                                                                                                                                                                    | 2131 | 470    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1159                      |
| 80399PFOSA-064 | PFOSA082.D | Day 14     | 4.5                                                                                                                                                                    | 2955 | 710    |                   |                  | 3.7                                                                                                                                             | 603  | 255    |                   |                  | 3.8                            | 1076                      |
| 80399PFOSA-085 | PFOSA083.D | Day 21     | 4.5                                                                                                                                                                    | 1943 | 479    | 1%                | 101%             | 3.7                                                                                                                                             | 0    | 0      | (A)               | 101%             | 3.8                            | 1037                      |
| 80399PFOSA-086 | PFOSA084.D | Day 21     | 4.5                                                                                                                                                                    | 2000 | 484    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1056                      |
| 80399PFOSA-087 | PFOSA085.D | Day 21     | 4.5                                                                                                                                                                    | 2109 | 490    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1100                      |
| 80399PFOSA-088 | PFOSA086.D | Day 21     | 4.5                                                                                                                                                                    | 3153 | 738    |                   |                  | 3.7                                                                                                                                             | 613  | 252    |                   |                  | 3.8                            | 1106                      |
| MeOH Blank     | PFOSA090.D |            | 4.5                                                                                                                                                                    |      | < 12.5 |                   |                  | 3.7                                                                                                                                             |      | < 13.0 |                   |                  | 3.8                            | 0                         |
| MeOH Blank     | PFOSA091.D |            | 4.5                                                                                                                                                                    |      | < 12.5 |                   |                  | 3.7                                                                                                                                             |      | < 13.0 |                   |                  | 3.8                            | 0                         |
| 80399PFOSA-110 | PFOSA092.D | Day 28     | 4.5                                                                                                                                                                    | 1813 | 446    | 2%                | 102%             | 3.7                                                                                                                                             | 0    | 0      | (A)               | 96%              | 3.8                            | 1038                      |
| 80399PFOSA-111 | PFOSA093.D | Day 28     | 4.5                                                                                                                                                                    | 1884 | 458    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1050                      |
| 80399PFOSA-112 | PFOSA094.D | Day 28     | 4.5                                                                                                                                                                    | 1878 | 463    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1037                      |
| 80399PFOSA-113 | PFOSA095.D | Day 28     | 4.5                                                                                                                                                                    | 2876 | 711    |                   |                  | 3.7                                                                                                                                             | 574  | 250    |                   |                  | 3.8                            | 1045                      |
| 80399PFOSA-133 | PFOSA096.D | Day 35     | 4.5                                                                                                                                                                    | 1935 | 474    | 3%                | 93%              | 3.7                                                                                                                                             | 0    | 0      | (A)               | 98%              | 3.8                            | 1044                      |
| 80399PFOSA-134 | PFOSA097.D | Day 35     | 4.5                                                                                                                                                                    | 2089 | 497    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1075                      |
| 80399PFOSA-135 | PFOSA098.D | Day 35     | 4.5                                                                                                                                                                    | 2009 | 493    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1043                      |
| 80399PFOSA-136 | PFOSA099.D | Day 35     | 4.5                                                                                                                                                                    | 2918 | 721    |                   |                  | 3.7                                                                                                                                             | 583  | 253    |                   |                  | 3.8                            | 1047                      |
| 80399PFOSA-157 | PFOSA100.D | Day 42     | 4.5                                                                                                                                                                    | 1993 | 487    | 2%                | 98%              | 3.7                                                                                                                                             | 0    | 0      | (A)               | 96%              | 3.8                            | 1047                      |
| 80399PFOSA-158 | PFOSA101.D | Day 42     | 4.5                                                                                                                                                                    | 1939 | 480    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1032                      |
| 80399PFOSA-159 | PFOSA102.D | Day 42     | 4.5                                                                                                                                                                    | 1932 | 471    |                   |                  | 3.7                                                                                                                                             | 0    | 0      |                   |                  | 3.8                            | 1049                      |
| 80399PFOSA-160 | PFOSA103.D | Day 42     | 4.5                                                                                                                                                                    | 2980 | 723    |                   |                  | 3.7                                                                                                                                             | 585  | 250    |                   |                  | 3.8                            | 1066                      |
| MeOH Blank     | PFOSA104.D |            | 4.5                                                                                                                                                                    |      | < 12.5 |                   |                  | 3.7                                                                                                                                             |      | < 13.0 |                   |                  | 3.8                            | 0                         |
| MeOH Blank     | PFOSA105.D |            | 4.5                                                                                                                                                                    |      | < 12.5 |                   |                  | 3.7                                                                                                                                             |      | < 13.0 |                   |                  | 3.8                            | 0                         |
| 99039-138-01   | PFOSA106.D |            |                                                                                                                                                                        |      |        |                   |                  | 3.7                                                                                                                                             | 26   | 12     |                   |                  |                                |                           |
| 99039-138-02   | PFOSA107.D |            | 4.5                                                                                                                                                                    | 200  | 49     | 97%               |                  | 3.7                                                                                                                                             | 116  | 51     | 98%               |                  | 3.8                            | 1023                      |
| 99039-138-03   | PFOSA108.D |            | 4.5                                                                                                                                                                    | 413  | 101    | 101%              |                  | 3.7                                                                                                                                             | 237  | 103    | 100%              |                  | 3.8                            | 1026                      |
| 99039-138-04   | PFOSA109.D |            | 4.5                                                                                                                                                                    | 1023 | 253    | 101%              |                  | 3.7                                                                                                                                             | 589  | 262    | 101%              |                  | 3.8                            | 1023                      |
| 99039-138-05   | PFOSA110.D |            | 4.5                                                                                                                                                                    | 2017 | 496    | 99%               |                  | 3.7                                                                                                                                             | 1144 | 517    | 100%              |                  | 3.8                            | 1040                      |
| 99039-138-06   | PFOSA111.D |            | 4.5                                                                                                                                                                    | 3998 | 996    | 100%              |                  | 3.7                                                                                                                                             | 2166 | 1032   | 99%               |                  | 3.8                            | 1052                      |
| Method ID:     | 0309_15B.M |            | Internal Standard quant: $r^2=0.999$<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 49.9 998<br>Original conc.= 499.4<br>Spike conc.= 249.7 |      |        |                   |                  | Internal Standard quant: $r^2=0.999$<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 51.9 1038<br>Spike conc.= 259.40 |      |        |                   |                  | Conc.<br>mean<br>S.D.<br>%S.D. | 225<br>1054<br>28<br>2.6% |

(A) The relative percent deviation (RPD) is not defined when the mean concentration is zero.

FOSA (Perfluorooctanesulfonamide) Hydrolysis Study  
 50°C; pH 9.0  
 All concentrations in ng/ml. All areas x 10<sup>-3</sup>

| All concentrations in ng/ml. All areas x 10 |            |            | FOSA                                                                                                                                                                     |      |        |                   |                  | PFOS                                                                                                                                             |      |        |                   |                  | FBSA                                    |      |
|---------------------------------------------|------------|------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|--------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|-----------------------------------------|------|
| Sample I.D.                                 | Data File  | Time Point | Ret Time                                                                                                                                                                 | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                                                                                                                                         | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                                | Area |
| MeOH Blank                                  | PFOSA003.D |            | 4.5                                                                                                                                                                      |      | < 12.5 |                   |                  | 3.7                                                                                                                                              |      | < 13.0 |                   |                  | 3.8                                     | 0    |
| 99039-139-01                                | PFOSA004.D |            |                                                                                                                                                                          |      |        |                   |                  | 3.7                                                                                                                                              | 26   | 22     |                   |                  |                                         |      |
| 99039-139-02                                | PFOSA005.D |            | 4.5                                                                                                                                                                      | 196  | 56     | 111%              |                  | 3.7                                                                                                                                              | 114  | 55     | 106%              |                  | 3.8                                     | 997  |
| 99039-139-03                                | PFOSA006.D |            | 4.5                                                                                                                                                                      | 420  | 98     | 98%               |                  | 3.7                                                                                                                                              | 248  | 102    | 99%               |                  | 3.8                                     | 1029 |
| 99039-139-04                                | PFOSA007.D |            | 4.5                                                                                                                                                                      | 1056 | 229    | 92%               |                  | 3.7                                                                                                                                              | 612  | 244    | 94%               |                  | 3.8                                     | 1016 |
| 99039-139-05                                | PFOSA008.D |            | 4.5                                                                                                                                                                      | 2258 | 498    | 100%              |                  | 3.7                                                                                                                                              | 1232 | 505    | 97%               |                  | 3.8                                     | 1022 |
| 99039-139-06                                | PFOSA009.D |            | 4.5                                                                                                                                                                      | 3943 | 953    | 95%               |                  | 3.7                                                                                                                                              | 2172 | 985    | 95%               |                  | 3.8                                     | 1038 |
| MeOH Blank                                  | PFOSA014.D |            | 4.5                                                                                                                                                                      |      | < 12.5 |                   |                  | 3.7                                                                                                                                              |      | < 13.0 |                   |                  | 3.8                                     | 0    |
| MeOH Blank                                  | PFOSA015.D |            | 4.5                                                                                                                                                                      |      | < 12.5 |                   |                  | 3.7                                                                                                                                              |      | < 13.0 |                   |                  | 3.8                                     | 0    |
| 80399PFOSA-017                              | PFOSA016.D | Day 0      | 4.5                                                                                                                                                                      | 1948 | 434    | 1%                | 102%             | 3.7                                                                                                                                              | 0    | 0      | (A)               | 89%              | 3.8                                     | 1002 |
| 80399PFOSA-018                              | PFOSA017.D | Day 0      | 4.5                                                                                                                                                                      | 1921 | 425    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 1008 |
| 80399PFOSA-019                              | PFOSA018.D | Day 0      | 4.5                                                                                                                                                                      | 1948 | 427    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 1016 |
| 80399PFOSA-020                              | PFOSA019.D | Day 0      | 4.5                                                                                                                                                                      | 2936 | 683    |                   |                  | 3.7                                                                                                                                              | 572  | 230    |                   |                  | 3.8                                     | 1007 |
| 80399PFOSA-041                              | PFOSA020.D | Day 7      | 4.5                                                                                                                                                                      | 1947 | 431    | 1%                | 79%              | 3.7                                                                                                                                              | 0    | 0      | (A)               | 89%              | 3.8                                     | 1006 |
| 80399PFOSA-042                              | PFOSA021.D | Day 7      | 4.5                                                                                                                                                                      | 1945 | 435    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 997  |
| 80399PFOSA-043                              | PFOSA022.D | Day 7      | 4.5                                                                                                                                                                      | 1894 | 423    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 996  |
| 80399PFOSA-044                              | PFOSA023.D | Day 7      | 4.5                                                                                                                                                                      | 2760 | 628    |                   |                  | 3.7                                                                                                                                              | 577  | 230    |                   |                  | 3.8                                     | 1018 |
| 80399PFOSA-065                              | PFOSA024.D | Day 14     | 4.5                                                                                                                                                                      | 2054 | 435    | 1%                | 93%              | 3.7                                                                                                                                              | 0    | 0      | (A)               | 90%              | 3.8                                     | 1052 |
| 80399PFOSA-066                              | PFOSA025.D | Day 14     | 4.5                                                                                                                                                                      | 1931 | 431    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 1000 |
| 80399PFOSA-067                              | PFOSA026.D | Day 14     | 4.5                                                                                                                                                                      | 1982 | 443    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 998  |
| 80399PFOSA-068                              | PFOSA027.D | Day 14     | 4.5                                                                                                                                                                      | 2872 | 669    |                   |                  | 3.7                                                                                                                                              | 581  | 235    |                   |                  | 3.8                                     | 1003 |
| 80399PFOSA-089                              | PFOSA028.D | Day 21     | 4.5                                                                                                                                                                      | 1893 | 430    | 2%                | 101%             | 3.7                                                                                                                                              | 0    | 0      | (A)               | 94%              | 3.8                                     | 981  |
| 80399PFOSA-090                              | PFOSA029.D | Day 21     | 4.5                                                                                                                                                                      | 1970 | 440    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 999  |
| 80399PFOSA-091                              | PFOSA030.D | Day 21     | 4.5                                                                                                                                                                      | 1985 | 444    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 998  |
| 80399PFOSA-092                              | PFOSA031.D | Day 21     | 4.5                                                                                                                                                                      | 2960 | 691    |                   |                  | 3.7                                                                                                                                              | 584  | 235    |                   |                  | 3.8                                     | 1005 |
| MeOH Blank                                  | PFOSA035.D |            | 4.5                                                                                                                                                                      |      | < 12.5 |                   |                  | 3.7                                                                                                                                              |      | < 13.0 |                   |                  | 3.8                                     | 0    |
| MeOH Blank                                  | PFOSA036.D |            | 4.5                                                                                                                                                                      |      | < 12.5 |                   |                  | 3.7                                                                                                                                              |      | < 13.0 |                   |                  | 3.8                                     | 0    |
| 80399PFOSA-113                              | PFOSA037.D | Day 28     | 4.5                                                                                                                                                                      | 1883 | 431    | 1%                | 103%             | 3.7                                                                                                                                              | 0    | 0      | (A)               | 90%              | 3.8                                     | 974  |
| 80399PFOSA-114                              | PFOSA038.D | Day 28     | 4.5                                                                                                                                                                      | 1849 | 422    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 975  |
| 80399PFOSA-115                              | PFOSA039.D | Day 28     | 4.5                                                                                                                                                                      | 1850 | 430    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 958  |
| 80399PFOSA-116                              | PFOSA040.D | Day 28     | 4.5                                                                                                                                                                      | 2813 | 686    |                   |                  | 3.7                                                                                                                                              | 557  | 235    |                   |                  | 3.8                                     | 962  |
| 80399PFOSA-137                              | PFOSA041.D | Day 35     | 4.5                                                                                                                                                                      | 1948 | 450    | 0%                | 104%             | 3.7                                                                                                                                              | 0    | 0      | (A)               | 93%              | 3.8                                     | 968  |
| 80399PFOSA-138                              | PFOSA042.D | Day 35     | 4.5                                                                                                                                                                      | 1936 | 452    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 958  |
| 80399PFOSA-139                              | PFOSA043.D | Day 35     | 4.5                                                                                                                                                                      | 1917 | 450    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 953  |
| 80399PFOSA-140                              | PFOSA044.D | Day 35     | 4.5                                                                                                                                                                      | 2933 | 711    |                   |                  | 3.7                                                                                                                                              | 579  | 241    |                   |                  | 3.8                                     | 974  |
| 80399PFOSA-161                              | PFOSA045.D | Day 42     | 4.5                                                                                                                                                                      | 1872 | 444    | 2%                | 98%              | 3.7                                                                                                                                              | 0    | 0      | (A)               | 91%              | 3.8                                     | 941  |
| 80399PFOSA-162                              | PFOSA046.D | Day 42     | 4.5                                                                                                                                                                      | 1929 | 452    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 955  |
| 80399PFOSA-163                              | PFOSA047.D | Day 42     | 4.5                                                                                                                                                                      | 1951 | 459    |                   |                  | 3.7                                                                                                                                              | 0    | 0      |                   |                  | 3.8                                     | 952  |
| 80399PFOSA-164                              | PFOSA048.D | Day 42     | 4.5                                                                                                                                                                      | 2831 | 697    |                   |                  | 3.7                                                                                                                                              | 559  | 237    |                   |                  | 3.8                                     | 955  |
| MeOH Blank                                  | PFOSA049.D |            | 4.5                                                                                                                                                                      |      | < 12.5 |                   |                  | 3.7                                                                                                                                              |      | < 13.0 |                   |                  | 3.8                                     | 0    |
| MeOH Blank                                  | PFOSA050.D |            | 4.5                                                                                                                                                                      |      | < 12.5 |                   |                  | 3.7                                                                                                                                              |      | < 13.0 |                   |                  | 3.8                                     | 0    |
| 99039-139-01                                | PFOSA051.D |            |                                                                                                                                                                          |      |        |                   |                  | 3.7                                                                                                                                              | 26   | 23     |                   |                  |                                         |      |
| 99039-139-02                                | PFOSA052.D |            | 4.5                                                                                                                                                                      | 190  | 57     | 113%              |                  | 3.7                                                                                                                                              | 112  | 57     | 109%              |                  | 3.8                                     | 941  |
| 99039-139-03                                | PFOSA053.D |            | 4.5                                                                                                                                                                      | 408  | 99     | 100%              |                  | 3.7                                                                                                                                              | 242  | 105    | 101%              |                  | 3.8                                     | 981  |
| 99039-139-04                                | PFOSA054.D |            | 4.5                                                                                                                                                                      | 1050 | 245    | 98%               |                  | 3.7                                                                                                                                              | 597  | 256    | 99%               |                  | 3.8                                     | 942  |
| 99039-139-05                                | PFOSA055.D |            | 4.5                                                                                                                                                                      | 2165 | 521    | 104%              |                  | 3.7                                                                                                                                              | 1218 | 547    | 105%              |                  | 3.8                                     | 942  |
| 99039-139-06                                | PFOSA056.D |            | 4.5                                                                                                                                                                      | 3835 | 1042   | 104%              |                  | 3.7                                                                                                                                              | 2127 | 1089   | 105%              |                  | 3.8                                     | 946  |
| Method ID:                                  | 0309_15C.M |            | Internal Standard quant: $r^2=0.999$<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 49.9 998<br>Original conc. = 499.4<br>Spike conc. = 249.7 |      |        |                   |                  | Internal Standard quant: $r^2=0.999$<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 51.9 1038<br>Spike conc. = 259.40 |      |        |                   |                  | Conc. mean 986<br>S.D. 30<br>%S.D. 3.0% |      |

(A) The relative percent deviation (RPD) is not defined when the mean concentration is zero.

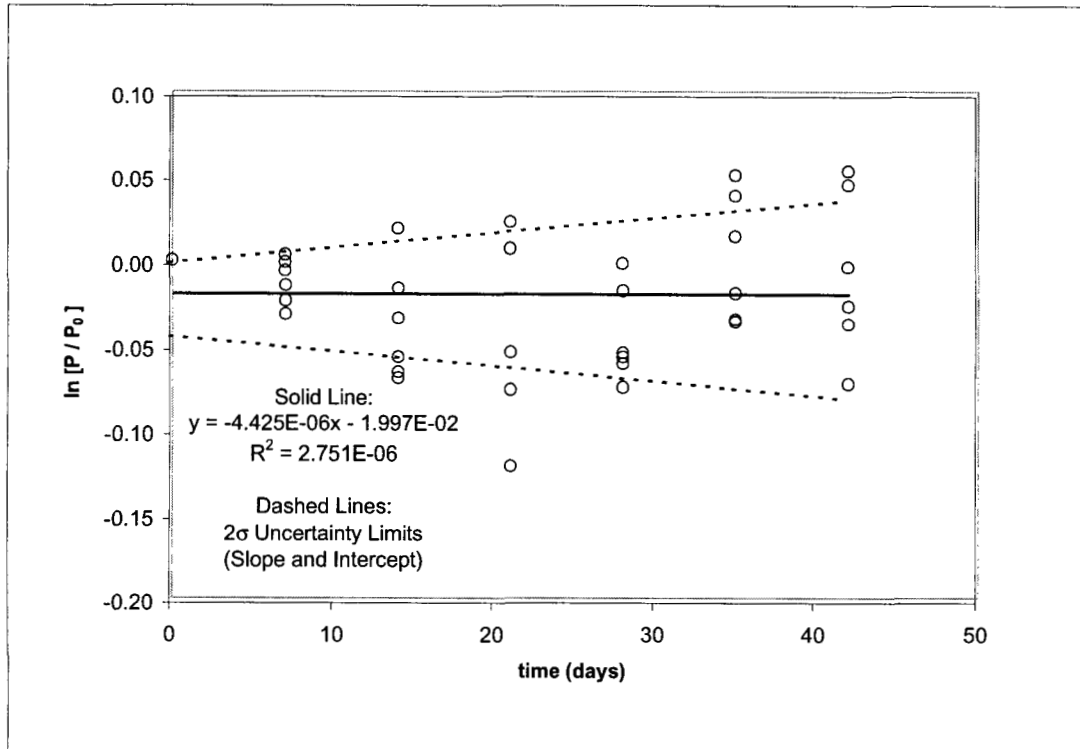
FOSA (Perfluorooctanesulfonamide) Hydrolysis Study  
50°C; pH 11All concentrations in ng/ml. All areas x 10<sup>-3</sup>

| FOSA           |            |            |                                                                                                                                                                              |      |        | PFOS              |                  |                                                                                                                                                      |      |        |                   | FBSA             |          |      |  |
|----------------|------------|------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|------------------------------------------------------------------------------------------------------------------------------------------------------|------|--------|-------------------|------------------|----------|------|--|
| Sample ID      | Data File  | Time Point | Ret Time                                                                                                                                                                     | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time                                                                                                                                             | Area | Conc.  | %Standard or %RSD | % Spike Recovery | Ret Time | Area |  |
| MeOH Blank     | PFOSA058.D |            | 4.5                                                                                                                                                                          |      | < 12.5 |                   |                  | 3.7                                                                                                                                                  |      | < 13.0 |                   |                  | 3.8      | 0    |  |
| 99039-140-01   | PFOSA059.D |            |                                                                                                                                                                              |      |        |                   |                  | 3.7                                                                                                                                                  | 27   | 20     |                   |                  |          |      |  |
| 99039-140-02   | PFOSA060.D |            | 4.5                                                                                                                                                                          | 196  | 53     | 107%              |                  | 3.7                                                                                                                                                  | 115  | 54     | 104%              |                  | 3.8      | 932  |  |
| 99039-140-03   | PFOSA061.D |            | 4.5                                                                                                                                                                          | 393  | 96     | 96%               |                  | 3.7                                                                                                                                                  | 227  | 99     | 95%               |                  | 3.8      | 934  |  |
| 99039-140-04   | PFOSA062.D |            | 4.5                                                                                                                                                                          | 1031 | 242    | 97%               |                  | 3.7                                                                                                                                                  | 596  | 255    | 98%               |                  | 3.8      | 927  |  |
| 99039-140-05   | PFOSA063.D |            | 4.5                                                                                                                                                                          | 1995 | 475    | 95%               |                  | 3.7                                                                                                                                                  | 1126 | 491    | 95%               |                  | 3.8      | 936  |  |
| 99039-140-06   | PFOSA064.D |            | 4.5                                                                                                                                                                          | 3844 | 998    | 100%              |                  | 3.7                                                                                                                                                  | 2139 | 1026   | 99%               |                  | 3.8      | 951  |  |
| MeOH Blank     | PFOSA069.D |            | 4.5                                                                                                                                                                          |      | < 12.5 |                   |                  | 3.7                                                                                                                                                  |      | < 13.0 |                   |                  | 3.8      | 0    |  |
| MeOH Blank     | PFOSA070.D |            | 4.5                                                                                                                                                                          |      | < 12.5 |                   |                  | 3.7                                                                                                                                                  |      | < 13.0 |                   |                  | 3.8      | 0    |  |
| 80399PFOSA-021 | PFOSA071.D | Day 0      | 4.5                                                                                                                                                                          | 1906 | 447    | 1%                | 105%             | 3.7                                                                                                                                                  | 0    | 0      | (A)               | 91%              | 3.8      | 946  |  |
| 80399PFOSA-022 | PFOSA072.D | Day 0      | 4.5                                                                                                                                                                          | 1884 | 450    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 929  |  |
| 80399PFOSA-023 | PFOSA073.D | Day 0      | 4.5                                                                                                                                                                          | 1856 | 440    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 935  |  |
| 80399PFOSA-024 | PFOSA074.D | Day 0      | 4.5                                                                                                                                                                          | 2893 | 707    |                   |                  | 3.7                                                                                                                                                  | 568  | 236    |                   |                  | 3.8      | 951  |  |
| 80399PFOSA-045 | PFOSA075.D | Day 7      | 4.5                                                                                                                                                                          | 1852 | 437    | 2%                | 99%              | 3.7                                                                                                                                                  | 0    | 0      | (A)               | 92%              | 3.8      | 938  |  |
| 80399PFOSA-046 | PFOSA076.D | Day 7      | 4.5                                                                                                                                                                          | 1884 | 446    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 936  |  |
| 80399PFOSA-047 | PFOSA077.D | Day 7      | 4.5                                                                                                                                                                          | 1906 | 453    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 935  |  |
| 80399PFOSA-048 | PFOSA078.D | Day 7      | 4.5                                                                                                                                                                          | 2810 | 692    |                   |                  | 3.7                                                                                                                                                  | 570  | 239    |                   |                  | 3.8      | 941  |  |
| 80399PFOSA-069 | PFOSA079.D | Day 14     | 4.5                                                                                                                                                                          | 1933 | 459    | 4%                | 101%             | 3.7                                                                                                                                                  | 0    | 0      | (A)               | 93%              | 3.8      | 937  |  |
| 80399PFOSA-070 | PFOSA080.D | Day 14     | 4.5                                                                                                                                                                          | 1782 | 427    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 923  |  |
| 80399PFOSA-071 | PFOSA081.D | Day 14     | 4.5                                                                                                                                                                          | 1855 | 430    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 956  |  |
| 80399PFOSA-072 | PFOSA082.D | Day 14     | 4.5                                                                                                                                                                          | 2845 | 692    |                   |                  | 3.7                                                                                                                                                  | 584  | 242    |                   |                  | 3.8      | 953  |  |
| 80399PFOSA-093 | PFOSA083.D | Day 21     | 4.5                                                                                                                                                                          | 1871 | 446    | (B)               | (B)              | 3.7                                                                                                                                                  | 0    | 0      | (A)               | 0%               | 3.8      | 932  |  |
| 80399PFOSA-094 | PFOSA084.D | Day 21     | 4.5                                                                                                                                                                          |      | 445    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 953  |  |
| 80399PFOSA-095 | PFOSA085.D | Day 21     | 4.5                                                                                                                                                                          |      | 445    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 953  |  |
| 80399PFOSA-096 | PFOSA086.D | Day 21     | 4.5                                                                                                                                                                          | 1913 | 445    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 953  |  |
| MeOH Blank     | PFOSA090.D |            | 4.5                                                                                                                                                                          |      | < 12.5 |                   |                  | 3.7                                                                                                                                                  |      | < 13.0 |                   |                  | 3.8      | 0    |  |
| MeOH Blank     | PFOSA091.D |            | 4.5                                                                                                                                                                          |      | < 12.5 |                   |                  | 3.7                                                                                                                                                  |      | < 13.0 |                   |                  | 3.8      | 0    |  |
| 80399PFOSA-117 | PFOSA092.D | Day 28     | 4.5                                                                                                                                                                          | 1829 | 435    | 1%                | 104%             | 3.7                                                                                                                                                  | 0    | 0      | (A)               | 91%              | 3.8      | 932  |  |
| 80399PFOSA-118 | PFOSA093.D | Day 28     | 4.5                                                                                                                                                                          | 1886 | 436    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 959  |  |
| 80399PFOSA-119 | PFOSA094.D | Day 28     | 4.5                                                                                                                                                                          | 1853 | 444    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 927  |  |
| 80399PFOSA-120 | PFOSA095.D | Day 28     | 4.5                                                                                                                                                                          | 2800 | 697    |                   |                  | 3.7                                                                                                                                                  | 558  | 237    |                   |                  | 3.8      | 931  |  |
| 80399PFOSA-141 | PFOSA096.D | Day 35     | 4.5                                                                                                                                                                          | 1949 | 461    | 0%                | 96%              | 3.7                                                                                                                                                  | 0    | 0      | (A)               | 93%              | 3.8      | 940  |  |
| 80399PFOSA-142 | PFOSA097.D | Day 35     | 4.5                                                                                                                                                                          | 1921 | 464    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 921  |  |
| 80399PFOSA-143 | PFOSA098.D | Day 35     | 4.5                                                                                                                                                                          | 1911 | 464    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 916  |  |
| 80399PFOSA-144 | PFOSA099.D | Day 35     | 4.5                                                                                                                                                                          | 2819 | 704    |                   |                  | 3.7                                                                                                                                                  | 567  | 241    |                   |                  | 3.8      | 930  |  |
| 80399PFOSA-165 | PFOSA100.D | Day 42     | 4.5                                                                                                                                                                          | 1955 | 473    | 4%                | 95%              | 3.7                                                                                                                                                  | 0    | 0      | (A)               | 91%              | 3.8      | 922  |  |
| 80399PFOSA-166 | PFOSA101.D | Day 42     | 4.5                                                                                                                                                                          | 1831 | 444    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 916  |  |
| 80399PFOSA-167 | PFOSA102.D | Day 42     | 4.5                                                                                                                                                                          | 1956 | 482    |                   |                  | 3.7                                                                                                                                                  | 0    | 0      |                   |                  | 3.8      | 905  |  |
| 80399PFOSA-168 | PFOSA103.D | Day 42     | 4.5                                                                                                                                                                          | 2793 | 703    |                   |                  | 3.7                                                                                                                                                  | 550  | 236    |                   |                  | 3.8      | 922  |  |
| MeOH Blank     | PFOSA104.D |            | 4.5                                                                                                                                                                          |      | < 12.5 |                   |                  | 3.7                                                                                                                                                  |      | < 13.0 |                   |                  | 3.8      | 0    |  |
| MeOH Blank     | PFOSA105.D |            | 4.5                                                                                                                                                                          |      | < 12.5 |                   |                  | 3.7                                                                                                                                                  |      | < 13.0 |                   |                  | 3.8      | 0    |  |
| 99039-140-01   | PFOSA106.D |            |                                                                                                                                                                              |      |        |                   |                  | 3.7                                                                                                                                                  | 25   | 19     |                   |                  |          |      |  |
| 99039-140-02   | PFOSA107.D |            | 4.5                                                                                                                                                                          | 187  | 53     | 107%              |                  | 3.7                                                                                                                                                  | 109  | 54     | 105%              |                  | 3.8      | 882  |  |
| 99039-140-03   | PFOSA108.D |            | 4.5                                                                                                                                                                          | 406  | 101    | 101%              |                  | 3.7                                                                                                                                                  | 234  | 104    | 100%              |                  | 3.8      | 913  |  |
| 99039-140-04   | PFOSA109.D |            | 4.5                                                                                                                                                                          | 998  | 247    | 99%               |                  | 3.7                                                                                                                                                  | 583  | 262    | 101%              |                  | 3.8      | 880  |  |
| 99039-140-05   | PFOSA110.D |            | 4.5                                                                                                                                                                          | 2105 | 533    | 107%              |                  | 3.7                                                                                                                                                  | 1185 | 549    | 106%              |                  | 3.8      | 890  |  |
| 99039-140-06   | PFOSA111.D |            | 4.5                                                                                                                                                                          | 3653 | 998    | 100%              |                  | 3.7                                                                                                                                                  | 2068 | 1049   | 101%              |                  | 3.8      | 903  |  |
| Method ID:     | 0309_15C.M |            | Internal Standard quant: r <sup>2</sup> =0.999<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 49.9<br>Original conc.= 499.4<br>Spike conc.= 249.7 |      |        |                   |                  | Internal Standard quant: r <sup>2</sup> =0.999<br>Curve Averaged, Quadratic, Origin ignored<br>Cal. range min. and max = 51.9<br>Spike conc.= 259.40 |      |        |                   |                  | Conc.    | 225  |  |
|                |            |            |                                                                                                                                                                              |      |        |                   |                  |                                                                                                                                                      |      |        |                   |                  | mean     | 930  |  |
|                |            |            |                                                                                                                                                                              |      |        |                   |                  |                                                                                                                                                      |      |        |                   |                  | S.D.     | 19   |  |
|                |            |            |                                                                                                                                                                              |      |        |                   |                  |                                                                                                                                                      |      |        |                   |                  | %S.D.    | 2.1% |  |

(A) The relative standard deviation (RSD) is not defined when the mean concentration is zero.

(B) Excluded; samples 80399PFOSA-094 and -095 not analyzed, due to human or mechanical error; no spike added to sample 80399PFOSA-096.

