US EPA ARCHIVE DOCUMENT

United States Environmental Protection Agency

Results of the Analyses of Screening Surface and Well Water Samples from Decatur, Alabama for Selected Perfluorinated Compounds

US EPA ARCHIVE DOCUMENT

EPA 600/C-09-013
April 2009
www.epa.gov

US EPA ARCHIVE DOCUMENT

Results of the Analyses of Screening Surface and Well Water Samples from Decatur, Alabama for Selected Perfluorinated Compounds

Andrew B. Lindstrom, Mark J. Strynar, Amy D. Delinsky, Larry McMillian

Human Exposure and Atmospheric Sciences Division
National Exposure Research Laboratory
U. S. Environmental Protection Agency
Research Triangle Park, NC 27711

Shoji F. Nakayama

Land Remediation and Pollution Control Division
National Risk Management Research Laboratory
U. S. Environmental Protection Agency
Research Triangle Park, NC 27711

U.S. Environmental Protection Agency
Office of Research and Development
Washington, DC 20460

Results of the Analyses of Screening Surface and Well Water Samples from Decatur, Alabama for Selected Perfluorinated Compounds

Report Date: April 16, 2009

Principle Investigators: Mike Neill**,** EPA/Region 4/SESD

Field Sampling: Mike Neill, EPA/Region 4/SESD

Sample Analysis: Andrew B. Lindstrom, Ph.D., Mark J.Strynar, Ph.D., Amy D. Delinsky, Ph.D., Larry McMillian, EPA/ORD/NERL; Shoji F. Nakayama, Ph.D. EPA/ORD/NRMRL

Disclaimer: The United States Environmental Protection Agency through its Office of Research and Development funded and managed the work described here. It has been subjected to the Agency's administrative review and approved for publication

Introduction

EPA has conducted testing of agricultural sites in Alabama where sewage sludge was applied from a local wastewater treatment plant that receives wastewater from numerous industrial sources, including facilities that manufacture and use perfluorooctanoic acid (PFOA) and other perfluorinated chemicals (PFCs). The results from the initial limited testing indicated elevated levels of PFCs in the sewage sludge and the soil that received the sewage sludge. As a result, EPA conducted sampling of public drinking water, private wells, springs, ponds, and soil in the area. In January 2009, EPA developed drinking water Provisional Health Advisories for PFOA and perfluorooctane sulfonate (PFOS) (Reference No. 1) to provide information in response to an urgent and rapidly developing situation. These short term Provisional Health Advisories reflect reasonable, health-based hazard concentrations above which action should be taken to reduce exposure to unregulated contaminants in drinking water. The Provisional Health Advisories are 0.4 ppb (400 ng/L) for PFOA and 0.2 ppb (200 ng/L) for PFOS. The levels of PFOA and PFOS recently analyzed in community water systems in Lawrence and Morgan Counties were all lower than 0.04 ppb (2).

EPA conducted a small scale reconnaissance screening level study in which Region 4 staff collected a limited number of groundwater and surface water samples during the period of February 16 – 19, 2009. A total of 51 samples collected from surface and potable water sources were submitted for PFC analysis. Analyses were performed in EPA's Office of Research and Development (ORD) laboratory to assess if perfluorochemicals have migrated into private drinking water supplies, ponds, and streams in the affected area. The purpose for collection and analyses of these 51 samples was to determine if the concentrations of PFOA and PFOS were at or above the Provisional Health Advisory levels. These analyses were not intended to measure low levels of the PFCs that may be present at concentrations below the calibration range discussed in this document.

US EPA ARCHIVE DOCUMENT

This report provides a description of the methods used for the collection and analyses, documentation of the quality control samples, and the resulting measured concentrations of the selected perfluorinated chemicals, including PFOA and PFOS, measured in the 51 water samples submitted by EPA Region 4 for analysis.

Methods

Sample Collection

The field portion of this study was conducted by USEPA Region 4 personnel following the procedures established in their Quality Assurance Project Plan (QAPP) entitled "Land Application Site Near Decatur, Alabama – Private Wells Perfluorinated Compounds Study" (3). The Perfluorinated Chemicals (PFC) laboratory in the National Exposure Research Laboratory (NERL) of ORD, located in Research Triangle Park, NC, prepared and sent Region 4 personnel the materials used for the collection of these samples. Sampling materials were shipped to Region 4 personnel on February 11, 2009. The shipment consisted of five individual shipping containers, with each container holding one prepared field blank, two prepared field spikes (one with each target analyte listed in Table 1 at 200 ng/L and another with each target analyte at 400 ng/L), and 12 pre-cleaned (triple rinsed with methanol and dried) 1-L HDPE (Nalgene) sampling bottles. A few additional wide-mouth containers were also included for use as water transfer vessels in cases where source access might make direct filling of the narrow-mouth sampling bottles difficult.

The selection of sampling locations and the collection of the surface and well water samples were the responsibility of Region 4 personnel. Methods used for sample collection are outlined in the NERL/HEASD SOP No. EMAB-113-0 entitled "Sample Collection Protocol for PFCs in Surface and Well Water (4). The specific sampling method was dependent on the source of water being sampled; however, the basic process involved rinsing the collection bottle with three volumes of water followed by filling on the fourth iteration and adding 5 mL of 35% nitric acid as a preservation agent. Duplicate samples were collected at a rate of 10%. Samples were labeled with unique codes as well as the appropriate information providing time, date, location and water source type. Samples were maintained at ambient temperature after collection. All samples were shipped on February 20, 2009 to the RTP PFC laboratory for analysis, with chain of custody (CoC) forms recording collection, shipment, and receipt (Appendix 1).

Sample Analysis

Analysis of samples was conducted by the RTP PFC team following the procedures established in the NERL/HEASD Quality Assurance Project Plan entitled "The Analysis of Screening Surface and Well Water Samples for Selected Perfluorinated Compounds from Decatur, Alabama" (5). The chemical analyses were performed using the method outlined in SOP # EMAB 114.0 "Improved Method for the Extraction and Analysis of Perfluorinated Compounds (PFCs) from Surface and Well Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS)" (6).

US EPA ARCHIVE DOCUMENT

Actual water volumes were determined, sample containers were rinsed with methanol and the rinsate combined with the original water sample. The sample was then spiked with an internal standard mixture. Waters Oasis WAX solid phase extraction (SPE) cartridges were used for the retention and purification of the target PFCs. After capture and washing, the target PFCs were eluted from the SPE cartridges and the eluates were analyzed using a UPLC-MS/MS operated in negative electrospray ionization (ESI) mode.

For the analyses of these water samples, a method previously developed for trace level analysis (7) was modified to measure midlevel concentrations (10 -1000 ng/L) of the target analytes to allow for more accurate comparison with the Provisional Health Advisories for PFOA and PFOS (400 ng/L and 200 ng/L, respectively). To achieve this purpose, it was necessary to calibrate the LC/MS/MS over a range of concentrations from 10 to 1000 ng/L. Quantitation was performed using a multipoint calibration curve and internal standard calculation. The Limit of Quantitation (LOQ) of the modified method, defined as the lowest point on the standard curve which back-predicted within ±30% of the theoretical value, was determined to be 10 ng/L (0.01 ppb) for all compounds except the C7 and C10 acids, which were 50 ng/L (0.05 ppb). It should be noted that the LOQ for these 51 water samples is much higher than the LOQ of 0.2 ng/L determined for the previously developed trace level method (7) and was based on the lowest calibration standard. The limit of detection (LOD) and LOQ have been defined in a number of ways in the scientific community, including EPA's Occupational and Residential Exposure Test Guidelines (8). The EPA Office of Prevention, Pesticides and Toxic Substances' (OPPTS') preferred procedure is to define LOD as the concentration having a signal-to-noise ratio of 3, and LOQ as 3.3 times the LOD. These are considered typical and acceptable definitions for LOD and LOQ. However, for the purposes of these analyses to identify samples with concentrations at or above 200 ng/L, the use of the higher LOQ was reasonable and most expeditious for reporting the measurement results.

Table 2 is a summary of the LC/MS/MS parameters used in this assessment.

Analyte concentrations were determined using the stable-isotope internal standard method using the response of the analyte (peak area counts) divided by the response of the internal standard to calculate unknown concentrations. Appendix 2 contains the standard curves used to calculate the concentrations of the samples measured in this survey.

Quality Control

The collection and analytical processes included prescribed quality control (QC) procedures to document data quality and assay performance. These QC procedures included the following:

Field Blanks

Field blanks were used to monitor for potential contamination in all steps of the process, to include the purity of all reagents used, possible contamination of sample storage bottles, potential contamination associated with shipping and sample collection procedures, and possible contamination during sample preparation and analysis in the laboratory. Field blanks were prepared in the RTP PFC laboratory by filling pre-cleaned 1 L collection bottles with deionized laboratory grade water, previously determined to be PFC-free. The samples were preserved with

US EPA ARCHIVE DOCUMENT

the addition of 5 mL of 35% nitric acid, and shipped into the field with the empty containers designated for collection of field samples. The field blanks were included at a rate of 10% of all planned samples.

Field Spikes

Field spikes were used to monitor how sample preparation, collection, storage, and analysis procedures potentially influence target analyte recovery. Field spikes were prepared at low (200 ng/L) and high (400 ng/L) levels of all of the compounds on the target list (Table 1). These samples were preserved with the addition of 5 mL of 35% nitric acid and shipped into the field with the empty containers designated for collection of field samples. Both low and high level field spikes were included at a rate of 10% of all planned field samples (e.g., 5 low level and 5 high level spikes).

Field Duplicates

Field duplicates were used to document precision (repeatability) of the entire process, to include potential variability in reagents, sample storage bottles, differences in sample collection technique, and possible variation in sample processing in the laboratory during analysis. Duplicate surface and well water samples were collected at a rate of 10% of all planned field samples.

As part of the laboratory analysis, QC procedures included:

Matrix Blank

Field samples that have high concentrations of the target analytes may need to be diluted before they can be accurately measured in the laboratory. It is therefore necessary to determine that this diluent (blank matrix) is free of potential contamination. A matrix blank sample was prepared in the RTP PFC laboratory by filling a pre-cleaned 1 L collection bottle with deionized laboratory grade water, previously determined to be PFC-free, and adding 5 mL of 35% nitric acid. After addition of the internal standard mix, this material was analyzed and determined to be free of potential contaminants before it was used as a diluent for samples that had concentrations of PFOA that exceeded 1000 ng/L in the preliminary screening analysis.

Solvent Blanks

Solvent blanks containing unprocessed methanol (MEOH) and 2 mM ammonium acetate at a ratio of 1:3 were analyzed to assure that the primary solvents used in the LC mobile phase were not contaminated with any of the target PFCs.

Fortified Water Samples

Water samples fortified with target analytes (standard addition) were prepared at a rate of 10% of all field samples. After successful analysis of the first 500 mL portion of selected 1 L samples, the remaining portion received a spike of the native standard solution containing all target analytes

US EPA ARCHIVE DOCUMENT

equivalent to 400 ng/L for each of the individual analytes. These samples were prepared to provide assurance that retention times, quantitiation and qualification ions, and calibration procedures were consistent between unknown and fortified samples.

Quantitiation and Qualification Ion Ratios

For each target analyte in the field samples, quantitiation and qualification ions were monitored and compared with the quantitiation and qualification ion ratios observed in the standards used to construct the standard curves. If the quantitiation/qualification ion ratio of the field samples differed by more than 2 standard deviations from the standard curve points, the sample was flagged and examined for potential errors associated with inappropriate peak integration, retention time, or ion suppression/enhancement. (Sufficient quantities of qualification ions were not produced for the C4 and C5 acids to allow for this analysis).

Method Deviations

The analytical procedures outlined in SOP # EMAB 114.0 "Improved Method for the Extraction and Analysis of Perfluorinated Compounds (PFCs) from Surface and Well Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS)" (6) were followed, with minor changes to improve performance.

Modification to SPE Elution

The original analytical protocol (single elution method) was designed to measure comparatively low levels (< 50 ng/L) of the target PFCs in surface water. The elution step was tailored to extract low levels of the target compounds from the SPE cartridge without removing other compounds that interfere with target ion ionization. Because this application of the analytical method focused on comparatively high levels of the PFCs, avoiding low levels of co-eluting interferences was not a substantial concern in this case and after an initial analysis of the extracts, it was observed that ion counts for the internal standards and native standards for some of the samples were lower than expected, possibly adversely impacting the precision and accuracy. It was decided, therefore, to use a more aggressive extraction procedure to increase signal, thereby improving sample accuracy and precision. Operating on the hypothesis that analyte recovery from the SPE cartridge was lower than expected, each cartridge was re-eluted with an additional 4 mL of elution solvent described in section 10.4.5 of the analytical SOP. This additional extract was combined with the initial extract, and the combined total was concentrated to a volume of 3 mL. This combined and concentrated extract was mixed with buffer and reanalyzed, giving greater ion counts and a corresponding increase in precision and accuracy.

Sample Batch Size

Samples were prepared and analyzed as a single batch containing all samples, QC samples, and calibration standards. This served to eliminate batch to batch variability, thereby improving precision of the analytical process.

US EPA ARCHIVE DOCUMENT

Results

Sample Completeness

As indicated in Table 3, all field samples were collected by Region 4 field personnel and analyzed by the RTP PFC analytical team. (Note: Fifty samples were scheduled for collection; however, a total of 51 samples were received from the field. The additional sample was collected in one of the water transfer bottles. It was also analyzed and reported.) All of the proposed field duplicates, field blanks, low level field spikes, high level field spikes, and laboratory spikes (n = 5 for each) were prepared and analyzed as planned.

Standard Curve Back-Prediction

Before the standard curves were applied to the sample data, an analysis was conducted to determine how well each standard point back-predicted its own theoretical value. Acceptable back-prediction is within ± 20 % for all but the lowest standard point, where ± 30 % is acceptable. Given these criteria, the LOQs were determined to be 10 ng/L for each compound except the C10 and C7 acids, which were assigned LOQs of 50 ng/L. Table 4 summarizes the mean back-predicted values for all standard curve points used in this analysis. After adjustment of the LOQ for C10 and C7, the mean back-prediction criteria were satisfied for all calibration points. The standard curves for each of the target compounds are included in Appendix 2.

Quality Control Samples

Table 5 contains a summary of the results from the field blank and spike samples. All of the field blank samples had concentrations of all of the target compounds that were less than the LOQ. The mean accuracy of the low (200 ng/L) and high level (400 ng/L) field spikes was in all cases within ± 25% of the theoretical spiked concentration.

Field Duplicates

The results of the duplicate field samples are presented in Table 6. Of the five duplicate samples that were collected, three of the samples had analyte concentrations that were near, or below the LOQ. The duplicate samples with low concentrations near, or below, the LOQs were in good agreement. Samples W36SW and W36SW Dup, for which most of the target analytes were in the measureable range, had relative percent difference values in most cases of < 20%. Duplicate values for PFOS in this duplicate set had a relative difference of 42%, but the concentrations were at the lowest portion of the calibration curve.

Fortified Water Sample (Standard Addition)

To help evaluate the response of the analytical assay at the midrange of the calibration curves, standard addition was performed on five selected field samples. After the initial analyses were performed, 40 µL of a mixed standard solution containing all of the native standards at 5 ng/µL was added to the remaining portion of each sample (500 mL) in order to provide an additional 400

US EPA ARCHIVE DOCUMENT

ng/L of each analyte to the samples. The samples were then reanalyzed to determine the response due to the addition of 400 ng/L of each analyte.

As summarized in Table 7, the average % recovery of standard addition at this level was within ± 12 % of the theoretical value for all compounds except C10 and PFOS, which showed 188% and 157% recovery, respectively. With the exception of the C10 and the PFOS, the results indicate good general performance of the assay at the 400 ng/L level, providing additional evidence of the accuracy of the analytical procedures. The reason for the higher than expected recoveries for C10 and PFOS has not been conclusively determined. However, considering the good performance of C10 and PFOS in the field spike tests, it appears that some other process influenced the results of this specific analysis. In reviewing the data from this set of standard addition samples, it was noted that the internal standards for PFOS and C10 had approximately 50% of their response recorded in the original analysis. This diminished recovery could explain the apparently elevated recoveries for these target compounds in this part of the evaluation. It should be noted that the higher recovery noted in these analyses, which would contribute to a positive bias for these compounds, suggests that the concentrations of the C10 and PFOS compounds reported for the field samples may be somewhat higher their actual "true" concentration. However, PFOS was not measured above 200 ng/L in any of the field samples and C10 was measureable above the LOQ in only six of the 51 samples.

