3M Specialty Materials

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January 29, 2003

VIA FEDERAL EXPRESS

Document Control Office (DCO)
Office of Pollution Prevention and Toxics (OPPT)
US Environmental Protection Agency
EPA East, Room 6428
1201 Constitution Avenue, NW
Washington, DC 20460

Attention: Docket No. AR-226 and the FYI Docket

Dear Sir or Madam:

This continues 3M's voluntary submissions of data on perfluorooctane sulfonates and related compounds, as part of our ongoing dialogue with EPA regarding fluorochemicals.

Included in this submission are the following:

1. Assay Validation Report, Quantitative Determination of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA, and PFHS in Human Serum by LC/MS/MS, Northwest Bioanalytical (NWB), Study No. NWBS98-082, Report No. NWBR99-005, May 13, 1999.

2. Assay Revalidation Report, Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS, Northwest Bioanalytical (NWB), Study No. NWBS00-040, Report No. NWBR00-108, January 24, 2001.

3. Assay Revalidation Addendum Report, Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS, Northwest Bioanalytical (NWB), Study No. NWBS00-040, Report No. NWBR00-122, November 20, 2001.

4. Sample Analysis Report for Protocol EPI-0011, Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS, Northwest Bioanalytical (NWB), Study No. NWBS00-088, Report No. NWBR01-001, November 30, 2001.

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5. Sample Analysis Report for Protocol EPI-0013, Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS, Northwest Bioanalytical (NWB), Study No. NWBS00-128, Report No. NWBR01-066, January 9, 2002.

6. Sample Analysis Report for Protocol EPI-0016, Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS, Northwest Bioanalytical (NWB), Study No. NWBS00-145, Report No. NWBR01-155, January 10, 2002.

The first three reports listed above refer to the assay validation methods developed by Northwest Bioanalytical. The fourth, fifth and sixth reports listed are the final analytical reports from Northwest Bioanalytical of the children, adult and elderly serum study data, respectively. The statistical analyses of these data have been previously submitted to the EPA by 3M.

We will continue to provide information as it becomes available. Please feel free to contact me if you have any questions.

Very truly yours,

Michael A. Santoro
Director of Environmental, Health
Safety and Regulatory Affairs
Specialty Materials
3M Center, Building 236-1B-10
St. Paul, MN 55144
651-733-6374 (phone)
651-733-1958 (fax)
E-mail: masantoro@mmm.com

cc: Dr. Charles Auer
Dr. Oscar Hernandez

Study No. NWBS98-082
Report No. NWBR99-005

AR226-1208

Quantitative Determination of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in Human Serum by LC/MS/MS

Assay Validation Report

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Northwest Bioanalytical (NWB)
A Division of NWT Inc.
1121 East 3900 South
Salt Lake City, UT 84124

PREPARED FOR: 3M
935 Bush Avenue
St. Paul, MN 55133

AUTHOR:

David L. Vollmer, Ph.D., Project Manager — DATE: 5/13/99

APPROVED FOR RELEASE BY:

Rodger L. Foltz, Ph.D., Laboratory Director — DATE: 5/13/99

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Quantitative Determination of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in Human Serum by LC/MS/MS

Assay Validation Report

1. INTRODUCTION

Northwest Bioanalytical (NWB) was contracted by 3M to develop and validate a liquid chromatography/tandem mass spectrometry method for the measurement of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in human serum. For chemical names and structures, see the Analytical Method contained in section 6 of the report.

This report summarizes the analytical results from the validation of the method for quantitation of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in human serum for 3M. Kris Hansen, Ph.D., at 3M served as the Study Monitor. The following is a list of NWB supervisory personnel involved in the completion of this work: David L. Vollmer, Ph.D. (Project Manager); Brad Coopersmith, Ph.D. (Senior Scientist); Rodger L. Foltz, Ph.D., (NWB Laboratory Director).

3M SOP AMDT-S-12 (effective 12/12/95) and NWB SOPs were used in the conduct of this project and were available to project personnel in electronic or hard copy formats.

Date Study Initiated: January 15, 1999 Date Analyses Completed: February 10, 1999

The method validation study described in this report is not included within the definition of a GLP regulated nonclinical study (Title 21 of the US Code of Federal Regulations Part 58). However, Northwest Bioanalytical conducts all studies within the guidelines of GLP principles.

Background

Early method development experiments demonstrated varying levels of endogenous PFOS, PFOSAA, POAA, and PFHS in several lots of "blank" human serum. These endogenous levels complicate the use of human serum for the preparation of calibration standards and quality control samples.

To circumvent the difficulty of preparing standards and controls in the presence of endogenous levels, several animal matrices were evaluated. Rabbit serum showed the lowest endogenous levels of the aforementioned analytes when directly compared to rat, dog, and monkey serum. Utilizing a calibration curve prepared in rabbit serum, however, did not demonstrate acceptable precision and accuracy for the determination of PFOSA, MeFOSE-OH, and EtFOSE-OH.

A serum matrix that was pre-extracted from human serum to remove any endogenous levels of analytes prior to preparation of the curve was also evaluated. This serum matrix, however, showed greater extraction recoveries for all the analytes in the calibration curve samples than in the human serum samples that were not pre-extracted, and therefore did not demonstrate acceptable accuracy for quantitation of the analytes.

Because the four analytes (PFOS, PFOSAA, POAA, and PFHS) that did show acceptable precision and accuracy with the rabbit serum curve were the same four analytes that showed measurable endogenous concentrations in "blank" human serum, an abbreviated rabbit curve was used to calculate the endogenous levels in specific lots of human serum. Once the endogenous levels were determined for those lots of human serum, those concentrations were added to the spiked concentrations for the human serum QCs and for the human serum calibration standards. This procedure is described in detail within this report.

Principles of the Method

After the addition of HPLC-grade water and sodium dodecyl sulfate (SDS, internal standard) to 0.20 mL of human or rabbit serum, the serum mixture is made basic with the addition of 0.5 M tetrabutylammonium hydrogen sulfate (TBA, pH 10) and 0.25 M carbonate buffer. This mixture is then extracted with methyl-tert-butyl ether. After sufficient mixing, the sample is centrifuged. The organic layer is transferred via pipette into a clean test tube. The organic layer is then evaporated to dryness and the sample is reconstituted into 2 mM ammonium acetate water:methanol (50:50 v/v). The extracts are then analyzed by liquid chromatography/tandem mass spectrometry using negative-ion electrospray ionization and multiple reaction monitoring.

2. VALIDATION SUMMARY

Four separate analytical runs were used in the determination of linearity, precision, and accuracy of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS. The validation included determination of the stability of the analytes maintained under various storage conditions, the extraction efficiencies of the analytes and the internal standard, the system precision of the analytes, and the reproducibility of the analytes for this assay.

All values reported in the validation summary and tables are based upon the actual concentrations of solutions utilized during this validation. Please refer to the *List of Tables* for the location of the validation test data and summary statistics.

2.1. Range of Quantitation

Rabbit Serum Calibration Curve

Each analytical run included calibration standards in duplicate at a minimum of four different concentrations and at least two rabbit serum blanks. Data for PFOSA, N-MeFOSE-OH and N-EtFOSE-OH are not included because there were no endogenous amounts detected in human serum.

	PFOS	PFOSAA	POAA	PFHS
Range (ppb)	1.00 to 50.0	1.00 to 50.0	1.00 to 50.0	1.00 to 50.0
Mean Correlation Coefficient	0.9961	0.9957	0.9987	0.9982

Human Serum Calibration Curve

Each analytical run included calibration standards in duplicate at a minimum of seven different concentrations, a minimum of 10 quality control samples (QCs) (two levels in replicates of five), two "blanks" and two "0-ppb QCs" (QCs containing internal standard, but no spiked analytes).

	PFOS	PFOSA	PFOSAA	N-MeFOSE-OH	N-EtFOSE-OH	POAA	PFHS
Spiked Concentration Range	1.00 to 500 ppb	1.00 to 500 ppb	0.538 to 53.8 ppb	1.00 to 500 ppb	1.00 to 500 ppb	1.00 to 500 ppb	1.00 to 500 ppb
Mean Correlation Coefficient	0.9934	0.9969	0.9966	0.9971	0.9976	0.9972	0.9978

2.2. Precision and Accuracy

The precision and accuracy of the LC/MS/MS method for the quantitation of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in human serum were determined by analyzing two levels of quality controls in replicates of five on four separate days.

The intra-assay %CV for PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS were less than or equal to 13.0% for each QC concentration.

The inter-assay %CV for PFOS, PFOSA, PFOSAA, POAA and PFHS were less than or equal to 13.3% for each QC concentration. The mean percent theoretical for all levels of quality controls ranged from 95.7 to 106.7.

The inter-assay %CV for N-MeFOSE-OH and N-EtFOSE-OH were less than or equal to 25.5% for each QC concentration. The mean percent theoretical for all levels of quality controls ranged from 93.5 to 99.4.

2.3. Repeatability and Reproducibility Test

A different extractionist prepared samples for run number 32 than the extractionist who had prepared the samples for run numbers 25, 26, and 28. The samples were analyzed under the same operating conditions and with the same instrument. The calibration standards and QCs from run number 32 met the same acceptance criteria used for run numbers 25, 26, and 28.

The analytical procedure demonstrated acceptable reproducibility for all the analytes except N-MeFOSE-OH and N-EtFOSE-OH.

2.4. System Precision Test

The system precision for PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS was determined by injecting unextracted low and high QC samples at the beginning and end of a normal validation run. Instrument response is defined as the peak area ratio (analyte peak area/internal standard peak area). The intra-assay %CV for PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS were less than or equal to 78.2% for each QC concentration.

The unextracted samples did not demonstrate acceptable precision for all the analytes (See section 4 Comments and Conclusions for discussion of system precision.).

2.5. Extraction Efficiency

The extraction efficiencies for PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA, PFHS and SDS (internal standard) from human serum were determined by comparing the peak areas obtained for the following three cases:

1. Both the analyte and internal standard added following the extraction.
2. The analyte added to serum prior to extraction and the internal standard added following extraction.
3. The internal standard added to serum prior to extraction and the analyte added following the extraction.

The extraction efficiencies were then determined by the changes in peak area for the various cases. The mean extraction efficiencies were PFOS (85.9%), PFOSA (82.3%), PFOSAA (71.5%), N-MeFOSE-OH (53.0%), N-EtFOSE-OH (44.6%), POAA (90.0%), PFHS (81.5%) and SDS (71.2%).

2.6. Stability Evaluation

For the following stability evaluations, the mean test QC sample concentrations must fall within 20% of the mean reference QC sample concentrations to demonstrate acceptable stability.

Freeze-thaw Stability

Stability of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in human serum subjected to freezing and thawing for three cycles before processing was determined for low and high QC samples. Test QCs were compared against reference QC samples subjected to one freeze/thaw cycle.

Samples demonstrated acceptable stability after three freeze/thaw cycles for all the analytes.

Room Temperature Matrix Stability

Stability of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in human serum stored under normal room temperature and light conditions for up to 4 hours was determined for low and high QC samples. Test QCs were compared against reference QC samples prepared immediately upon thawing.

Samples demonstrated acceptable stability after 4 hours at normal room temperature and light conditions for all the analytes except N-MeFOSE-OH and N-EtFOSE-OH.

Room Temperature Extract Stability

Stability of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS extracts stored at room temperature for approximately 24 hours prior to analysis was determined for low and high QC extracts. Test QCs were compared against reference QC samples analyzed immediately following extraction.

Extracted samples demonstrated acceptable stability after 24 hours at normal room temperature for all the analytes except PFOS, PFOSAA, POAA and PFHS analytes (See section 4 Comments and Conclusions for discussion of room temperature extract stability).

Stock Solution Stability

The stock solution stability was evaluated by comparing the concentrations for a set of unextracted standards made from a stock solution prepared on January 12, 1999 with a set made from a stock solution prepared February 5, 1999. The stock solution stability evaluation was performed in replicates of three at the same level of unextracted standard.

Stock solutions demonstrated acceptable stability after storage at -20 °C for 24 days for all the analytes except N-MeFOSE-OH and N-EtFOSE-OH.

Long-Term Matrix Stability and Reduced Temperature (-20 °C) Extract Stability

These stability tests will be performed and reported at a later date in a separate report.

3. DATA MANAGEMENT

The concentration values for the endogenous levels of PFOS, PFOSAA, POAA and PFHS were calculated using PE Sciex MacQuan software (v. 1.6). All other concentration values were calculated using the Watson® DMLIMS software. Data presented in this report may be formatted to fewer significant figures than may have been used by the computer to generate means, standard deviations and coefficients of variation. Summary statistics may be derived from unrounded data and may differ slightly from the values obtained using the rounded values.

4. COMMENTS AND CONCLUSIONS

During the assay validation, three potential internal standards were evaluated. They were nonanesulfonic acid, octanesulfonic acid and SDS. A fourth internal standard, $C_8H_5F_{13}SO_3$, was evaluated during method development, but was unsuitable for this assay owing to a serum interferent that demonstrated different ionization suppression from lot to lot of human serum. Of the three internal standards, SDS gave the best results for all the analytes, and thus it was used as the internal standard for each analyte.

To calculate the endogenous levels of the analytes in human serum, each validation run included: (1) "blank" human serum samples, prepared in duplicate or greater, from the lot used to prepare

the human serum calibration curve, (2) "blank" human serum samples in five replicates from the lot used to prepare the QCs, and (3) a calibration curve in duplicate prepared using rabbit serum with four different concentrations covering the range 1 to 50 ppb for each analyte. See Tables 5-7 for human serum endogenous concentration data.

If endogenous analyte was detected in the "blank" human serum samples, that concentration was calculated using the rabbit serum calibration curve. The calibration standard and the analytical QC target concentrations were then adjusted to include those endogenous levels.

For example, PFOS was determined to have an average endogenous level of 30.4 ppb in the human serum lot used for the calibration standards on run numbers 26, 28, and 32. The curve range was then adjusted to be 31.4 to 530 ppb; that is, the sum of the endogenous concentration plus the concentration spiked into each calibration standard. The average endogenous PFOS level for the human serum lot used for the analytical QCs was determined to be 22.3 ppb. Thus, the analytical low and high QC target levels were adjusted to 42.3 and 372 ppb (sum of the endogenous concentration plus the concentration spiked into each QC), respectively.

These average endogenous amounts for each analyte were used over the course of the validation except for run number 25. A different lot of human serum was used for the calibration curve on run number 25 than on run numbers 26, 28, and 29, thus yielding a different adjusted human calibration curve range for run number 25. The same human serum lot for the analytical and stability QCs was utilized over the course of the validation.

The method described in this report has been validated for the determination of PFOS, PFOSA, PFOSAA, POAA and PFHS in human serum. The method is not considered validated for N-MeFOSE-OH and N-EtFOSE-OH. The reason the results for N-MeFOSE-OH and N-EtFOSE-OH did not meet the acceptance criteria for validation could be due to the following factors: (1) poor extraction efficiencies, (2) poor ionization efficiencies (i. e., no efficiently ionizable functional groups), and (3) because the only useable MRM transition monitored for these two analytes is not derived from a molecular fragmentation, but rather fragmentation of the acetate adduct of each analyte to the acetate ion (i. e., $[M + CH_3CO_2]^- \rightarrow [CH_3CO_2]^-$).

The poor system precision for all the analytes can be attributed to a "column conditioning" affect. That is, the analytical column used for this assay yields more precise results when it is primed by injection of two or more extracted samples. When unextracted samples are injected onto the column, the condition of the column is affected and the precision decreases. This was verified by injecting two validation runs, with and without unextracted samples. The run without the unextracted samples gave more accurate and precise results than the run that included the unextracted samples.

Acceptable room temperature extract stability was not demonstrated for the high QCs of PFOS, PFOSAA, POAA, and PFHS. Owing to the apparent instability of the room temperature extracts, the samples must be extracted and analyzed on the same day and the analytical run times must be less than 24 hours, which is the longest time period for an accepted validation run.

4.1. Proposed Sample Analysis Acceptance Criteria

Subject Sample Determination

Subject sample concentrations for all the analytes will be determined using the human serum calibration curve, unless an analyte concentration for PFOS, PFOSAA, POAA or PFHS is less that the adjusted LLOQ concentration. If a subject sample has a concentration for any of those analytes that is below the adjusted LLOQ, then the rabbit curve will be used to quantitate that analyte in that sample. In these cases, the accuracy of the rabbit serum curve will be verified by analyzing human serum QCs containing known analyte concentrations between 1 and 50 ppb and calculating their concentrations using the rabbit serum curve.

Calibration Curve

Each run will include calibration standards in duplicate at seven or more concentrations covering the lower to upper limit of quantitation. At least two thirds of the non-LLOQ calibration standard's back-calculated concentrations must be within ± 15% (LLOQ must be within ± 20%) of their individual target concentrations. If a standard is not within the acceptance criteria, it is deactivated. This process starts from the highest standard down to the lowest standard until all the active standards are within the acceptance criteria.

Lower Limit of Quantitation

The back-calculated concentrations of at least one of the duplicate lowest points in the calibration curve must be within ± 20% of the target concentration to qualify as the LLOQ. If this criterion is not met, the next level is subjected to the same test and the LLOQ raised accordingly.

Quality Control Samples

Each analytical run will include low and high QC samples in triplicate. The measured concentrations of at least two-thirds of all analytical QCs must be within ± 20 % of their target concentrations. If study samples require dilution, a dilution QC will be analyzed in triplicate for each dilution level. At least one dilution QC at each level must be within ± 20% of the target concentration. The dilution QC acceptance is independent of the undiluted analytical QC acceptance.

5. REFERENCES

[5.1] L. Clemen, G. Langenburg, "Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Serum or Blood Extracts Using HPLC-Electrospray/Mass Spectrometry," 3M Environmental Laboratory, FACT-M-4.1.

[5.2] L. Clemen, G. Langenburg, "Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals Compounds from Serum or Other Fluids for Analysis Using HPLC-Electrospray/Mass Spectrometry," 3M Environmental Laboratory, FACT-M-3.1.

[5.3] L. Clemen, "Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Sulfactants from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry," 3M Environmental Laboratory, FACT-M-1.0.

The raw data and final report for this study will be stored in the NWB Archives, 1121 East 3900 South, Salt Lake City, UT 84124 per regulations and contract agreement. 3M will be notified concerning final disposition of records at completion of contract obligations.

Table 1. Summary of Rabbit Serum Calibration Curves for PFOS

Linear (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	1.00	5.00	25.0	50.0	Slope	Intercept	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	0.896	5.42	20.0	53.1	1524	266	0.9950	1.00	50.0
		1.03	5.61	24.6	51.4					
01-Feb-1999	26	*0.671	4.44	24.3	47.2	1407	553	0.9950	1.00	50.0
		1.20	4.80	22.7	56.3					
02-Feb-1999	28	0.903	6.22	26.1	47.0	1414	657	0.9977	1.00	50.0
		0.887	4.65	26.3	50.0					
03-Feb-1999	29	0.826	4.96	21.7	51.9	1580	944	0.9966	1.00	50.0
		1.02	6.07	26.8	48.7					
Mean		0.928	5.27	24.1	50.7	1481	605	0.9961		
S.D.		0.146	0.619	2.27	2.95	74	243	0.0011		
%CV		15.8	11.7	9.4	5.8					
n		7	8	8	8	4	4	4		

* sample deactivated – poor instrument response

Table 2. Summary of Rabbit Serum Calibration Curves for PFOSAA

Linear (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	1.00	5.00	25.0	50.0	Slope	Intercept	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	0.857	5.41	19.9	55.0	212	-30	0.9921	1.00	50.0
		2.07	6.16	23.0	50.8					
01-Feb-1999	26	*	4.80	23.2	47.6	215	103	0.9967	1.00	50.0
		1.21	4.25	25.3	54.7					
02-Feb-1999	28	0.724	5.29	25.3	46.8	266	188	0.9962	1.00	50.0
		0.863	6.73	26.8	49.5					
03-Feb-1999	29	0.702	4.98	24.9	47.2	313	159	0.9979	1.00	50.0
		1.09	6.16	25.1	51.8					
Mean		1.07	5.47	24.2	50.4	252	105	0.9957		
S.D.		0.442	0.770	1.98	3.02	41	84	0.0022		
%CV		41.2	14.1	8.2	6.0					
n		7	8	8	8	4	4	4		

* sample deactivated – poor instrument response

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Table 3. Summary of Rabbit Serum Calibration Curves for POAA

Linear (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	1.00	5.00	25.0	50.0	Slope	Intercept	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	1.02	5.57	21.9	52.6	2543	81	0.9976	1.00	50.0
		0.917	5.22	24.2	50.5					
01-Feb-1999	26	*0.643	4.51	23.8	52.2	2782	318	0.9991	1.00	50.0
		1.13	5.01	24.3	50.1					
02-Feb-1999	28	0.835	5.56	23.4	48.2	2983	859	0.9985	1.00	50.0
		1.01	5.31	26.6	51.1					
03-Feb-1999	29	0.857	5.26	24.0	50.3	2988	1141	0.9994	1.00	50.0
		1.00	5.66	24.8	50.1					
Mean		0.925	5.26	24.1	50.6	2824	600	0.9987		
S.D.		0.139	0.352	1.22	1.27	182	421	0.0007		
%CV		15.0	6.7	5.1	2.5					
n		7	8	8	8	4	4	4		

* sample deactivated – poor instrument response

Table 4. Summary of Rabbit Serum Calibration Curves for PFHS

Linear (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	1.00	5.00	25.0	50.0	Slope	Intercept	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	0.833	5.87	22.5	52.9	2677	567	0.9955	1.00	50.0
		0.876	5.50	23.4	52.3					
01-Feb-1999	26	*0.742	4.85	23.2	50.7	2824	701	0.9990	1.00	50.0
		1.06	5.10	24.2	51.9					
02-Feb-1999	28	0.882	5.45	23.2	49.5	3013	629	0.9990	1.00	50.0
		1.08	4.94	25.4	51.6					
03-Feb-1999	29	0.862	5.12	23.8	49.2	3018	974	0.9994	1.00	50.0
		1.03	5.54	25.4	51.0					
Mean		0.921	5.30	23.9	51.1	2883	718	0.9982		
S.D.		0.113	0.326	1.00	1.22	142	155	0.0016		
%CV		12.3	6.2	4.2	2.4					
n		7	8	8	8	4	4	4		

* sample deactivated – poor instrument response

Table 5. Summary Data for Endogenous Analyte Concentrations in Human Serum Calibration Standards for Run 25

	PFOS	PFOSA	PFOSAA	N-MeFOSE-OH	N-EtFOSE-OH	PFHS	POAA
Replicate							
1	16.1	ND	1.60	ND	ND	0.497	3.08
2	15.4	ND	1.94	ND	ND	0.666	3.00
3	16.0	ND	0.943	ND	ND	0.696	3.18
4	17.3	ND	1.34	ND	ND	0.602	3.52
Mean	16.2	N/A	1.46	N/A	N/A	0.615	3.20
S.D.	0.796	N/A	0.421	N/A	N/A	0.088	0.228
% CV	4.9	N/A	28.9	N/A	N/A	14.3	7.1
n	4	4	4	4	4	4	4

ND = none detected
N/A = not applicable

Table 6. Summary Data for Endogenous Analyte Concentrations in Human Serum Calibration Standards for Runs 26, 28 and 29

	PFOS	PFOSA	PFOSAA	N-MeFOSE-OH	N-EtFOSE-OH	POAA	PFHS
Run 26							
1	25.4	ND	0.646	ND	ND	3.91	1.33
2	31.3	ND	1.07	ND	ND	5.04	1.66
3	27.5	ND	2.94	ND	ND	4.59	1.68
4	33.3	ND	1.55	ND	ND	4.79	1.68
Mean	29.4	N/A	1.55	N/A	N/A	4.58	1.59
S.D.	3.61	N/A	0.998	N/A	N/A	0.482	0.172
% CV	12.3	N/A	64.3	N/A	N/A	10.5	10.8
Run 28							
1	36.8	ND	2.88	ND	ND	4.22	1.79
2	35.8	ND	2.30	ND	ND	5.05	1.94
3	39.2	ND	1.25	ND	ND	4.97	2.01
4	27.8	ND	0.953	ND	ND	4.02	1.57
Mean	34.9	N/A	1.85	N/A	N/A	4.56	1.82
S.D.	4.93	N/A	0.901	N/A	N/A	0.521	0.195
% CV	14.1	N/A	48.7	N/A	N/A	11.4	10.7
Run 29							
1	23.4	ND	2.07	ND	ND	3.10	1.24
2	23.9	ND	1.23	ND	ND	2.97	1.26
Mean	23.6	N/A	1.65	N/A	N/A	3.04	1.25
S.D.	0.417	N/A	0.598	N/A	N/A	0.091	0.017
% CV	1.8	N/A	36.3	N/A	N/A	3.0	1.4
Overall Mean	30.4	N/A	1.69	N/A	N/A	4.27	1.61
Overall S.D.	5.66	N/A	0.814	N/A	N/A	0.767	0.269
Overall % CV	18.6	N/A	48.2	N/A	N/A	18.0	16.6

ND = none detected
N/A = not applicable

Table 7. Summary Data for Endogenous Analyte Concentrations in Human Serum Quality Control Samples for Runs 26, 28 and 29

	PFOS	PFOSA	PFOSAA	N-MeFOSE-OH	N-EtFOSE-OH	POAA	PFHS
Run 26							
1	21.6	ND	1.71	ND	ND	1.62	3.22
2	21.4	ND	1.69	ND	ND	1.99	2.85
3	21.0	ND	2.86	ND	ND	1.77	3.54
4	22.5	ND	3.13	ND	ND	2.13	3.32
5	23.4	ND	1.74	ND	ND	2.13	3.38
Mean	22.0	N/A	2.23	N/A	N/A	1.93	3.26
S.D.	0.982	N/A	0.706	N/A	N/A	0.225	0.258
% CV	4.5	N/A	31.7	N/A	N/A	11.7	7.9
Run 28							
1	18.2	ND	1.66	ND	ND	1.66	3.13
2	22.3	ND	1.95	ND	ND	2.02	3.11
3	20.7	ND	1.51	ND	ND	2.04	3.18
4	23.0	ND	1.53	ND	ND	2.13	3.33
5	22.1	ND	2.09	ND	ND	2.07	3.20
Mean	21.3	N/A	1.75	N/A	N/A	1.98	3.19
S.D.	1.91	N/A	0.258	N/A	N/A	0.187	0.087
% CV	0.1	N/A	0.1	N/A	N/A	0.1	0.0
Run 29							
1	21.8	ND	2.41	ND	ND	1.73	3.41
2	23.9	ND	4.01	ND	ND	2.15	3.55
3	22.8	ND	2.59	ND	ND	1.96	2.88
4	23.5	ND	2.50	ND	ND	1.92	3.24
5	26.2	ND	2.80	ND	ND	2.31	3.42
Mean	23.6	N/A	2.86	N/A	N/A	2.01	3.30
S.D.	1.63	N/A	0.658	N/A	N/A	0.224	0.258
% CV	6.9	N/A	23.0	N/A	N/A	11.1	7.8
Overall Mean	22.3	N/A	1.99	N/A	N/A	1.97	3.25
Overall S.D.	1.77	N/A	0.560	N/A	N/A	0.200	0.206
Overall % CV	7.9		28.2			10.2	6.3

ND = none detected
N/A = not applicable

Table 8. Summary of Human Serum Calibration Curve Parameters for PFOS

Quadratic (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	A*	B*	C*	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	-0.000002	0.003075	0.006751	0.9909	17.2	516
01-Feb-1999	26	-0.000002	0.002977	0.000357	0.9930	31.4	530
02-Feb-1999	28	-0.000001	0.001649	0.007710	0.9937	31.4	530
10-Feb-1999	32	-0.000003	0.004162	-0.009869	0.9960	31.4	530
Mean		-0.000002	0.002966	0.001237	0.9934		
S.D.		0.000001	0.001029	0.008092	0.0021		
n		4	4	4	4		

* A, B, and C are coefficients used to define the quadratic curve.

Table 9. Summary of Human Serum Calibration Curve Parameters for PFOSA

Quadratic (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	A*	B*	C*	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	-0.000002	0.003444	0.000622	0.9946	1.00	500
01-Feb-1999	26	-0.000002	0.003308	0.000757	0.9965	1.00	500
02-Feb-1999	28	-0.000001	0.001834	0.000789	0.9982	1.00	500
10-Feb-1999	32	-0.000002	0.003945	0.001714	0.9982	1.00	500
Mean		-0.000002	0.003133	0.000971	0.9969		
S.D.		0.000001	0.000908	0.000501	0.0017		
n		4	4	4	4		

* A, B, and C are coefficients used to define the quadratic curve.

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Table 10. Summary of Human Serum Calibration Curve Parameters for PFOSAA

Quadratic (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	A*	B*	C*	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	0.000000	0.000676	-0.000208	0.9964	1.99	270
01-Feb-1999	26	-0.000001	0.000764	-0.000653	0.9971	2.23	271
02-Feb-1999	28	0.000000	0.000469	-0.000089	0.9963	2.23	271
10-Feb-1999	32	-0.000001	0.001170	-0.000458	0.9965	2.23	271
Mean		0.000000	0.000770	-0.000352	0.9966		
S.D.		0.000001	0.000294	0.000253	0.0004		
n		4	4	4	4		

* A, B, and C are coefficients used to define the quadratic curve.

Table 11. Summary of Human Serum Calibration Curve Parameters for N-MeFOSE-OH

Quadratic (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	A*	B*	C*	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	-0.000007	0.021767	-0.001921	0.9981	1.00	500
01-Feb-1999	26	-0.000004	0.012302	0.005797	0.9962	1.00	500
02-Feb-1999	28	0.000001	0.007315	0.001464	0.9970	1.00	500
10-Feb-1999	32	-0.000010	0.019016	0.003235	0.9970	1.00	500
Mean		-0.000005	0.015100	0.002144	0.9971		
S.D.		0.000005	0.006538	0.003241	0.0008		
n		4	4	4	4		

* A, B, and C are coefficients used to define the quadratic curve.

Table 12. Summary of Human Serum Calibration Curve Parameters for N-EtFOSE-OH

Quadratic (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	A*	B*	C*	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	-0.000002	0.015048	-0.003325	0.9994	1.00	500
01-Feb-1999	26	-0.000002	0.008104	0.002172	0.9954	1.00	500
02-Feb-1999	28	0.000000	0.005031	-0.005001	0.9986	5.00**	500
10-Feb-1999	32	-0.000004	0.011968	-0.001308	0.9969	1.00	500
Mean		-0.000002	0.010038	-0.001866	0.9976		
S.D.		0.000002	0.004383	0.003086	0.0018		
n		4	4	4	4		

* A, B, and C are coefficients used to define the quadratic curve.
** Both Standards at 1.00 ppb were statistical outliers, so the LLOQ was raised to 5.00 ppb.

Table 13. Summary of Human Serum Calibration Curve Parameters for POAA

Quadratic (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	A*	B*	C*	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	-0.000003	0.005601	0.000463	0.9972	4.20	503
01-Feb-1999	26	-0.000004	0.006274	-0.001033	0.9963	5.27	504
02-Feb-1999	28	-0.000002	0.004036	0.003976	0.9981	5.27	504
10-Feb-1999	32	-0.000005	0.008652	-0.001660	0.9970	5.27	504
Mean		-0.000004	0.006141	0.000437	0.9972		
S.D.		0.000001	0.001919	0.002522	0.0007		
n		4	4	4	4		

* A, B, and C are coefficients used to define the quadratic curve.

Table 14. Summary of Human Serum Calibration Curve Parameters for PFHS

Quadratic (weighted 1/x). All concentrations are expressed as ppb.

Run Date	Run Number	A*	B*	C*	Multiple Rsquared	LLOQ (ppb)	ULOQ (ppb)
30-Jan-1999	25	-0.000004	0.005961	0.002977	0.9979	1.62	501
01-Feb-1999	26	-0.000004	0.006088	0.000767	0.9977	2.61	502
02-Feb-1999	28	-0.000002	0.003814	0.003933	0.9982	2.61	502
10-Feb-1999	32	-0.000006	0.007750	0.002353	0.9973	2.61	502
Mean		-0.000004	0.005903	0.002508	0.9978		
S.D.		0.000002	0.001614	0.001330	0.0004		
n		4	4	4	4		

* A, B, and C are coefficients used to define the quadratic curve.

Table 15. Back-Calculated Concentrations of Human Serum Calibration Standards for PFOS

All concentrations are expressed as ppb.

Run Date	Run Number	17.2	21.2	26.2	31.4	35.4	40.4	41.2	55.4	66.2	80.4	116	130	280	516	530
30-Jan-1999	25	16.6	19.9	27.2				44.2		65.1		103			460	
		16.4	18.3	29.4				45.0		69.3		120			584	
01-Feb-1999	26				35.6	37.2	39.6		50.8		73.0		130			498
					32.8	32.4	*32.6		53.0		*56.4		143			563
02-Feb-1999	28				28.4	36.6	39.1		53.6		76.1		126	288		566
					34.6	35.7	42.1		62.6		68.5		135	290		486
10-Feb-1999	32				29.7	33.6	43.3		57.7		*65.5		140	278		501
					32.4	33.8	40.5		56.7		73.6		135	262		580
Mean		16.5	19.1	28.3	32.3	34.9	40.9	44.6	55.7	67.2	72.8	112	135	280	522	532
S.D.		0.141	1.13	1.56	2.77	1.90	1.75	0.566	4.20	2.97	3.17	12.0	6.24	12.8	87.7	41.6
%CV		0.9	5.9	5.5	8.6	5.4	4.3	1.3	7.5	4.4	4.4	10.7	4.6	4.6	16.8	7.8
%Bias		-4.1	-9.9	8.0	2.9	-1.4	1.2	8.3	0.5	1.5	-9.5	-3.4	3.8	0.0	1.2	0.4
n		2	2	2	6	6	5	2	6	2	4	2	6	4	2	6

* sample deactivated-statistical outlier

Table 16. Back-Calculated Concentrations of Human Serum Calibration Standards for PFOSA

All concentrations are expressed as ppb.

Run Date	Run Number	1.00	5.00	10.0	25.0	50.0	100	250	500
30-Jan-1999	25	0.818	4.39	10.0	25.3	51.0	86.9		456
		1.14	5.02	*11.8	28.4	54.3	103		550
01-Feb-1999	26	1.19	5.06	10.1	23.2	48.3	107		454
		0.929	4.93	9.82	23.4	*40.6	*120		550
02-Feb-1999	28	1.00	5.01	10.2	23.9	47.7	99.9	262	525
		1.05	4.80	*12.2	26.4	47.0	107	236	476
10-Feb-1999	32	0.998	4.72	*11.8	23.9	*41.4	*118	247	471
		0.989	4.94	11.0	24.8	49.7	108	241	538
Mean		1.01	4.86	10.2	24.9	49.7	102	247	503
S.D.		0.116	0.222	0.456	1.77	2.69	7.99	11.3	42.2
%CV		11.5	4.6	4.5	7.1	5.4	7.8	4.6	8.4
%Bias		1.0	-2.8	2.0	-0.4	-0.6	2.0	-1.2	0.6
n		8	8	5	8	6	6	4	8

* sample deactivated-statistical outlier

Table 17. Back-Calculated Concentrations of Human Serum Calibration Standards for PFOSAA

All concentrations are expressed as ppb.

Run Date	Run Number	1.99	2.23	4.15	4.38	6.84	7.07	14.9	15.1	28.4	28.6	55.3	55.5	136	270	271
30-Jan-1999	25	1.74		4.03		7.29		13.7		27.3		53.2			250	
		2.10		4.37		7.06		*20.1		30.6		56.7			291	
01-Feb-1999	26		*3.80		4.16		6.75		14.2		26.4		54.6			264
			2.47		4.53		7.95		13.3		*22.7		61.5			278
02-Feb-1999	28		2.08		4.39		*9.11		*18.3		28.1		57.0	140		294
			2.35		4.29		*8.77		*18.4		29.2		57.8	126		252
10-Feb-1999	32		2.19		3.94		7.94		13.0		25.4		63.5	131		264
			2.29		4.62		7.02		16.0		28.5		56.7	132		282
Mean		1.92	2.28	4.20	4.32	7.18	7.42	13.7	14.1	29.0	27.5	55.0	58.5	132	271	272
S.D.		0.255	0.149	0.240	0.249	0.163	0.622		1.35	2.33	1.57	2.47	3.32	5.80	29.0	15.1
%CV		13.3	6.5	5.7	5.8	2.3	8.4		9.6	8.0	5.7	4.5	5.7	4.4	10.7	5.6
%Bias		-3.5	2.2	1.2	-1.4	5.0	5.0	-8.1	-6.6	2.1	-3.8	-0.5	5.4	-2.9	0.4	0.4
n		2	5	2	6	2	4	1	4	2	5	2	6	4	2	6

* sample deactivated-statistical outlier

Table 18. Back-Calculated Concentrations of Human Serum Calibration Standards for N-MeFOSE-OH

All concentrations are expressed as ppb.

Run Date	Run Number	1.00	5.00	10.0	25.0	50.0	100	250	500
30-Jan-1999	25	1.18	4.54	9.73	22.8	47.7	*63.1		478
		0.862	*4.04	11.3	*30.7	53.4	101		522
01-Feb-1999	26	1.10	4.69	10.1	23.6	52.8	*132		463
		1.04	4.77	*7.56	24.2	*39.4	*152		538
02-Feb-1999	28	0.971	4.61	10.2	24.9	45.1	87.0	252	493
		1.13	4.72	11.3	*29.5	*37.3	106	265	*385
10-Feb-1999	32	0.945	*3.84	11.4	21.8	*40.2	103	268	460
		1.19	4.41	10.6	21.9	*36.5	98.6	240	537
Mean		1.05	4.62	10.7	23.2	49.8	99.1	256	499
S.D.		0.118	0.133	0.678	1.25	4.02	7.30	12.9	33.6
%CV		11.2	2.9	6.3	5.4	8.1	7.4	5.0	6.7
%Bias		5.0	-7.6	7.0	-7.2	-0.4	-0.9	2.4	-0.2
n		8	6	7	6	4	5	4	7

* sample deactivated-statistical outlier

Table 19. Back-Calculated Concentrations of Human Serum Calibration Standards for N-EtFOSE-OH

All concentrations are expressed as ppb.

Run Date	Run Number	1.00	5.00	10.0	25.0	50.0	100	250	500
30-Jan-1999	25	1.19	5.01	10.1	23.4	49.8	*60.9		491
		0.963	4.25	*12.0	*30.1	52.4	100		509
01-Feb-1999	26	1.11	4.65	10.0	23.7	52.5	*133		461
		1.03	4.75	*7.79	24.5	*38.1	*153		540
02-Feb-1999	28	*1.88	5.20	9.64	22.5	*40.9	*78.6	244	497
		*2.06	4.98	10.9	25.5	*34.0	96.1	264	*386
10-Feb-1999	32	1.16	4.38	*11.7	22.8	42.6	107	272	477
		1.14	4.75	11.3	22.2	*37.5	97.5	246	513
Mean		1.10	4.75	10.4	23.5	49.3	100	257	498
S.D.		0.0861	0.321	0.687	1.17	4.65	4.84	13.7	25.7
%CV		7.8	6.8	6.6	5.0	9.4	4.8	5.3	5.2
%Bias		10.0	-5.0	4.0	-6.0	-1.4	0.0	2.8	-0.4
n		6	8	5	7	4	4	4	7

* sample deactivated-statistical outlier

Table 20. Back-Calculated Concentrations of Human Serum Calibration Standards for POAA

All concentrations are expressed as ppb.

Run Date	Run Number	4.2	5.27	8.2	9.27	13.2	14.3	28.2	29.3	53.2	54.3	103	104	254	503	504
30-Jan-1999	25	4.49		7.26		13.4		27.6		51.7		100			461	
		4.30		7.69		14.0		29.6		53.3		107			549	
01-Feb-1999	26		6.09		9.12		13.9		*18.9		48.7		105			500
			5.14		9.55		14.1		26.7		*44.3		113			
02-Feb-1999	28		4.92		9.49		14.8		27.2		53.2		104	270		528
			5.05		9.39		15.6		31.0		52.1		106	239		481
10-Feb-1999	32		5.47		8.49		*16.9		27.3		*44.5		113	241		489
			5.20		9.59		14.4		28.7		55.6		113	235		541
Mean		4.40	5.31	7.48	9.27	13.7	14.6	28.6	28.2	52.5	52.4	104	109	246	505	508
S.D.		0.134	0.423	0.304	0.418	0.424	0.673	1.41	1.74	1.13	2.87	4.95	4.43	16.0	62.2	25.7
%CV		3.0	8.0	4.1	4.5	3.1	4.6	4.9	6.2	2.2	5.5	4.8	4.1	6.5	12.3	5.1
%Bias		4.8	0.8	-8.8	0.0	3.8	2.1	1.4	-3.8	-1.3	-3.5	1.0	4.8	-3.1	0.4	0.8
n		2	6	2	6	2	5	2	5	2	4	2	6	4	2	5

* sample deactivated-statistical outlier

Table 21. Back-Calculated Concentrations of Human Serum Calibration Standards for PFHS

All concentrations are expressed as ppb.