Pooled FOSA Data and Slope Regression



## SUMMARY OUTPUT

| Regression Statistics |              |
|-----------------------|--------------|
| Multiple R            | 0.001658616  |
| R Square              | 2.75101E-06  |
| Adjusted R Square     | -0.025638204 |
| Standard Error        | 0.038763091  |
| Observations          | 41           |

% 2σ slope uncertainty  
19309

| ANOVA      |    |             |             |            |                |
|------------|----|-------------|-------------|------------|----------------|
|            | df | SS          | MS          | F          | Significance F |
| Regression | 1  | 1.61211E-07 | 1.61211E-07 | 0.00010729 | 0.991788409    |
| Residual   | 39 | 0.058600511 | 0.001502577 |            |                |
| Total      | 40 | 0.058600673 |             |            |                |

|              | Coefficients | Standard Error | t Stat      | P-value     | Lower 95%    | Upper 95%   | Lower 95.0%  | Upper 95.0% |
|--------------|--------------|----------------|-------------|-------------|--------------|-------------|--------------|-------------|
| Intercept    | -0.019972663 | 0.010823282    | -1.84534255 | 0.072590288 | -0.041864795 | 0.00191947  | -0.041864795 | 0.00191947  |
| X Variable 1 | -4.42532E-06 | 0.000427234    | -0.01035807 | 0.991788409 | -0.000868587 | 0.000859736 | -0.000868587 | 0.000859736 |



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**Rate and T<sub>1/2</sub> Calculations**Author: **GMP**Date: **4/02/01**

Shaded results included in the report.

|                               |           |
|-------------------------------|-----------|
| <b>Incubation time (days)</b> | 4.200E+01 |
|-------------------------------|-----------|

| Parent | Parent Mass (gm/mole) | Parent P0 (ng/ml) | Parent P0 (nm/ml) |
|--------|-----------------------|-------------------|-------------------|
| FOSA   | 4.990E+02             | 4.994E+02         | 1.001E+00         |

| Analyte (LOQ) | Analyte Mass (gm/mole) | Analyte LOQ (ng/ml) | Analyte LOQ (nm/ml) |
|---------------|------------------------|---------------------|---------------------|
| PFOS          | 5.379E+02              | 5.200E+01           | 9.667E-02           |

| Analyte (LOD) | Analyte Mass (gm/mole) | Analyte LOD (ng/ml) | Analyte LOD (nm/ml) |
|---------------|------------------------|---------------------|---------------------|
| PFOS          | 5.379E+02              | 1.300E+01           | 2.417E-02           |

| Rates and Half-Lives from LOQ<br>(Eq's B32, B33, and B41) | Max Rate @50C (day-1)<br>2.300E-03 | Max Rate @25C (day-1)<br>2.300E-04 | Min Half-Life @25C (year)<br>8.3 |
|-----------------------------------------------------------|------------------------------------|------------------------------------|----------------------------------|
|-----------------------------------------------------------|------------------------------------|------------------------------------|----------------------------------|

| Rates and Half-Lives from LOD<br>(Eq's B38, B39, and B41) | Max Rate @50C (day-1)<br>5.749E-04 | Max Rate @25C (day-1)<br>5.749E-05 | Min Half-Life @25C (year)<br>33 |
|-----------------------------------------------------------|------------------------------------|------------------------------------|---------------------------------|
|-----------------------------------------------------------|------------------------------------|------------------------------------|---------------------------------|

| Rates and Half-Lives from mean ( $\mu$ ) and standard deviation ( $2\sigma$ ) concentrations:<br>(Eq's B36, B37, and B41) | Max Rate @50C (day-1)<br>1.802E-03 | Max Rate @25C (day-1)<br>1.802E-04 | Min Half-Life @25C (year)<br>11 |
|---------------------------------------------------------------------------------------------------------------------------|------------------------------------|------------------------------------|---------------------------------|
| mean =                                                                                                                    | 4.551E+02                          |                                    |                                 |
| stdev =                                                                                                                   | 1.722E+01                          |                                    |                                 |

| Pooled Data                                 | Slopes @50C (day-1)     | Slopes @25C (day-1)      |                            |
|---------------------------------------------|-------------------------|--------------------------|----------------------------|
| Regression Slope 2 $\sigma$ Minimum (day-1) | -8.686E-04              | -8.686E-05               |                            |
| Regression Slope Value (day-1)              | -4.425E-06              | -4.425E-07               |                            |
| Regression Slope 2 $\sigma$ Maximum (day-1) | 8.597E-04               | 8.597E-05                |                            |
|                                             | Max. H-Life @25C (year) | Calc. H-Life @25C (year) | Min. Half-Life @25C (year) |
| (Eq's B8, B9, and B41)                      | (N/A - slope positive)  | 4291                     | 22                         |

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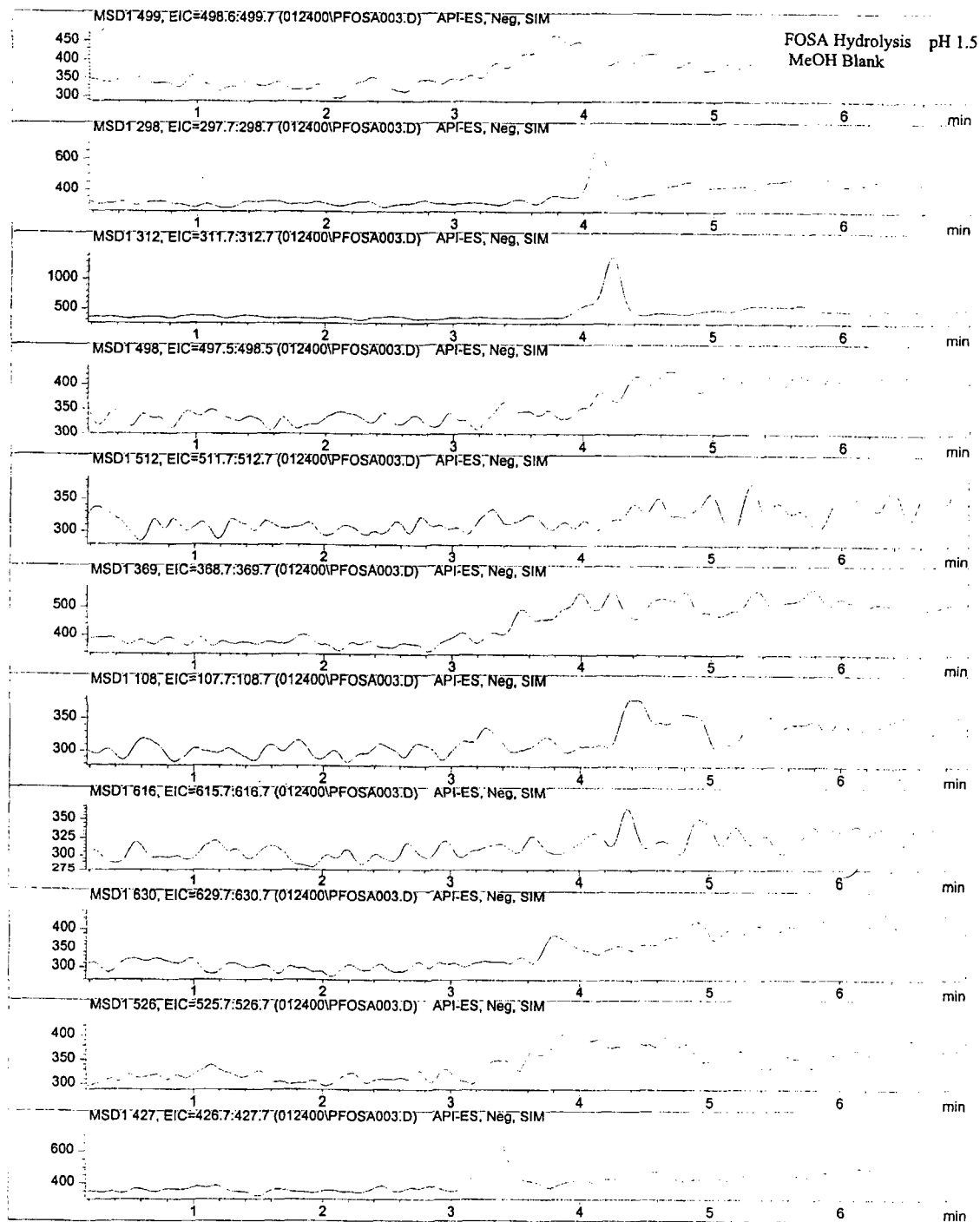
## **Appendix D: Selected Chromatograms**

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

Batch Run # 3 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA003.D

Sample Name: MeOH Blank



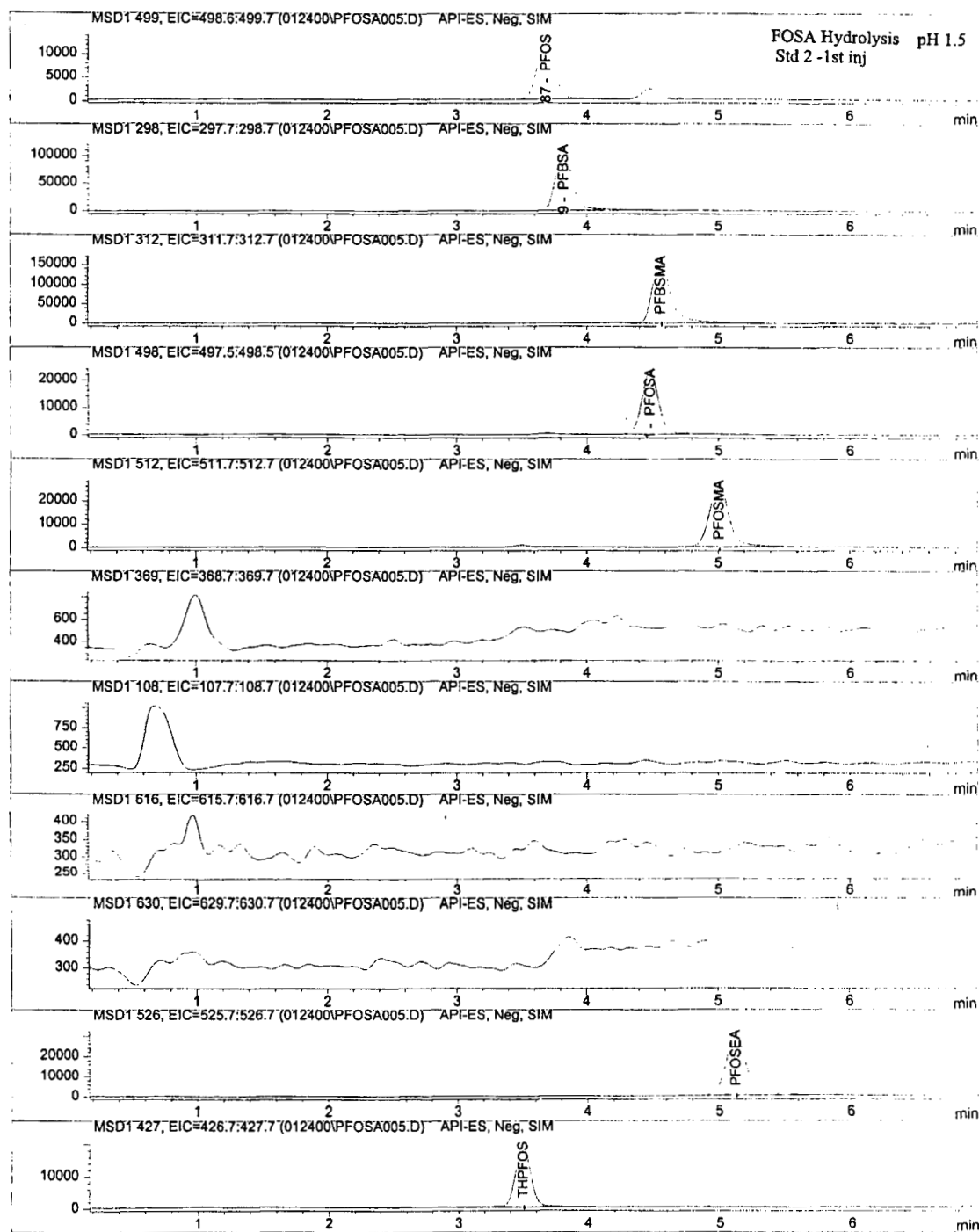
Data4 9/21/00 12:38:00 PM AES/ALS

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Batch Run # 5 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA005.D

Sample Name: 99039-135-02



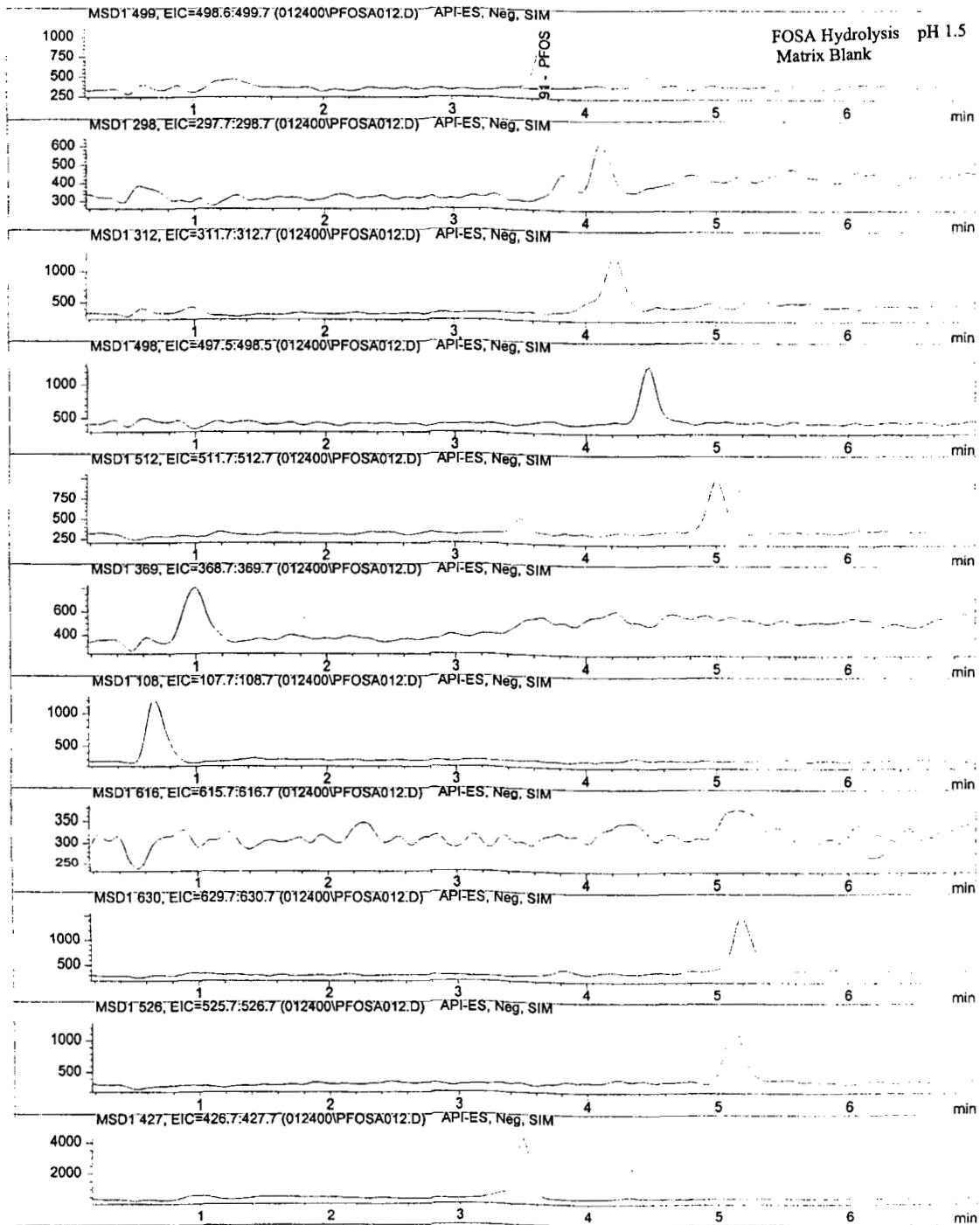
Data4 9/21/00 12:38:21 PM AES/ALS

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Batch Run # 12 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA012.D

Sample Name: 081399-BLK-1.5



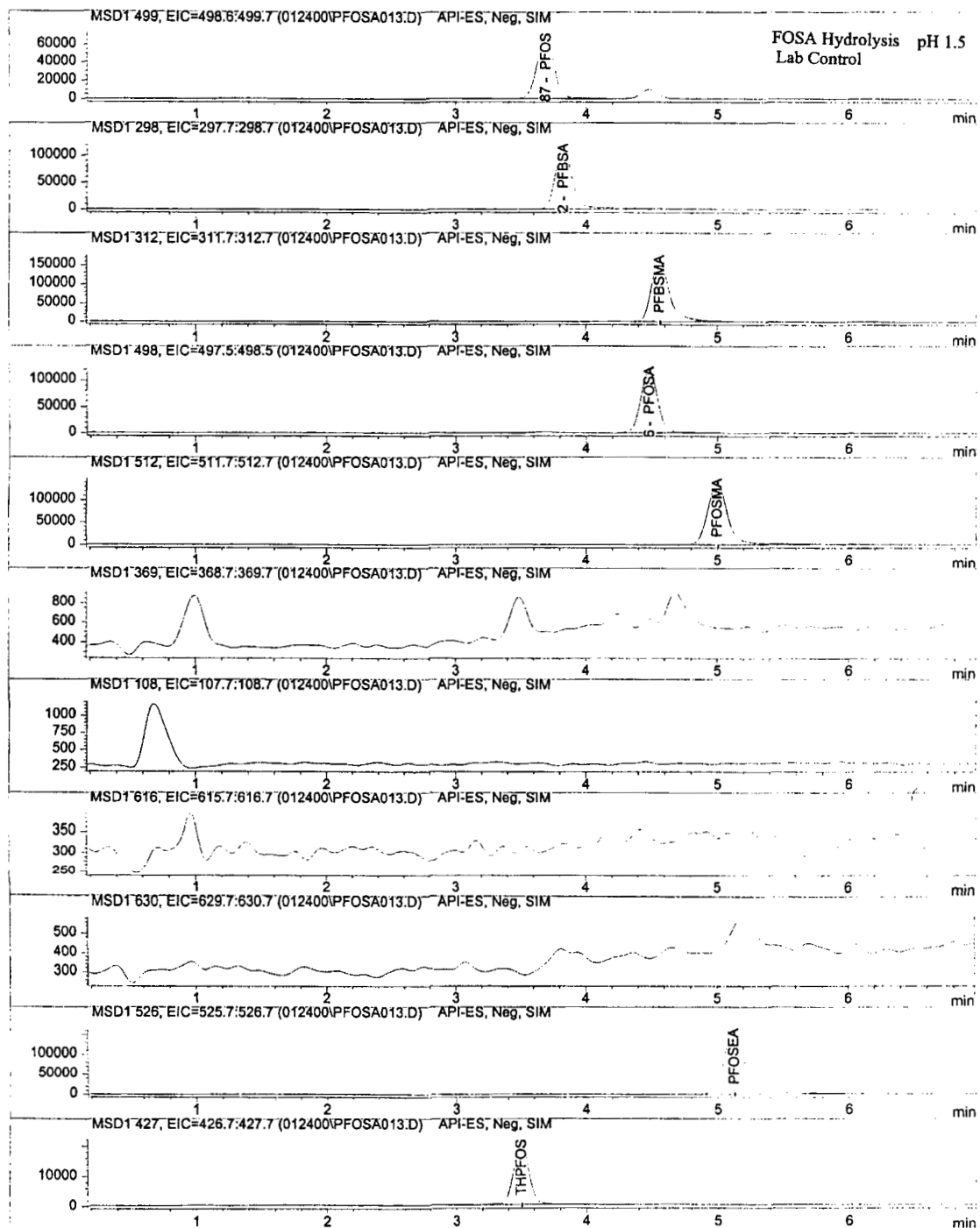
Data4 9/21/00 12:39:27 PM AES/ALS

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Batch Run # 13 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA013.D

Sample Name: 081399-LCS-1.5



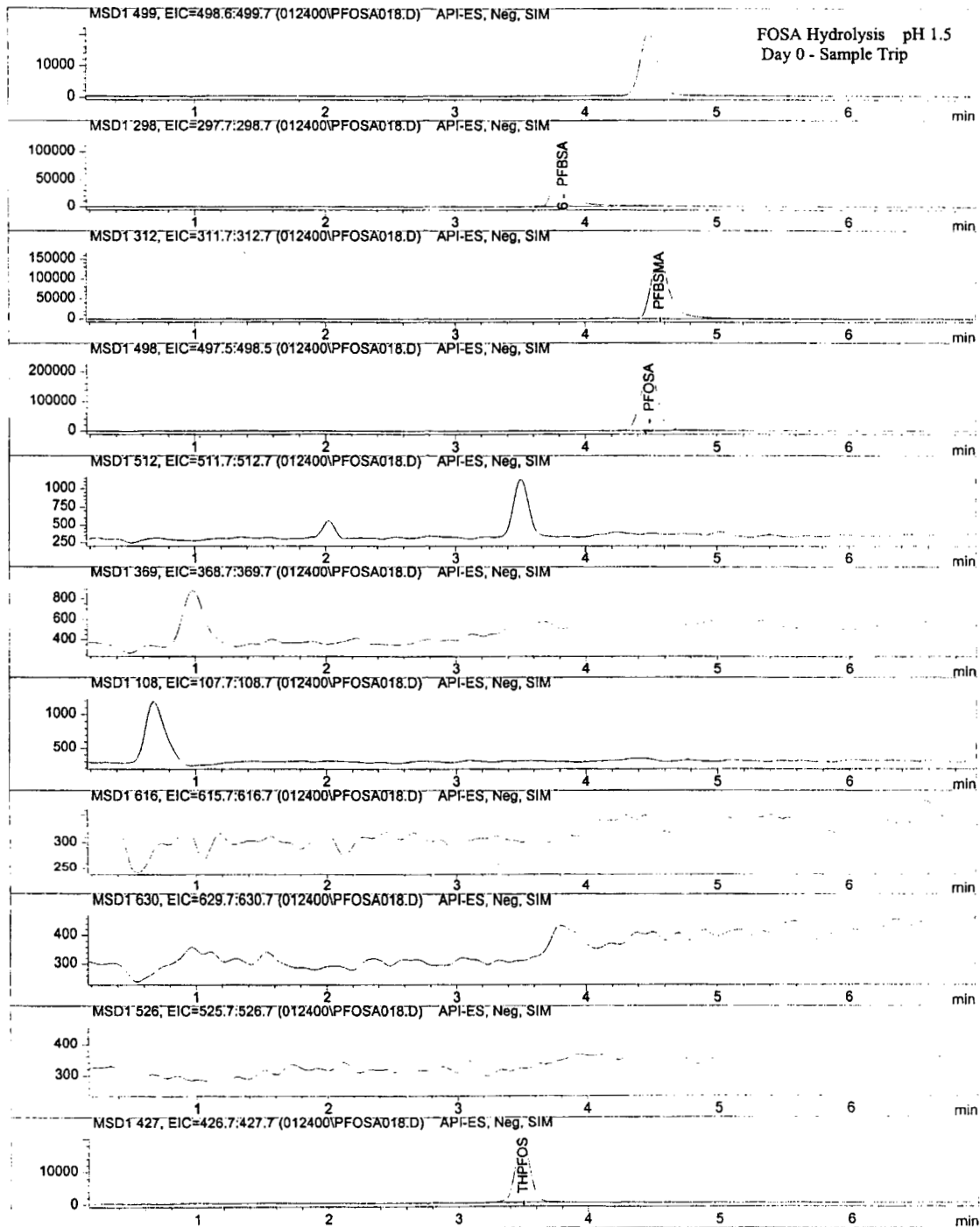
Data4 9/21/00 12:39:36 PM AES/ALS

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Batch Run # 18 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA018.D

Sample Name: 80399PFOSA-003



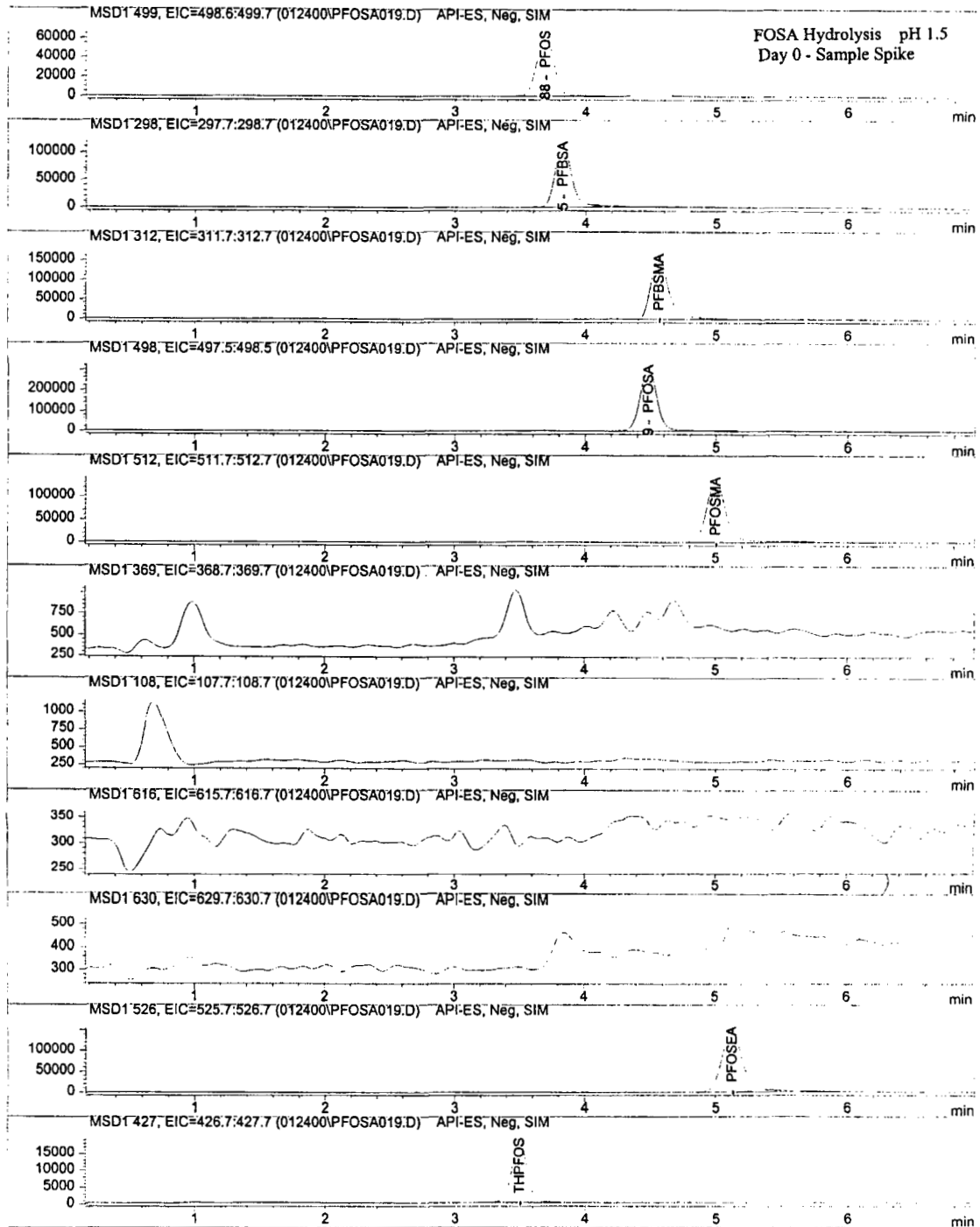
Data4 9/21/00 12:40:24 PM AES/ALS

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Batch Run # 19 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA019.D

Sample Name: 80399PFOSA-004



Data4 9/21/00 12:40:33 PM AES/ALS

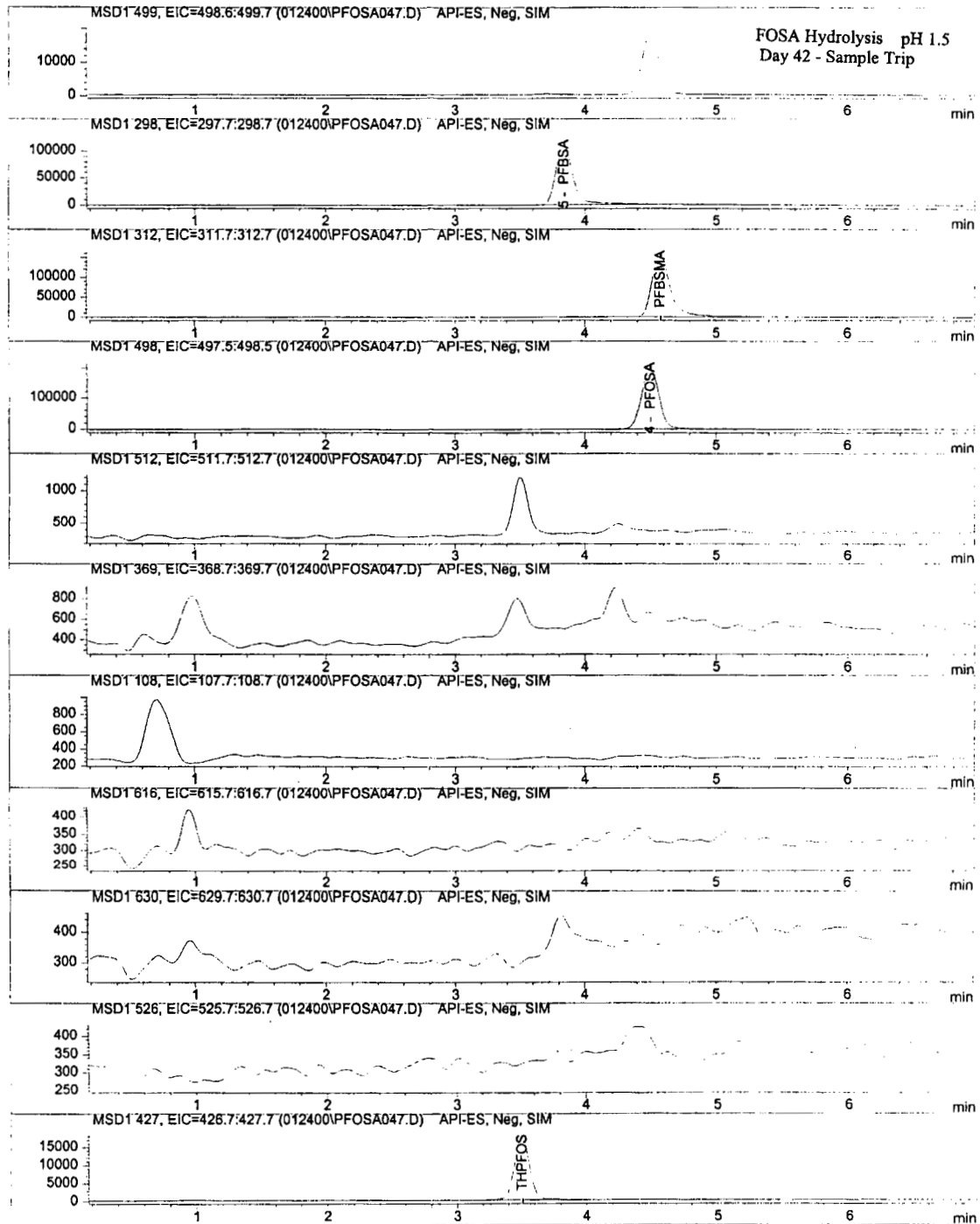
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Batch Run # 47 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA047.D

Sample Name: 80399PFOSA-147



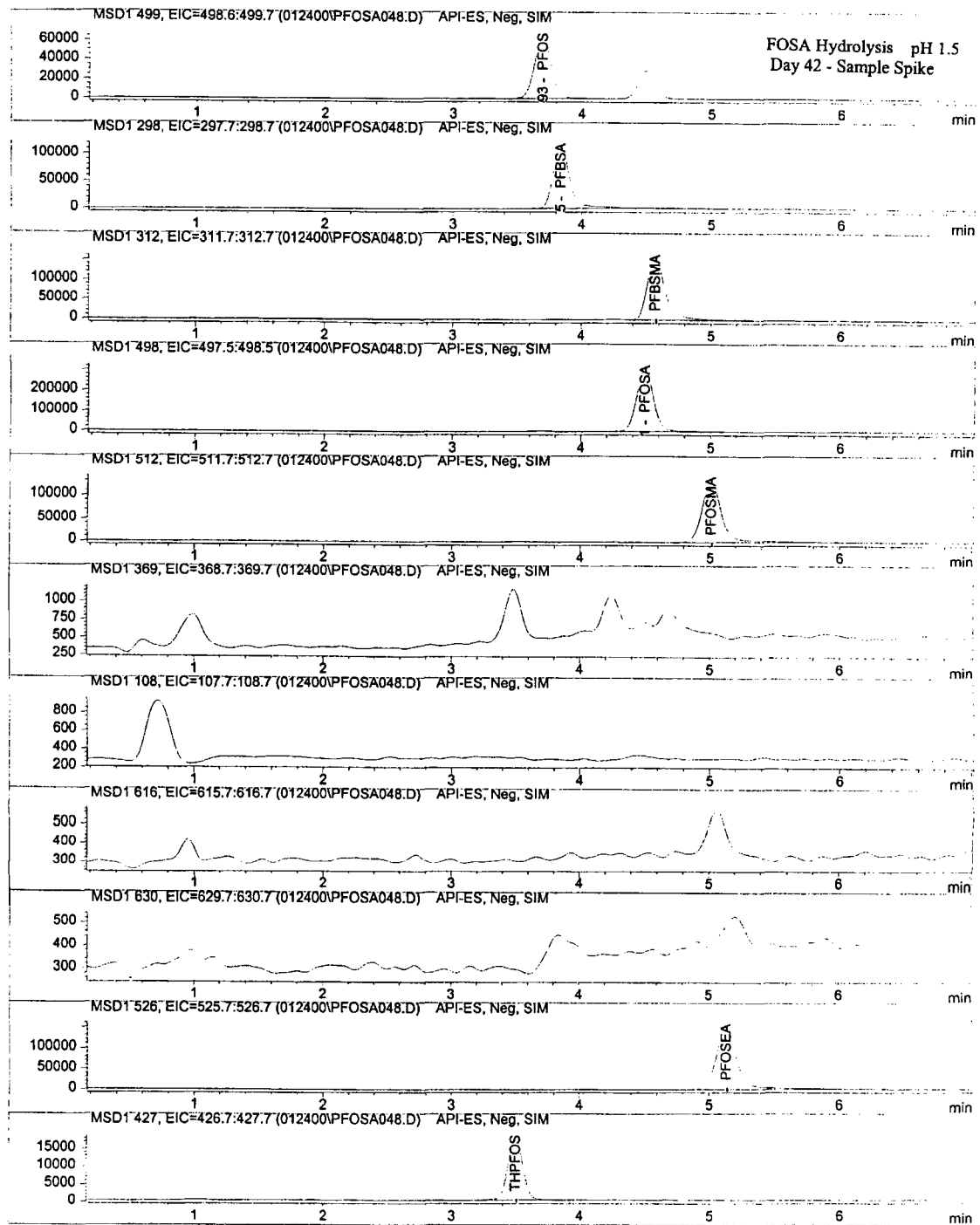
Data4 9/21/00 12:44:55 PM AES/ALS

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Batch Run # 48 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA048.D