Field Samples

Table 8 summarizes all of the data from the field samples. Samples W01PW, W11PW, W22PW, W54PW, and W62PW were identified by Region 4 personnel as samples from wells used for drinking water (indicated in Table 8). Of the 51 unique field samples collected (duplicates excluded), PFOA was detected in 29 (57%) of the samples. The PFOA concentrations ranged from < LOQ to a high of 11,000 ng/L, with 11 samples out of 51 (22%) above 400 ng/L and two samples had concentrations (389 ng/L and 397 ng/L), which are not significantly different from the 400 ng/L Provisional Health Advisory level. PFOA occurred in only one drinking water sample, W54PW, at a concentration of 2,070 ng/L. PFOS was measured in 15 samples (29%) at concentrations ranging from < LOQ to a high of 151 ng/L; all PFOS concentrations were below the 200 ng/L Provisional Health Advisory level. The only drinking water sample with PFOS was W11PW with a concentration of 12.0 ng/L. Of the 51 samples, 42 (82%) had at least one target compound at concentrations above the LOQ. Five of the target compounds were measured in more than half of the samples, with C4 in 39 samples (77%), both C6 and PFOA in 29 (57%), PFBS in 27 (53%), and C5 in 26 (51%). The C9 acid was detected in 10 (20%) samples with the highest concentration being 286 ng/L. The C10 acid was detected in 6 (12%) samples with a high value of 838 ng/L. Neither compound was measured in the drinking water samples.

It should be noted again that this method was optimized for performance in the 200 ng/L to 400 ng/L range to allow for accurate comparison with the Provisional Health Advisories for PFOS and PFOA. Concentrations of PFCs in samples listed as being below the LOQ have not been reported, but should not be assumed to be zero.

US EPA ARCHIVE DOCUMENT

Chromatographic Profiles

As an indicator of chromatographic conditions and instrument response, Appendix 3 contains examples of mass spectral data from three blank samples, three spiked samples, and three field samples. The first three chromatograms are from field blank samples which contain only mass-labeled internal standards. The following three examples are field samples which have received the mass-labeled internal standards and standard addition of 400 ng/L of each of the target analytes. The final three chromatograms are field samples which have received only mass-labeled internal standards.

Discussion

Results of field blanks, field spikes, field duplicates, standard curve back-prediction, and standard addition indicate that the methods used in this assessment generally provide data of acceptable precision and accuracy. As shown in Table 8, PFOA was measured at concentrations exceeding 388 ng/L in 13 of the 51 samples. PFOS was not measured at concentrations exceeding 200 ng/L in any of the samples. All 10 target compounds were measurable above the LOQ in one or more samples. Nine of the samples contained none of the target compounds at concentrations above the LOQ.

References

(1) Provisional Health Advisories for Perfluorooctanoic Acid (PFOA) and Perfluorooctane Sulfonate (PFOS), USEPA Office of Water, January 8, 2009.

(2) Analytical Data Report: Perfluorinated Compounds in Water Samples Collected from Public Water Supplies Near Decatur, Alabama. National Exposure Research Laboratory, Office of Research and Development, USEPA, November 25, 2008.

(3) Quality Assurance Project Plan "Land Application Site Near Decatur, Alabama – Private Wells Perfluorinated Compounds Study," Science and Ecosystem Support Division, USEPA Region 4, January 11, 2009.

(4) Standard Operating Procedure EMAB-113-0. "Sample Collection Protocol for PFCs in Surface and Well Water," Human Exposure and Atmospheric Sciences Division, National Exposure Research Laboratory, Office of Research and Development, USEPA, February 12, 2009.

(5) Quality Assurance Project Plan "The Analysis of Screening Surface and Well Water Samples for Selected Perfluorinated Compounds from Decatur, Alabama," Human Exposure and Atmospheric Sciences Division, National Exposure Research Laboratory, Office of Research and Development, USEPA, March 13, 2009.

(6) Standard Operating Procedure EMAB 114.0 "Improved Method for the Extraction and Analysis of Perfluorinated Compounds (PFCs) from Surface and Well Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS)," Human

US EPA ARCHIVE DOCUMENT

Exposure and Atmospheric Sciences Division, National Exposure Research Laboratory, Office of Research and Development, USEPA, March 13, 2009.

(7) S. Nakayama, M. Strynar, L. Helfant, P. Egeghy, X.Ye, and A.B. Lindstrom, (2007) Perfluorinated Compounds in the Cape Fear Drainage Basin in North Carolina. *Environmental Science and Technology,* 41(16):5271-5276.

(8) EPA's Occupational and Residential Exposure Test Guidelines: OPPTS 875.2000 – Part B – Chapter 1; Background – General Provisions Guideline, USEPA, Office of Prevention, Pesticides and Toxic Substances, 1998. This document is available at http://www.epa.gov/scipoly/sap/1998/march/chapb-1.pdf .

List of Tables

Table 1. Perfluorinated analytes, abbreviations, internal standards, LC/MS/MS transitions, confirmation ions, and ion ratios monitored

Table 2. Summary of the LC/MS/MS method including target and qualifier ions

Table 3. Proposed, Collected, and Analyzed Samples Summarizing Completeness

Table 4. Mean Back-Predicted Values for all Standard Curve Points

Table 5. Summary of Field Blanks, Low Level Field Spikes, and High Level Field Spikes in ng/L

Table 6. Summary of Duplicate Field Samples in ng/L

Table 7. Standard Addition of 400 ng/L of Each Analyte to Selected Field Samples

Table 8. Perfluorinated Compound Concentrations in Surface and Well Water Samples in ng/L

List of Appendices

Appendix 1. Chain of custody forms (CoC) for water samples collected, shipped and received by the analytical laboratory.

Appendix 2. Calibration curves for all target analytes X's correspond to standard points, ◊'s correspond to quality controls samples at either 200 or 400 ng/L

Appendix 3. Mass spectral data from 3 blank samples, 3 spiked samples, and 3 field samples.

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

Table 1. Perfluorinated analytes, abbreviations, internal standards, LC/MS/MS transitions, confirmation ions, and ion ratios monitored in analysis

Target Analyte	**Quantitation transition**	**Confirmation transition**	**IS**	**†ion ratio (mean)**	**ion ratio (SD)**	**LOQ (ng/L)**
Perfluorobutanoic acid (C4)	212.80 → 168.75	*NA	$^{13}C_2$-C6	NA	NA	10
Perfluoropentanoic acid (C5)	262.85 → 218.75	NA				10
Perfluorohexanoic acid (C6)	312.70 →268.70	312.70 →118.70		16.26	2.05	10
Perfluoroheptanoic acid (C7)	362.65 → 318.70	362.65 → 168.65		4.81	0.23	50
Perfluorooctanoic acid (C8)	412.60 → 368.65	412.60 → 168.70	$^{13}C_8$-C8	3.63	0.26	10
Perfluorononanoic acid (C9)	462.60 → 418.60	462.60 → 218.75		3.89	0.27	10
Perfluorodecanoic acid (C10)	512.60 → 468.55	512.60 → 468.55	$^{13}C_2$-C11	6.31	0.50	50
Perfluorobutane sulfonate (PFBS)	298.70 → 98.80	298.70 → 79.90	$^{18}O_2$-PFHS	0.62	0.04	10
Perfluorohexane sulfonate (PFHS)	398.65 → 98.80	398.65 → 79.90		1.15	0.10	10
Perfluorooctane sulfonate (PFOS)	498.65 → 98.80	498.65 → 79.90	$^{18}O_2$-PFOS	0.62	0.03	10
1,2-$^{13}C_2$- Perfluorohexanoic acid ($^{13}C_2$-C6)	314.75 → 269.75	‡Internal Standards (IS)				
$^{18}O_2$-Sodium perfluorohexanesulfonate ($^{18}O_2$-PFHS)	402.65 → 83.90					
1,2,3,4,5,6,7,8-$^{13}C_2$-Perfluorooctanoic ($^{13}C_8$-C8)	429.65 → 375.75					
$^{18}O_2$-Ammonium perfluorooctanesulfonate ($^{18}O_2$-PFOS)	502.60 → 83.90					
$^{13}C_2$ Perfluoroundecanoic acid ($^{13}C_2$-C11)	564.60 → 519.65					

* Mass spectrometer conditions did not produce secondary qualification ions that can be used for compound confirmation
† Ratio of quantitation ion to confirmation ion, used to help confirm the identity of target compounds
‡ Parameters not used with internal standards

US EPA ARCHIVE DOCUMENT

Table 2. Summary of the LC/MS/MS method including target and qualifier ions

Reservoirs: A: 2 mM ammonium acetate in deionized water with 5% methanol, B: 2 mM ammonium acetate in 100% methanol
Column: BEH C18 reverse phase, 2.1×50 mm, 1.7 µm particle size
Flow rate: 500 µL/min
Column temperature: 50°C
Injection Volume: 40 µL
Gradient mobile phase program:

Time	A	B	curve
0.00	75	25	initial
0.50	75	25	6
3.50	10	90	6
3.60	0	100	6
4.50	0	100	6
4.60	75	25	6
6.00	75	25	6

The Quatro Premier mass spectrometer is operated in the multiple reaction monitoring (MRM) mode using negative-ion-spray ionization under the following conditions:

Instrument Parameters	
Capillary (kV)	−0.40
Source temperature	150°C
Desolvation temperature	350°C
Cone gas flow	2 L/hr
Desolvation gas flow	1200 L/hr
Cone voltage	Optimized for
Collision energy	each compound

US EPA ARCHIVE DOCUMENT

Table 2. (Continued) Compound specific parameters for Quatro Premier XE (MS/MS)

Compound	Quantitation MRM	Qualification MRM	Cone Voltage	Collision Energy
PFBS	298.70 > 98.80	298.70 > 79.90	40	28 (30)
PFHS	398.65 > 98.80	398.65 > 79.90	50	32 (38)
PFOS	498.65 > 98.80	498.65 > 79.90	60	38 (48)
C4	212.80 > 168.75		15	10
C5	262.85 > 218.75		15	9
C6	312.70 > 268.70	312.70 > 118.70	13	10 (21)
C7	362.65 > 318.70	362.65 > 168.65	14	10 (17)
PFOA or C8	412.60 > 368.65	412.60 > 168.70	15	11 (18)
C9	462.60 > 418.60	462.60 > 218.75	15	11 (17)
C10	512.60 > 468.55	512.60 > 218.75	16	12 (18)
Internal Standards				
$^{18}O_2$-PFHS	402.65 > 83.90		50	38
$^{13}C_2$-PFOS	502.65 > 83.90		60	48
$^{13}C_2$-C6	314.75 > 269.75		13	9
$^{13}C_8$-PFOA	420.65 > 375.75		15	11
$^{13}C_2$-C11	564.60 > 519.65		17	12

Note: Collision energies for qualification ions are in parenthesis

US EPA ARCHIVE DOCUMENT

Table 3. Proposed, Collected, and Analyzed Samples Summarizing Completeness

Sample Type	Proposed	Collected/prepared	Analyzed
Field Samples	50	51*	51*
Field Duplicates	5	5	5
Field Blanks	5	5	5
Low Level Field Spikes	5	5	5
High Level Field Spikes	5	5	5
Laboratory Spikes	5	5	5

*One additional sample was collected in a bottle intended for water transfer.
This sample was analyzed and is noted in Table 8 below.

Table 4. Mean Back-Predicted Values for all Standard Curve Points

	C10	**C9**	**PFOA**	**C7**	**C6**	**C5**	**C4**	**PFOS**	**PFHS**
10 ng/L standard	---	9.09	9.34	---	9.40	7.17	9.40	9.51	9.06
50 ng/L standard	50.8	51.0	52.0	48.9	52.1	63.8	52.4	54.9	55.7
100 ng/L standard	100	103	101	98.7	103	102	101	92.5	96.1
200 ng/L standard	199	213	208	210	199	215	202	206	203
400 ng/L standard	389	410	400	398	407	345	401	408	412
600 ng/L standard	574	574	593	599	591	629	601	580	594
800 ng/L standard	873	771	784	786	788	797	779	807	764
1000 ng/L standard	959	1030	1010	1010	1010	1000	1010	1000	1030

US EPA ARCHIVE DOCUMENT

Table 5. Summary of Field Blanks, Low Level Field Spikes, and High Level Field Spikes in ng/L

Sample Type	C10	C9	PFOA	C7	C6	C5	C4	PFOS	PFHS	PFBS
*Trip Blanks	< 50	< 10	< 10	< 50	< 10	< 10	< 10	< 10	< 10	< 10
* Low Level Trip Spike (SD)	210 (17)	156 (45)	162 (36)	171 (31)	195 (23)	217 (33)	218 (60)	172 (39)	198 (18)	205 (22)
Percent Accuracy (RSD)	105 (8.2)	78.1 (28.8)	80.9 (22.5)	85.5 (18.3)	97.3 11.9)	108 (15.4)	109 (27.5)	86.1 (22.7)	98.9 (9.1)	103 (10.6)
* High Level Trip Spike (SD)	448 (56.8)	301 (59.7)	318 (51.1)	339 (58.0)	388(29.3)	393 (41.5)	382 (19.2)	364 (30.9)	386 (26.5)	387 (24.2)
Percent Accuracy (RSD)	112 (12.7)	75.2 (19.9)	79.4 (16.1)	84.7 (17.1)	97.1 (7.6)	98.3 (10.6)	95.4 (5.0)	90.9 (8.5)	96.6 (6.9)	96.8 (6.2)

* Mean of 5 determinations; Low Level Field Spikes prepared at 200 ng/L; High Level Field Spikes prepared at 400 ng/L

US EPA ARCHIVE DOCUMENT

Table 6. Summary of Duplicate Field Samples in ng/L

	C10	C9	PFOA	C7	C6	C5	C4	PFOS	PFHS	PFBS
W06PW	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
W06PW dup	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
Rel % Diff	---	---	---	---	---	---	---	---	---	---
W53SW	< LOQ	< LOQ	18.4	< LOQ	< LOQ	< LOQ	< LOQ	51.1	< LOQ	< LOQ
W53SW dup	< LOQ	< LOQ	14.8	< LOQ	< LOQ	< LOQ	< LOQ	56.2	< LOQ	< LOQ
Rel % Diff	---	---	21.3	---	---	---	---	9.26	---	---
W24SW	< LOQ	< LOQ	< LOQ	< LOQ	22.1	56.6	62.6	< LOQ	< LOQ	< LOQ
W24SW dup	< LOQ	< LOQ	32.9	< LOQ	18.2	70.2	76.0	< LOQ	< LOQ	< LOQ
Rel % Diff	---	---	---	---	19.3	21.4	19.3	---	---	---
W36SW	54.2	12.4	389	394	505	333	236	30.3	16.8	38.2
W36SW dup	< LOQ	21.8	397	407	511	369	274	19.8	17.7	41.2
Rel % Diff	---	54.8	2.04	3.52	1.11	10.1	15.2	42.2	5.42	7.67
W17PW	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	13.2	< LOQ	< LOQ	< LOQ
W17PW dup	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	13.8	< LOQ	< LOQ	< LOQ
Rel % Diff	---	---	---	---	---	---	4.33	---	---	---

Rel % Diff = Relative percent difference between duplicate samples:
Absolute value of [(conc 1- conc 2)/ (mean of conc 1 and conc 2) x 100%]

US EPA ARCHIVE DOCUMENT

Table 7. Standard Addition (SA†) of 400 ng/L of Each Analyte to Selected Field Samples (ng/L)