Run Date	Run Number	1.62	2.61	5.62	6.61	10.6	11.6	25.6	26.6	50.6	51.6	101	102	252	501	502
30-Jan-1999	25	1.49		5.65		11.3		26.2		49.2		98.1			458	
		1.50		5.31		12.1		26.1		50.5		102			554	
01-Feb-1999	26		2.68		7.13		12.0		24.0		48.7		102			478
			2.65		6.74		11.4		24.3		*39.0		111			525
02-Feb-1999	28		*1.82		6.86		12.2		25.3		50.3		103	250		522
			2.16		6.55		12.4		29.2		48.6		111	238		494
10-Feb-1999	32		2.28		6.27		13.3		25.6		*42.5		112	243		475
			2.59		6.79		12.2		25.6		50.0		106	234		566
Mean		1.50	2.47	5.48	6.72	11.7	12.3	26.2	25.7	49.9	49.4	100	108	241	506	510
S.D.		0.00707	0.236	0.240	0.291	0.566	0.619	0.0707	1.86	0.919	0.876	2.76	4.42	6.90	67.9	34.7
%CV		0.5	9.6	4.4	4.3	4.8	5.0	0.3	7.2	1.8	1.8	2.8	4.1	2.9	13.4	6.8
%Bias		-7.4	-5.4	-2.5	1.7	10.4	6.0	2.3	-3.4	-1.4	-4.3	-1.0	5.9	-4.4	1.0	1.6
n		2	5	2	6	2	6	2	6	2	4	2	6	4	2	6

* sample deactivated-statistical outlier

Table 22. Intra-Assay Precision for PFOS Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Number: 26 Run Date: 01-Feb-1999	Low QC (42.3 ppb)	High QC (372 ppb)
Replicate		
1	42.0	412
2	44.6	* 459
3	45.0	324
4	41.4	392
5	43.5	443
Mean	43.3	406
S.D.	1.57	52.8
CV%	3.6	13.0
%Theoretical	102.4	109.1
n	5	5

* > 20% theoretical

Table 23. Intra-Assay Precision for PFOSA Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Number: 26 Run Date: 01-Feb-1999	Low QC (20.0 ppb)	High QC (350 ppb)
Replicate		
1	21.8	387
2	*26.1	410
3	23.2	324
4	21.5	374
5	23.2	409
Mean	23.2	381
S.D.	1.82	35.2
CV%	7.8	9.2
%Theoretical	116.0	108.9
n	5	5

* > 20% theoretical

Table 24. Intra-Assay Precision for PFOSAA Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Number: 26 Run Date: 01-Feb-1999	Low QC (12.7 ppb)	High QC (190 ppb)
Replicate		
1	12.0	187
2	14.0	200
3	13.2	163
4	13.9	189
5	13.9	196
Mean	13.4	187
S.D.	0.846	14.4
%CV	6.3	7.7
%Theoretical	105.5	98.4
n	5	5

Table 25. Intra-Assay Precision for N-MeFOSE-OH Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Number: 26 Run Date: 01-Feb-1999	Low QC (20.0 ppb)	High QC (350 ppb)
Replicate		
1	23.2	*424
2	22.1	*425
3	23.2	336
4	21.7	*426
5	21.5	407
Mean	22.3	404
S.D.	0.814	38.6
CV%	3.7	9.6
%Theoretical	111.5	115.4
n	5	5

* > 20% theoretical

Table 26. Intra-Assay Precision for N-EtFOSE-OH Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Number: 26 Run Date: 01-Feb-1999	Low QC (20.0 ppb)	High QC (350 ppb)
Replicate		
1	23.1	*431
2	22.2	*431
3	22.7	341
4	21.4	*440
5	22.1	403
Mean	22.3	409
S.D.	0.644	40.6
CV%	2.9	9.9
%Theoretical	111.5	116.9
n	5	5

* > 20% theoretical

Table 27. Intra-Assay Precision for POAA Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Number: 26 Run Date: 01-Feb-1999	Low QC (23.3 ppb)	High QC (353 ppb)
Replicate		
1	21.8	366
2	24.0	374
3	22.2	*281
4	22.2	341
5	18.8	365
Mean	21.8	345
S.D.	1.88	38.1
CV%	8.6	11.0
%Theoretical	93.6	97.7
n	5	5

* > 20% theoretical

Table 28. Intra-Assay Precision for PFHS Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Number: 26 Run Date: 01-Feb-1999	Low QC (22.0 ppb)	High QC (352 ppb)
Replicate		
1	22.9	343
2	25.2	384
3	23.3	*278
4	23.3	332
5	24.9	360
Mean	23.9	339
S.D.	1.05	39.5
CV%	4.4	11.7
%Theoretical	108.6	96.3
n	5	5

* > 20% theoretical

Table 29. Inter-Assay Precision for PFOS Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (42.3 ppb)	High QC (372 ppb)
30-Jan-1999	25	40.9	414
		45.2	409
		**33.6	408
		**29.2	354
		41.2	398
01-Feb-1999	26	42.0	412
		44.6	**459
		45.0	324
		41.4	392
		43.5	443
02-Feb-1999	28	*19.0	**503
		38.9	401
		44.7	418
		43.2	397
		36.9	435
10-Feb-1999	32	40.7	352
		43.3	340
		41.9	377
		38.6	344
		41.5	355
Mean		40.9	397
S.D.		4.08	44.3
%CV		10.0	11.2
%Theoretical		96.7	106.7
%Bias		-3.3	6.7
n		19	20

* sample deactivated – poor instrument response
** > 20% theoretical

Table 30. Inter-Assay Precision for PFOSA Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (20.0 ppb)	High QC (350 ppb)
30-Jan-1999	25	23.9	386
		**24.7	357
		17.8	410
		**14.2	343
		21.5	381
01-Feb-1999	26	21.8	387
		**26.1	410
		23.2	324
		21.5	374
		23.2	409
02-Feb-1999	28	*10.5	**456
		20.5	342
		20.9	370
		22.2	339
		19.2	388
10-Feb-1999	32	20.7	338
		23.4	329
		20.7	347
		17.0	331
		20.1	317
Mean		21.2	367
S.D.		2.81	36.5
%CV		13.3	9.9
%Theoretical		106.0	104.9
%Bias		6.0	4.9
n		19	20

* sample deactivated– poor instrument response
** > 20% theoretical

Table 31. Inter-Assay Precision for PFOSAA Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (12.7 ppb)	High QC (190 ppb)
30-Jan-1999	25	**16.6	223
		13.6	225
		11.1	**246
		**9.97	208
		14.0	213
01-Feb-1999	26	12.0	187
		14.0	200
		13.2	163
		13.9	189
		13.9	196
02-Feb-1999	28	*9.11	212
		12.2	186
		14.8	194
		13.9	177
		11.8	189
10-Feb-1999	32	13.4	202
		14.3	195
		12.5	205
		10.9	192
		12.2	175
Mean		13.1	199
S.D.		1.56	19.1
%CV		11.9	9.6
%Theoretical		103.1	104.7
%Bias		3.1	4.7
n		19	20

* sample deactivated– poor instrument response
** > 20% theoretical

Table 32. Inter-Assay Precision for N-MeFOSE-OH Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (20.0 ppb)	High QC (350 ppb)
30-Jan-1999	25	16.4	310
		19.0	316
		**15.2	355
		**12.2	301
		17.4	311
01-Feb-1999	26	23.2	**424
		22.1	**425
		23.2	336
		21.7	**426
		21.5	407
02-Feb-1999	28	*11.6	**446
		23.6	352
		**28.8	383
		**26.5	335
		22.8	401
10-Feb-1999	32	**14.8	**252
		16.0	**244
		**15.0	328
		**11.5	315
		**14.7	292
Mean		19.2	348
S.D.		4.90	58.9
%CV		25.5	16.9
%Theoretical		96.0	99.4
%Bias		-4.0	-0.6
n		19	20

* sample deactivated-- poor instrument response
** > 20% theoretical

Table 33. Inter-Assay Precision for N-EtFOSE-OH Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (20.0 ppb)	High QC (350 ppb)
30-Jan-1999	25	**15.5	316
		18.7	308
		**15.0	340
		**12.1	294
		17.3	316
01-Feb-1999	26	23.1	**431
		22.2	**431
		22.7	341
		21.4	**440
		22.1	403
02-Feb-1999	28	*11.4	**436
		23.0	329
		23.5	363
		23.1	332
		21.2	378
10-Feb-1999	32	**15.1	**251
		16.8	**254
		**15.4	339
		**12.5	314
		**14.9	301
Mean		18.7	346
S.D.		3.97	57.6
%CV		21.2	16.6
%Theoretical		93.5	98.9
%Bias		-6.5	-1.1
n		19	20

* sample deactivated– poor instrument response
** > 20% theoretical

Table 34. Inter-Assay Precision for POAA Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (23.3 ppb)	High QC (353 ppb)
30-Jan-1999	25	**28.0	349
		24.4	396
		**18.3	377
		**17.4	318
		24.2	379
01-Feb-1999	26	21.8	366
		24.0	374
		22.2	**281
		22.2	341
		18.8	365
02-Feb-1999	28	*11.6	376
		23.1	325
		24.8	345
		22.0	324
		19.1	351
10-Feb-1999	32	23.5	348
		25.2	321
		22.1	321
		19.5	326
		22.6	302
Mean		22.3	344
S.D.		2.70	29.5
%CV		12.1	8.6
%Theoretical		95.7	97.5
%Bias		-4.3	-2.5
n		19	20

* sample deactivated– poor instrument response
** > 20% theoretical

Table 35. Inter-Assay Precision for PFHS Human Serum Quality Control Samples

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (22.0 ppb)	High QC (352 ppb)
30-Jan-1999	25	**26.6	379
		24.4	391
		18.7	370
		**17.1	328
		24.0	361
01-Feb-1999	26	22.9	343
		25.2	384
		23.3	**278
		23.3	332
		24.9	360
02-Feb-1999	28	*11.9	393
		23.1	359
		23.1	382
		23.3	366
		21.0	377
10-Feb-1999	32	21.7	357
		22.6	344
		22.6	354
		20.8	300
		21.4	308
Mean		22.6	353
S.D.		2.22	31.1
%CV		9.8	8.8
%Theoretical		102.7	100.3
%Bias		2.7	0.3
n		19	20

* sample deactivated– statistical outlier
** > 20% theoretical

Table 36. System Precision for PFOS

Run Date	Run Number	Unextracted Low QC (20.0 ppb)	Unextracted High QC (350 ppb)
		Instrument Response	
10-Feb-1999	32	0.101895	1.622610
		0.141723	1.870562
		0.169329	1.990953
Mean		0.137649	1.828042
S.D.		0.033901	0.187817
%CV		24.6	10.3
n		3	3

Table 37. System Precision for PFOSA

Run Date	Run Number	Unextracted Low QC (20.0 ppb)	Unextracted High QC (350 ppb)
		Instrument Response	
10-Feb-1999	32	0.006447	0.207807
		0.006939	0.558348
		0.018200	0.347834
Mean		0.010529	0.371330
S.D.		0.006648	0.176448
%CV		63.1	47.5
n		3	3

Table 38. System Precision for PFOSAA

Run Date	Run Number	Unextracted Low QC (12.7 ppb)	Unextracted High QC (190 ppb)
		Instrument Response	
10-Feb-1999	32	0.017247	0.247510
		0.015952	0.338792
		0.029981	0.357733
Mean		0.021060	0.314678
S.D.		0.007753	0.058935
%CV		36.8	18.7
n		3	3

Table 39. System Precision for N-MeFOSE-OH

Run Date	Run Number	Unextracted Low QC (20.0 ppb)	Unextracted High QC (350 ppb)
		Instrument Response	
10-Feb-1999	32	0.015063	0.583610
		0.010676	2.230467
		0.032584	1.338583
Mean		0.019441	1.384220
S.D.		0.011592	0.824376
%CV		59.6	59.6
n		3	3

Table 40. System Precision for N-EtFOSE-OH

Run Date	Run Number	Unextracted Low QC (20.0 ppb)	Unextracted High QC (350 ppb)
		Instrument Response	
10-Feb-1999	32	0.011565	0.525858
		0.008637	2.211409
		0.034819	1.323620
Mean		0.018340	1.353629
S.D.		0.014346	0.843176
%CV		78.2	62.3
n		3	3

Table 41. System Precision for POAA

Run Date	Run Number	Unextracted Low QC (20.0 ppb)	Unextracted High QC (350 ppb)
		Instrument Response	
10-Feb-1999	32	0.148240	2.629881
		0.197618	3.363888
		0.313308	3.570008
Mean		0.219722	3.187926
S.D.		0.084725	0.494147
%CV		38.6	15.5
n		3	3

Table 42. System Precision for PFHS

Run Date	Run Number	Unextracted Low QC (20.0 ppb)	Unextracted High QC (350 ppb)
		Instrument Response	
10-Feb-1999	32	0.179242	2.770742
		0.244403	3.162425
		0.295965	3.428575
Mean		0.239870	3.120581
S.D.		0.058493	0.330907
%CV		24.4	10.6
n		3	3

Table 43. PFOS Extraction Efficiency

Time of Spiking	Analyte Peak Areas	Istd Peak Areas
1. Analyte and IS after extraction	81470	539547
	86784	554615
	77797	523549
Mean	82017	539237
2. Analyte prior and IS after extraction	61442	
	94927	
	54973	
Mean	70447	
3. IS prior and Analyte after extraction		200162
		461938
		490278
Mean		384126

Mean extraction efficiency for the analyte = 85.9%
Mean extraction efficiency for the internal standard = 71.2%

Table 44. PFOSA Extraction Efficiency

Time of Spiking	Analyte Peak Areas	Istd Peak Areas
1. Analyte and IS after extraction	45280	539547
	45068	554615
	38724	523549
Mean	43024	539237
2. Analyte prior and IS after extraction	33229	
	47251	
	25711	
Mean	35397	
3. IS prior and Analyte after extraction		200162
		461938
		490278
Mean		384126

Mean extraction efficiency for the analyte = 82.3%
Mean extraction efficiency for the internal standard = 71.2%

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Table 45. PFOSAA Extraction Efficiency

Time of Spiking	Analyte Peak Areas	Istd Peak Areas
1. Analyte and IS after extraction	6484	539547
	6197	554615
	6383	523549
Mean	6355	539237
2. Analyte prior and IS after extraction	4229	
	5443	
	3961	
Mean	4544	
3. IS prior and Analyte after extraction		200162
		461938
		490278
Mean		384126

Mean extraction efficiency for the analyte = 71.5%
Mean extraction efficiency for the internal standard = 71.2%

Table 46. N-MeFOSE-OH Extraction Efficiency

Time of Spiking	Analyte Peak Areas	Istd Peak Areas
1. Analyte and IS after extraction	257654	539547
	262358	554615
	154951	523549
Mean	224988	539237
2. Analyte prior and IS after extraction	85560	
	226651	
	45573	
Mean	119261	
3. IS prior and Analyte after extraction		200162
		461938
		490278
Mean		384126

Mean extraction efficiency for the analyte = 53.0%
Mean extraction efficiency for the internal standard = 71.2%

Table 47. N-EtFOSE-OH Extraction Efficiency

Time of Spiking	Analyte Peak Areas	Istd Peak Areas
1. Analyte and IS after extraction	191529	539547
	190650	554615
	122094	523549
Mean	168091	539237
2. Analyte prior and IS after extraction	52797	
	139842	
	32402	
Mean	75014	
3. IS prior and Analyte after extraction		200162
		461938
		490278
Mean		384126

Mean extraction efficiency for the analyte = 44.6%
Mean extraction efficiency for the internal standard = 71.2%

Table 48. POAA Extraction Efficiency

Time of Spiking	Analyte Peak Areas	Istd Peak Areas
1. Analyte and IS after extraction	87822	539547
	84427	554615
	75701	523549
Mean	82650	539237
2. Analyte prior and IS after extraction	76904	
	83445	
	62754	
Mean	74368	
3. IS prior and Analyte after extraction		200162
		461938
		490278
Mean		384126

Mean extraction efficiency for the analyte = 90.0%
Mean extraction efficiency for the internal standard = 71.2%

Table 49. PFHS Extraction Efficiency

Time of Spiking	Analyte Peak Areas	Istd Peak Areas
1. Analyte and IS after extraction	87124	539547
	86589	554615
	82852	523549
Mean	85522	539237
2. Analyte prior and IS after extraction	66595	
	81391	
	61113	
Mean	69700	
3. IS prior and Analyte after extraction		200162
		461938
		490278
Mean		384126

Mean extraction efficiency for the analyte = 81.5%
Mean extraction efficiency for the internal standard = 71.2%

Table 50. Freeze/Thaw Stability for PFOS

All concentrations are expressed as ppb.

Run Number	**25**
Run Date	**30-Jan-1999**
	LOW QC
	After 3 Freeze/Thaw cycles
1	43.3
2	33.1
3	40.5
Mean	39.0
S.D.	5.27
% CV	13.5%
Control (ng/mL)	38.0
Mean % Dev. from Control	**2.54%**
	HIGH QC
	After 3 Freeze/Thaw cycles
1	330
2	315*
3	410
Mean	370
S.D.	56.6
% CV	15.3%
Control (ng/mL)	397
Mean % Dev. from Control	**-6.80%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

Table 51. Freeze/Thaw Stability for PFOSA

All concentrations are expressed as ppb.

Run Number	**25**
Run Date	**30-Jan-1999**
	LOW QC
	After 3 Freeze/Thaw cycles
1	23.5
2	19.9
3	20.0
Mean	21.1
S.D.	2.05
% CV	9.70%
Control (ng/mL)	20.4
Mean % Dev. from Control	**3.59%**
	HIGH QC
	After 3 Freeze/Thaw cycles
1	336
2	248*
3	401
Mean	369
S.D.	46.0
% CV	12.5%
Control (ng/mL)	375
Mean % Dev. from Control	**-1.73%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

Table 52. Freeze/Thaw Stability for PFOSAA

All concentrations are expressed as ppb.

Run Number	**25**
Run Date	**30-Jan-1999**
	LOW QC
	After 3 Freeze/Thaw cycles
1	13.6
2	12.5
3	12.0
Mean	12.7
S.D.	0.819
% CV	6.45%
Control (ng/mL)	13.1
Mean % Dev. from Control	**-3.05%**
	HIGH QC
	After 3 Freeze/Thaw cycles
1	205
2	*180
3	**237
Mean	221
S.D.	22.6
% CV	10.2%
Control (ng/mL)	223
Mean % Dev. from Control	**-0.897%**

* sample deactivated – poor instrument response
** > ±20% theoretical
Control = mean of reference QC samples

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Table 53. Freeze/Thaw Stability for N-MeFOSE-OH

All concentrations are expressed as ppb.

Run Number	**25**
Run Date	**30-Jan-1999**
	LOW QC
	After 3 Freeze/Thaw cycles
1	18.9
2	11.8
3	15.5
Mean	15.4
S.D.	3.55
% CV	23.1%
Control (ng/mL)	16.0
Mean % Dev. from Control	**-3.75%**
	HIGH QC
	After 3 Freeze/Thaw cycles
1	320
2	213*
3	324
Mean	322
S.D.	2.83
% CV	0.878%
Control (ng/mL)	319
Mean % Dev. from Control	**0.940%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

Table 54. Freeze/Thaw Stability for N-EtFOSE-OH

All concentrations are expressed as ppb.

Run Number	**25**
Run Date	**30-Jan-1999**
	LOW QC
	After 3 Freeze/Thaw cycles
1	19.1
2	11.4
3	15.9
Mean	15.5
S.D.	3.87
% CV	25.0%
Control (ng/mL)	15.7
Mean % Dev. from Control	**-1.49%**
	HIGH QC
	After 3 Freeze/Thaw cycles
1	302
2	214*
3	307
Mean	305
S.D.	3.54
% CV	1.16%
Control (ng/mL)	315
Mean % Dev. from Control	**-3.33%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

Table 55. Freeze/Thaw Stability for POAA

All concentrations are expressed as ppb.

Run Number	**25**
Run Date	**30-Jan-1999**
	LOW QC
	After 3 Freeze/Thaw cycles
1	25.3
2	23.8
3	25.5
Mean	24.9
S.D.	0.929
% CV	3.74%
Control (ng/mL)	22.5
Mean % Dev. from Control	**10.5%**
	HIGH QC
	After 3 Freeze/Thaw cycles
1	330
2	144*
3	385
Mean	358
S.D.	38.9
% CV	10.9%
Control (ng/mL)	364
Mean % Dev. from Control	**-1.79%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

Table 56. Freeze/Thaw Stability for PFHS

All concentrations are expressed as ppb.

Run Number	**25**
Run Date	**30-Jan-1999**
	LOW QC
	After 3 Freeze/Thaw cycles
1	24.4
2	21.7
3	23.5
Mean	23.2
S.D.	1.37
% CV	5.93%
Control (ng/mL)	22.2
Mean % Dev. from Control	**4.50%**
	HIGH QC
	After 3 Freeze/Thaw cycles
1	310
2	145*
3	365
Mean	338
S.D.	38.9
% CV	11.5%
Control (ng/mL)	366
Mean % Dev. from Control	**-7.79%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

Table 57. Room Temperature Matrix Stability for PFOS

All concentrations are expressed as ppb.

Run Number	**32**	
Run Date	**10-Feb-1999**	
	LOW QC	
	2 Hour	***4 Hour***
1	39.5	38.4
2	41.0	36.9
3	40.8	37.5
Mean	40.4	37.6
S.D.	0.814	0.755
% CV	2.01%	2.01%
Control (ng/mL)	41.2	41.2
Mean % Dev. from Control	**-1.94%**	**-8.74%**
	HIGH QC	
	2 Hour	***4 Hour***
1	322	343
2	271	173*
3	276	349
Mean	290	346
S.D.	28.1	4.24
% CV	9.69%	1.23%
Control (ng/mL)	354	354
Mean % Dev. from Control	**-18.1%**	**-2.26%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

000067

Table 58. Room Temperature Matrix Stability for PFOSA

All concentrations are expressed as ppb.

Run Number	32	
Run Date	10-Feb-1999	
	LOW QC	
	2 Hour	*4 Hour*
1	20.0	18.7
2	20.3	17.2
3	20.7	18.2
Mean	20.3	18.0
S.D.	0.351	0.764
% CV	1.73%	4.24%
Control (ng/mL)	20.4	20.4
Mean % Dev. from Control	**-0.490%**	**-11.8%**
	HIGH QC	
	2 Hour	*4 Hour*
1	310	317
2	261	174*
3	276	336
Mean	282	327
S.D.	25.1	13.4
% CV	8.89%	4.1%
Control (ng/mL)	332	332
Mean % Dev. from Control	**-15.0%**	**-1.51%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

000068

Table 59. Room Temperature Matrix Stability for PFOSAA

All concentrations are expressed as ppb.

Run Number	32	
Run Date	10-Feb-1999	
	LOW QC	
	2 Hour	*4 Hour*
1	12.8	11.5
2	13.3	11.5
3	13.3	10.3
Mean	13.1	11.1
S.D.	0.289	0.693
% CV	2.21%	6.24%
Control (ng/mL)	12.7	12.7
Mean % Dev. from Control	**3.15%**	**-12.6%**
	HIGH QC	
	2 Hour	*4 Hour*
1	179	200
2	**138	*103
3	152	185
Mean	156	193
S.D.	20.8	10.6
% CV	13.3%	5.49%
Control (ng/mL)	194	194
Mean % Dev. from Control	**-19.6%**	**-0.515%**

* sample deactivated – poor instrument response
** > ±20% theoretical
Control = mean of reference QC samples

Table 60. Room Temperature Matrix Stability for N-MeFOSE-OH

All concentrations are expressed as ppb.

Run Number	**32**	
Run Date	**10-Feb-1999**	
	LOW QC	
	2 Hour	***4 Hour***
1	14.3	10.5
2	14.9	12.5
3	13.0	10.8
Mean	14.1	11.3
S.D.	0.971	1.08
% CV	6.90%	9.57%
Control (ng/mL)	14.4	14.4
Mean % Dev. from Control	**-2.31%**	**-21.8%**
	HIGH QC	
	2 Hour	***4 Hour***
1	248	218
2	199	131*
3	197	335
Mean	215	277
S.D.	28.9	82.7
% CV	13.5%	29.9%
Control (ng/mL)	286	286
Mean % Dev. from Control	**-24.9%**	**-3.15%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

Table 61. Room Temperature Matrix Stability for N-EtFOSE-OH

All concentrations are expressed as ppb.

Run Number	32	
Run Date	10-Feb-1999	
	LOW QC	
	2 Hour	*4 Hour*
1	15.1	11.9
2	14.8	12.7
3	13.1	12.0
Mean	14.3	12.2
S.D.	1.08	0.436
% CV	7.52%	3.57%
Control (ng/mL)	14.9	14.9
Mean % Dev. from Control	**-3.80%**	**-18.12%**
	HIGH QC	
	2 Hour	*4 Hour*
1	255	235
2	224	139*
3	210	339
Mean	230	287
S.D.	23.0	73.5
% CV	10.0%	25.6%
Control (ng/mL)	292	292
Mean % Dev. from Control	**-21.3%**	**-1.71%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

000071

Table 62. Room Temperature Matrix Stability for POAA

All concentrations are expressed as ppb.

Run Number	**32**	
Run Date	**10-Feb-1999**	
	LOW QC	
	2 Hour	***4 Hour***
1	22.3	20.9
2	22.8	20.1
3	24.4	20.1
Mean	23.2	20.4
S.D.	1.10	0.462
% CV	4.74%	2.27%
Control (ng/mL)	22.6	22.6
Mean % Dev. from Control	**2.51%**	**-9.88%**
	HIGH QC	
	2 Hour	***4 Hour***
1	313	327
2	221	171*
3	280	308
Mean	271	318
S.D.	46.6	13.4
% CV	17.2%	4.21%
Control (ng/mL)	324	324
Mean % Dev. from Control	**-16.3%**	**-1.85%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

Table 63. Room Temperature Matrix Stability for PFHS

All concentrations are expressed as ppb.

Run Number	32	
Run Date	10-Feb-1999	
	LOW QC	
	2 Hour	*4 Hour*
1	20.8	21.5
2	21.3	19.5
3	23.9	21.1
Mean	22.0	20.7
S.D.	1.66	1.06
% CV	7.57%	5.11%
Control (ng/mL)	21.8	21.8
Mean % Dev. from Control	**0.917%**	**-5.05%**
	HIGH QC	
	2 Hour	*4 Hour*
1	333	341
2	251	172*
3	281	306
Mean	288	324
S.D.	41.5	24.7
% CV	14.4%	7.62%
Control (ng/mL)	333	333
Mean % Dev. from Control	**-13.4%**	**-2.70%**

* sample deactivated – poor instrument response
Control = mean of reference QC samples

Table 64. Room Temperature Extract Stability for PFOS

All concentrations are expressed as ppb.

Run Number	**32**
Run Date	**10-Feb-1999**
	LOW QC *After 24 Hours*
1	35.9
2	36.5
3	32.3
Mean	34.9
S.D.	2.27
% CV	6.51%
Control (ng/mL)	41.2
Mean % Dev. from Control	**-15.3%**
	HIGH QC *After 24 Hours*
1	300
2	235
3	271
Mean	269
S.D.	32.6
% CV	12.1%
Control (ng/mL)	354
Mean % Dev. from Control	**-24.1%**

Control = mean of reference QC samples

Table 65. Room Temperature Extract Stability for PFOSA

All concentrations are expressed as ppb.

Run Number	**32**
Run Date	**10-Feb-1999**
	LOW QC *After 24 Hours*
1	19.7
2	19.4
3	16.1
Mean	18.4
S.D.	1.997
% CV	10.9%
Control (ng/mL)	20.4
Mean % Dev. from Control	**-9.80%**
	HIGH QC *After 24 Hours*
1	311
2	239
3	270
Mean	273
S.D.	36.12
% CV	13.2%
Control (ng/mL)	332
Mean % Dev. from Control	**-17.7%**

Control = mean of reference QC samples

Table 66. Room Temperature Extract Stability for PFOSAA

All concentrations are expressed as ppb.

Run Number	**32**
Run Date	**10-Feb-1999**
	LOW QC *After 24 Hours*
1	12.6
2	11.7
3	10.2
Mean	11.5
S.D.	1.21
% CV	10.5%
Control (ng/mL)	12.7
Mean % Dev. from Control	-9.45%
	HIGH QC *After 24 Hours*
1	167
2	*133
3	*147
Mean	149
S.D.	17.1
% CV	11.5%
Control (ng/mL)	194
Mean % Dev. from Control	-23.2%

* > ±20% theoretical
Control = mean of reference QC samples

000076

Table 67. Room Temperature Extract Stability for N-MeFOSE-OH

All concentrations are expressed as ppb.

Run Number	**32**
Run Date	**10-Feb-1999**
	LOW QC *After 24 Hours*
1	12.8
2	13.5
3	10.1
Mean	12.1
S.D.	1.80
% CV	14.8%
Control (ng/mL)	14.4
Mean % Dev. from Control	**-15.7%**
	HIGH QC *After 24 Hours*
1	276
2	257
3	242
Mean	258
S.D.	17.0
% CV	6.60%
Control (ng/mL)	286
Mean % Dev. from Control	**-9.67%**

Control = mean of reference QC samples

Table 68. Room Temperature Extract Stability for N-EtFOSE-OH

All concentrations are expressed as ppb.

Run Number	**32**
Run Date	**10-Feb-1999**
	LOW QC *After 24 Hours*
1	12.8
2	13.5
3	10.5
Mean	12.3
S.D.	1.57
% CV	12.8%
Control (ng/mL)	14.9
Mean % Dev. from Control	-17.7%
	HIGH QC *After 24 Hours*
1	289
2	265
3	247
Mean	267
S.D.	21.1
% CV	7.89%
Control (ng/mL)	292
Mean % Dev. from Control	-8.56%

Control = mean of reference QC samples

Table 69. Room Temperature Extract Stability for POAA

All concentrations are expressed as ppb.

Run Number	**32**
Run Date	**10-Feb-1999**
	LOW QC *After 24 Hours*
1	21.7
2	22.4
3	19.1
Mean	21.1
S.D.	1.74
% CV	8.25%
Control (ng/mL)	22.6
Mean % Dev. from Control	**-6.78%**
	HIGH QC *After 24 Hours*
1	290
2	222
3	247
Mean	253
S.D.	34.4
% CV	13.6%
Control (ng/mL)	324
Mean % Dev. from Control	**-21.9%**

Control = mean of reference QC samples

Table 70. Room Temperature Extract Stability for PFHS

All concentrations are expressed as ppb.

Run Number	**32**
Run Date	**10-Feb-1999**
	LOW QC *After 24 Hours*
1	20.7
2	19.9
3	18.1
Mean	19.6
S.D.	1.33
% CV	6.81%
Control (ng/mL)	21.8
Mean % Dev. from Control	**-10.2%**
	HIGH QC *After 24 Hours*
1	283
2	216
3	241
Mean	247
S.D.	33.9
% CV	13.7%
Control (ng/mL)	333
Mean % Dev. from Control	**-25.9%**

Control = mean of reference QC samples

Table 71. Stock Solution Stability for PFOS

	Analyte Peak Area
Prepared day of analytical run	121195
	128494
	125134
Mean	124941
SD	3653
% CV	2.92
n	3
Stored for 24 days	143208
	132499
	122485
Mean	132731
SD	10363
% CV	7.81
n	3
% Difference	6.23%

Table 72. Stock Solution Stability for PFOSA

	Analyte Peak Area
Prepared day of analytical run	41058
	34117
	84507
Mean	53227
SD	27310
% CV	51.3
n	3
Stored for 24 days	63582
	58872
	46572
Mean	56342
SD	8783
% CV	15.6
n	3
% Difference	5.85%

Table 73. Stock Solution Stability for PFOSAA

	Analyte Peak Area
Prepared day of analytical run	14044
	15654
	15150
Mean	14949
SD	824
% CV	5.51
n	3
Stored for 24 days	17264
	16136
	13572
Mean	15657
SD	1892
% CV	12.1
n	3
% Difference	4.74%

Table 74. Stock Solution Stability for N-MeFOSE-OH

	Analyte Peak Area
Prepared day of analytical run	115190
	90190
	52253
Mean	85878
SD	31689
% CV	36.9
n	3
Stored for 24 days	169447
	175317
	125816
Mean	156860
SD	27045
% CV	17.2
n	3
% Difference	82.7%

000084

Table 75. Stock Solution Stability for N-EtFOSE-OH

	Analyte Peak Area
Prepared day of analytical run	113772
	87610
	52034
Mean	84472
SD	30988
% CV	36.7
n	3
Stored for 24 days	160189
	166768
	115012
Mean	147323
SD	28175
% CV	19.1
n	3
% Difference	74.4%

Table 76. Stock Solution Stability for POAA

	Analyte Peak Area
Prepared day of analytical run	200388
	206992
	205145
Mean	204175
SD	3407
% CV	1.67
n	3
Stored for 24 days	200970
	200039
	183527
Mean	194845
SD	9813
% CV	5.04
n	3
% Difference	-4.57%

000086

Table 77. Stock Solution Stability for PFHS

	Analyte Peak Area
Prepared day of analytical run	212505
	221778
	218307
Mean	217530
SD	4685
% CV	2.15
n	3
Stored for 24 days	230603
	224907
	200109
Mean	218540
SD	16214
% CV	7.42
n	3
% Difference	0.464%

6. ANALYTICAL METHOD

Principles of the Method

After the addition of HPLC-grade water and sodium dodecyl sulfate (SDS, internal standard) to 0.20 mL of human or rabbit serum, the serum mixture is made basic with the addition of 0.5 M tetrabutylammonium hydrogen sulfate (TBA, pH 10) and 0.25 M carbonate buffer. This mixture is then extracted with methyl-tert-butyl ether. After sufficient mixing, the sample is centrifuged. The organic layer is transferred via pipette into a clean test tube. The organic layer is then evaporated to dryness and the sample is reconstituted into 2 mM ammonium acetate water:methanol (50:50 v/v). The extracts are then analyzed by liquid chromatography/tandem mass spectrometry using negative-ion electrospray ionization and multiple reaction monitoring.

CHEMICAL FORMULAS

Abbreviation	Chemical Name	Formula
PFOS	Perfluorooctane sulfonate	$C_8F_{17}SO_3^-$
PFOSA	Perfluorooctane sulfonylamide	$C_8F_{17}SO_2NH_2$
PFOSAA	Perfluorooctane sulfonylamido(ethyl)acetate	$C_8F_{17}SO_2N(CH_2CH_3)CH_2CO_2^-$
PFHS	Perfluorohexane sulfonate	$C_6F_{13}SO_3^-$
POAA	Perfluorooctanoate	$C_7F_{15}CO_2^-$
N-EtFOSE-OH	2(N-ethylperfluorooctanesulfonamidoethanol	$C_8F_{17}\ SO_2\ N(CH_2CH_3)(CH_2CH_2OH)$
N-MeFOSE-OH	2(N-methylperfluorooctanesulfonamidoethanol	$C_8F_{17}\ SO_2\ N\ (CH_3)(CH_2CH_2OH)$
SDS	Dodecyl sulfate (sodium)	$CH_3(CH_2)_{10}\ CH_2\ OSO_3^-$

6.1. Reference Materials and Matrices

Reference Material	*Lot Number*	*Purity*	*Expiration Date*	*Source*	*Storage Conditions*
PFOS	193	100%	12/31/2010	3M	Room Temperature
PFOSA	214	100%	12/31/2010	3M	Room Temperature
PFOSAA	617	53.8%	12/31/2010	3M	Room Temperature
N-MeFOSE-OH	212	100%	12/31/2010	3M	Room Temperature
N-EtFOSE-OH	936	100%	12/31/2010	3M	Room Temperature

Reference Material	*Lot Number*	*Purity*	*Expiration Date*	*Source*	*Storage Conditions*
POAA	245	100%	12/31/2010	3M	Room Temperature
PFHS (in methanol)	S398-182	100% (6200 ppm)	12/31/2010	3M	- 20 °C
SDS (sodium dodecyl sulfate)	17H0459	91.0%	1/16/2001	Sigma	Room Temperature

Matrix	*Source*
Human serum	Biochemed
Rabbit serum	Biochemed

6.2. Chemicals and Equipment

Chemicals
Ammonium Acetate, 99.9 %
Water, HPLC-grade
Methanol, HPLC-grade
Methyl-*tert*-Butyl Ether, HPLC-grade
Sodium Hydroxide, 98.4%
Sodium Carbonate, 101.0%
Sodium Bicarbonate, 100.3%
Tetrabutylammonium Hydrogen Sulfate (TBA), approx. 97%

Equipment / Supplies	
Model	*Manufacturer*
Balance: Mettler Toledo AT261	Mettler-Toledo, Inc., Hightstown, NJ
Balance: Mettler Toledo MT5	Mettler-Toledo, Inc., Hightstown, NJ
Centrifuge: Beckman GS-6R	Beckman Instruments, Fullerton, CA
Evaporator: Turbo Vap LV, Model 43750	Zymark Corp., Hopkinton, MA
Liquid Chromatograph: Hewlett Packard 1100	Hewlett Packard, Palo Alto, CA
Mass Spectrometer: Perkin Elmer Sciex API 3000	Perkin Elmer Sciex, Thornhill,

Equipment / Supplies	
Model	*Manufacturer*
	Ontario
pH Meter: Orion 230A	Orion, Boston, MA
Pipettes: Finnpipette: Digital 1-5 mL	Fisher Scientific, Pittsburgh, PA
Pipettes: Rainin Pipetman™ Volumes: fixed20; adjust.: 20, 100; 200; 1000 µL	Rainin Instrument Co., Woburn, MA
Sonicator	Branson, Danbury, CT
Stir Plate: Nuova II	Thermolyne Corp., Dubuque, IA
Vortex: Fisher Genie 2	Fisher Scientific, Fair Lawn, NJ

6.3. Reagents, Calibration Standard and Quality Control (QC) Solutions

The calibrator, quality control and stock solution concentrations listed below are reported as they were prepared for the validation study and serve as a general guideline for future preparations.

Reagents

All reagent solutions are stored at room temperature.

- *0.25 M carbonate buffer*

  Add approximately 500 mL HPLC-grade water to an appropriate 1-L container. Add 26.5 g of sodium carbonate (Na_2CO_3) to the 1-L container. Add 21.0 g of sodium bicarbonate ($NaHCO_3$) to the 1-L container. Fill to volume with HPLC-grade water. Use a stir plate to mix.

- *10 N sodium hydroxide*

  Add 400 g of sodium hydroxide (NaOH) to an appropriate 1-L container. ***Slowly*** add approximately 900 mL of HPLC-grade water while mixing. After the NaOH is completely dissolved, allow the solution to cool to room temperature. Fill to volume with HPLC-grade water and mix. ***Caution:*** The solution will become very hot as water is added! Prepare in the hood.

- *0.5 M Tetrabutylammonium Hydrogen Sulfate (TBA, pH 10)*

  Add approximately 50 mL of HPLC-grade water to an appropriate 100-mL container. Add 16.9 g of TBA to the container. Adjust the pH with 10 N sodium hydroxide. Fill to volume with HPLC-grade water and mix. ***Note:*** Be sure to check the pH daily when in use.

- *2 mM ammonium acetate in water*

  Add 1 mL of 1 M ammonium acetate to a 500-mL container approximately half full with HPLC-grade water. QS to volume with HPLC-grade water and mix by inversion.

- *2 mM ammonium acetate in methanol*

  Fill an appropriate 1-L container approximately half full with methanol. Weigh out 0.154 g of ammonium acetate (NH_4 OAc) and transfer to the container. Use a stir plate to mix. Adjust the pH if desired and fill to volume with methanol.

- *2 mM ammonium acetate water:methanol (50:50 v/v)*

  Fill an appropriate 1-L container with 500 mL of 2 mM ammonium acetate in water and 2 mM ammonium acetate in methanol.

<u>Calibration Standard and Quality Control (QC) Solutions</u>

All calibration standard solutions are transferred to 16 x 100 mm polypropylene screw-cap tubes and stored in a -20 °C freezer.

- *PFOS Stock Standard (1053 ppm)*

  Weigh 10.527 mg of PFOS and transfer to a 10-mL volumetric flask. QS to volume with methanol. Sonicate for approximately 5 minutes.

- *PFOSA Stock Standard (1078 ppm)*

  Weigh 10.778 mg of PFOSA and transfer to a 10-mL volumetric flask. QS to volume with methanol. Sonicate for approximately 5 minutes.

- *PFOSAA Stock Standard (1031 ppm)*

  Weigh 10.310 mg of PFOSAA and transfer to a 10-mL volumetric flask. QS to volume with methanol. Sonicate for approximately 5 minutes.

- *N-MeFOSE-OH Stock Standard (1210 ppm)*

  Weigh 12.103 mg of N-MeFOSE-OH and transfer to a 10-mL volumetric flask. QS to volume with methanol. Sonicate for approximately 5 minutes.

- *N-EtFOSE-OH Stock Standard (1127 ppm)*

  Weigh 11.266 mg of N-EtFOSE-OH and transfer to a 10-mL volumetric flask. QS to volume with methanol. Sonicate for approximately 5 minutes.

- *POAA Stock Standard (1090 ppm)*

  Weigh 10.9 mg of POAA and transfer to a 10-mL volumetric flask. QS to volume with methanol. Sonicate for approximately 5 minutes.

- *PFHS Stock Standard (1000 ppm)*

  Add 807 µL of PFHS (6200 ppm) to a 5-mL volumetric flask. QS to volume with methanol. Mix by inversion.

- *Diluted Stock Solution (10.0 ppm)*

  Add 95 µL of PFOS Stock Solution (1053 ppm), 93 µL of PFOSA Stock Solution (1078 ppm), 97 µL of PFOSAA Stock Solution (1031 ppm), 83 µL of N-MeFOSE-OH Stock Solution (1210 ppm), 89 µL of N-EtFOSE-OH Stock Solution (1127 ppm), 92 µL of POAA Stock Solution (1090 ppm) and 100 µL of PFHS Stock Solution (1000 ppm) to a 10-mL volumetric flask. QS to volume with methanol. Mix by inversion.

- *Spiking Standard 8 (5.00 ppm)*

  Add 500 µL of methanol and 500 µL of Diluted Stock Solution (10.0 ppm) to a 13 x 100 mm silanized glass tube. Mix by vortexing.

- *Spiking Standard 7 (2.50 ppm)*

  Add 500 µL of methanol and 500 µL of Spiking Standard 8 (5.00 ppm) to a 13 x 100 mm silanized glass tube. Mix by vortexing.

- *Spiking Standard 6 (1.00 ppm)*

  Add 800 µL of methanol and 200 µL of Spiking Standard 7 (5.00 ppm) to a 13 x 100 mm silanized glass tube. Mix by vortexing.

- *Spiking Standard 5 (0.500 ppm)*

  Add 900 µL of methanol and 100 µL of Spiking Standard 7 (5.00 ppm) to a 13 x 100 mm silanized glass tube. Mix by vortexing.

- *Spiking Standard 4 (0.250 ppm)*

  Add 950 µL of methanol and 50 µL of Spiking Standard 7 (5.00 ppm) to a 13 x 100 mm silanized glass tube. Mix by vortexing.

- *Spiking Standard 3 (0.100 ppm)*

  Add 900 µL of methanol and 100 µL of Spiking Standard 6 (1.00 ppm) to a 13 x 100 mm silanized glass tube. Mix by vortexing

- *Spiking Standard 2 (0.0500 ppm)*

  Add 900 µL of methanol and 100 µL of Spiking Standard 5 (0.500 ppm) to a 13 x 100 mm silanized glass tube. Mix by vortexing

- *Spiking Standard 1 (0.0100 ppm)*

  Add 900 µL of methanol and 100 µL of Spiking Standard 3 (0.100 ppm) to a 13 x 100 mm silanized glass tube. Mix by vortexing

<u>Internal Standard Solutions</u>

All internal standard solutions are transferred to 16 x 100 mm polypropylene screw-cap tubes and stored in a -20 °C freezer.

- *SDS Stock Solution (1091 ppm)*

  Weigh 5.986 mg of SDS and transfer to a 5-mL volumetric flask. QS to volume with methanol. Sonicate for 10 minutes.

- *SDS Diluted Stock Solution (10.0 ppm)*

  Add 91.7 µL of SDS Stock Solution (1091 ppm) to a 10-mL volumetric flask. QS to volume with methanol and mix by inversion.

- *SDS Working Internal Standard (1.00 ppm)*

  Add 2.50 mL of SDS Diluted Stock Solution (10 ppm) to a 25-mL class A volumetric flask. QS to volume with methanol and mix by inversion.

6.4. Preparation of Quality Control Samples

High Quality Control (350 ppb spiked)

Quantitatively transfer 1.05 mL of the Diluted Stock Solution (10.0 ppm) and 28.95 mL of blank human serum into a polypropylene tube. Sonicate for approximately 10 minutes and equilibrate for approximately 10 minutes.

Low Quality Control (20.0 ppb spiked)

Quantitatively transfer 60 µL of the Diluted Stock Solution (10.0 ppm) and 29.94 mL of blank human serum into a polypropylene tube. Sonicate for approximately 10 minutes and equilibrate for approximately 10 minutes.

Storage of QC Samples

After preparation, place aliquots of the low and high QC pools into 2-mL cryogenic vials, and store in a -20 °C freezer.

6.5. Preparation of Calibration Standards

If endogenous analyte concentrations in human serum need to be calculated, a rabbit serum calibration curve is prepared by spiking standards as shown in the table below. The calibration curve range is 1.00 ppb to 50.0 ppb.

Standard Number	Concentration of Spiking Solution (ppm)	Volume of Spiking Solution (µL)	Volume of Blank Rabbit Serum (mL)	Final Spiked Concentration (ppb)
5	0.500	20	0.20	50.0
4	0.250	20	0.20	25.0
2	0.0500	20	0.20	5.00
1	0.0100	20	0.20	1.00

The human serum calibration curve is prepared on the day of each run by spiking standards as shown in the table below. The unadjusted calibration curve range is 1.00 ppb to 500 ppb.

Standard Number	Concentration of Spiking Solution (ppm)	Volume of Spiking Solution (µL)	Volume of Blank Human Serum (mL)	Final Spiked Concentration (ppb)
8	5.00	20	0.20	500
7	2.50	20	0.20	250
6	1.00	20	0.20	100
5	0.500	20	0.20	50.0
4	0.250	20	0.20	25.0
3	0.100	20	0.20	10.0
2	0.0500	20	0.20	5.00
1	0.0100	20	0.20	1.00

6.6. Sample Preparation

Calibration Curve Samples (prepare in duplicate)

Rabbit Serum Curve

Transfer 0.20 mL of HPLC-grade water and 0.20 mL rabbit serum into appropriately labeled 13 x 100 mm polypropylene tubes. Add 20 µL of Calibration Standard Spiking Solutions 1, 2, 4 and 5 to the corresponding labeled tube.

Human Serum Curve

Transfer 0.20 mL of HPLC-grade water and 0.20 mL human serum into appropriately labeled 13 x 100 mm polypropylene tubes. Add 20 µL of Calibration Standard Spiking Solutions 1-8 to the corresponding labeled tube.

Quality Control Samples (prepare in duplicate)

Aliquot 0.20 mL of each of the Low and High controls into appropriately labeled 13 x 100 mm polypropylene tubes.

Aliquot 0.20 mL of blank human serum into separate 13 x 100 mm polypropylene tubes and label as QC0.

Aliquot 0.20 mL of blank human serum into separate 13 x 100 mm polypropylene tubes and label as BLANK

Aliquot 0.20 mL of blank rabbit serum into separate 13 x 100 mm polypropylene tubes and label as RABBIT BLANK.

Study Samples

Transfer 0.20-mL aliquots of each study sample into appropriately labeled 13 x 100 mm polypropylene tubes.

Extraction Procedure

1. Add 0.20 mL of HPLC-grade water (this should be performed before adding the calibration standard spiking solutions to calibration curve samples).
2. Add 20 µL of SDS internal standard (1.00 ppm) to all samples, excluding BLANKS.
3. Vortex samples for at least 5 seconds.
4. Add 0.40 mL 0.5 M tetrabutylammonium hydrogen sulfate (TBA), pH 10, and 0.40 mL of 0.25 M carbonate buffer. NOTE: Check the pH of TBA daily.
5. Add 3.0 mL methyl-*tert*-butyl ether.

6. Cap each sample, vortex and rotate for approximately 10 minutes.
7. Centrifuge at approximately 3500 rpm for 10 minutes (or until the layers are well separated).
8. Transfer the organic layer to a clean 13 x 100 mm polypropylene tube with a disposable transfer pipet.
9. Dry samples under nitrogen at 45 °C until dry (approximately 10 minutes).
10. Add 75 µL of 2 mM ammonium acetate water:methanol (50:50 v/v) to each tube.
11. Vortex for at least 5 seconds.
12. Transfer into autosampler vial insert.
13. Cap and store extracts at 4 °C until analysis.

6.7. LC/MS/MS Conditions

LC Conditions

Column I.D.	Betasil C-18
Mobile Phase(s)	A: 2mM ammonium acetate in water
	B: 2mM ammonium acetate in methanol
Flow Rate	300 µL/min
Column Temperature	50 °C
Injection Volume	5 - 15 µL
LC Conditions	Gradient

The HPLC gradient required is shown below.

Time (min.)	% Mobile Phase A	% Mobile Phase B
0.00	50	50
4.50	50	50
11.0	2.7	97.3
12.0	2.7	97.3
13.0	50	50

MS Conditions

Post Column Split	2:1
Source	Turbo Ion Spray
Source Temp.	300 °C
Analysis type	Multiple reaction monitoring (MRM)

Compounds	Transitions monitored	Dwell Time (ms)	Collision Energy (v)
PFOS	499 / 80	100	91
PFOSA	498 / 78	100	38
PFOSAA	584 / 419	200	30
N-MeFOSE-OH	616 / 59	100	52
N-EtFOSE-OH	630 / 59	100	58
POAA	413 / 369	100	15
PFHS	399 / 80	100	48
SDS	265 / 97	100	41

The prepared standards and QCs are loaded onto the autosampler tray in a random order and injected into the instrument LC/MS/MS system.

6.8. Quantitation

PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA, PFHS and SDS chromatographic peaks are integrated using the instrument manufacturer supplied software with a smooth factor of 1. Quantitation of the rabbit calibration curve is based upon linear regression analysis of calibration curves (weighted 1/x) using the analyte peak areas. This quantitation is performed using the instrument manufacturer supplied software. Quantitation of the human calibration curve is based upon quadratic regression analysis of calibration curves (weighted 1/x) using the peak area ratio (analyte peak area/internal standard peak area) vs. concentration. This quantitation is performed using the Watson® DMLIMS software.