Sample Name: 80399PFOSA-148



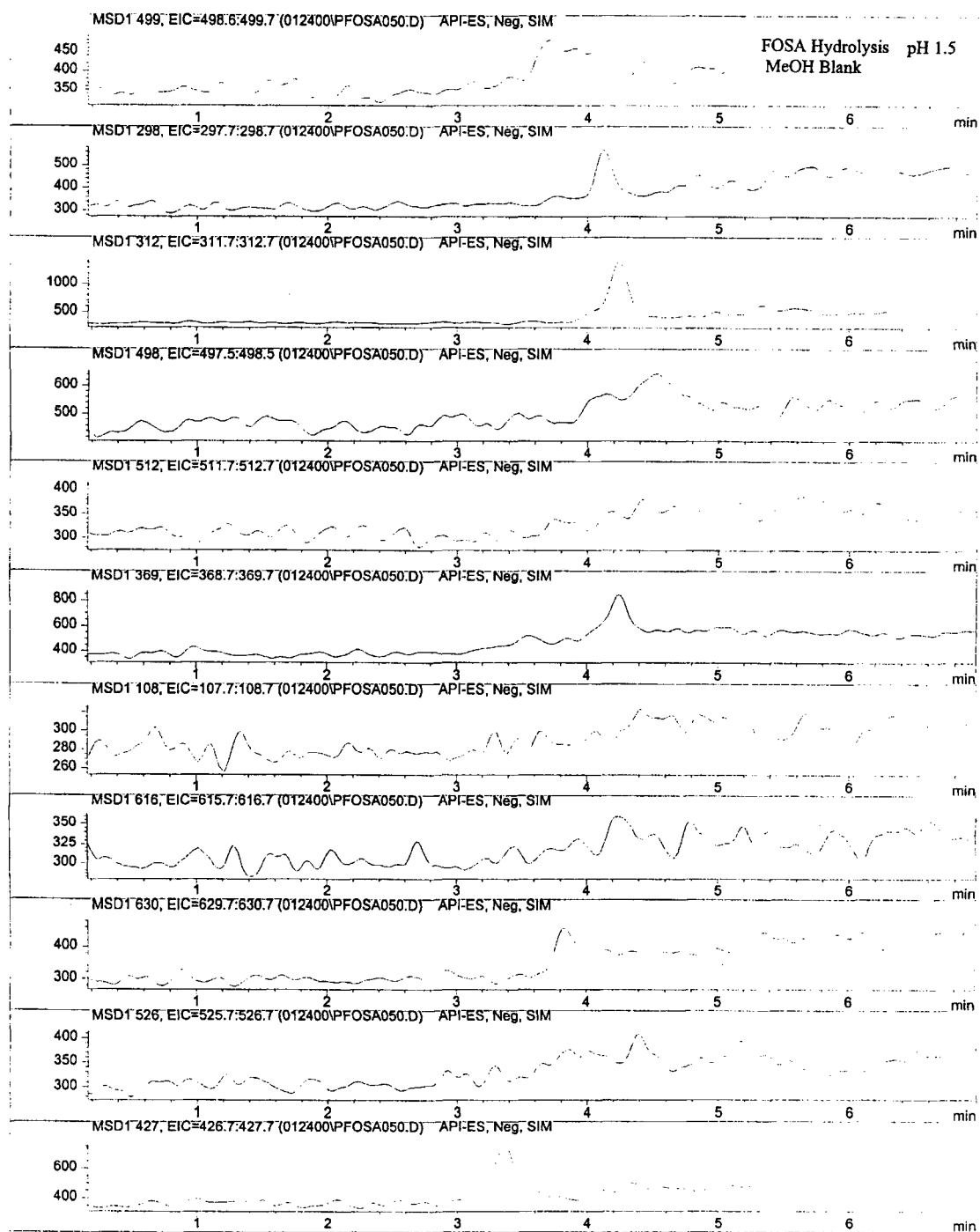
Data4 9/21/00 12:45:03 PM AES/ALS

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Batch Run # 50 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA050.D

Sample Name: MeOH Blank



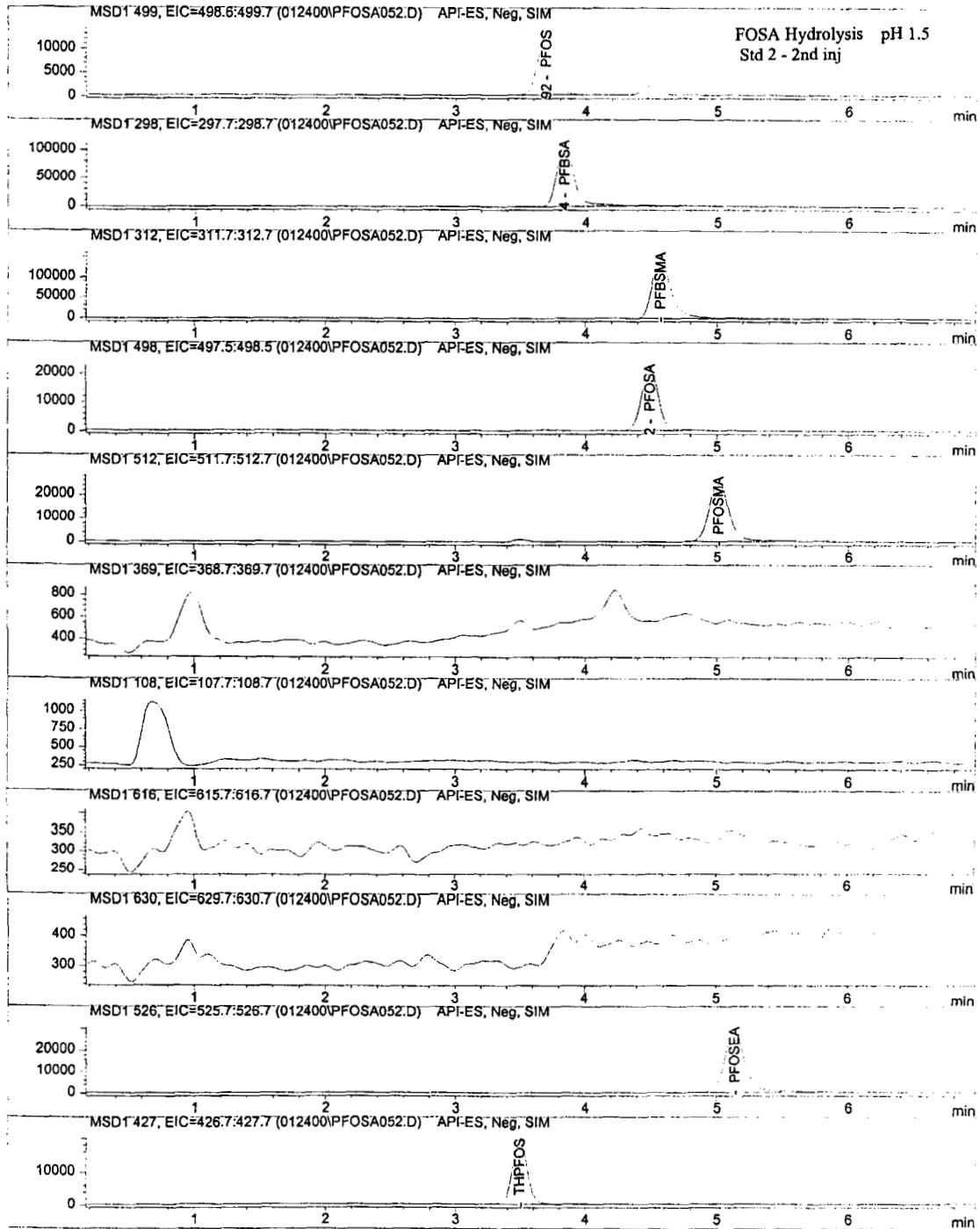
Data4 9/21/00 12:45:23 PM AES/ALS

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Batch Run # 52 of 57

Data File C:\HPCHEM\1\DATA\012400\PFOSA052.D

Sample Name: 99039-135-02



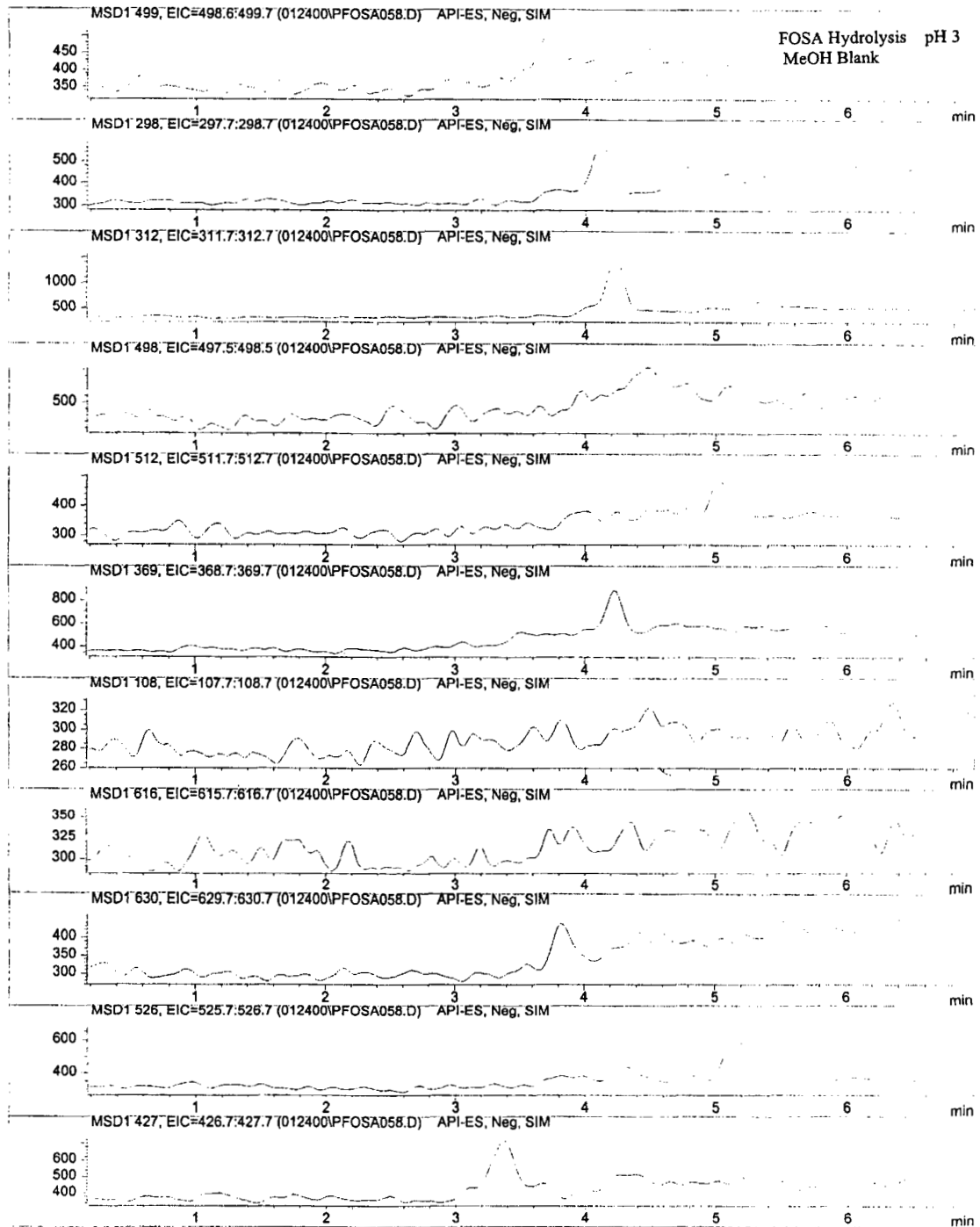
Data4 9/21/00 12:45:43 PM AES/ALS

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Batch Run # 2 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA058.D

Sample Name: MeOH Blank



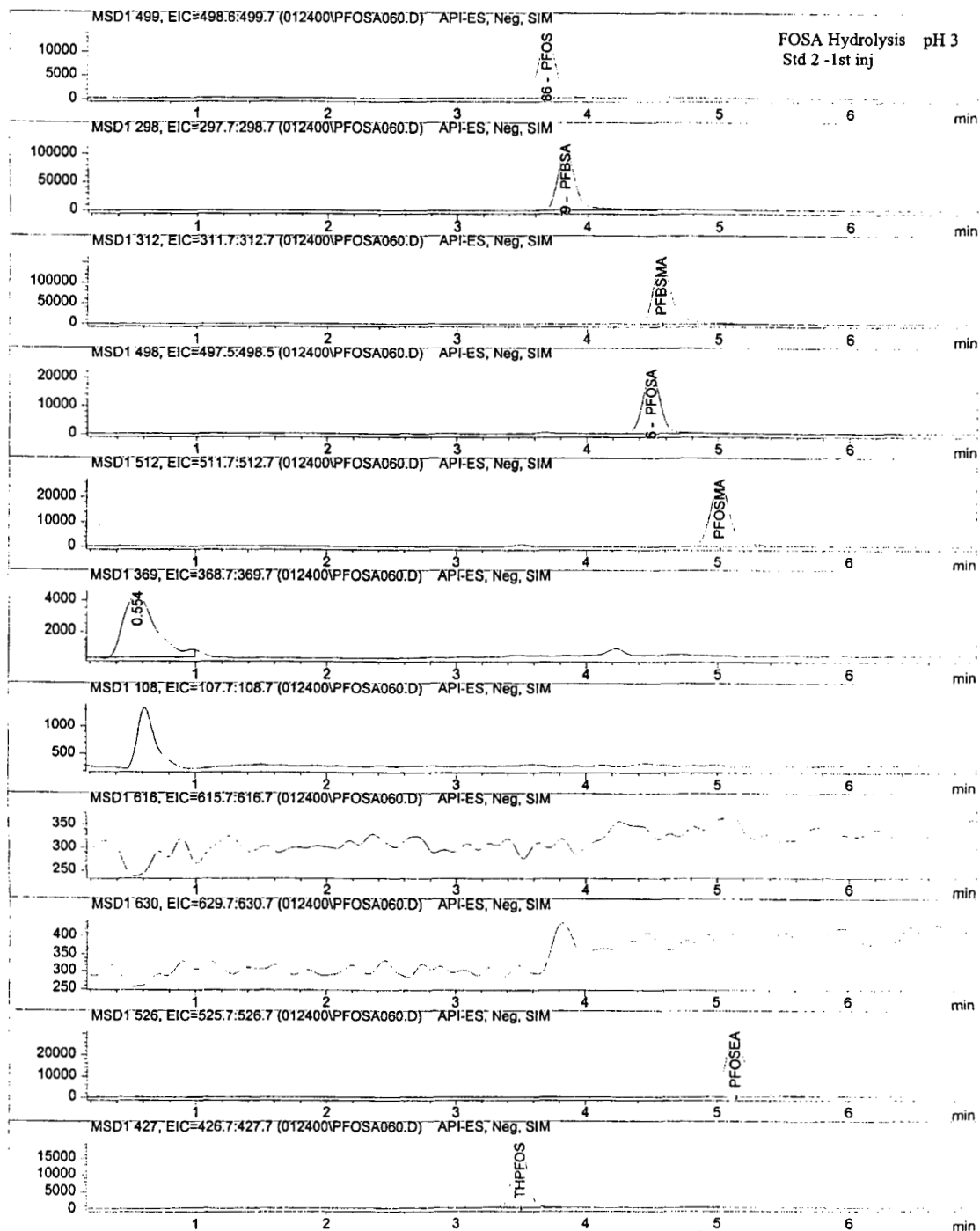
Data4 9/26/00 1:04:37 PM AES/ALS

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Batch Run # 4 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA060.D

Sample Name: 99039-136-02



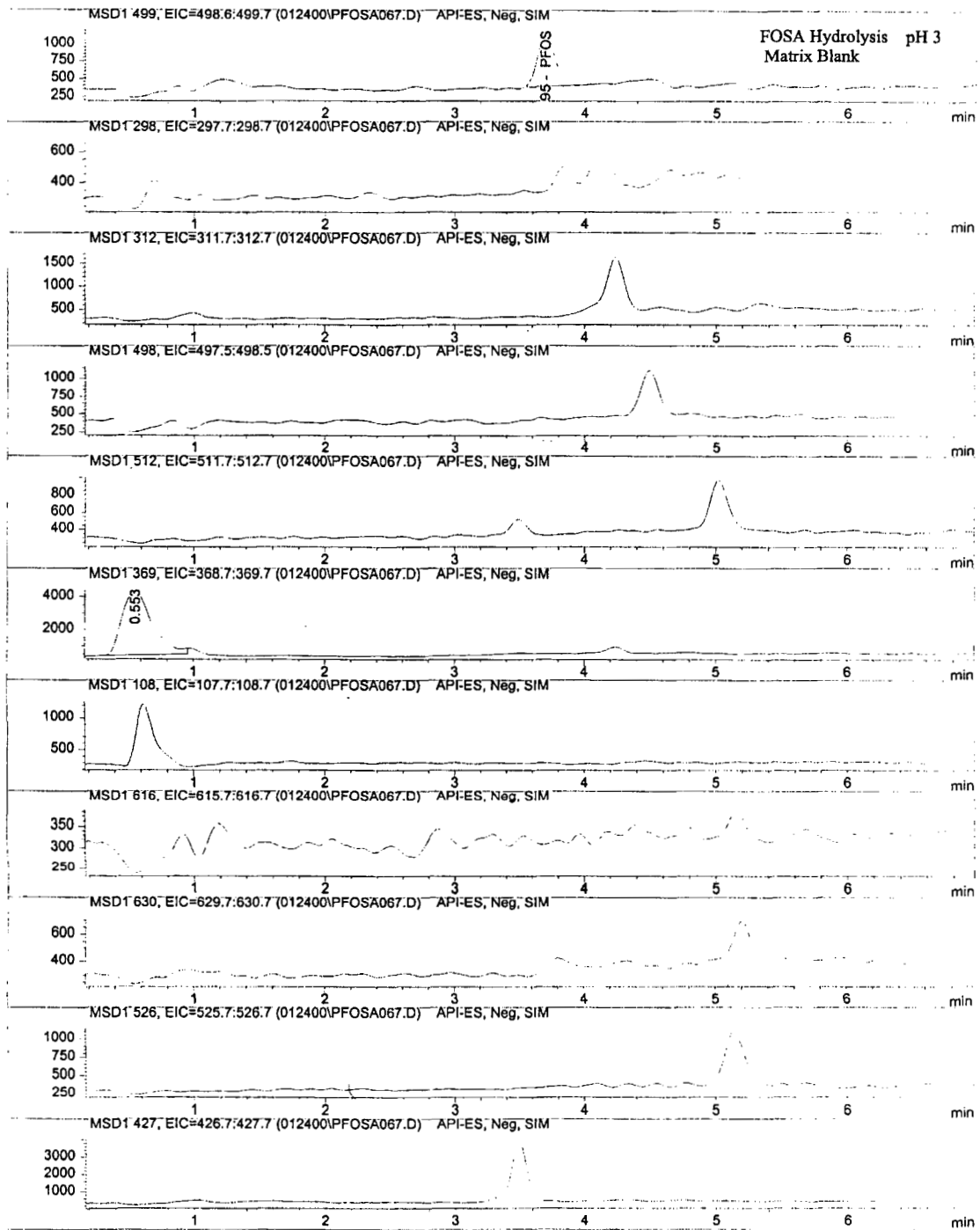
Data4 9/26/00 1:04:57 PM AES/ALS

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Batch Run # 11 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA067.D

Sample Name: 081399-BLK-3.0



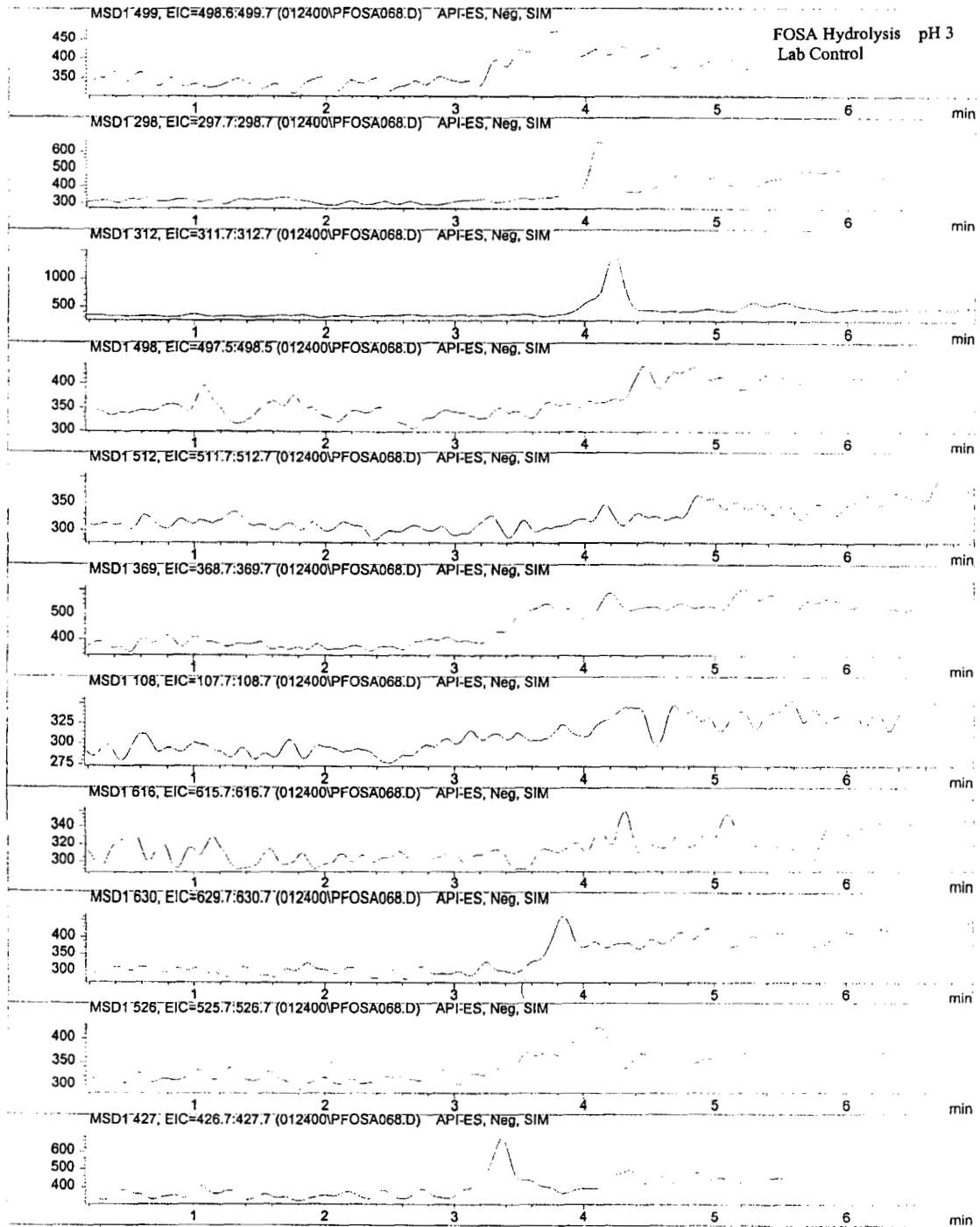
Data4 9/26/00 1:05:59 PM AES/ALS

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Batch Run # 12 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA068.D

Sample Name: 081399-LCS-3.0



Data4 9/26/00 1:06:08 PM AES/ALS

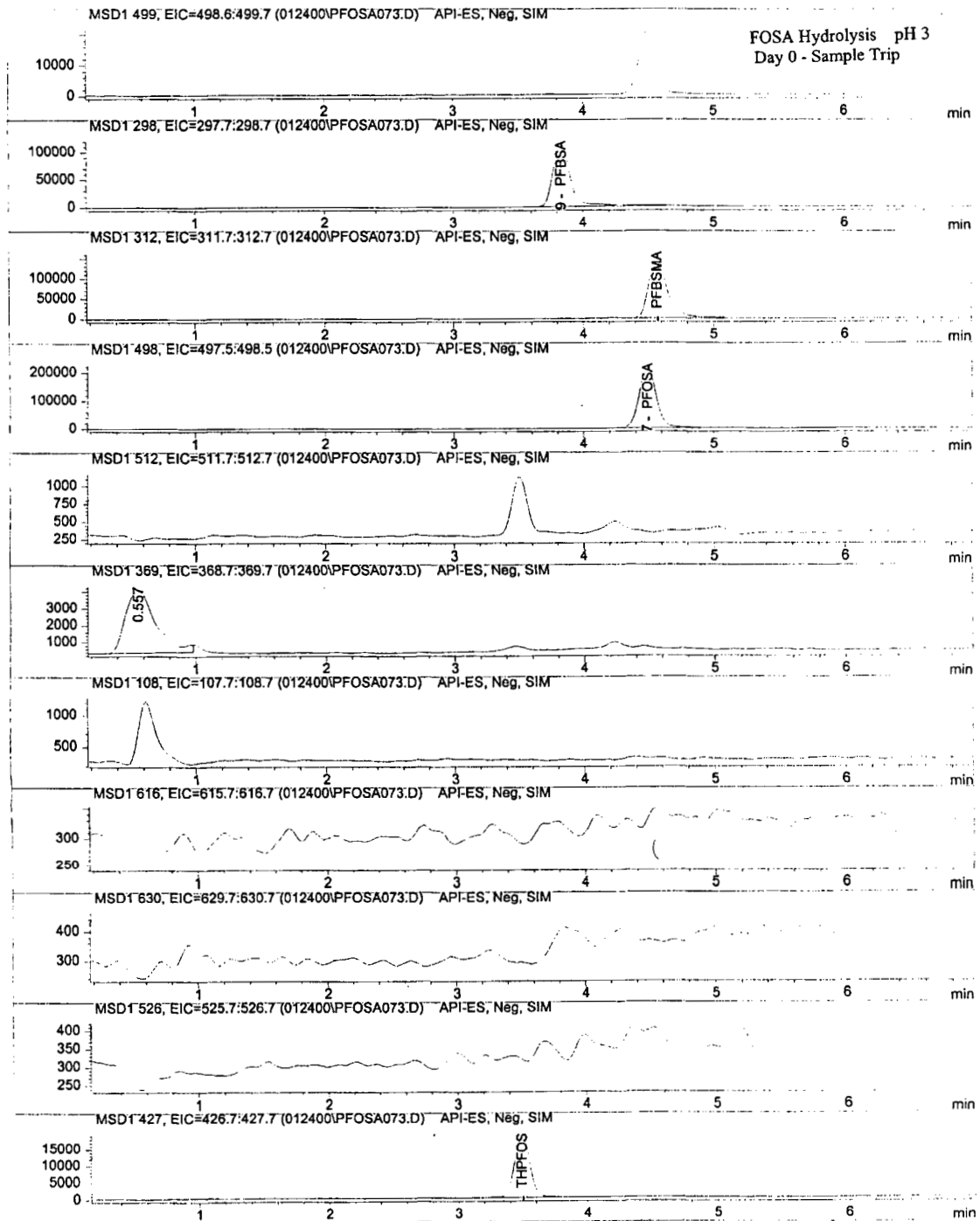
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Batch Run # 17 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA073.D

Sample Name: 80399PFOSA-007



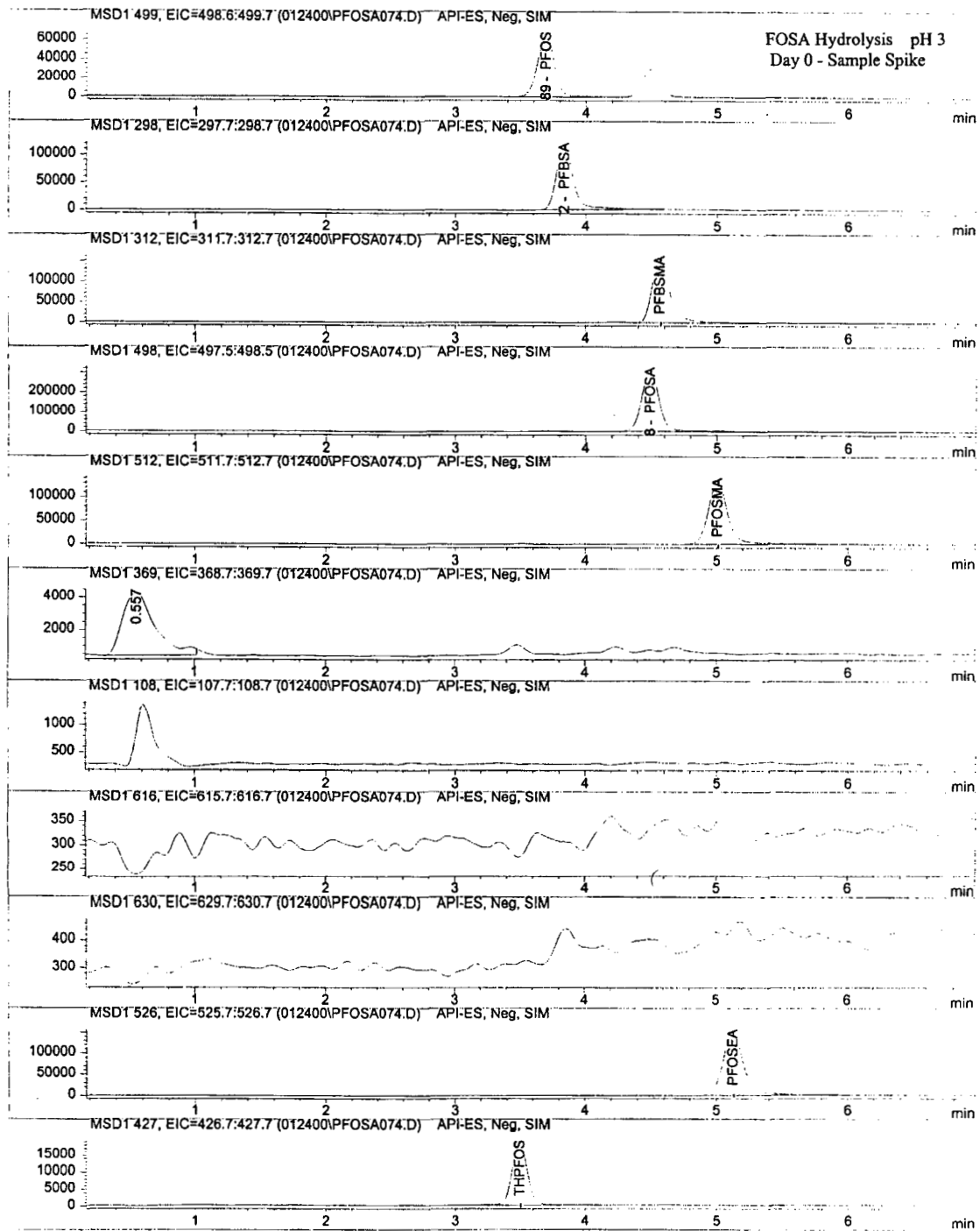
Data4 9/26/00 1:06:54 PM AES/ALS

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Batch Run # 18 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA074.D

Sample Name: 80399PFOSA-008



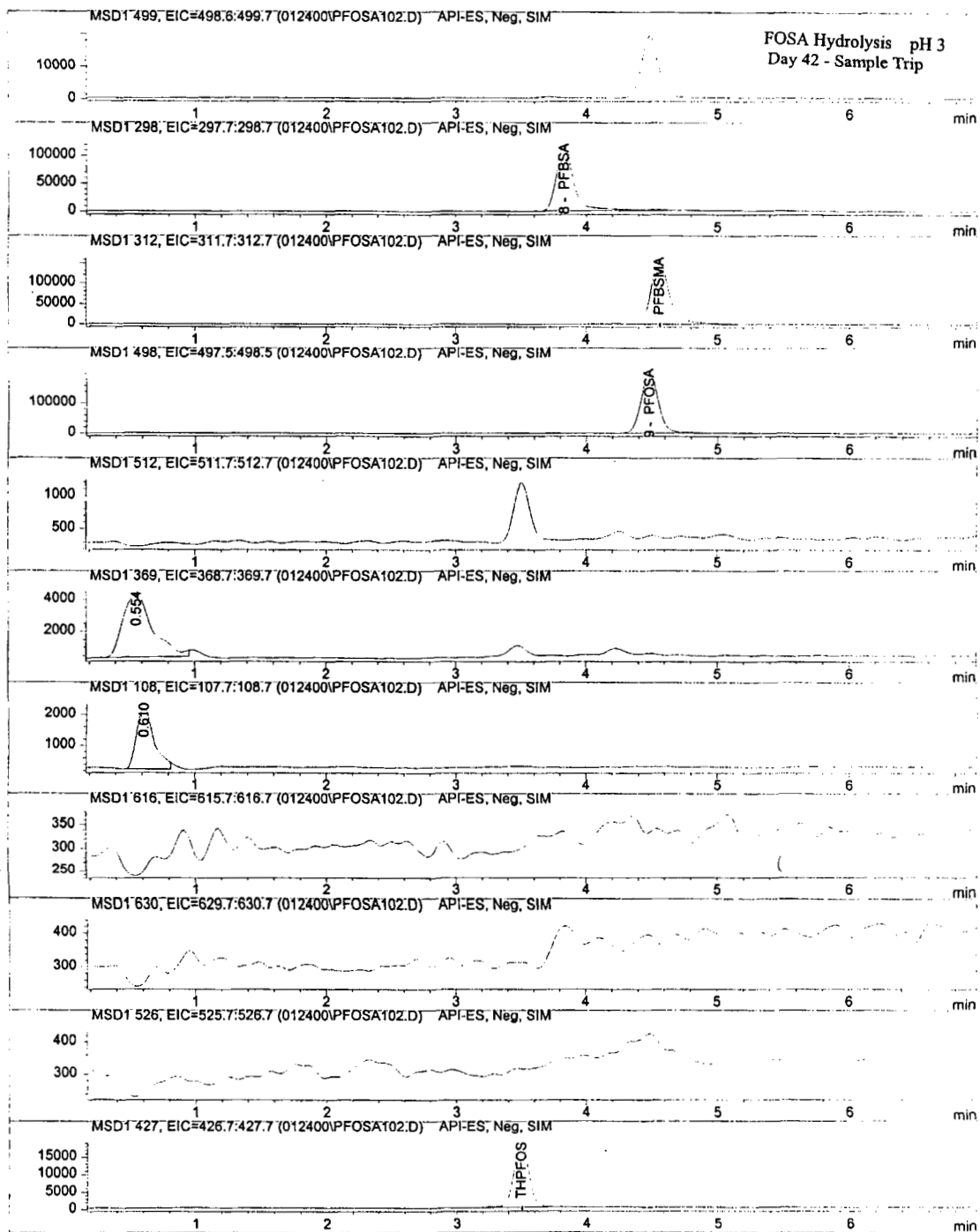
Data4 9/26/00 1:07:01 PM AES/ALS

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Batch Run # 46 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA102.D