	C10	C9	PFOA	C7	C6	C5	C4	PFOS	PFHS	PFBS
W06PW-SA†	614	433	477	460	386	369	393	551	450	420
W63PW-SA†	677	412	471	489	405	427	412	646	485	504
W02PW-SA†	1029	301	339	347	392	459	444	688	420	401
W13SW-SA†	628	403	653	731	515	480	426	595	422	450
W34SW-SA†	805	318	559	512	451	520	558	663	396	426
W06PW	*	*	*	*	*	*	*	*	*	*
W63PW	*	*	*	*	*	*	*	*	*	*
W02PW	*	*	*	*	*	*	*	*	*	*
W13SW	*	27.7	321	234	182	76.4	62.5	*	*	13.4
W34SW	*	16.2	204	73.6	103	162	234	*	*	*
(W06PW-SA†) – (W06PW)	614	433	477	460	385	369	393	551	450	420
(W63PW-SA†)- (W63PW)	677	412	471	489	405	427	412	646	485	504
(W02PW-SA†)- (W02PW)	1029	301	339	347	392	459	444	688	420	401
(W13SW-SA†)- (W13SW)	628	375	332	498	333	403	364	595	422	437
(W34SW-SA†)- (W34SW)	805	302	355	439	348	358	324	663	396	426
% recovery	153	108	119	115	96	92	98	138	113	105
	169	103	118	122	101	107	103	161	121	126
	257	75	85	87	98	115	111	172	105	100
	157	94	83	124	83	101	91	149	105	109
	201	76	89	110	87	90	81	166	99	107
Ave % Recovery	188	91.1	98.8	112	93.2	101	96.9	157	109	109
SD % Recovery	43.2	15.4	18.2	15.1	7.7	10.3	11.5	13.8	8.5	9.8

SA† = Sample received laboratory spike equivalent to 400 ng/L of each compound

* Values below the limit of quantitation, assumed to be 0 for the calculation of difference

Table 8. Perfluorinated Compound Concentrations in Surface and Well Water Samples in ng/L

Sample Name	C10	C9	PFOA	C7	C6	C5	C4	PFOS	PFHS	PFBS
W06PW	*	*	*	*	*	*	*	*	*	*
W06PW dup	*	*	*	*	*	*	*	*	*	*
W51SW	*	*	29.5	*	12.0	*	*	*	*	*
W27SW	*	*	134	81.5	65.9	68.4	72.7	‡11.6	*	*
W10SW	*	*	13.6	*	20.2	20.8	52.7	*	*	30.9
W28SW	*	*	94.8	127	153	91.1	70.8	*	*	15.6
W14PW	*	25.7	594	619	570	333	180	‡14.1	20.7	25.4
W46SW	838	286	1100	491	205	192	188	83.9	*	10.4
W42SW	‡125	93.3	993	777	729	434	303	‡16.5	17.5	40.8
W43SW	68.0	54.4	396	216	201	180	152	‡14.6	*	10.0
W32SW	230	70.9	750	839	961	571	439	‡66.3	20.6	90.2
W53SW	*	*	18.3	*	*	*	*	51.1	*	*
W53SW dup	*	*	14.8	*	*	*	*	56.1	*	*
† W03SW	*	*	*	*	*	*	19.4	13.2	*	‡20.9
W33SW	*	*	*	*	*	*	30.4	*	*	23.9
W63PW	*	*	*	*	*	*	*	*	*	*
W07PW	*	*	*	*	9.72	*	45.8	*	*	*
W101PW	*	*	*	*	*	*	14.6	*	*	22.9
W61SW	*	*	*	*	*	*	*	*	*	*
W52SW	*	*	2230	3180	3750	1970	1030	*	12.1	91.3
W58PW	*	*	*	*	*	*	*	*	*	*
W09PW	*	*	*	*	*	*	10.4	*	*	*
W24SW	*	*	*	*	22.1	56.6	62.6	*	*	*
W24SW dup	*	*	33.7	*	18.7	72.0	77.9	*	*	*
W102SW	*	*	*	*	*	*	*	*	*	*
W02PW	*	*	*	*	*	*	*	*	*	*
W64SW	*	*	758	1200	1730	1060	825	*	12.3	56.7
[β]W54PW	*	*	2070	2100	2150	1180	680	*	46.4	56.5
W15PW	*	*	*	*	‡15.8	12.2	42.6	*	*	*
[β]W62PW	*	*	*	*	*	*	*	*	*	*
[β]W22PW	*	*	*	*	*	*	*	*	*	*
[β]W11PW	*	*	*	*	*	*	34.6	12.0	12.7	26.4
W60PW	*	*	149	77.2	150	57.2	98.1	151	56.5	33.9
W36SW	54.2	‡12.4	389	393	505	333	236	‡30.3	16.7	38.2
W36SW dup	*	21.8	397	407	511	369	274	19.8	17.7	41.2
W12PW	*	*	6410	5220	3970	2330	1260	*	87.5	76.6
W29SW	*	*	*	*	*	*	*	‡21.1	*	14.8
W31SW	*	*	30.1	*	*	*	44.6	31.7	*	26.0
W30SW	*	*	24.1	*	13.7	*	40.0	31.5	*	13.5
W08PW	*	*	*	*	*	*	*	*	*	*
W35SW	*	*	*	*	*	*	14.4	*	*	9.51

US EPA ARCHIVE DOCUMENT

Sample Name	C10	C9	PFOA	C7	C6	C5	C4	PFOS	PFHS	PFBS
[β]W01PW	*	*	*	*	*	*	24.1	*	*	[‡]10.1
W48SW	*	*	26.0	*	16.4	17.2	33.0	*	*	*
W13SW	*	27.7	321	234	182	76.4	62.5	*	*	13.4
W34SW	*	16.2	204	73.6	103	162	234	*	*	*
W26SW	*	*	67.9	30.0	141	305	394	*	*	[‡]11.2
W17PW	*	*	*	*	*	*	13.2	*	*	*
W17PW dup	*	*	*	*	*	*	13.8	*	*	*
W57SW	*	*	32.2	*	*	*	10.7	*	*	*
W47SW	*	*	1250	1360	1310	478	330	*	40.6	63.9
W50SW	*	40.0	1160	715	762	354	199	*	*	54.5
W44SW	*	*	11000	8250	6710	3770	1750	*	218	208
W45SW	129	26.4	176	61.0	69.4	143	194	38.2	*	*
W41SW	*	*	90.5	*	50.6	90.7	102	*	*	*
W49SW	*	*	35.7	*	42.3	28.3	29.4	*	*	*
W19PW	*	*	*	*	*	*	11.6	*	*	*

* Values below the limit of quantitation (LOQ) – cannot be assumed to be zero.

‡ Values flagged for having confirmatory/quantification ion ratios more than 2 standard deviations away from mean values determined for standards

† W03SW sample collected in a container intended for water transfer only

β indicates sample from a well used for drinking water

dup indicates duplicate sample

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

Appendix 1.

Chain of custody forms (CoC) for water samples collected, shipped and received by the analytical laboratory

USEPA Contract Laboratory Program
Generic Chain of Custody

Original

Reference Case: R
Client No:

Region:	4	Date Shipped:	2/19/2009	Chain of Custody Record	Sampler Signature:
Project Code:	09-0227	Carrier Name:	FedEx	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code:	09-0227	Airbill:	865935737060	1	2/23/09
CERCLIS ID:		Shipped to:	NERL RTP 109 TW Alexander Dr Building E, Room E-178 Durham NC 27711 (919) 541-3706	2.	
Spill ID:				3.	
Site Name/State:	LAS Near Decatur AL Private Well PFC			4.	
Project Leader:	Mike Neill				
Action:					
Sampling Co:	PL-SESD				

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	QC Type
W01-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	1 (HNO3) (1)	W01-PW	S: 2/19/2009 13:12	--
W02-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	2 (HNO3) (1)	W02-PW	S: 2/18/2009 10:48	--
W06-PW	Groundwater/ Marty Allen	L/G	PFCs (0)	4 (HNO3) (1)	W06-PW	S: 2/17/2009 17:00	--
W06-PW-DUP	Groundwater/ Marty Allen	L/G	PFCs (0)	5 (HNO3) (1)	W06-PW-DUP	S: 2/17/2009 17:00	--
W07-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	6 (HNO3) (1)	W07-PW	S: 2/19/2009 13:44	--
W08-PW	Groundwater/ Art Masters	L/G	PFCs (0)	7 (HNO3) (1)	W08-PW	S: 2/18/2009 9:05	--
W09-PW	Groundwater/ Art Masters	L/G	PFCs (0)	8 (HNO3) (1)	W09-PW	S: 2/19/2009 11:49	--
W101-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	10 (HNO3) (1)	W101-PW	S: 2/18/2009 14:05	--
W102-SW	Surface Water/ Kevin Simmons	L/G	PFCs (0)	11 (HNO3) (1)	W102-SW	S: 2/18/2009 14:23	--
W10-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	9 (HNO3) (1)	W10-SW	S: 2/18/2009 9:35	--
W11-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	12 (HNO3) (1)	W11-PW	S: 2/17/2009 15:57	--

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s): Marty Allen	Chain of Custody Seal Number:
Analysis Key:	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced?
PFCs = Perfluorinated Compounds			

TR Number: 4-043013577-021909-0001

REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.

Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

USEPA Contract Laboratory Program
Generic Chain of Custody

Reference Case: R
Client No:

Region:	4	Date Shipped:	2/19/2009	Chain of Custody Record	Sampler Signature:
Project Code:	09-0227	Carrier Name:	FedEx	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code:	09-0227	Airbill:	865935737060	1	2/23/09
CERCLIS ID:		Shipped to:	NERL RTP 109 TW Alexander Dr Building E, Room E-178 Durham NC 27711 (919) 541-3706	2.	
Spill ID:				3.	
Site Name/State:	LAS Near Decatur AL Private Well PFC			4.	
Project Leader:	Mike Neill				
Action:					
Sampling Co:	PL-SESD				

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	QC Type
W12-PW	Groundwater/ Art Masters	L/G	PFCs (0)	13 (HNO3) (1)	W12-PW	S: 2/18/2009 11:50	--
W13-SW	Surface Water/ Art Masters	L/G	PFCs (0)	14 (HNO3) (1)	W13-SW	S: 2/18/2009 11:20	--
W14-PW	Groundwater/ Marty Allen	L/G	PFCs (0)	15 (HNO3) (1)	W14-PW	S: 2/18/2009 14:15	--
W15-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	16 (HNO3) (1)	W15-PW	S: 2/17/2009 16:38	--
W17-PW	Groundwater/ Marty Allen	L/G	PFCs (0)	17 (HNO3) (1)	W17-PW	S: 2/19/2009 11:16	--
W17-PW-DUP	Groundwater/ Marty Allen	L/G	PFCs (0)	18 (HNO3) (1)	W17-PW-DUP	S: 2/19/2009 11:16	--
W19-PW	Groundwater/ Marty Allen	L/G	PFCs (0)	19 (HNO3) (1)	W19-PW	S: 2/19/2009 13:10	--
W22-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	20 (HNO3) (1)	W22-PW	S: 2/18/2009 10:00	--
W24-SW	Surface Water/ Kevin Simmons	L/G	PFCs (0)	21 (HNO3) (1)	W24-SW	S: 2/19/2009 9:05	--
W24-SW-DUP	Surface Water/ Kevin Simmons	L/G	PFCs (0)	22 (HNO3) (1)	W24-SW-DUP	S: 2/19/2009 9:05	--
W26-SW	Surface Water/ Art Masters	L/G	PFCs (0)	23 (HNO3) (1)	W26-SW	S: 2/18/2009 14:00	--

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key:	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced?

PFCs = Perfluorinated Compounds

TR Number: 4-043013577-021909-0001

REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.

Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

USEPA Contract Laboratory Program
Generic Chain of Custody

Reference Case: R
Client No:

Region:	4	Date Shipped:	2/19/2009	Chain of Custody Record	Sampler Signature:
Project Code:	09-0227	Carrier Name:	FedEx	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code:	09-0227	Airbill:	865935737060	1	[illegible] 2/[illegible]
CERCLIS ID:		Shipped to:	NERL RTP 109 TW Alexander Dr Building E, Room E-178 Durham NC 27711 (919) 541-3706	2.	
Spill ID:				3.	
Site Name/State:	LAS Near Decatur AL Private Well PFC			4.	
Project Leader:	Mike Neill				
Action:					
Sampling Co:	PL-SESD				

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME		QC Type
W27-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	24 (HNO3) (1)	W27-SW	S: 2/19/2009	13:15	--
W28-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	25 (HNO3) (1)	W28-SW	S: 2/18/2009	10:05	--
W29-SW	Surface Water/ Art Masters	L/G	PFCs (0)	26 (HNO3) (1)	W29-SW	S: 2/17/2009	15:54	--
W30-SW	Surface Water/ Art Masters	L/G	PFCs (0)	27 (HNO3) (1)	W30-SW	S: 2/17/2009	16:35	--
W31-SW	Surface Water/ Art Masters	L/G	PFCs (0)	28 (HNO3) (1)	W31-SW	S: 2/17/2009	16:11	--
W32-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	29 (HNO3) (1)	W32-SW	S: 2/17/2009	15:55	--
W33-SW	Surface Water/ Art Masters	L/G	PFCs (0)	30 (HNO3) (1)	W33-SW	S: 2/18/2009	14:55	--
W34-SW	Surface Water/ Art Masters	L/G	PFCs (0)	31 (HNO3) (1)	W34-SW	S: 2/18/2009	10:05	--
W35-SW	Surface Water/ Art Masters	L/G	PFCs (0)	32 (HNO3) (1)	W35-SW	S: 2/18/2009	9:20	--
W36-SW	Surface Water/ Art Masters	L/G	PFCs (0)	33 (HNO3) (1)	W36-SW	S: 2/18/2009	9:35	--
W36-SW-DUP	Surface Water/ Art Masters	L/G	PFCs (0)	34 (HNO3) (1)	W36-SW-DUP	S: 2/18/2009	9:35	--

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s): [illegible] Marty [illegible]	Chain of Custody Seal Number:
Analysis Key:	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____
PFCs = Perfluorinated Compounds			

TR Number: 4-043013577-021909-0001

REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.

Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

USEPA Contract Laboratory Program
Generic Chain of Custody

Reference Case: R
Client No:

Region:	4	Date Shipped:	2/19/2009	Chain of Custody Record	Sampler Signature:
Project Code:	09-0227	Carrier Name:	FedEx	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code:	09-0227	Airbill:	865935737060	1	2/23/09
CERCLIS ID:		Shipped to:	NERL RTP 109 TW Alexander Dr Building E, Room E-178 Durham NC 27711 (919) 541-3706	2.	
Spill ID:				3.	
Site Name/State:	LAS Near Decatur AL Private Well PFC			4.	
Project Leader:	Mike Neill				
Action:					
Sampling Co:	PL-SESD				

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME		QC Type
W41-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	35 (HNO3) (1)	W41-SW	S: 2/18/2009	11:25	--
W42-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	36 (HNO3) (1)	W42-SW	S: 2/18/2009	11:35	--
W43-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	37 (HNO3) (1)	W43-SW	S: 2/18/2009	11:10	--
W44-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	38 (HNO3) (1)	W44-SW	S: 2/18/2009	14:35	--
W45-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	39 (HNO3) (1)	W45-SW	S: 2/18/2009	13:35	--
W46-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	40 (HNO3) (1)	W46-SW	S: 2/18/2009	13:20	--
W47-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	41 (HNO3) (1)	W47-SW	S: 2/18/2009	12:55	--
W48-SW	Surface Water/ Art Masters	L/G	PFCs (0)	42 (HNO3) (1)	W48-SW	S: 2/18/2009	10:50	--
W49-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	43 (HNO3) (1)	W49-SW	S: 2/19/2009	14:25	--
W50-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	44 (HNO3) (1)	W50-SW	S: 2/19/2009	14:50	--
W51-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	R4-56 (HNO3) (1)	W51-SW	S: 2/19/2009	15:15	--

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: PFCs = Perfluorinated Compounds	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced?

TR Number: 4-043013577-021909-0001

PR provides preliminary results. Requests for preliminary results will increase analytical costs.