Figure 1. Total Ion Chromatogram of Rabbit Serum Blank

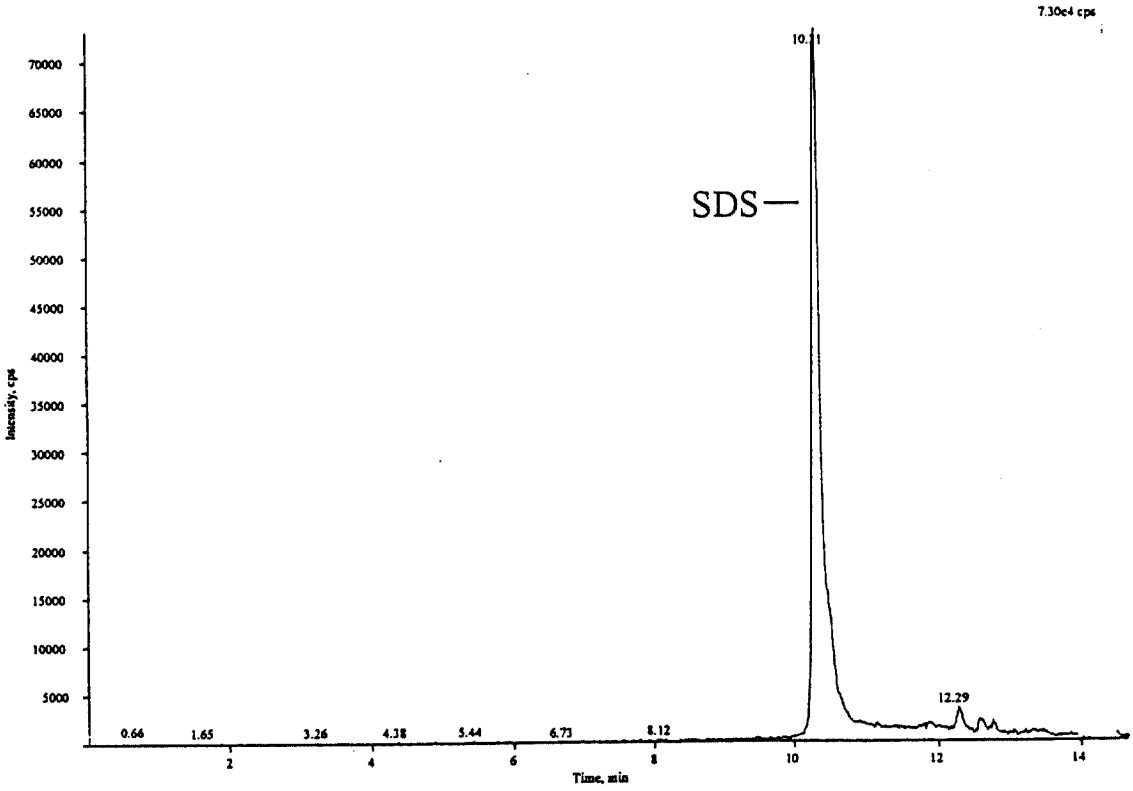
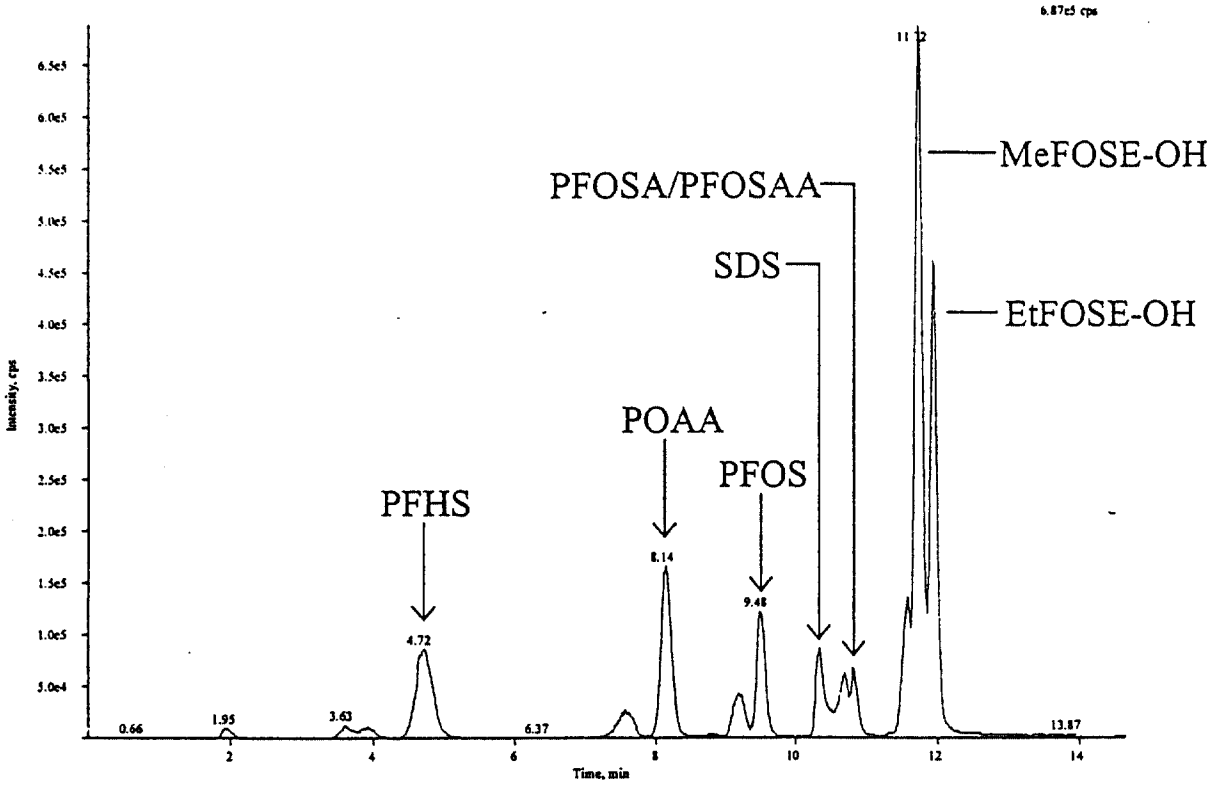


Figure 3. Total Ion Chromatogram of High Standard (500 ppb)



AR226 - 1209

Study No. NWBS00-040
Report No. NWBR00-108

Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS

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Assay Revalidation Report

Northwest Bioanalytical (NWB)
A Division of NWT Inc.
1121 East 3900 South
Salt Lake City, UT 84124

PREPARED FOR: 3M Environmental Technology and Services (3M)
935 Bush Avenue
St. Paul, MN 55133

AUTHOR:
Connie O Sakashita
Connie O. Sakashita, B.S., NWB Project Manager
DATE: 1/24/01

APPROVED FOR RELEASE BY:
Patrick Bennett
Patrick Bennett, M.S., M.B.A., NWB Laboratory Director
DATE: 1/24/01

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QUALITY ASSURANCE STATEMENT

LABORATORY:	Northwest Bioanalytical (NWB) A Division of NWT, Inc. 1121 East 3900 South Salt Lake City, UT 84124
SPONSOR:	3M Environmental Technology and Services (3M) 935 Bush Avenue St. Paul, MN 55133
COMPOUND(S):	PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570
NWB STUDY NUMBER:	NWBS00-040
SPONSOR STUDY NUMBER:	NA, Assay Validation
NWB STUDY TITLE:	Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS, Assay Validation

The assay validation study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all studies within the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.) The following inspections were performed by the NWB QAU per SOP.

Inspection and Reporting Statement.

Inspection Date	Phase of Study	Date Inspection Report Issued To: NWB Project Manager	Date Inspection Report Issued To: *NWB Management
15 May 2000	Analytical Plan	15 May 2000	31 May 2000
15 – 18 May 2000	Assay Validation	18 May 2000	31 May 2000
07 – 18 Sep 2000	Report Draft/Raw Data	20 Sep 2000	29 Sep 2000
24 Jan 2001	Final Report	24 Jan 2001	31 Jan 2001

*Reports to NWB Management are issued monthly.

As can reasonably be established, the methods and procedures described and the results incorporated into this final report accurately reflect the raw data.

Shaundel Percey, B.S., M.T., ASCP
Quality Assurance Compliance Specialist

24 January 2001
Date

000102

COMPLIANCE STATEMENT

The method validation study described in this report is not included within the definition of a GLP nonclinical regulated study. However, to the best of our knowledge, this study was conducted in accordance with the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58) and according to the methods and procedures described within this report. In addition, the study followed the guidelines of the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.) Any known circumstances that may have affected the quality or integrity of the study or data are discussed within the report. This report represents an accurate record of the raw data.

Connie O Sakashita DATE: 1/24/01

Connie O. Sakashita, B.S.
NWB Project Manager

Patrick Bennett DATE: 1/24/01

Patrick Bennett, M.S., M.B.A.
NWB Laboratory Director

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A – Inter Assay P/A for PFOSA + PFOSAA QC samples from NWL Study 00-062 July/Aug 2000

B – Calculation of persistent levels in diluted human serum (examples)

000107

Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS

Assay Revalidation Report

1. INTRODUCTION

Northwest Bioanalytical (NWB) was contracted by 3M Environmental Technology and Services to develop and validate a liquid chromatography/tandem mass spectrometry method for the measurement of perfluorooctanesulfonate (PFOS), perfluorooctanesulfonamide (PFOSA), N-ethyl perfluorooctanesulfonamidoacetate (PFOSAA), perfluorooctanoate (POAA), perfluorohexanesulfonate (PFHS), perfluorooctanesulfonamidoacetate (M556) and N-methyl perfluorooctanesulfonamidoacetate (M570) in human serum.

The reference material purity for PFOS, PFOSA, PFHS and POAA was not available prior to the conduct of this study. Therefore, all concentrations included in the report for these analytes are based upon an assumed purity of 100%.

After the validation was completed, 3M contracted with Centre Analytical Laboratories, Inc. in State College, Pennsylvania to determine the absolute concentration of PFOS, POAA and PFHS in the stock solutions used to prepare the analytical standards and controls used for this validation and subsequent analyses. Based on the results obtained, the concentrations included in this report should be corrected according to the following table:

Analyte	Correction Factor
PFOS	0.836
POAA	0.909
PFHS	0.855

This report summarizes the accuracy, precision and the Northern Chinese plasma extraction efficiency results from the validation of the method for the quantitation of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in human serum for 3M Environmental Technology and Services. Stability results will be reported in a separate addendum report. Kris Hansen at 3M Environmental Technology and Services served as the Study Monitor. The following is a list of NWB supervisory personnel involved in the completion of this work: Connie O. Sakashita, B.S. (NWB Project Manager); Brad I. Coopersmith, Ph.D. (NWB Senior Scientist); Licong Jiang, Ph.D. (NWB Senior Scientist); Patrick Bennett, M.S., M.B.A. (NWB Laboratory Director); Rodger Foltz, Ph.D. (NWB Technical Director).

NWB SOPs were used in the conduct of this project and were available to project personnel in both electronic and hard copy formats.

Date Study Initiated: April 13, 2000 Date Analyses Completed: July 16, 2000

The method validation study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all studies within the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424). Any changes to or deviations from the original protocol (Analytical Plan) were documented through approved protocol amendments or deviation memos and are retained within the raw data.

Principles of the Method

The newly developed method is a modification of a previous method developed under study NWBS98-082 and reported in NWB report NWBR99-005 [5.1]. The new method was developed to provide improved accuracy, precision and ruggedness using less sample volume, and to add M556 and M570 to the method.

The analytical method consisted of a liquid:liquid extraction procedure followed by evaporation and reconstitution of the extract residue with 30:70 20 mM ammonium acetate in water: 20 mM ammonium acetate in methanol (v/v). The samples were analyzed by liquid chromatography/tandem mass spectrometry using a PE Sciex API 3000. The instrument was operated in the multiple reaction monitoring (MRM) mode under optimized conditions for PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 detection.

2. VALIDATION SUMMARY

Three separate analytical runs were used in the determination of linearity, precision, and accuracy. An additional run was used to determine intra-assay precision and accuracy for PFOSA and PFOSAA. The extraction efficiencies for all analytes and the internal standard 1H,1H,2H,2H-perfluorooctane sulfonic acid (THPFOS) were also determined.

2.1. Persistent Levels of Analytes in Matrix

Because PFOS, PFOSAA, POAA, PFHS, M556 and M570 demonstrate measurable levels in control human serum, a procedure to account for these persistent levels is needed for this method validation.

The sponsor provided NWB with blank matrix (Northern Chinese human plasma). This matrix was tested and demonstrated no quantifiable concentrations for any of the analytes. In order to determine the persistent levels of these analytes in the lot of human serum used for the validation, a partial calibration curve was prepared with the blank Northern Chinese human plasma and extracted along with blank human serum samples. The human serum samples were quantitated against the Northern Chinese human plasma calibration curve to determine the persistent levels of the analytes in the lot of human serum being tested.

Once the persistent level of each analyte was determined, the target concentrations for each calibration standard and quality control sample were adjusted to account for the persistent amount of analyte. For example, if the target concentration at the lower

limit of quantitation was 1.00 ng/mL and the persistent level of the analyte in human serum was 2.00 ng/mL, the target concentration of the LLOQ was adjusted to 3.00 ng/mL. Appendix B outlines the procedure used to calculate persistent levels of analytes in diluted human serum samples.

This procedure to account for persistent levels of the analytes in matrix must be performed whenever a new lot of human serum is used during the course of a study.

2.2. Range of Quantitation

Each analytical run included calibration standards in duplicate at nine different concentrations (eight for M556), a minimum of six quality control samples (QCs) (three levels in replicates of two), two serum blanks and two 0-ng/mL QCs (serum blank with internal standard).

The target calibrator concentrations were approximately 1.00 , 2.50, 10.0, 25.0, 50.0, 100, 250, 400 and 500 ng/mL for all analytes. Each analyte has a different final curve range based upon the persistent levels of the analyte in the human serum used. For the validation study, the calibrator concentrations were as follows:

PFOS	48.1, 49.6, 57.1, 72.1, 97.1, 147, 297, 447, 547
PFOSA	1.00, 2.51, 10.1, 25.1, 50.3, 100, 251, 402, 502
PFOSAA	6.00, 7.50, 15.0, 30.0, 55.0, 105, 255, 405, 505
POAA	5.76, 7.26, 14.8, 29.8, 54.8, 105, 255, 405, 505
PFHS	3.15, 4.65, 12.2, 27.2, 52.2, 102, 252, 402, 502
M556	4.30, 11.8, 26.8, 51.8, 102, 252, 402, 502
M570	5.60, 7.10, 14.6, 29.6, 54.6, 105, 255, 405, 505

	PFOS	PFOSA	PFOSAA	POAA
Range	48.1 to 547 ng/mL	1.00 to 502 ng/mL	6.00 to 505 ng/mL	5.76 to 505 ng/mL
Mean Coefficient of Determination	0.9925 (Table 1)	0.9899 (Table 2)	0.9968 (Table 3)	0.9922 (Table 4)

	PFHS	M556	M570
Range	3.15 to 502 ng/mL	4.30 to 502 ng/mL	5.60 to 505 ng/mL
Mean Coefficient of Determination	0.9954 (Table 5)	0.9935 (Table 6)	0.9967 (Table 7)

The results for the individual calibration standards can be found in Tables 8 – 14.

2.3. Precision and Accuracy

The target concentrations for the quality control samples were 4.00, 150, 400 and 4000 ng/mL for all analytes. All quality control target concentrations were corrected for the persistent levels of the analytes in the human serum used for preparation. For the validation study, the quality control concentrations were as follows:

	Low (ng/mL)	Medium (ng/mL)	High (ng/mL)	Dilution (ng/mL)
PFOS	51.1	197	446	4460
PFOSA	4.00	150	400	4000
PFOSAA	9.00	155	405	4050
POAA	8.74	154	403	4030
PFHS	6.15	152	402	4020
M556	5.80	152	402	4020
M570	8.60	155	405	4050

2.3.1. Precision and Accuracy for PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 Quality Controls

The precision and accuracy of the LC/MS/MS method for the quantitation of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in human serum were determined by analyzing three levels of quality controls in replicates of five on three separate days.

The intra-assay precision (%CV) for PFOS, POAA, PFHS, M556 and M570 were less than or equal to 9.5% for each undiluted QC concentration. The mean accuracy (%Theoretical) for all levels of undiluted quality controls ranged from 84.8% to 111.2% (Tables 15 and 18 – 21). The intra-assay precision (%CV) for PFOSA and PFOSAA were less than or equal to 19.8% for each undiluted QC concentration. The mean accuracy (%Theoretical) for all levels of undiluted QCs ranged from 68.7% to 91.7% (Tables 16 – 17).

The inter-assay precision (%CV) for PFOS, POAA, PFHS, M556 and M570 were less than or equal to 9.7% for each undiluted QC concentration. The mean accuracy (%Theoretical) for all levels of undiluted QCs ranged from 87.4% to 108.5% (Tables 22 and 25 – 28). The inter-assay precision (%CV) for PFOSA and PFOSAA were less than or equal to 14.0% for each undiluted QC concentration. The mean accuracy (%Theoretical) for all levels of undiluted QCs ranged from 73.1% to 88.9% (Tables 23 – 24).

For diluted QCs, the inter-assay precision (%CV) for PFOS, POAA, PFHS, M556 and M570 were less than or equal to 6.6%. The mean accuracy (%Theoretical) for the diluted QCs ranged from 92.8% to 113.9% (Tables 31 and 34 – 37). The inter-assay precision (%CV) for PFOSA and PFOSAA were less than or equal to 9.7% for each diluted QC. The mean accuracy (%Theoretical) for the diluted QCs ranged from 79.0% to 81.3% (Tables 32 – 33).

The slightly high negative bias for the calculated concentrations of the PFOSA and PFOSAA QC samples was a result of the QC preparation procedure. A stock solution containing all the analytes was prepared and evaporated to dryness in order to obtain a concentration high enough for the QC concentration range. This methodology is commonly used when stock solution concentrations are too dilute to obtain the targeted concentration or to reduce the organic content of spiked serum samples. For this preparation, evaporation was required to obtain appropriate concentrations. However, because of the high volatility of PFOSA and PFOSAA, approximately 25% of these analytes evaporated from the solution. The quality control samples from this preparation were biased approximately 25% lower (Tables 23 – 25) than those prepared from a solution that was not evaporated (Tables A.1. – A.2.).

2.3.2. Precision and Accuracy for PFOSA and PFOSAA Specific Quality Controls

Because of the bias demonstrated for PFOSA and PFOSAA calculated concentrations from the original QCs prepared as described in section 2.3.1, a new set of QCs containing only PFOSA and PFOSAA were prepared using concentrated solutions that did not require evaporation.

For the PFOSA and PFOSA specific QCs, the intra-assay precision (%CV) for PFOSA and PFOSAA was less than or equal to 23.8% for each undiluted QC concentration. The mean accuracy (%Theoretical) for all levels of undiluted QCs ranged from 72.9% to 107.7% (Tables 29 – 30).

The PFOSAA LLOQ calibration standards for the intra-assay Run 17 demonstrated unacceptable accuracies resulting in both replicates being rejected and a raised LLOQ of 7.50 ng/mL for the run. The low QCs for PFOSAA also demonstrated variability greater than 20%. This resulted in a negative bias (72.9% of theoretical) for the low PFOSAA QCs. The mid-level and high-level QCs demonstrated acceptable precision and accuracy. Because this bias only appears at the Low QC level and is a result of within-run

variability, study sample analysis acceptance criteria will be maintained at 20% precision and accuracy for QCs.

For the PFOSA and PFOSAA specific QCs containing PFOSA or PFOSAA concentrations above the ULOQ, the intra-assay precision (%CV) for PFOSA and PFOSAA was 19.8% and 20.9%, respectively. The mean accuracy (%Theoretical) for diluted PFOSA and PFOSAA specific QCs were 78.3% and 78.0%, respectively (Tables 38 – 39). The deviation of the calculated concentrations from theoretical concentrations for these QCs diluted with control matrix were both greater than 20%. This indicates that samples with experimentally calculated PFOSA or PFOSAA concentrations above the ULOQ should not be diluted with control matrix. However, the levels of PFOSA and PFOSAA in study samples are expected to be within the range of the calibration curve.

The inter-assay precision and accuracy of the PFOSA and PFOSAA specific QCs was demonstrated with the analytical QC performance during sample analysis study NWBS00-062 (medical surveillance). The results can be found in Appendix A, Tables A.1. and A.2. These tables only contain analyses for reported PFOSA and PFOSAA sample data, and do not include runs that did not meet the acceptance criteria for PFOSA or PFOSAA.

For the PFOSA and PFOSA specific QCs, the inter-assay precision (%CV) was less than or equal to 11.4% for each undiluted QC concentration. The mean accuracy (%Theoretical) for all levels of undiluted QCs ranged from 98.5% to 107.7%.

2.4. Extraction Efficiency

The extraction efficiencies of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 were determined using Northern Chinese human plasma by comparing the area ratios obtained for the following three cases:

1. Both the analyte and internal standard added following the extraction (post-extract).
2. The analyte added to plasma prior to extraction and the internal standard added following extraction (pre-extract analyte).
3. The internal standard added to plasma prior to extraction and the analyte added following the extraction (pre-extract internal standard).

The extraction efficiencies were then determined by the area ratio of the pre-extract samples to the post-extract samples. The extraction efficiency experiments were performed at both low, medium and high concentrations to determine that there was no concentration bias. The mean extraction efficiencies were: PFOS (42.1%), PFOSA (65.3%), PFOSAA (73.4%), POAA (18.0%), PFHS (19.7%), M556 (43.9%), M570 (66.9%). The overall mean extraction efficiency for the internal standard THPFOS was 7.86% (Tables 40 – 46).

The recovery for many of the analytes and for the internal standard are very low. This low recovery is a result of the following factors: a neutral pH was required to minimize any matrix effects that were observed from the previous extraction method which used a basic pH and, the wide range of polarities for the analytes prevented the use of acidic pH during the extraction and prevented the use of an effective SPE extraction for all of the analytes. While the recovery is low, the intra and inter-assay precision and accuracy values demonstrated good reproducibility. The low recovery should not affect the assay performance.

The Chinese plasma containing very low persistent levels of most analytes was used to estimate the extraction recovery of the analytes from matrix. This plasma was used to provide the most accurate results possible at the lower concentrations. The recovery from general population control serum will be evaluated and reported in an addendum report.

2.5. Stability Evaluation

The stability of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 maintained under various storage conditions will be documented in a separate NWB report.

3. DATA MANAGEMENT

PFOS, PFOSA, PFOSAA, POAA, PFHS, M556, M570 chromatographic peaks are integrated using PE Sciex MacQuan software (version 1.6) with a smooth factor of 1. Quantitation is based upon quadratic regression analysis of calibration curves (weighted $1/x^2$) using the area ratio vs. concentration calculated by the Watson® DMLIMS software (version 6.1.1.04).

4. COMMENTS AND CONCLUSIONS

Per agreement with the Sponsor, the regressions were not recalculated based upon the updated purity information for PFOS, POAA and PFHS. Some differences might occur due to the effect of rounding if the regressions were performed with the purity corrected concentrations, but these differences would have a negligible effect on the overall interpretation of the validation results.

Only THPFOS was used as an internal standard because of concerns over the specificity of one of the original internal standards, N-Et-FOSE-OH, which gave an acetate ion as a product.

The volatility of PFOSA and PFOSAA is an important aspect to be cautious of during the evaporation step of the assay. An evaporation step was used for the initial preparation of QC samples to obtain an adequate concentration of the analytes. This set demonstrated acceptable precision, but slightly high negative bias for PFOSA and PFOSAA. A separate set of QCs containing only PFOSA and PFOSAA were prepared without using an evaporation step. The second set (PFOSA and PFOSAA specific QCs) demonstrated

both acceptable precision and accuracy. As a result, two sets of intra-assay and inter-assay precision and accuracy are shown in the report.

The method described in this report has been validated for the determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in human serum. However, study samples with experimentally calculated PFOSA or PFOSAA concentrations above the ULOQ should not be diluted with control matrix. Any known circumstances that may have affected the quality or integrity of the data are discussed in this report.

4.1. Proposed Sample Analysis Acceptance Criteria

Calibration Curve

Each run will include in duplicate calibration standards at six or more concentrations covering the lower to upper limit of quantitation. For all analytes except PFOSA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 15% (± 20% for LLOQ) of their individual target concentrations. For PFOSA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 20% (± 25% for LLOQ) of their individual target concentrations. A calibration standard will be considered a statistical outlier if the back-calculated concentration is greater than two times the acceptance criteria for that standard.

Lower Limit of Quantitation

The back-calculated concentrations of at least one of the duplicate lowest points in the calibration curve must be within ± 25% of the target concentration for PFOSA to qualify as the LLOQ and within ± 20% of the target concentration to qualify as the LLOQ for all other analytes. If this criterion is not met, the next level is subjected to the same test and the LLOQ raised accordingly.

Quality Control Samples

Each analytical run will include low, medium and high QC samples in duplicate. The measured concentrations of at least two-thirds of all analytical QCs must be within ± 20% of their target concentrations (± 25% for PFOSA), and no two QCs at the same concentration can be outside the limit. If study samples require dilution, a dilution QC will be analyzed in triplicate for each dilution level (except for PFOSA and PFOSAA which should not be diluted with control matrix). At least two dilution QC at each level must be within ± 20% of its target concentrations in order to accept diluted study samples at that level. The dilution QC acceptance is independent of the undiluted analytical QC acceptance.

5. REFERENCES

[5.1] D. Vollmer, "Quantitative Determination of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in Human Serum by LC/MS/MS," NWB study NWBS98-082, NWB report NWBR99-005, May 13, 1999.

6. DATA RETENTION

The raw data and final report for this study will be stored in the NWB Archives, 1121 East 3900 South, Salt Lake City, UT 84124 per regulations and contract agreement. 3M Environmental Technology and Services will be notified concerning final disposition of records at completion of contract obligations.

Table 1. Summary of Calibration Curve Parameters for PFOS

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
29-Jun-2000	13	-0.000009	0.035473	0.368276	0.9877	48.1	547
30-Jun-2000	15	-0.000001	0.018768	0.148749	0.9949	48.1	547
02-Jul-2000	16	-0.000005	0.028829	0.235310	0.9948	48.1	547
Mean		-0.000005	0.027690	0.250778	0.9925		
S.D.		0.000004	0.008411	0.110578	0.0041		
%CV		-80.0	30.4	44.1	0.4		
n		3	3	3	3		

A, B and C are coefficients used to define the quadratic curve.

Table 2. Summary of Calibration Curve Parameters for PFOSA

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
29-Jun-2000	13	-0.000030	0.093690	0.023501	0.9761	1.00	502
30-Jun-2000	15	-0.000012	0.051196	0.004685	0.9975	1.00	502
02-Jul-2000	16	-0.000020	0.088242	0.025293	0.9960	1.00	502
Mean		-0.000021	0.077709	0.017826	0.9899		
S.D.		0.000009	0.023122	0.011416	0.0119		
%CV		-42.9	29.8	64.0	1.2		
n		3	3	3	3		

A, B and C are coefficients used to define the quadratic curve.

Table 3. Summary of Calibration Curve Parameters for PFOSAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
29-Jun-2000	13	-0.000003	0.023347	-0.035788	0.9969	6.00	505
30-Jun-2000	15	0.000000	0.013890	-0.020748	0.9971	6.00	505
02-Jul-2000	16	0.000001	0.023426	-0.033118	0.9964	6.00	505
Mean		-0.000001	0.020221	-0.029885	0.9968		
S.D.		0.000002	0.005483	0.008024	0.0004		
%CV		-200.0	27.1	-26.8	0.0		
n		3	3	3	3		

A, B and C are coefficients used to define the quadratic curve.

Table 4. Summary of Calibration Curve Parameters for POAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
29-Jun-2000	13	0.000002	0.018721	0.026984	0.9847	5.76	505
30-Jun-2000	15	0.000000	0.012512	0.009407	0.9958	5.76	505
02-Jul-2000	16	-0.000002	0.014490	0.015595	0.9962	5.76	505
Mean		0.000000	0.015241	0.017329	0.9922		
S.D.		0.000002	0.003172	0.008916	0.0065		
%CV		-	20.8	51.5	0.7		
n		3	3	3	3		

A, B and C are coefficients used to define the quadratic curve.

Table 5. Summary of Calibration Curve Parameters for PFHS

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
29-Jun-2000	13	-0.000006	0.023682	0.021811	0.9947	3.15	502
30-Jun-2000	15	-0.000005	0.016787	0.011507	0.9946	3.15	502
02-Jul-2000	16	-0.000009	0.021344	0.020627	0.9968	3.15	502
Mean		-0.000007	0.020604	0.017982	0.9954		
S.D.		0.000002	0.003507	0.005638	0.0012		
%CV		-28.6	17.0	31.4	0.1		
n		3	3	3	3		

A, B and C are coefficients used to define the quadratic curve.

Table 6. Summary of Calibration Curve Parameters for M556

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
29-Jun-2000	13	0.000001	0.005000	0.004952	0.9881	4.30	502
30-Jun-2000	15	0.000000	0.002882	0.000748	0.9975	4.30	502
02-Jul-2000	16	0.000000	0.003891	0.000548	0.9949	4.30	502
Mean		0.000000	0.003924	0.002083	0.9935		
S.D.		0.000001	0.001059	0.002487	0.0049		
%CV		-	27.0	119.4	0.5		
n		3	3	3	3		

A, B and C are coefficients used to define the quadratic curve.

000122

Table 7. Summary of Calibration Curve Parameters for M570

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
29-Jun-2000	13	-0.000002	0.006621	-0.007115	0.9962	5.60	505
30-Jun-2000	15	-0.000001	0.003911	-0.005406	0.9972	5.60	505
02-Jul-2000	16	0.000000	0.006433	-0.009231	0.9967	5.60	505
Mean		-0.000001	0.005655	-0.007251	0.9967		
S.D.		0.000001	0.001513	0.001916	0.0005		
%CV		-100.0	26.8	-26.4	0.1		
n		3	3	3	3		

A, B and C are coefficients used to define the quadratic curve.

000123

Table 8. Back-Calculated Concentrations of Calibration Standards for PFOS

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	48.1	49.6	57.1	72.1	97.1	147	297	447	547
29-Jun-2000	13	44.9	52.1	56.6	71.7	81.1	153	302	449	515
		46.5	51.5	58.5	80.8	94.6	146	316	445	560
30-Jun-2000	15	46.5	49.8	58.6	72.7	92.2	147	321	438	541
		50.3	48.3	58.4	71.2	98.9	135	306	471	518
02-Jul-2000	16	51.6	50.5	60.3	74.9	94.3	151	299	415	571
		45.2	47.6	54.3	71.2	94.9	148	298	449	547
Mean		47.5	50.0	57.8	73.8	92.7	147	307	445	542
S.D.		2.78	1.77	2.07	3.72	6.07	6.28	9.47	18.2	22.3
%CV		5.9	3.5	3.6	5.0	6.5	4.3	3.1	4.1	4.1
%Bias		-1.2	0.8	1.2	2.4	-4.5	0.0	3.4	-0.4	-0.9
n		6	6	6	6	6	6	6	6	6

Table 9. Back-Calculated Concentrations of Calibration Standards for PFOSA

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	1.00	2.51	10.1	25.1	50.3	100	251	402	502
29-Jun-2000	13	1.00	2.44	10.6	25.7	30.8	104	280	420	483
		1.03	2.23	11.1	33.3	45.7	95.0	256	379	507
30-Jun-2000	15	1.03	2.46	9.90	25.7	48.6	98.5	255	401	518
		0.957	2.68	9.71	25.7	50.4	94.8	277	418	447
02-Jul-2000	16	0.952	2.48	11.5	26.2	47.6	99.0	244	391	506
		1.04	2.57	9.79	22.7	48.7	107	237	431	501
Mean		1.00	2.48	10.4	26.6	45.3	99.7	258	407	494
S.D.		0.0388	0.150	0.753	3.54	7.27	4.90	17.3	19.7	25.6
%CV		3.9	6.0	7.2	13.3	16.0	4.9	6.7	4.8	5.2
%Bias		0.0	-1.2	3.0	6.0	-9.9	-0.3	2.8	1.2	-1.6
n		6	6	6	6	6	6	6	6	6

000124

Table 10. Back-Calculated Concentrations of Calibration Standards for PFOSAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	6.00	7.50	15.0	30.0	55.0	105	255	405	505
29-Jun-2000	13	5.90	7.74	14.4	31.3	*31.8	110	262	412	476
		5.92	7.85	14.1	30.8	49.4	106	266	400	512
30-Jun-2000	15	6.00	7.82	14.9	29.0	53.3	105	271	403	494
		5.58	7.81	15.7	30.1	53.3	99.9	271	426	475
02-Jul-2000	16	5.92	7.22	15.8	28.2	49.8	102	255	392	501
		6.15	7.65	14.8	30.2	55.8	119	260	413	504
Mean		5.91	7.68	15.0	29.9	52.3	107	264	408	494
S.D.		0.187	0.237	0.683	1.15	2.69	6.83	6.37	11.9	15.2
%CV		3.2	3.1	4.6	3.8	5.1	6.4	2.4	2.9	3.1
%Bias		-1.5	2.4	0.0	-0.3	-4.9	1.9	3.5	0.7	-2.2
n		6	6	6	6	5	6	6	6	6

* Sample deactivated as an outlier (> 2 times the acceptance criteria). Result not included in summary statistics.

Table 11. Back-Calculated Concentrations of Calibration Standards for POAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	5.76	7.26	14.8	29.8	54.8	105	255	405	505
29-Jun-2000	13	5.15	8.55	14.6	30.9	*36.6	104	246	377	450
		6.41	6.41	13.4	25.3	56.6	116	287	423	541
30-Jun-2000	15	6.26	7.24	14.0	29.5	54.4	108	280	390	517
		5.67	6.80	15.0	29.7	55.2	97.6	276	406	466
02-Jul-2000	16	5.36	7.09	15.2	28.2	50.7	103	250	385	532
		*11.8	7.74	15.9	32.2	54.9	106	255	394	521
Mean		5.77	7.31	14.7	29.3	54.4	106	266	396	505
S.D.		0.550	0.754	0.891	2.38	2.20	6.12	17.4	16.4	37.3
%CV		9.5	10.3	6.1	8.1	4.0	5.8	6.5	4.1	7.4
%Bias		0.2	0.7	-0.7	-1.7	-0.7	1.0	4.3	-2.2	0.0
n		5	6	6	6	5	6	6	6	6

* Sample deactivated as an outlier (> 2 times the acceptance criteria). Result not included in summary statistics.

000125

Table 12. Back-Calculated Concentrations of Calibration Standards for PFHS

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	3.15	4.65	12.2	27.2	52.2	102	252	402	502
		2.5-3.9	4-5.3	10.4-14	23-31.3	44-60	87-117	214-290	342-462	427-577
29-Jun-2000	13	3.28	5.17	11.8	28.0	55.3	107	252	377	448
		2.85	4.59	11.7	27.3	48.8	97.6	250	414	574
30-Jun-2000	15	2.96	5.08	13.2	26.2	47.6	101	264	395	515
		3.00	4.70	13.8	26.6	50.4	94.6	255	415	484
02-Jul-2000	16	3.21	4.70	12.9	28.4	50.0	101	244	382	545
		3.12	4.26	13.5	26.5	50.3	99.7	249	399	509
Mean		3.07	4.75	12.8	27.2	50.4	100	252	397	513
S.D.		0.162	0.333	0.880	0.887	2.63	4.14	6.77	15.8	44.3
%CV		5.3	7.0	6.9	3.3	5.2	4.1	2.7	4.0	8.6
%Bias		-2.5	2.2	4.9	0.0	-3.4	-2.0	0.0	-1.2	2.2
n		6	6	6	6	6	6	6	6	6

Table 13. Back-Calculated Concentrations of Calibration Standards for M556

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	4.30	11.8	26.8	51.8	102	252	402	502
		3.1-5	10-13.6	23-30.8	44-60	↑	↑	342-462	427-577
29-Jun-2000	13	5.11	11.3	30.6	*33.3	111	266	396	468
		3.78	9.88	24.0	49.7	101	272	395	514
30-Jun-2000	15	4.25	11.6	27.4	49.8	107	273	403	499
		4.39	11.7	26.1	51.1	95.1	266	412	466
02-Jul-2000	16	3.78	11.5	25.6	47.5	98.2	250	379	513
		4.74	13.0	26.9	53.6	110	254	408	506
Mean		4.34	11.5	26.8	50.3	104	264	399	494
S.D.		0.527	0.996	2.22	2.23	6.56	9.46	11.8	21.9
%CV		12.1	8.7	8.3	4.4	6.3	3.6	3.0	4.4
%Bias		0.9	-2.5	0.0	-2.9	2.0	4.8	-0.7	-1.6
n		6	6	6	5	6	6	6	6

* Sample deactivated as an outlier (> 2 times the acceptance criteria). Result not included in summary statistics.

000126

Table 14. Back-Calculated Concentrations of Calibration Standards for M570

Quadratic weighted $1/x^2$. All concentrations are expressed as ng/mL.

Run Date	Run Number	5.60	7.10	14.6	29.6	54.6	105	255	405	505
		4.8-6.4 ✓	6-8.2 ✓	12.4-16.8 ✓	25.2-34 ✓	46.4-62.8 63%	87-121 ✓	217-293 ✓	344-466 ✓	429-581 ✓
29-Jun-2000	13	5.50	7.08	13.7	31.6	*34.5	107	264	418	466
		5.47	7.61	14.6	30.5	50.5	98.9	269	388	530
30-Jun-2000	15	5.26	7.11	14.8	29.1	55.2	101	266	408	498
		6.00	6.94	14.5	30.7	54.7	97.0	272	422	474
02-Jul-2000	16	5.67	6.55	15.7	27.5	49.8	108	255	394	509
		5.82	6.98	14.9	29.5	56.3	113	251	401	513
Mean		5.62	7.05	14.7	29.8	53.3	104	263	405	498
S.D.		0.266	0.342	0.648	1.44	2.94	6.16	8.18	13.4	24.4
%CV		4.7	4.9	4.4	4.8	5.5	5.9	3.1	3.3	4.9
%Bias		0.4	-0.7	0.7	0.7	-2.4	-1.0	3.1	0.0	-1.4
n		6	6	6	6	5	6	6	6	6

* Sample deactivated as an outlier (> 2 times the acceptance criteria). Result not included in summary statistics.

000127

Table 15. Intra-Assay Precision and Accuracy for PFOS Quality Control Samples

LOQ = 48.1

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 51.1 ng/mL	Medium QC 197 ng/mL	High QC 446 ng/mL
29-Jun-2000	13	45.9	191	392
		44.8	177	403
		47.6	183	429
		48.8	175	383
		48.6	179	377
Mean		47.1	181	397
S.D.		1.74	6.32	20.5
%CV		3.7	3.5	5.2
%Theoretical		92.2	91.9	89.0
n		5	5	5
Run Date	**Run Number**	**Low QC 51.1 ng/mL**	**Medium QC 197 ng/mL**	**High QC 446 ng/mL**
30-Jun-2000	15	48.8	173	382
		46.7	174	395
		47.8	175	402
		45.8	166	382
		47.1	176	406
Mean		47.2	173	393
S.D.		1.13	3.96	11.1
%CV		2.4	2.3	2.8
%Theoretical		92.4	87.8	88.1
n		5	5	5

000128

Table 15. Intra-Assay Precision and Accuracy for PFOS Quality Control Samples (continued)

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 51.1 ng/mL	Medium QC 197 ng/mL	High QC 446 ng/mL
02-Jul-2000	16	45.2	170	380
		48.7	160	393
		46.4	166	385
		47.7	169	387
		44.0	172	361
Mean		46.4	167	381
S.D.		1.88	4.67	12.2
%CV		4.1	2.8	3.2
%Theoretical		90.8	84.8	85.4
n		5	5	5

000129

Table 16. Intra-Assay Precision and Accuracy for PFOSA Quality Control Samples

LLOQ = 4.00

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 4.00 ng/mL	Medium QC 150 ng/mL	High QC 400 ng/mL
29-Jun-2000	13	3.43	133	325
		3.21	122	345
		3.39	128	357
		3.58	129	322
		3.67	139	342
Mean		3.46	130	338
S.D.		0.178	6.30	14.6
%CV	✓	5.1	4.8	4.3
%Theoretical		86.5	86.7	84.5
n		5	5	5
Run Date	**Run Number**	**Low QC 4.00 ng/mL**	**Medium QC 150 ng/mL**	**High QC 400 ng/mL**
30-Jun-2000	15	* 2.88	* 100	* 278
		* 2.76	* 105	* 291
		* 2.89	* 103	318
		* 2.78	* 97.8	* 297
		* 2.77	* 110	314
Mean		2.82	103	300
S.D.		0.0635	4.71	16.5
%CV	✓	2.3	4.6	5.5
%Theoretical		70.5	68.7	75.0
n		5	5	5

* > ±25% theoretical

Fortification problem see p. 14

Repeated Table 29

000130

Table 16. Intra-Assay Precision and Accuracy for PFOSA Quality Control Samples (continued)

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 4.00 ng/mL	Medium QC 150 ng/mL	High QC 400 ng/mL
02-Jul-2000	16	* 2.58	* 105	* 289
		* 2.96	* 102	311
		3.50	* 103	315
		3.94	* 102	304
		* 2.51	* 112	305
Mean		3.10	105	305
S.D.		0.613	4.21	9.91
%CV		19.8	4.0	3.2
%Theoretical		77.5	70.0	76.3
n		5	5	5

* > ±25% theoretical

000131

Table 17. Intra-Assay Precision and Accuracy for PFOSAA Quality Control Samples

All concentrations are expressed as ng/mL. LLOQ = 6.00

Run Date	Run Number	Low QC 9.00 ng/mL	Medium QC 155 ng/mL	High QC 405 ng/mL
29-Jun-2000	13	7.98	129	* 272
		7.43	* 120	* 303
		8.05	* 123	* 317
		8.81	* 121	* 260
		8.46	127	* 286
Mean		8.15	124	288
S.D.		0.522	3.87	23.0
%CV		6.4	3.1	8.0
%Theoretical		90.6	80.0	71.1
n		5	5	5
Run Date	**Run Number**	**Low QC 9.00 ng/mL**	**Medium QC 155 ng/mL**	**High QC 405 ng/mL**
30-Jun-2000	15	7.65	* 112	* 285
		7.76	* 117	* 300
		7.73	* 116	* 315
		7.29	* 104	* 290
		7.52	* 117	* 313
Mean		7.59	113	301
S.D.		0.192	5.54	13.4
%CV		2.5	4.9	4.5
%Theoretical		84.3	72.9	74.3
n		5	5	5

* > ±20% theoretical

Fortification problems (p. 14) Repeated table 30

Table 17. Intra-Assay Precision and Accuracy for PFOSAA Quality Control Samples (continued)

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 9.00 ng/mL	Medium QC 155 ng/mL	High QC 405 ng/mL
02-Jul-2000	16	7.37	* 117	* 287
		8.11	* 107	* 309
		8.47	* 111	* 301
		9.67	* 114	* 312
		7.63	* 116	* 291
Mean		8.25	113	300
S.D.		0.900	4.06	10.9
%CV		10.9	3.6	3.6
%Theoretical		91.7	72.9	74.1
n		5	5	5

* > ±20% theoretical

000133

Table 18. Intra-Assay Precision and Accuracy for POAA Quality Control Samples

All concentrations are expressed as ng/mL. LLOQ: 5.70

Run Date	Run Number	Low QC 8.74 ng/mL	Medium QC 154 ng/mL	High QC 403 ng/mL
29-Jun-2000	13	7.74	157	404
		8.83	156	401
		9.28	154	434
		10.1	153	422
		9.01	163	377
Mean		8.99	157	408
S.D.		0.852	3.91	21.8
%CV	✓	9.5	2.5	5.3
%Theoretical		102.9	101.9	101.2
n		5	5	5
Run Date	**Run Number**	**Low QC 8.74 ng/mL**	**Medium QC 154 ng/mL**	**High QC 403 ng/mL**
30-Jun-2000	15	8.79	144	374
		8.00	153	389
		8.26	147	382
		7.59	136	376
		7.88	139	398
Mean		8.10	144	384
S.D.		0.453	6.69	9.86
%CV	✓	5.6	4.6	2.6
%Theoretical		92.7	93.5	95.3
n		5	5	5

000134

Table 18. Intra-Assay Precision and Accuracy for POAA Quality Control Samples (continued)

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 8.74 ng/mL	Medium QC 154 ng/mL	High QC 403 ng/mL
02-Jul-2000	16	7.15	150	401
		8.15	136	393
		8.42	140	398
		8.72	148	405
		7.12	141	376
Mean		7.91	143	395
S.D.		0.737	5.83	11.3
%CV		9.3	4.1	2.9
%Theoretical		90.5	92.9	98.0
n		5	5	5

000135

Table 19. Intra-Assay Precision and Accuracy for PFHS Quality Control Samples

All concentrations are expressed as ng/mL. LLOD= 2.15

Run Date	Run Number	Low QC 6.15 ng/mL	Medium QC 152 ng/mL	High QC 402 ng/mL
29-Jun-2000	13	7.03	175	454
		6.44	166	431
		6.77	171	468
		6.89	158	434
		7.05	165	424
Mean		6.84	167	442
S.D.		0.249	6.44	18.2
%CV	✓	3.6	3.9	4.1
%Theoretical		111.2	109.9	110.0
n		5	5	5
Run Date	**Run Number**	**Low QC 6.15 ng/mL**	**Medium QC 152 ng/mL**	**High QC 402 ng/mL**
30-Jun-2000	15	6.53	163	429
		6.22	157	455
		6.06	158	463
		6.00	149	429
		6.31	164	447
Mean		6.22	158	445
S.D.		0.211	5.97	15.3
%CV	✓	3.4	3.8	3.4
%Theoretical		101.1	103.9	110.7
n		5	5	5

000136

Table 19. Intra-Assay Precision and Accuracy for PFHS Quality Control Samples (continued)

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 6.15 ng/mL	Medium QC 152 ng/mL	High QC 402 ng/mL
02-Jul-2000	16	6.43	154	408
		6.52	150	441
		6.23	152	422
		6.86	153	430
		5.93	151	409
Mean		6.39	152	422
S.D.		0.345	1.58	14.1
%CV		5.4	1.0	3.3
%Theoretical		103.9	100.0	105.0
n		5	5	5