Sample Name: 80399PFOSA-151



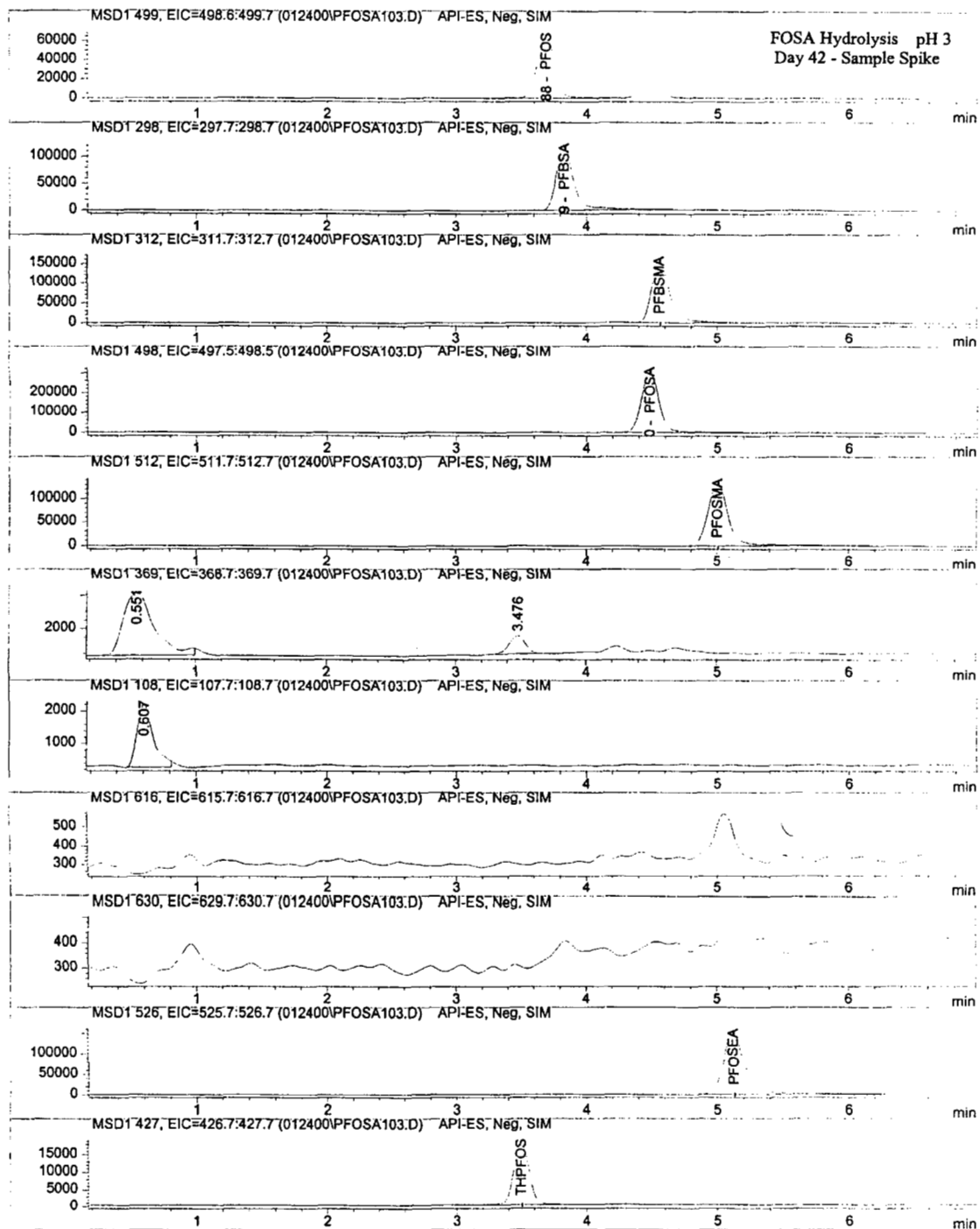
Data4 9/26/00 1:10:54 PM AES/ALS

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Batch Run # 47 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA103.D

Sample Name: 80399PFOSA-152



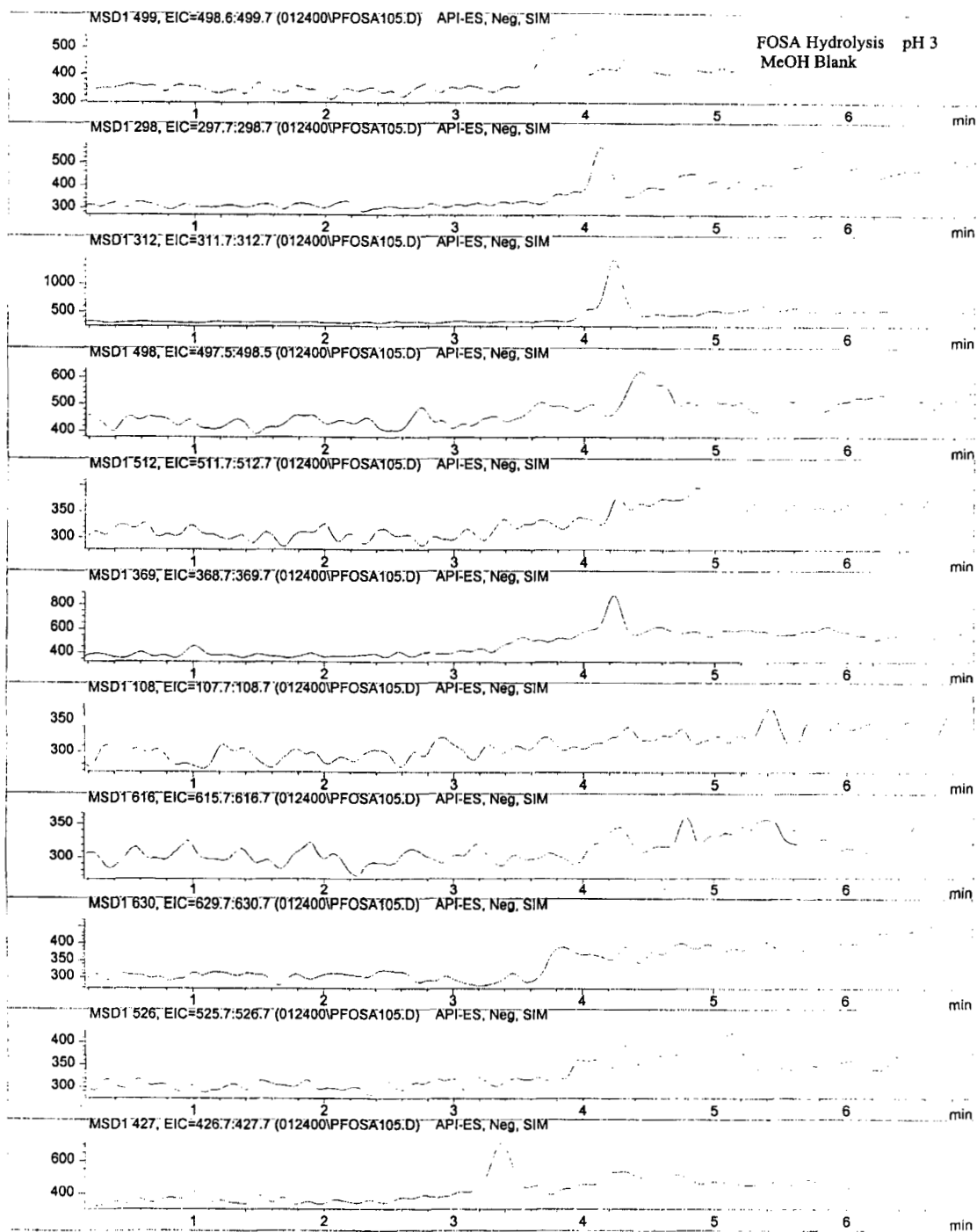
Data4 9/26/00 1:11:02 PM AES/ALS

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Batch Run # 49 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA105.D

Sample Name: MeOH Blank



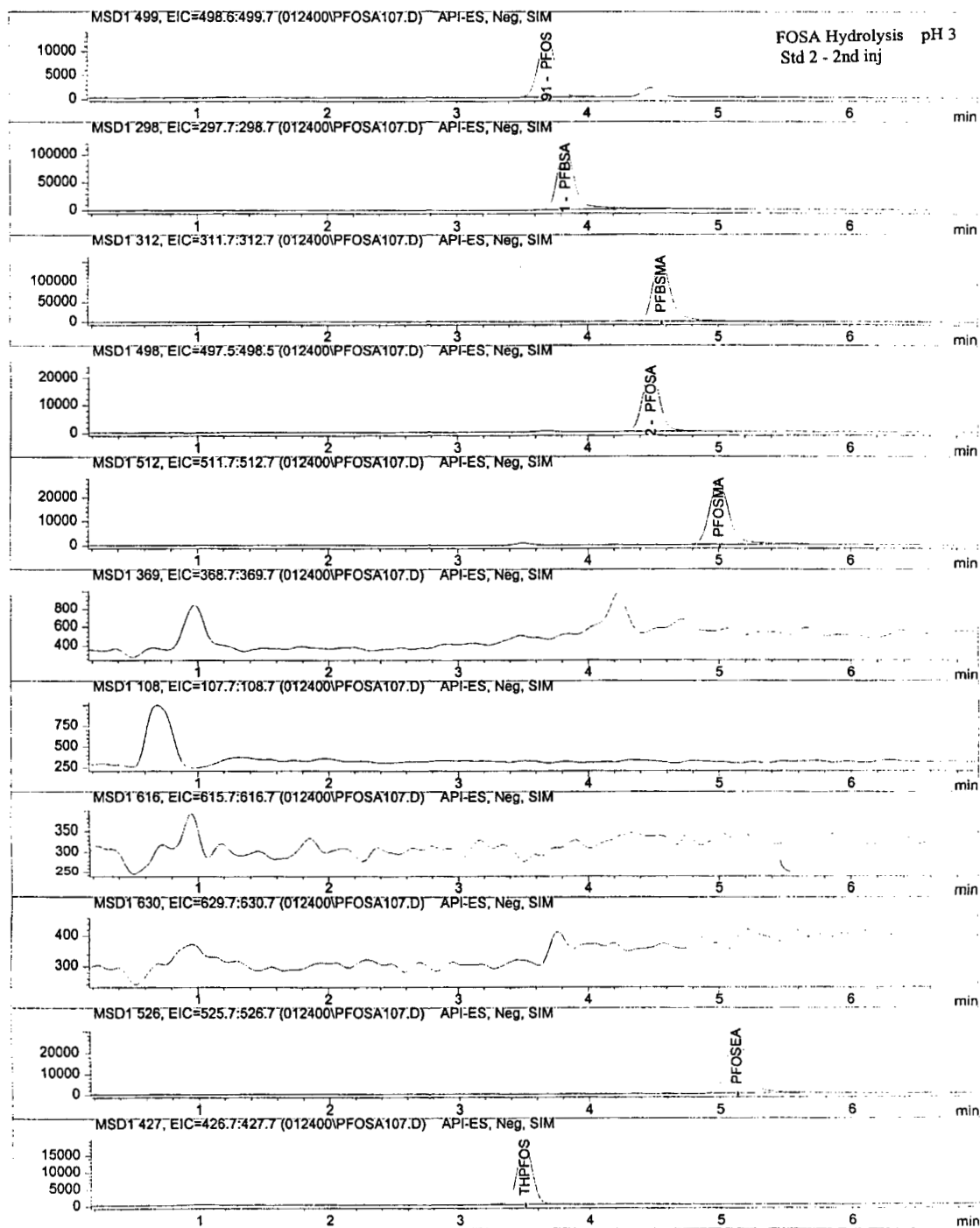
Data4 9/26/00 1:11:20 PM AES/ALS

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Batch Run # 51 of 55

Data File C:\HPCHEM\1\DATA\012400\PFOSA107.D

Sample Name: 99039-136-02



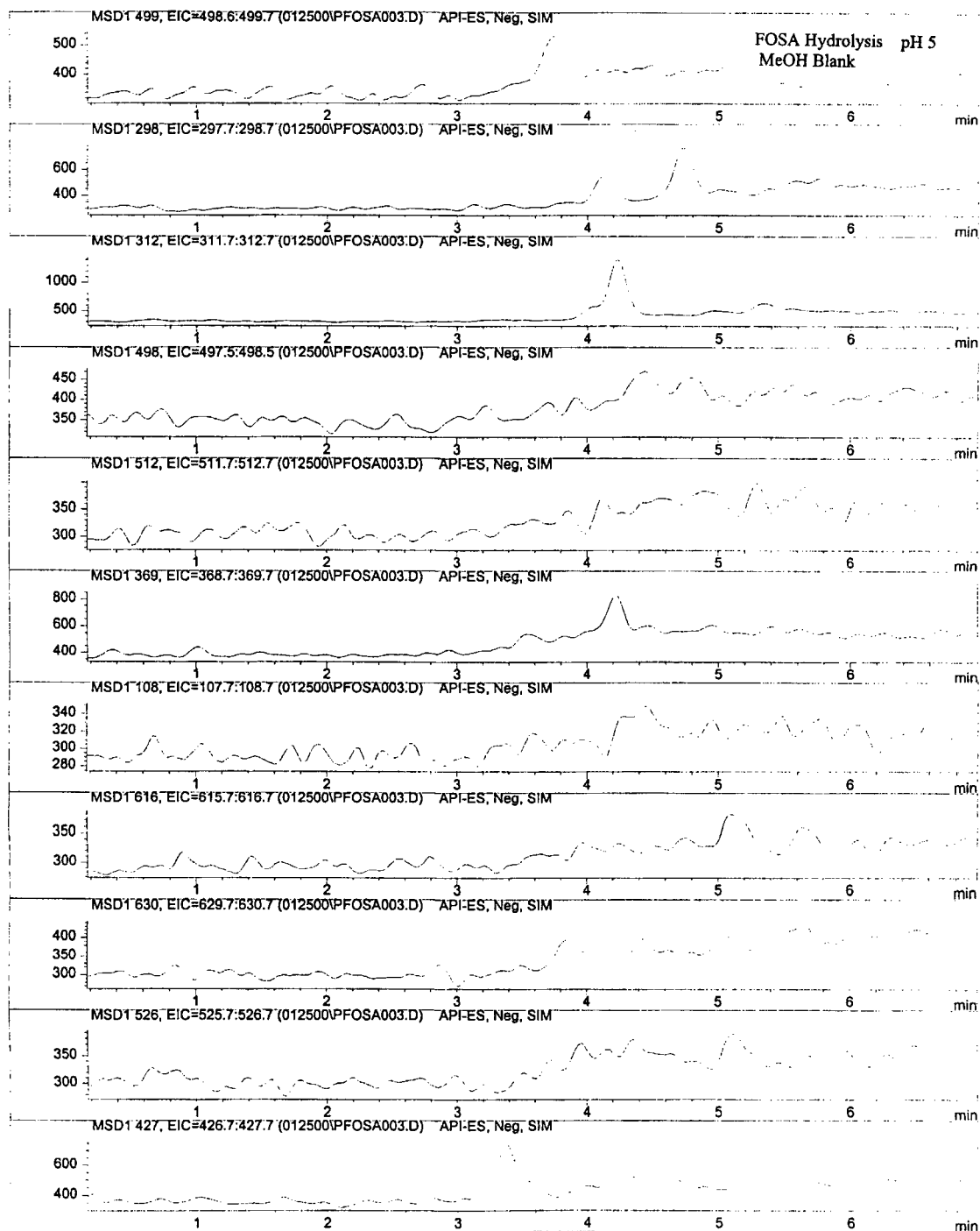
Data4 9/26/00 1:11:40 PM AES/ALS

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Batch Run # 3 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA003.D

Sample Name: MeOH Blank



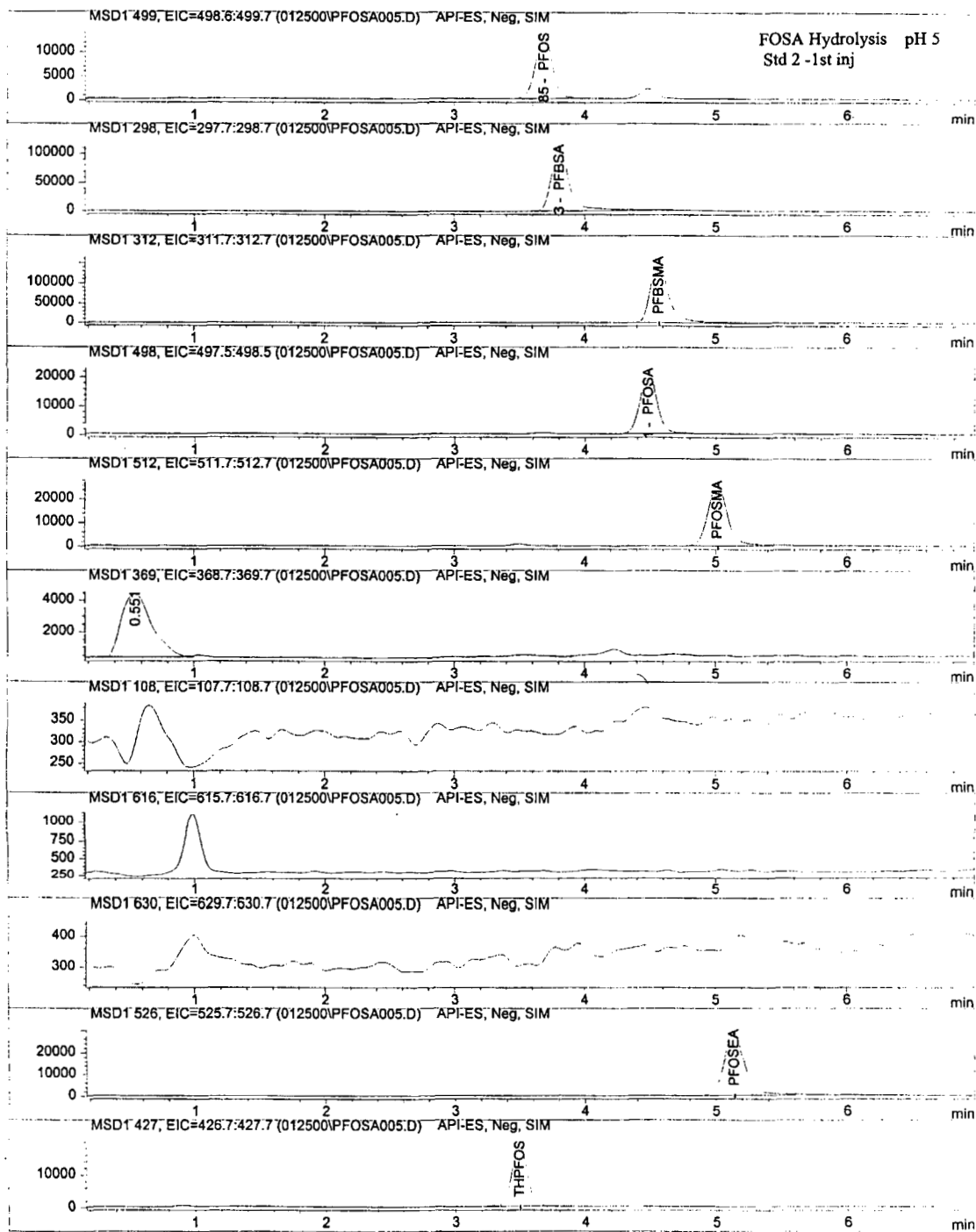
Data4 9/26/00 2:25:05 PM AES/ALS

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Batch Run # 5 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA005.D

Sample Name: 99039-137-02



Data4 9/26/00 2:25:24 PM AES/ALS

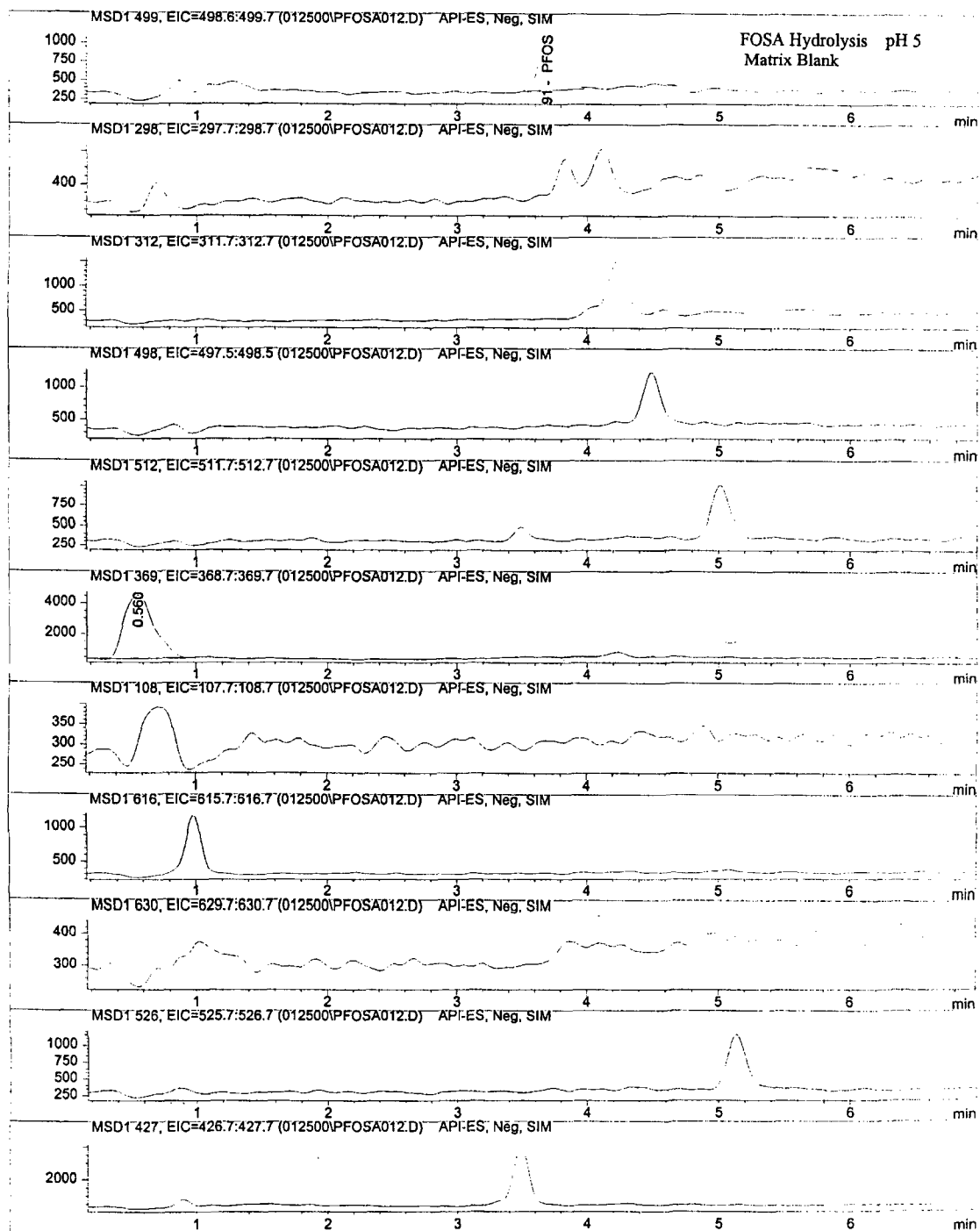
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Batch Run # 12 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA012.D

Sample Name: 081399-BLK-5.0



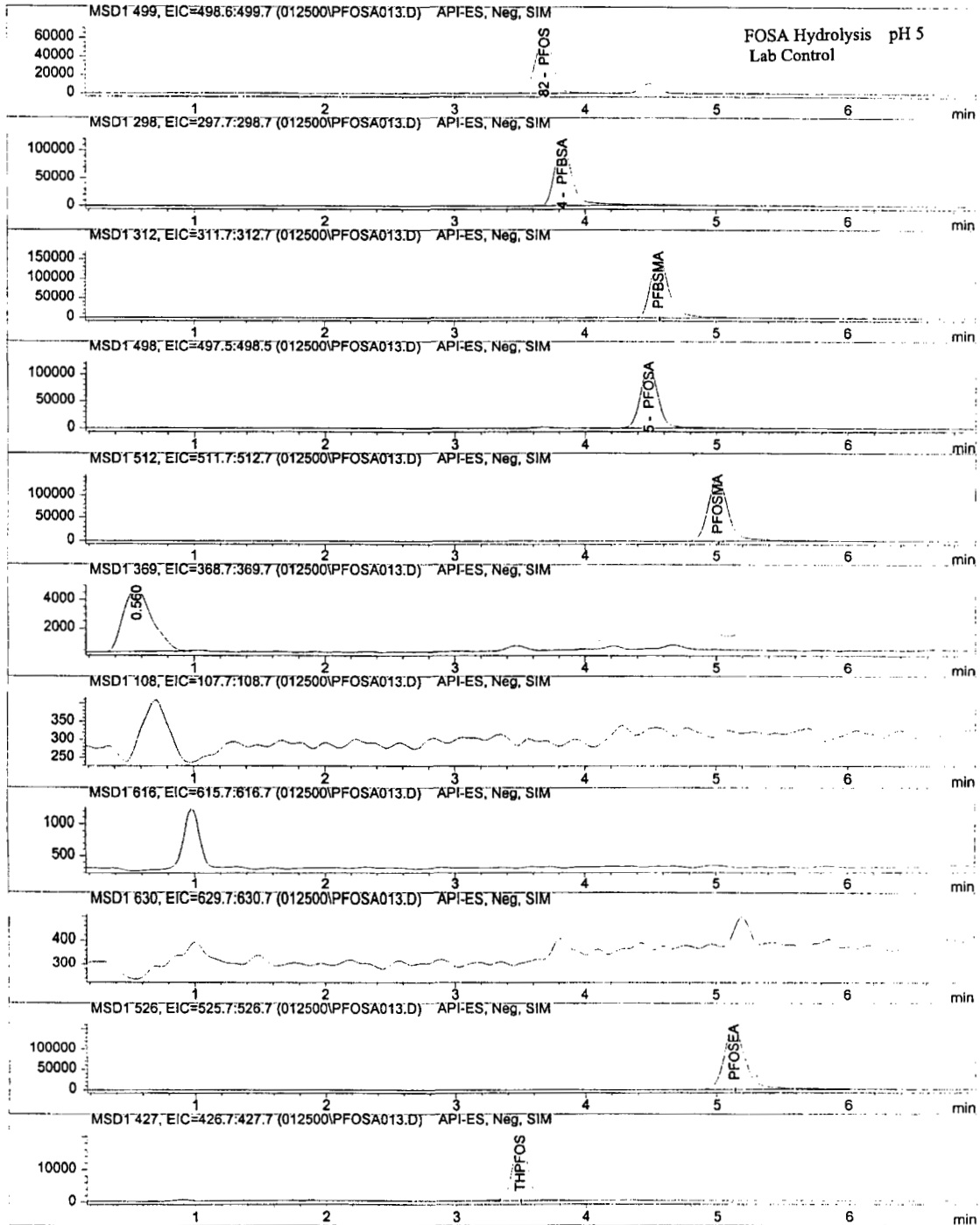
Data4 9/26/00 2:26:27 PM AES/ALS

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Batch Run # 13 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA013.D

Sample Name: 081399-LCS-5.0



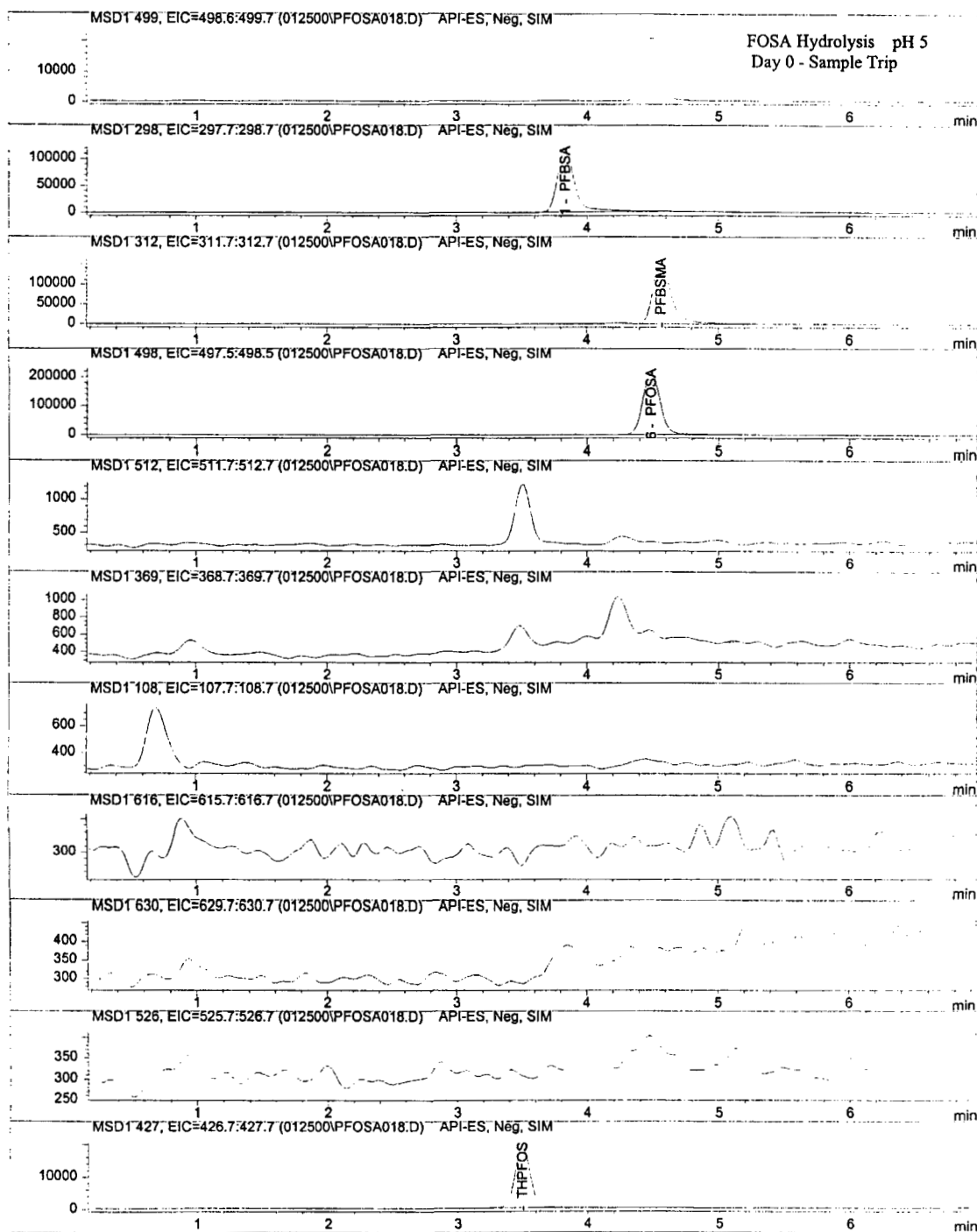
Data4 9/26/00 2:26:35 PM AES/ALS

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Batch Run # 18 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA018.D