REGION COPY

Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

USEPA Contract Laboratory Program
Generic Chain of Custody

Reference Case: R

Client No:

Region:	4	Date Shipped:	2/19/2009	Chain of Custody Record	Sampler Signature:
Project Code:	09-0227	Carrier Name:	FedEx	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code:	09-0227	Airbill:	865935737060	1	2/23/09
CERCLIS ID:		Shipped to:	NERL RTP 109 TW Alexander Dr Building E, Room E-178 Durham NC 27711 (919) 541-3706	2.	
Spill ID:				3.	
Site Name/State:	LAS Near Decatur AL Private Well PFC			4.	
Project Leader:	Mike Neill				
Action:					
Sampling Co:	PL-SESD				

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME		QC Type
W52-SW	Surface Water/ Art Masters	L/G	PFCs (0)	45 (HNO3) (1)	W52-SW	S: 2/18/2009	14:15	--
W53-SW	Surface Water/ Art Masters	L/G	PFCs (0)	46 (HNO3) (1)	W53-SW	S: 2/19/2009	10:25	--
W53-SW-DUP	Surface Water/ Art Masters	L/G	PFCs (0)	47 (HNO3) (1)	W53-SW-DUP	S: 2/19/2009	10:25	--
W54-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	48 (HNO3) (1)	W54-PW	S: 2/18/2009	9:25	--
W57-SW	Surface Water/ Marty Allen	L/G	PFCs (0)	49 (HNO3) (1)	W57-SW	S: 2/19/2009	9:55	--
W58-PW	Groundwater/ Art Masters	L/G	PFCs (0)	50 (HNO3) (1)	W58-PW	S: 2/19/2009	10:45	--
W60-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	51 (HNO3) (1)	W60-PW	S: 2/17/2009	17:25	--
W61-SW	Surface Water/ Art Masters	L/G	PFCs (0)	52 (HNO3) (1)	W61-SW	S: 2/19/2009	13:30	--
W62-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	53 (HNO3) (1)	W62-PW	S: 2/18/2009	15:16	--
W63-PW	Groundwater/ Kevin Simmons	L/G	PFCs (0)	54 (HNO3) (1)	W63-PW	S: 2/19/2009	11:10	--
W64-SW	Surface Water/ Kevin Simmons	L/G	PFCs (0)	55 (HNO3) (1)	W64-SW	S: 2/19/2009	10:10	--

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: PFCs = Perfluorinated Compounds	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced?

TR Number: 4-043013577-021909-0001

REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.

Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

USEPA Contract Laboratory Program
Generic Chain of Custody

Reference Case: R
Client No:

Region:	4	Date Shipped:	2/19/2009	Chain of Custody Record	Sampler Signature:
Project Code:	09-0227	Carrier Name:	FedEx	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code:	09-0227	Airbill:	865935737060	1	2/23/09
CERCLIS ID:		Shipped to:	NERL RTP 109 TW Alexander Dr Building E, Room E-178 Durham NC 27711 (919) 541-3706	2.	
Spill ID:				3.	
Site Name/State:	LAS Near Decatur AL Private Well PFC			4.	
Project Leader:	Mike Neill				
Action:					
Sampling Co:	PL-SESD				

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	QC Type
WO3-SW	Surface Water/ Kevin Simmons	L/G	PFCs (0)	3 (HNO3) (1)	W03-SW	S: 2/19/2009 14:00	--

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: PFCs = Perfluorinated Compounds	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced?

TR Number: 4-043013577-021909-0001

REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.

Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

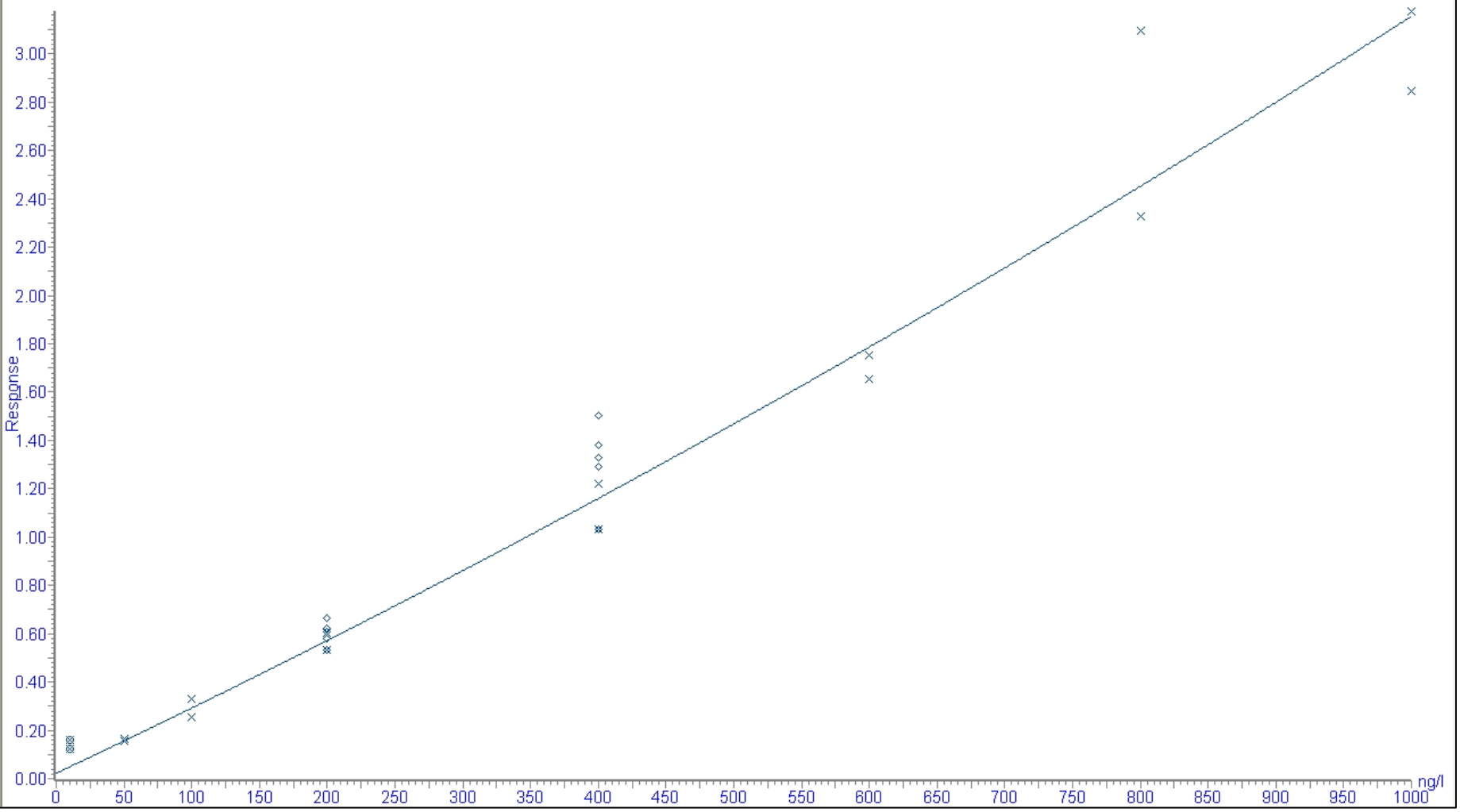
US EPA ARCHIVE DOCUMENT

Appendix 2.

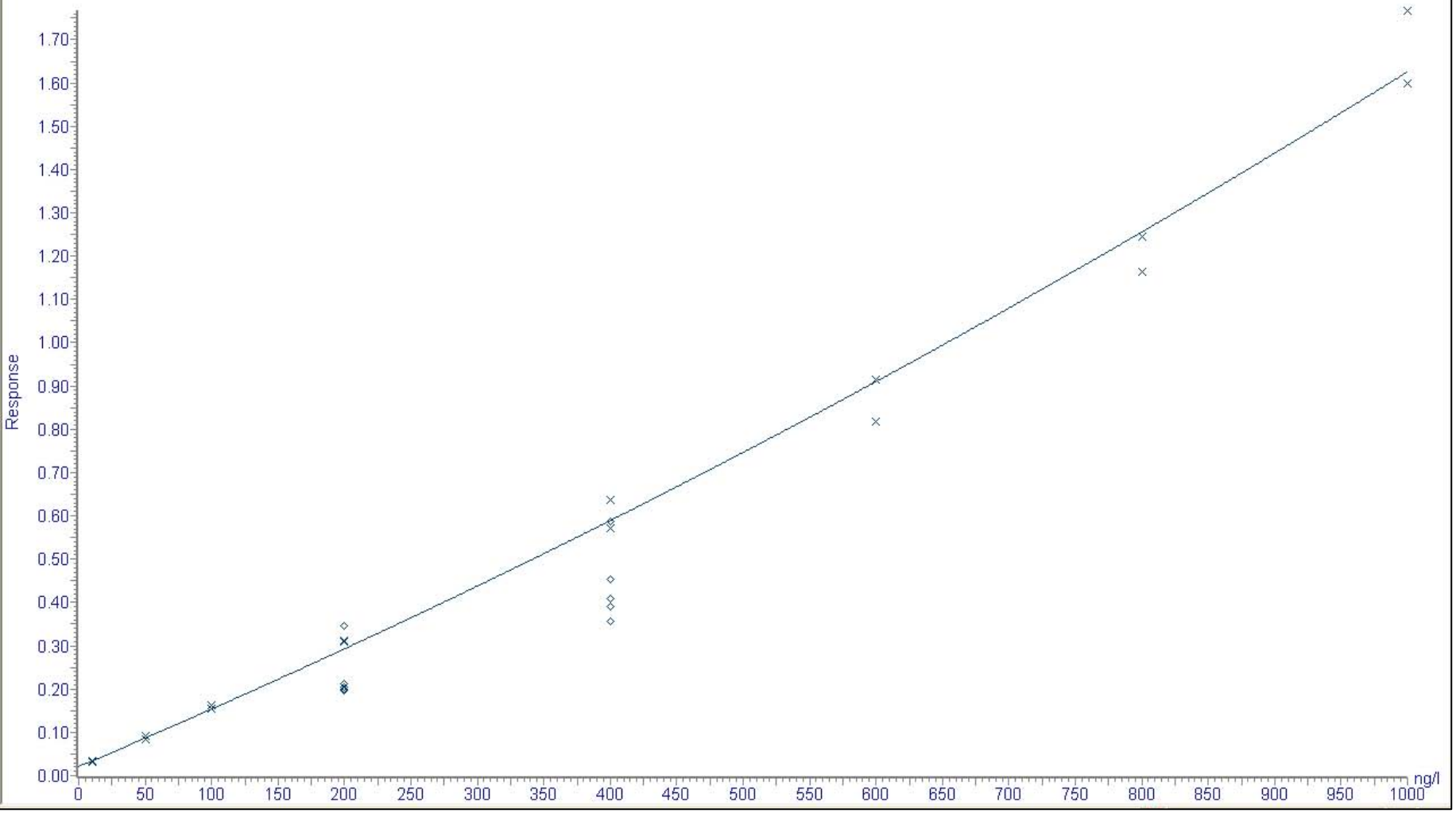
Calibration curves for all target analytes

X's correspond to standard points, ◊'s correspond to Quality Control samples at the 200 or 400 ng/L levels

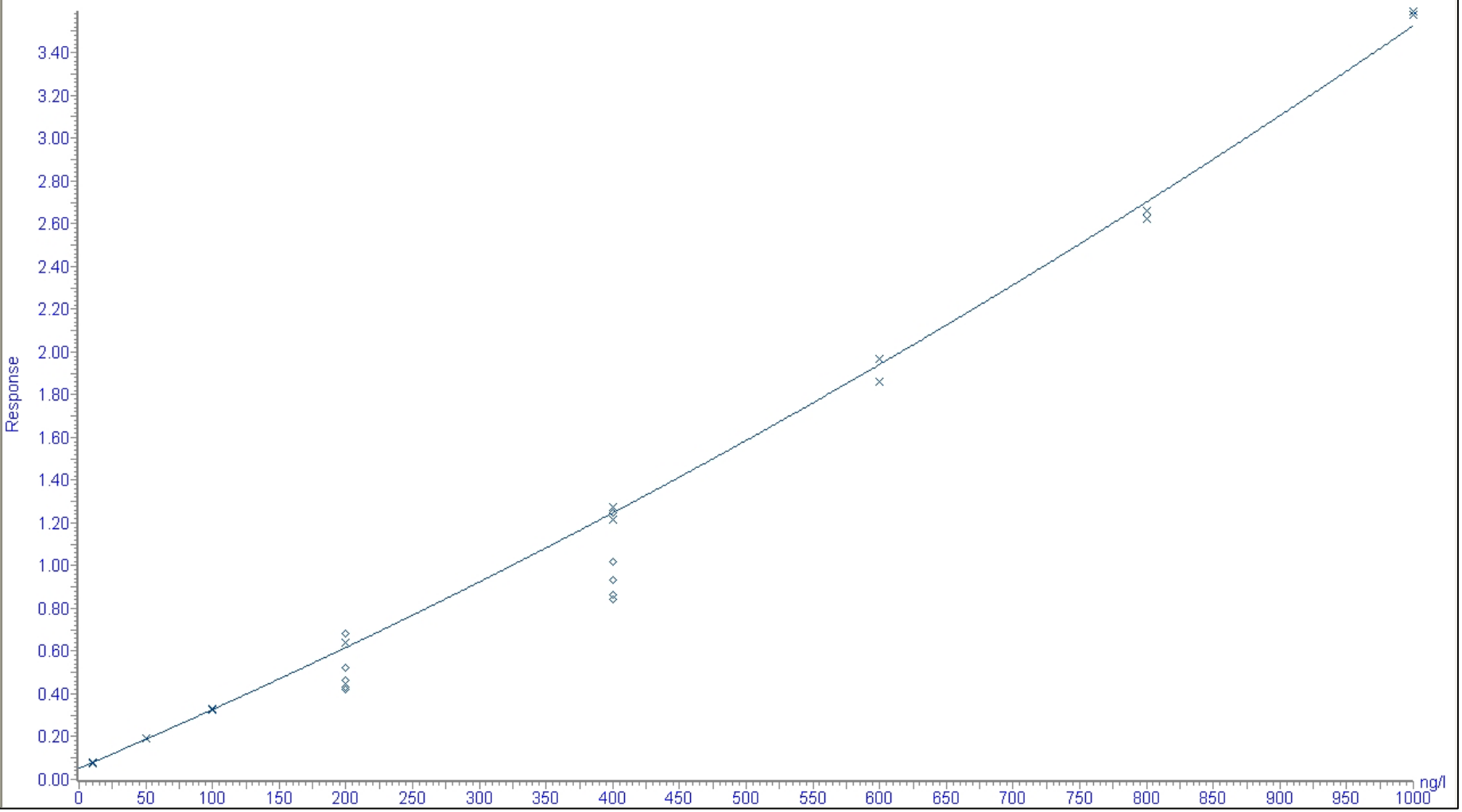
Compound name: C10
Coefficient of Determination: R^2 = 0.979851
Calibration curve: 4.84144e-007 * x^2 + 0.00264988 * x + 0.0225038
Response type: Internal Std (Ref 1), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