000137

Table 20. Intra-Assay Precision and Accuracy for M556 Quality Control Samples

LOD = 4.30

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 5.80 ng/mL	Medium QC 152 ng/mL	High QC 402 ng/mL
29-Jun-2000	13	** 8.98	165	427
		6.34	159	429
		** 7.98	161	439
		6.55	163	413
		5.86	170	366
Mean		6.25	164	415
S.D.		0.354	4.22	28.8
%CV	✓	5.7	2.6	6.9
%Theoretical		107.8	107.9	103.2
n		3	5	5
Run Date	**Run Number**	**Low QC 5.80 ng/mL**	**Medium QC 152 ng/mL**	**High QC 402 ng/mL**
30-Jun-2000	15	5.74	143	380
		5.89	147	395
		5.46	152	408
		5.33	139	390
		5.61	153	407
Mean		5.61	147	396
S.D.		0.221	5.93	11.8
%CV	✓	3.9	4.0	3.0
%Theoretical		96.7	96.7	98.5
n		5	5	5

** Sample deactivated due to injector carryover (not included in summary statistics)

000138

Table 20. Intra-Assay Precision and Accuracy for M556 Quality Control Samples (continued)

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 5.80 ng/mL	Medium QC 152 ng/mL	High QC 402 ng/mL
02-Jul-2000	16	6.23	149	391
		6.17	137	418
		6.17	149	390
		6.33	152	417
		5.61	150	378
Mean		6.10	147	399
S.D.		0.283	5.94	17.8
%CV		4.6	4.0	4.5
%Theoretical		105.2	96.7	99.3
n		5	5	5

000139

Table 21. Intra-Assay Precision and Accuracy for M570 Quality Control Samples

All concentrations are expressed as ng/mL. LLOQ = 5.60

Run Date	Run Number	Low QC 8.60 ng/mL	Medium QC 155 ng/mL	High QC 405 ng/mL
29-Jun-2000	13	8.23	168	393
		8.07	155	403
		8.34	164	424
		8.87	153	378
		8.04	170	377
Mean		8.31	162	395
S.D.		0.336	7.65	19.5
%CV	✓	4.0	4.7	4.9
%Theoretical		96.6	104.5	97.5
n		5	5	5
Run Date	**Run Number**	**Low QC 8.60 ng/mL**	**Medium QC 155 ng/mL**	**High QC 405 ng/mL**
30-Jun-2000	15	8.17	151	396
		9.09	149	396
		8.72	148	412
		7.71	138	388
		7.88	150	413
Mean		8.31	147	401
S.D.		0.579	5.26	11.0
%CV	✓	7.0	3.6	2.7
%Theoretical		96.6	94.8	99.0
n		5	5	5

Table 21. Intra-Assay Precision and Accuracy for M570 Quality Control Samples (continued)

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 8.60 ng/mL	Medium QC 155 ng/mL	High QC 405 ng/mL
02-Jul-2000	16	8.67	148	382
		8.49	137	391
		9.73	148	384
		9.69	145	412
		7.95	151	368
Mean		8.91	146	387
S.D.		0.780	5.36	16.1
%CV		8.8	3.7	4.2
%Theoretical		103.6	94.2	95.6
n		5	5	5

Table 22. Inter-Assay Precision for PFOS Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 51.1 ng/mL	Medium QC 197 ng/mL	High QC 446 ng/mL
29-Jun-2000	13	45.9	191	392
		44.8	177	403
		47.6	183	429
		48.8	175	383
		48.6	179	377
30-Jun-2000	15	48.8	173	382
		46.7	174	395
		47.8	175	402
		45.8	166	382
		47.1	176	406
02-Jul-2000	16	45.2	170	380
		48.7	160	393
		46.4	166	385
		47.7	169	387
		44.0	172	361
Mean		46.9	174	390
S.D.		1.55	7.46	15.7
%CV		3.3	4.3	4.0
%Theoretical		91.8	88.3	87.4
%Bias		-8.2	-11.7	-12.6
n		15	15	15

Table 23. Inter-Assay Precision for PFOSA Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 4.00 ng/mL	Medium QC 150 ng/mL	High QC 400 ng/mL
29-Jun-2000	13	3.43	133	325
		3.21	122	345
		3.39	128	357
		3.58	129	322
		3.67	139	342
30-Jun-2000	15	*2.88	*100	*278
		*2.76	*105	*291
		*2.89	*103	318
		*2.78	*97.8	*297
		*2.77	*110	314
02-Jul-2000	16	*2.58	*105	*289
		*2.96	*102	311
		3.50	*103	315
		3.94	*102	304
		*2.51	*112	305
Mean		3.12	113	314
S.D.		0.437	13.7	21.9
%CV		14.0	12.1	7.0
%Theoretical		78.0	75.3	78.5
%Bias		-22.0	-24.7	-21.5
n		15	15	15

* > ±25% theoretical

Table 24. Inter-Assay Precision for PFOSAA Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 9.00 ng/mL	Medium QC 155 ng/mL	High QC 405 ng/mL
29-Jun-2000	13	7.98	129	*272
		7.43	*120	*303
		8.05	*123	*317
		8.81	*121	*260
		8.46	127	*286
30-Jun-2000	15	7.65	*112	*285
		7.76	*117	*300
		7.73	*116	*315
		7.29	*104	*290
		7.52	*117	*313
02-Jul-2000	16	7.37	*117	*287
		8.11	*107	*309
		8.47	*111	*301
		9.67	*114	*312
		7.63	*116	*291
Mean		8.00	117	296
S.D.		0.640	6.79	16.6
%CV		8.0	5.8	5.6
%Theoretical		88.9	75.5	73.1
%Bias		-11.1	-24.5	-26.9
n		15	15	15

* > ±20% theoretical

Table 25. Inter-Assay Precision for POAA Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 8.74 ng/mL	Medium QC 154 ng/mL	High QC 403 ng/mL
29-Jun-2000	13	7.74	157	404
		8.83	156	401
		9.28	154	434
		10.1	153	422
		9.01	163	377
30-Jun-2000	15	8.79	144	374
		8.00	153	389
		8.26	147	382
		7.59	136	376
		7.88	139	398
02-Jul-2000	16	7.15	150	401
		8.15	136	393
		8.42	140	398
		8.72	148	405
		7.12	141	376
Mean		8.34	148	395
S.D.		0.812	8.27	17.4
%CV		9.7	5.6	4.4
%Theoretical		95.4	96.1	98.0
%Bias		-4.6	-3.9	-2.0
n		15	15	15

000145

Table 26. Inter-Assay Precision for PFHS Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 6.15 ng/mL	Medium QC 152 ng/mL	High QC 402 ng/mL
29-Jun-2000	13	7.03	175	454
		6.44	166	431
		6.77	171	468
		6.89	158	434
		7.05	165	424
30-Jun-2000	15	6.53	163	429
		6.22	157	455
		6.06	158	463
		6.00	149	429
		6.31	164	447
02-Jul-2000	16	6.43	154	408
		6.52	150	441
		6.23	152	422
		6.86	153	430
		5.93	151	409
Mean		6.48	159	436
S.D.		0.368	7.96	18.1
%CV		5.7	5.0	4.2
%Theoretical		105.4	104.6	108.5
%Bias		5.4	4.6	8.5
n		15	15	15

Table 27. Inter-Assay Precision for M556 Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 5.80 ng/mL	Medium QC 152 ng/mL	High QC 402 ng/mL
29-Jun-2000	13	**8.98	165	427
		6.34	159	429
		**7.98	161	439
		6.55	163	413
		5.86	170	366
30-Jun-2000	15	5.74	143	380
		5.89	147	395
		5.46	152	408
		5.33	139	390
		5.61	153	407
02-Jul-2000	16	6.23	149	391
		6.17	137	418
		6.17	149	390
		6.33	152	417
		5.61	150	378
Mean		5.95	153	403
S.D.		0.381	9.49	21.0
%CV		6.4	6.2	5.2
%Theoretical		102.6	100.7	100.2
%Bias		2.6	0.7	0.2
n		13	15	15

** Sample deactivated due to injector carryover (not included in summary statistics)

000147

Table 28. Inter-Assay Precision for M570 Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 8.60 ng/mL	Medium QC 155 ng/mL	High QC 405 ng/mL
29-Jun-2000	13	8.23	168	393
		8.07	155	403
		8.34	164	424
		8.87	153	378
		8.04	170	377
30-Jun-2000	15	8.17	151	396
		9.09	149	396
		8.72	148	412
		7.71	138	388
		7.88	150	413
02-Jul-2000	16	8.67	148	382
		8.49	137	391
		9.73	148	384
		9.69	145	412
		7.95	151	368
Mean		8.51	152	394
S.D.		0.621	9.51	15.8
%CV		7.3	6.3	4.0
%Theoretical		99.0	98.1	97.3
%Bias		-1.0	-1.9	-2.7
n		15	15	15

Table 29. Intra-Assay Precision and Accuracy for PFOSA Specific Quality Controls

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC (4.00 ng/mL)	Medium QC (150 ng/mL)	High QC (400 ng/mL)
16-Jul-2000	17	3.55	145	322
		3.58	135	324
		3.84	171	326
		4.01	149	330
		3.46	144	341
Mean		3.69	149	329
S.D.		0.229	13.4	7.54
%CV		6.2	9.0	2.3
%Theoretical		92.3	99.3	82.3
n		5	5	5

Note: The following calibration curve and standard statistics are included for analytical run review. (not included in overall validation summary statistics)
Quadratic weighted $1/x^2$.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
16-Jul-2000	17	0.000002	0.020014	0.006490	0.9985	1.00	502
n		1	1	1	1		

A, B and C are coefficients used to define the quadratic curve.

Run Date	Run Number	1.00	2.51	10.1	25.1	50.3	100	251	402	502
16-Jul-2000	17	0.998	2.48	10.1	24.5	52.6	96.2	244	390	531
		0.980	2.67	10.4	24.2	50.4	104	239	399	505
Mean		0.989	2.58	10.3	24.4	51.5	100	242	395	518
%Bias		-1.1	2.8	2.0	-2.8	2.4	0.0	-3.6	-1.7	3.2
n		2	2	2	2	2	2	2	2	2

Table 30. Intra-Assay Precision and Accuracy for PFOSAA Specific Quality Controls

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC (9.00 ng/mL)	Medium QC (155 ng/mL)	High QC (405 ng/mL)
16-Jul-2000	17	7.30	161	368
		*4.93	176	382
		*5.65	*190	395
		8.36	159	365
		**21.4	151	346
Mean		6.56	167	371
S.D.		1.56	15.5	18.5
%CV		23.8	9.3	5.0
%Theoretical		72.9	107.7	91.6
n		4	5	5

* > ±20% theoretical

** Deactivated as an outlier (not included in summary statistics)

Note: The following calibration curve and standard statistics are included for analytical run review. (not included in overall validation summary statistics)

Quadratic weighted $1/x^2$.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
16-Jul-2000	17	0.000001	0.005669	0.008710	0.9764	7.50	505
n		1	1	1	1		

A, B and C are coefficients used to define the quadratic curve.

Run Date	Run Number	6.00	7.50	15.0	30.0	55.0	105	255	405	505
16-Jul-2000	17	**8.89	8.60	*17.8	32.5	*65.3	107	287	413	559
		**3.30	6.49	*12.1	*25.4	47.6	104	231	372	465
Mean		-	7.55	15.0	29.0	56.5	106	259	393	512
%Bias		-	0.7	0.0	-3.3	2.7	1.0	1.6	-3.0	1.4
n		-	2	2	2	2	2	2	2	2

* > ±15% theoretical

** Sample deactivated as an outlier (> 2 times the acceptance criteria). Result not included in summary statistics.

Table 31. PFOS Dilution Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Dilution QC 4460 ng/mL
29-Jun-2000	13	4310
		4170
		4420
		3900
		4110
30-Jun-2000	15	4490
		3990
		4180
		4050
		4400
02-Jul-2000	16	4160
		4160
		4020
		3790
		3970
Mean		4140
S.D.		199
%CV		4.8
%Theoretical		92.8
%Bias		-7.2
n		15

Table 32. PFOSA Dilution Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Dilution QC 4000 ng/mL
29-Jun-2000	13	3720
		3570
		3720
		3380
		3690
30-Jun-2000	15	3020
		*2890
		3100
		*2750
		*2860
02-Jul-2000	16	3270
		3130
		3130
		3210
		3240
Mean		3250
S.D.		315
%CV		9.7
%Theoretical		81.3
%Bias		-18.8
n		15

* > ±25% theoretical

Table 33. PFOSAA Dilution Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Dilution QC 4050 ng/mL
29-Jun-2000	13	3490
		3280
		3270
		*3170
		3390
30-Jun-2000	15	3420
		*3030
		3240
		*3060
		*3120
02-Jul-2000	16	*3220
		*3110
		*3210
		*2960
		*3000
Mean		3200
S.D.		157
%CV		4.9
%Theoretical		79.0
%Bias		-21.0
n		15

* > ±20% theoretical

000153

Table 34. POAA Dilution Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Dilution QC 4030 ng/mL
29-Jun-2000	13	4220
		3820
		4250
		4040
		4000
30-Jun-2000	15	4550
		3850
		4010
		4000
		4020
02-Jul-2000	16	4170
		4090
		4230
		3910
		4040
Mean		4080
S.D.		184
%CV		4.5
%Theoretical		101.2
%Bias		1.2
n		15

Table 35. PFHS Dilution Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Dilution QC 4020 ng/mL
29-Jun-2000	13	4830
		4430
		4530
		4150
		4190
30-Jun-2000	15	*4980
		4500
		4550
		4490
		4790
02-Jul-2000	16	4700
		*5030
		4550
		4260
		4760
Mean		4580
S.D.		267
%CV		5.8
%Theoretical		113.9
%Bias		13.9
n		15

* > ±20% theoretical

000155

Table 36. M556 Dilution Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Dilution QC 4020 ng/mL
29-Jun-2000	13	4330
		4050
		4260
		3940
		4000
30-Jun-2000	15	4490
		3980
		4130
		4000
		4170
02-Jul-2000	16	4270
		3990
		4240
		3890
		3980
Mean		4110
S.D.		172
%CV		4.2
%Theoretical		102.2
%Bias		2.2
n		15

Table 37. M570 Dilution Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Dilution QC 4050 ng/mL
29-Jun-2000	13	4710
		4590
		4550
		4100
		4490
30-Jun-2000	15	4610
		3980
		4080
		4080
		4280
02-Jul-2000	16	4270
		3950
		4120
		3890
		3960
Mean		4240
S.D.		278
%CV		6.6
%Theoretical		104.7
%Bias		4.7
n		15

000157

Table 38. PFOSA Specific Dilution Quality Controls

All concentrations are expressed as ng/mL.

Run Date	Run Number	Dilution QC 4000 ng/mL
16-Jul-2000	17	*2630
		*2570
		3110
		3250
		4110
Mean		3130
S.D.		620
%CV		19.8
%Theoretical		78.3
%Bias		-21.8
n		5

* > ±25% theoretical

000158

Table 39. PFOSAA Specific Dilution Quality Controls

All concentrations are expressed as ng/mL.

Run Date	Run Number	Dilution QC 4050 ng/mL
16-Jul-2000	17	*2560
		*2650
		*2900
		3640
		4070
Mean		3160
S.D.		661
%CV		20.9
%Theoretical		78.0
%Bias		-22.0
n		5

* > ±20% theoretical

000159

Table 40. PFOS Extraction Efficiency

Run 7

Low Concentration = 2.50 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.00936	107
		0.0101	99.2
		0.0103	97.5
	Mean	0.00992	101
2. Analyte prior and IS after extraction		0.00532	
		0.00568	
		0.00517	
	Mean	0.00539	
3. IS prior and Analyte after extraction			6.94
			7.51
			8.42
	Mean		7.62

Mean extraction efficiency for the analyte = 54.3%
Mean extraction efficiency for the internal standard = 7.54%

Medium Concentration = 100 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.326	3.07
		0.297	3.37
		0.312	3.21
	Mean	0.312	3.22
2. Analyte prior and IS after extraction		0.117	
		0.121	
		0.113	
	Mean	0.117	
3. IS prior and Analyte after extraction			0.263
			0.269
			0.280
	Mean		0.271

Mean extraction efficiency for the analyte = 37.5%
Mean extraction efficiency for the internal standard = 8.42%

High Concentration = 400 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		1.17	0.855
		1.25	0.797
		1.21	0.828
	Mean	1.21	0.827
2. Analyte prior and IS after extraction		0.435	
		0.398	
		0.418	
	Mean	0.417	
3. IS prior and Analyte after extraction			0.0692
			0.0652
			0.0668
	Mean		0.0671

Mean extraction efficiency for the analyte = 34.5%
Mean extraction efficiency for the internal standard = 8.11%

Overall mean extraction efficiency for analyte = 42.1%
Overall mean extraction efficiency for internal standard = 8.02%

000160

Table 41. PFOSA Extraction Efficiency

Run 7

Low Concentration = 2.50 ng/mL for Analyte

Time of Spiking	Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction	0.00603	166
	0.00611	164
	0.00587	170
Mean	0.00600	167
2. Analyte prior and IS after extraction	0.00375	
	0.00434	
	0.00383	
Mean	0.00397	
3. IS prior and Analyte after extraction		11.4
		11.2
		13.4
Mean		12.0

Mean extraction efficiency for the analyte = 66.2%
Mean extraction efficiency for the internal standard = 7.19%

Medium Concentration = 100 ng/mL for Analyte

Time of Spiking	Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction	0.240	4.16
	0.203	4.92
	0.214	4.68
Mean	0.219	4.59
2. Analyte prior and IS after extraction	0.148	
	0.128	
	0.152	
Mean	0.143	
3. IS prior and Analyte after extraction		0.331
		0.357
		0.358
Mean		0.349

Mean extraction efficiency for the analyte = 65.3%
Mean extraction efficiency for the internal standard = 7.60%

High Concentration = 400 ng/mL for Analyte

Time of Spiking	Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction	0.950	1.05
	0.901	1.11
	0.973	1.03
Mean	0.941	1.06
2. Analyte prior and IS after extraction	0.595	
	0.574	
	0.648	
Mean	0.606	
3. IS prior and Analyte after extraction		0.0880
		0.0778
		0.0817
Mean		0.0825

Mean extraction efficiency for the analyte = 64.4%
Mean extraction efficiency for the internal standard = 7.78%

Overall mean extraction efficiency for analyte = 65.3%
Overall mean extraction efficiency for internal standard = 7.52%

000161

Table 42. PFOSAA Extraction Efficiency

Run 7

Low Concentration = 2.50 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.00526	190
		0.00589	170
		0.00559	179
	Mean	0.00558	180
2. Analyte prior and IS after extraction		0.00401	
		0.00427	
		0.00399	
	Mean	0.00409	
3. IS prior and Analyte after extraction			12.8
			14.1
			15.6
	Mean		14.2

Mean extraction efficiency for the analyte = 73.3%
Mean extraction efficiency for the internal standard = 7.89%

Medium Concentration = 100 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.373	2.68
		0.325	3.08
		0.342	2.93
	Mean	0.347	2.90
2. Analyte prior and IS after extraction		0.254	
		0.270	
		0.249	
	Mean	0.258	
3. IS prior and Analyte after extraction			0.234
			0.238
			0.248
	Mean		0.240

Mean extraction efficiency for the analyte = 74.4%
Mean extraction efficiency for the internal standard = 8.28%

High Concentration = 400 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.864	1.16
		0.900	1.11
		0.889	1.12
	Mean	0.884	1.13
2. Analyte prior and IS after extraction		0.642	
		0.624	
		0.658	
	Mean	0.641	
3. IS prior and Analyte after extraction			0.0944
			0.0878
			0.0885
	Mean		0.0902

Mean extraction efficiency for the analyte = 72.5%
Mean extraction efficiency for the internal standard = 7.98%

Overall mean extraction efficiency for analyte = 73.4%
Overall mean extraction efficiency for internal standard = 8.05%

000162

Table 43. POAA Extraction Efficiency

Run 7

Low Concentration = 2.50 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.00921	109
		0.00982	102
		0.0105	95.0
	Mean	0.00984	102
2. Analyte prior and IS after extraction		0.00209	
		0.00213	
		0.00224	
	Mean	0.00215	
3. IS prior and Analyte after extraction			7.02
			7.35
			7.97
	Mean		7.45

Mean extraction efficiency for the analyte = 21.8%
Mean extraction efficiency for the internal standard = 7.30%

Medium Concentration = 100 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.358	2.79
		0.318	3.15
		0.336	2.98
	Mean	0.337	2.97
2. Analyte prior and IS after extraction		0.0526	
		0.0570	
		0.0516	
	Mean	0.0537	
3. IS prior and Analyte after extraction			0.227
			0.237
			0.248
	Mean		0.237

Mean extraction efficiency for the analyte = 15.9%
Mean extraction efficiency for the internal standard = 7.98%

High Concentration = 400 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		1.19	0.841
		1.31	0.762
		1.29	0.775
	Mean	1.26	0.793
2. Analyte prior and IS after extraction		0.205	
		0.201	
		0.216	
	Mean	0.207	
3. IS prior and Analyte after extraction			0.0638
			0.0595
			0.0627
	Mean		0.0620

Mean extraction efficiency for the analyte = 16.4%
Mean extraction efficiency for the internal standard = 7.82%

Overall mean extraction efficiency for analyte = 18.0%
Overall mean extraction efficiency for internal standard = 7.70%

Table 44. PFHS Extraction Efficiency

Run 7

Low Concentration = 2.50 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.0158	63.2
		0.0168	59.6
		0.0169	59.3
	Mean	0.0165	60.7
2. Analyte prior and IS after extraction		0.00371	
		0.00384	
		0.00399	
	Mean	0.00385	
3. IS prior and Analyte after extraction			4.43
			4.47
			5.27
	Mean		4.72

Mean extraction efficiency for the analyte = 23.3%
Mean extraction efficiency for the internal standard = 7.78%

Medium Concentration = 100 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.632	1.58
		0.567	1.76
		0.598	1.67
	Mean	0.599	1.67
2. Analyte prior and IS after extraction		0.105	
		0.113	
		0.108	
	Mean	0.109	
3. IS prior and Analyte after extraction			0.134
			0.146
			0.146
	Mean		0.142

Mean extraction efficiency for the analyte = 18.2%
Mean extraction efficiency for the internal standard = 8.50%

High Concentration = 400 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		2.07	0.484
		2.25	0.444
		2.15	0.465
	Mean	2.16	0.464
2. Analyte prior and IS after extraction		0.387	
		0.363	
		0.385	
	Mean	0.378	
3. IS prior and Analyte after extraction			0.0394
			0.0360
			0.0365
	Mean		0.0373

Mean extraction efficiency for the analyte = 17.5%
Mean extraction efficiency for the internal standard = 8.04%

Overall mean extraction efficiency for analyte = 19.7%
Overall mean extraction efficiency for internal standard = 8.11%

000164

Table 45. M556 Extraction Efficiency

Run 7

Low Concentration = 2.50 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.00635	157
		0.00659	152
		0.00684	146
	Mean	0.00659	152
2. Analyte prior and IS after extraction		0.00293	
		0.00299	
		0.00279	
	Mean	0.00290	
3. IS prior and Analyte after extraction			10.7
			10.7
			12.4
	Mean		11.3

Mean extraction efficiency for the analyte = 44.0%
Mean extraction efficiency for the internal standard = 7.43%

Medium Concentration = 100 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.270	3.70
		0.240	4.17
		0.252	3.97
	Mean	0.254	3.95
2. Analyte prior and IS after extraction		0.106	
		0.124	
		0.111	
	Mean	0.114	
3. IS prior and Analyte after extraction			0.309
			0.320
			0.325
	Mean		0.318

Mean extraction efficiency for the analyte = 44.9%
Mean extraction efficiency for the internal standard = 8.05%

High Concentration = 400 ng/mL for Analyte

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		1.02	0.984
		1.07	0.934
		1.05	0.953
	Mean	1.05	0.957
2. Analyte prior and IS after extraction		0.444	
		0.441	
		0.463	
	Mean	0.449	
3. IS prior and Analyte after extraction			0.0800
			0.0744
			0.0765
	Mean		0.0770

Mean extraction efficiency for the analyte = 42.8%
Mean extraction efficiency for the internal standard = 8.05%

Overall mean extraction efficiency for analyte = 43.9%
Overall mean extraction efficiency for internal standard = 7.84%

Table 46. M570 Extraction Efficiency

Run 7

Low Concentration = 2.50 ng/mL for Analyte

Time of Spiking	Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction	0.00470	213
	0.00487	205
	0.00530	189
Mean	0.00496	202
2. Analyte prior and IS after extraction	0.00317	
	0.00355	
	0.00320	
Mean	0.00331	
3. IS prior and Analyte after extraction		14.4
		14.0
		15.8
Mean		14.7

Mean extraction efficiency for the analyte = 66.7%
Mean extraction efficiency for the internal standard = 7.28%

Medium Concentration = 100 ng/mL for Analyte

Time of Spiking	Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction	0.217	4.61
	0.191	5.24
	0.204	4.90
Mean	0.204	4.92
2. Analyte prior and IS after extraction	0.130	
	0.143	
	0.136	
Mean	0.136	
3. IS prior and Analyte after extraction		0.391
		0.396
		0.422
Mean		0.403

Mean extraction efficiency for the analyte = 66.7%
Mean extraction efficiency for the internal standard = 8.19%

High Concentration = 400 ng/mL for Analyte

Time of Spiking	Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction	0.772	1.29
	0.831	1.20
	0.796	1.26
Mean	0.800	1.25
2. Analyte prior and IS after extraction	0.533	
	0.521	
	0.563	
Mean	0.539	
3. IS prior and Analyte after extraction		0.102
		0.0970
		0.0984
Mean		0.0991

Mean extraction efficiency for the analyte = 67.4%
Mean extraction efficiency for the internal standard = 7.93%

Overall mean extraction efficiency for analyte = 66.9%
Overall mean extraction efficiency for internal standard = 7.80%

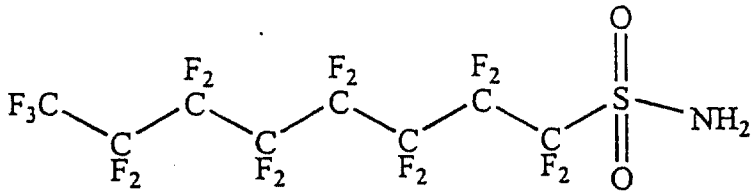
000166

7. ANALYTICAL METHOD

Principles of the Method

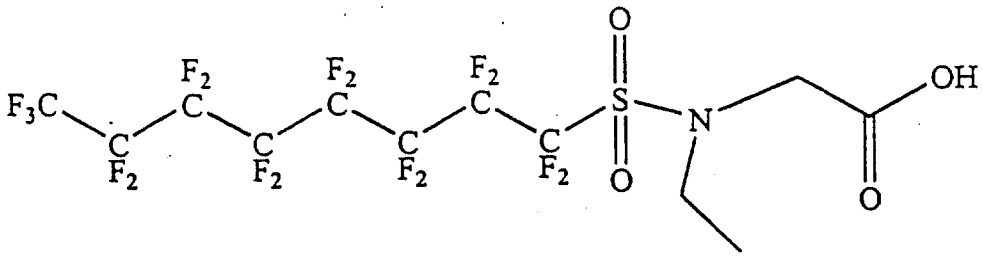
The analytical method consisted of a liquid:liquid extraction procedure followed by evaporation and reconstitution of the extract residue with 30:70 (v/v) 20 mM ammonium acetate in water: 20 mM ammonium acetate in methanol. The samples were analyzed by liquid chromatography/tandem mass spectrometry using a PE Sciex API 3000. The instrument was operated in the multiple reaction monitoring (MRM) mode under optimized conditions for PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 detection.

CHEMICAL STRUCTURES



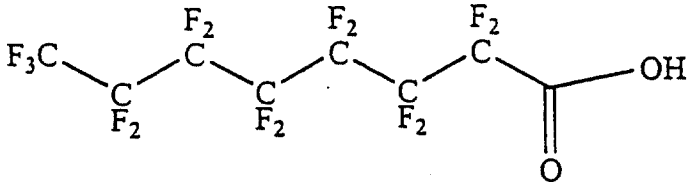
PFOSA

MW=499.0
[M-H]=498.0



PFOSAA

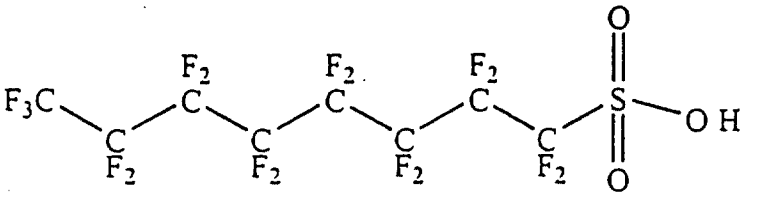
MW=585.0
[M-H]=584.0



POAA

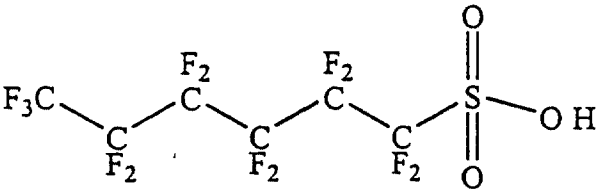
MW=414.0
[M-H]=413.0

000167



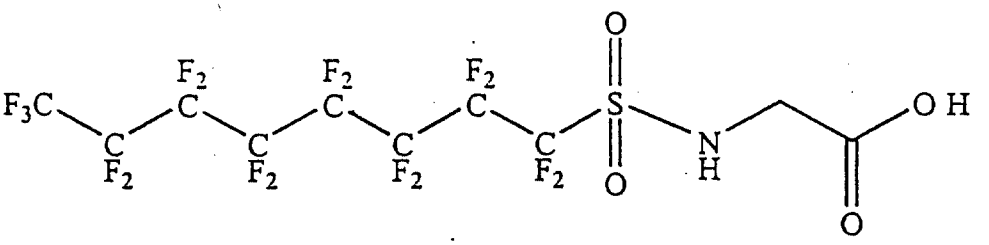
PFOS

MW=499.9
[M-H]=498.9



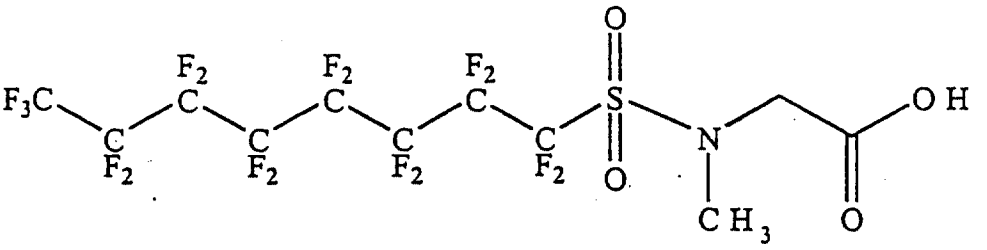
PFHS

MW=399.9
[M-H]=398.9



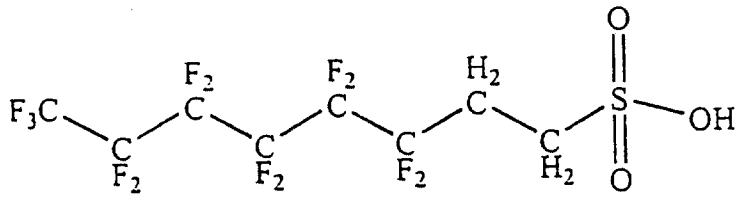
M556

MW=556.9
[M-H]=555.9



M570

MW=571.0
[M-H]=570.0



THPFOS
MW=428

7.1. Reference Materials and Matrices

Analyte	*Lot Number*	*Purity*	*Expiration Date*	*Source*	*Storage Conditions*
PFOS (FC-95)	193	100%	12/31/2010	3M	Room Temperature
PFOSA	214	100%	12/31/2010	3M	Room Temperature
PFOSAA (FC-129)	617	53.8%	12/31/2010	3M	Room Temperature
POAA (FC-143)	245	100%	12/31/2010	3M	Room Temperature**
PFHS*	S398-182	100%	12/31/2010	3M	-20°C
M556	NB113047-8D	99.89%	12/31/2010	3M	Room Temperature
M570	118506-26	99.75%	12/31/2010	3M	Room Temperature
THPFOS	59909	90%	12/31/2010	3M	Room Temperature

* Received as a 6,200 ppm solution in methanol. ** Kept dry.

The reference material purity for PFOS, PFOSA, PFHS and POAA was not available prior to the conduct of this study. Therefore, all concentrations included in the report for these analytes are based upon an assumed purity of 100%.

Prior to the completion of this final report, 3M contracted with Centre Analytical Laboratories, Inc. in State College, Pennsylvania to determine the absolute concentration of PFOS, POAA and PFHS in the stock standard solution prepared using seven reference materials (PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and

M570) at a target concentration of 5000 ppb. Absolute determination of the concentration of PFOSA in the stock standard solutions cannot be made until a full purity determination of these reference materials is completed.

Based on the results obtained, the concentrations included in this report should be corrected according to the following table:

Analyte	Correction Factor
PFOS	0.836
POAA	0.909
PFHS	0.855

Matrix	*Lot Numbers*
Human serum	BC30399-4
Human plasma (Northern Chinese)	C5186; C4929; C5517; C5200

7.2. Chemicals and Equipment

Chemicals
Ammonium acetate, 99.9%
Di (ethylene glycol) methyl ether [may be written as 2-(2-methoxyethoxy) ethanol]
Methanol, HPLC-grade
Methyl-*Tert*-Butyl Ether (MTBE), HPLC-grade
Water, HPLC-grade

Equipment/Supplies	
Name	**Source**
Autosampler: PE Series 200	Perkin Elmer, Norwalk, CT
Balance: Mettler Toledo MT5	Mettler-Toledo, Inc., Hightstown, NJ

Equipment/Supplies	
Name	Source
Centrifuge: Beckman GS-6R	Beckman Instruments, Fullerton, CA
Evaporator: Turbo Vap LV, Model 43750	Zymark Corp., Hopkinton, MA
Hematology/Chemistry Mixer, Model 346	Fisher Scientific, Pittsburgh, PA
HPLC Chromatographic column: Genesis Lightning, C_{18}, 4 µm, 2 x 50 mm	Jones Chromatography, Lakewood, CA
Liquid Chromatograph: Hewlett Packard 1100	Agilent (Hewlett Packard), Palo Alto, CA
Liquid Chromatograph: PE Sciex Series 200	Perkin Elmer, Norwalk, CT
Liquid Chromatograph: Shimadzu SCL-10A controller with CL-10AD pump and CTO-10A column oven	Shimadzu, Columbia, MD
Mass Spectrometer: Perkin Elmer Sciex API 3000	PE Sciex, Concord, Ontario
Multi-tube Vortexer	VWR Scientific Products, Bridgeport, NJ
Pipettes: Eppendorf Repeater Pipette	Brinkman Instruments, Inc., Westbury, NY
Pipettes: Finnpipette: Digital 1-5 mL	Fisher Scientific, Pittsburgh, PA
Pipettes: Rainin EDP Digital 100-1000µL ™	Rainin Instrument Co., Woburn, MA
Pipettes: Rainin M Volumes: adjustable 20-50, 10-100, 100-1000 µL	Rainin Instrument Co., Woburn, MA
Pipettes: Rainin RL Volumes: adjustable 5-20, 20-100, 40-200, 200-1000 µL	Rainin Instrument Co., Woburn, MA
Sonicator, Branson	Branson, Danbury, CT
Sonicator, Fisher	Fisher Scientific, Fair Lawn, NJ
Vortex: Fisher Genie 2	Fisher Scientific, Fair Lawn, NJ
Water Pro Plus	Labconco, Co., Kansas City, MO

7.3. Reagents, Calibration Standard and Quality Control (QC) Solutions

The calibrator, quality control and stock solution preparations listed below serve as a general guideline for attaining the targeted concentrations. Due to the fact that PFHS was received at NWB as a solution, both the calibration standard and quality control stocks are from the same solution and are not from separate weighings of reference material as per NWB SOP LABOP003. However, separate aliquots were used for calibration standard and QC stock solution preparation.

Reagents

All reagent solutions are stored at room temperature unless otherwise noted.

- *50 mM ammonium acetate in water (unadjusted: pH ~6.9)*
  Fill a 1-L volumetric flask approximately half full with water. Weigh out 3.85 g of ammonium acetate and transfer to the flask. Use a stir plate to mix and fill to volume with water.
- *20 mM ammonium acetate in water (unadjusted: pH ~6.9)*
  Fill a 1-L volumetric flask approximately half full with water. Weigh out 1.54 g of ammonium acetate and transfer to the flask. Use a stir plate to mix and fill to volume with water.
- *20 mM ammonium acetate in methanol*
  Prepare as described above, substituting methanol for water.
- *30:70 20 mM ammonium acetate in water (unadjusted: pH ~6.9): 20 mM ammonium acetate in methanol (v/v)*
- *50:50 water:methanol (v/v)*

Calibration Standard Solutions

All calibration standard solutions are transferred to polypropylene containers and stored in a –20 °C freezer. The PFHS reference material was provided as a 6200 ppm solution.

- *PFOS Stock Standard (0.05904 mg/mL)*
  Weigh 1.476 mg of PFOS and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *PFOSA Stock Standard (0.07652 mg/mL)*
  Weigh 1.913 mg of PFOSA and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *PFOSAA Stock Standard (0.02264 mg/mL)*
  Weigh 1.052 mg of PFOSAA and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *POAA Stock Standard (0.03868 mg/mL)*
  Weigh 0.967 mg of POAA and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *M556 Stock Standard (0.04579 mg/mL)*
  Weigh 1.146 mg of M556 and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *M570 Stock Standard (0.04796 mg/mL)*
  Weigh 1.202 mg of M570 and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *Spiking Standard 9 (5000 ng/mL for PFOS, PFOSAA, POAA, PFHS, M556 and M570; 5020 ng/mL for PFOSA)*
  Add 2.117 mL of PFOS Stock Standard (0.05904 mg/mL), 1.639 mL of PFOSA

Stock Standard (0.07652 mg/mL), 5.521 mL of PFOSAA Stock Standard (0.02264 mg/mL), 3.232 mL of POAA Stock Standard (0.03868 mg/mL), 20.16 µL of PFHS (6200 ppm), 2.73 mL of M556 Stock Standard (0.04579 mg/mL) and 2.606 mL of M570 Stock Standard (0.04796 mg/mL) to a 25-mL volumetric flask. Fill to volume with 50:50 water:methanol and mix by inverting 10 times and sonicating for approximately 10 minutes.

- *Spiking Standard 8 (4000 ng/mL for PFOS, PFOSAA, POAA, PFHS, M556, M570; 4020 ng/mL for PFOSA)*
  Add 8.00 mL of Spiking Standard 9 to a 16 x 100 mm polypropylene tube and add 2.00 mL of 50:50 water:methanol. Mix by inverting 20 times.

- *Spiking Standard 7 (2500 ng/mL for PFOS, PFOSAA, POAA, PFHS, M556,M570; 2510 ng/mL for PFOSA)*
  Add 5.00 mL of Spiking Standard 9 to a 16 x 100 mm polypropylene tube and add 5.00 mL of 50:50 water:methanol. Mix by inverting 20 times.

- *Spiking Standard 6 (1000 ng/mL for PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570)*
  Add 2.00 mL of Spiking Standard 9 to a 16 x 100 mm polypropylene tube and add 8.00 mL of 50:50 water:methanol. Mix by inverting 20 times.

- *Spiking Standard 5 (500 ng/mL for PFOS, PFOSAA, POAA, PFHS, M556, M570; 503ng/mL for PFOSA)*
  Add 1.25 mL of Spiking Standard 8 to a 16 x 100 mm polypropylene tube and add 8.75 mL of 50:50 water:methanol. Mix by inverting 20 times.

- *Spiking Standard 4 (250 ng/mL for PFOS, PFOSAA, POAA, PFHS, M556, M570; 251 ng/mL for PFOSA)*
  Add 0.500 mL of Spiking Standard 9 to a 16 x 100 mm polypropylene tube and add 9.50 mL of 50:50 water:methanol. Mix by inverting 20 times.

- *Spiking Standard 3 (100 ng/mL for PFOS, PFOSAA, POAA, PFHS, M556, M570; 101 ng/mL for PFOSA)*

  Add 0.250 mL of Spiking Standard 8 to a 16 x 100 mm polypropylene tube and add 9.75 mL of 50:50 water:methanol. Mix by inverting 20 times.

- *Spiking Standard 2 (25.0 ng/mL for PFOS, PFOSAA, POAA, PFHS, M556, M570; 25.1 ng/mL for PFOSA)*

  Add 0.100 mL of Spiking Standard 7 to a 16 x 100 mm polypropylene tube and add 9.90 mL of 50:50 water:methanol. Mix by inverting 20 times.

- *Spiking Standard 1 (10.0 ng/mL for PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570)*

  Add 0.100 mL of Spiking Standard 6 to a 16 x 100 mm polypropylene tube and add 9.90 mL of 50:50 water:methanol. Mix by inverting 20 times.

Internal Standard Solutions

All internal standard solutions are transferred to polypropylene containers and stored in a –20 °C freezer.

- *THPFOS Stock Solution (0.04842 mg/mL)*

  Weigh 0.2690 mg of THPFOS and add to a 5-mL volumetric flask. Fill to volume with 50:50 water:methanol and mix by inverting 5 times and sonicating approximately 10 minutes.

- *Working Internal Standard (400 ng/mL THPFOS)*

  Add 413 µL of the THPFOS Stock Solution (0.04842 mg/mL) to a 50-mL volumetric flask and fill to volume with 50:50 water:methanol. Mix by inverting 5 to 10 times and sonicating approximately 5 to 10 minutes.

Quality Control (QC) Solutions

All quality control solutions are transferred to polypropylene containers and stored in a –20 °C freezer.

- *PFOS QC Stock (0.07732 mg/mL)*

  Weigh 1.933 mg of PFOS and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *PFOSA QC Stock (0.04876 mg/mL)*

  Weigh 1.219 mg of PFOSA and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *PFOSAA QC Stock (0.02920 mg/mL)*

  Weigh 1.357 mg of PFOSAA and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *POAA QC Stock (0.04228 mg/mL)*

  Weigh 1.057 mg of POAA and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *PFHS QC Stock (1000 ppm)*

  Aliquot 807µL of PFHS stock solution (6200 ppm) to a 5-mL volumetric flask. Fill to volume with methanol and mix by inversion.

- *M556 QC Stock (0.05230 mg/mL)*

  Weigh 1.309 mg of M556 and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *M570 QC Stock (0.05474 mg/mL)*

  Weigh 1.372 mg of M570 and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *PFOS Concentrated QC Solution (997,000 ng/mL)*
  Evaporate 1.69 mL of PFOS *(0.07732 mg/mL)* at 20 °C and below 5 P.S.I. nitrogen. Reconstitute with 131 µL of methanol.

- *PFOSA Concentrated QC Solution (1,000,000 ng/mL)*
  Evaporate 2.05 mL of PFOSA *(0.04876 mg/mL)* at 20 °C and below 5 P.S.I. nitrogen. Reconstitute with 100 µL of methanol.

- *PFOSAA Concentrated QC Solution (1,000,000 ng/mL)*
  Evaporate 4.42 mL of PFOSAA *(0.02920 mg/mL)* at 20 °C and below 5 P.S.I. nitrogen. Reconstitute with 129 µL of methanol.

- *POAA Concentrated QC Solution (996,000 ng/mL)*
  Evaporate 2.59 mL of POAA *(0.04228 mg/mL)* at 20 °C and below 5 P.S.I. nitrogen. Reconstitute with 110 µL of methanol.

- *PFHS Concentrated QC Solution*
  No PFHS concentrated solution was prepared.

- *M556 Concentrated QC Solution (1,000,000 ng/mL)*
  Evaporate 2.18 mL of M556 *(0.05230 mg/mL)* at 20 °C and below 5 P.S.I. nitrogen. Reconstitute with 114 µL of methanol.

- *M570 Concentrated QC Solution (1,000,000 ng/mL)*
  Evaporate 2.09 mL of M570 *(0.05474 mg/mL)* at 20 °C and below 5 P.S.I. nitrogen. Reconstitute with 114.4 µL of methanol.

7.4. Preparation of Validation Quality Control Samples

All quality control target concentrations will be corrected for the persistent levels of the analytes in human serum.

Dilution Quality Control (3990 ng/mL PFOS, 4000 ng/mL PFOSA, 4000 ng/mL PFOSAA, 3980 ng/mL POAA, 4000 ng/mL PFHS, 4000 ng/mL M556 and 4000 ng/mL M570)

Transfer 100 µL of each Concentrated QC Solution (997,000 ng/mL PFOS, 1,000,000 ng/mL PFOSA, 1,000,000 ng/mL PFOSAA, 996,000 ng/mL POAA, 1,000,000 ng/mL M556, and 1,000,000 ng/mL M570) and 100 µL of the 1000 ppm PFHS QC Stock into a 25-mL volumetric flask. Fill to volume with human serum and mix by inversion. Sonicate for approximately 10 minutes and equilibrate for approximately 10 minutes.

High Quality Control (399 ng/mL PFOS, 400 ng/mL PFOSA, 400 ng/mL PFOSAA, 398 ng/mL POAA, , 400 ng/mL PFHS, 400 ng/mL M556 and 400 ng/mL M570)

Transfer 5.00 mL of the Dilution Quality Control into a 50-mL volumetric flask. Fill to volume with human serum and mix by inversion. Sonicate for approximately 10 minutes and equilibrate for approximately 10 minutes.

Medium Quality Control (150 ng/mL PFOS, 150 ng/mL PFOSA, 150 ng/mL PFOSAA, 149 ng/mL POAA, 150 ng/mL PFHS, 150 ng/mL M556 and 150 ng/mL M570)

Transfer 1.875 mL of the Dilution Quality Control into a 50-mL volumetric flask. Fill to volume with human serum and mix by inversion. Sonicate for approximately 10 minutes and equilibrate for approximately 10 minutes.

Low Quality Control (3.99 ng/mL PFOS, 4.00 ng/mL PFOSA, 4.00 ng/ mL PFOSAA, 3.98 ng/mL POAA, 4.00 ng/mL PFHS, 4.00 ng/mL M556 and 4.00 ng/mL M570)

Transfer 0.500 mL of the High Quality Control into a 50-mL volumetric flask. Fill to volume with human serum and mix by inversion. Sonicate for approximately 10 minutes and equilibrate for approximately 10 minutes.

Storage of QC Samples

After preparation, place aliquots of the low, medium, high and dilution QC pools into cryogenic vials, and store in a -20°C freezer.