Sample Name: 80399PFOSA-011



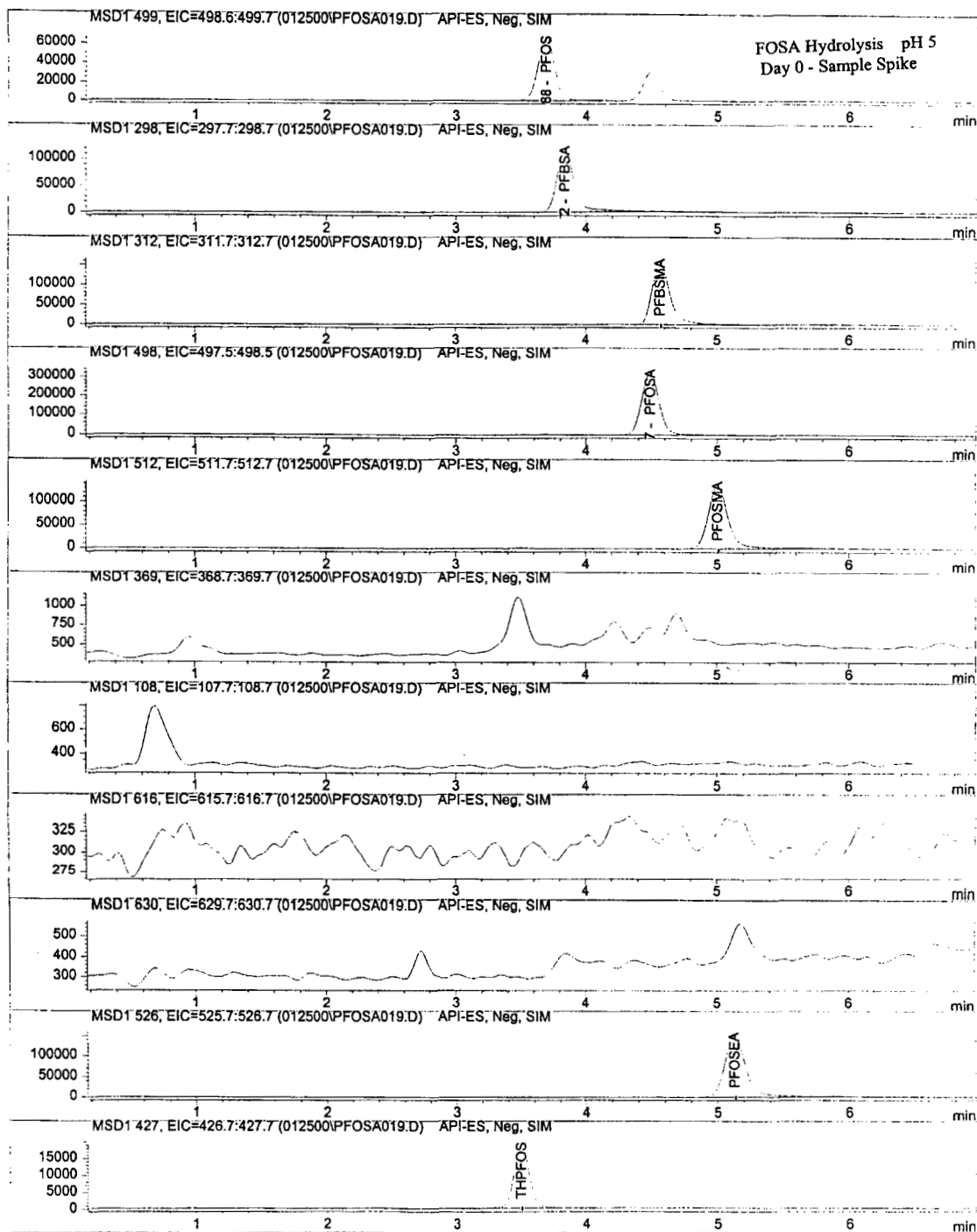
Data4 9/26/00 2:27:21 PM AES/ALS

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Batch Run # 19 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA019.D

Sample Name: 80399PFOSA-012



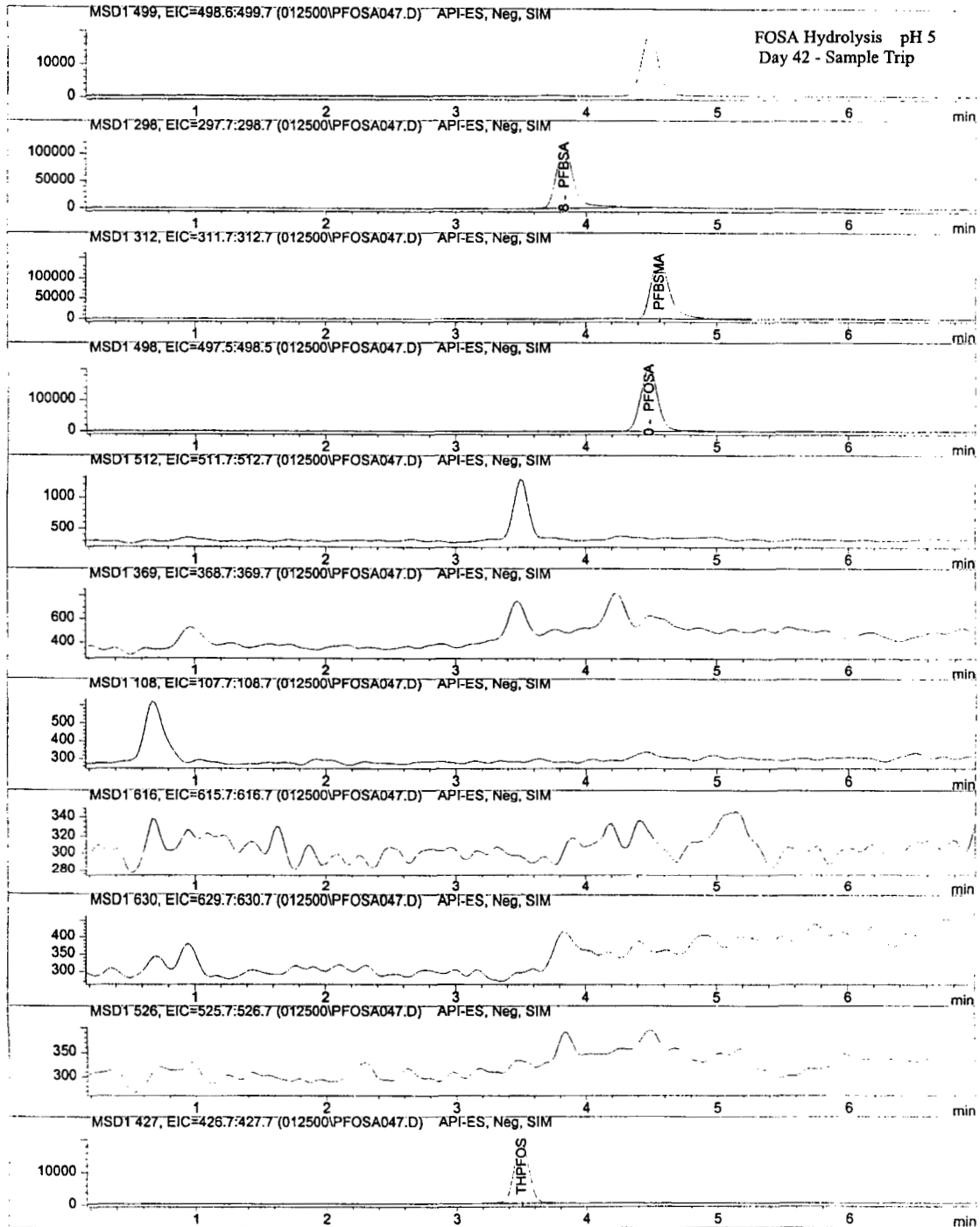
Data4 9/26/00 2:27:29 PM AES/ALS

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Batch Run # 47 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA047.D

Sample Name: 80399PFOSA-155



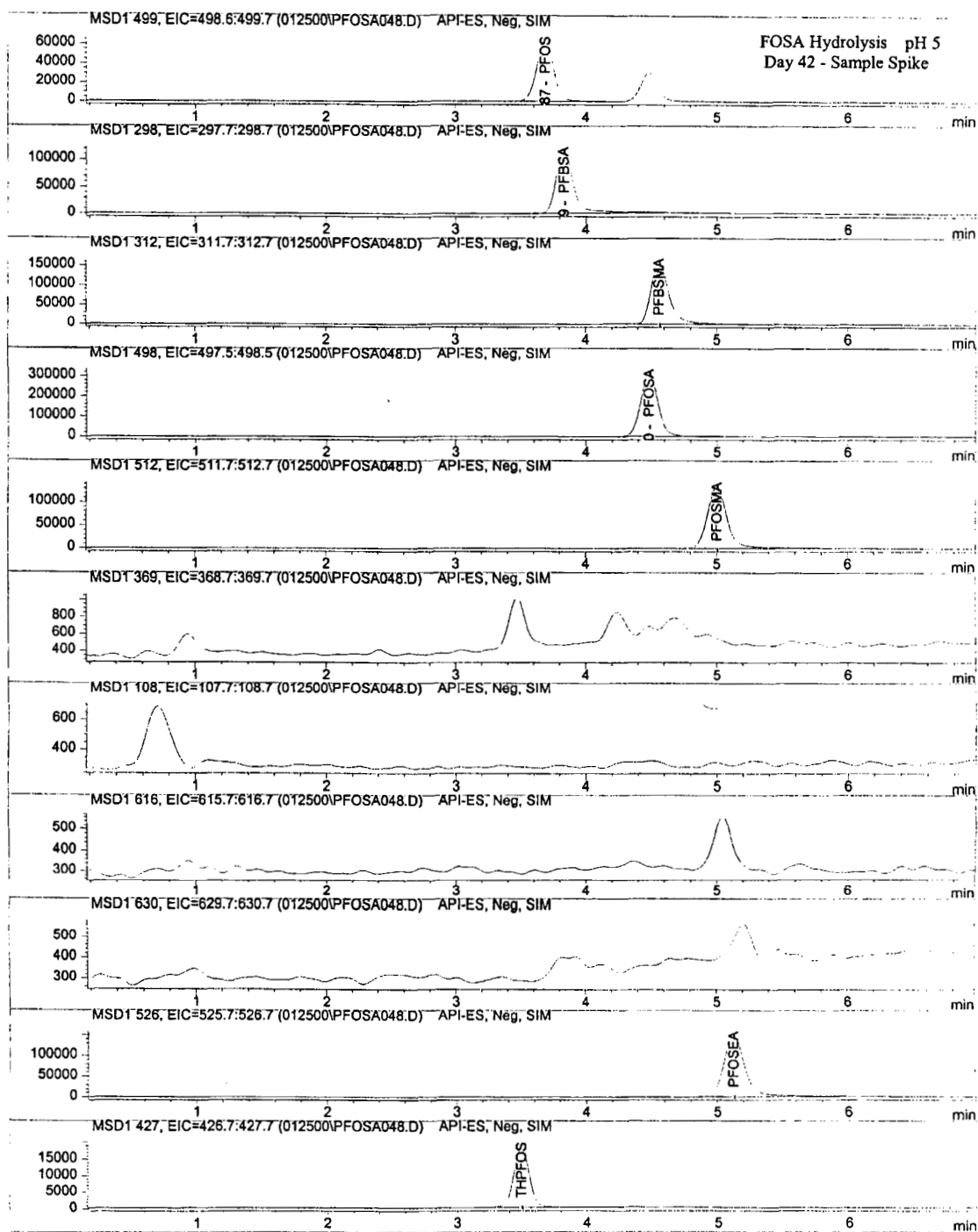
Data4 9/26/00 2:31:39 PM AES/ALS

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Batch Run # 48 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA048.D

Sample Name: 80399PFOSA-156



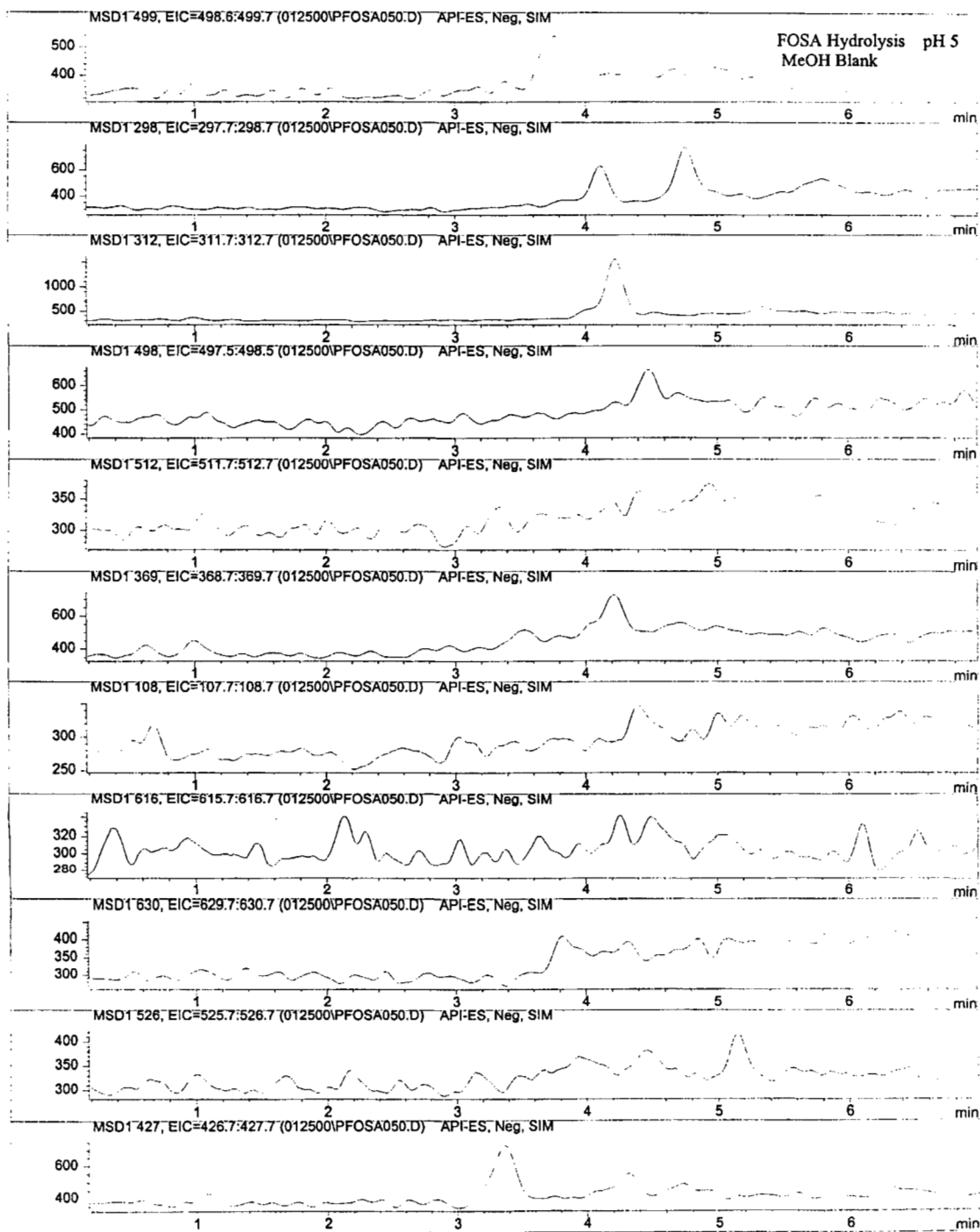
Data4 9/26/00 2:31:47 PM AES/ALS

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Batch Run # 50 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA050.D

Sample Name: MeOH Blank



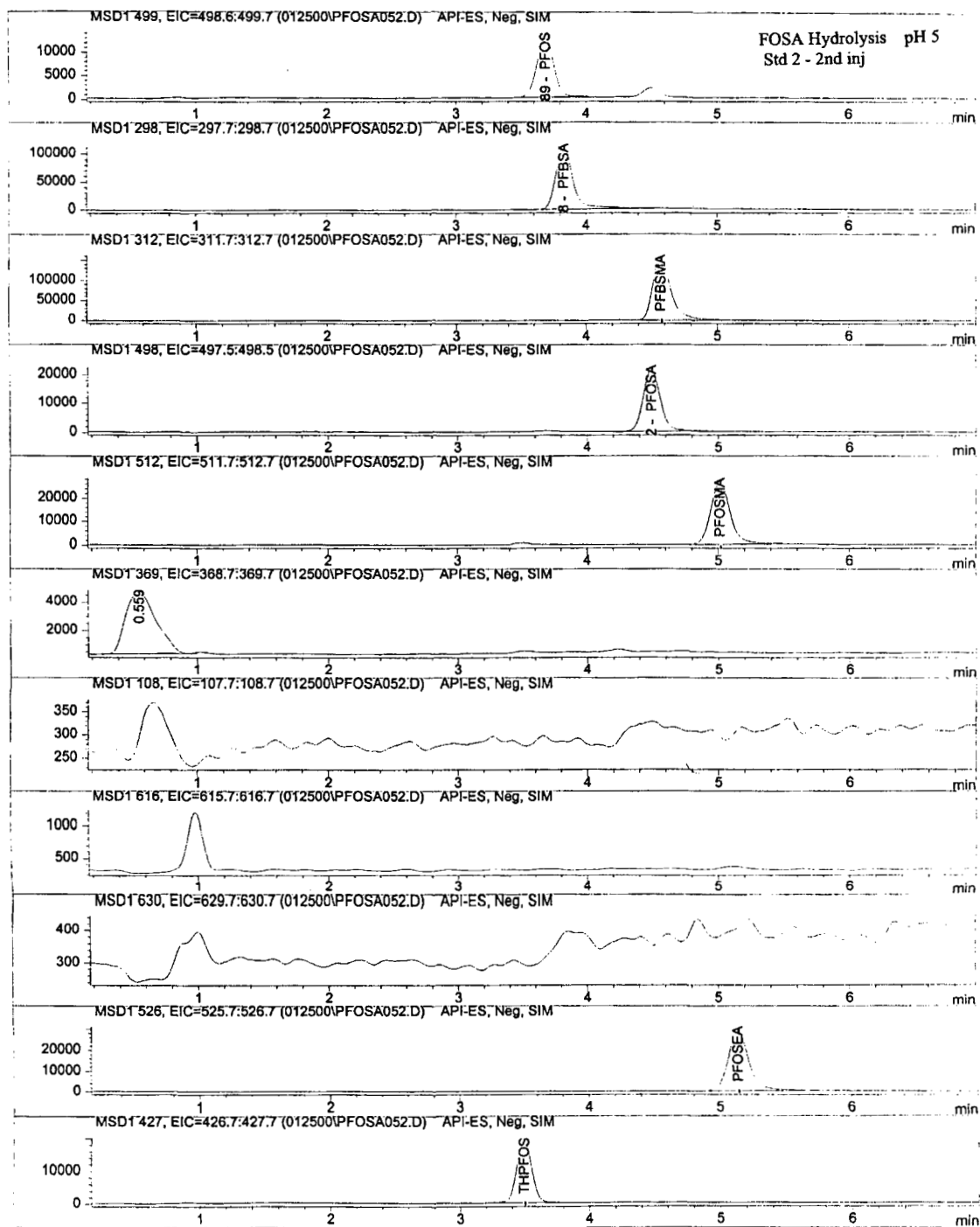
Data4 9/26/00 2:32:06 PM AES/ALS

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Batch Run # 52 of 56

Data File C:\HPCHEM\1\DATA\012500\PFOSA052.D

Sample Name: 99039-137-02



Data4 9/26/00 2:32:25 PM AES/ALS

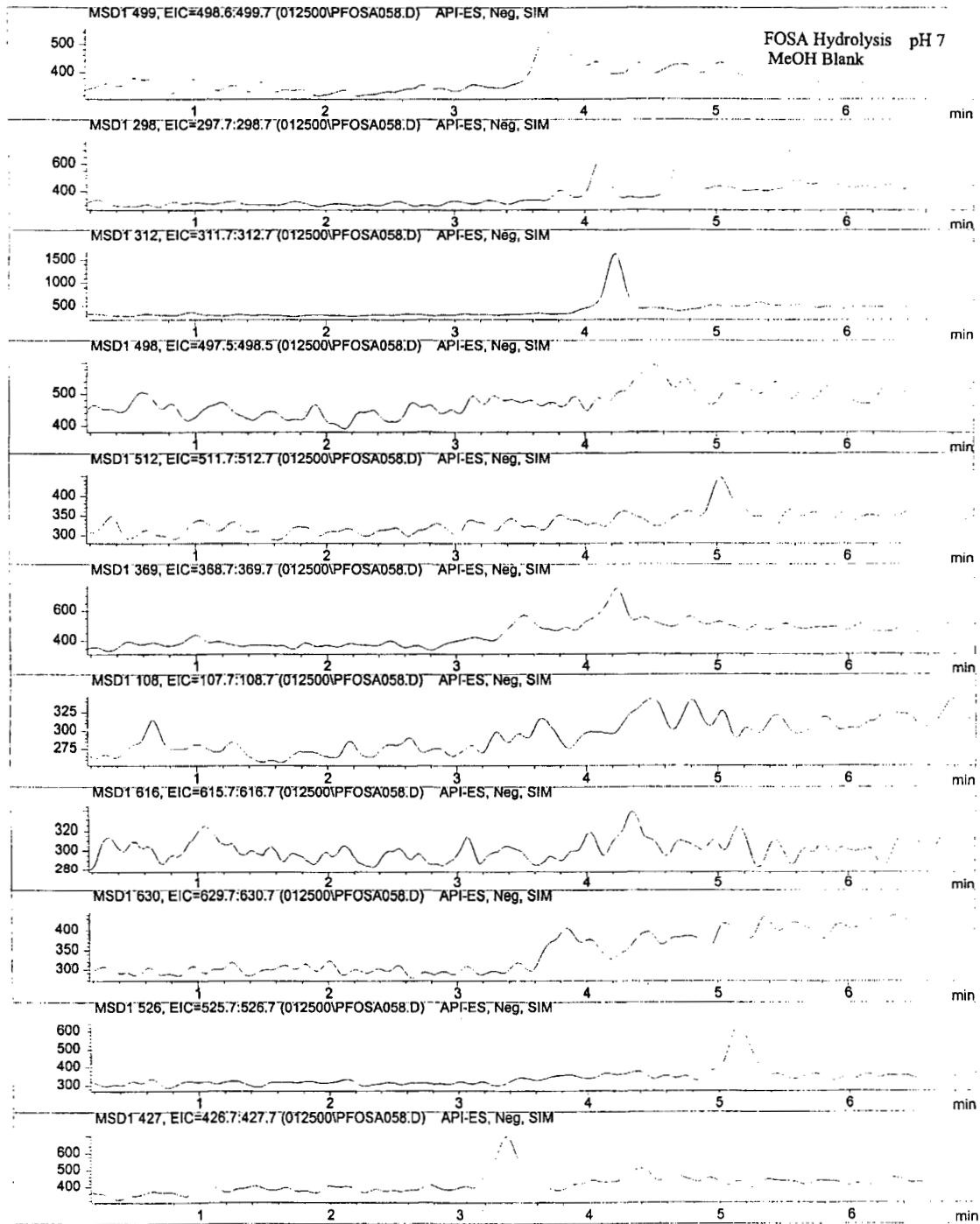
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Batch Run # 2 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA058.D

Sample Name: MeOH Blank



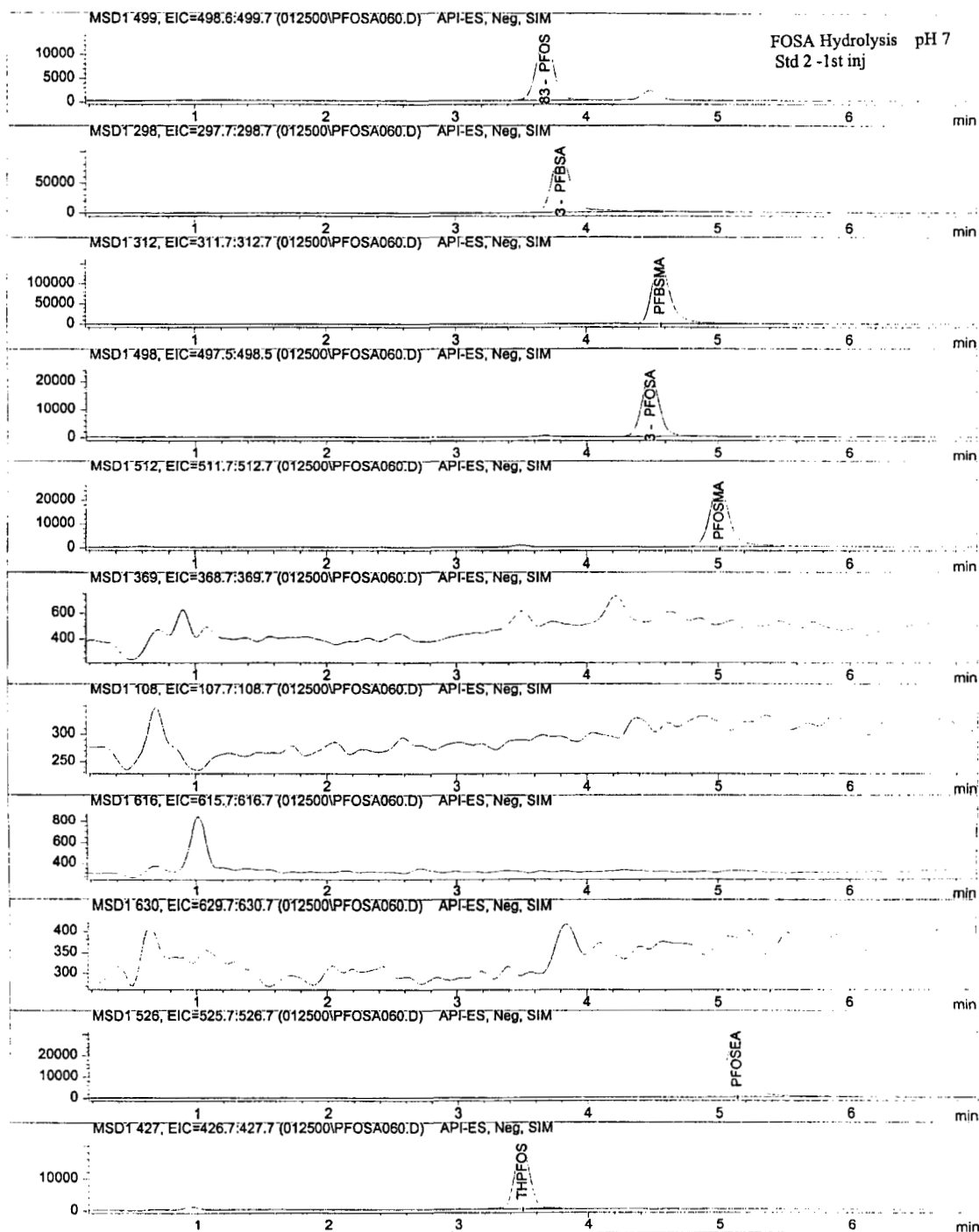
Data4 9/27/00 11:10:45 AM ABS/ALS

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Batch Run # 4 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA060.D

Sample Name: 99039-138-02



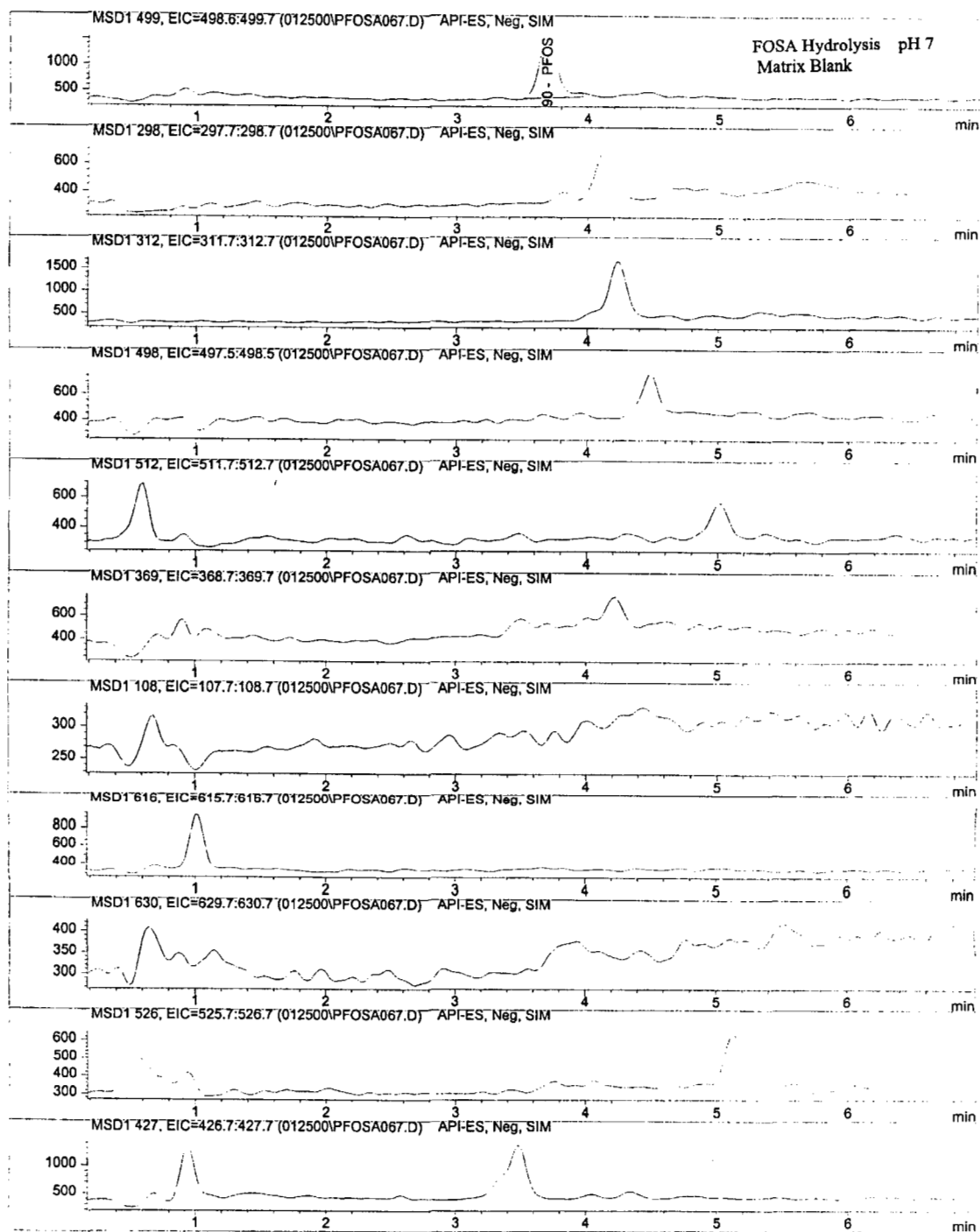
Data4 9/27/00 11:11:04 AM AES/ALS

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Batch Run # 11 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA067.D

Sample Name: 081399-BLK-7.0



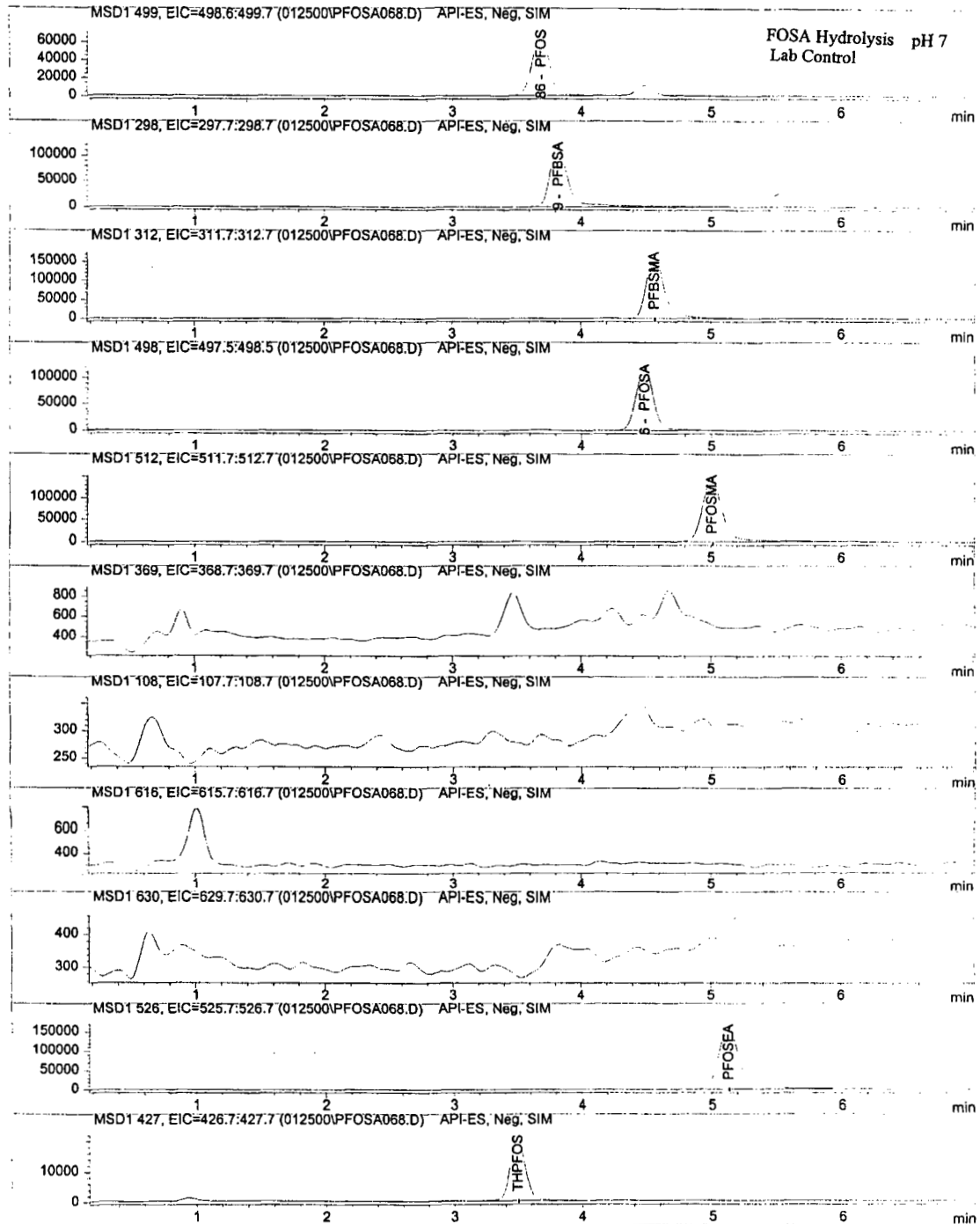
Data4 9/27/00 11:12:07 AM AES/ALS

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Batch Run # 12 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA068.D