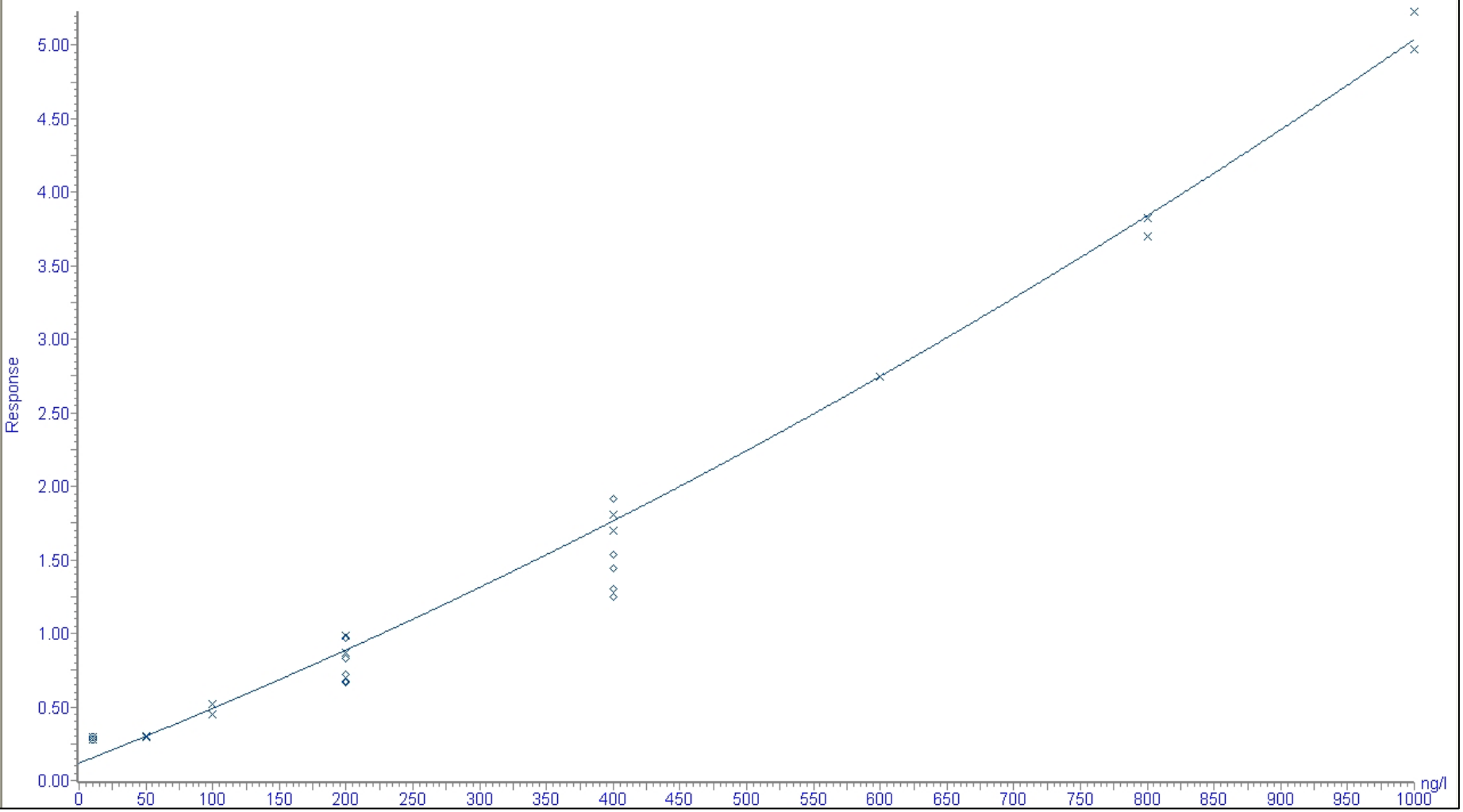
Compound name: C9
Coefficient of Determination: R^2 = 0.995520
Calibration curve: 3.07374e-007 * x^2 + 0.00129746 * x + 0.0210335
Response type: Internal Std (Ref 5), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



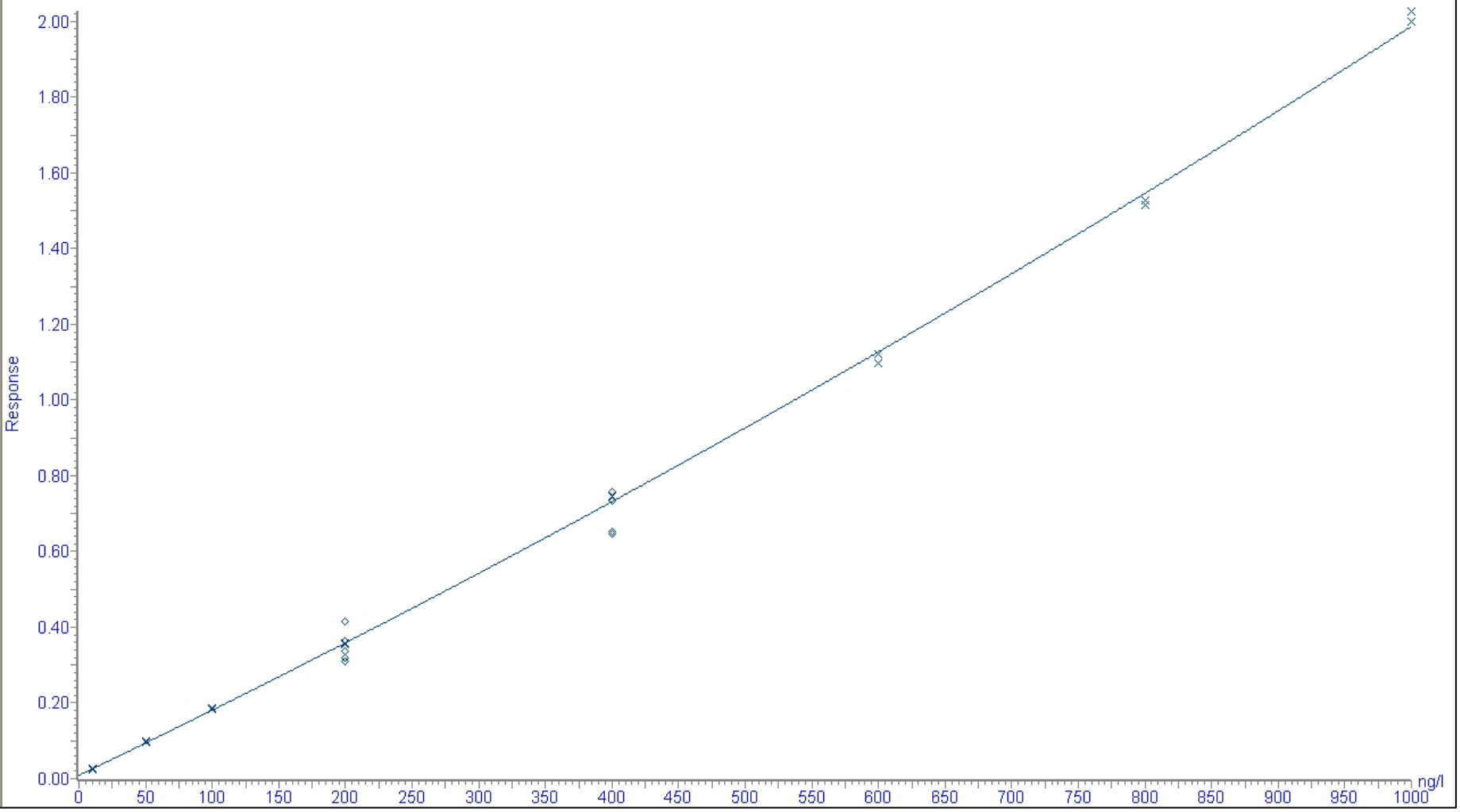
Compound name: PFOA
Coefficient of Determination: R^2 = 0.999293
Calibration curve: 8.13297e-007 * x^2 + 0.00266009 * x + 0.0526481
Response type: Internal Std (Ref 5), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



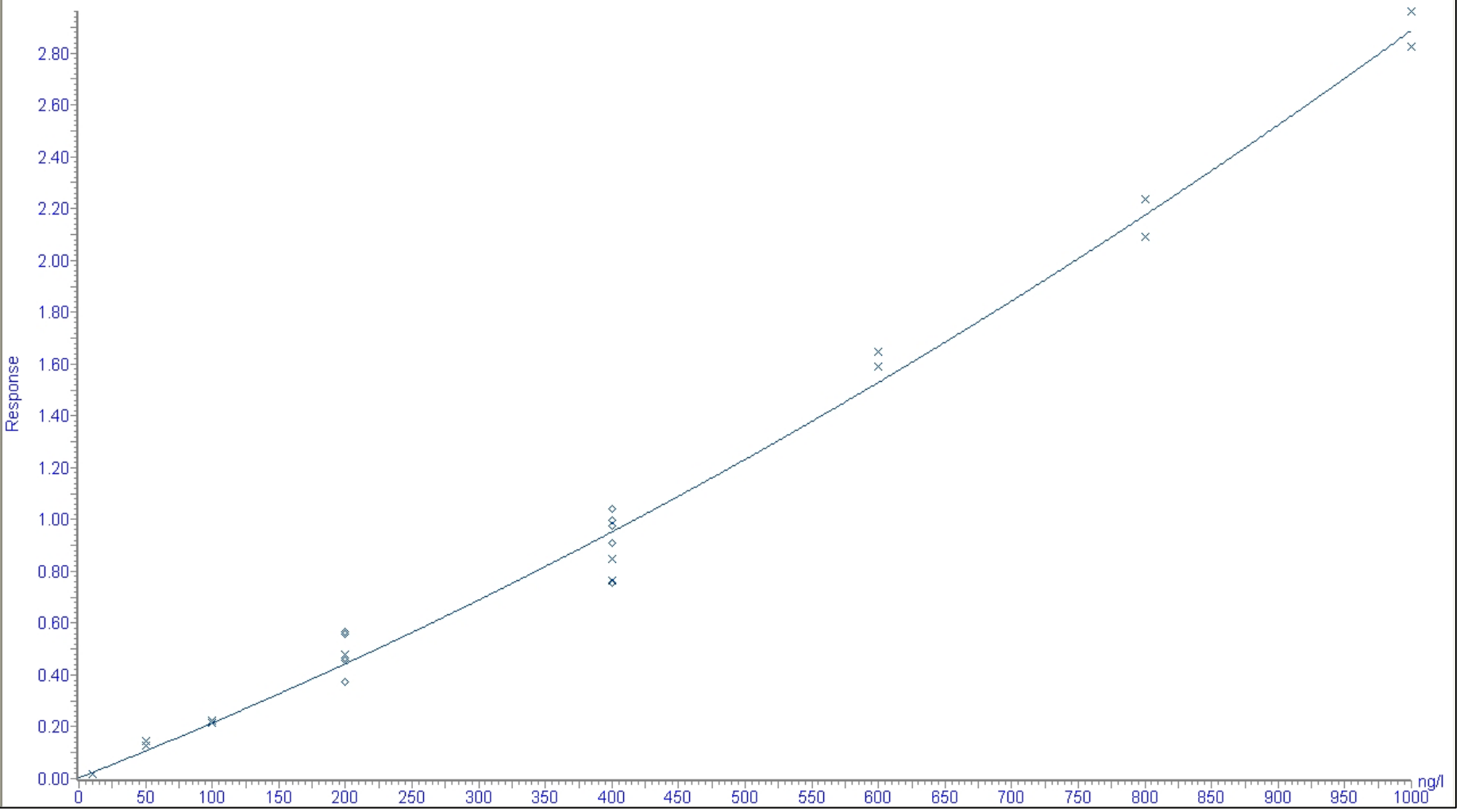
Compound name: C7
Coefficient of Determination: R^2 = 0.998188
Calibration curve: 1.34265e-006 * x^2 + 0.00357712 * x + 0.118878
Response type: Internal Std (Ref 5), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



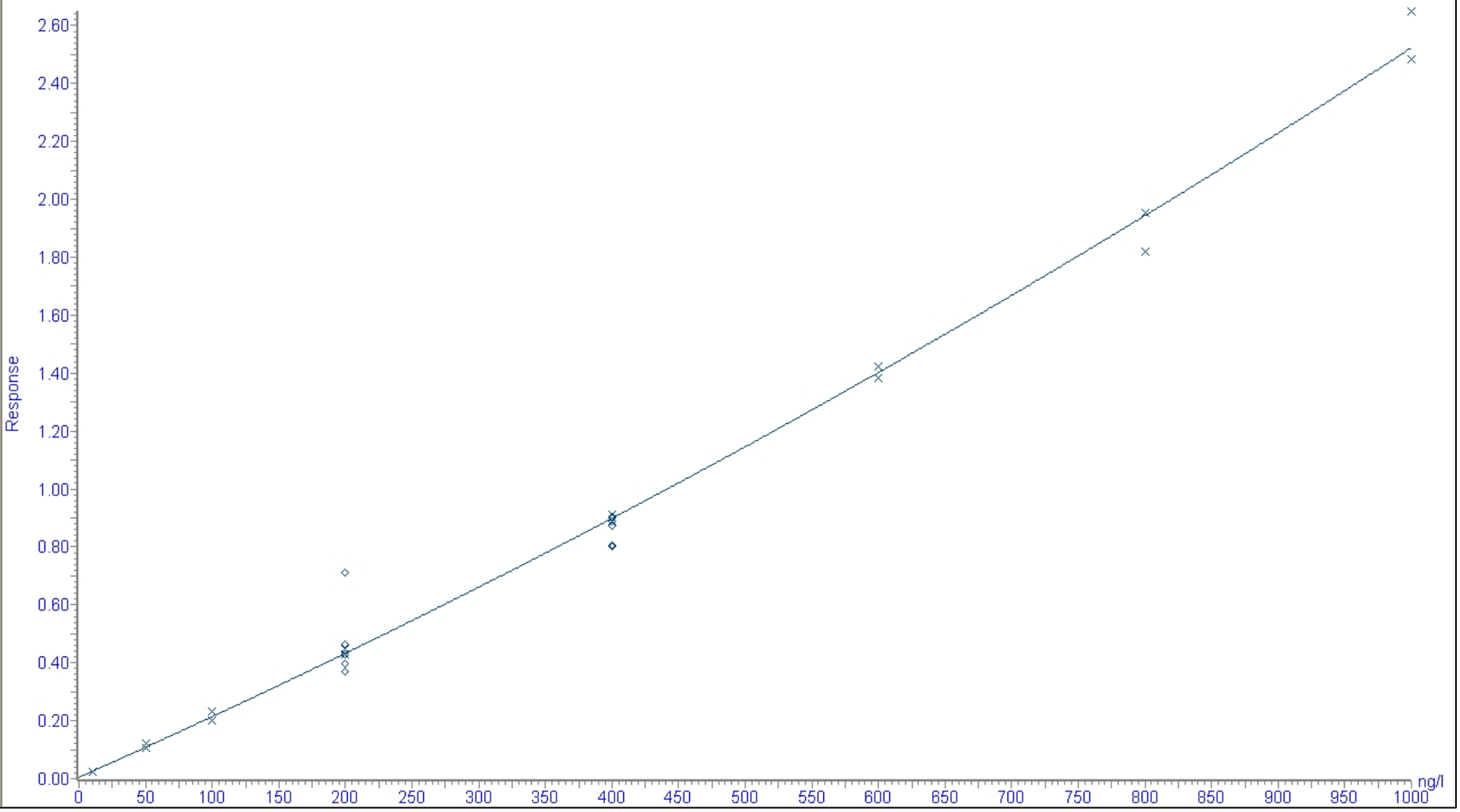
Compound name: C6
Coefficient of Determination: R^2 = 0.999621
Calibration curve: 2.86242e-007 * x^2 + 0.00169204 * x + 0.00881371
Response type: Internal Std (Ref 10), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