7.5. Preparation of PFOSA and PFOSAA Specific Validation Quality Control Samples

Quality Control (QC) Solutions

All quality control solutions are transferred to polypropylene containers and stored in a –20 °C freezer.

- *PFOSA QC Stock (0.2166 mg/mL)*
  Weigh 5.414 mg of PFOSA and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

- *PFOSAA QC Stock (0.2322 mg/mL)*
  Weigh 10.790 mg of PFOSAA and add to a 25-mL volumetric flask. Fill to volume with methanol, mix by inverting 5 times and sonicating for approximately 10 minutes.

Preparation of Quality Control Samples

All quality control target concentrations will be corrected for the persistent levels of the analytes in human serum.

Dilution Quality Control (4000 ng/mL PFOSA and PFOSAA)

Transfer 0.462 mL of the PFOSA QC Stock Solution (*0.2166 mg/mL)* and 0.431 mL of the PFOSAA QC Stock Solution (*0.2322 mg/mL*) into a 25-mL volumetric flask. Fill to volume with human serum and mix by inverting approximately 10 times. Sonicate for 5 minutes and equilibrate for approximately 5 minutes.

High Quality Control (400 ng/mL PFOSA and PFOSAA)

Transfer 2.50 mL of the Dilution Quality Control into a 25-mL volumetric flask. Fill to volume with human serum and mix by inverting approximately 10 times. Sonicate for 5 minutes and equilibrate for approximately 5 minutes.

Medium Quality Control (150 ng/mL PFOSA and PFOSAA)

Transfer 0.938 mL of the Dilution Quality Control into a 25-mL volumetric flask. Fill to volume with human serum and mix by inverting approximately 10 times. Sonicate for 5 minutes and equilibrate for approximately 5 minutes.

Low Quality Control (4.00 ng/mL PFOSA and PFOSAA)

Transfer 0.250 mL of the High Quality Control into a 25-mL volumetric flask. Fill to volume with human serum and mix by inverting approximately 10 times. Sonicate for 5 minutes and equilibrate for approximately 5 minutes.

Storage of QC Samples

After preparation, place aliquots of the low, medium, high and dilution QC pools into cryogenic vials, and store in a -20°C freezer.

7.6. Recommended Calibration Standard and Quality Control Preparation for Analysis

While concentrated solutions of the analytes can be used to prepare calibration standards and quality control samples, the evaporation of stock solutions to prepare concentrated stock solutions is not recommended for PFOSA and PFOSAA.

7.7. Preparation of Calibration Standards

The calibration curve is prepared on the day of each run by adding 100 µL blank human serum and 400 µL of 50 mM ammonium acetate in water (unadjusted: pH ~6.9) to 13 x 100 mm polypropylene tubes. After a brief vortex mixing, spike 10.0 µL of the appropriate spiking solution into the tubes as shown in the table below.

Standard Number	Concentration of Spiking Solution (ng/mL) PFOS, PFOSAA, POAA, PFHS, M556, M570 / PFOSA	Volume of Spiking Solution (µL)	Volume of Blank Human Serum (µL)	Final Concentration* (ng/mL)
9	5000 / 5020	10.0	100	500 / 502
8	4000 / 4020	10.0	100	400 / 402
7	2500 / 2510	10.0	100	250 / 251

Standard Number	Concentration of Spiking Solution (ng/mL) PFOS, PFOSAA, POAA, PFHS, M556, M570 / PFOSA	Volume of Spiking Solution (µL)	Volume of Blank Human Serum (µL)	Final Concentration* (ng/mL)
6	1000	10.0	100	100
5	500 / 503	10.0	100	50.0 / 50.3
4	250 / 251	10.0	100	25.0 / 25.1
3	100 / 101	10.0	100	10.0 / 10.1
2	25.0 / 25.1	10.0	100	2.50 / 2.51
1	10.0	10.0	100	1.00

* The target calibration curve range is 1.00 ng/mL to 500 ng/mL. Each analyte has a different final curve range based upon the persistent levels of the analyte in the human serum used.

7.8. Sample Preparation

Calibration Curve Samples (prepare in duplicate) as indicated above

Quality Control Samples (prepare in duplicate)

Aliquot 100 µL of each of the Low, Medium and High controls into separate 13 x 100 mm polypropylene tubes. If needed, prepare a dilution control (in triplicate) according to the following formula: for DF=X, aliquot (0.500 mL/X) of dilution control into [0.500 mL – (0.500 mL/X)] of blank human serum. Aliquot 100 µL of the prepared dilution control into separate 13 x 100 mm polypropylene tubes.

Blank Control Samples (prepare in duplicate)

Aliquot 100 µL of blank human serum into separate 13 x 100 mm polypropylene tubes and label as QC0.

Aliquot 100 µL of blank human serum into separate 13 x 100 mm polypropylene tubes and label as BLANK.

Study Samples

Transfer 100-µL aliquots of each study sample into appropriately labeled 13 x 100 mm polypropylene tubes. If necessary, dilute study samples in the same manner as the Dilution QC.

Extraction Procedure

1. Add 400 µL of 50 mM ammonium acetate in water (unadjusted: pH ~6.9) to each sample except calibrators and vortex mix briefly.
2. Add 50.0 µL of Working Internal Standard to each sample (except Blanks) and vortex mix for approximately 30 seconds.
3. Centrifuge samples at 3000 rpm for approximately 2 minutes.
4. Add 3 mL of methyl-*tert*-butyl ether to each tube, cap and vortex mix for approximately 30 seconds.
5. Rotate samples for approximately 10 minutes.
6. Centrifuge samples at 3000 rpm for approximately 10 minutes.
7. Transfer the top MTBE layer into clean 13 x 100 mm polypropylene tubes using a transfer pipette.
8. Evaporate the organic layer to dryness in a TurboVap set at 25°C under gentle nitrogen stream (setting ≤5 psi) for approximately 40 minutes.
9. Reconstitute in 100 µL of 30:70 20 mM ammonium acetate in water (unadjusted: pH ~6.9): 20 mM ammonium acetate in methanol and vortex mix for approximately 15 seconds.
10. Transfer samples to autosampler vials and centrifuge at 3000 rpm for approximately 2 minutes

7.9. LC/MS/MS Conditions

LC Conditions

HPLC Column	Genesis Lightning C_{18}, 4 µm, 2x50 mm
Mobile Phases	A: 20 mM ammonium acetate in water (unadjusted: pH ~6.9) B: 20 mM ammonium acetate in methanol
LC Conditions	Gradient

Time (min.)	% B
0	50
1	50
7	97.5
9	97.5
9.1	50
11	end

Flow Rate	300 µL/minute
Post Column Addition	50 µL/minute of 2-(2-methoylethoxy) ethanol
Column Temperature	40°C
Injection Volume	2 – 20 µL

MS Conditions

Post Column Split	None
Source	TurboIonSpray™ (flow rate 8 L/min.)
Source Temp.	400 °C
Ionization Mode	Negative Ion
Analysis type	Multiple reaction monitoring (MRM)

	Transitions monitored (±0.3)	Dwell Time (ms)	Collision Energy (eV)
PFOS	499 → 80	70	80
PFOSA	498 → 78	70	65

	Transitions monitored (±0.3)	Dwell Time (ms)	Collision Energy (eV)
PFOSAA	584 → 419	70	29
POAA	413 → 169	70	25
PFHS	399 → 80	70	70
M556	556 → 498	400	40
M570	570 → 419	70	42
THPFOS	427 → 407	70	42

The prepared standards and QCs were injected into the instrument LC/MS/MS system in a systematic order.

7.10. Quantitation

PFOS, PFOSA, PFOSAA, POAA, PFHS, M556, M570 and THPFOS chromatographic peaks are integrated using PE Sciex MacQuan software (version 1.6) with a smooth factor of 1. Several of the analytes possess iosmers. The chromatographic conditions of this method can achieve baseline separation of these isomers. Because the goal of this method is to determine the total amount for each analyte, all isomer peaks are integrated together and the peak areas are treated as a single peak by MacQuan. Quantitation is based upon quadratic regression analysis of calibration curves (weighted $1/x^2$) using the area ratio vs. concentration by the Watson® DMLIMS software (version 6.1.1.04)

Figure 1. Representative Calibration Curve for PFOS

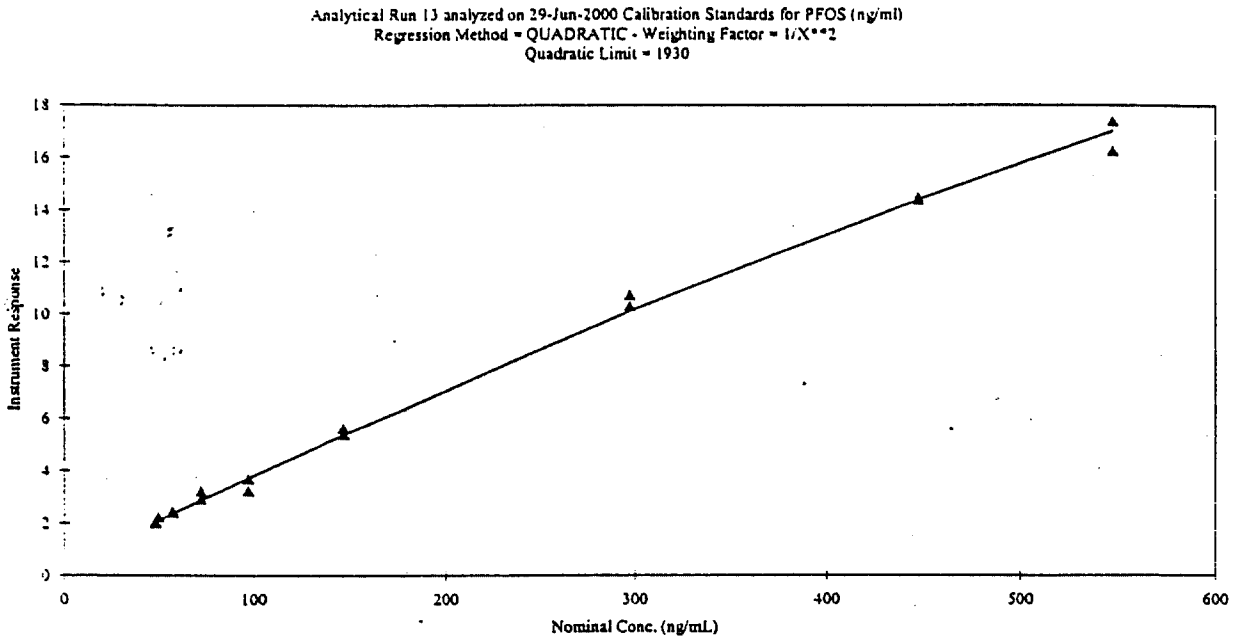
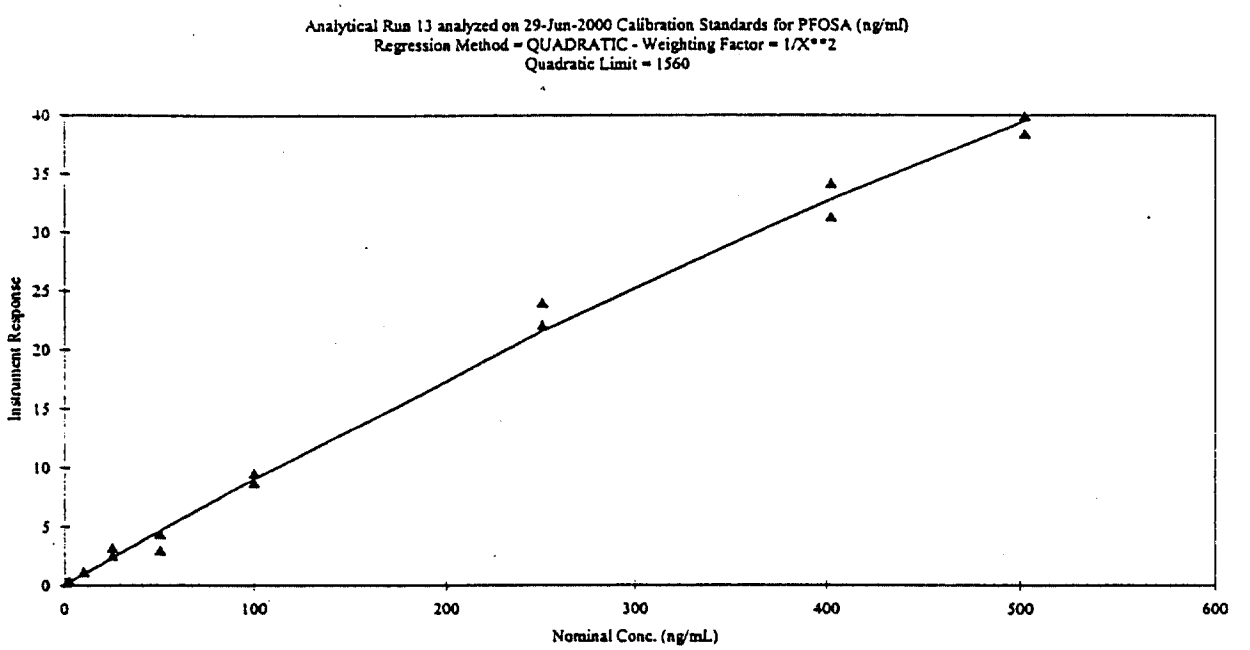


Figure 2. Representative Calibration Curve for PFOSA



000185

Figure 3. Representative Calibration Curve for PFOSAA

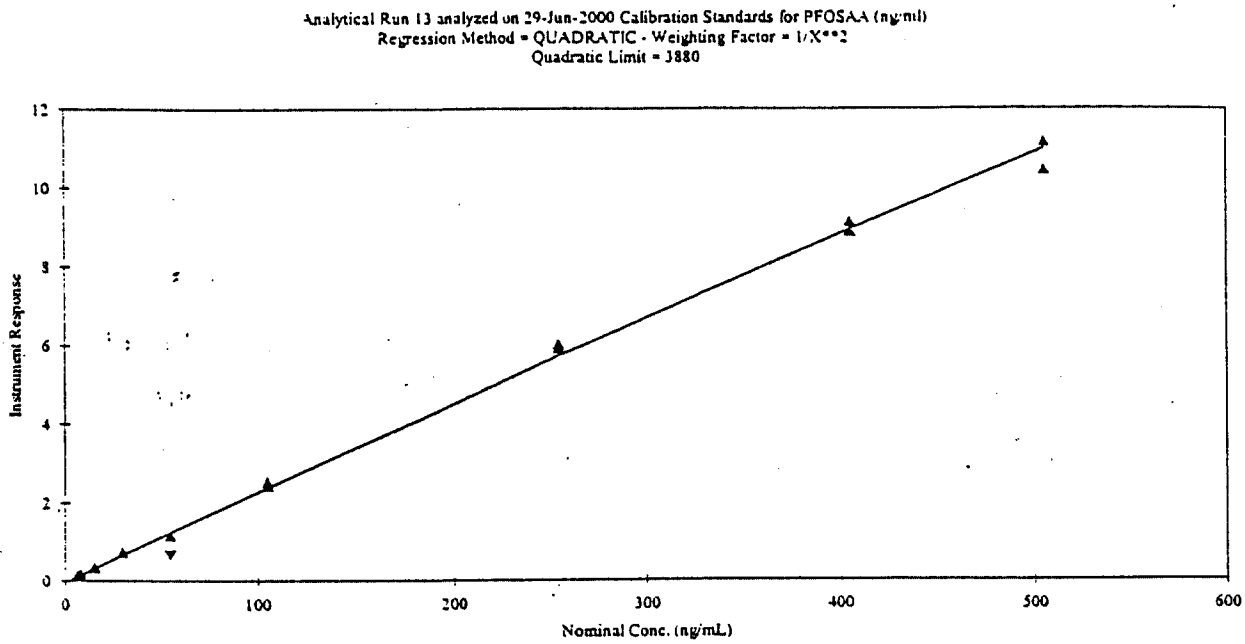
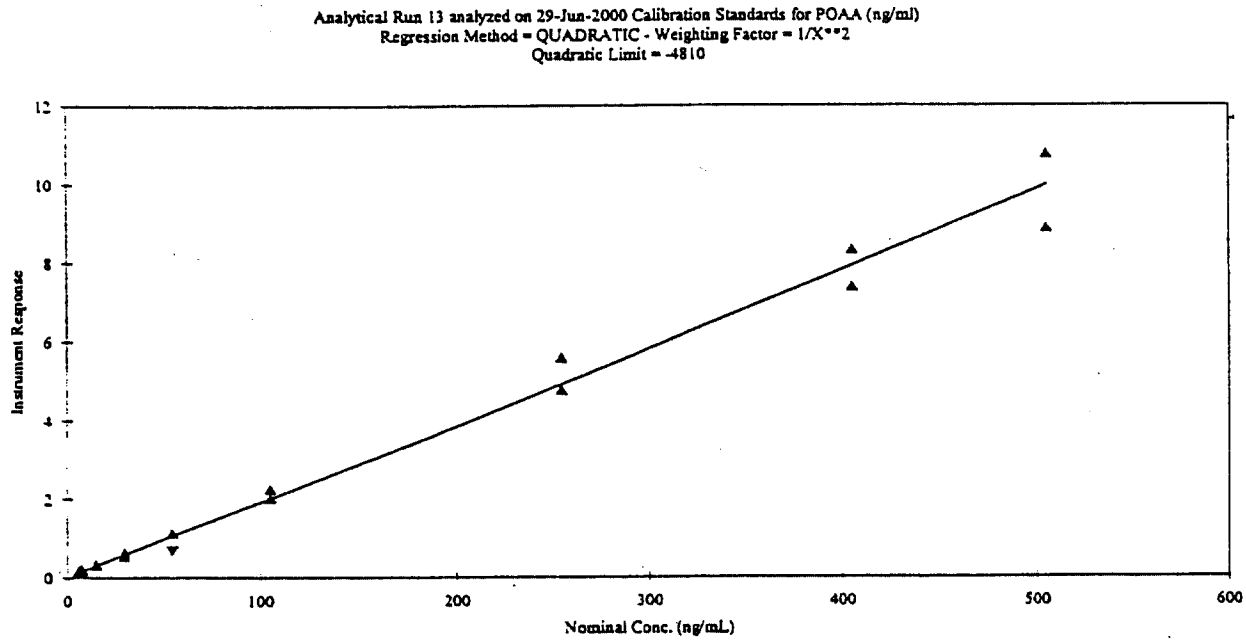


Figure 4. Representative Calibration Curve for POAA



000186

Figure 5. Representative Calibration Curve for PFHS

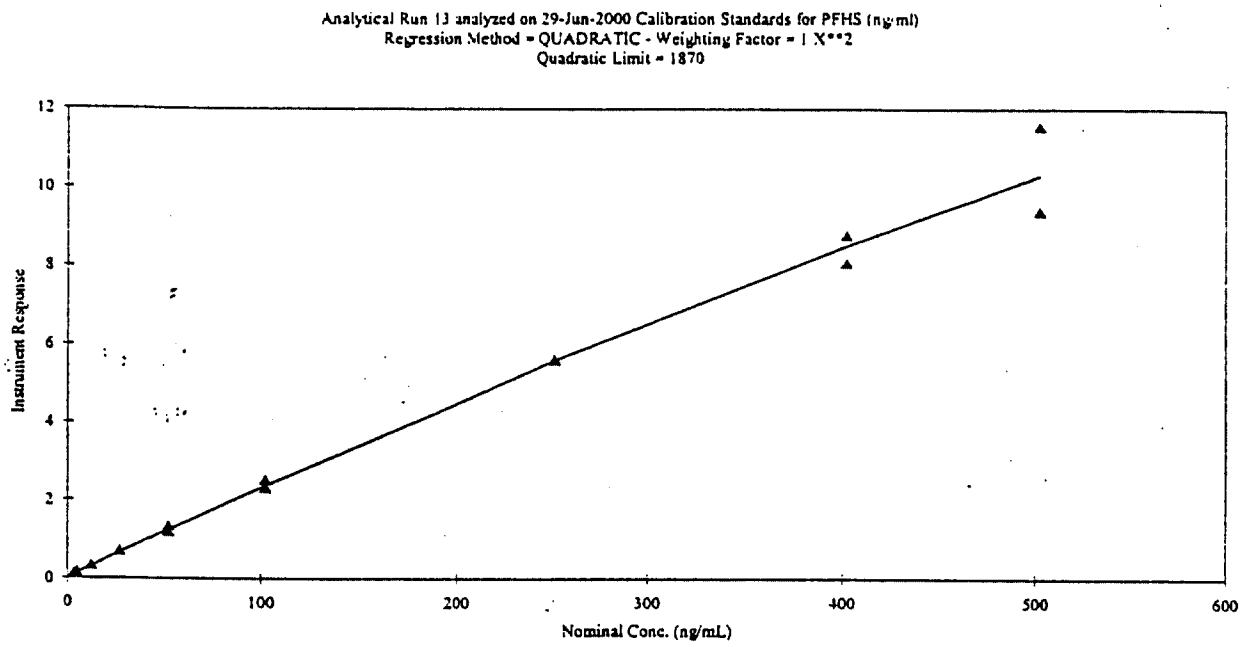
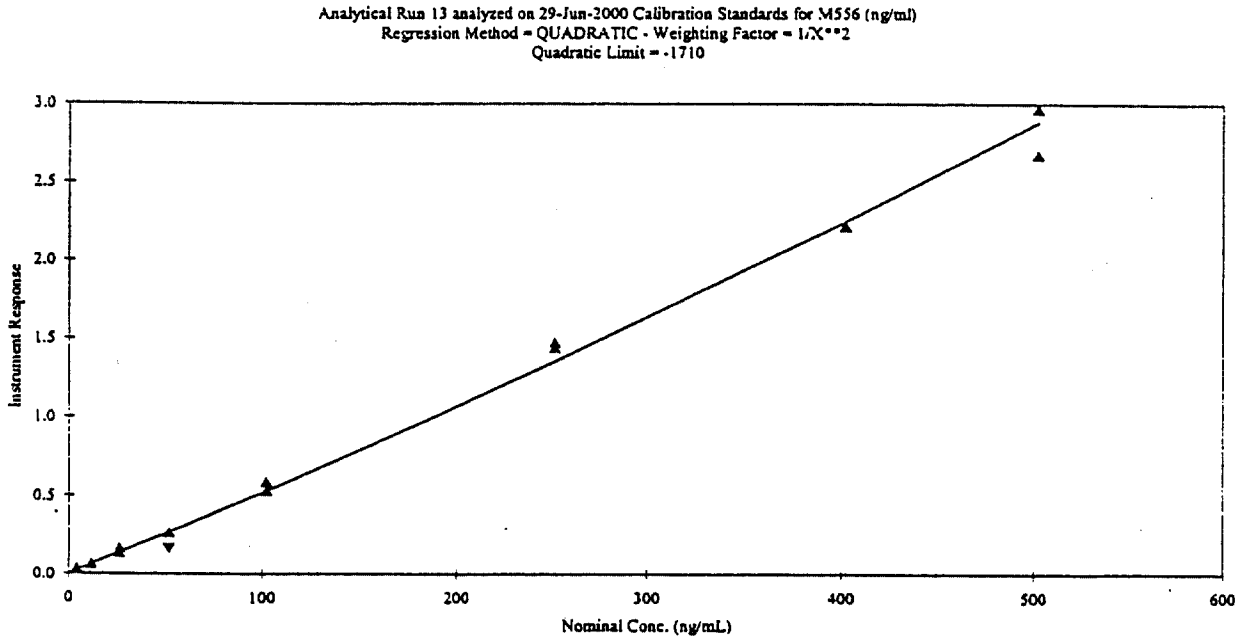
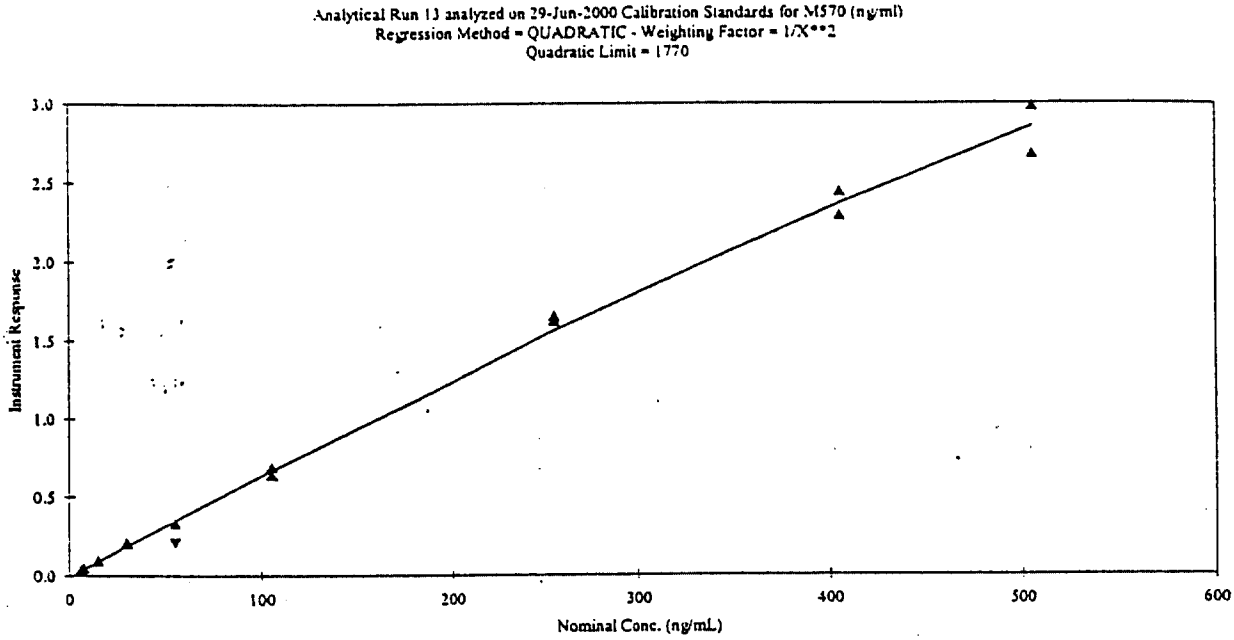


Figure 6. Representative Calibration Curve for M556



000187

Figure 7. Representative Calibration Curve for M570



000188

Figure 8. Standard (57.1 ng/mL) Chromatogram for PFOS

PFOS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.35

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	2.0	2.0
Min. Width	5	5
Mult. Width	20	20
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

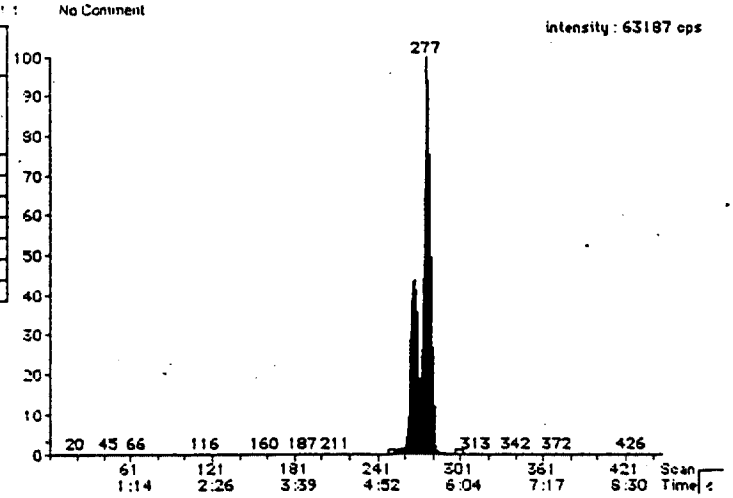
006207003 7.003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8:59 MRM, 446 scans

499.0->80.0

Area	602815
Height	63160
Start Time	5:05
End Time	8:03
Integration Width	0:58.1
Retention Time	5:35
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:58

	Current	Method
Noise Thres.	50.0	50.0
Quant Thres.	5.0	5.0
Min. Width	12	12
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

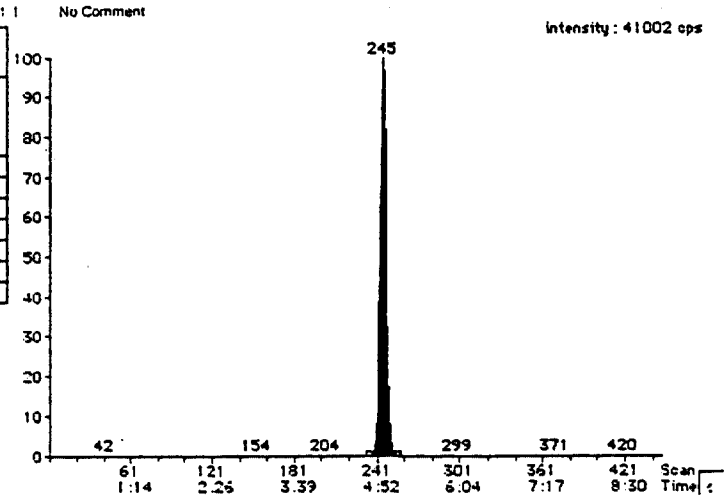
006207003 7.003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8:59 MRM, 446 scans

427.0->407.0

Area	256706
Height	40943
Start Time	4:47
End Time	5:09
Integration Width	0:21.8
Retention Time	4:57
Integration Type	A - BB



000189

Figure 9. Standard (10.1 ng/mL) Chromatogram for PFOSA

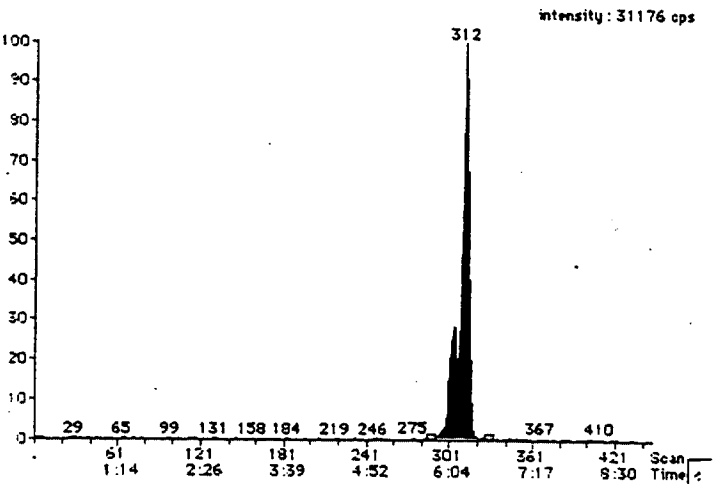
PFOSA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 6:18

	Current	Method
Noise Thres.	20.0	20.0
Quant Thres.	4.0	4.0
Min. Width	5	5
Mult. Width	12	12
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

006207003 7 003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8:59 in 1 period
1: 8:59 MRM, 446 scans
498.0->78.0

Area	261240
Height	31081
Start Time	5:50
End Time	6:38
Integration Width	0:48.4
Retention Time	6:18
Integration Type	A - BB



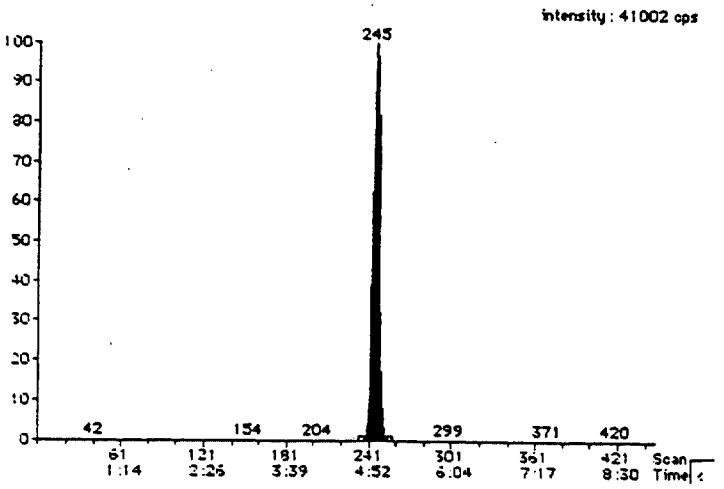
THPFOS
use as Internal Standard
Expected RT 4:58

	Current	Method
Noise Thres.	50.0	50.0
Quant Thres.	5.0	5.0
Min. Width	12	12
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

006207003 7 003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8:59 in 1 period
1: 8:59 MRM, 446 scans
427.0->407.0

Area	256706
Height	40943
Start Time	4:47
End Time	5:09
Integration Width	0:21.8
Retention Time	4:57
Integration Type	A - BB



000190

Figure 10. Standard (15.0 ng/mL) Chromatogram for PFOSAA

PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 6:32

	Current	Method
Noise Thres.	12.0	12.0
Quant Thres.	4.0	4.0
Min. Width	6	6
Mult. Width	15	15
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

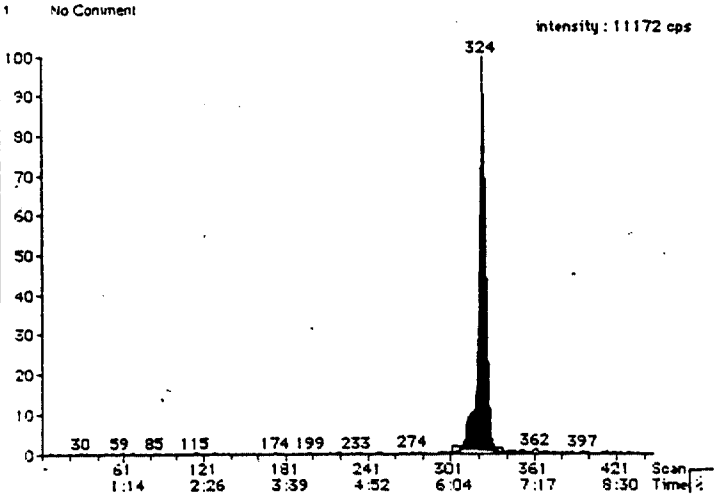
006207003 7.003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8:59 MRM, 446 scans

584.0->419.1

Area	76838
Height	11102
Start Time	6:10
End Time	6:44
Integration Width	0:33.9
Retention Time	6:32
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:58

	Current	Method
Noise Thres.	50.0	50.0
Quant Thres.	5.0	5.0
Min. Width	12	12
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

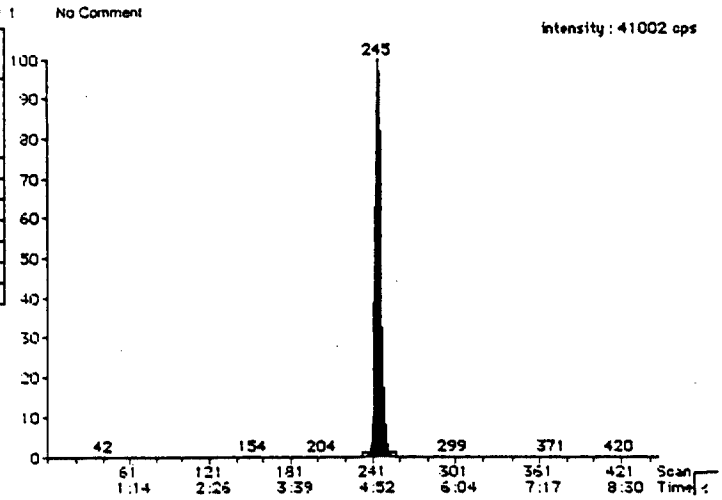
006207003 7.003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8:59 MRM, 446 scans

427.0->407.0

Area	256706
Height	40943
Start Time	4:47
End Time	5:09
Integration Width	0:21.8
Retention Time	4:57
Integration Type	A - BB



000191

Figure 11. Standard (14.8 ng/mL) Chromatogram for POAA

POAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4.59

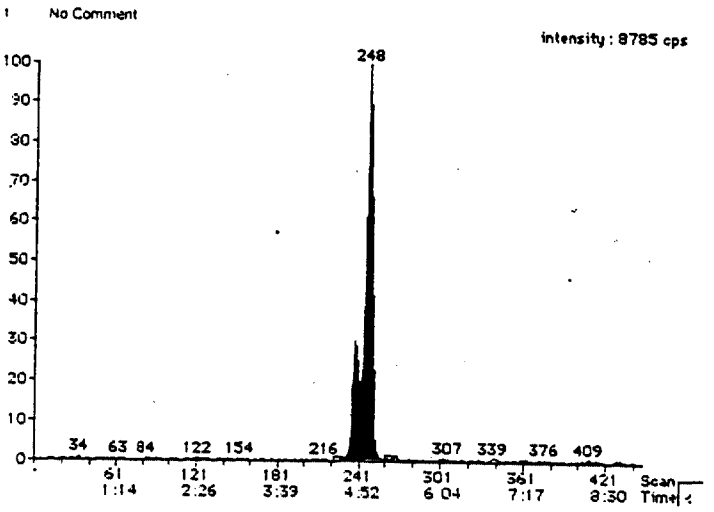
	Current	Method
Noise Thres.	20.0	20.0
Quant Thres.	2.0	2.0
Min. Width	6	6
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

006207003 7.003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8:59 MRM, 446 scans
413.0->169.1

Area	77412
Height	8757
Start Time	4:34
End Time	5:18
Integration Width	0:44.8
Retention Time	5:00
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:58

	Current	Method
Noise Thres.	50.0	50.0
Quant Thres.	5.0	5.0
Min. Width	12	12
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

006207003 7.003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8:59 MRM, 446 scans
427.0->407.0

Area	256706
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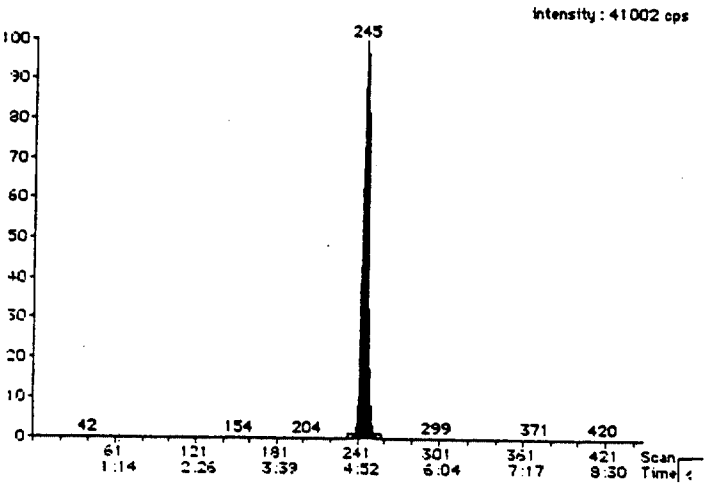
End Time	5:09
Integration Width	0:21.8
Retention Time	4:57
Integration Type	A - BB



000192

Figure 12. Standard (12.2 ng/mL) Chromatogram for PFHS

PFHS
Internal Standard. THPFOS
Use Area
Absolute Retention Time
Expected RT 4.17

	Current	Method
Noise Thres.	20.0	20.0
Quant Thres.	2.0	2.0
Min. Width	5	5
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

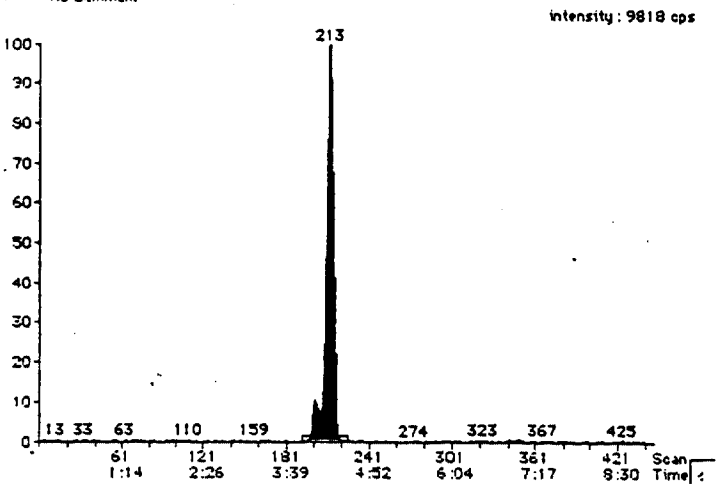
006207003 7.003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8:59 MRM, 446 scans

399.0->79.9

Area	77376
Height	9782
Start Time	3:57
End Time	4:30
Integration Width	0:32.7
Retention Time	4:18
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:58

	Current	Method
Noise Thres.	50.0	50.0
Quant Thres.	5.0	5.0
Min. Width	12	12
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

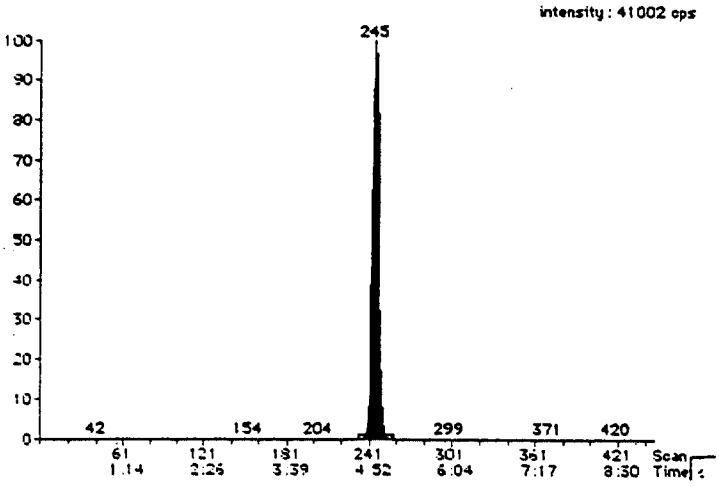
006207003 7.003 S00062 Std3 1 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8:59 MRM, 446 scans

427.0->407.0

Area	256708
Height	40943
Start Time	4:47
End Time	5:09
Integration Width	0:21.8
Retention Time	4:57
Integration Type	A - BB



000193

Figure 13. Standard (11.8 ng/mL) Chromatogram for M556

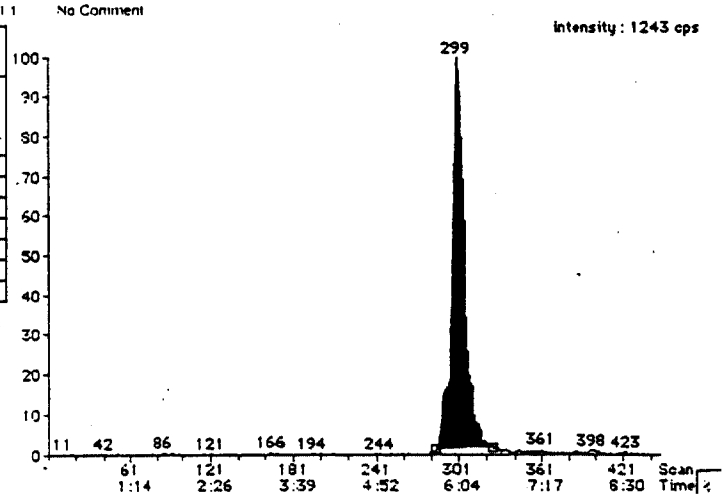
M556
Internal Standard. THPFOS
Use Area
Absolute Retention Time
Expected RT 8.04

	Current	Method
Noise Thres.	12.0	12.0
Quant Thres.	4.0	4.0
Min. Width	5	5
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

006207003 7.003 S00062 Std3 1.1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period
1: 8:59 MRM, 446 scans
556.0->498.0

Area	15835
Height	1227
Start Time	5:45
End Time	6:33
Integration Width	0:48.4
Retention Time	6:02
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:58

	Current	Method
Noise Thres.	50.0	50.0
Quant Thres.	5.0	5.0
Min. Width	12	12
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

006207003 7.003 S00062 Std3 1.1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period
1: 8:59 MRM, 446 scans
427.0->407.0

Area	256706
Height	40943
Start Time	4:47
End Time	5:09
Integration Width	0:21.8
Retention Time	4:57
Integration Type	A - BB

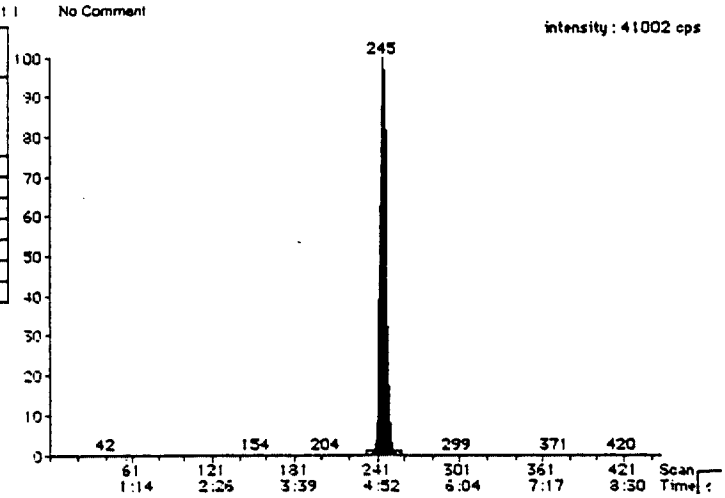


Figure 14. Standard (14.6 ng/mL) Chromatogram for M570

M570
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 6.18

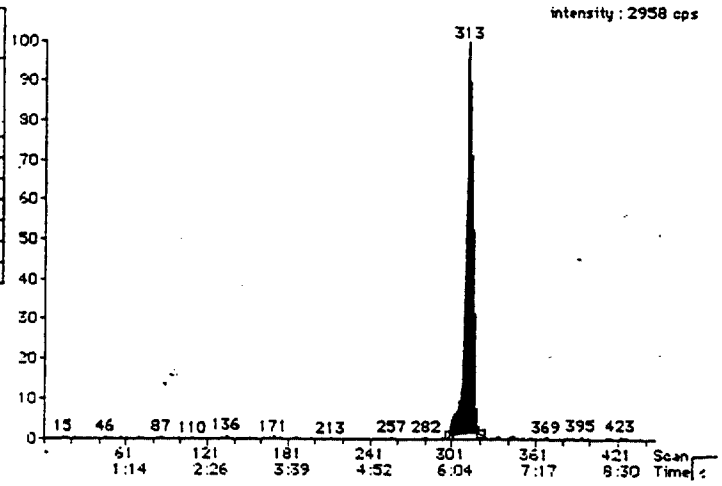
	Current	Method
Noise Thres.	12.0	12.0
Quant Thres.	2.0	2.0
Min. Width	5	5
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

008207003 7.003 S00062 Std3 : 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8.59 MRM, 446 scans
570.0->419.1

Area	21349
Height	2928
Start Time	6:02
End Time	6:28
Integration Width	0:26.8
Retention Time	6:19
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:58

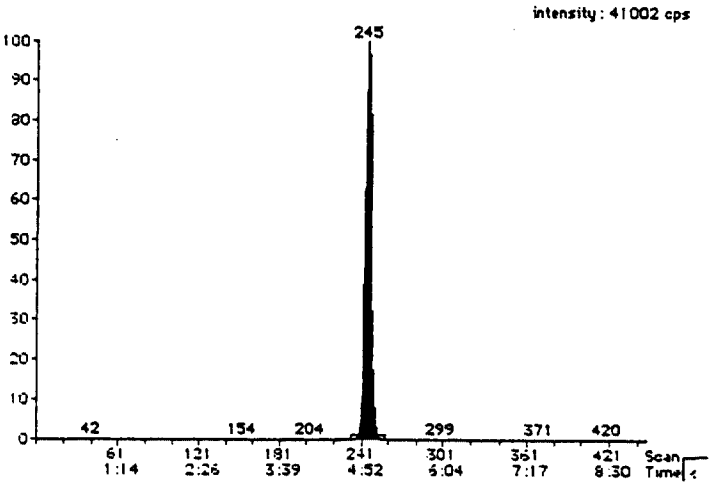
	Current	Method
Noise Thres.	50.0	50.0
Quant Thres.	5.0	5.0
Min. Width	12	12
Mult. Width	10	10
Base. Width	100	100
RT Win. (secs)	20	20
Smooth	1	1

008207003 7.003 S00062 Std3 : 1 No Comment

Thu, Jun 29, 2000 2:10 AM
8.59 in 1 period

1: 8.59 MRM, 446 scans
427.0->407.0

Area	256706
Height	40943
Start Time	4:47
End Time	5:09
Integration Width	0:21.8
Retention Time	4:57
Integration Type	A - BB



000195

APPENDIX A

The following quality control results are from sample analysis study NWBS00-062.