Sample Name: 081399-LCS-7.0



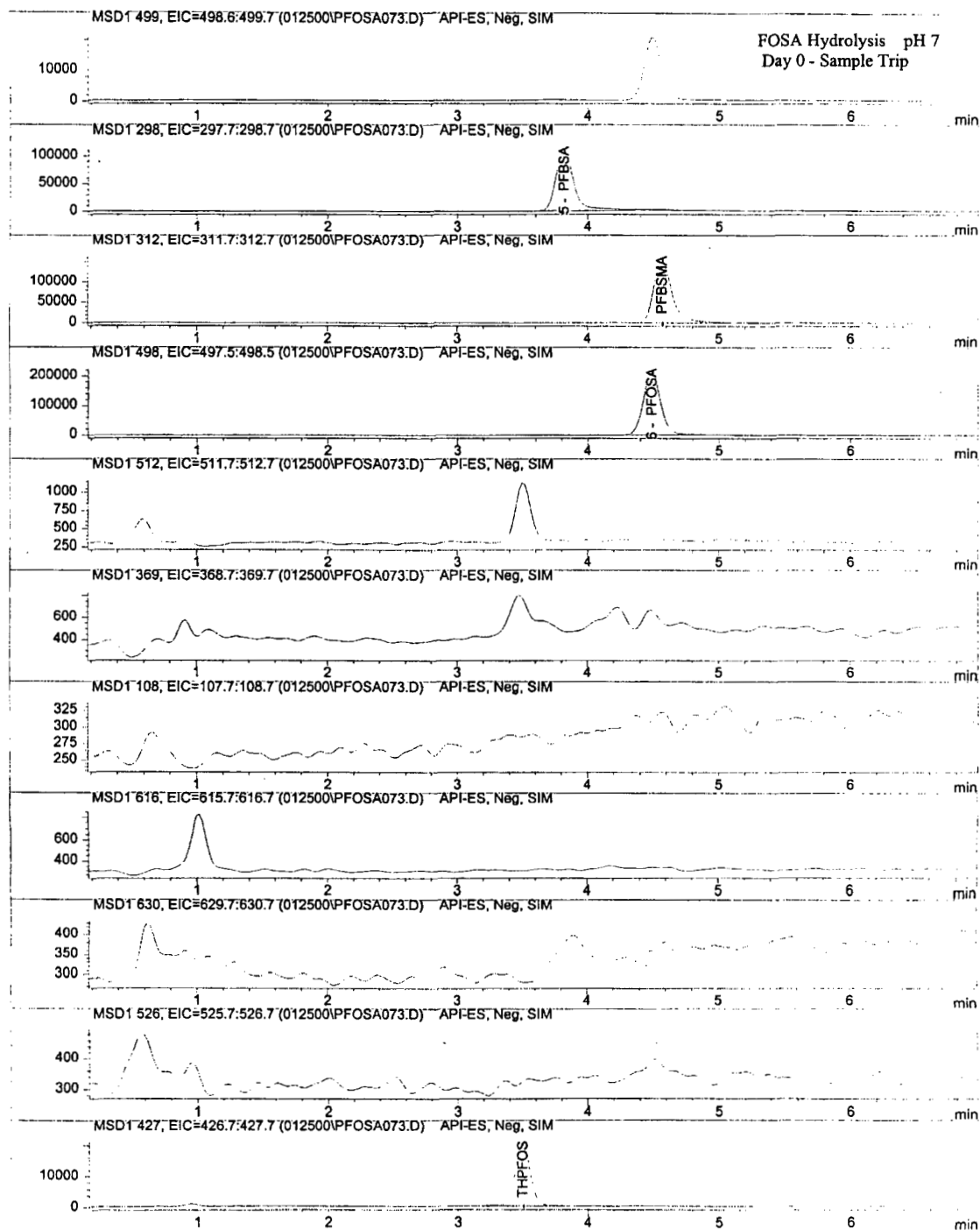
Data4 9/27/00 11:12:16 AM AES/ALS

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Batch Run # 17 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA073.D

Sample Name: 80399PFOSA-015



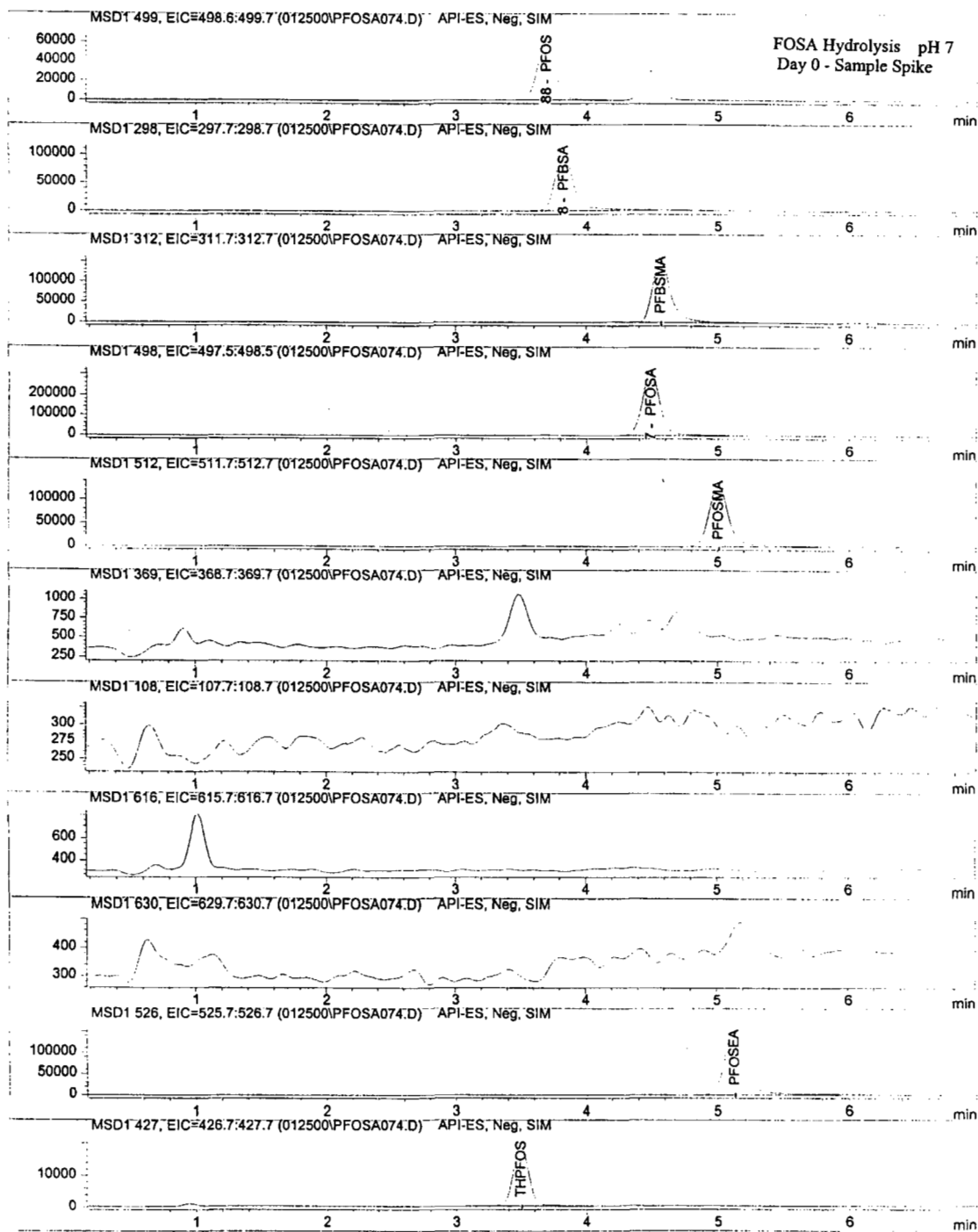
Data4 9/27/00 11:13:02 AM AES/ALS

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Batch Run # 18 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA074.D

Sample Name: 80399PFOSA-016



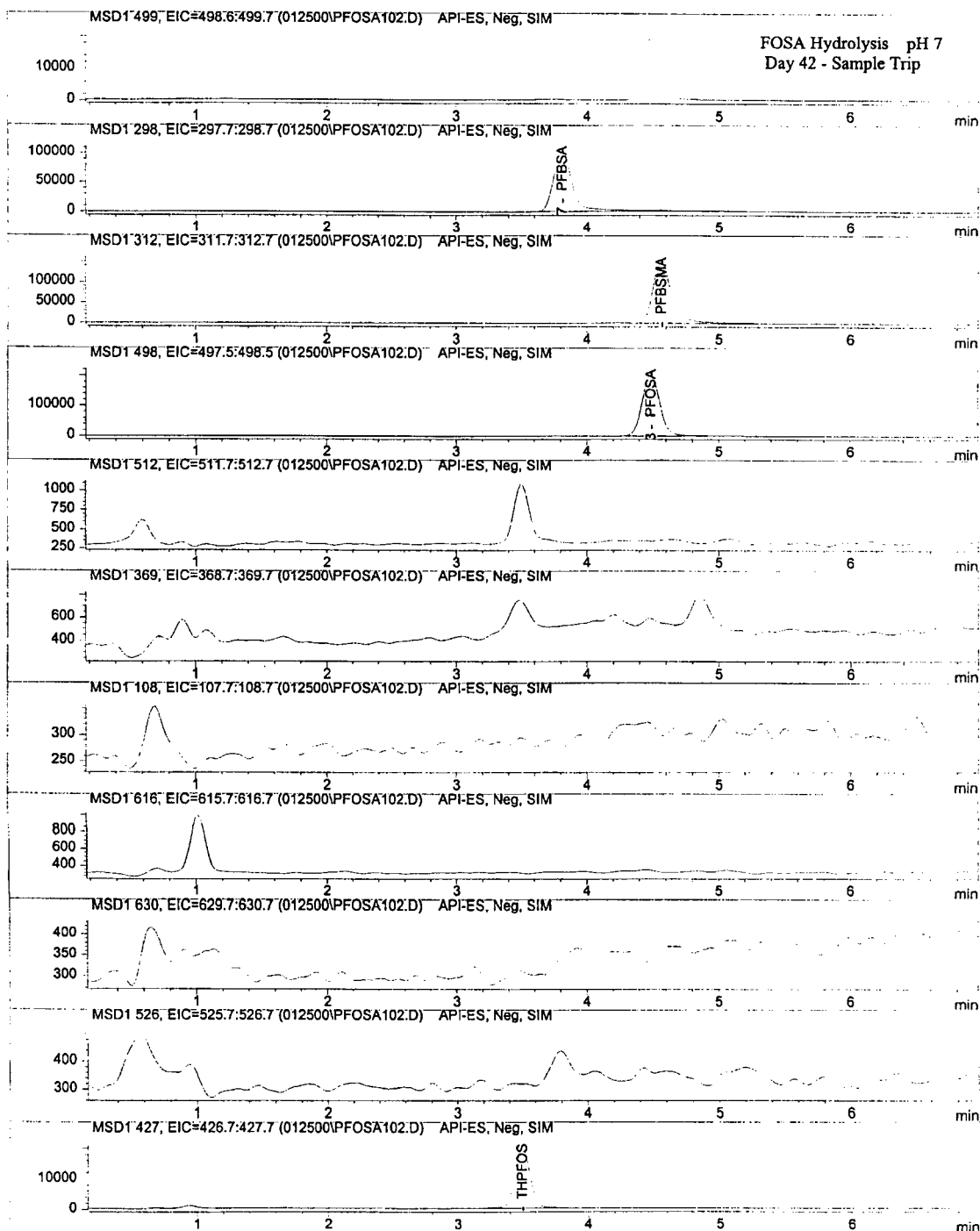
Data4 9/27/00 11:13:10 AM AES/ALS

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Batch Run # 46 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA102.D

Sample Name: 80399PFOSA-159



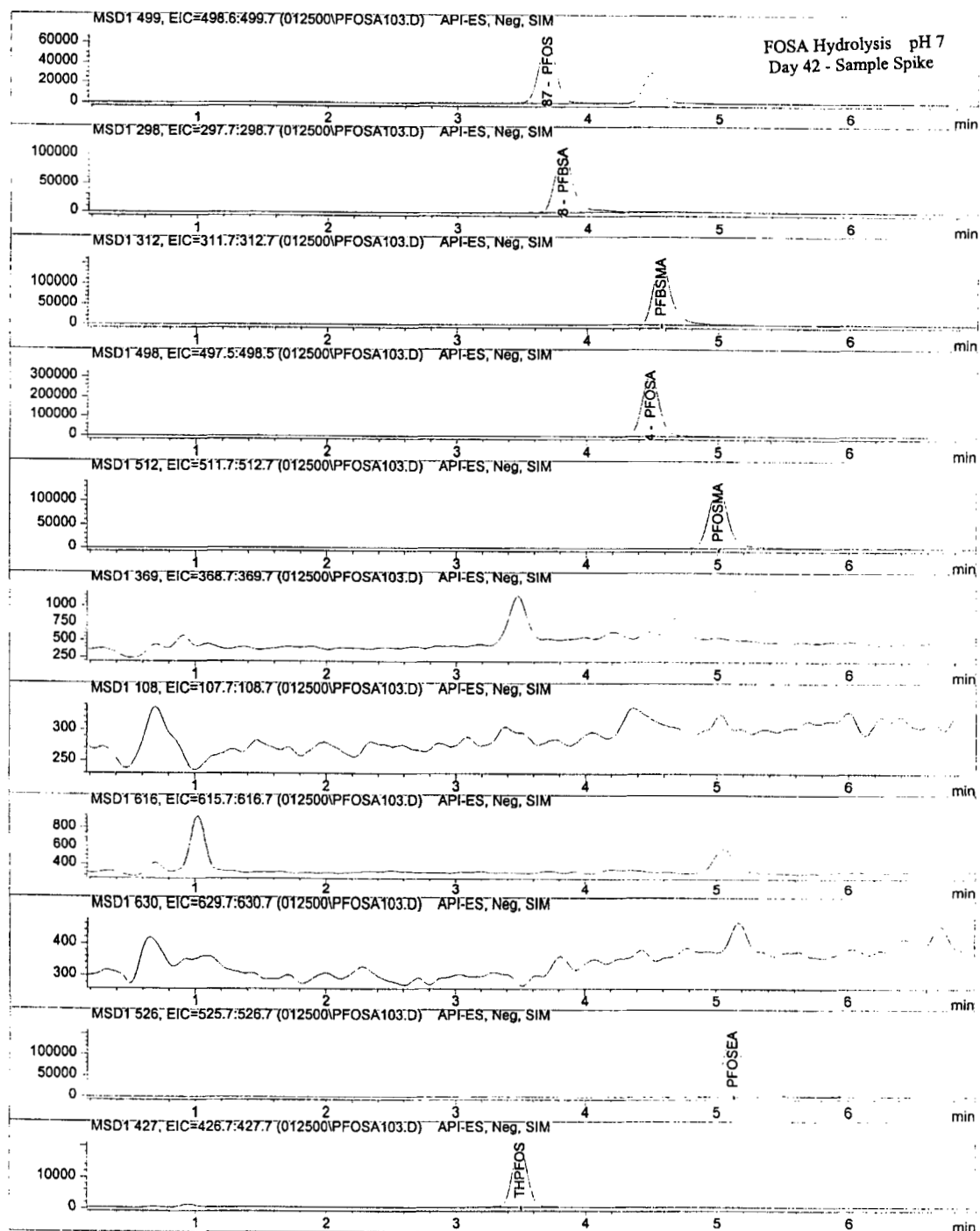
Data4 9/27/00 11:17:11 AM AES/ALS

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Batch Run # 47 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA103.D

Sample Name: 80399PFOSA-160



Data4 9/27/00 11:17:20 AM AES/ALS

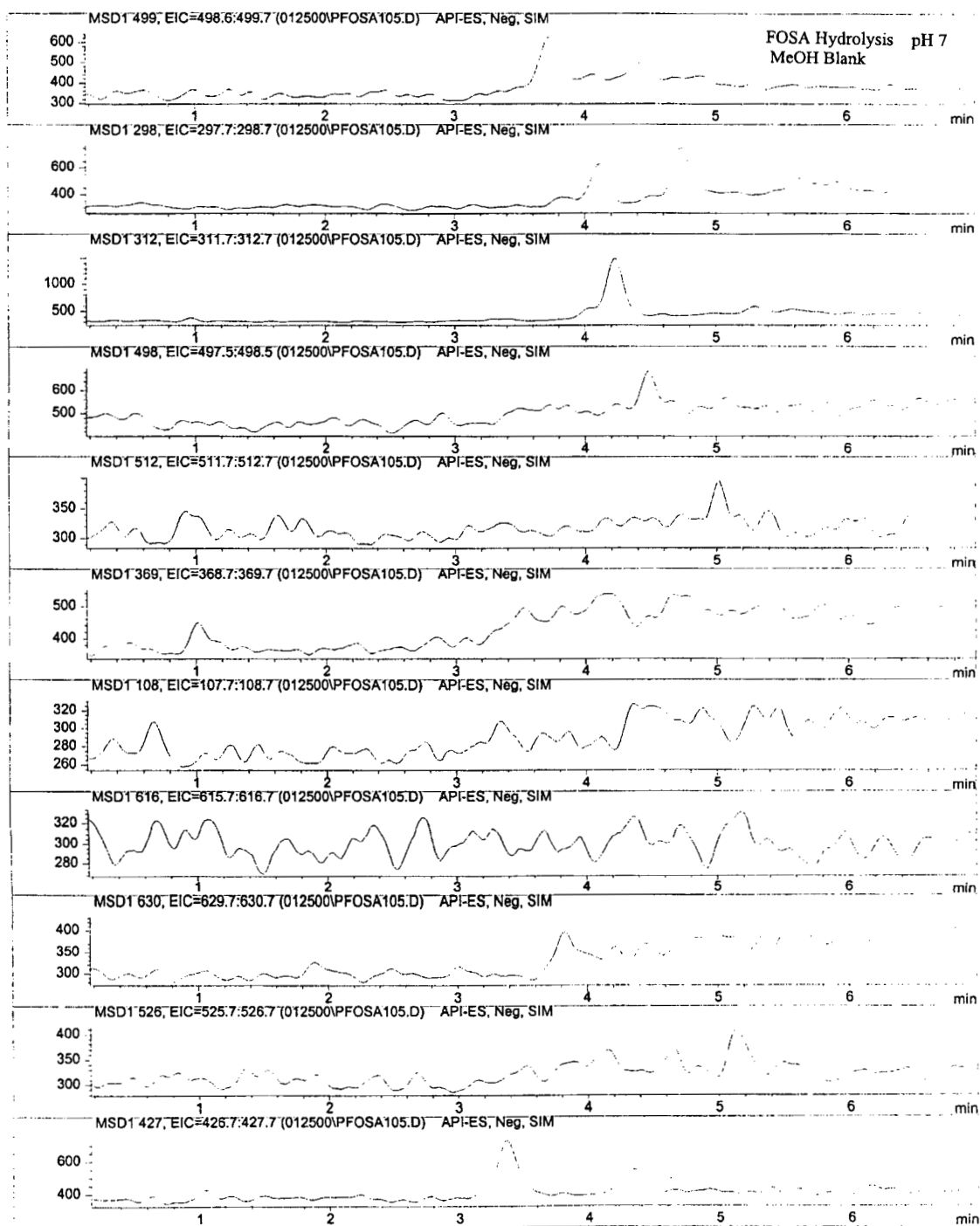
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Batch Run # 49 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA105.D

Sample Name: MeOH Blank



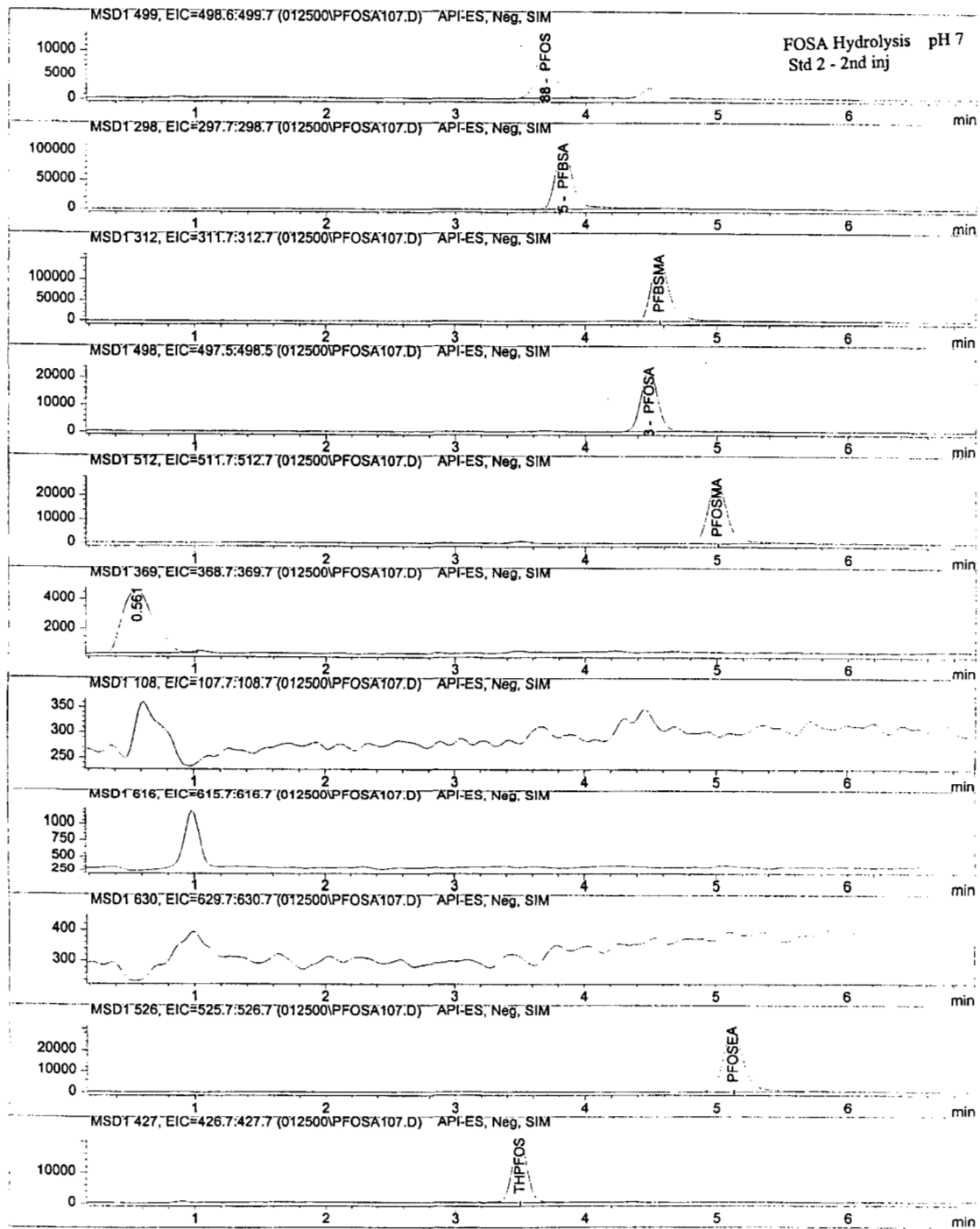
Data4 9/27/00 11:17:38 AM AES/ALS

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Batch Run # 51 of 55

Data File C:\HPCHEM\1\DATA\012500\PFOSA107.D

Sample Name: 99039-138-02



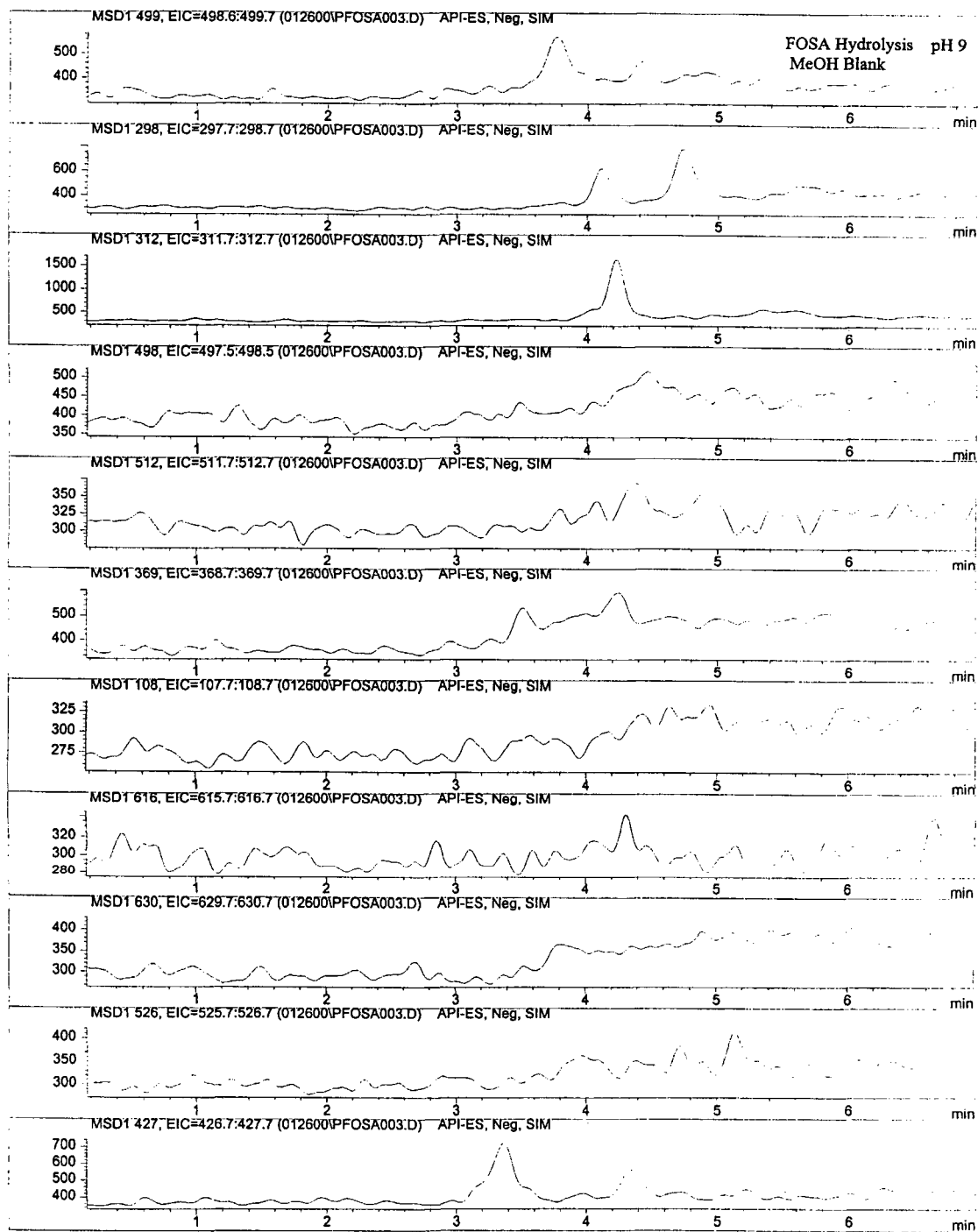
Data4 9/27/00 11:17:57 AM AES/ALS

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Batch Run # 3 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA003.D

Sample Name: MeOH Blank



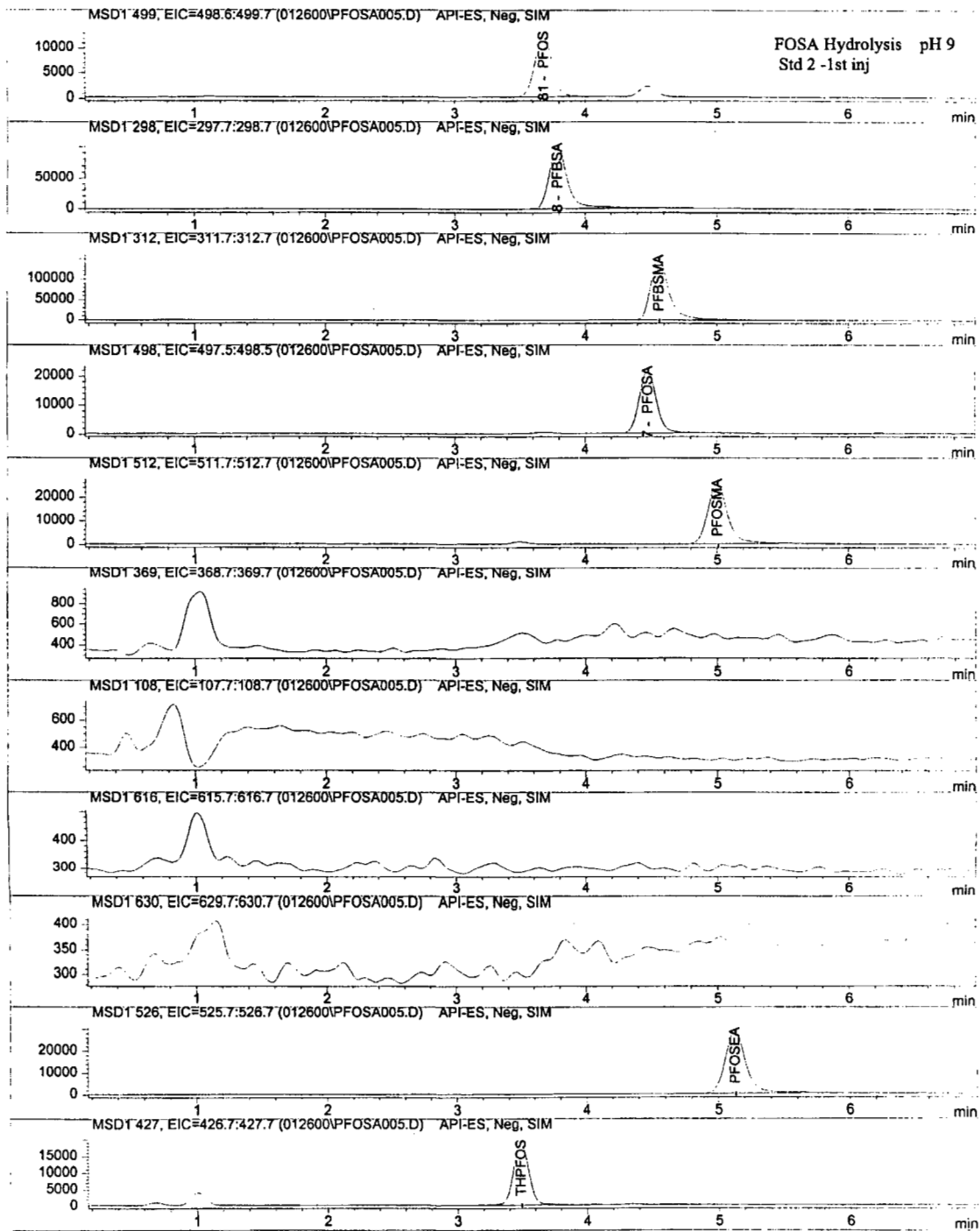
Data4 9/27/00 3:46:28 PM AES/ALS

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Batch Run # 5 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA005.D

Sample Name: 99039-139-02



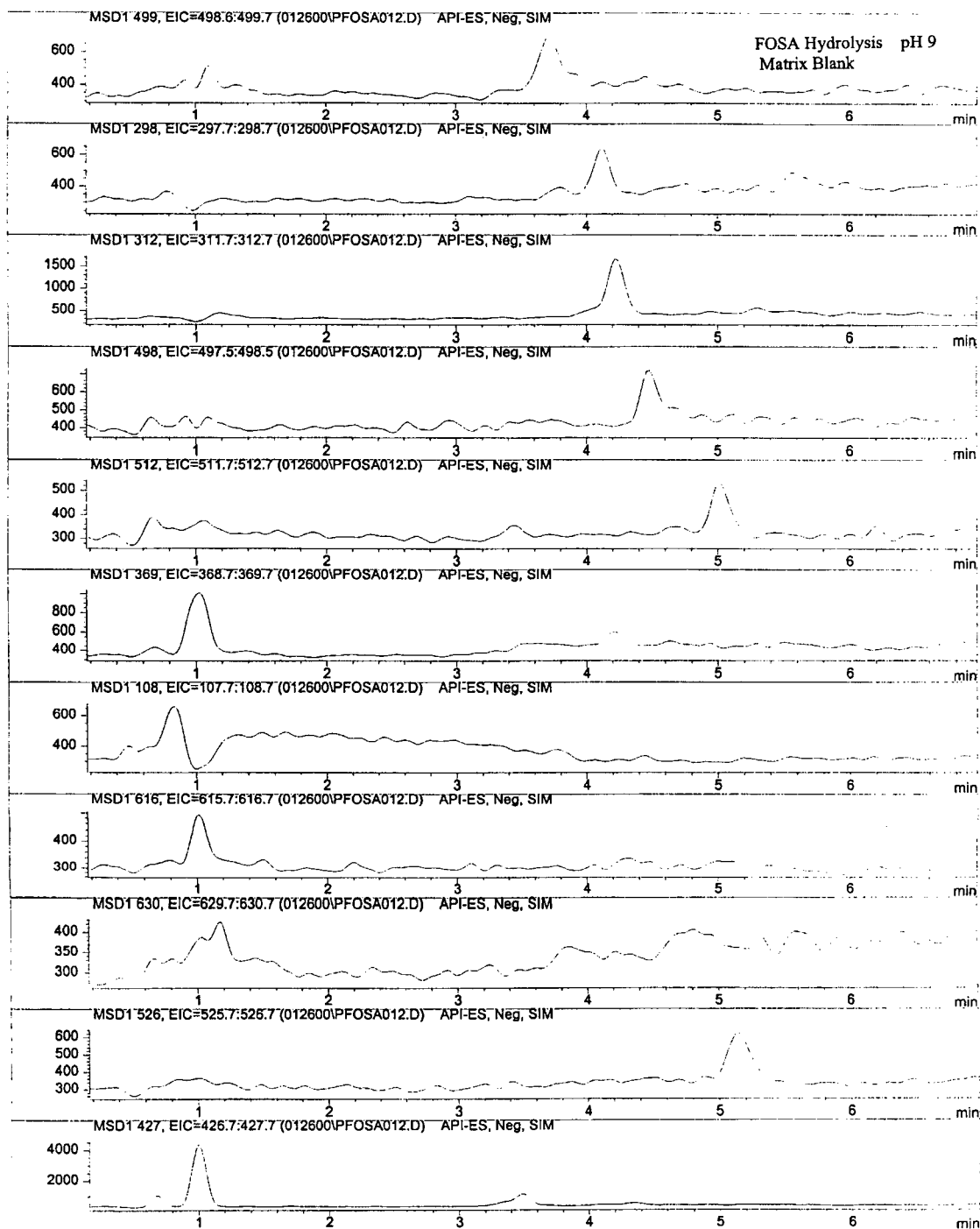
Data4 9/27/00 3:46:47 PM AES/ALS

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Batch Run # 12 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA012.D