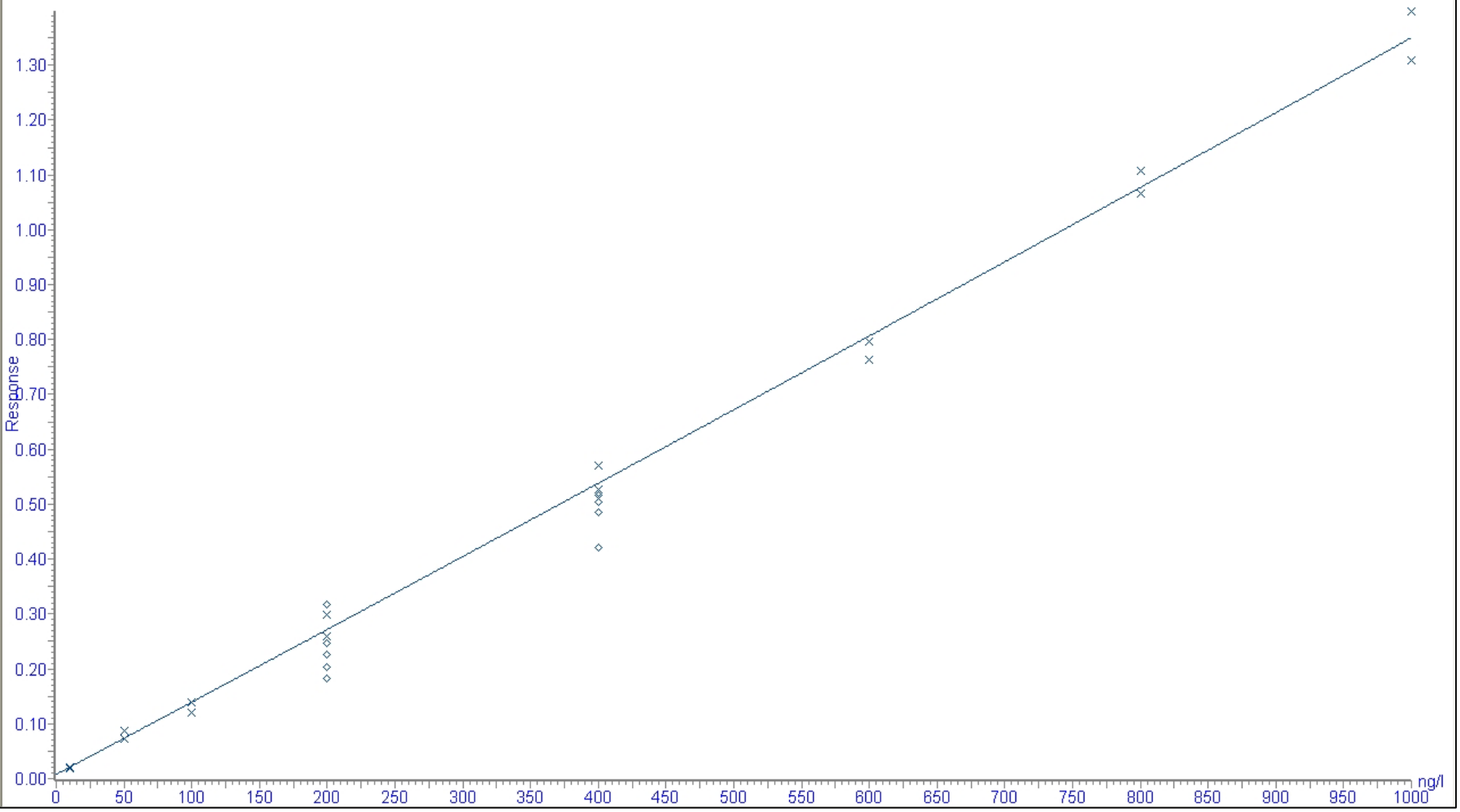
Compound name: C5
Coefficient of Determination: R^2 = 0.994211
Calibration curve: 8.52527e-007 * x^2 + 0.00203243 * x + 0.00293018
Response type: Internal Std (Ref 10), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



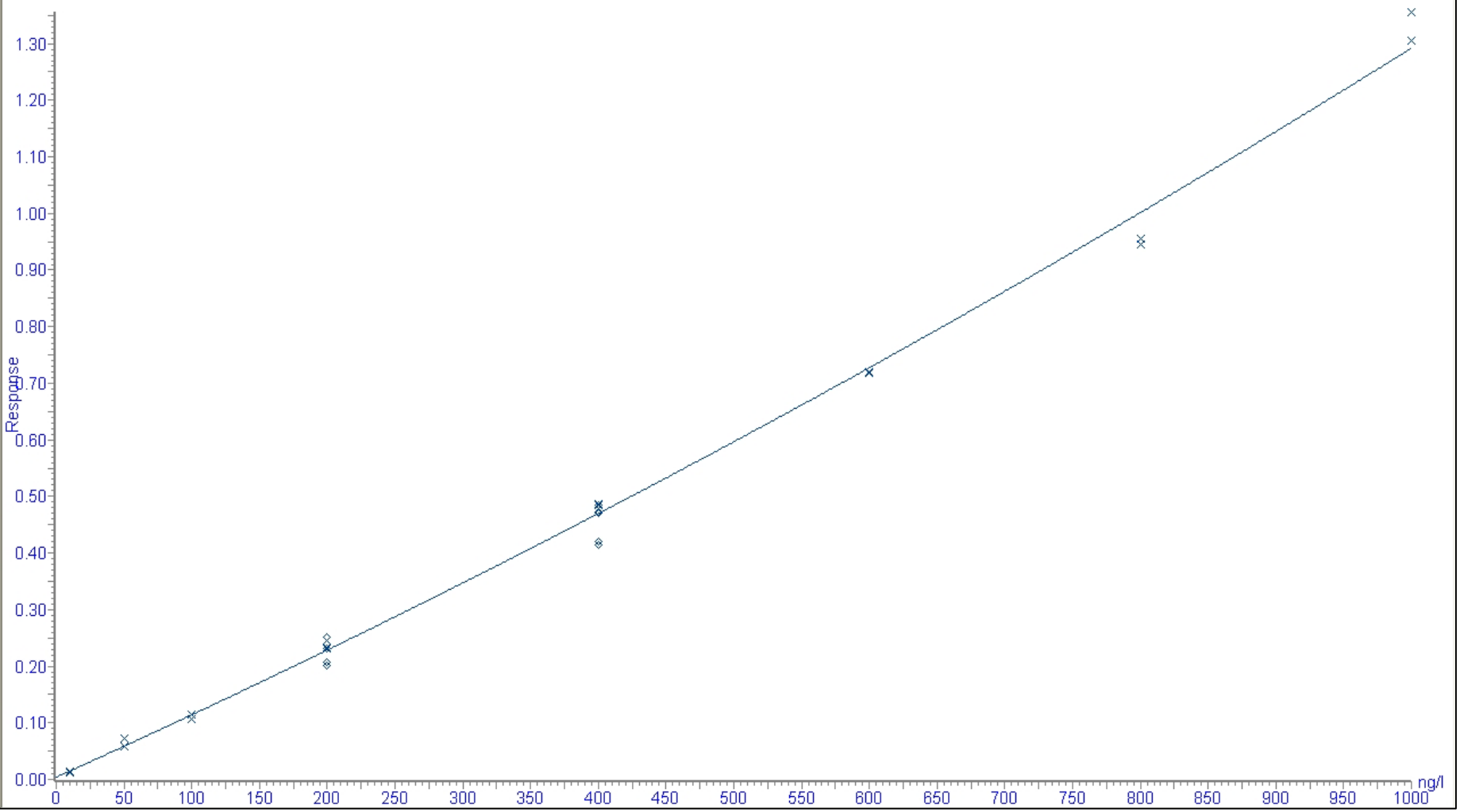
Compound name: C4
Coefficient of Determination: R^2 = 0.998470
Calibration curve: 4.73404e-007 * x^2 + 0.00204506 * x + 0.00485309
Response type: Internal Std (Ref 10), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



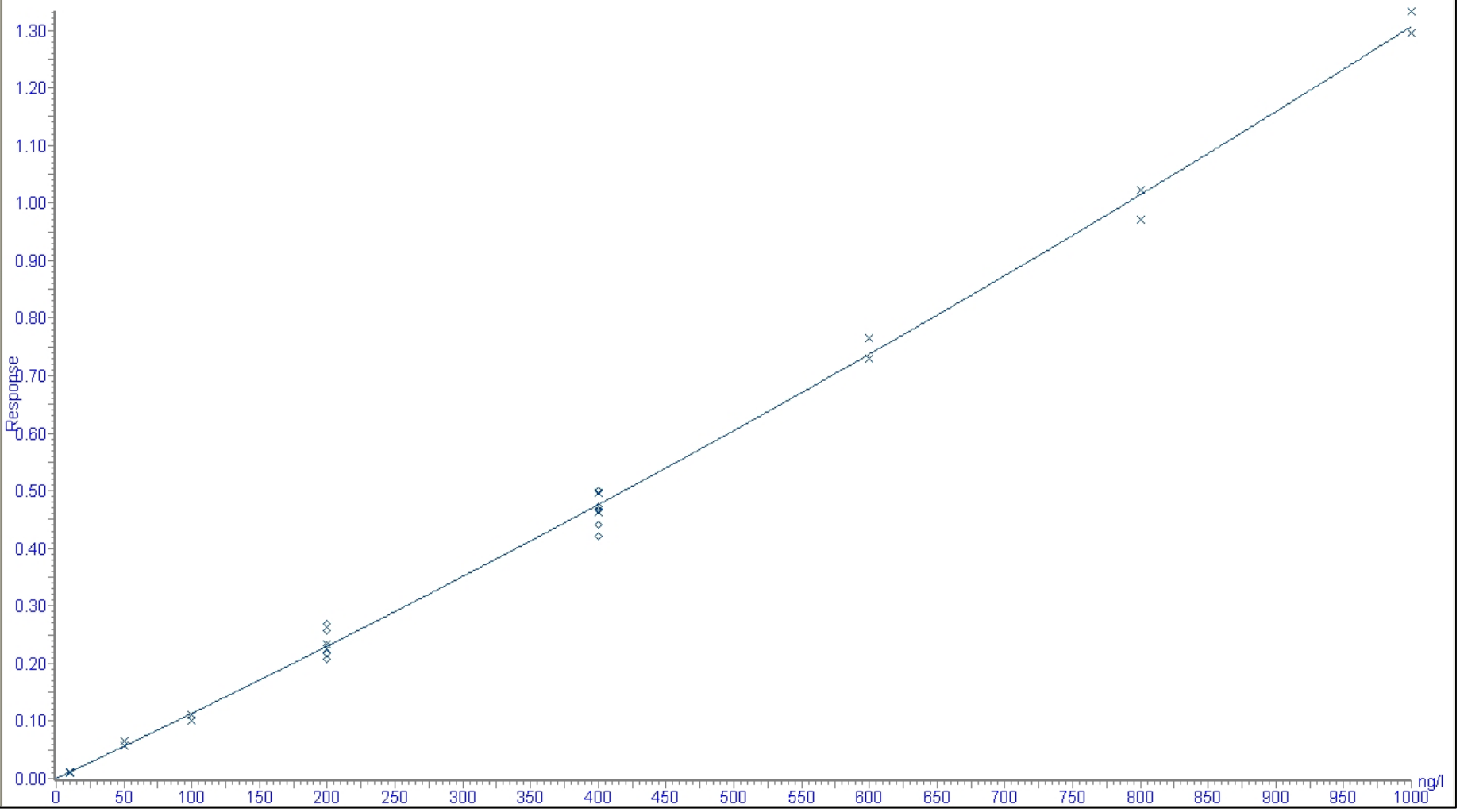
Compound name: PFOS
Coefficient of Determination: R^2 = 0.997563
Calibration curve: 2.63976e-008 * x^2 + 0.00131589 * x + 0.0075659
Response type: Internal Std (Ref 12), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound name: PFHS
Coefficient of Determination: R^2 = 0.997949
Calibration curve: 2.05369e-007 * x^2 + 0.00108281 * x + 0.00381447
Response type: Internal Std (Ref 14), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



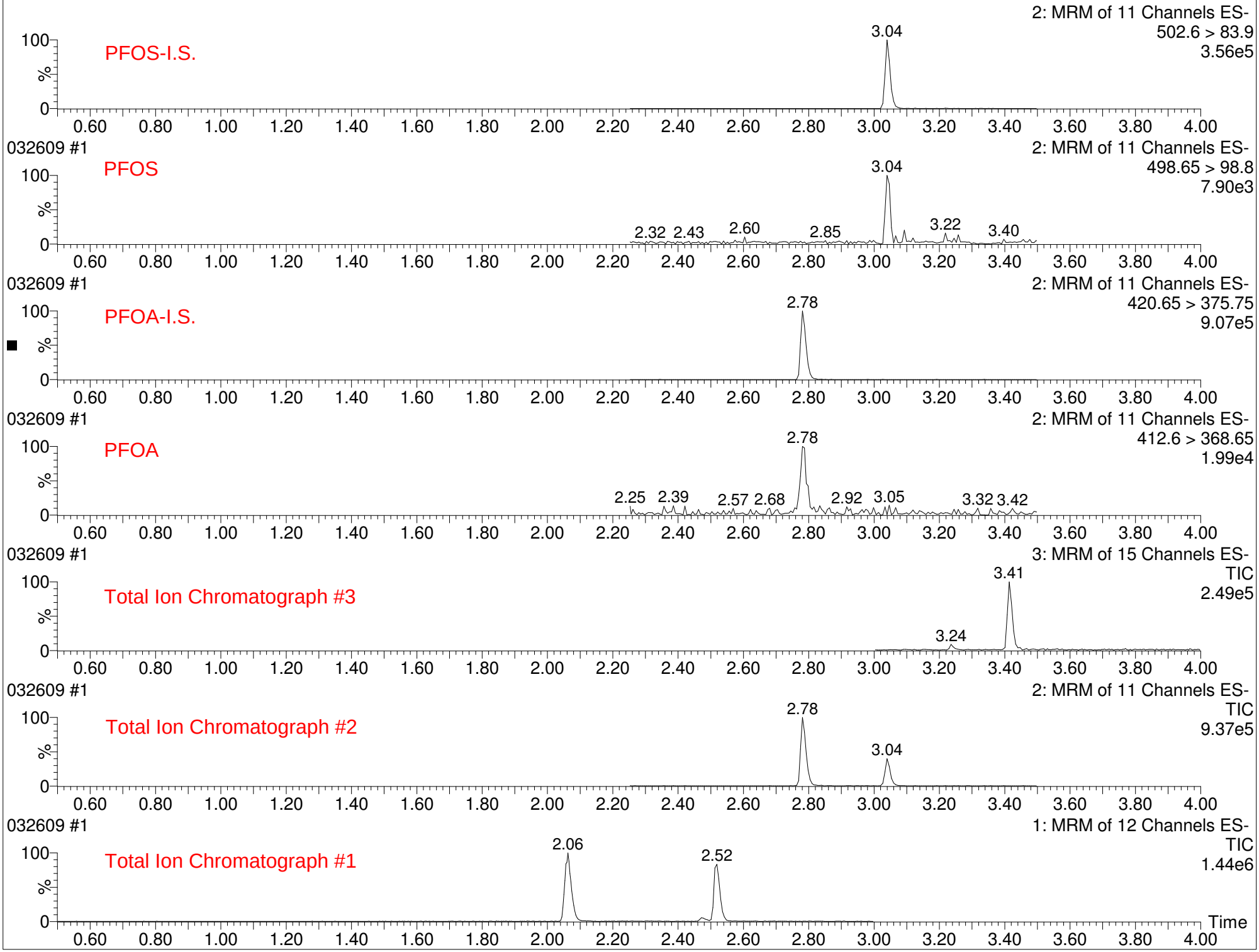
Compound name: PFBS
Coefficient of Determination: R^2 = 0.998882
Calibration curve: 1.95929e-007 * x^2 + 0.00111091 * x + 0.000157238
Response type: Internal Std (Ref 14), Area * (IS Conc. / IS Area)
Curve type: 2nd Order, Origin: Exclude, Weighting: 1/x, Axis trans: None



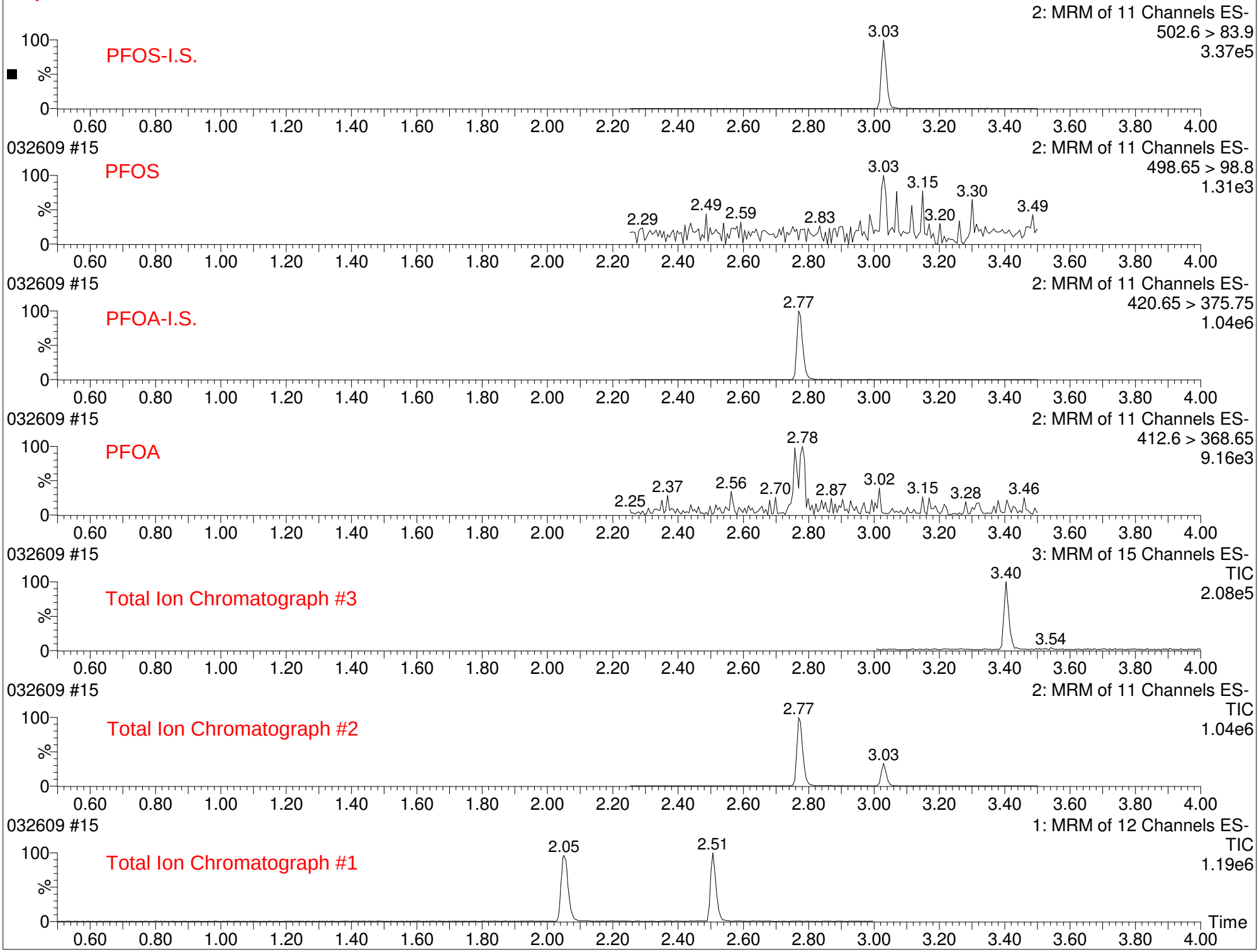
US EPA ARCHIVE DOCUMENT

Appendix 3. Mass spectral data from 3 blank samples, 3 spiked samples, and 3 field samples