Table A.1. Inter -Assay Precision and Accuracy for PFOSA Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 4.00 ng/mL	Medium QC 150 ng/mL	High QC 400 ng/mL
22-Jul-2000	21	3.95	158	461
		*5.18	153	364
25-Jul-2000	22	4.14	171	394
		3.50	152	379
31-Jul-2000	23	3.68	151	345
		4.35	141	356
01-Aug-2000	24	4.14	150	395
		3.84	144	359
02-Aug-2000	26	4.51	169	378
		3.96	161	406
09-Aug-2000	29	3.70	141	390
		4.09	134	364
12-Aug-2000	31	4.17	148	382
		3.95	148	346
14-Aug-2000	32	3.92	142	319
		3.86	147	364
15-Aug-2000	33	4.98	156	403
		3.81	157	413
16-Aug-2000	36	4.20	144	409
		3.33	126	382
17-Aug-2000	37	4.56	149	384
		3.43	121	470

* >±25% theoretical

000196

Table A.1. Inter -Assay Precision and Accuracy for PFOSA Quality Control Samples (continued)

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 4.00 ng/mL	Medium QC 150 ng/mL	High QC 400 ng/mL
24-Aug-2000	42	4.57	148	471
		3.06	136	425
26-Aug-2000	43	4.61	162	403
		4.02	161	388
28-Aug-2000	44	3.97	142	409
		3.87	147	392
30-Aug-2000	46	3.85	137	381
		3.84	145	474
Mean		4.03	148	394
S.D.		0.458	11.3	37.8
%CV		11.4	7.6	9.6
%Theoretical		100.8	98.7	98.5
%Bias		0.8	-1.3	-1.5
n		30	30	30

Table A.2. Inter -Assay Precision and Accuracy for PFOSAA Quality Control Samples

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 9.00 ng/mL	Medium QC 155 ng/mL	High QC 405 ng/mL
25-Jul-2000	22	8.50	147	359
		7.33	142	431
31-Jul-2000	23	10.2	*115	426
		10.5	170	396
01-Aug-2000	24	10.5	156	416
		10.4	161	444
02-Aug-2000	26	*11.4	177	452
		9.24	171	434
04-Aug-2000	27	*11.3	153	430
		8.37	155	424
09-Aug-2000	29	8.59	152	393
		9.46	152	399
12-Aug-2000	31	10.2	149	395
		10.2	168	455
14-Aug-2000	32	8.46	140	370
		8.89	142	391
15-Aug-2000	33	*10.9	160	439
		9.51	161	449
16-Aug-2000	36	9.40	151	438
		8.70	163	449
17-Aug-2000	37	10.5	158	409
		9.74	157	421

* >±20% theoretical

030198

Table A.2. Inter -Assay Precision and Accuracy for PFOSAA Quality Control Samples (continued)

All concentrations are expressed as ng/mL.

Run Date	Run Number	Low QC 9.00 ng/mL	Medium QC 155 ng/mL	High QC 405 ng/mL
24-Aug-2000	42	9.83	144	461
		8.00	137	475
26-Aug-2000	43	*11.0	169	424
		10.4	167	438
28-Aug-2000	44	9.71	155	436
		10.4	159	447
30-Aug-2000	46	9.30	152	445
		9.65	165	*518
Mean		9.69	155	429
S.D.		1.01	12.5	31.7
%CV		10.4	8.1	7.4
%Theoretical		107.7	100.0	105.9
%Bias		7.7	0.0	5.9
n		30	30	30

* >±20% theoretical

000199

APPENDIX B

Calculation of Persistent Levels of Analytes in Diluted Human Serum Samples

Because there are persistent levels of the analytes in human serum, diluted samples must account for the amount of analyte added from the sample and the amount of analyte added from the human serum matrix. The following section describes equations used to correct for the amount of analyte added from the human serum matrix used to dilute samples.

The total volume (V_T) of a diluted sample is defined as the volume aliquoted from the original sample (V_S) plus the volume of matrix (V_M) added. The dilution factor (DF) is defined as the total volume divided by the volume aliquoted from the original sample.

$$V_T = V_S + V_M$$

$$DF = V_T / V_S$$

Example

$$V_T = 0.1 \text{ mL sample} + 0.4 \text{ mL control matrix}$$

$$V_T = 0.5$$

The amount of analyte A in a diluted sample is equal to the volume aliquoted from the original sample times the concentration of analyte in the sample (C_S) plus the volume of control matrix added times the concentration of analyte in the control matrix (C_M). The concentration of analyte in the sample (C_S) is determined during sample analyses. The concentration of analyte in the control matrix (C_M) is determined experimentally for each lot of control matrix as described in Section 2.1.

Equation 1.

$$\textbf{Total Amount of analyte} = V_S * C_S + V_M * C_M$$

Example

Total Amount of analyte = [0.1 mL sample * 100 ng/mL]+ [0.4 mL control matrix * 10 ng/mL]

Total Amount of analyte = 14 ng

000200

Amount of analyte from sample = 10 ng

Amount of analyte from control matrix = 4 ng

The concentration of the analyte (C_A) in the diluted sample is equal to the sum of the following: 1) the ratio of the volume of sample (V_S) to the total volume of the diluted sample (V_T) times the concentration in the sample (C_S), and 2) the ratio of the volume of control matrix in the diluted sample (V_M) to the total volume of the diluted sample(V_T) times the concentration in the control matrix (C_M). (This is the concentration of analyte in the diluted sample prior to any adjustment for the actual dilution.)

The equation for the above is:

Equation 2.

$$C_A = (V_S/V_T)*C_S + (V_M/V_T)*C_M$$

Example

$$C_A = [(0.1\ mL/0.5\ mL) * 100\ ng/mL] + [(0.4\ mL/0.5\ mL) * 10\ ng/mL]$$

$$C_A = 28\ ng/mL$$

(28 ng/mL = 14 ng total amount calculated above/0.5 mL total volume)

Replacing the volume of control matrix V_M with (V_T-V_S) into this equation produces:

Equation 3.

$$C_A = (V_S/V_T)*C_S + ((V_T-V_S)/V_T)*C_M$$

or

$$C_A = (V_S/V_T)*C_S + (1-V_S/V_T)*C_M$$

Example

$$C_A = [(0.1\ mL/0.5\ mL)*100\ ng/mL] + [(1-(0.1\ mL/0.5\ mL))*10\ ng/mL]$$

$$C_A = 20\ ng/mL + 8\ ng/mL$$

$$C_A = 28\ ng/mL$$

Inserting 1/DF for the term V_S/V_T yields the final equation for C_A:

000201

Equation 4.

$$C_A = (1/DF)*C_S + (1-1/DF)*C_M$$

Example

$$C_A = [(1/5)*100\ ng/mL] + [(1-(1/5))*10\ ng/mL]$$

$$C_A = 28\ ng/mL$$

The concentration determined by the Watson LIMS system (C_W) for any sample is:

Equation 5.

$$C_W = DF * C_A$$

Example

$$C_W = 5 * 28\ ng/mL$$

$$C_W = 140\ ng/mL$$

(This is the concentration of analyte after adjusting for the actual dilution.)

Substituting the final equation for C_A (Equation 4.) into the equation for C_W gives:

Equation 6.

$$C_W = DF *[(1/DF)*C_S + (1-1/DF)*C_M]$$

Example

$$C_W = 5 * [((1/5) *100\ ng/mL) + (((1-(1/5)) * 10\ ng/mL)]$$

$$C_W = 5* [20\ ng/mL + 8\ ng/mL]$$

$$C_W = 140\ ng/mL$$

Solving this last equation for C_S produces the equation for correction due to persistent levels which is used to adjust theoretical calibration and QC concentrations.

Equation 7.

$$C_S = C_W - (DF-1)*C_M$$

Example

$$C_S = 140\ ng/mL - [(5-1) * 10\ ng/mL]$$

060202

$$C_S = 140 \text{ ng/mL} - 40 \text{ ng/mL}$$

$$C_S = 100 \text{ ng/mL}$$

060203

AR226_1210

Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS

Assay Revalidation Addendum Report

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Northwest Bioanalytical (NWB)
A Division of NWT Inc.
1121 East 3900 South
Salt Lake City, UT 84124

PREPARED FOR: 3M Environmental Technology and Services
935 Bush Avenue
St. Paul, MN 55133

AUTHOR:
Connie O Sakashita DATE: 11-20-01
Connie O. Sakashita, B.S., NWB Project Manager

APPROVED FOR RELEASE BY:
Patrick Bennett DATE: 11-20-01
Patrick Bennett, M.S., M.B.A., NWB Laboratory Director

CONTAIN NO CBI

030204

QUALITY ASSURANCE STATEMENT

LABORATORY:	Northwest Bioanalytical (NWB) A Division of NWT, Inc. 1121 East 3900 South Salt Lake City, UT 84124
SPONSOR:	3M Environmental Technology Services 935 Bush Avenue St Paul, MN 55133
COMPOUND(S):	PFOS, PFOSA, PFOSAA, PFHS, M556 and M570
NWB STUDY NUMBER:	NWBS00-040
SPONSOR STUDY NUMBER:	Method Validation
NWB STUDY TITLE:	Quantitative Determination of PFOS and Metabolites in Human Serum by LC/MS/MS, Assay Validation

The method validation study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all projects in accordance with the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.) The following inspections were performed by the NWB QAU per SOP.

Inspection and Reporting Statement.

		Date Inspection Report Issued To	
Inspection Date	Phase of Study	NWB Project Manager	[1]NWB Management
15 May 2000	Analytical Plan	15 May 2000	31 May 2000
15-18 May 2000	Assay Validation	18 May 2000	31 May 2000
07-18 Sep 2000	Report Draft/Raw Data (NWBR00-108)	20 Sep 2000	29 Sep 2000
24 Jan 2000	Final Report (NWBR00-108)	24 Jan 2000	31 Jan 2000
19-30 Jul 2001	Report Draft/Raw Data (NWBR00-122)	30 Jul 2001	31 Jul 2001
05-07 Nov 2001	Report Draft/Raw Data (NWBR00-122)	08 Nov 2001	30 Nov 2001
20 Nov 2001	Final Report (NWBR00-122)	20 Nov 2001	30 Nov 2001

[1]Reports to NWB Management are issued monthly.

As can reasonably be established, the methods and procedures described and the results incorporated into this final report accurately reflect the raw data.

Sheryl A. Mathews

Sheryl A. Mathews, A.A.S.
NWB QAU Compliance Auditor

26 Nov 2001
Date

030205

COMPLIANCE STATEMENT

The method validation study described in this addendum report is not included within the definition of a GLP nonclinical regulated study. However, to the best of our knowledge, the stability, extraction efficiency, and plasma abbreviated validation experiments were conducted in accordance with the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 48) and according to the methods and procedures described within this report. In addition, these experiments followed the guidelines of the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.)

This addendum report also included results from research experiments conducted outside of the validation study. These experiments were conducted according to the procedures described within section 7 of this report.

Serum sample results obtained using plasma curves for PFOSA, PFOSAA, POAA, PFHS, M556 and M570 did not meet NWB SOP requirements for validation.

Any known circumstances that may have affected the quality or integrity of the study or data are discussed within the report. This report represents an accurate record of the raw data.

Connie O Sakashita

Date: 11-20-01

Connie O. Sakashita, B.S.
NWB Project Manager

Patrick K. Bennett

Date: 11-20-01

Patrick K. Bennett, M.S., M.B.A.
NWB Laboratory Director

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Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS

Assay Revalidation Addendum Report

1. INTRODUCTION

Northwest Bioanalytical (NWB) was contracted by 3M Environmental Technology and Services (3M) to modify and revalidate a liquid chromatography/tandem mass spectrometry method for the measurement of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in human serum. The accuracy and precision results of the method revalidation were reported in NWB report NWBR00-108 [9.1]. This report summarizes the results from the stability tests conducted as part of the method revalidation for PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in human serum for 3M Environmental Technology and Services. The results from the following tests are also included: extraction efficiency from human serum; ion suppression experiment; and diluted matrix experiment. The suitability of the method for use with plasma was determined by demonstrating intra-assay and LLOQ accuracy and precision and comparing serum and plasma results.

Kris Hansen and James Lundberg at 3M Environmental Technology and Services served as the Study Monitors. The following is a list of NWB supervisory personnel involved in the completion of this work: Connie O. Sakashita, B.S. (Current NWB Project Manager); Brad I. Coopersmith, Ph.D. (NWB Project Manager until October 28, 2000); Licong Jiang, Ph.D. (NWB Senior Scientist); Patrick Bennett, M.S., M.B.A. (NWB Laboratory Director). The following NWB laboratory personnel contributed to the completion of this work: Suzanne Newman, B.S. (NWB Scientist).

NWB SOPs were used in the conduct of this project and were available to project personnel in both electronic and hard copy formats.

Date Study Initiated: 13-Apr-2000 Date Analyses Completed: 25-Apr-2001

The method revalidation study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all studies within the guidelines of the US FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424). Any changes to or deviations from the original protocol (Analytical Plan) were documented through approved protocol amendments or deviation memos and are retained within the raw data.

2. METHODOLOGY

Reference Materials and Matrices

Analyte	*Lot Number*	*Purity*	*Expiration Date*	*Source*	*Storage Conditions*
PFOS (FC-95)	193	100%	12/31/2010	3M	Room Temperature
PFOSA	214	100%	12/31/2010	3M	Room Temperature
PFOSAA (FC-129)	617	53.8%	12/31/2010	3M	Room Temperature
POAA (FC-143)	245	100%	12/31/2010	3M	Room Temperature**
PFHS* (used for serum)	S398-182	100%	12/31/2010	3M	-20°C
PFHS (used for plasma)	SE-036	91.1%	1/01/2010	3M	-20°C
M556	NB113047-8D	99.89%	12/31/2010	3M	Room Temperature
M570	118506-26	99.75%	12/31/2010	3M	Room Temperature
THPFOS	59909	90%	12/31/2010	3M	Room Temperature

* Received as a 6,200 ppm solution in methanol. ** Kept dry.

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Matrix	*Lot Numbers*
Human serum	BC30399-4; BC061900-1
Human plasma (Northern Chinese)	C5186; C4929; C5517; C5200

The reference material purity for PFOS, PFOSA, PFHS and POAA was not available prior to the conduct of this study. Therefore, all serum concentrations included in the report for these analytes are based upon an assumed purity of 100%.

After the revalidation was completed, 3M contracted with Centre Analytical Laboratories, Inc. in State College, Pennsylvania to determine the absolute concentration of PFOS, POAA and PFHS in NWB stock solutions used to prepare the analytical standards and controls used for this revalidation and subsequent analyses. All arrangements for purity determinations and transfers of NWB solutions to Centre Analytical Laboratories, Inc. were performed by 3M. Per agreement with the Sponsor, NWB quantitative results were not corrected for the purity corrections. Therefore, based on the results obtained, the serum concentrations included in this report should be corrected according to the following table (except for the serum samples included in the serum versus plasma experiments, Section 7.3):

Analyte	Correction Factor
PFOS	0.836
POAA	0.909
PFHS	0.855

Principles of the Method

The analytical method consisted of a liquid:liquid extraction procedure followed by evaporation and reconstitution of the extract residue with 30:70 (v/v) 20 mM ammonium acetate in water: 20 mM ammonium acetate in methanol. The samples were analyzed by liquid chromatography/tandem mass spectrometry using a PE Sciex API 3000. The instrument was operated in the multiple reaction monitoring (MRM) mode under

optimized conditions for PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 detection.

A detailed description of the methodology is included in NWB report NWBR00-108 [9.1].

Acceptance Criteria for Human Serum

Calibration Curve

Each run will include in duplicate calibration standards at six or more concentrations covering the lower to upper limit of quantitation. For all analytes except PFOSA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 15% (± 20% for LLOQ) of their individual target concentrations. For PFOSA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 20% (± 25% for LLOQ) of their individual target concentrations. A calibration standard will be considered a statistical outlier if the back-calculated concentration is greater than two times the acceptance criteria for that standard.

Lower Limit of Quantitation

The back-calculated concentrations of at least one of the duplicate lowest points in the calibration curve must be within ± 25% of the target concentration for PFOSA to qualify as the LLOQ and within ± 20% of the target concentration to qualify as the LLOQ for all other analytes. If this criterion is not met, the next level is subjected to the same test and the LLOQ raised accordingly.

Quality Control Samples

Each analytical run will include low, medium and high QC samples in duplicate. The measured concentrations of at least two-thirds of all analytical QCs must be within ± 20% of their target concentrations (± 25% for PFOSA), and no two QCs at the same concentration can be outside the limit. If study samples require dilution, a dilution QC will be analyzed in triplicate for each dilution level (except for PFOSA and PFOSAA

which should not be diluted with control matrix). At least two dilution QC at each level must be within ± 20% of its target concentrations in order to accept diluted study samples at that level. The dilution QC acceptance is independent of the undiluted analytical QC acceptance.

3. STABILITY EVALUATION

The revalidated method's final acceptance criteria is used as a basis for evaluating fluorochemical stability results. If the mean concentration obtained for stability test QC samples falls within ± 20% (± 25% for PFOSA) of the mean control QC concentrations, then stability is considered acceptable under the specified test conditions. Control QC samples are defined as samples not subjected to test conditions.

The calibration curve parameters summary and individual calibration standard results for the serum stability runs can be found in Tables 1-14. The analytical QC results for the stability runs can be found in Tables 15-21.

3.1. Freeze-Thaw and Room Temperature Matrix Stability

The stability of PFOS, PFOSA, PFOSAA, POAA, and PFHS in human serum for three freeze-thaw cycles was demonstrated and reported under the original validation study NWBS98-092, NWB report NWBR99-005 [9.2]. Therefore, only freeze-thaw stability for M556 and M570 was needed. Acceptable results for the three freeze-thaw cycle stability test was obtained on Run 4 for M556 and M570 (data not included).

However, since some analyzed subject samples subsequently underwent more than three freeze-thaw cycles, the stability of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 was evaluated after seven freeze-thaw cycles. All analytes demonstrated acceptable stability when compared against controls that underwent one freeze-thaw cycle (Tables 22 – 28).

During the seven freeze-thaw cycle test, samples were left at room temperature for up to 6.75 hour periods between each freeze cycle. Since the freeze-thaw samples

demonstrated acceptable stability, the stability of serum samples left at room temperature for up to 6.75 hours is also demonstrated.

3.2. Reinjection Stability

The quantitative reproducibility of standards and controls after reinjection was determined for PFOS, PFOSA, PFOSAA, POAA, PFHS, M556, and M570. The samples were extracted and analyzed on June 30, 2000 (Run 15 – data shown in NWBR00-108). These samples were reinjected on July 3, 2000 after storage at room temperature (Run 23). The results from Run 23 indicate reinjection stability when compared with data obtained from the original injection [Run 15] (Tables 29 – 35).

3.3. Extract Storage Stability

Extract stability was evaluated at different storage temperatures and for various lengths of time. The evaluations were performed using QC lots prepared on June 27, 2000 for all analytes and conditions. Although the same QC lots (prepared on June 27, 2000) were not used for PFOSA and PFOSAA run acceptance determination, they still provided a control value for stability evaluations.

3.3.1. Room Temperature Extract Stability

The extract stability of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 was determined for low and high serum QC extracts stored at room temperature for seven days prior to analysis. These test QC samples were compared against serum control samples that were analyzed immediately following extraction.

The extracted test QC samples demonstrated acceptable stability after seven days at normal room temperature (Tables 36 – 42).

3.3.2. Reduced Temperature (1 to 8°C) Extract Stability

The extract stability of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 was determined for low and high serum QC extracts stored at 1 to 8°C for four

days prior to analysis. These test QC samples were compared against serum control samples analyzed immediately following extraction.

The extracted test QC samples demonstrated acceptable stability after four days at 1 to 8 °C (Tables 43 – 49).

3.3.3. Reduced Temperature (-20 °C) Extract Stability

The extract stability of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556, and M570 was determined for low and high serum QC extracts stored at -20 °C for five days prior to analysis. These test QC samples were compared against serum control samples analyzed immediately following extraction.

The extracted test QC samples demonstrated acceptable stability after five days at -20 °C (Tables 50 – 56).

3.4. Long-Term Matrix Stability in Human Serum

The long-term matrix stability of PFOS, POAA, PFHS, M556 and M570 in human serum was determined by storing QC samples for approximately 55 days at –20 °C. The long-term matrix stability of PFOSA and PFOSAA in human serum was determined by storing QC samples for approximately 42 days at –20 °C. The mean test QC concentrations were compared against theoretical.

The test QC samples demonstrated acceptable stability after approximately 42 days for PFOSA & PFOSAA or 55 days for other analytes, at –20 °C (Tables 57 – 63).

4. EXTRACTION EFFICIENCY FROM HUMAN SERUM

The extraction efficiencies of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 were determined by comparing the area ratios obtained for the following three cases:

1. Both the analyte and internal standard added following the extraction (post-extract).

2. The analyte added to serum prior to extraction and the internal standard added following extraction (pre-extract analyte).
3. The internal standard added to serum prior to extraction and the analyte added following the extraction (pre-extract internal standard).

The extraction efficiencies were then determined by the area ratio of the pre-extract samples to the post-extract samples. The extraction efficiency experiments were performed at both low and high concentrations to determine that there was no concentration bias.

The total amount of analyte in the sample prior to extraction (pre-extract) is equal to the amount of analyte added plus the persistent amount in the blank matrix. The amount of analyte in the post-extract solution is the amount of analyte added plus the amount of analyte recovered (extracted) from the blank matrix. Therefore, the following formula was used to determine the extraction efficiency for PFOS, PFOSAA, POAA, PFHS, M556 and M570:

$$((Spk + Per) * EE) / (Spk + (Per * EE)) = Rpre / Rpost; \text{ where}$$

Spk = the amount of analyte added
Per = the persistent amount of analyte in the blank matrix
EE = the extraction efficiency
Rpre = the ratio of analyte/internal standard determined for the analyte added prior and internal standard added after extraction
Rpost = the ratio of analyte/internal standard determined for the analyte and internal standard added after extraction

	PFOS	PFOSA	PFOSAA
Mean extraction efficiency (%)	37.3	69.6	62.7

	POAA	PFHS	M556
Mean extraction efficiency (%)	15.6	15.3	39.1

	M570	THPFOS
Mean extraction efficiency (%)	57.1	5.99

The results are shown in Tables 64 – 70.

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5. COMMENTS AND CONCLUSIONS

Per agreement with the Sponsor, the regressions were not recalculated based upon the updated purity information for PFOS, POAA and PFHS. Some differences might occur due to the effect of rounding if the regressions were performed with the purity corrected concentrations, but these differences would have a negligible effect on the overall interpretation of the revalidation results.

PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in human serum appear to be stable for seven freeze-thaw cycles and demonstrated matrix stability for up to 6.75 hours at room temperature. PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 exhibit extract stability for seven days at room temperature, four days at 1 to 8 °C, and five days at -20 °C. The analytes also demonstrated matrix stability for approximately 55 days (for PFOS, POAA, PFHS, M556 and M570) and 42 days (for PFOSA and PFOSAA) at -20 °C in human serum.

6. ABBREVIATED VALIDATION – HUMAN PLASMA

The choice of matrix for calibration curve preparation is dependent upon the desired LLOQ. 3M supplied NWB with human plasma identified as "Northern Chinese" plasma (referred to as Chinese plasma or CP). This matrix has very low levels of fluorochemicals and will be used for studies with a desired LLOQ of ≤ 5.00 ppb. The following sections summarize the accuracy and precision of the fluorochemicals in human plasma and the quantitative accuracy of serum sample results obtained against plasma calibration curves using the revalidated serum method.

The absolute concentrations of PFOS, PFHS and POAA in the stock solution used for plasma analyses were determined by Centre Analytical Laboratories, Inc. in State College, Pennsylvania. Based upon the results, the concentrations of plasma standard and quality control samples were corrected using the following factors:

Analyte	Correction Factor
PFOS	0.821
POAA	0.961
PFHS	1.043

All plasma experiment concentrations included in this report reflect adjustments based upon the correction factors above. The serum target concentrations for these experiments have also been adjusted based upon the correction factors (listed on page 15 of report).

The tests for the abbreviated human plasma validation were conducted after the serum revalidation was completed. They were performed outside of the revalidation or in sample analysis studies conducted after the serum revalidation. However, all applicable NWB SOPs were followed.

Persistent Levels of Analytes in Chinese Plasma

The persistent levels of the analytes in Chinese plasma varied in each tube of plasma tested. Therefore, several tubes of plasma were pooled and the amounts of persistent analytes were determined. Previous determination of persistent levels was achieved by comparison with rabbit serum curves. For the experiments included in this report, the persistent levels were determined as follows:

Known amounts of the analytes were added to Chinese plasma in the range of 1 to 50 or 1 to 100 ppb. The standards were then analyzed according to the revalidated method on at least two separate occasions. A linear regression was performed using the spiked analyte concentration as the y-value and the ratio of the analyte area to internal standard area as the x-value (Microsoft® Excel 97 formulas). The mean resultant y-intercept determined from multiple runs was then used as an estimate of the persistent level of analyte if it was greater than 0.2 ppb.

6.1. Range of Quantitation

The target calibrator concentrations were approximately 1.00 , 2.50, 10.0, 25.0, 50.0, 100, 250, 400 and 500 ppb for all analytes except M556. The target calibrator concentrations for M556 were approximately 2.50, 10.0, 25.0, 50.0, 100, 250, 400 and 500 ppb. The final curve range can vary based upon the persistent levels of the analyte in the human plasma pool used. For the validation study, the calibration curve ranges were as follows:

PFOS	3.94 to 414 ppb
PFOSA	1.00 to 500 ppb
PFOSAA	1.60 to 501 ppb
POAA	1.92 to 481 ppb
PFHS	1.36 to 523 ppb
M556	2.50 to 500 ppb
M570	1.00 to 500 ppb

The calibration curve parameter summaries and individual calibration standard results for the plasma runs can be found in Tables 71 – 84.

6.2. Lower Limit of Quantitation

The precision at the Lower Limit of Quantitation (LLOQ) of the assay in human plasma was determined by using six 100-µL aliquots of the pooled Chinese plasma fortified with PFOS, PFOSA, PFOSAA, POAA, PFHS, M556, and M570 at concentrations equal to the LLOQ. The LLOQ concentration was dependent upon the persistent levels found in the plasma.

	PFOS	PFOSA	PFOSAA	POAA
Lower Limit of Quantitation	3.94 ppb	1.00 ppb	1.60 ppb	1.92 ppb
Precision (%CV)	5.4	10.1	5.7	4.4
Accuracy (%Bias)	-1.5	1.0	-2.5	-8.9

	PFHS	M556	M570
Lower Limit of Quantitation	1.36 ppb	2.50 ppb	1.00 ppb
Precision (%CV)	9.9	5.7	5.2
Accuracy (%Bias)	-5.1	-2.0	3.0

The results are shown in Tables 85 – 91.

6.3. Intra-Assay Precision and Accuracy

The precision and accuracy of the LC/MS/MS method for PFOS, PFOSA, PFOSAA, POAA, PFHS, M556, and M570 in human plasma were determined by analyzing three levels of quality control samples in replicates of five on a single run.

	PFOS Intra-assay	PFOSA Intra-assay	PFOSAA Intra-assay	POAA Intra-assay
Precision (%CV)	1.5 to 2.8	1.8 to 5.7	2.6 to 5.8	0.8 to 5.8
Accuracy (%Theoretical)	101.2 to 112.3	104.0 to 116.3	102.6 to 111.7	103.4 to 106.2
Accuracy (%Bias)	1.2 to 12.3	4.0 to 16.3	2.6 to 11.7	3.4 to 6.2

	PFHS Intra-assay	M556 Intra-assay	M570 Intra-assay
Precision (%CV)	1.7 to 3.1	4.7 to 7.6	1.9 to 5.6
Accuracy (%Theoretical)	95.7 to 106.0	98.7 to 111.3	101.3 to 118.0
Accuracy (%Bias)	-4.3 to 6.0	-1.3 to 11.3	1.3 to 18.0

The results are shown in Tables 92 – 98.

6.4. Proposed Acceptance Criteria for Human Plasma

Calibration Curve

Each run will include in duplicate calibration standards at six or more concentrations covering the lower to upper limit of quantitation. For all analytes except PFOSA and PFOSAA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 15% (± 20% for LLOQ) of their individual target

concentrations. For PFOSA and PFOSAA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 20% (± 25% for LLOQ) of their individual target concentrations. A calibration standard will be considered a statistical outlier if the back-calculated concentration is greater than two times the acceptance criteria for that standard.

Lower Limit of Quantitation

The back-calculated concentrations of at least one of the duplicate lowest points in the calibration curve must be within ± 25% of the target concentration for PFOSA and PFOSAA to qualify as the LLOQ and within ± 20% of the target concentration to qualify as the LLOQ for all other analytes. If this criterion is not met, the next level is subjected to the same test and the LLOQ raised accordingly.

Quality Control Samples

Each analytical run will include low, medium and high QC samples in duplicate. The measured concentrations of at least two-thirds of all analytical QCs must be within ± 20% of their target concentrations (± 25% for PFOSA and PFOSAA), and no two QCs at the same concentration can be outside the limit.

Based upon the results, the method is suitable for use with human plasma.

7. ADDITIONAL POST-REVALIDATION EXPERIMENTS

Additional experiments were conducted subsequent to completion of the revalidation to further elucidate the applicability of the revalidated method to sample analysis. These tests were performed outside of any revalidation or sample analysis studies.

7.1. Ion Suppression Experiment

During analysis of medical surveillance serum samples, it was noted that the internal standard response for the study samples differed from the standards and quality controls. POAA and the internal standard (THPFOS) have very similar retention

times. Therefore, THPFOS and POAA are not chromatographically resolved in extracted samples containing high concentrations of POAA. This co-elution can suppress the internal standard response and introduce a (high) bias in the measured concentration of analytes when compared against the standard curve. The suppression of the internal standard signal is proportional to the concentration of POAA in the sample and dependent on the LC resolution of the two compounds.

An experiment was performed to evaluate the magnitude of this effect. Low control samples were fortified with various (high) levels of POAA. The test samples were analyzed in triplicate in a single run (PFOSMD-41). The mean concentration of the test samples were then compared against the mean of the analytical low control samples. A summary of the results is shown in the following table:

	PFOS	*PFOSA*	*PFOSAA*	*PFHS*	*M556*	*M570*	*IS Area*
Control Mean Conc. (ppb)	51.8	4.05	6.83	8.04	5.41	6.87	108857
POAA added at ~59200 ppb							
% change from control mean	182%	152%	180%	180%	216%	207%	-75%
POAA added at ~11800 ppb							
% change from control mean	28%	39%	52%	29%	52%	56%	-43%
POAA added at ~5920 ppb							
% change from control mean	16%	21%	30%	16%	36%	34%	-32%
POAA added at ~592 ppb							
% change from control mean	-2%	-11%	-5%	-7%	-8%	-8%	+11%

The experiment verified that very high levels of POAA resulted in a lower response for the internal standard, which then results in a falsely elevated concentration for the other 6 analytes. No significant bias is introduced when the extracted concentration of POAA is within the revalidated range of the analytical method (approximately 500 ppb). However, some samples can contain POAA at concentrations above the revalidated range of the analytical method, and will likely introduce a bias in the reported concentrations of PFOS, PFHS, PFOSA, PFOSAA, M556, or M570 when the concentration of these analytes are determined prior to sample dilution. The amount of bias could range up to approximately 56%. However, the amount of

PFOS, PFHS, PFOSA, PFOSAA, M556, or M570 impurities in the POAA reference material is not known. This prevents accurate determination of the % bias. It should be noted that since the amount of ion suppression is dependent upon the amount of extracted POAA, no concentration bias is expected for an analyte result determined from a pre-diluted sample wherein the extracted POAA concentration is less than approximately 500 ppb. This bias will not significantly effect the interpretation of the analytical results.

7.2. Dilution Matrix Experiment

The amount of Chinese plasma supplied by 3M is limited. Therefore, it cannot be used on a routine basis for dilution. Human serum (control serum) is used to dilute test samples as needed to obtain measurable concentrations within the range of the calibration curve. The measured concentrations of fluorochemicals (FC) obtained for diluted samples must then be corrected for persistent levels of the analytes in the control serum. Experiments were performed to determine whether a substitute matrix that did not contain persistent levels of FC analytes could be used when performing dilutions.

Previous experiments had shown that samples diluted with buffer instead of serum did not accurately quantitate against a typical serum curve (data not reported). This indicated that the matrix to buffer ratio of the sample affected the extraction recovery. For the experiments described in this report, QC samples were diluted with buffer. The calibration curve was prepared in the same buffer to matrix ratio as the QC samples to determine if this was a viable method for diluting samples. A different dilution ratio was prepared to determine if samples could be quantitated from a curve prepared in a matrix to buffer ratio that was different from the QC samples (e.g., if QC samples diluted with matrix by a factor of 50 (2% matrix in buffer) could be quantitated against a curve prepared in 10% matrix in buffer). For the experiments described below, ammonium acetate buffer was used to dilute the matrix for the QCs and calibration curves.

Calibration Curve and QC Preparation

The experiment (PFOSMD run 39) used a curve prepared in 10% serum in buffer, and three levels of QC samples diluted with buffer by a factor of 10 (10% matrix to buffer ratio). The QC concentrations prior to dilution were 150, 400, and 4000 ppb plus the persistent level for each analyte. QC samples at 4000 ppb (plus persistent level) were also diluted 1:50 with buffer.

The calibration curve parameter summaries and individual calibration standard results for the runs can be found in Tables 99 – 112. The quality control sample results can be found in Tables 113 – 119.

Results for 10% Serum in Buffer (PFOSMD Run 39)

The mean accuracy (% theoretical) for all analytes at all QC concentration levels diluted with buffer by a factor of 10 (the same serum to buffer ratio as the curve) ranged from 94.1% to 114.3%. The intra-assay precision (%CV) for all analytes were less than or equal to 5.6% for each QC concentration.

The mean accuracy (% theoretical) for the QC samples diluted with buffer by a factor of 50 ranged from 56.5% to 84.8%. The inter-assay precision (%CV) for all analytes were less than or equal to 4.4%. All analytes were biased low, and all analytes except for POAA and PFHS were not within ±20% of the target concentrations.

QC samples met the acceptance criteria of ± 20% of target concentration (±25% for PFOSAA) when diluted with buffer if quantitated against a curve prepared in the same serum to buffer ratio. QC samples diluted with buffer at a different ratio than the calibration curve did not meet the acceptance criteria for all analytes.

It appears that quantitating samples diluted with buffer against a curve prepared in the same serum to buffer ratio is a viable way minimizing the corrections needed due to persistent levels of analytes in the dilution matrix. However, since most analytes cannot be accurately quantitated against a calibration curve that is prepared in a serum/buffer ratio different from that of the diluted samples, a separate curve with the same buffer to matrix ratio would need to be prepared for each dilution factor needed.

7.3. Serum and Plasma Comparison

Because the choice of calibration curve and quality control matrix is dependent upon the desired LLOQ, it is not always possible to match the subject sample matrix with that of the calibration curve and quality control matrix. For instance, it is necessary to use Chinese plasma to prepare the calibration standards and quality control samples when analyzing serum samples where a LLOQ of ≤ 5.00 ppb is desired. Therefore, to evaluate if, and to what extent, the use of plasma calibration standards affects the quantitative analysis of fluorochemicals in serum, a comparison of quantitative results obtained from plasma curves versus serum curves was performed.

Calibration curves were prepared in both human serum and Chinese plasma, and were analyzed along with serum and Chinese plasma quality control samples in replicates of four. The results for the calibration and quality control samples are shown in Tables 120 – 140.

The confidence interval around the serum QC mean was determined for serum samples quantitated against the plasma curve using Microsoft® Excel 97. Alpha (α) was set to 0.05 for a 95% confidence level. The confidence interval around the mean was compared to the analytical acceptance range of the QC which is ± 20% (± 25% for PFOSA and PFOSAA) of the target concentration. The summary results of the experiment are shown in Table 141.

The 10/27/00 serum curve for M570 had a raised LLOQ of 5.20 ppb. Data for PFOSA and PFOSAA analyzed on 10/27/00 and data for PFHS and M556 analyzed on 4/25/01 are not used because either the serum or plasma calibration curve or the QC samples did not meet the acceptance criteria. One high QC replicate analyzed on 10/27/00 was not included in the statistical calculations because of an extraction error.

The confidence intervals around the mean serum QC results for PFOS are within the acceptance criteria at all levels.

The confidence intervals around the mean serum QC results for PFOSA are greater than the acceptance range of ±25% deviation. The confidence intervals ranged up to 78% deviation from target.

The confidence intervals around the mean serum medium QC result for PFOSAA was 33.9% deviation which is greater than the plasma acceptance range of ±25% deviation. The confidence interval for the PFOSAA low and high QC samples are within the acceptance criteria.

The confidence intervals around the mean serum QC results for POAA and PFHS are greater than the acceptance range of ±20% deviation. The confidence intervals ranged up to 27.4% deviation from target for POAA and up to 37.6% deviation from target for PFHS.

The confidence interval around the mean serum low QC results for M556 was –26.9% deviation. This is greater than the acceptance range of ±20% deviation and may be due a low value obtained for one replicate. The confidence intervals for the M556 medium and high QC samples are within the acceptance criteria.

The confidence intervals around the mean serum medium QC result for M570 was 21.9% deviation which is slightly greater than the plasma acceptance range of ±20% deviation. The confidence interval for the M570 low and high QC samples are within the acceptance criteria.

CONCLUSION

Serum sample results obtained using plasma curves for PFOSA, PFOSAA, POAA, PFHS, M556, and M570 did not meet NWB SOP requirements for validation. Results may vary on average up to 26% from QC results obtained using human serum calibration curves at some concentration levels for PFHS, PFOSAA, POAA, M556, and M570. For PFOSA, results may vary on average up to 43% from QC results obtained using human serum calibration curves. However, when a lower limit of

quantitation less than 5 ppb is required by the Sponsor, it will be necessary to use plasma curves.

8. DATA MANAGEMENT

PFOS, PFOSA, PFOSAA, POAA, PFHS, M556, M570, and THPFOS chromatographic peaks were integrated using PE Sciex MacQuan software (version 1.6) with a smooth factor of one. Quantitation was based upon quadratic regression analysis of calibration curves (weighted $1/x^2$) using the area ratio vs. concentration calculated by the Watson® DMLIMS software (versions 6.1.1.03 and 6.1.1.04).

9. REFERENCES

[9.1.] C. Sakashita. "Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS." Assay Revalidation Report. NWB Study NWBS00-040. NWB Report NWBR00-108. January 24, 2001.

[9.2.] D. Vollmer. "Quantitative Determination of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in Human Serum by LC/MS/MS." Assay Validation Report. NWB Study NWBS98-082. NWB Report NWBR99-005. May 13, 1999.

10. DATA RETENTION

The raw data and final report for this study will be stored in the NWB Archive, 1121 East 3900 South, Salt Lake City, UT 84124 per regulations and contract agreement. 3M Environmental Technology and Services will be notified concerning final disposition of records at completion of contract obligations.

Table 1. Summary of Calibration Curve Parameters for PFOS in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
03-Jul-2000	23	-0.000004	0.019854	0.112640	0.9862	48.1	547
21-Aug-2000	18	0.000001	0.028989	0.257857	0.9849	48.1	547

A, B, and C are coefficients used to define the calibration curve.

Table 2. Summary of Calibration Curve Parameters for PFOSA in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
03-Jul-2000	23	-0.000017	0.054058	0.006149	0.9952	1.00	502
21-Aug-2000	18	0.000005	0.082782	0.019970	0.9952	1.00	502

A, B, and C are coefficients used to define the calibration curve.

Table 3. Summary of Calibration Curve Parameters for PFOSAA in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
03-Jul-2000	23	-0.000002	0.013656	-0.020557	0.9903	6.00	505
21-Aug-2000	18	0.000005	0.025511	-0.020269	0.9978	6.00	505

A, B, and C are coefficients used to define the calibration curve.

Table 4. Summary of Calibration Curve Parameters for POAA in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
03-Jul-2000	23	-0.000002	0.013683	0.010580	0.9979	5.76	505
21-Aug-2000	18	0.000003	0.013704	0.017950	0.9966	5.76	505

A, B, and C are coefficients used to define the calibration curve.

Table 5. Summary of Calibration Curve Parameters for PFHS in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
03-Jul-2000	23	-0.000006	0.018323	0.012882	0.9971	3.15	502
21-Aug-2000	18	-0.000001	0.022486	0.017181	0.9970	3.15	502

A, B, and C are coefficients used to define the calibration curve.

Table 6. Summary of Calibration Curve Parameters for M556 in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
03-Jul-2000	23	0.000000	0.002714	0.000585	0.9925	4.30	502
21-Aug-2000	18	0.000001	0.011156	0.005643	0.9958	4.30	502

A, B, and C are coefficients used to define the calibration curve.

Table 7. Summary of Calibration Curve Parameters for M570 in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
03-Jul-2000	23	0.000000	0.003818	-0.004160	0.9963	5.60	505
21-Aug-2000	18	0.000005	0.036404	-0.017720	0.9977	5.60	505

A, B, and C are coefficients used to define the calibration curve.