Sample Name: 081399-BLK-9.0



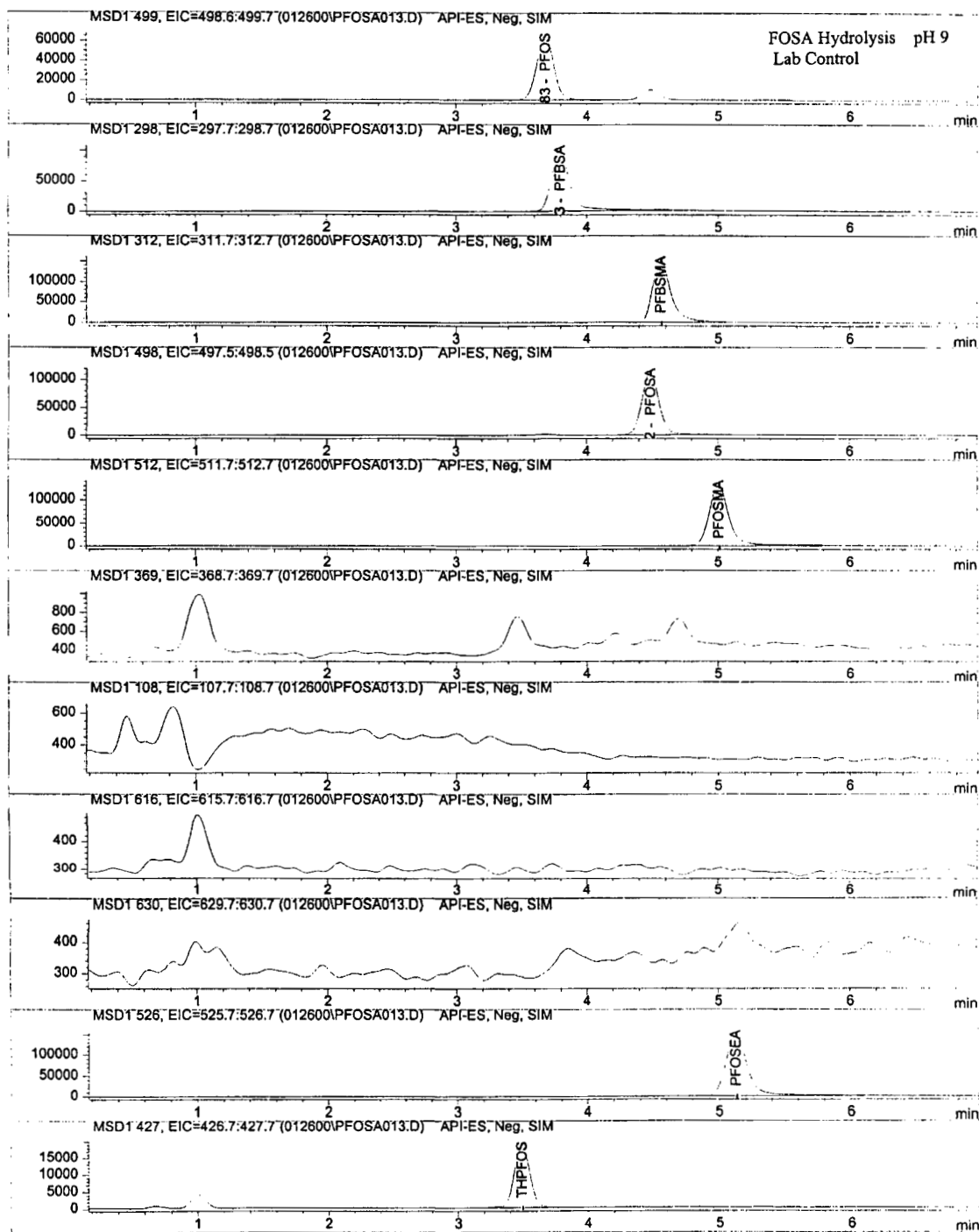
Data4 9/27/00 3:47:50 PM AES/ALS

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Batch Run # 13 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA013.D

Sample Name: 081399-LCS-9.0



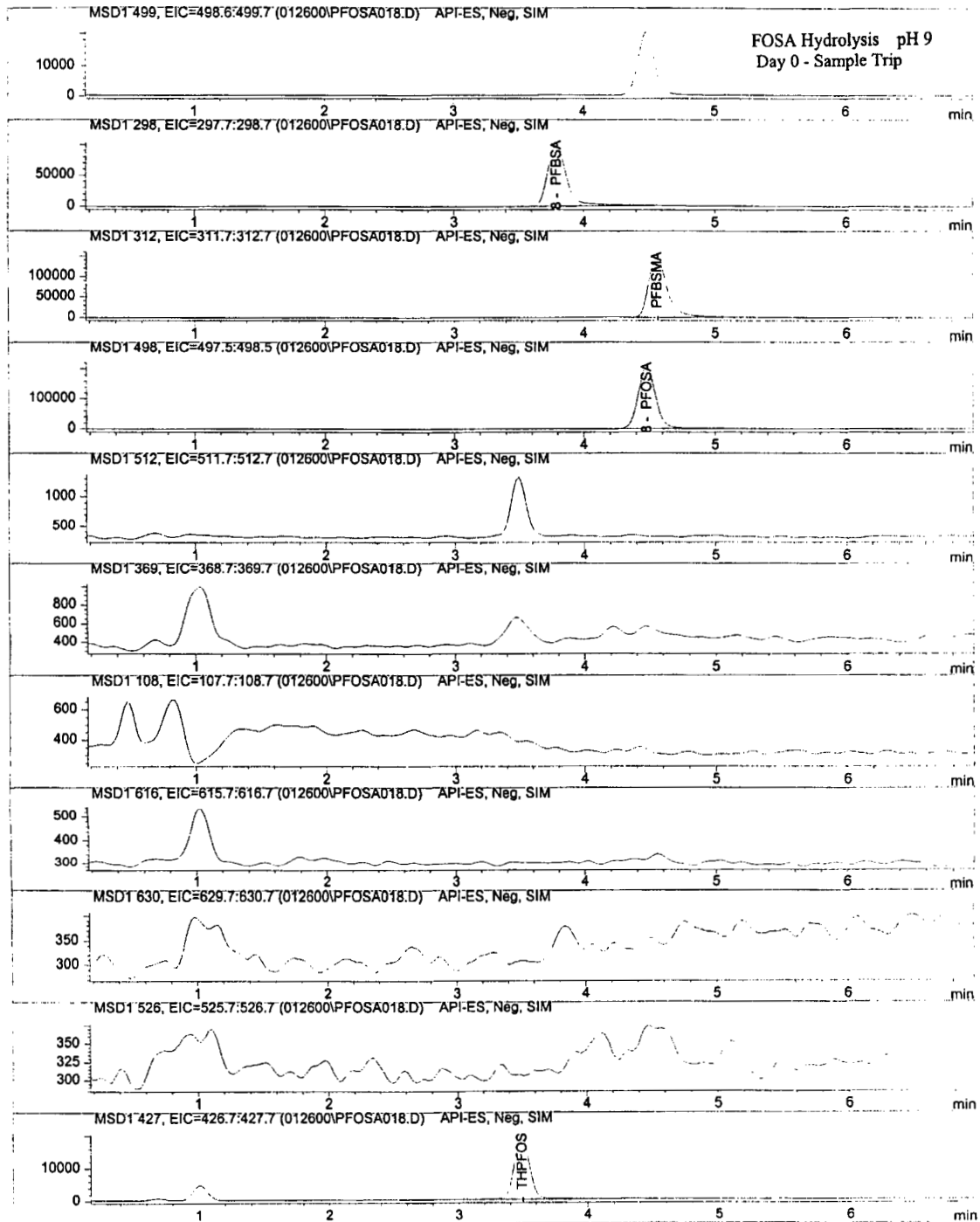
Data4 9/27/00 3:48:01 PM AES/ALS

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Batch Run # 18 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA018.D

Sample Name: 80399PFOSA-019



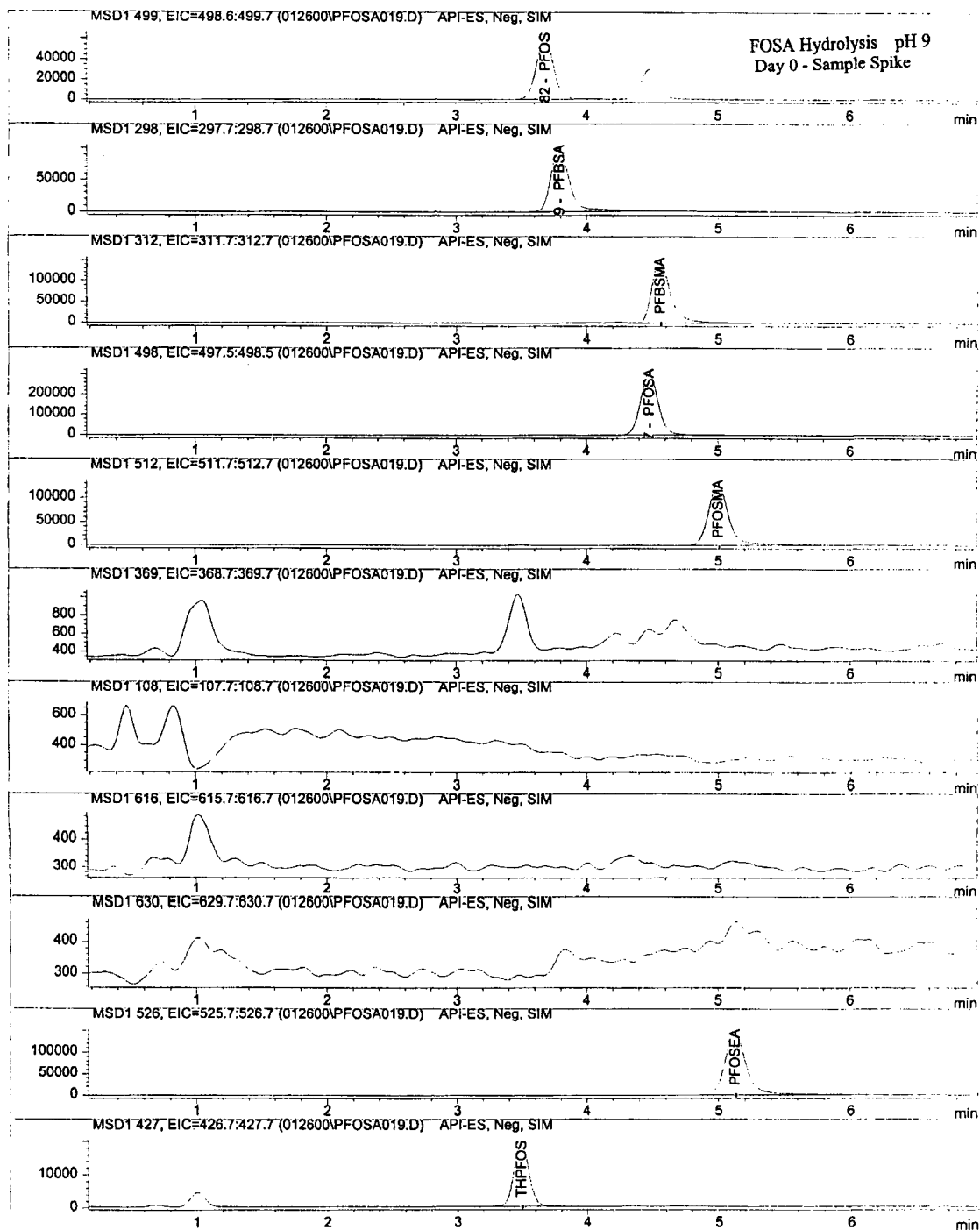
Data4 9/27/00 3:48:47 PM AES/ALS

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Batch Run # 19 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA019.D

Sample Name: 80399PFOSA-020



Data4 9/27/00 3:48:55 PM AES/ALS

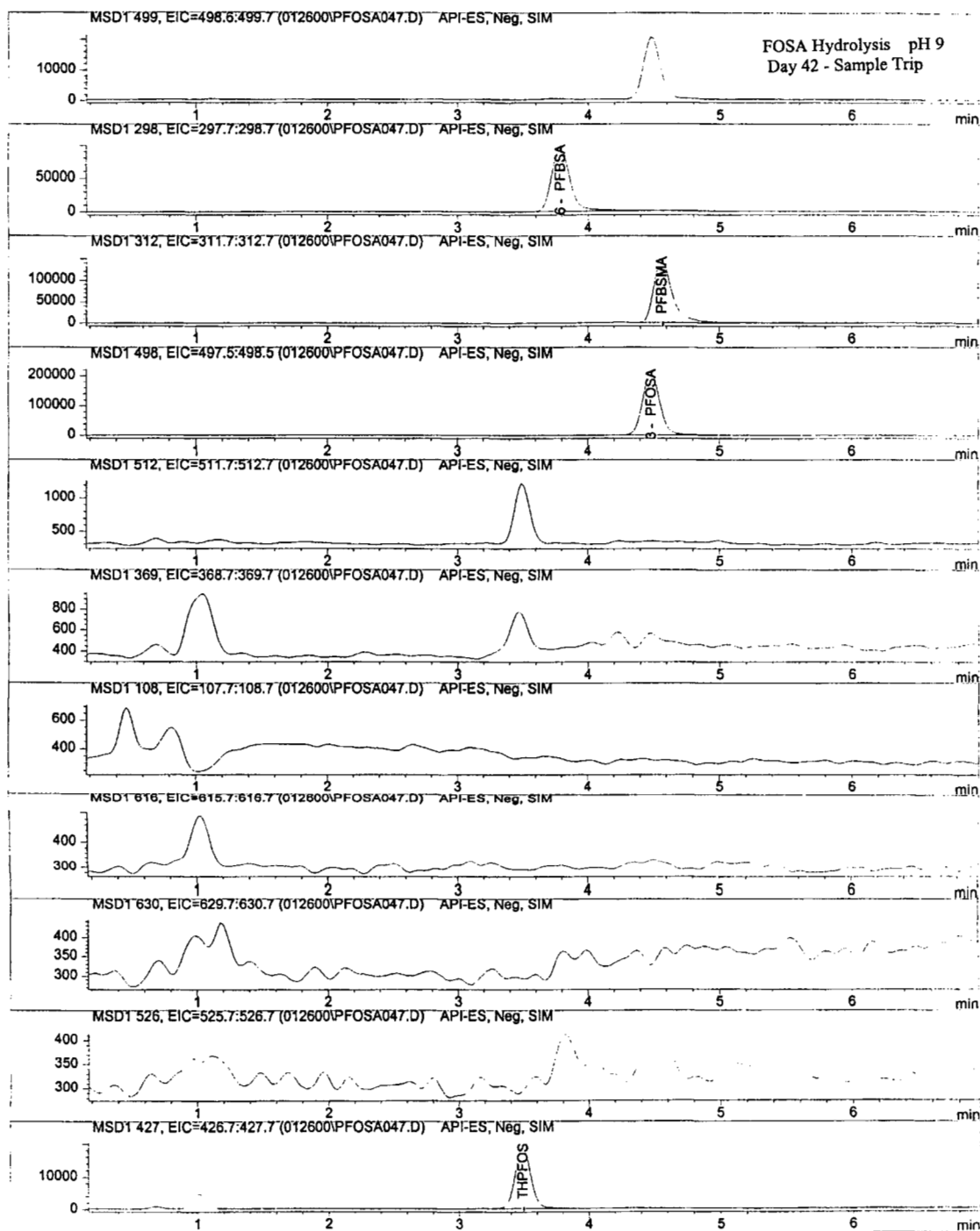
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Batch Run # 47 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA047.D

Sample Name: 80399PFOSA-163



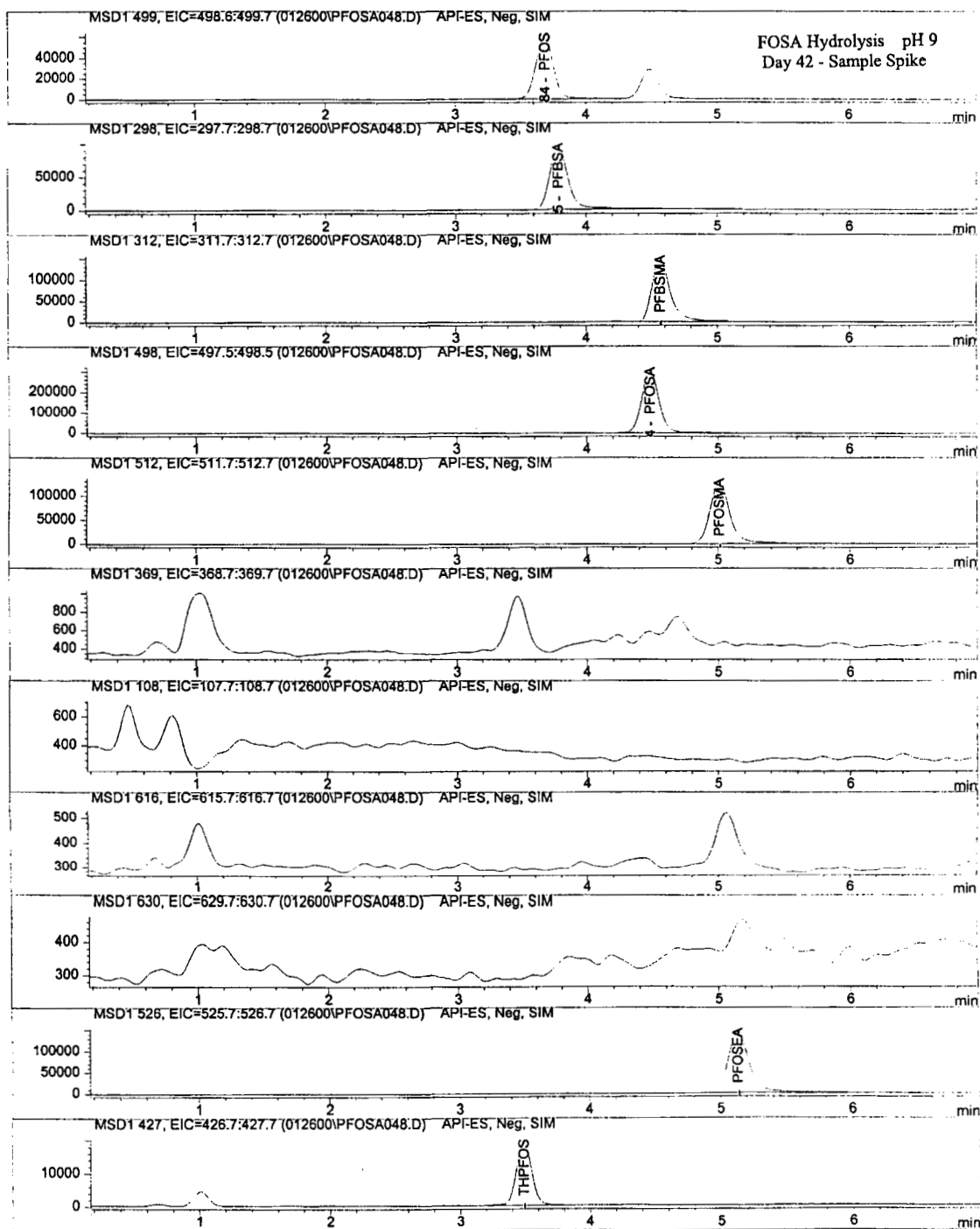
Data4 9/27/00 3:52:59 PM AES/ALS

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Batch Run # 48 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA048.D

Sample Name: 80399PFOSA-164



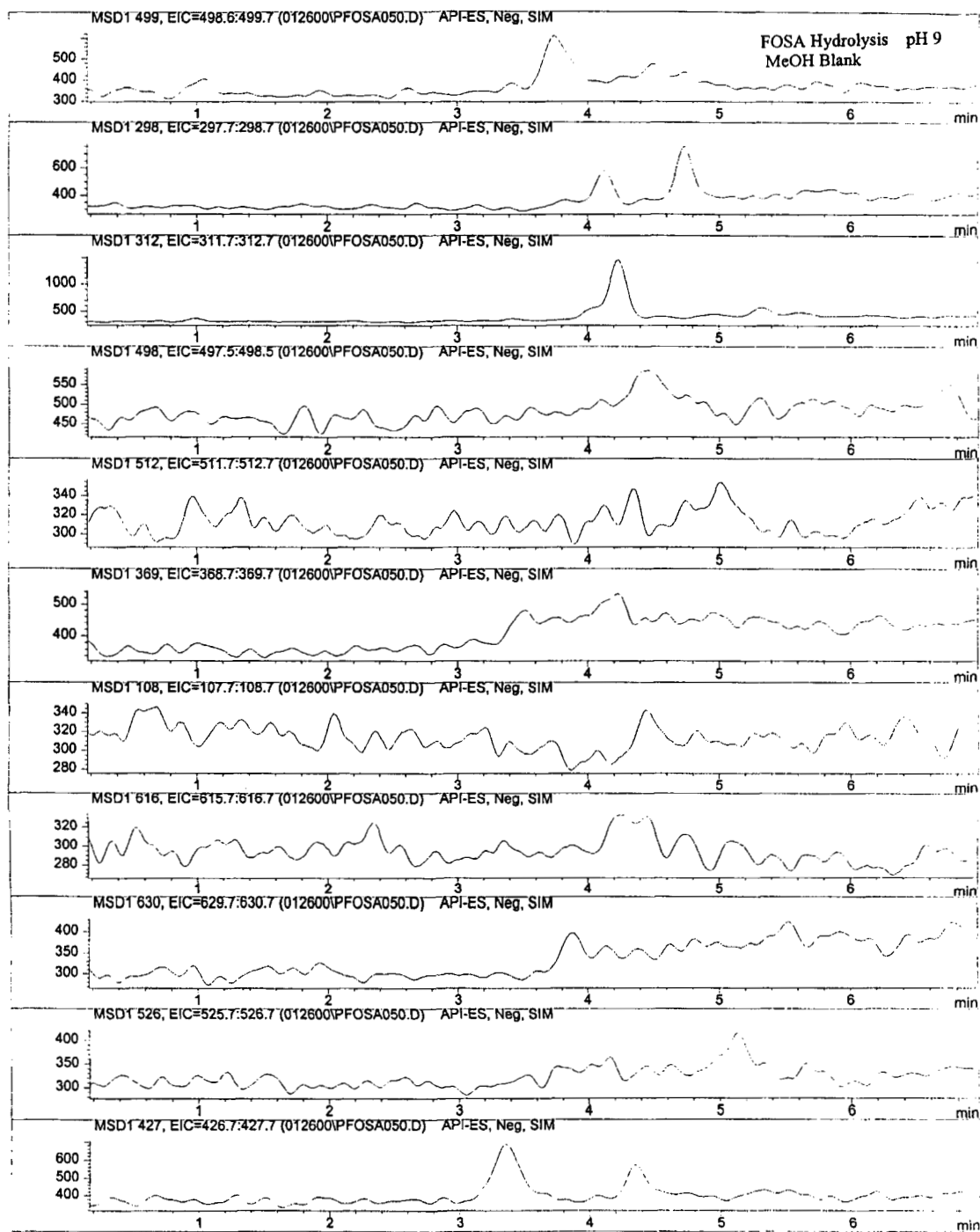
Data4 9/27/00 3:53:08 PM AES/ALS

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Batch Run # 50 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA050.D

Sample Name: MeOH Blank



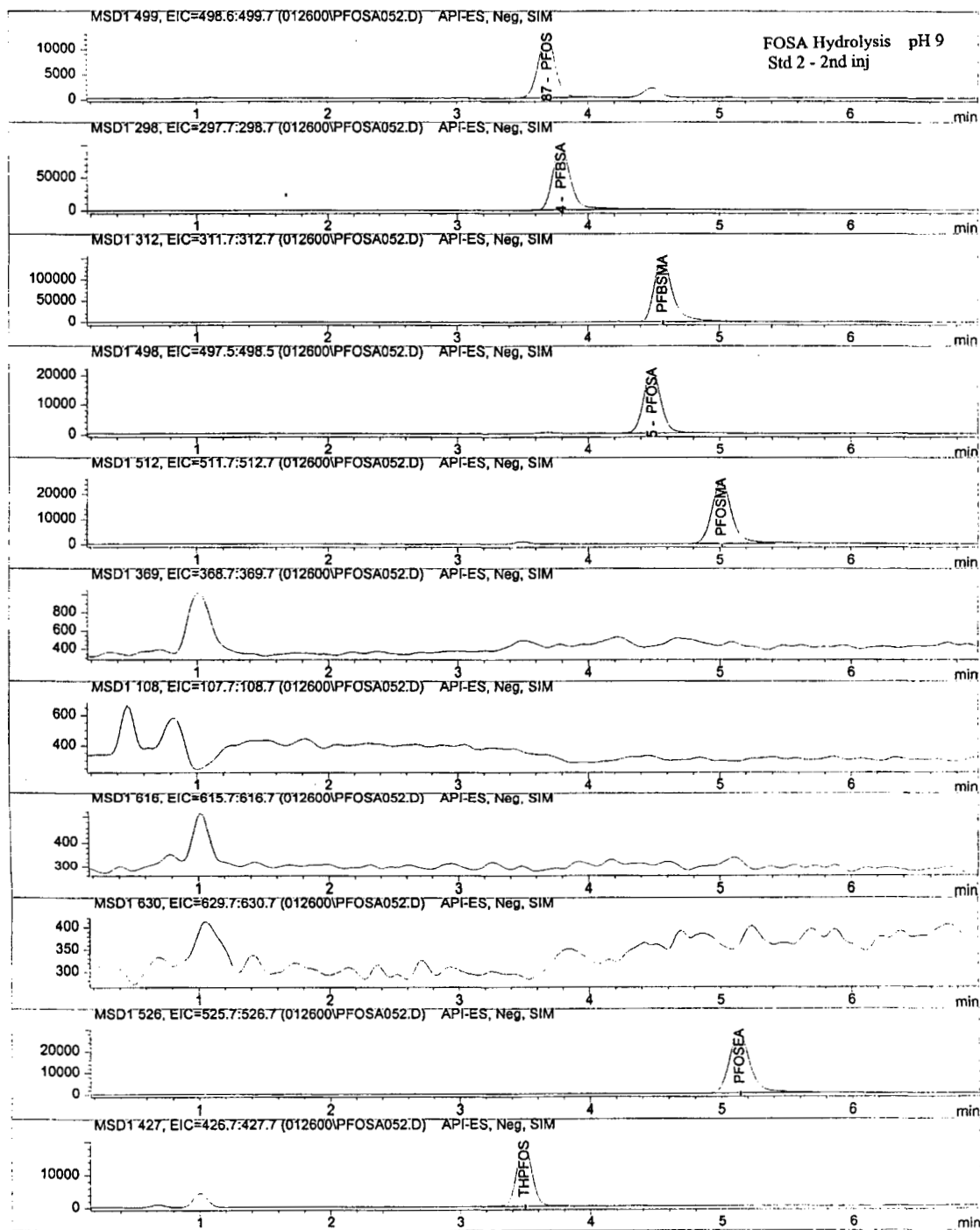
Data4 9/27/00 3:53:27 PM AES/ALS

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Batch Run # 52 of 56

Data File C:\HPCHEM\1\DATA\012600\PFOSA052.D

Sample Name: 99039-139-02



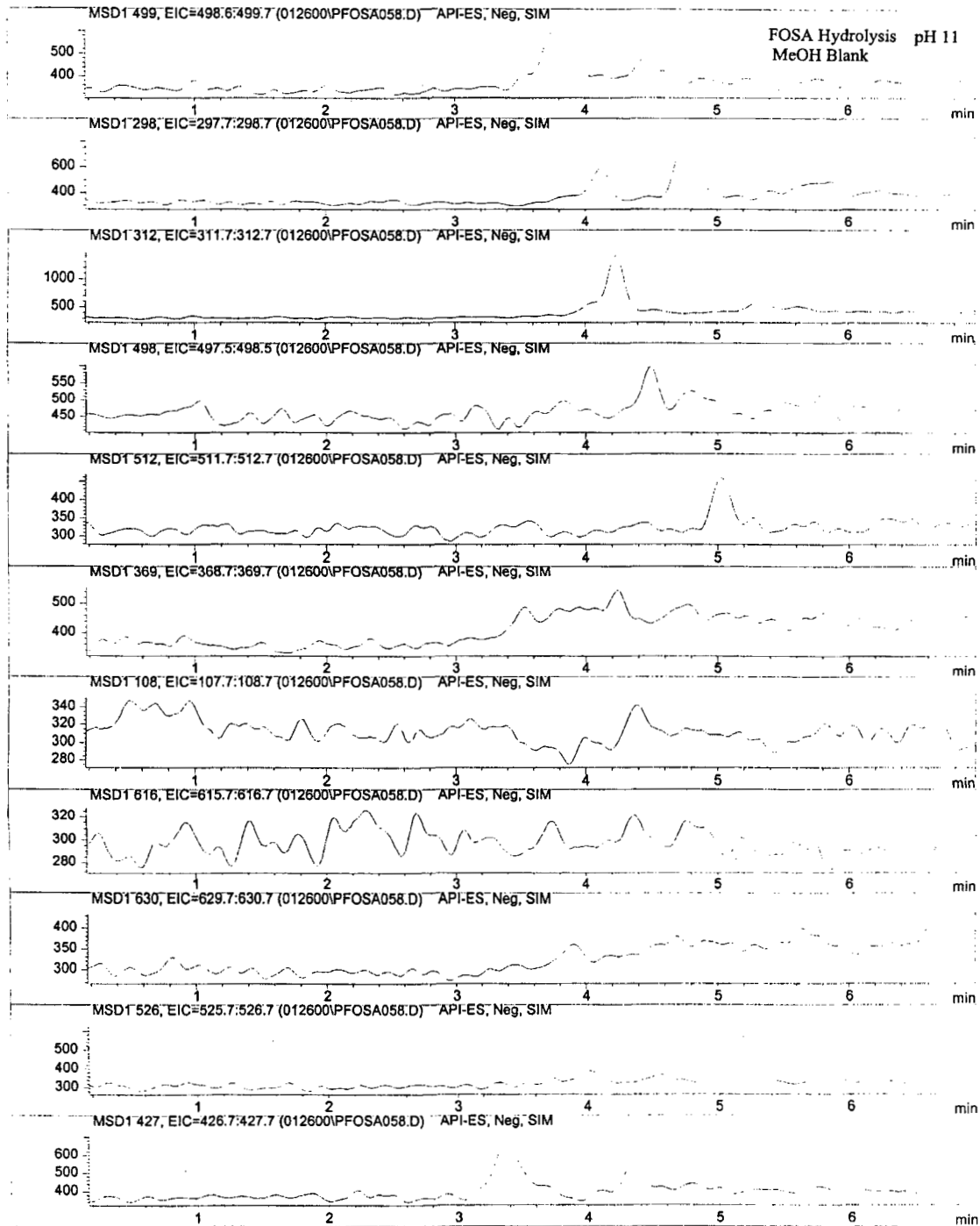
Data4 9/27/00 3:53:50 PM AES/ALS

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Batch Run # 2 of 55

Data File C:\HPCHEM\1\DATA\012600\PFOSA058.D

Sample Name: MeOH Blank

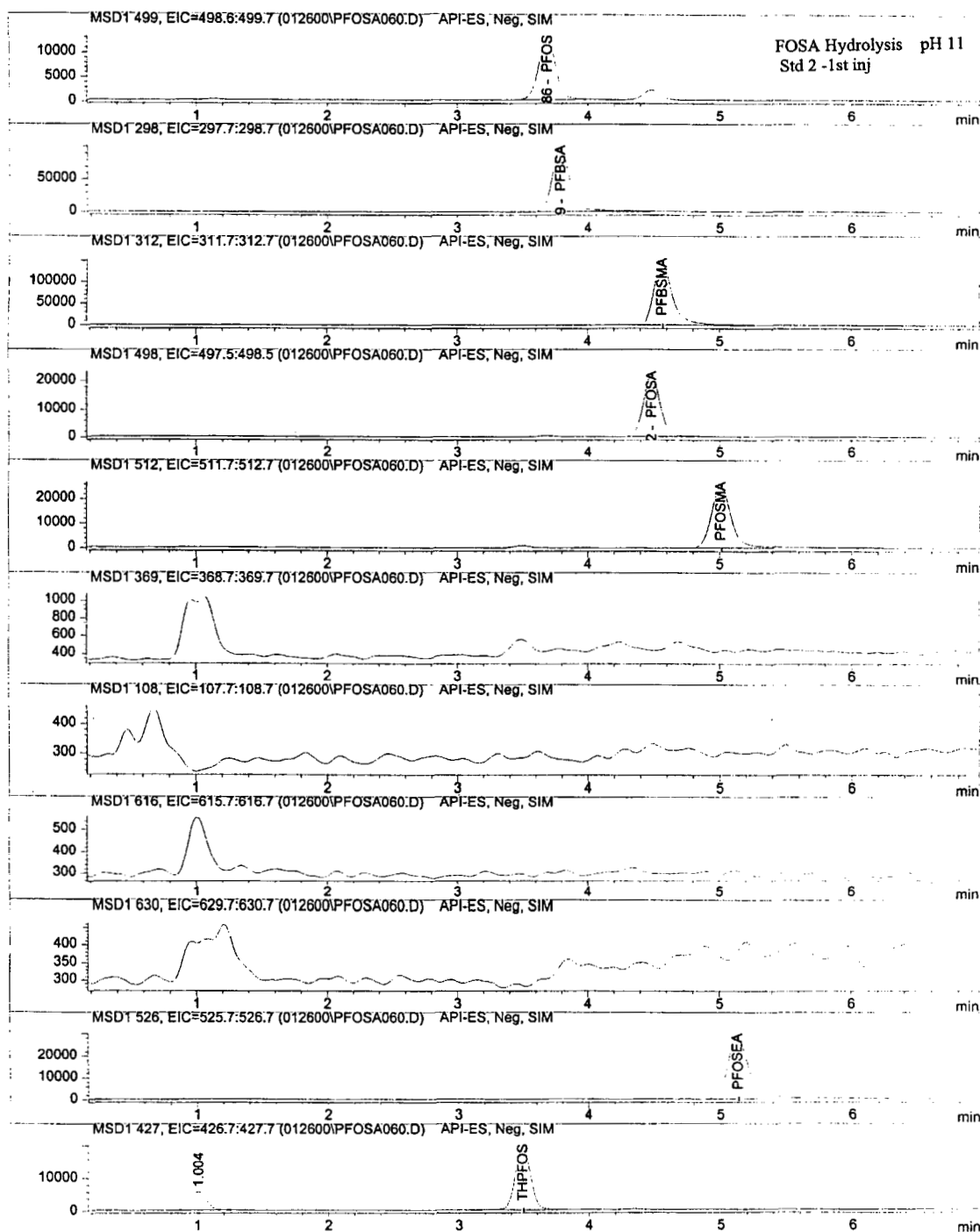


Data4 9/28/00 9:58:36 AM AES/ALS

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Batch Run # 4 of 55  
Data File C:\HPCHEM\1\DATA\012600\PFOSA060.D