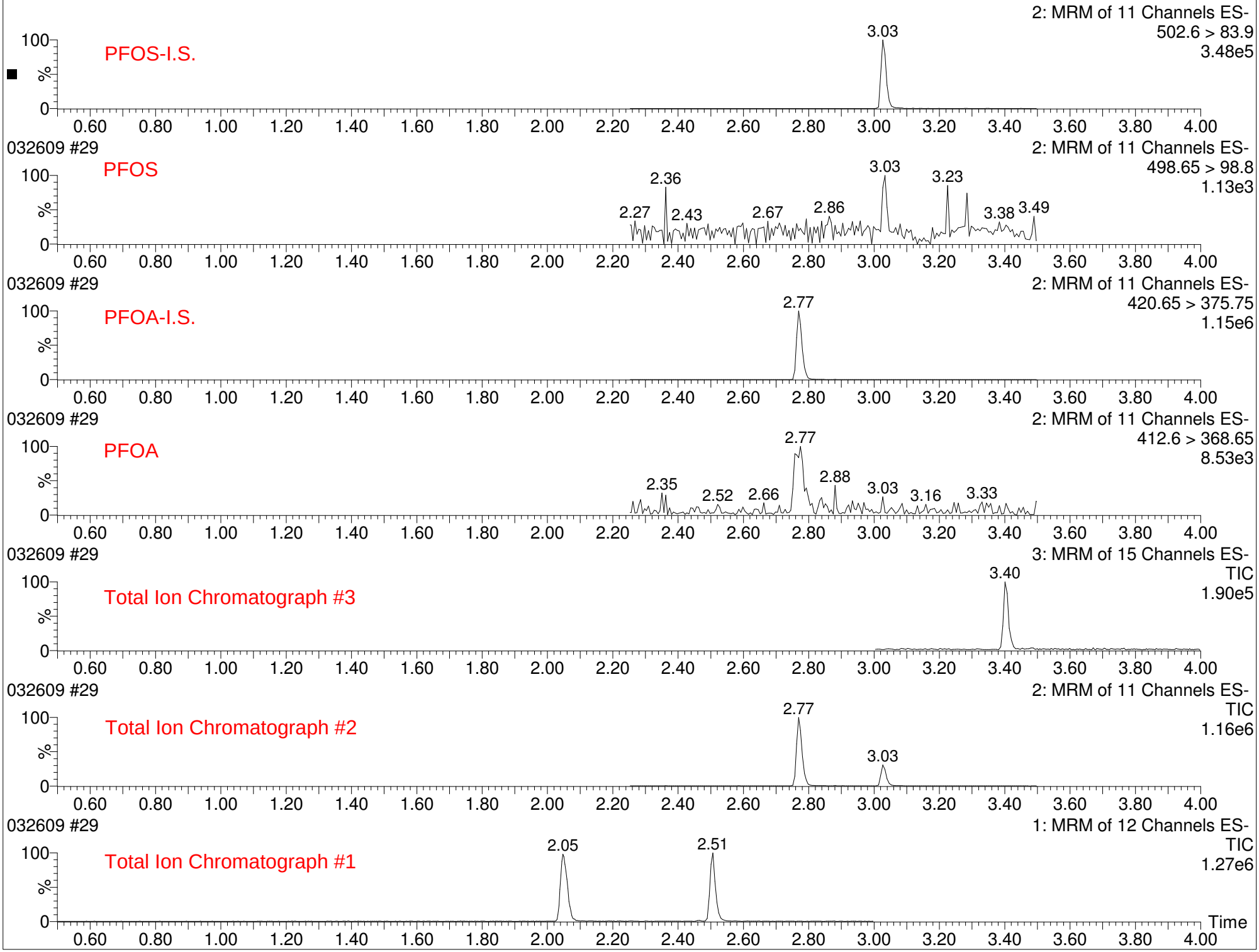
Trip Blank #1


PFOS-I.S.
2: MRM of 11 Channels ES-
502.6 > 83.9
3.56e5
3.04
032609 #1
PFOS
2: MRM of 11 Channels ES-
498.65 > 98.8
7.90e3
3.04
2.32 2.43 2.60 2.85 3.22 3.40
032609 #1
PFOA-I.S.
2: MRM of 11 Channels ES-
420.65 > 375.75
9.07e5
2.78
032609 #1
PFOA
2: MRM of 11 Channels ES-
412.6 > 368.65
1.99e4
2.78
2.25 2.39 2.57 2.68 2.92 3.05 3.32 3.42
032609 #1
Total Ion Chromatograph #3
3: MRM of 15 Channels ES-
TIC
2.49e5
3.41
3.24
032609 #1
Total Ion Chromatograph #2
2: MRM of 11 Channels ES-
TIC
9.37e5
2.78
3.04
032609 #1
Total Ion Chromatograph #1
1: MRM of 12 Channels ES-
TIC
1.44e6
2.06
2.52
100
%
0
0.60 0.80 1.00 1.20 1.40 1.60 1.80 2.00 2.20 2.40 2.60 2.80 3.00 3.20 3.40 3.60 3.80 4.00
Time

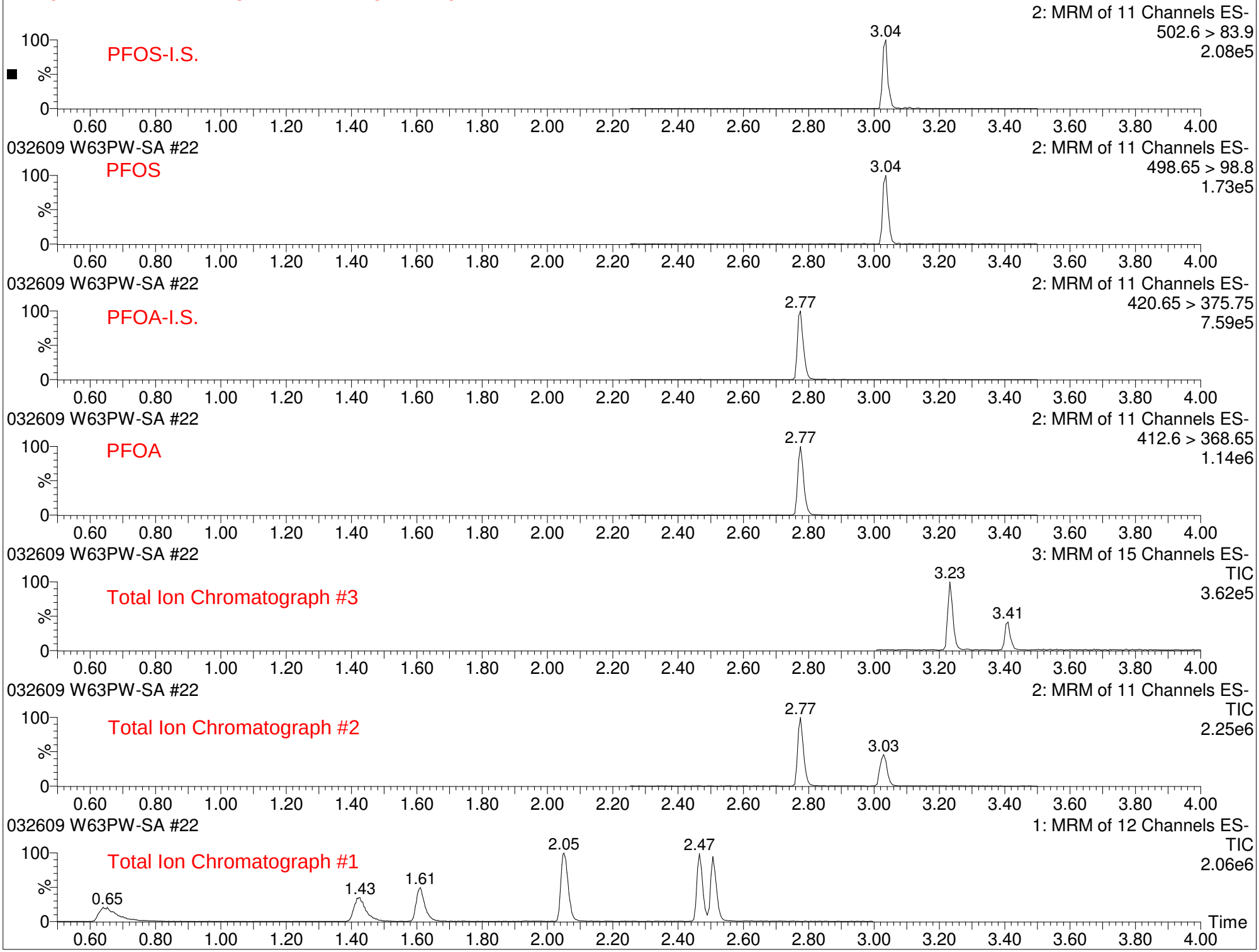
Trip Blank #2


PFOS-I.S.
2: MRM of 11 Channels ES-
502.6 > 83.9
3.37e5
3.03
032609 #15
PFOS
2: MRM of 11 Channels ES-
498.65 > 98.8
1.31e3
2.29
2.49
2.59
2.83
3.03
3.15
3.20
3.30
3.49
032609 #15
PFOA-I.S.
2: MRM of 11 Channels ES-
420.65 > 375.75
1.04e6
2.77
032609 #15
PFOA
2: MRM of 11 Channels ES-
412.6 > 368.65
9.16e3
2.25
2.37
2.56
2.70
2.78
2.87
3.02
3.15
3.28
3.46
032609 #15
Total Ion Chromatograph #3
3: MRM of 15 Channels ES-
TIC
2.08e5
3.40
3.54
032609 #15
Total Ion Chromatograph #2
2: MRM of 11 Channels ES-
TIC
1.04e6
2.77
3.03
032609 #15
Total Ion Chromatograph #1
1: MRM of 12 Channels ES-
TIC
1.19e6
2.05
2.51
Time
100
%
0
0.60
0.80
1.00
1.20
1.40
1.60
1.80
2.00
2.20
2.40
2.60
2.80
3.00
3.20
3.40
3.60
3.80
4.00

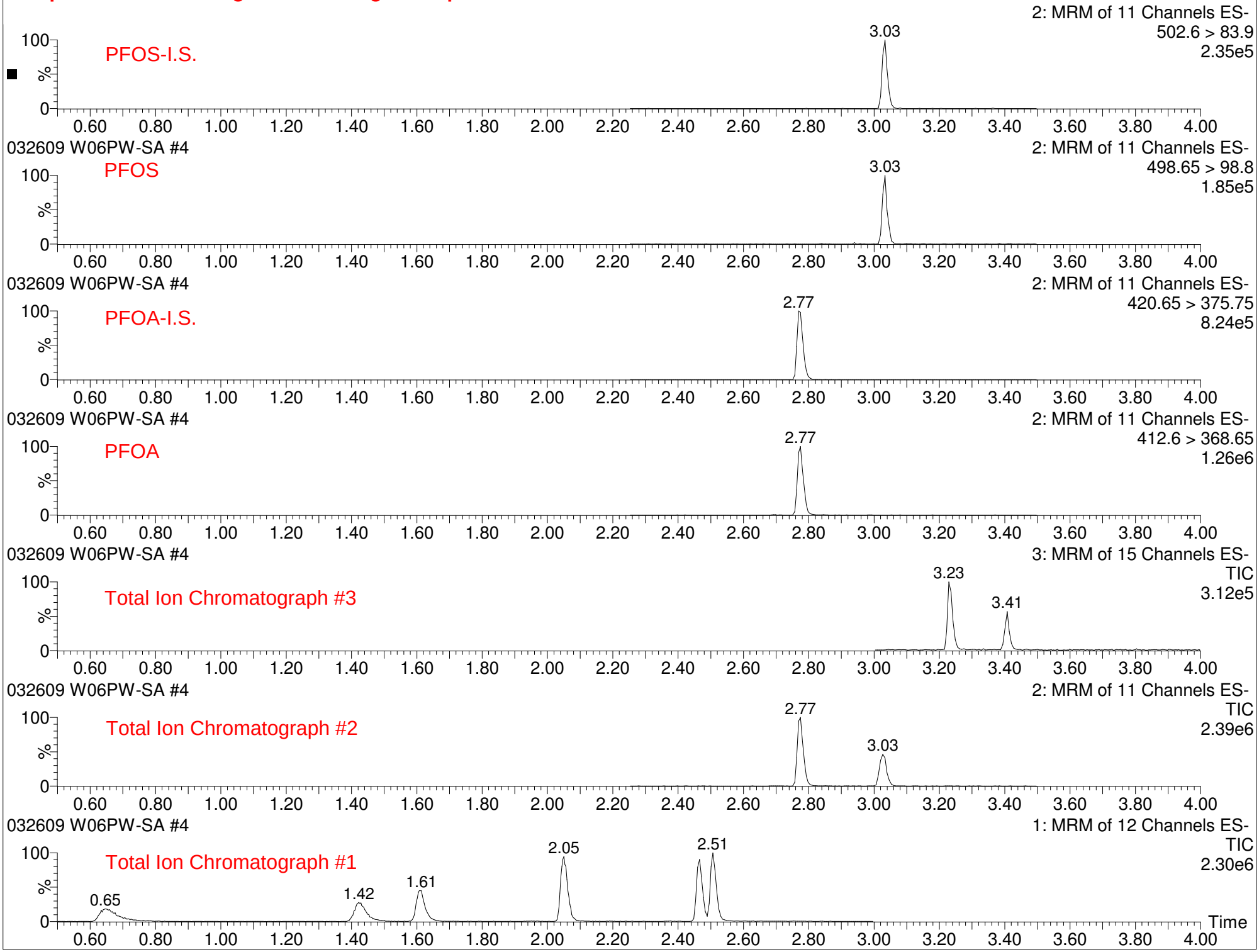
Trip Blank #3


PFOS-I.S.
2: MRM of 11 Channels ES-
502.6 > 83.9
3.48e5
3.03
032609 #29
PFOS
2: MRM of 11 Channels ES-
498.65 > 98.8
1.13e3
2.27
2.36
2.43
2.67
2.86
3.03
3.23
3.38
3.49
032609 #29
PFOA-I.S.
2: MRM of 11 Channels ES-
420.65 > 375.75
1.15e6
2.77
032609 #29
PFOA
2: MRM of 11 Channels ES-
412.6 > 368.65
8.53e3
2.35
2.52
2.66
2.77
2.88
3.03
3.16
3.33
032609 #29
Total Ion Chromatograph #3
3: MRM of 15 Channels ES-
TIC
1.90e5
3.40
032609 #29
Total Ion Chromatograph #2
2: MRM of 11 Channels ES-
TIC
1.16e6
2.77
3.03
032609 #29
Total Ion Chromatograph #1
1: MRM of 12 Channels ES-
TIC
1.27e6
2.05
2.51
100
%
0
0.60
0.80
1.00
1.20
1.40
1.60
1.80
2.00
2.20
2.40
2.60
2.80
3.00
3.20
3.40
3.60
3.80
4.00
Time