Table 8. Back-Calculated Concentrations of Calibration Standards for PFOS in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	48.1	49.6	57.1	72.1	97.1	147	297	447	547
03-Jul-2000	23	50.7	52.1	64.0	77.3	98.4	158	309	472	564
		45.8	45.6	53.4	68.2	86.6	138	298	435	508
21-Aug-2000	18	51.7	54.3	60.3	80.5	101	152	304	489	566
		44.1	45.6	50.5	70.5	91.1	139	286	417	521

Table 9. Back-Calculated Concentrations of Calibration Standards for PFOSA in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.51	10.1	25.1	50.3	100	251	402	502
03-Jul-2000	23	1.10	2.54	10.6	27.3	51.6	103	259	419	534
		0.918	2.38	9.36	24.6	46.2	95.4	265	398	436
21-Aug-2000	18	1.01	2.73	10.4	27.9	52.7	106	253	416	530
		0.990	2.30	9.62	25.1	44.7	95.8	229	378	502

Table 10. Back-Calculated Concentrations of Calibration Standards for PFOSAA in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	6.00	7.50	15.0	30.0	55.0	105	255	405	505
03-Jul-2000	23	6.74	7.73	16.6	31.3	57.6	111	278	446	524
		5.49	6.93	13.6	27.3	52.0	99.6	249	382	451
21-Aug-2000	18	5.83	7.71	14.5	29.8	53.7	99.3	239	397	499
		6.13	7.50	14.4	32.2	57.0	108	268	414	513

Table 11. Back-Calculated Concentrations of Calibration Standards for POAA in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	5.76	7.26	14.8	29.8	54.8	105	255	405	505
03-Jul-2000	23	5.93	7.11	15.2	30.6	52.0	109	265	406	524
		5.74	6.93	15.6	29.4	53.1	99.6	261	404	474
21-Aug-2000	18	5.98	7.19	13.9	28.2	52.8	102	256	401	481
		6.06	6.58	15.2	32.5	55.2	108	270	412	511

Table 12. Back-Calculated Concentrations of Calibration Standards for PFHS in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	3.15	4.65	12.2	27.2	52.2	102	252	402	502
03-Jul-2000	23	3.13	4.45	12.8	27.7	49.3	103	256	405	515
		3.20	4.55	13.7	26.3	49.1	99.7	253	402	487
21-Aug-2000	18	2.85	4.72	11.8	28.1	48.8	98.3	245	408	497
		3.17	5.23	12.1	27.9	51.9	105	258	404	502

Table 13. Back-Calculated Concentrations of Calibration Standards for M556 in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	4.30	11.8	26.8	51.8	102	252	402	502
03-Jul-2000	23	4.44	12.6	28.9	53.2	113	274	434	528
		4.18	11.1	23.8	47.5	95.1	247	381	448
21-Aug-2000	18	4.36	11.0	25.5	49.0	101	234	391	469
		4.35	11.8	27.2	55.4	106	284	415	516

Table 14. Back-Calculated Concentrations of Calibration Standards for M570 in Human Serum

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	5.60	7.10	14.6	29.6	54.6	105	255	405	505
03-Jul-2000	23	6.16	6.90	15.4	29.8	54.0	107	273	414	510
		5.54	6.43	14.3	28.7	52.6	104	265	404	464
21-Aug-2000	18	5.11	7.35	13.7	30.2	53.3	101	249	401	503
		5.84	7.43	14.4	31.1	54.5	106	269	406	505

Table 15. Quality Control Samples for PFOS in Human Serum

Mean of replicate QCs reported. All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 51.1 ppb	*%Dif.*	Medium QC 197 ppb	*%Dif.*	High QC 446 ppb	*%Dif.*
03-Jul-2000	23	47.7	*-6.65*	168	*-14.7*	382	*-14.3*
21-Aug-2000	18	46.7	*-8.61*	170	*-13.7*	378	*-15.2*

Table 16. Quality Control Samples for PFOSA in Human Serum

Mean of replicate QCs reported. All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	*%Dif.*	Medium QC 150 ppb	*%Dif.*	High QC 400 ppb	*%Dif.*
03-Jul-2000	23	2.73	*-31.8*	100	*-33.3*	286	*-28.5*
21-Aug-2000	18	4.34	*8.50*	150	*0.00*	395	*-1.25*

Table 17. Quality Control Samples for PFOSAA in Human Serum

Mean of replicate QCs reported. All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 9.00 ppb	*%Dif.*	Medium QC 155 ppb	*%Dif.*	High QC 405 ppb	*%Dif.*
03-Jul-2000	23	7.73	*-14.1*	110	*-29.0*	288	*-28.9*
21-Aug-2000	18	9.95	*10.6*	160	*3.23*	413	*1.98*

Table 18. Quality Control Samples for POAA in Human Serum

Mean of replicate QCs reported. All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 8.74 ppb	*%Dif.*	Medium QC 154 ppb	*%Dif.*	High QC 403 ppb	*%Dif.*
03-Jul-2000	23	8.45	*-3.32*	150	*-2.60*	400	*-0.744*
21-Aug-2000	18	7.82	*-10.5*	149	*-3.25*	372	*-7.69*

Table 19. Quality Control Samples for PFHS in Human Serum

Mean of replicate QCs reported. All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 6.15 ppb	*%Dif.*	Medium QC 152 ppb	*%Dif.*	High QC 402 ppb	*%Dif.*
03-Jul-2000	23	6.24	*1.46*	157	*3.29*	427	*6.22*
21-Aug-2000	18	6.86	*11.5*	164	*7.89*	403	*0.249*

Table 20. Quality Control Samples for M556 in Human Serum

Mean of replicate QCs reported. All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 5.80 ppb	*%Dif.*	Medium QC 152 ppb	*%Dif.*	High QC 402 ppb	*%Dif.*
03-Jul-2000	23	5.52	*-4.83*	145	*-4.61*	381	*-5.22*
21-Aug-2000	18	5.42	*-6.55*	141	*-7.24*	367	*-8.71*

Table 21. Quality Control Samples for M570 in Human Serum

Mean of replicate QCs reported. All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 8.60 ppb	*%Dif.*	Medium QC 155 ppb	*%Dif.*	High QC 405 ppb	*%Dif.*
03-Jul-2000	23	8.20	*-4.65*	146	*-5.81*	390	*-3.70*
21-Aug-2000	18	8.02	*-6.74*	149	*-3.87*	386	*-4.69*

Table 22. Freeze-Thaw and Room Temperature Matrix Stability for PFOS in Human Serum

All concentrations are expressed as ppb.

Run Number	18
Run Date	21-Aug-2000
	LOW QC
	After 7 Freeze-Thaw cycles
	46.8
	45.1
	44.0
Mean	45.3
S.D.	1.41
% CV	3.1
Control (ng/mL)	46.7
Mean % Dev. from Control	-3.0
	MEDIUM QC
	After 7 Freeze-Thaw cycles
	170
	158
	164
Mean	164
S.D.	6.00
% CV	3.7
Control (ng/mL)	170
Mean % Dev. from Control	-3.5
	HIGH QC
	After 7 Freeze-Thaw cycles
	369
	376
	361
Mean	369
S.D.	7.51
% CV	2.0
Control (ng/mL)	378
Mean % Dev. from Control	-2.4

Control = mean of analytical QC samples analyzed in the same run
NOTE: Samples were left at room temperature for up to 6.75 hour periods between each freeze cycle

Table 23. Freeze-Thaw and Room Temperature Matrix Stability for PFOSA in Human Serum

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	After 7 Freeze-Thaw cycles
	3.73
	3.53
	3.48
Mean	3.58
S.D.	0.132
% CV	3.7
Control (ng/mL)	4.34
Mean % Dev. from Control	-17.5
	MEDIUM QC
	After 7 Freeze-Thaw cycles
	108
	126
	125
Mean	120
S.D.	10.1
% CV	8.4
Control (ng/mL)	150
Mean % Dev. from Control	-20.0
	HIGH QC
	After 7 Freeze-Thaw cycles
	348
	361
	350
Mean	353
S.D.	7.00
% CV	2.0
Control (ng/mL)	395
Mean % Dev. from Control	-10.6

Control = mean of analytical QC samples analyzed in the same run
NOTE: Samples were left at room temperature for up to 6.75 hour periods between each freeze cycle

Table 24. Freeze-Thaw Stability and Room Temperature Matrix for PFOSAA in Human Serum

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	After 7 Freeze-Thaw cycles
	9.08
	9.85
	9.46
Mean	9.46
S.D.	0.385
% CV	4.1
Control (ng/mL)	9.95
Mean % Dev. from Control	-4.9
	MEDIUM QC
	After 7 Freeze-Thaw cycles
	114
	147
	147
Mean	136
S.D.	19.1
% CV	14.0
Control (ng/mL)	160
Mean % Dev. from Control	-15.0
	HIGH QC
	After 7 Freeze-Thaw cycles
	383
	403
	391
Mean	392
S.D.	10.1
% CV	2.6
Control (ng/mL)	413
Mean % Dev. from Control	-5.1

Control = mean of analytical QC samples analyzed in the same run
NOTE: Samples were left at room temperature for up to 6.75 hour periods between each freeze cycle

Table 25. Freeze-Thaw and Room Temperature Matrix Stability for POAA in Human Serum

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	After 7 Freeze-Thaw cycles
	8.06
	8.83
	8.25
Mean	8.38
S.D.	0.401
% CV	4.8
Control (ng/mL)	7.82
Mean % Dev. from Control	7.2
	MEDIUM QC
	After 7 Freeze-Thaw cycles
	151
	149
	154
Mean	151
S.D.	2.52
% CV	1.7
Control (ng/mL)	149
Mean % Dev. from Control	1.3
	HIGH QC
	After 7 Freeze-Thaw cycles
	364
	379
	372
Mean	372
S.D.	7.51
% CV	2.0
Control (ng/mL)	372
Mean % Dev. from Control	0.0

Control = mean of analytical QC samples analyzed in the same run
NOTE: Samples were left at room temperature for up to 6.75 hour periods between each freeze cycle

Table 26. Freeze-Thaw and Room Temperature Matrix Stability for PFHS in Human Serum

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	After 7 Freeze-Thaw cycles
	6.05
	6.27
	7.12
Mean	6.48
S.D.	0.565
% CV	8.7
Control (ng/mL)	6.86
Mean % Dev. from Control	-5.5
	MEDIUM QC
	After 7 Freeze-Thaw cycles
	165
	157
	163
Mean	162
S.D.	4.16
% CV	2.6
Control (ng/mL)	164
Mean % Dev. from Control	-1.2
	HIGH QC
	After 7 Freeze-Thaw cycles
	397
	417
	405
Mean	406
S.D.	10.1
% CV	2.5
Control (ng/mL)	403
Mean % Dev. from Control	0.7

Control = mean of analytical QC samples analyzed in the same run
NOTE: Samples were left at room temperature for up to 6.75 hour periods between each freeze cycle

Table 27. Freeze-Thaw and Room Temperature Matrix Stability for M556 in Human Serum

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	After 7 Freeze-Thaw cycles
	4.95
	5.07
	5.09
Mean	5.04
S.D.	0.0757
% CV	1.5
Control (ng/mL)	5.42
Mean % Dev. from Control	-7.0
	MEDIUM QC
	After 7 Freeze-Thaw cycles
	128
	140
	135
Mean	134
S.D.	6.03
% CV	4.5
Control (ng/mL)	141
Mean % Dev. from Control	-5.0
	HIGH QC
	After 7 Freeze-Thaw cycles
	359
	372
	374
Mean	368
S.D.	8.14
% CV	2.2
Control (ng/mL)	367
Mean % Dev. from Control	0.3

Control = mean of analytical QC samples analyzed in the same run
NOTE: Samples were left at room temperature for up to 6.75 hour periods between each freeze cycle

Table 28. Freeze-Thaw and Room Temperature Matrix Stability for M570 in Human Serum

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	After 7 Freeze-Thaw cycles
	7.68
	8.76
	8.51
Mean	8.32
S.D.	0.565
% CV	6.8
Control (ng/mL)	8.02
Mean % Dev. from Control	3.7
	MEDIUM QC
	After 7 Freeze-Thaw cycles
	142
	142
	143
Mean	142
S.D.	0.577
% CV	0.4
Control (ng/mL)	149
Mean % Dev. from Control	-4.7
	HIGH QC
	After 7 Freeze-Thaw cycles
	356
	371
	370
Mean	366
S.D.	8.39
% CV	2.3
Control (ng/mL)	386
Mean % Dev. from Control	-5.2

Control = mean of analytical QC samples analyzed in the same run
NOTE: Samples were left at room temperature for up to 6.75 hour periods between each freeze cycle

Table 29. Reinjection Stability for PFOS

All concentrations are expressed as ppb.

Run Number	**23**
Run Date	**3-Jul-2000**
	LOW QC
	Reinjection
	(After 2.5 Days Room Temp.)
	49.1
	48.0
	50.0
	46.1
	45.1
Mean	47.7
S.D.	2.04
% CV	4.3
Control (ng/mL)	47.2
Mean % Dev. from Control	**1.1**
	MEDIUM QC
	Reinjection
	(After 2.5 Days Room Temp.)
	164
	172
	170
	164
	170
Mean	168
S.D.	3.74
% CV	2.2
Control (ng/mL)	173
Mean % Dev. from Control	**-2.9**
	HIGH QC
	Reinjection
	(After 2.5 Days Room Temp.)
	391
	369
	368
	372
	411
Mean	382
S.D.	18.6
% CV	4.9
Control (ng/mL)	393
Mean % Dev. from Control	**-2.8**

Control = mean of analytical QC samples analyzed in run 15 (original injection)

Table 30. Reinjection Stability for PFOSA

All concentrations are expressed as ppb.

Run Number	**23**
Run Date	**3-Jul-2000**
	LOW QC
	Reinjection
	(After 2.5 Days Room Temp.)
	2.79
	2.69
	2.85
	2.66
	2.68
Mean	2.73
S.D.	0.0820
% CV	3.0
Control (ng/mL)	2.82
Mean % Dev. from Control	-3.2
	MEDIUM QC
	Reinjection
	(After 2.5 Days Room Temp.)
	102
	102
	97.9
	94.4
	105
Mean	100
S.D.	4.14
% CV	4.1
Control (ng/mL)	103
Mean % Dev. from Control	-2.9
	HIGH QC
	Reinjection
	(After 2.5 Days Room Temp.)
	275
	273
	290
	284
	307
Mean	286
S.D.	13.7
% CV	4.8
Control (ng/mL)	300
Mean % Dev. from Control	-4.7

Control = mean of analytical QC samples analyzed in run 15 (original injection)

Table 31. Reinjection Stability for PFOSAA

All concentrations are expressed as ppb.

Run Number	**23**
Run Date	**3-Jul-2000**
	LOW QC *Reinjection* *(After 2.5 Days Room Temp.)*
	7.78
	7.58
	8.15
	7.41
	7.74
Mean	7.73
S.D.	0.276
% CV	3.6
Control (ng/mL)	7.59
Mean % Dev. from Control	**1.8**
	MEDIUM QC *Reinjection* *(After 2.5 Days Room Temp.)*
	109
	112
	113
	104
	110
Mean	110
S.D.	3.51
% CV	3.2
Control (ng/mL)	113
Mean % Dev. from Control	**-2.7**
	HIGH QC *Reinjection* *(After 2.5 Days Room Temp.)*
	287
	285
	284
	278
	305
Mean	288
S.D.	10.2
% CV	3.5
Control (ng/mL)	301
Mean % Dev. from Control	**-4.3**

Control = mean of analytical QC samples analyzed in run 15 (original injection)

Table 32. Reinjection Stability for POAA

All concentrations are expressed as ppb.

Run Number	**23**
Run Date	**3-Jul-2000**
	LOW QC *Reinjection (After 2.5 Days Room Temp.)*
	8.68
	8.43
	8.31
	8.41
	8.41
Mean	8.45
S.D.	0.138
% CV	1.6
Control (ng/mL)	8.10
Mean % Dev. from Control	4.3
	MEDIUM QC *Reinjection (After 2.5 Days Room Temp.)*
	150
	152
	156
	144
	147
Mean	150
S.D.	4.60
% CV	3.1
Control (ng/mL)	144
Mean % Dev. from Control	4.2
	HIGH QC *Reinjection (After 2.5 Days Room Temp.)*
	417
	408
	386
	388
	401
Mean	400
S.D.	13.2
% CV	3.3
Control (ng/mL)	384
Mean % Dev. from Control	4.2

Control = mean of analytical QC samples analyzed in run 15 (original injection)

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Table 33. Reinjection Stability for PFHS

All concentrations are expressed as ppb.

Run Number	**23**
Run Date	**3-Jul-2000**
	LOW QC
	Reinjection
	(After 2.5 Days Room Temp.)
	6.30
	6.32
	6.48
	5.98
	6.11
Mean	6.24
S.D.	0.195
% CV	3.1
Control (ng/mL)	6.22
Mean % Dev. from Control	**0.3**
	MEDIUM QC
	Reinjection
	(After 2.5 Days Room Temp.)
	153
	156
	156
	159
	159
Mean	157
S.D.	2.51
% CV	1.6
Control (ng/mL)	158
Mean % Dev. from Control	**-0.6**
	HIGH QC
	Reinjection
	(After 2.5 Days Room Temp.)
	441
	416
	421
	409
	450
Mean	427
S.D.	17.4
% CV	4.1
Control (ng/mL)	445
Mean % Dev. from Control	**-4.0**

Control = mean of analytical QC samples analyzed in run 15 (original injection)

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Table 34. Reinjection Stability for M556

All concentrations are expressed as ppb.

Run Number	**23**
Run Date	**3-Jul-2000**
	LOW QC *Reinjection (After 2.5 Days Room Temp.)*
	5.78
	5.73
	5.45
	5.29
	5.33
Mean	5.52
S.D.	0.227
% CV	4.1
Control (ng/mL)	5.61
Mean % Dev. from Control	-1.6
	MEDIUM QC *Reinjection (After 2.5 Days Room Temp.)*
	144
	147
	147
	143
	146
Mean	145
S.D.	1.82
% CV	1.3
Control (ng/mL)	147
Mean % Dev. from Control	-1.4
	HIGH QC *Reinjection (After 2.5 Days Room Temp.)*
	388
	373
	381
	367
	398
Mean	381
S.D.	12.2
% CV	3.2
Control (ng/mL)	396
Mean % Dev. from Control	-3.8

Control = mean of analytical QC samples analyzed in run 15 (original injection)

Table 35. Reinjection Stability for M570

All concentrations are expressed as ppb.

Run Number	**23**
Run Date	**3-Jul-2000**
	LOW QC
	Reinjection
	(After 2.5 Days Room Temp.)
	8.31
	8.36
	8.52
	8.10
	7.71
Mean	8.20
S.D.	0.312
% CV	3.8
Control (ng/mL)	8.31
Mean % Dev. from Control	-1.3
	MEDIUM QC
	Reinjection
	(After 2.5 Days Room Temp.)
	145
	149
	146
	144
	145
Mean	146
S.D.	1.92
% CV	1.3
Control (ng/mL)	147
Mean % Dev. from Control	-0.7
	HIGH QC
	Reinjection
	(After 2.5 Days Room Temp.)
	391
	384
	390
	380
	405
Mean	390
S.D.	9.51
% CV	2.4
Control (ng/mL)	401
Mean % Dev. from Control	-2.7

Control = mean of analytical QC samples analyzed in run 15 (original injection)

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Table 36. Room Temperature Extract Stability for PFOS

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC *After 7 Days*
	51.7
	46.0
	46.2
Mean	48.0
S.D.	3.23
% CV	6.7
Control (ng/mL)	46.7
Mean % Dev. from Control	**2.8**
	HIGH QC *After 7 Days*
	373
	402
	397
Mean	391
S.D.	15.5
% CV	4.0
Control (ng/mL)	378
Mean % Dev. from Control	**3.4**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 37. Room Temperature Extract Stability for PFOSA

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC *After 7 Days*
	3.48
	3.34
	3.30
Mean	3.37
S.D.	0.0945
% CV	2.8
Control (ng/mL)	3.13
Mean % Dev. from Control	**7.7**
	HIGH QC *After 7 Days*
	337
	362
	334
Mean	344
S.D.	15.4
% CV	4.5
Control (ng/mL)	322
Mean % Dev. from Control	**6.8**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 38. Room Temperature Extract Stability for PFOSAA

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC *After 7 Days*
	8.2
	9.2
	9.3
Mean	8.89
S.D.	0.607
% CV	6.8
Control (ng/mL)	8.02
Mean % Dev. from Control	**10.8**
	HIGH QC *After 7 Days*
	321
	337
	343
Mean	334
S.D.	11.4
% CV	3.4
Control (ng/mL)	297
Mean % Dev. from Control	**12.5**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 39. Room Temperature Extract Stability for POAA

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC *After 7 Days*
	6.92
	7.98
	8.15
Mean	7.68
S.D.	0.667
% CV	8.7
Control (ng/mL)	7.82
Mean % Dev. from Control	**-1.8**
	HIGH QC *After 7 Days*
	360
	395
	378
Mean	378
S.D.	17.5
% CV	4.6
Control (ng/mL)	372
Mean % Dev. from Control	**1.6**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 40. Room Temperature Extract Stability for PFHS

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC *After 7 Days*
	6.31
	6.21
	5.90
Mean	6.14
S.D.	0.214
% CV	3.5
Control (ng/mL)	6.86
Mean % Dev. from Control	**-10.5**
	HIGH QC *After 7 Days*
	396
	405
	412
Mean	404
S.D.	8.02
% CV	2.0
Control (ng/mL)	403
Mean % Dev. from Control	**0.2**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 41. Room Temperature Extract Stability for M556

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC *After 7 Days*
	5.13
	5.65
	5.50
Mean	5.43
S.D.	0.268
% CV	4.9
Control (ng/mL)	5.42
Mean % Dev. from Control	**0.2**
	HIGH QC *After 7 Days*
	373
	376
	387
Mean	379
S.D.	7.37
% CV	1.9
Control (ng/mL)	367
Mean % Dev. from Control	**3.3**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 42. Room Temperature Extract Stability for M570

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC *After 7 Days*
	8.86
	9.13
	8.74
Mean	8.91
S.D.	0.200
% CV	2.2
Control (ng/mL)	8.02
Mean % Dev. from Control	**11.1**
	HIGH QC *After 7 Days*
	409
	429
	423
Mean	420
S.D.	10.3
% CV	2.5
Control (ng/mL)	386
Mean % Dev. from Control	**8.8**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 43. Reduced Temperature (1 to 8 °C) Extract Stability for PFOS

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC **1 to 8°C** ***After 4 Days***
	49.3
	45.6
	44.8
Mean	46.6
S.D.	2.40
% CV	5.2
Control (ng/mL)	46.7
Mean % Dev. from Control	-0.2
	HIGH QC **1 to 8°C** ***After 4 Days***
	391
	385
	377
Mean	384
S.D.	7.02
% CV	1.8
Control (ng/mL)	378
Mean % Dev. from Control	**1.6**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 44. Reduced Temperature (1 to 8 °C) Extract Stability for PFOSA

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC **1 to 8°C** ***After 4 Days***
	3.22
	2.79
	2.86
Mean	2.96
S.D.	0.231
% CV	7.8
Control (ng/mL)	3.13
Mean % Dev. from Control	**-5.4**
	HIGH QC **1 to 8°C** ***After 4 Days***
	350
	297
	304
Mean	317
S.D.	28.8
% CV	9.1
Control (ng/mL)	322
Mean % Dev. from Control	**-1.6**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 45. Reduced Temperature (1 to 8 °C) Extract Stability for PFOSAA

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC **1 to 8°C** ***After 4 Days***
	7.78
	8.55
	8.32
Mean	8.22
S.D.	0.395
% CV	4.8
Control (ng/mL)	8.02
Mean % Dev. from Control	**2.5**
	HIGH QC **1 to 8°C** ***After 4 Days***
	312
	306
	311
Mean	310
S.D.	3.21
% CV	1.0
Control (ng/mL)	297
Mean % Dev. from Control	**4.4**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 46. Reduced Temperature (1 to 8 °C) Extract Stability for POAA

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC **1 to 8°C** ***After 4 Days***
	7.78
	8.75
	8.39
Mean	8.31
S.D.	0.490
% CV	5.9
Control (ng/mL)	7.82
Mean % Dev. from Control	**6.3**
	HIGH QC **1 to 8°C** ***After 4 Days***
	381
	394
	385
Mean	387
S.D.	6.66
% CV	1.7
Control (ng/mL)	372
Mean % Dev. from Control	**4.0**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 47. Reduced Temperature (1 to 8 °C) Extract Stability for PFHS

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC **1 to 8°C** ***After 4 Days***
	6.76
	6.34
	7.74
Mean	6.95
S.D.	0.718
% CV	10.3
Control (ng/mL)	6.86
Mean % Dev. from Control	**1.3**
	HIGH QC **1 to 8°C** ***After 4 Days***
	400
	411
	423
Mean	411
S.D.	11.5
% CV	2.8
Control (ng/mL)	403
Mean % Dev. from Control	**2.0**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 48. Reduced Temperature (1 to 8 °C) Extract Stability for M556

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC **1 to 8°C** ***After 4 Days***
	5.47
	6.57
	6.82
Mean	6.29
S.D.	0.718
% CV	11.4
Control (ng/mL)	5.42
Mean % Dev. from Control	**16.1**
	HIGH QC **1 to 8°C** ***After 4 Days***
	398
	404
	403
Mean	402
S.D.	3.21
% CV	0.8
Control (ng/mL)	367
Mean % Dev. from Control	**9.5**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 49. Reduced Temperature (1 to 8 °C) Extract Stability for M570

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC **1 to 8°C** ***After 4 Days***
	7.96
	7.90
	9.21
Mean	8.36
S.D.	0.740
% CV	8.9
Control (ng/mL)	8.02
Mean % Dev. from Control	**4.2**
	HIGH QC **1 to 8°C** ***After 4 Days***
	396
	394
	402
Mean	397
S.D.	4.16
% CV	1.0
Control (ng/mL)	386
Mean % Dev. from Control	**2.8**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 50. Reduced Temperature (-20 °C) Extract Stability for PFOS

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	-20°C
	After 5 Days
	47.2
	45.5
	40.9
Mean	44.5
S.D.	3.26
% CV	7.3
Control (ng/mL)	46.7
Mean % Dev. from Control	**-4.7**
	HIGH QC
	-20°C
	After 5 Days
	376
	373
	365
Mean	371
S.D.	5.69
% CV	1.5
Control (ng/mL)	378
Mean % Dev. from Control	**-1.9**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 51. Reduced Temperature (-20 °C) Extract Stability for PFOSA

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	-20°C
	After 5 Days
	2.95
	3.16
	3.14
Mean	3.08
S.D.	0.116
% CV	3.8
Control (ng/mL)	3.13
Mean % Dev. from Control	**-1.6**
	HIGH QC
	-20°C
	After 5 Days
	312
	312
	296
Mean	307
S.D.	9.24
% CV	3.0
Control (ng/mL)	322
Mean % Dev. from Control	**-4.7**

Control = mean of QC samples analyzed in the same run immediately following extraction

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Table 52. Reduced Temperature (-20 °C) Extract Stability for PFOSAA

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	-20°C
	After 5 Days
	7.52
	8.32
	7.89
Mean	7.91
S.D.	0.400
% CV	5.1
Control (ng/mL)	8.02
Mean % Dev. from Control	-1.4
	HIGH QC
	-20°C
	After 5 Days
	294
	306
	287
Mean	296
S.D.	9.61
% CV	3.2
Control (ng/mL)	297
Mean % Dev. from Control	-0.3

Control = mean of QC samples analyzed in the same run immediately following extraction

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Table 53. Reduced Temperature (-20 °C) Extract Stability for POAA

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	-20°C
	After 5 Days
	7.84
	10.2
	6.85
Mean	8.30
S.D.	1.72
% CV	20.7
Control (ng/mL)	7.82
Mean % Dev. from Control	**6.1**
	HIGH QC
	-20°C
	After 5 Days
	351
	373
	386
Mean	370
S.D.	17.7
% CV	4.8
Control (ng/mL)	372
Mean % Dev. from Control	**-0.5**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 54. Reduced Temperature (-20 °C) Extract Stability for PFHS

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC -20°C *After 5 Days*
	6.68
	6.06
	7.70
Mean	6.81
S.D.	0.828
% CV	12.2
Control (ng/mL)	6.86
Mean % Dev. from Control	**-0.7**
	HIGH QC -20°C *After 5 Days*
	403
	407
	417
Mean	409
S.D.	7.21
% CV	1.8
Control (ng/mL)	403
Mean % Dev. from Control	**1.5**

Control = mean of QC samples analyzed in the same run immediately following extraction

Table 55. Reduced Temperature (-20 °C) Extract Stability for M556

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	-20°C
	After 5 Days
	4.51
	5.69
	5.97
Mean	5.39
S.D.	0.775
% CV	14.4
Control (ng/mL)	5.42
Mean % Dev. from Control	**-0.6**
	HIGH QC
	-20°C
	After 5 Days
	358
	373
	378
Mean	370
S.D.	10.4
% CV	2.8
Control (ng/mL)	367
Mean % Dev. from Control	**0.8**

Control = mean of QC samples analyzed in the same run immediately following extraction

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Table 56. Reduced Temperature (-20 °C) Extract Stability for M570

All concentrations are expressed as ppb.

Run Number	**18**
Run Date	**21-Aug-2000**
	LOW QC
	-20°C
	After 5 Days
	8.43
	8.52
	7.94
Mean	8.30
S.D.	0.312
% CV	3.8
Control (ng/mL)	8.02
Mean % Dev. from Control	**3.5**
	HIGH QC
	-20°C
	After 5 Days
	366
	388
	369
Mean	374
S.D.	11.9
% CV	3.2
Control (ng/mL)	386
Mean % Dev. from Control	**-3.1**

Control = mean of QC samples analyzed in the same run immediately following extraction

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Table 57. Long-Term Matrix Stability for PFOS in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 51.1 ppb 55 Days	Medium QC 197 ppb 55 Days	High QC 446 ppb 55 Days
21-Aug-2000	18	52.2	175	381
		50.1	171	391
		42.3	174	361
		43.8	166	371
		45.0	165	388
Mean		46.7	170	378
SD		4.26	4.55	12.4
%CV		9.1	2.7	3.3
%Bias		-8.6	-13.7	-15.2
N		5	5	5

Table 58. Long-Term Matrix Stability for PFOSA in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb 42 Days	Medium QC 150 ppb 42 Days	High QC 400 ppb 42 Days
21-Aug-2000	18	4.28	155	376
		4.56	158	420
		4.40	154	381
		4.12	142	397
		4.32	143	400
Mean		4.34	150	395
SD		0.161	7.37	17.4
%CV		3.7	4.9	4.4
%Bias		8.5	0.0	-1.3
N		5	5	5

Table 59. Long-Term Matrix Stability for PFOSAA in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 9.00 ppb 42 Days	Medium QC 155 ppb 42 Days	High QC 405 ppb 42 Days
21-Aug-2000	18	9.64	157	403
		9.11	158	429
		10.5	164	406
		10.2	159	413
		10.3	160	416
Mean		9.95	160	413
SD		0.568	2.70	10.2
%CV		5.7	1.7	2.5
%Bias		10.6	3.2	2.0
N		5	5	5

Table 60. Long-Term Matrix Stability for POAA in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 8.74 ppb 55 Days	Medium QC 154 ppb 55 Days	High QC 403 ppb 55 Days
21-Aug-2000	18	7.74	145	364
		7.54	138	380
		7.72	155	354
		8.05	151	371
		8.03	157	389
Mean		7.82	149	372
SD		0.219	7.76	13.6
%CV		2.8	5.2	3.7
%Bias		-10.5	-3.2	-7.7
N		5	5	5

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Table 61. Long-Term Matrix Stability for PFHS in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 6.15 ppb 55 Days	Medium QC 152 ppb 55 Days	High QC 402 ppb 55 Days
21-Aug-2000	18	7.01	163	387
		7.06	158	414
		7.10	169	375
		6.57	161	404
		6.56	168	435
Mean		6.86	164	403
SD		0.271	4.66	23.4
%CV		4.0	2.8	5.8
%Bias		11.5	7.9	0.2
N		5	5	5

Table 62. Long-Term Matrix Stability for M556 in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 5.80 ppb 55 Days	Medium QC 152 ppb 55 Days	High QC 402 ppb 55 Days
21-Aug-2000	18	6.07	141	355
		5.67	137	382
		*4.60	149	340
		4.97	137	362
		5.79	143	394
Mean		5.42	141	367
SD		0.612	4.98	21.5
%CV		11.3	3.5	5.9
%Bias		-6.6	-7.2	-8.7
N		5	5	5

* > ± 20% deviation from theoretical

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Table 63. Long-Term Matrix Stability for M570 in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 8.60 ppb 55 Days	Medium QC 155 ppb 55 Days	High QC 405 ppb 55 Days
21-Aug-2000	18	7.93	147	373
		8.07	147	391
		7.87	158	373
		8.04	144	384
		8.20	150	408
Mean		8.02	149	386
SD		0.128	5.36	14.6
%CV		1.6	3.6	3.8
%Bias		-6.7	-3.9	-4.7
N		5	5	5

Table 64. PFOS Extraction Efficiency from Human Serum

Run 22

Low Concentration = 4.00 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.0679	14.7
		0.0683	14.6
		0.0735	13.6
	Mean	0.0699	14.3
2. Analyte prior and IS after extraction		0.0656	
		0.0605	
		0.0649	
	Mean	0.0637	
3. IS prior and Analyte after extraction			0.775
			0.726
			0.805
	Mean		0.769

Adjusted mean extraction efficiency = 44.4%
Mean extraction efficiency for the internal standard = 5.38%

High Concentration = 400 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		1.65	0.605
		1.55	0.643
		1.65	0.607
	Mean	1.62	0.618
2. Analyte prior and IS after extraction		0.522	
		0.538	
		0.518	
	Mean	0.526	
3. IS prior and Analyte after extraction			0.0396
			0.0379
			0.0401
	Mean		0.0392

Adjusted mean extraction efficiency = 30.1%
Mean extraction efficiency for the internal standard = 6.34%

Overall mean extraction efficiency for analyte = 37.3%
Overall mean extraction efficiency for internal standard = 5.86%

* Due to rounding, calculations based on displayed values may differ from actual.

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Table 65. PFOSA Extraction Efficiency from Human Serum

Run 22

Low Concentration = 4.00 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.0275	36.3
		0.0222	45.0
		0.0214	46.8
	Mean	0.0237	42.7
2. Analyte prior and IS after extraction		0.0171	
		0.0147	
		0.0172	
	Mean	0.0163	
3. IS prior and Analyte after extraction			2.47
			2.25
			2.45
	Mean		2.39

Mean extraction efficiency for the internal standard = 5.60%

High Concentration = 400 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		2.58	0.387
		2.47	0.405
		2.65	0.377
	Mean	2.57	0.390
2. Analyte prior and IS after extraction		1.80	
		1.87	
		1.75	
	Mean	1.81	
3. IS prior and Analyte after extraction			0.0243
			0.0223
			0.0233
	Mean		0.0233

Mean extraction efficiency for the analyte = 70.4%

Overall mean extraction efficiency for analyte = 69.6%
Overall mean extraction efficiency for internal standard = 5.79%

* Due to rounding, calculations based on displayed values may differ from actual.

030282

Table 66. PFOSAA Extraction Efficiency from Human Serum

Run 22

Low Concentration = 4.00 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.0246	40.6
		0.0257	39.0
		0.0238	42.0
	Mean	0.0247	40.5
2. Analyte prior and IS after extraction		0.0206	
		0.0186	
		0.0194	
	Mean	0.0195	
3. IS prior and Analyte after extraction			2.71
			2.45
			2.58
	Mean		2.58

Adjusted mean extraction efficiency = 62.6%
Mean extraction efficiency for the internal standard = 6.37%

High Concentration = 400 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		1.50	0.665
		1.44	0.693
		1.56	0.641
	Mean	1.50	0.666
2. Analyte prior and IS after extraction		0.962	
		0.959	
		0.917	
	Mean	0.946	
3. IS prior and Analyte after extraction			0.0419
			0.0394
			0.0413
	Mean		0.0409

Adjusted mean extraction efficiency = 62.7%
Mean extraction efficiency for the internal standard = 6.14%

Overall mean extraction efficiency for analyte = 62.7%
Overall mean extraction efficiency for internal standard = 6.26%

* Due to rounding, calculations based on displayed values may differ from actual.

030283

Table 67. POAA Extraction Efficiency from Human Serum

Run 22

Low Concentration = 4.00 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.0288	34.7
		0.0291	34.3
		0.0302	33.1
	Mean	0.0294	34.0
2. Analyte prior and IS after extraction		0.00934	
		0.00760	
		0.0101	
	Mean	0.00901	
3. IS prior and Analyte after extraction			2.02
			1.93
			2.05
	Mean		2.00

Adjusted mean extraction efficiency = 16.8%
Mean extraction efficiency for the internal standard = 5.88%

High Concentration = 400 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		2.61	0.383
		2.59	0.386
		2.72	0.367
	Mean	2.64	0.379
2. Analyte prior and IS after extraction		0.377	
		0.385	
		0.383	
	Mean	0.382	
3. IS prior and Analyte after extraction			0.0248
			0.0227
			0.0241
	Mean		0.0239

Adjusted mean extraction efficiency = 14.3%
Mean extraction efficiency for the internal standard = 6.31%

Overall mean extraction efficiency for analyte = 15.6%
Overall mean extraction efficiency for internal standard = 6.10%

* Due to rounding, calculations based on displayed values may differ from actual.

030284

Table 68. PFHS Extraction Efficiency from Human Serum

Run 22

Low Concentration = 4.00 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.0367	27.3
		0.0361	27.7
		0.0366	27.3
	Mean	0.0365	27.4
2. Analyte prior and IS after extraction		0.00870	
		0.00778	
		0.00878	
	Mean	0.00842	
3. IS prior and Analyte after extraction			1.72
			1.63
			1.71
	Mean		1.69

Adjusted mean extraction efficiency = 16.3%
Mean extraction efficiency for the internal standard = 6.17%

High Concentration = 400 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		3.49	0.286
		3.29	0.304
		3.58	0.279
	Mean	3.45	0.290
2. Analyte prior and IS after extraction		0.486	
		0.514	
		0.485	
	Mean	0.495	
3. IS prior and Analyte after extraction			0.0192
			0.0181
			0.0192
	Mean		0.0188

Adjusted mean extraction efficiency = 14.3%
Mean extraction efficiency for the internal standard = 6.48%

Overall mean extraction efficiency for analyte = 15.3%
Overall mean extraction efficiency for internal standard = 6.33%

* Due to rounding, calculations based on displayed values may differ from actual.

Table 69. M556 Extraction Efficiency from Human Serum

Run 22

Low Concentration = 4.00 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.00786	127
		0.00750	133
		0.00810	123
	Mean	0.00782	128
2. Analyte prior and IS after extraction		0.00421	
		0.00366	
		0.00310	
	Mean	0.00366	
3. IS prior and Analyte after extraction			7.54
			6.18
			6.78
	Mean		6.83

Mean extraction efficiency for the analyte = 46.8%
Adjusted mean extraction efficiency = 37.7%

High Concentration = 400 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.788	1.27
		0.758	1.32
		0.836	1.20
	Mean	0.794	1.26
2. Analyte prior and IS after extraction		0.337	
		0.326	
		0.303	
	Mean	0.322	
3. IS prior and Analyte after extraction			0.0753
			0.0690
			0.0709
	Mean		0.0717

Mean extraction efficiency for the analyte = 40.6%
Adjusted mean extraction efficiency = 40.4%

Overall mean extraction efficiency for analyte = 39.1%
Overall mean extraction efficiency for internal standard = 5.52%

* Due to rounding, calculations based on displayed values may differ from actual.

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Table 70. M570 Extraction Efficiency from Human Serum

Run 22

Low Concentration = 4.00 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		0.0333	30.1
		0.0317	31.5
		0.0335	29.9
	Mean	0.0328	30.5
2. Analyte prior and IS after extraction		0.0244	
		0.0220	
		0.0244	
	Mean	0.0236	
3. IS prior and Analyte after extraction			1.92
			1.72
			1.74
	Mean		1.79

Adjusted mean extraction efficiency = 54.4%
Mean extraction efficiency for the internal standard = 5.87%

High Concentration = 400 ng/mL

Time of Spiking		Ratio Analyte/IS	Ratio IS/Analyte
1. Analyte and IS after extraction		2.20	0.454
		2.16	0.463
		2.35	0.426
	Mean	2.24	0.448
2. Analyte prior and IS after extraction		1.34	
		1.37	
		1.32	
	Mean	1.34	
3. IS prior and Analyte after extraction			0.0294
			0.0270
			0.0280
	Mean		0.0281

Adjusted mean extraction efficiency = 59.7%
Mean extraction efficiency for the internal standard = 6.27%

Overall mean extraction efficiency for analyte = 57.1%
Overall mean extraction efficiency for internal standard = 6.07%

* Due to rounding, calculations based on displayed values may differ from actual.

Table 71. Summary of Calibration Curve Parameters for PFOS in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
12-Oct-2000	S00091-2	-0.000007	0.030663	-0.005620	0.9986	3.94	414
27-Oct-2000	PFOSMD-40	0.000000	0.028132	0.010586	0.9931	3.94	414

A, B, and C are coefficients used to define the calibration curve.

Table 72. Summary of Calibration Curve Parameters for PFOSA in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
12-Oct-2000	S00091-2	-0.000011	0.073810	0.006421	0.9970	1.00	500
27-Oct-2000	PFOSMD-40	0.000001	0.052188	0.010966	0.9912	1.00	500

A, B, and C are coefficients used to define the calibration curve.

Table 73. Summary of Calibration Curve Parameters for PFOSAA in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
12-Oct-2000	S00091-2	-0.000003	0.032601	-0.006279	0.9988	1.60	501
27-Oct-2000	PFOSMD-40	0.000005	0.021209	-0.002450	0.9873	1.60	501

A, B, and C are coefficients used to define the calibration curve.

Table 74. Summary of Calibration Curve Parameters for POAA in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
12-Oct-2000	S00091-2	0.000000	0.017245	0.001072	0.9985	1.92	481
27-Oct-2000	PFOSMD-40	0.000002	0.014346	0.002192	0.9918	1.92	481

A, B, and C are coefficients used to define the calibration curve.

Table 75. Summary of Calibration Curve Parameters for PFHS in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
12-Oct-2000	S00091-2	-0.000005	0.025818	-0.000970	0.9983	1.36	523
27-Oct-2000	PFOSMD-40	-0.000001	0.025534	0.011247	0.9953	1.36	523

A, B, and C are coefficients used to define the calibration curve.

Table 76. Summary of Calibration Curve Parameters for M556 in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
12-Oct-2000	S00091-2	-0.000002	0.018315	0.005561	0.9954	2.50	500
31-Oct-2000	24	-0.000001	0.018373	0.001923	0.9972	2.50	500

A, B, and C are coefficients used to define the calibration curve.

Table 77. Summary of Calibration Curve Parameters for M570 in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
12-Oct-2000	S00091-2	-0.000006	0.048518	0.005367	0.9978	1.00	500
27-Oct-2000	PFOSMD-40	0.000001	0.034031	0.011681	0.9910	1.00	500

A, B, and C are coefficients used to define the calibration curve.

Table 78. Back-Calculated Concentrations of Calibration Standards for PFOS in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	3.94	5.17	11.3	23.6	44.2	85.4	209	332	414
12-Oct-2000	S00091-2	4.00	5.01	11.2	25.0	42.9	83.7	203	335	390
		3.86	5.35	11.1	24.3	43.5	85.3	209	350	424
27-Oct-2000	PFOSMD-40	3.72	5.18	11.3	19.4	45.5	82.1	214	337	369
		3.90	5.67	11.7	24.1	45.9	84.4	221	367	407

Table 79. Back-Calculated Concentrations of Calibration Standards for PFOSA in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
12-Oct-2000	S00091-2	1.04	2.60	10.4	25.1	49.1	95.9	252	380	462
		0.941	2.53	8.93	25.7	50.5	102	255	444	508
27-Oct-2000	PFOSMD-40	0.961	2.59	9.81	20.2	53.4	103	259	356	443
		0.999	2.71	10.4	22.1	52.3	101	267	458	515

Table 80. Back-Calculated Concentrations of Calibration Standards for PFOSAA in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.60	3.10	10.6	25.6	50.6	101	251	401	501
12-Oct-2000	S00091-2	1.65	3.12	10.7	25.3	50.5	98.2	255	393	469
		1.54	3.10	10.4	26.3	51.0	101	245	432	513
27-Oct-2000	PFOSMD-40	1.66	3.50	11.7	21.3	53.1	104	299	406	440
		1.47	2.96	10.8	23.5	47.3	90.3	261	441	475

Table 81. Back-Calculated Concentrations of Calibration Standards for POAA in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.92	3.36	10.6	25.0	49.0	97.1	241	385	481
12-Oct-2000	S00091-2	2.00	3.24	10.5	26.1	49.3	97.5	241	386	477
		1.96	3.11	10.1	25.2	52.1	95.0	239	392	477
27-Oct-2000	PFOSMD-40	1.73	3.01	10.5	22.9	49.7	99.1	250	367	439
		2.30	3.12	10.9	25.1	50.7	96.7	262	422	474

Table 82. Back-Calculated Concentrations of Calibration Standards for PFHS in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.36	2.92	10.7	26.5	52.6	104	262	418	523
12-Oct-2000	S00091-2	1.28	2.74	10.6	27.5	50.2	104	253	441	518
		1.44	3.08	10.8	26.9	53.2	104	259	413	524
27-Oct-2000	PFOSMD-40	1.52	2.72	10.6	24.2	54.6	107	259	466	489
		1.28	2.78	10.4	28.4	54.1	102	270	425	491

Table 83. Back-Calculated Concentrations of Calibration Standards for M556 in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	2.50	10.0	25.0	50.0	100	250	400	500
12-Oct-2000	S00091-2	2.48	11.3	27.9	49.7	101	259	398	521
		2.47	9.43	24.5	43.7	96.7	245	384	499
31-Oct-2000	24	2.33	9.67	24.3	51.9	95.2	255	388	499
		2.70	9.81	25.8	49.3	104	249	438	470

Table 84. Back-Calculated Concentrations of Calibration Standards for M570 in Human Plasma

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
12-Oct-2000	S00091-2	0.991	2.57	10.2	25.0	50.8	97.0	250	380	451
		1.00	2.48	9.46	25.4	50.3	101	244	448	528
27-Oct-2000	PFOSMD-40	0.993	2.71	10.6	21.2	53.6	104	284	401	444
		0.951	2.65	10.4	20.9	48.6	93.5	254	443	481

Table 85. Lower Limit of Quantitation for PFOS in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	LLOQ (3.94 ppb)
27-Oct-2000	PFOS MD 40	3.98
		3.51
		3.89
		4.04
		4.06
		3.77
Mean		3.88
S.D.		0.208
%CV		5.4
%Theoretical		98.5
%Bias		-1.5
N		6

Table 86. Lower Limit of Quantitation for PFOSA in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	LLOQ (1.00 ppb)
27-Oct-2000	PFOS MD 40	1.08
		1.07
		1.11
		0.900
		1.01
		0.865
Mean		1.01
S.D.		0.102
%CV		10.1
%Theoretical		101.0
%Bias		1.0
N		6

Table 87. Lower Limit of Quantitation for PFOSAA in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	LLOQ (1.60 ppb)
27-Oct-2000	PFOS MD 40	1.63
		1.58
		1.54
		1.39
		1.63
		1.57
Mean		1.56
S.D.		0.0889
%CV		5.7
%Theoretical		97.5
%Bias		-2.5
N		6

030296

Table 88. Lower Limit of Quantitation for POAA in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	LLOQ (1.92 ppb)
27-Oct-2000	PFOS MD 40	1.72
		1.84
		1.80
		1.74
		1.79
		1.62
Mean		1.75
S.D.		0.0776
%CV		4.4
%Theoretical		91.1
%Bias		-8.9
N		6

Table 89. Lower Limit of Quantitation for PFHS in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	LLOQ (1.36 ppb)
27-Oct-2000	PFOS MD 40	1.23
		1.29
		1.36
		1.35
		*1.06
		1.42
Mean		1.29
S.D.		0.128
%CV		9.9
%Theoretical		94.9
%Bias		-5.1
N		6

* > ± 20% deviation from theoretical

Table 90. Lower Limit of Quantitation for M556 in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	LLOQ (2.50 ppb)
31-Oct-2000	24	2.44
		2.25
		2.50
		2.65
		2.50
		2.34
Mean		2.45
S.D.		0.139
%CV		5.7
%Theoretical		98.0
%Bias		-2.0
N		6

030299

Table 91. Lower Limit of Quantitation for M570 in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	LLOQ (1.00 ppb)
27-Oct-2000	PFOS MD 40	1.14
		1.02
		1.01
		1.01
		1.01
		0.997
Mean		1.03
S.D.		0.0538
%CV		5.2
%Theoretical		103.0
%Bias		3.0
N		6

Table 92. Intra-Assay Precision for PFOS Quality Controls in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 6.40 ppb	Medium QC 126 ppb	High QC 332 ppb
12-Oct-2000	S00091-2	7.33	132	343
		7.38	129	331
		*7.89	138	334
		7.07	132	334
		6.98	137	340
Mean		7.19	134	336
S.D.		0.195	3.78	4.93
%CV		2.7	2.8	1.5
%Theoretical		112.3	106.3	101.2
%Bias		12.3	6.3	1.2
n		4	5	5

* Sample deactivated due to unacceptable internal standard response.