Sample Name: 99039-140-02



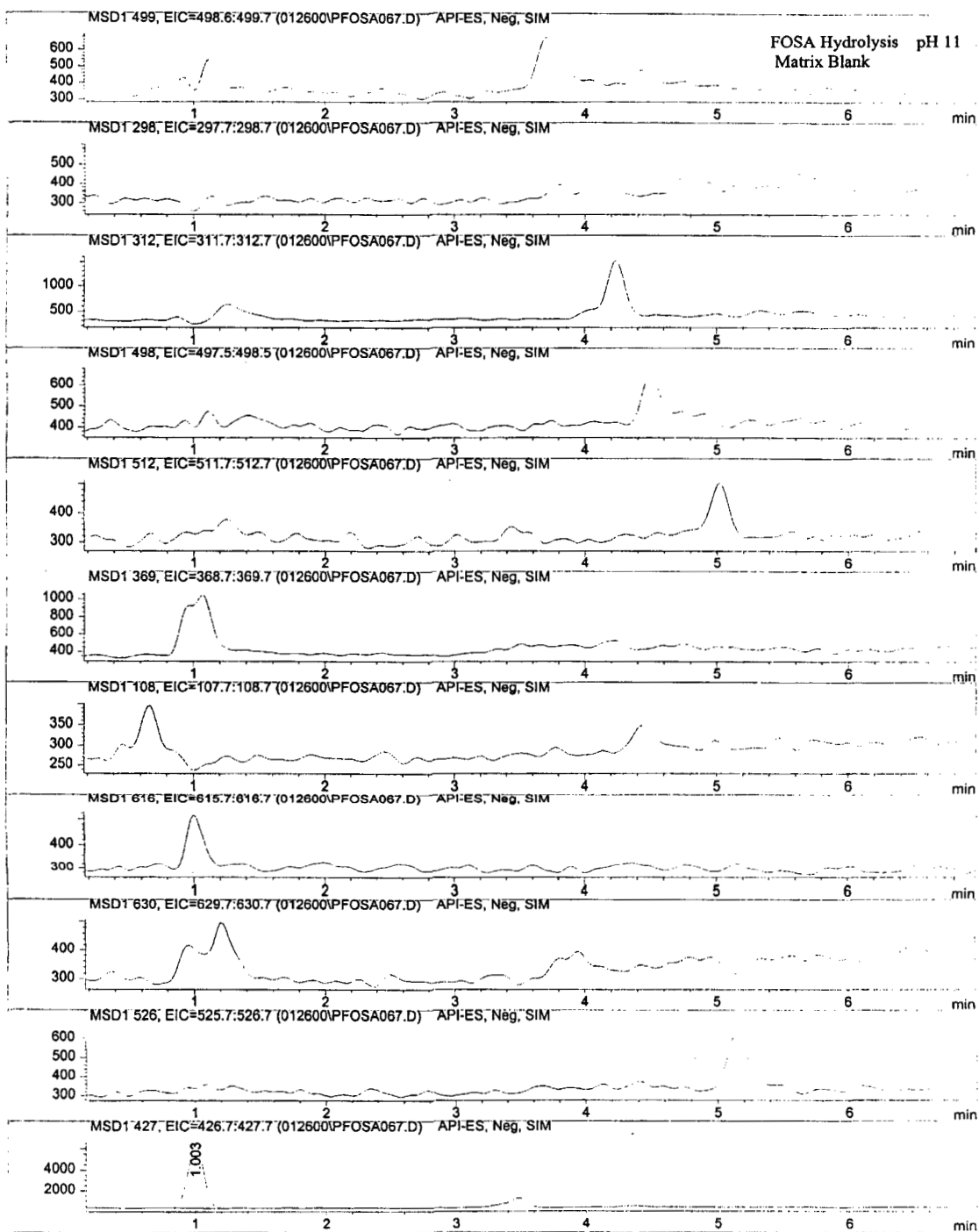
Data4 9/28/00 9:58:55 AM AES/ALS

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Batch Run # 11 of 55

Data File C:\HPCHEM\1\DATA\012600\PFOSA067.D

Sample Name: 081399-BLK-11.0



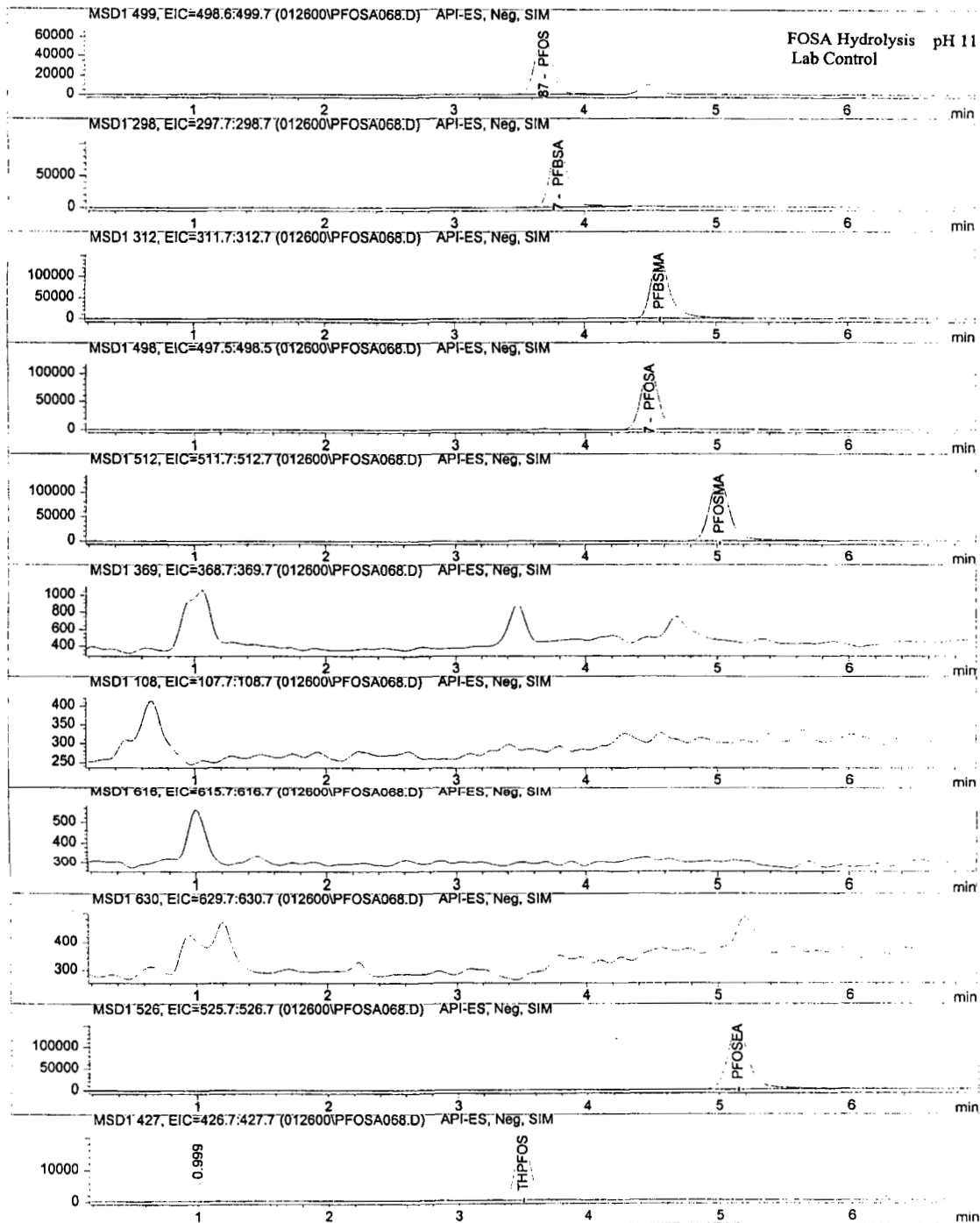
Data4 9/28/00 9:59:57 AM AES/ALS

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Batch Run # 12 of 55

Data File C:\HPCHEM\1\DATA\012600\PFOSA068.D

Sample Name: 081399-LCS-11.0



Data4 9/28/00 10:00:08 AM AES/ALS

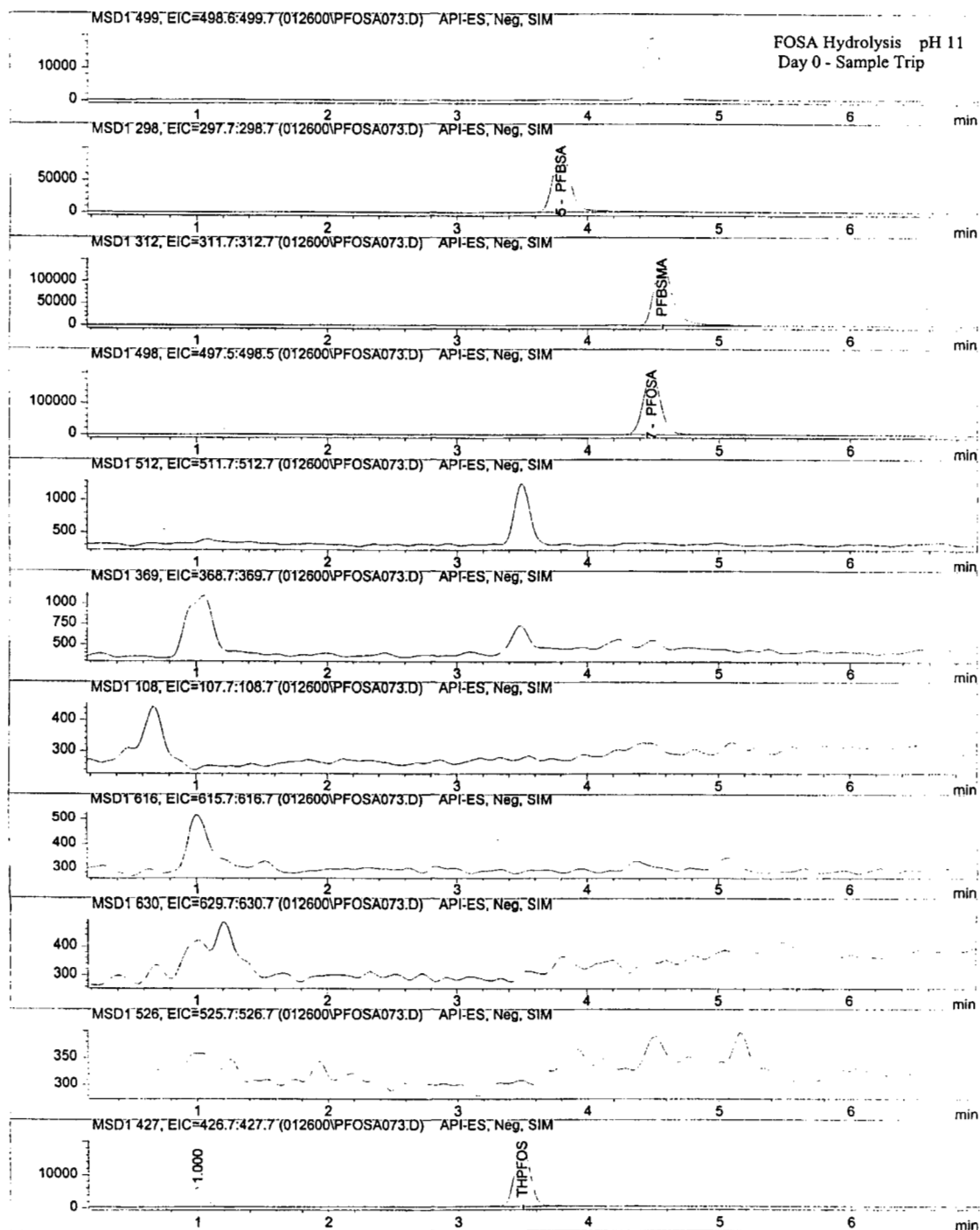
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Batch Run # 17 of 55

Data File C:\HPCHEM\1\DATA\012600\PFOSA073.D

Sample Name: 80399PFOSA-023



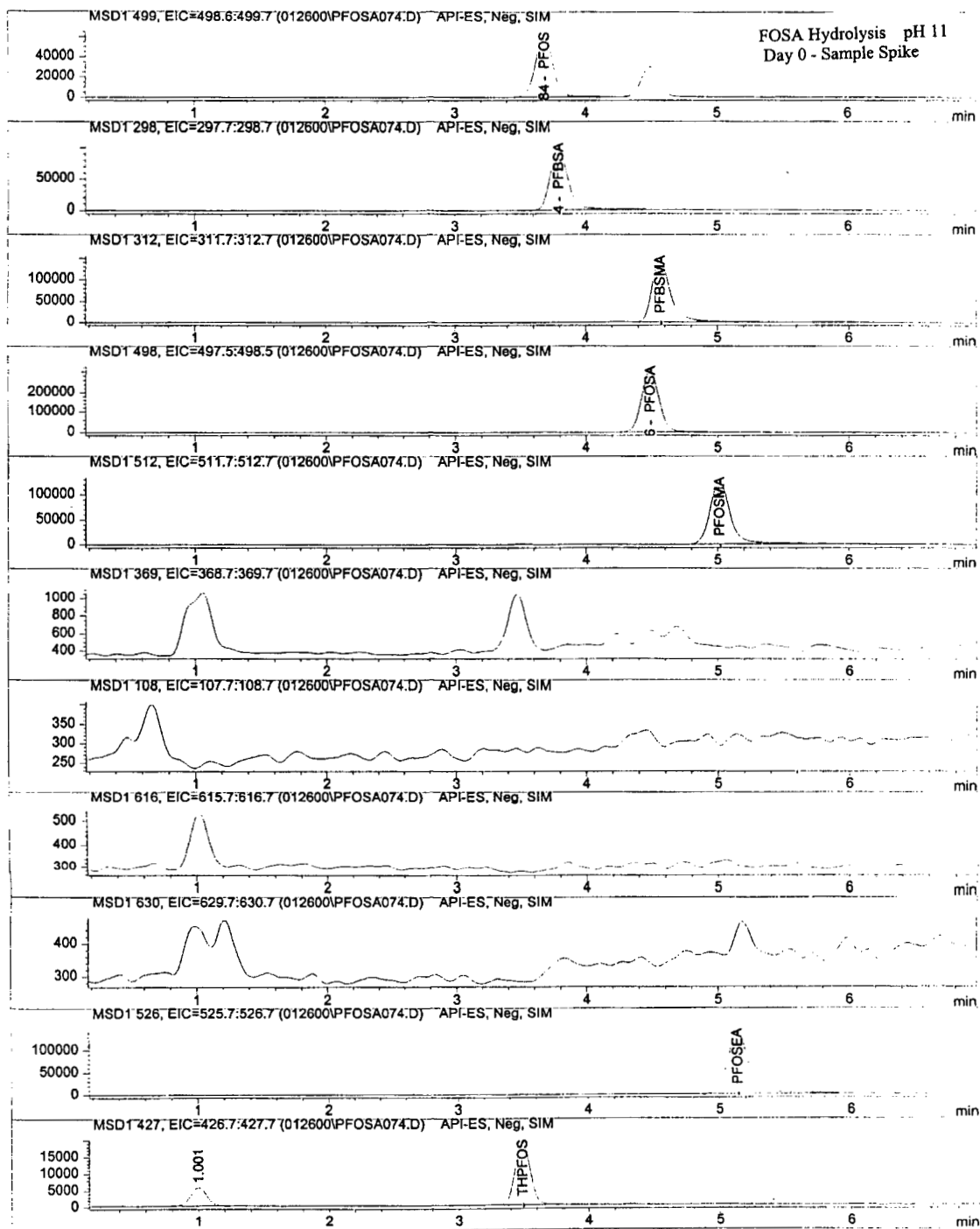
Data4 9/28/00 10:00:55 AM AES/ALS

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Batch Run # 18 of 55

Data File C:\HPCHEM\1\DATA\012600\PFOSA074.D

Sample Name: 80399PFOSA-024



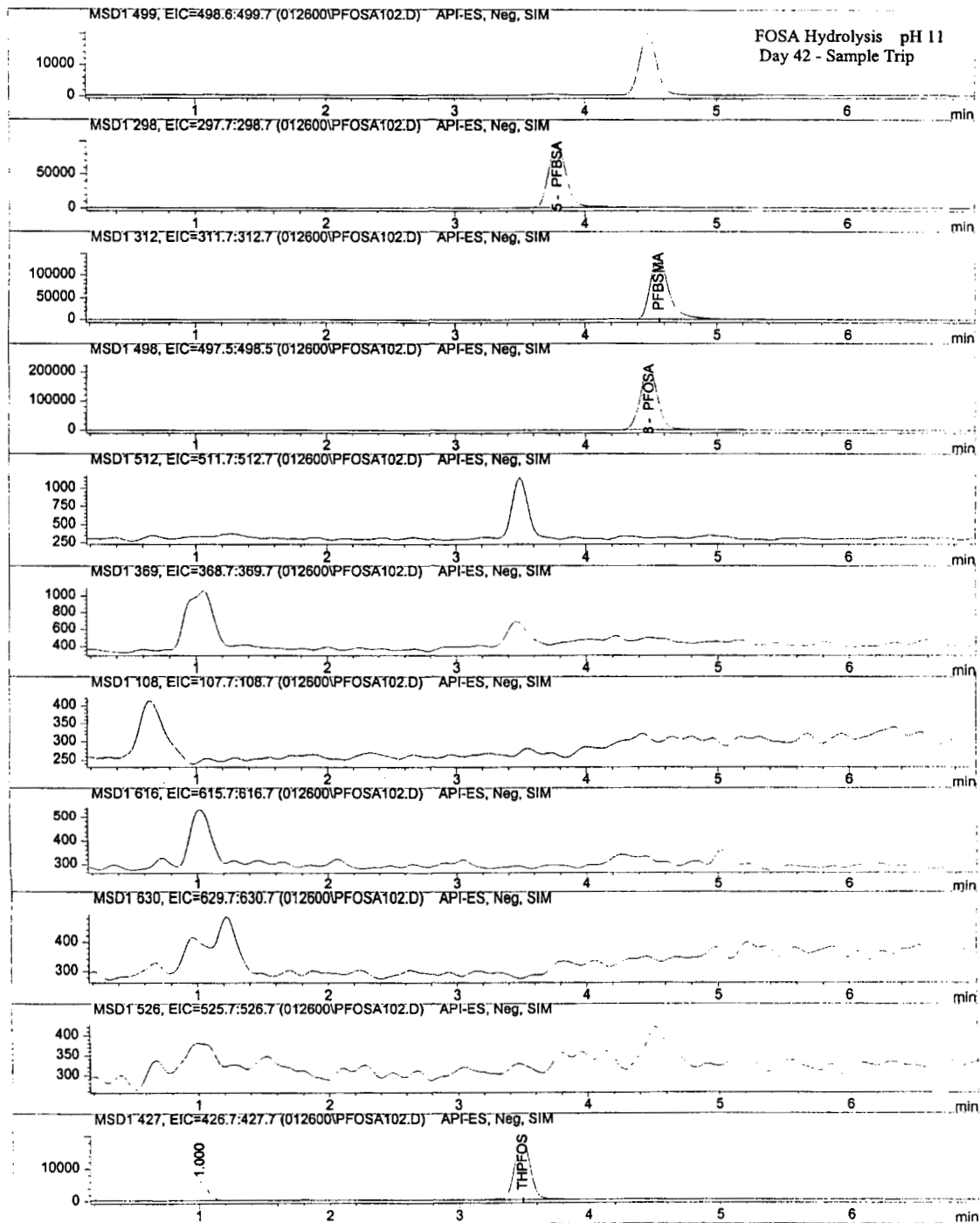
Data4 9/28/00 10:01:03 AM AES/ALS

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Batch Run # 46 of 55

Data File C:\HPCHEM\1\DATA\012600\PFOSA102.D

Sample Name: 80399PFOSA-167



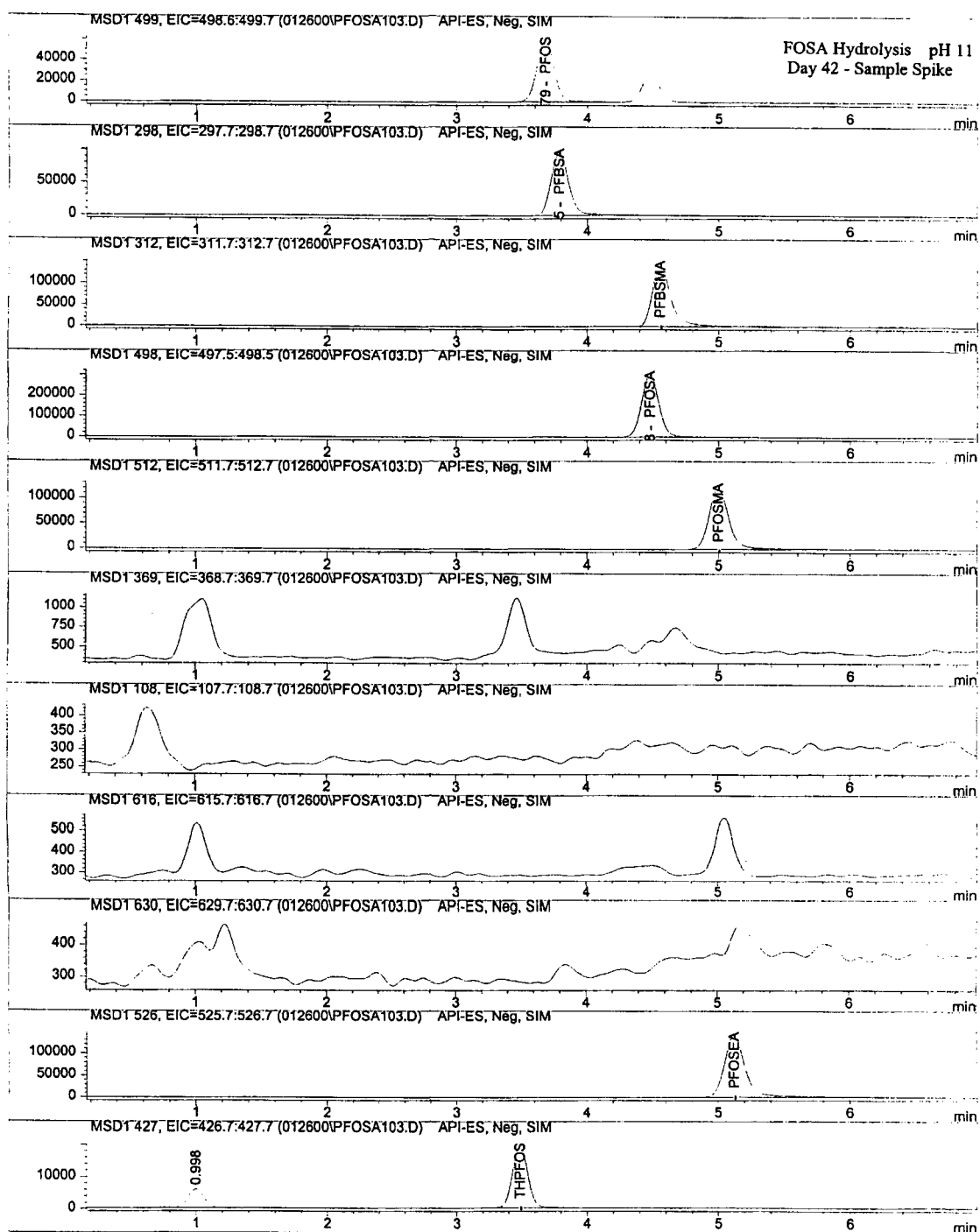
Data4 9/28/00 10:05:07 AM AES/ALS

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Batch Run # 47 of 55

Data File C:\HPCHEM\1\DATA\012600\PFOSA103.D

Sample Name: 80399PFOSA-168



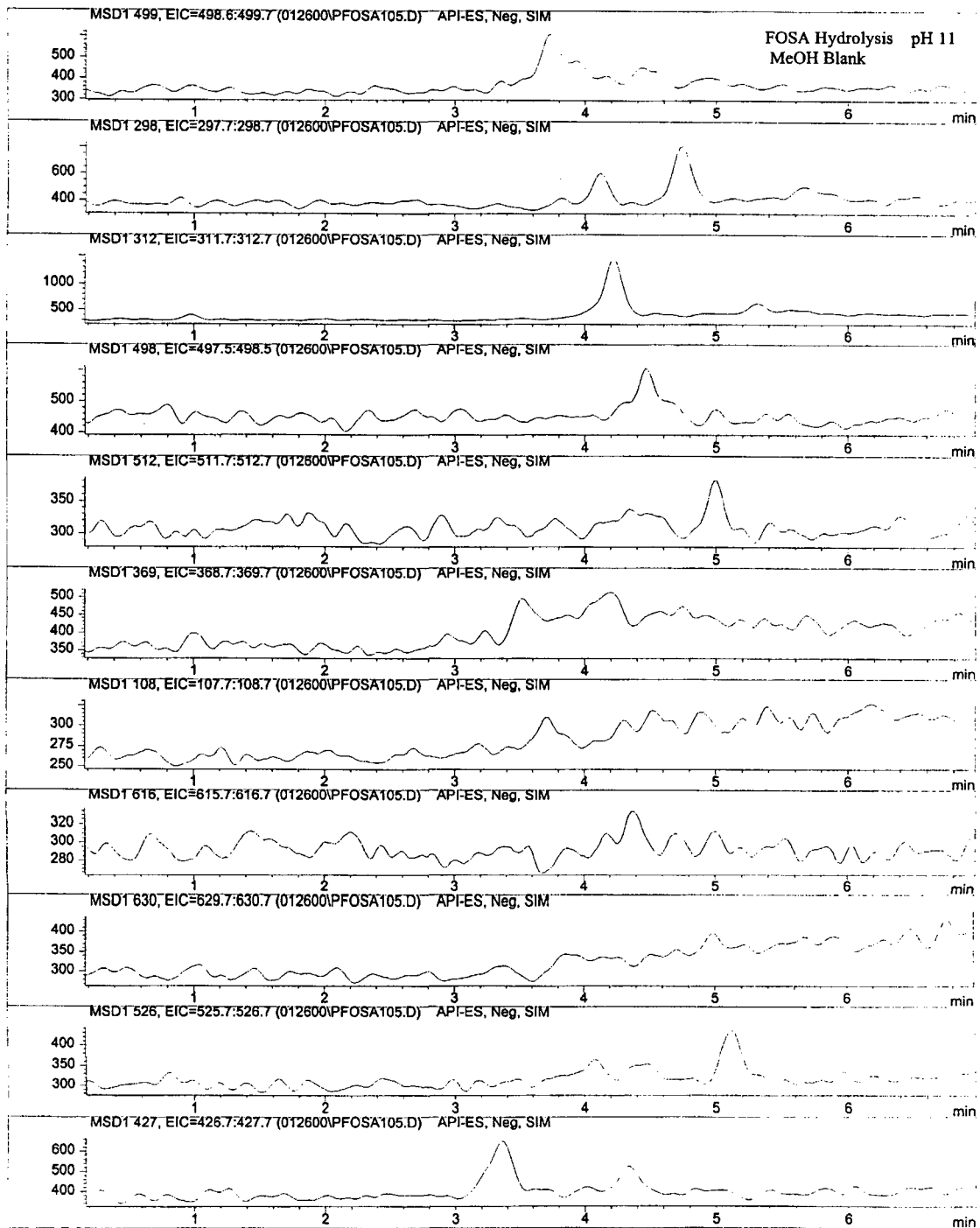
Data4 9/28/00 10:05:15 AM AES/ALS

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Batch Run # 49 of 55

Data File C:\HPCHEM\1\DATA\012600\PFOSA105.D

Sample Name: MeOH Blank

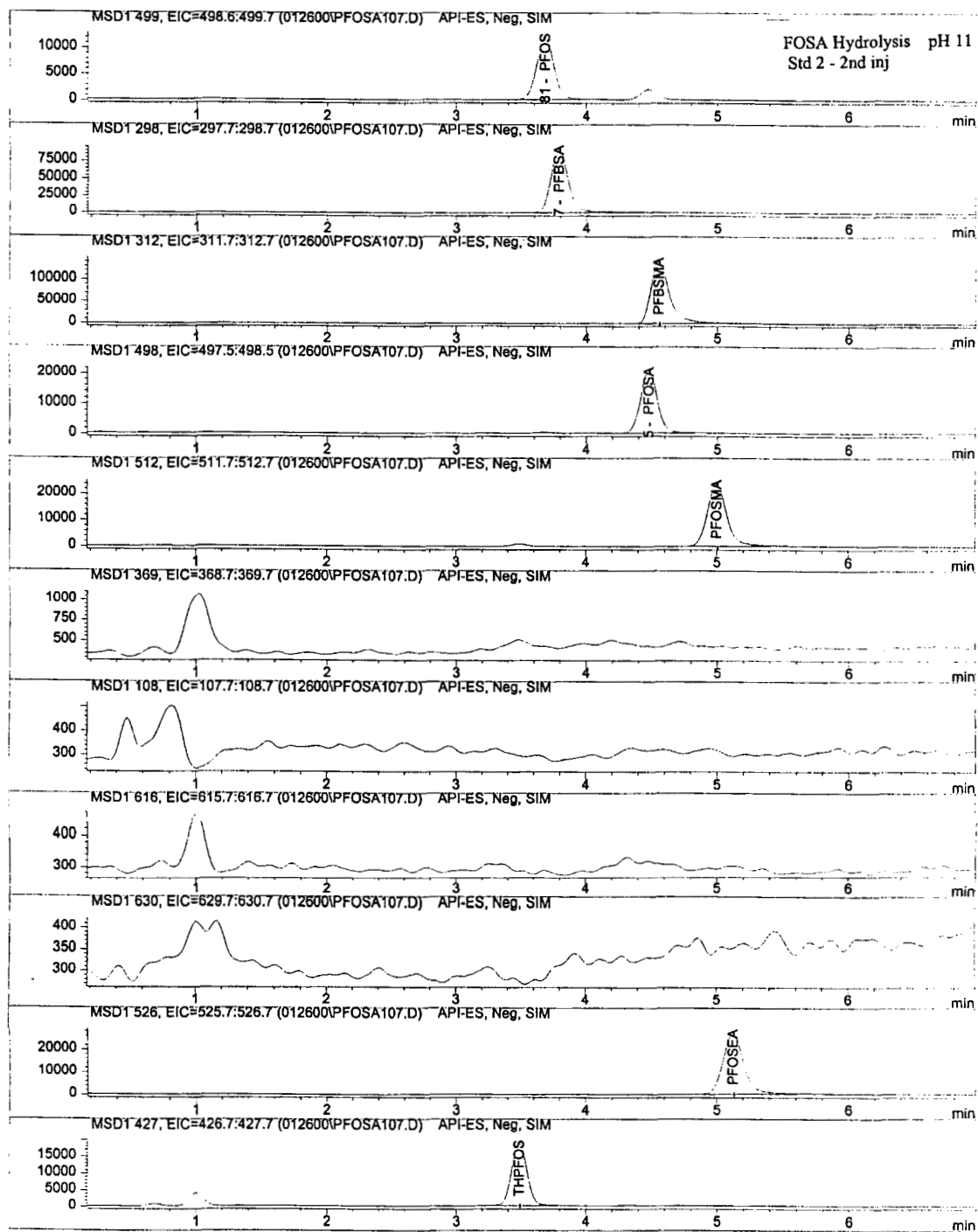


Data4 9/28/00 10:05:34 AM AES/ALS

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Batch Run # 51 of 55  
Data File C:\HPCHEM\1\DATA\012600\PFOSA107.D

Sample Name: 99039-140-02



Data4 9/28/00 10:05:53 AM AES/ALS

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