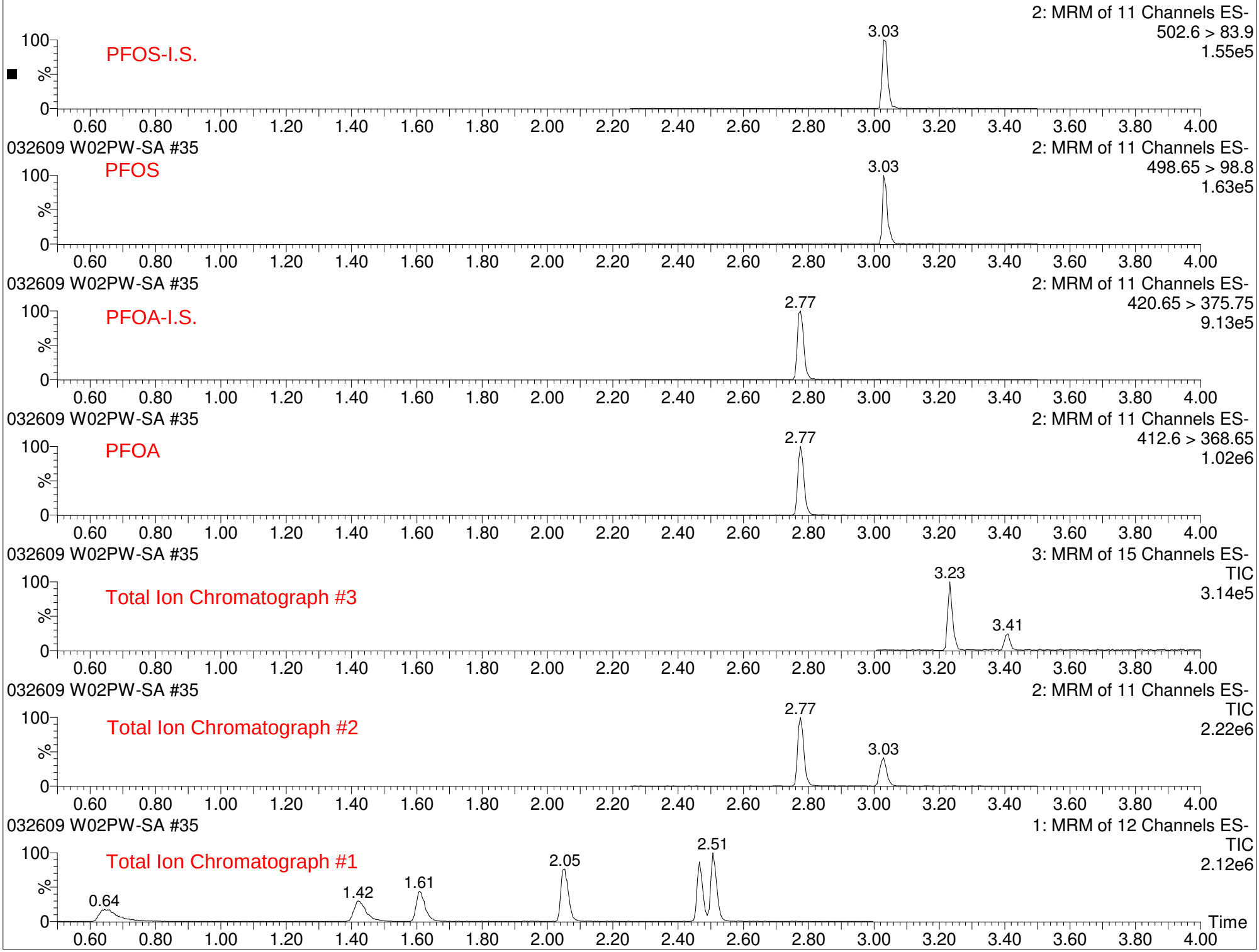
Sample W63PW + 400 ng/L of each target compound



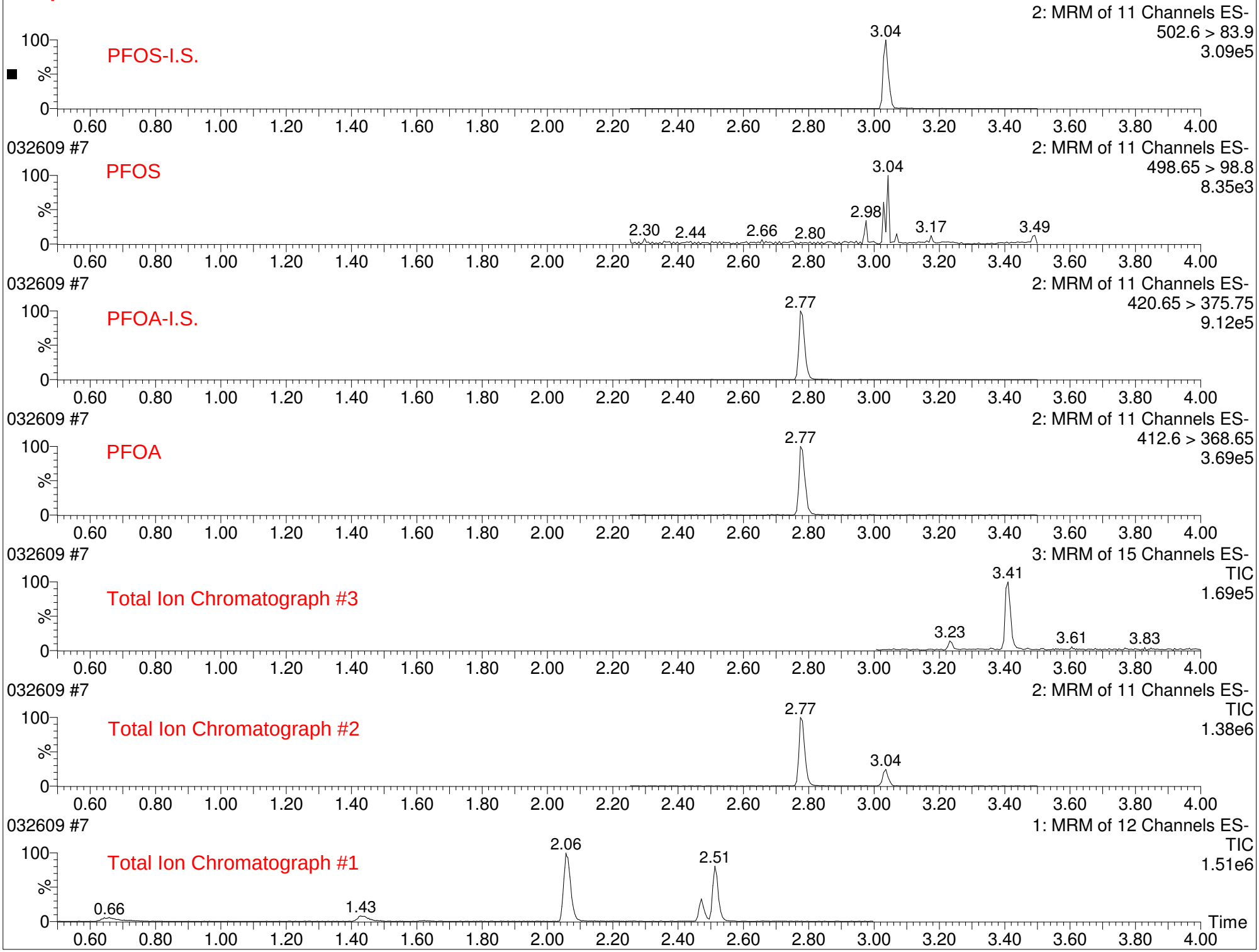
Sample W06PW + 400 ng/L of each target compound



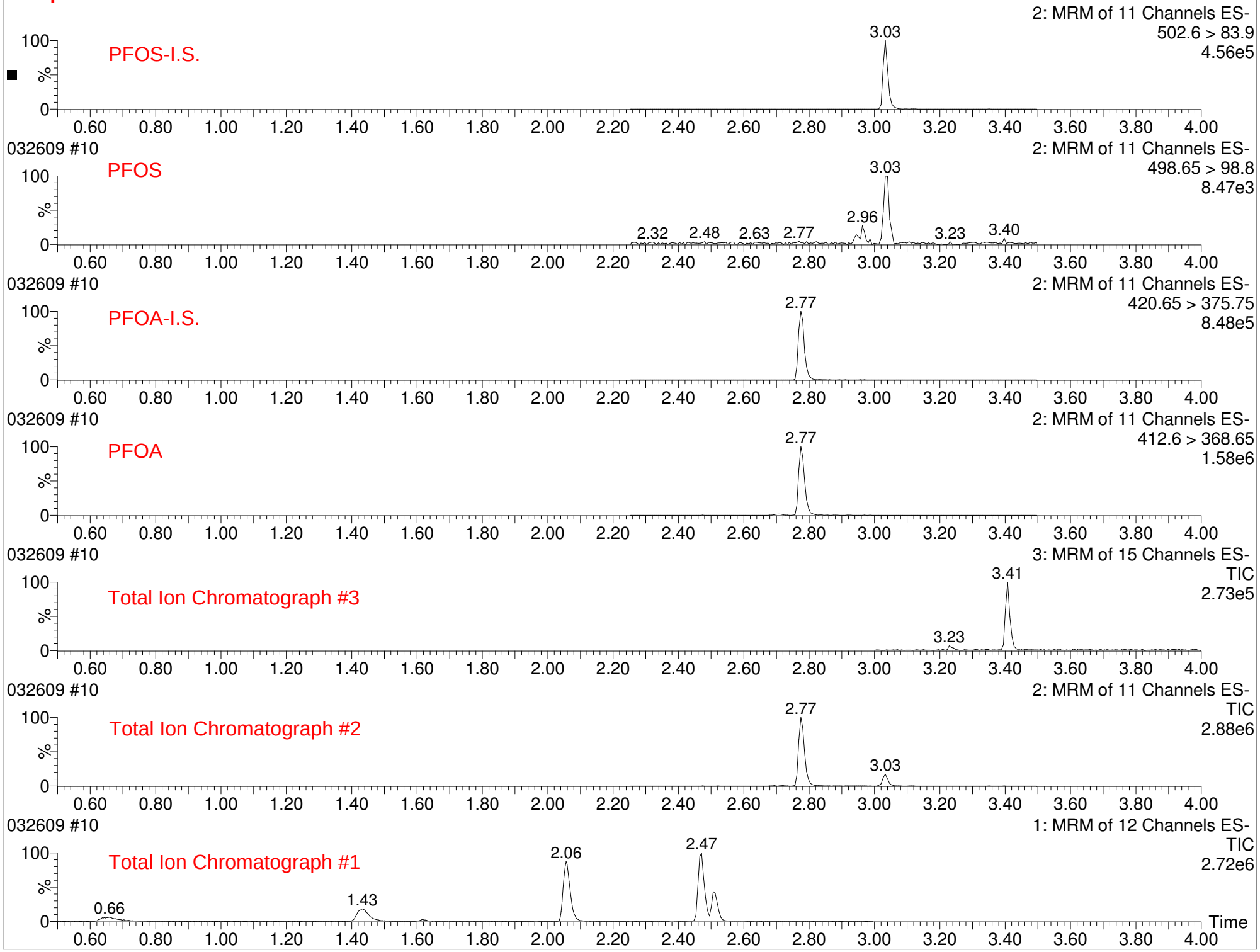
Sample W02PW + 400 ng/L of each target compound



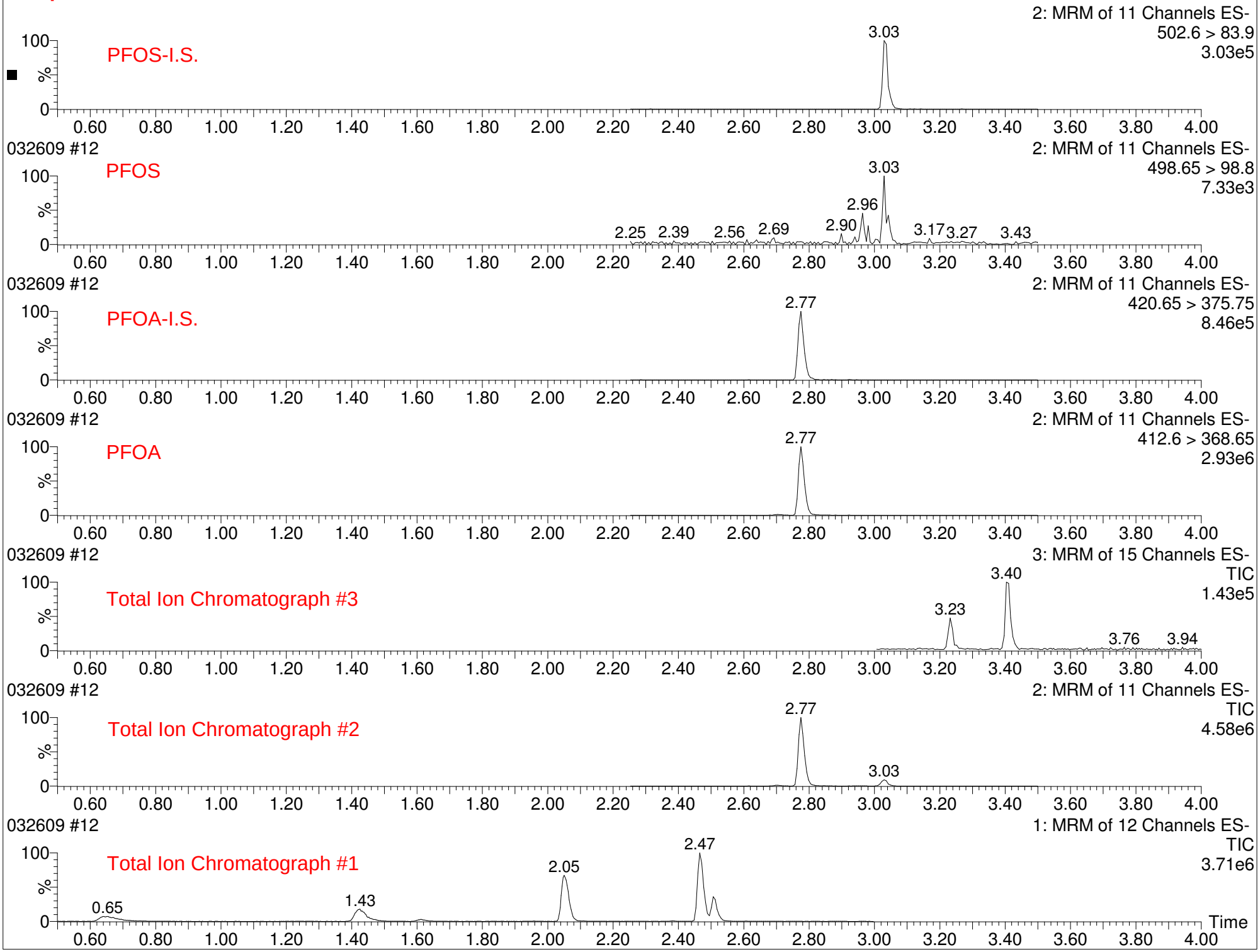
Sample W27SW



Sample W14PW



Sample W42SW



US EPA ARCHIVE DOCUMENT

United States
Environmental Protection
Agency

Office of Research and Development
(8101R)
Washington, DC 20460

Official Business
Penalty for Private Use
$300

EPA 600/C-09/003
April 2009
www.epa.gov

Recycled/Recyclable
Printed with vegetable-based ink on paper that contains a minimum of 50% post-consumer fiber and is processed chlorine free.

US EPA ARCHIVE DOCUMENT