Table 93. Intra-Assay Precision for PFOSA Quality Controls in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb
12-Oct-2000	S00091-2	4.69	148	423
		4.73	157	409
		*4.67	172	425
		4.53	166	414
		4.66	163	409
Mean		4.65	161	416
S.D.		0.0866	9.15	7.62
%CV		1.9	5.7	1.8
%Theoretical		116.3	107.3	104.0
%Bias		16.3	7.3	4.0
n		4	5	5

* Sample deactivated due to unacceptable internal standard response.

Table 94. Intra-Assay Precision for PFOSAA Quality Controls in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.60 ppb	Medium QC 151 ppb	High QC 401 ppb
12-Oct-2000	S00091-2	5.20	146	411
		5.50	148	423
		*7.43	169	431
		4.96	156	404
		4.89	155	425
Mean		5.14	155	419
S.D.		0.276	9.04	11.0
%CV		5.4	5.8	2.6
%Theoretical		111.7	102.6	104.5
%Bias		11.7	2.6	4.5
n		4	5	5

* Sample deactivated due to unacceptable internal standard response.

Table 95. Intra-Assay Precision for POAA Quality Controls in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.81 ppb	Medium QC 145 ppb	High QC 385 ppb
12-Oct-2000	S00091-2	5.33	144	400
		5.17	157	395
		*7.82	163	399
		4.86	155	394
		4.69	152	401
Mean		5.01	154	398
S.D.		0.290	6.98	3.11
%CV		5.8	4.5	0.8
%Theoretical		104.2	106.2	103.4
%Bias		4.2	6.2	3.4
n		4	5	5

* Sample deactivated due to unacceptable internal standard response.

090304

Table 96. Intra-Assay Precision for PFHS Quality Controls in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.48 ppb	Medium QC 156 ppb	High QC 417 ppb
12-Oct-2000	S00091-2	4.91	162	406
		4.80	158	388
		*5.41	162	399
		4.72	150	403
		4.58	158	401
Mean		4.75	158	399
S.D.		0.139	4.90	6.88
%CV		2.9	3.1	1.7
%Theoretical		106.0	101.3	95.7
%Bias		6.0	1.3	-4.3
n		4	5	5

* Sample deactivated due to unacceptable internal standard response.

Table 97. Intra-Assay Precision for M556 Quality Controls in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb
12-Oct-2000	S00091-2	4.65	147	441
		4.35	140	**492
		*4.76	150	459
		4.14	146	403
		3.89	159	431
Mean		4.26	148	445
S.D.		0.322	6.95	33.1
%CV		7.6	4.7	7.4
%Theoretical		106.5	98.7	111.3
%Bias		6.5	-1.3	11.3
n		4	5	5

* Sample deactivated due to unacceptable internal standard response.
** > ±20% deviation from theoretical

Table 98. Intra-Assay Precision for M570 Quality Controls in Human Plasma

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb
12-Oct-2000	S00091-2	**4.82	146	407
		4.75	161	407
		*5.89	170	414
		4.56	165	393
		4.74	158	403
Mean		4.72	160	405
S.D.		0.111	9.03	7.69
%CV		2.4	5.6	1.9
%Theoretical		118.0	106.7	101.3
%Bias		18.0	6.7	1.3
n		4	5	5

* Sample deactivated due to unacceptable internal standard response.
** > ±20% deviation from theoretical

Table 99. Summary of Calibration Curve Parameters for PFOS in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
23-Oct-2000	PFOSMD-39	-0.000001	0.009444	-0.004315	0.9954	5.30	504

A, B, and C are coefficients used to define the calibration curve.

Table 100. Summary of Calibration Curve Parameters for PFOSA in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
23-Oct-2000	PFOSMD-39	0.000000	0.014752	0.002265	0.9946	1.00	500

A, B, and C are coefficients used to define the calibration curve.

Table 101. Summary of Calibration Curve Parameters for PFOSAA in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
23-Oct-2000	PFOSMD-39	0.000002	0.009880	0.004993	0.9960	1.00	500

A, B, and C are coefficients used to define the calibration curve.

Table 102. Summary of Calibration Curve Parameters for POAA in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
23-Oct-2000	PFOSMD-39	-0.000001	0.011351	-0.000137	0.9967	1.70	501

A, B, and C are coefficients used to define the calibration curve.

Table 103. Summary of Calibration Curve Parameters for PFHS in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
23-Oct-2000	PFOSMD-39	-0.000003	0.018220	0.007453	0.9980	1.00	500

A, B, and C are coefficients used to define the calibration curve.

Table 104. Summary of Calibration Curve Parameters for M556 in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
23-Oct-2000	PFOSMD-39	0.000000	0.008729	-0.000146	0.9936	1.00	500

A, B, and C are coefficients used to define the calibration curve.

030309

Table 105. Summary of Calibration Curve Parameters for M570 in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
23-Oct-2000	PFOSMD-39	0.000000	0.015628	0.005711	0.9969	1.00	500

A, B, and C are coefficients used to define the calibration curve.

Table 106. Back-Calculated Concentrations of Calibration Standards for PFOS in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	5.30	6.80	14.3	29.3	54.3	104	254	404	504
23-Oct-2000	PFOSMD-39	5.32	6.18	14.0	25.6	52.3	102	264	410	476
		5.45	7.23	14.8	30.1	54.6	113	265	417	488

Table 107. Back-Calculated Concentrations of Calibration Standards for PFOSA in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
23-Oct-2000	PFOSMD-39	1.03	2.21	9.29	22.6	50.2	98.2	265	410	455
		1.03	2.45	10.1	25.8	50.6	112	271	423	468

Table 108. Back-Calculated Concentrations of Calibration Standards for PFOSAA in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
23-Oct-2000	PFOSMD-39	1.12	2.50	9.73	23.6	50.0	104	267	408	485
		0.901	2.41	9.41	24.1	50.6	111	254	399	479

Table 109. Back-Calculated Concentrations of Calibration Standards for POAA in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.70	3.20	10.7	25.7	50.7	101	254	401	501
23-Oct-2000	PFOSMD-39	1.56	3.22	10.4	24.7	50.9	99.8	260	401	458
		1.88	3.10	10.6	25.3	50.8	109	263	432	491

Table 110. Back-Calculated Concentrations of Calibration Standards for PFHS in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
23-Oct-2000	PFOSMD-39	1.01	2.40	10.1	24.2	49.6	99.3	254	424	450
		1.01	2.53	10.0	24.3	49.0	106	260	433	473

Table 111. Back-Calculated Concentrations of Calibration Standards for M556 in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	2.50	10.0	25.0	50.0	100	250	400	500
23-Oct-2000	PFOSMD-39	2.68	10.6	22.9	51.3	109	283	432	505
		2.36	9.33	22.9	48.6	102	242	379	451

Table 112. Back-Calculated Concentrations of Calibration Standards for M570 in Serum Diluted with Buffer

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
23-Oct-2000	PFOSMD-39	1.07	2.48	9.93	22.5	51.6	103	268	408	489
		0.964	2.34	9.59	24.6	50.8	110	254	393	476

Table 113. PFOS Quality Controls Samples Prepared in Serum Diluted with Buffer

Run Date	Run Number	193 ng/mL DF=10	443 ng/mL DF=10	4040 ng/mL DF=10	4040 ng/mL DF=50
23-Oct-2000	PFOSMD-39	204	464	4240	*3180
		219	423	4420	*3020
		205	451	4320	*3150
		202	477	4320	*3100
Mean		208	454	4330	3110
S.D.		7.77	23.1	73.7	69.9
%CV		3.7	5.1	1.7	2.2
%Theoretical		107.8	102.5	107.2	77.0
%Bias		7.8	2.5	7.2	-23.0
n		4	4	4	4

* >±20% deviation from theoretical

Table 114. PFOSA Quality Control Samples Prepared in Serum Diluted with Buffer

Run Date	Run Number	150 ng/mL DF=10	400 ng/mL DF=10	4000 ng/mL DF=10	4000 ng/mL DF=50
23-Oct-2000	PFOSMD-39	147	388	4020	*2380
		147	360	3850	*2240
		145	370	3980	*2210
		136	409	3960	*2210
Mean		144	382	3950	2260
S.D.		5.25	21.5	72.7	81.2
%CV		3.6	5.6	1.8	3.6
%Theoretical		96.0	95.5	98.8	56.5
%Bias		-4.0	-4.5	-1.3	-43.5
n		4	4	4	4

* >±20% deviation from theoretical

090314

Table 115. PFOSAA Quality Control Samples Prepared in Serum Diluted with Buffer

Run Date	Run Number	153 ng/mL DF=10	403 ng/mL DF=10	4000 ng/mL DF=10	4000 ng/mL DF=50
23-Oct-2000	PFOSMD-39	144	405	3920	*2750
		146	379	4100	*2560
		142	389	3970	*2620
		142	417	4010	*2550
Mean		144	398	4000	2620
S.D.		1.91	16.8	76.2	92.0
%CV		1.3	4.2	1.9	3.5
%Theoretical		94.1	98.8	100.0	65.5
%Bias		-5.9	-1.2	0.0	-34.5
n		4	4	4	4

* >±20% deviation from theoretical

Table 116. POAA Quality Control Samples Prepared in Serum Diluted with Buffer

Run Date	Run Number	157 ng/mL DF=10	407 ng/mL DF=10	4010 ng/mL DF=10	4010 ng/mL DF=50
23-Oct-2000	PFOSMD-39	163	410	4230	3610
		174	387	4190	3310
		164	418	4280	3330
		166	411	4140	3350
Mean		167	407	4210	3400
S.D.		4.99	13.5	59.4	141
%CV		3.0	3.3	1.4	4.1
%Theoretical		106.4	100.0	105.0	84.8
%Bias		6.4	0.0	5.0	-15.2
n		4	4	4	4

090315

Table 117. PFHS Quality Control Samples Prepared in Serum Diluted with Buffer

Run Date	Run Number	154 ng/mL DF=10	404 ng/mL DF=10	4000 ng/mL DF=10	4000 ng/mL DF=50
23-Oct-2000	PFOSMD-39	157	421	4200	3490
		163	389	4210	3300
		161	416	4330	3410
		166	419	4300	3260
Mean		162	411	4260	3370
S.D.		3.77	15.0	64.8	105
%CV		2.3	3.6	1.5	3.1
%Theoretical		105.2	101.7	106.5	84.3
%Bias		5.2	1.7	6.5	-15.8
n		4	4	4	4

Table 118. M556 Quality Control Samples Prepared in Serum Diluted with Buffer

Run Date	Run Number	151 ng/mL DF=10	401 ng/mL DF=10	4000 ng/mL DF=10	4000 ng/mL DF=50
23-Oct-2000	PFOSMD-39	156	421	4480	*2980
		157	385	4600	*2740
		145	403	4710	*2780
		148	417	4490	*2710
Mean		152	407	4570	2800
S.D.		5.92	16.3	108	122
%CV		3.9	4.0	2.4	4.4
%Theoretical		100.7	101.5	114.3	70.0
%Bias		0.7	1.5	14.3	-30.0
n		4	4	4	4

* >±20% deviation from theoretical

Table 119. M570 Quality Control Samples Prepared in Serum Diluted with Buffer

Run Date	Run Number	157 ng/mL DF=10	407 ng/mL DF=10	4010 ng/mL DF=10	4010 ng/mL DF=50
23-Oct-2000	PFOSMD-39	159	430	4240	*2910
		167	401	4390	*2730
		158	408	4290	*2700
		160	441	4330	*2680
Mean		161	420	4310	2760
S.D.		4.08	18.7	63.4	105
%CV		2.5	4.5	1.5	3.8
%Theoretical		102.5	103.2	107.5	68.8
%Bias		2.5	3.2	7.5	-31.2
n		4	4	4	4

* >±20% deviation from theoretical

Table 120. Summary of Calibration Curve Parameters for PFOS (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	A	B	C	R-Squared	LLOQ	ULOQ
27-Oct-2000	PFOSMD-40	Plasma	0.000000	0.028132	0.010586	0.9931	3.94	414
27-Oct-2000	PFOSMD-46	Serum	-0.000003	0.031081	0.084427	0.9506	36.8	454
25-Apr-2001	25	Plasma	-0.000003	0.048192	0.045435	0.9841	3.28	413
25-Apr-2001	26	Serum	-0.000020	0.057680	-0.301033	0.9934	36.8	454

A, B, and C are coefficients used to define the calibration curve.

Table 121. Summary of Calibration Curve Parameters for PFOSA (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	A	B	C	R-Squared	LLOQ	ULOQ
25-Apr-2001	25	Plasma	-0.000005	0.120662	0.028023	0.9887	1.00	500
25-Apr-2001	26	Serum	-0.000032	0.166445	0.051786	0.9873	1.00	502

A, B, and C are coefficients used to define the calibration curve.

Table 122. Summary of Calibration Curve Parameters for PFOSAA (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	A	B	C	R-Squared	LLOQ	ULOQ
25-Apr-2001	25	Plasma	0.000009	0.033608	0.035470	0.9938	1.30	500
25-Apr-2001	26	Serum	0.000007	0.044621	-0.040847	0.9947	4.20	503

A, B, and C are coefficients used to define the calibration curve.

Table 123. Summary of Calibration Curve Parameters for POAA (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	A	B	C	R-Squared	LLOQ	ULOQ
27-Oct-2000	PFOSMD-40	Plasma	0.000002	0.014346	0.002192	0.9918	1.92	481
27-Oct-2000	PFOSMD-46	Serum	0.000003	0.016839	0.003246	0.9894	6.82	461
25-Apr-2001	25	Plasma	-0.000003	0.011215	0.005552	0.9929	1.44	481
25-Apr-2001	26	Serum	0.000001	0.011414	0.000898	0.9931	6.82	461

A, B, and C are coefficients used to define the calibration curve.

Table 124. Summary of Calibration Curve Parameters for PFHS (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	A	B	C	R-Squared	LLOQ	ULOQ
27-Oct-2000	PFOSMD-40	Plasma	-0.000001	0.025534	0.011247	0.9953	1.36	523
27-Oct-2000	PFOSMD-46	Serum	-0.000012	0.033256	0.018733	0.9890	3.85	431

A, B, and C are coefficients used to define the calibration curve.

Table 125. Summary of Calibration Curve Parameters for M556 (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	A	B	C	R-Squared	LLOQ	ULOQ
27-Oct-2000	PFOSMD-40	Plasma	0.000002	0.016888	0.007835	0.9949	2.50	500
27-Oct-2000	PFOSMD-46	Serum	0.000005	0.014650	0.008960	0.9903	3.80	501

A, B, and C are coefficients used to define the calibration curve.

Table 126. Summary of Calibration Curve Parameters for M570 (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	A	B	C	R-Squared	LLOQ	ULOQ
27-Oct-2000	PFOSMD-40	Plasma	0.000001	0.034031	0.011681	0.9910	1.00	500
27-Oct-2000	PFOSMD-46	Serum	0.000003	0.034699	0.028660	0.9919	5.20	503
25-Apr-2001	25	Plasma	0.000009	0.039312	0.045599	0.9964	1.00	500
25-Apr-2001	26	Serum	0.000002	0.046817	0.021413	0.9931	3.70	503

A, B, and C are coefficients used to define the calibration curve.

Table 127. Back-Calculated Concentrations of Calibration Standards for PFOS (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	**Run Number**	**Matrix**	**3.94**	**5.17**	**11.3**	**23.6**	**44.2**	**85.4**	**209**	**332**	**414**
27-Oct-2000	PFOSMD-40	Plasma	3.72	5.18	11.3	19.4	45.5	82.1	214	337	369
			3.90	5.67	11.7	24.1	45.9	84.4	221	367	407
Run Date	**Run Number**	**Matrix**	**36.8**	**38.0**	**44.3**	**56.8**	**77.7**	**120**	**245**	**370**	**454**
27-Oct-2000	PFOSMD-46	Serum	34.0	31.7	37.3	49.3	70.7	114	247	374	392
			50.5	39.0	45.8	60.7	78.7	125	282	418	436
Run Date	**Run Number**	**Matrix**	**3.28**	**4.52**	**10.7**	**23.0**	**43.5**	**84.6**	**208**	**331**	**413**
25-Apr-2001	25	Plasma	2.70	3.90	9.65	21.4	42.2	80.3	190	324	392
			4.15	4.63	11.3	25.0	47.6	90.3	217	336	441
Run Date	**Run Number**	**Matrix**	**36.8**	**38.0**	**44.3**	**56.8**	**77.7**	**120**	**245**	**370**	**454**
25-Apr-2001	26	Serum	38.3	35.5	44.2	54.7	71.1	124	234	363	420
			37.4	39.0	46.1	56.1	75.9	128	256	400	459

000321

Table 128. Back-Calculated Concentrations of Calibration Standards for PFOSA (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	1.00	2.50	10.0	25.0	50.0	100	250	400	500
25-Apr-2001	25	Plasma	0.935	2.52	7.89	23.0	45.4	102	223	387	477
			*1.77	2.99	10.3	27.3	52.5	113	254	402	546
25-Apr-2001	26	Serum	0.951	1.96	9.62	25.6	48.1	109	240	403	422
			1.18	2.21	10.7	24.3	52.4	111	279	450	491

* Sample deactivated as an outlier

000322

Table 129. Back-Calculated Concentrations of Calibration Standards for PFOSAA (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	1.30	2.80	10.3	25.3	50.3	100	250	400	500
25-Apr-2001	25	Plasma	1.28	2.65	8.27	24.7	50.7	102	253	399	480
			*2.23	3.22	9.79	24.9	54.4	110	257	391	509
Run Date	**Run Number**	**Matrix**	**4.20**	**5.70**	**13.2**	**28.2**	**53.2**	**103**	**253**	**403**	**503**
25-Apr-2001	26	Serum	4.27	5.31	12.8	26.9	50.7	119	259	391	477
			4.38	5.78	13.6	25.0	51.9	111	260	407	511

* Sample deactivated as an outlier

000323

Table 130. Back-Calculated Concentrations of Calibration Standards for POAA (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	1.92	3.36	10.6	25.0	49.0	97.1	241	385	481
27-Oct-2000	PFOSMD-40	Plasma	1.73	3.01	10.5	22.9	49.7	99.1	250	367	439
			2.30	3.12	10.9	25.1	50.7	96.7	262	422	474
Run Date	**Run Number**	**Matrix**	**6.82**	**8.18**	**15.0**	**28.6**	**51.4**	**97.3**	**234**	**370**	**461**
27-Oct-2000	PFOSMD-46	Serum	7.32	6.86	13.9	27.2	51.9	105	252	399	421
			*18.3	9.23	14.9	29.4	46.2	94.9	246	392	424
Run Date	**Run Number**	**Matrix**	**1.44**	**2.88**	**10.1**	**24.5**	**48.5**	**96.6**	**241**	**385**	**481**
25-Apr-2001	25	Plasma	1.35	2.65	10.5	24.1	43.4	91.1	225	396	470
			*2.14	3.35	11.2	26.6	46.5	90.9	241	405	498
Run Date	**Run Number**	**Matrix**	**6.82**	**8.18**	**15.0**	**28.6**	**51.4**	**97.3**	**234**	**370**	**461**
25-Apr-2001	26	Serum	7.98	8.28	15.6	28.8	50.2	103	242	383	457
			6.23	7.23	15.0	26.9	48.0	100	235	378	433

* Sample deactivated as an outlier

000324

Table 131. Back-Calculated Concentrations of Calibration Standards for PFHS (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	1.36	2.92	10.7	26.5	52.6	104	262	418	523
27-Oct-2000	PFOSMD-40	Plasma	1.52✓	2.72	10.6	24.2	54.6	107	259	466	489
			1.28	2.78	10.4	28.4	54.1	102	270	425	491
Run Date	**Run Number**	**Matrix**	**3.85**	**5.13**	**11.5**	**24.4**	**45.7**	**88.9**	**217**	**345**	**431**
27-Oct-2000	PFOSMD-46	Serum	4.08	4.36	11.2	21.8	47.5	89.1	235	371	363
			*15.4	5.80	10.6	23.5	49.0	89.7	230	388	389

* Sample deactivated as an outlier

090325

Table 132. Back-Calculated Concentrations of Calibration Standards for M556 (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	2.50	10.0	25.0	50.0	100	250	400	500
27-Oct-2000	PFOSMD-40	Plasma	2.64	10.7	23.4	52.9	101	276	396	456
			2.35	9.95	23.3	48.3	94.1	260	440	481
Run Date	**Run Number**	**Matrix**	**3.80**	**11.3**	**26.3**	**51.3**	**101**	**251**	**401**	**501**
27-Oct-2000	PFOSMD-46	Serum	3.89	11.1	25.3	53.7	116	280	441	480
			3.91	10.0	25.7	42.9	102	258	396	450

090326

Table 133. Back-Calculated Concentrations of Calibration Standards for M570 (Serum vs. Plasma)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	Matrix	1.00	2.50	10.0	25.0	50.0	100	250	400	500
27-Oct-2000	PFOSMD-40	Plasma	0.993	2.71	10.6	21.2	53.6	104	284	401	444
			0.951	2.65	10.4	20.9	48.6	93.5	254	443	481
Run Date	**Run Number**	**Matrix**	**3.70**	**5.20**	**12.7**	**27.7**	**52.7**	**103**	**253**	**403**	**503**
27-Oct-2000	PFOSMD-46	Serum	*5.54	4.92	11.8	27.4	51.0	116	277	432	464
			*16.4	5.75	12.4	26.9	45.5	106	271	404	468
Run Date	**Run Number**	**Matrix**	**1.00**	**2.50**	**10.0**	**25.0**	**50.0**	**100**	**250**	**400**	**500**
25-Apr-2001	25	Plasma	1.01	2.43	8.50	24.6	52.8	105	246	420	483
			*1.90	2.66	9.29	25.4	51.9	108	247	387	504
Run Date	**Run Number**	**Matrix**	**3.70**	**5.20**	**12.7**	**27.7**	**52.7**	**103**	**253**	**403**	**503**
25-Apr-2001	26	Serum	4.07	4.60	12.5	27.4	50.3	118	262	395	478
			3.94	4.60	13.3	26.0	49.7	109	264	405	502

* Sample deactivated as an outlier

000327

Table 134. PFOS Quality Controls (Serum vs. Plasma)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 6.40 ppb	Medium QC (Plasma) 126 ppb	High QC (Plasma) 332 ppb
27-Oct-2000	PFOSMD-40	6.44	115	316
		5.99	136	326
		5.91	118	298
Mean		6.11	123	313
SD		0.286	11.4	14.2
%CV		4.7	9.3	4.5
%Bias		-4.5	-2.4	-5.7
n		3	3	3
Run Date	**Run Number**	**Low QC (Serum) 39.3 ppb**	**Medium QC (Serum) 161 ppb**	**High QC (Serum) 370 ppb**
27-Oct-2000	PFOSMD-46	35.0	164	336
		35.6	151	*199
		35.5	149	343
		35.8	152	346
Mean		35.5	154	342
SD		0.340	6.78	5.13
%CV		1.0	4.4	1.5
%Bias		-9.7	-4.3	-7.6
n		4	4	3

* Sample deactivated due to a sample preparation error.

** > ±20% deviation from theoretical

000328

Table 134. PFOS Quality Controls (Serum vs. Plasma) Continued

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 5.75 ppb	Medium QC (Plasma) 126 ppb	High QC (Plasma) 331 ppb
25-Apr-2001	25	5.44	143	333
		5.62	139	325
		5.59	136	347
		5.90	132	333
Mean		5.64	138	335
SD		0.192	4.65	9.15
%CV		3.4	3.4	2.7
%Bias		-1.9	9.5	1.2
n		4	4	4
Run Date	**Run Number**	**Low QC (Serum) 39.3 ppb**	**Medium QC (Serum) 161 ppb**	**High QC (Serum) 370 ppb**
25-Apr-2001	26	41.3	174	377
		45.4	182	388
		45.8	172	408
		45.6	169	381
Mean		44.5	174	389
SD		2.16	5.56	13.8
%CV		4.9	3.2	3.5
%Bias		13.2	8.1	5.1
n		4	4	4

** > ±20% deviation from theoretical

000329

Table 135. PFOSA Quality Controls (Serum vs. Plasma)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 4.00 ppb	Medium QC (Plasma) 150 ppb	High QC (Plasma) 400 ppb
25-Apr-2001	25	4.06	158	395
		3.99	175	412
		4.49	170	438
		4.73	159	424
Mean		4.32	166	417
SD		0.353	8.35	18.2
%CV		8.2	5.0	4.4
%Bias		8.0	10.7	4.3
n		4	4	4
Run Date	**Run Number**	**Low QC (Serum) 4.00 ppb**	**Medium QC (Serum) 150 ppb**	**High QC (Serum) 400 ppb**
25-Apr-2001	26	3.52	153	390
		4.75	163	431
		4.55	155	417
		**4.85	151	377
Mean		4.42	156	404
SD		0.611	5.26	24.6
%CV		13.8	3.4	6.1
%Bias		10.5	4.0	1.0
n		4	4	4

* Sample deactivated due to a sample preparation error.

** > ±20% deviation from theoretical

090330

Table 136. PFOSAA Quality Controls (Serum vs. Plasma)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 4.30 ppb	Medium QC (Plasma) 150 ppb	High QC (Plasma) 400 ppb
25-Apr-2001	25	3.59	**187	429
		3.85	175	412
		3.74	172	440
		4.96	169	429
Mean		4.04	176	428
SD		0.626	7.89	11.6
%CV		15.5	4.5	2.7
%Bias		-6.0	17.3	7.0
n		4	4	4
Run Date	**Run Number**	**Low QC (Serum) 7.20 ppb**	**Medium QC (Serum) 153 ppb**	**High QC (Serum) 403 ppb**
25-Apr-2001	26	6.74	150	363
		8.11	162	409
		8.29	159	396
		8.37	157	376
Mean		7.88	157	386
SD		0.766	5.10	20.5
%CV		9.7	3.2	5.3
%Bias		9.4	2.6	-4.2
n		4	4	4

* Sample deactivated due to a sample preparation error.

** > ±20% deviation from theoretical

090331

Table 137. POAA Quality Controls (Serum vs. Plasma)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 4.81 ppb	Medium QC (Plasma) 145 ppb	High QC (Plasma) 385 ppb
27-Oct-2000	PFOSMD-40	4.57	146	373
		4.31	167	419
		4.52	147	393
Mean		4.47	153	395
SD		0.138	11.8	23.1
%CV		3.1	7.7	5.8
%Bias		-7.1	5.5	2.6
n		3	3	3
Run Date	**Run Number**	**Low QC (Serum) 9.54 ppb**	**Medium QC (Serum) 143 ppb**	**High QC (Serum) 370 ppb**
27-Oct-2000	PFOSMD-46	9.42	158	373
		9.17	146	*253
		8.53	148	375
		9.62	150	388
Mean		9.19	151	379
SD		0.474	5.26	8.14
%CV		5.2	3.5	2.1
%Bias		-3.7	5.6	2.4
n		4	4	3

* Sample deactivated due to a sample preparation error.

** > ±20% deviation from theoretical

090332

Table 137. POAA Quality Controls (Serum vs. Plasma) Continued

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 4.32 ppb	Medium QC (Plasma) 145 ppb	High QC (Plasma) 385 ppb
25-Apr-2001	25	5.13	143	394
		4.47	145	383
		4.35	145	412
		4.44	137	404
Mean		4.60	143	398
SD		0.359	3.79	12.6
%CV		7.8	2.7	3.2
%Bias		6.5	-1.4	3.4
n		4	4	4
Run Date	**Run Number**	**Low QC (Serum) 9.54 ppb**	**Medium QC (Serum) 143 ppb**	**High QC (Serum) 370 ppb**
25-Apr-2001	26	10.1	157	374
		11.4	152	389
		**13.2	149	385
		9.82	152	391
Mean		11.1	153	385
SD		1.54	3.32	7.59
%CV		13.9	2.2	2.0
%Bias		16.4	7.0	4.1
n		4	4	4

** > ±20% deviation from theoretical

060333

Table 138. PFHS Quality Controls (Serum vs. Plasma)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 4.48 ppb	Medium QC (Plasma) 156 ppb	High QC (Plasma) 417 ppb
27-Oct-2000	PFOSMD-40	4.62	157	397
		4.35	173	407
		4.39	151	384
Mean		4.45	160	396
SD		0.146	11.4	11.5
%CV		3.3	7.1	2.9
%Bias		-0.7	2.6	-5.0
n		3	3	3
Run Date	**Run Number**	**Low QC (Serum) 6.41 ppb**	**Medium QC (Serum) 132 ppb**	**High QC (Serum) 345 ppb**
27-Oct-2000	PFOSMD-46	5.65	144	343
		5.70	136	*221
		6.87	129	395
		5.71	135	367
Mean		5.98	136	368
SD		0.592	6.16	26.0
%CV		9.9	4.5	7.1
%Bias		-6.7	3.0	6.7
n		4	4	3

* Sample deactivated due to a sample preparation error.

** > ±20% deviation from theoretical

090334

Table 139. M556 Quality Controls (Serum vs. Plasma)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 4.00 ppb	Medium QC (Plasma) 150 ppb	High QC (Plasma) 400 ppb
27-Oct-2000	PFOSMD-40	3.73	133	389
		3.45	168	420
		3.34	139	387
Mean		3.51	147	399
SD		0.201	18.7	18.5
%CV		5.7	12.7	4.6
%Bias		-12.3	-2.0	-0.3
n		3	3	3
Run Date	**Run Number**	**Low QC (Serum) 5.30 ppb**	**Medium QC (Serum) 151 ppb**	**High QC (Serum) 401 ppb**
27-Oct-2000	PFOSMD-46	5.03	166	383
		5.23	152	**243
		**4.15	152	371
		5.01	155	388
Mean		4.86	156	381
SD		0.480	6.65	8.74
%CV		9.9	4.3	2.3
%Bias		-8.3	3.3	-5.0
n		4	4	3

** > ±20% deviation from theoretical

090335

Table 140. M570 Quality Controls (Serum vs. Plasma)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 4.00 ppb	Medium QC (Plasma) 150 ppb	High QC (Plasma) 400 ppb
27-Oct-2000	PFOSMD-40	3.95	133	379
		3.54	161	403
		3.38	136	360
Mean		3.62	143	381
SD		0.294	15.4	21.5
%CV		8.1	10.8	5.6
%Bias		-9.5	-4.7	-4.8
n		3	3	3
Run Date	**Run Number**	**Low QC (Serum) 6.70 ppb**	**Medium QC (Serum) 153 ppb**	**High QC (Serum) 403 ppb**
27-Oct-2000	PFOSMD-46	6.36	169	397
		6.35	150	*227
		**5.33	156	367
		6.39	155	400
Mean		6.11	158	388
SD		0.519	8.10	18.2
%CV		8.5	5.1	4.7
%Bias		-8.8	3.3	-3.7
n		4	4	3

* Sample deactivated due to a sample preparation error.

** > ±20% deviation from theoretical

090336

Table 140. M570 Quality Controls (Serum vs. Plasma) Continued

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC (Plasma) 4.00 ppb	Medium QC (Plasma) 150 ppb	High QC (Plasma) 400 ppb
25-Apr-2001	25	3.24	180	420
		3.49	175	417
		3.58	164	440
		4.05	171	432
Mean		3.59	173	427
SD		0.339	6.76	10.7
%CV		9.4	3.9	2.5
%Bias		-10.3	15.3	6.8
n		4	4	4
Run Date	**Run Number**	**Low QC (Serum) 6.70 ppb**	**Medium QC (Serum) 153 ppb**	**High QC (Serum) 403 ppb**
25-Apr-2001	26	5.90	166	392
		7.49	167	445
		7.03	167	437
		8.03	161	409
Mean		7.11	165	421
SD		0.906	2.87	24.6
%CV		12.7	1.7	5.8
%Bias		6.1	7.8	4.5
n		4	4	4

** > ±20% deviation from theoretical

090337

Table 141. Serum QC Results Based on Plasma Curves

Sample ID	PFOS (ppb)	PFOSA (ppb)*	PFOSAA (ppb)*	POAA (ppb)	PFHS (ppb)*	M556 (ppb)*	M570 (ppb)
Low (analyzed 10/27/00)	41.2			11.1	7.64	4.44	6.99
Low (analyzed 10/27/00)	41.8			10.8	7.71	4.61	6.98
Low (analyzed 10/27/00)	41.8			10.1	9.22	3.67	5.93
Low (analyzed 10/27/00)	42.1			11.4	7.72	4.42	7.02
Low (analyzed 4/25/01)	41.7	5.04	6.68	9.62			6.41
Low (analyzed 4/25/01)	46.5	6.47	8.5	9.93			7.74
Low (analyzed 4/25/01)	46.7	6.75	8.72	11.3			8.29
Low (analyzed 4/25/01)	47.0	6.88	8.83	13.0			8.94
mean *	43.6	6.29	8.18	10.9	8.07	4.29	7.29
std. dev. *	2.61	0.847	1.01	1.07	0.766	0.419	0.986
%CV *	6.0%	13.5%	12.4%	9.9%	9.5%	9.8%	13.5%
%dev *	10.9%	57.1%	13.6%	14.3%	25.9%	-19.2%	8.8%
acceptance range *	31.4 to 47.2	3.00 to 5.00	5.40 to 9.00	7.63 to 11.4	5.13 to 7.69	4.24 to 6.36	5.36 to 8.04
95% confidence range *	**41.8 to 45.4**	**5.45 to 7.12**	**7.48 to 8.88**	**10.2 to 11.7**	**7.32 to 8.82**	**3.87 to 4.7**	**6.60 to 7.97**
Med (analyzed 10/27/00)	182			186	180	149	175
Med (analyzed 10/27/00)	168			172	170	136	155
Med (analyzed 10/27/00)	165			174	162	137	161
Med (analyzed 10/27/00)	168			177	169	139	160
Med (analyzed 4/25/01)	185	205	192	160			184
Med (analyzed 4/25/01)	189	207	200	164			190
Med (analyzed 4/25/01)	191	210	203	164			191
Med (analyzed 4/25/01)	199	220	207	170			191
mean *	181	211	201	171	170	140	176
std. dev. *	12.5	6.66	6.4	8.37	7.41	5.97	15.3
%CV *	6.9%	3.2%	3.2%	4.9%	4.4%	4.3%	8.7%
%dev *	12.3%	40.3%	31.0%	19.5%	29.0%	-7.1%	15.0%
acceptance range *	129 to 193	113 to 188	115 to 191	114 to 172	106 to 158	121 to 181	122 to 184
95% confidence range *	**172 to 190**	**204 to 217**	**196 to 205**	**165 to 177**	**163 to 178**	**134 to 146**	**165 to 186**
High (analyzed 10/27/00)	364			441	400	361	416
High (analyzed 10/27/00)	370			443	452	349	384
High (analyzed 10/27/00)	374			459	424	366	420
High (analyzed 4/25/01)	396	493	451	453			433
High (analyzed 4/25/01)	399	509	467	470			450
High (analyzed 4/25/01)	406	543	490	476			478
High (analyzed 4/25/01)	424	560	506	478			486
mean *	390	526	479	460	425	359	438
std. dev. *	21.8	30.7	24.3	15.2	26.0	8.74	36.0
%CV *	5.6%	5.8%	5.1%	3.3%	6.1%	2.4%	8.2%
%dev *	5.5%	31.6%	18.7%	24.3%	23.3%	-10.6%	8.7%
acceptance range *	296 to 444	300 to 500	302 to 504	296 to 444	276 to 414	321 to 481	322 to 484
95% confidence range *	**374 to 407**	**492 to 561**	**460 to 497**	**449 to 471**	**396 to 455**	**349 to 369**	**411 to 465**

* Due to rounding, calculations based on displayed values may differ from actual.
NOTE: Only data from acceptable runs included in the Table (see report text).
(Data obtained on 10/27/00 from run PFOSMD-40. Data obtained on 4/25/01 from run S00040-25.)

050338

AR226_1211

Study No. NWBS00-088
Report No. NWBR01-001

Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS

Sample Analysis Report for Protocol EPI-0011

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Northwest Bioanalytical (NWB)
A Division of NWT Inc.
1121 East 3900 South
Salt Lake City, UT 84124

PREPARED FOR: 3M Company
Corporate Occupational Medicine, Medical Department
220-3W-05
St. Paul, MN 55144

AUTHOR:
Connie O Sakashita
Connie O. Sakashita, B.S., NWB Project Manager
DATE: 11-30-01

APPROVED FOR RELEASE BY:
R L Foltz
Rodger L. Foltz, Ph.D., NWB Technical Director
DATE: 11-30-01

CONTAIN NO CBI

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QUALITY ASSURANCE STATEMENT

LABORATORY:	Northwest Bioanalytical (NWB) A Division of NWT Inc. 1121 East 3900 South Salt Lake City, UT 84124
SPONSOR:	3M Company Corporate Occupational Medicine, Medical Department 220-3W-05 St. Paul, MN 55144
COMPOUND(S):	PFOS and Related Compounds
NWB STUDY NUMBER:	NWBS00-088
SPONSOR STUDY NUMBER:	EPI-0011
NWB STUDY TITLE:	Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS

The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all studies within the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.) The following inspections were performed by the NWB QAU per SOP.

Inspection and Reporting Statement.

Date of Inspection	Phase of Study	Date Report Sent to NWB Project Manager	Date Report Sent to NWB Management*
22-25 Sep 2000	Sample Receipt	25 Sep 2000	29 Sep 2000
16 Oct 2000	Analytical Plan	16 Oct 2000	31 Oct 2000
07-13 Nov 2000	Sample Analysis	14 Nov 2000	30 Nov 2000
20-25 Oct 2001	Report Draft/Raw Data	25 Oct 2001	31 Oct 2001
27 Nov 2001	2nd Report Draft/Raw Data	27 Nov 2001	30 Nov 2001
03 Dec 2001	Final Report	03 Dec 2001	31 Dec 2001

*Reports to NWB Management are issued monthly.

As can reasonably be established, the methods and procedures described and the results incorporated into this final report accurately reflect the raw data.

Stacy Lynn Chain, B.A.
NWB Quality Assurance Compliance Auditor

12-03-01
Date

COMPLIANCE STATEMENT

The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, to the best of our knowledge, this project was conducted in accordance with the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58) and according to the methods and procedures described within this report. In addition, the study followed the guidelines of the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.) Any known incidents that may have affected the quality or integrity of the project or reported data is included in this report. This report represents an accurate record of the raw data.

Connie O. Sakashita

Connie O. Sakashita, B.S.
NWB Project Manager

11-30-01
Date

Rodger L. Foltz

Rodger L. Foltz, Ph.D.
NWB Technical Director

11-30-01
Date

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Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS

Sample Analysis Report for Protocol EPI-0011

1. INTRODUCTION

This report summarizes the analytical results from the quantitation of PFOS, PFOA, PFHS, PFOSAA, PFOSA, M556, and M570 in human serum samples for 3M Company in support of Protocol EPI-0011 [5.1]. The LC/MS/MS method for the analytes exhibited a quadratic response for the following LLOQ and ULOQ values:

Analyte	LLOQ	ULOQ
PFOS	3.94 ppb	414 ppb
PFOA	1.92 ppb	481 ppb
PFHS	1.36 ppb	523 ppb
PFOSAA	1.60 ppb	501 ppb
PFOSA	1.00 ppb	500 ppb
M556	2.50 ppb	500 ppb
M570	1.00 ppb	500 ppb

The testing facility was 3M Company (Corporate Occupational Medicine, Medical Department, 220-3W-05, St. Paul, MN 55144), and the Study Director was Jeffrey H. Mandel, M.D., M.P.H. James K. Lundberg served as the technical contact at 3M. The following is a list of NWB supervisory personnel involved in the completion of this work: Connie O. Sakashita, B.S. (NWB Project Manager); Patrick Bennett, M.S., M.B.A. (NWB Laboratory Director); Rodger L. Foltz, Ph.D. (NWB Technical Director). Laboratory personnel involved in the completion of this study included Suzanne Newman, B.S. (NWB Research Scientist); Emily Yardimci, B.S. (NWB Scientist).

NWB SOPs and guidelines were used in the conduct of this study and were available to study personnel in electronic and/or hard copy formats.

Date Study Initiated: 22-Sep-2000 Date Analyses Completed: 25-Mar-2001

However, Northwest Bioanalytical conducts all studies within the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424). Any changes to or deviations from the original protocol (Analytical Plan) were documented through approved protocol amendments or deviation memos and are retained within the raw data.

2. METHODOLOGY

The assay used for this study is reported in Northwest Bioanalytical reports NWBR00-108 [5.2] and NWBR00-122 [5.3]. Chinese human plasma was used for the calibration curves and quality control samples. Serum sample results obtained using plasma curves are discussed in NWBR00-122.

Samples for this study were received at NWB on the following dates:

Receipt Date	*Number of Samples Received*	*Storage Condition (except during analysis)*
21-Sep-2000	526	-20 °C
22-Sep-2000	73	-20 °C
21-Feb-2001	62	-20 °C

Reference Material

Analyte	*Lot Number*	*Purity*	*Expiration Date*	*Source*	*Storage Conditions*
PFOS	193	100%	31-Dec-2010	3M	Room temperature*
PFOA	245	100%	31-Dec-2010	3M	Room temperature*
PFHS	SE-036	100%	01-Jan-2010	3M	-20 °C
PFOSAA	617	53.8%	31-Dec-2010	3M	Room temperature
PFOSA	214	100%	31-Dec-2010	3M	Room temperature
M556	NB113047-8D	99.89%	31-Dec-2010	3M	Room temperature
M570	118506-26	99.75%	31-Dec-2010	3M	Room temperature
THPFOS	59909	90%	31-Dec-2010	3M	Room temperature

*Stored dry

The reference material purity for PFOS, PFOSA, PFHS, and PFOA was not available prior to the conduct of this study. Therefore, the reference material purity was assumed to be 100%.

3M contracted with Centre Analytical Laboratories, Inc., in State College, Pennsylvania to determine the absolute concentration of PFOS, PFOA, and PFHS in NWB stock solutions used to prepare the analytical standards and controls used for this studies. All arrangements for purity determinations and the transfer of NWB solutions to Centre Analytical Laboratories, Inc., were performed by 3M. Based on Centre Analytical Laboratories' results, the concentrations of plasma calibrator and quality control samples were corrected using the following factors:

Analyte	*Correction Factor*
PFOS	0.821
PFOA	0.961
PFHS	1.043

Eight or more calibration standards were prepared on the day of each run by adding 100 µL of blank human Chinese plasma and 400 µL of 50 mM ammonium acetate in

water (unadjusted pH ~6.9) to 13 x 100 mm polypropylene tubes. After a brief vortex mixing, 10.0 µL of the appropriate spiking solution was added.The final calibration standard concentrations in human Chinese plasma for each analyte are listed below:

Analyte	Calibration Standard Concentrations*
PFOS	3.94, 5.17, 11.3, 23.6, 44.2, 85.4, 209, 332, and 414 ppb
PFOA	1.92, 3.36, 10.6, 25.0, 49.0, 97.1, 241, 385, and 481 ppb
PFHS	1.36, 2.92, 10.7, 26.5, 52.6, 104, 262, 418, and 523 ppb
PFOSAA	1.60, 3.10, 10.6, 25.6, 50.6, 101, 251, 401, and 501 ppb
PFOSA	1.00, 2.50, 10.0, 25.0, 50.0, 100, 250, 400, and 500 ppb
M556	2.50, 10.0, 25.0, 50.0, 100, 250, 400, and 500 ppb
M570	1.00, 2.50, 10.0, 25.0, 50.0, 100, 250, 400, and 500 ppb

* The target calibration curve range is 1.00 ppb to 500 ppb (except for M556 which has a LLOQ of 2.50 ppb). Each analyte has a different final curve range based upon the persistent levels of the analyte in the human Chinese plasma used.

In addition, blank plasma samples, both with and without internal standard (designated as QC0s and Blanks, respectively), were assayed in each analytical run.

Analytical QCs were prepared in human Chinese plasma on October 6, 2000, October 11, 2000, and October 27, 2000 and stored in a -20 °C freezer. For each run, analytical QC levels were assayed in duplicate. In addition, for each dilution level, dilution QCs were run in triplicate in any analytical run that contained diluted subject samples. The QC concentration levels are shown in the following table.

	QC Concentrations All concentrations are expressed as ppb.			
Analyte	Low	Medium	High	High, DF = 2
PFOS	6.40	126	332	335
PFOA	4.81	145	385	386
PFHS	4.48	156	417	418
PFOSAA	4.60	151	401	401
PFOSA	4.00	150	400	400
M556	4.00	150	400	400
M570	4.00	150	400	400

The internal standard (THPFOS) was added to all plasma and serum samples (except Blanks) for a final concentration of approximately 200 ppb.

The analytical method consisted of a liquid:liquid extraction procedure followed by evaporation and reconstitution of the extract residue with 20 mM ammonium acetate in water: 20 mM ammonium acetate in methanol (30:70 v/v). The samples were analyzed by liquid chromatography/tandem mass spectrometry using a PE Sciex API 3000. The instrument was operated in the multiple reaction monitoring (MRM) mode under optimized conditions for PFOS, PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 detection of the negative ions formed by TurboIonSpray™ ionization.

3. SAMPLE ANALYSIS

PFOS, PFOA, PFHS, PFOSAA, PFOSA, M556, M570, and THPFOS chromatographic peaks were integrated using the PE Sciex MacQuan software (version 1.6) with a smooth factor of one. Quantitation was based upon quadratic regression analysis of weighted ($1/x^2$) calibration curves using area ratio vs. concentration utilizing Watson® DMLIMS software (version 6.1.1.04). The samples were injected in a systematic order.

3.1. Acceptance Criteria

For an analytical run to be accepted, it must have met the acceptance criteria listed below which are consistent with regulatory and industry recommendations.

Calibration Curve

Each run will include in duplicate calibration standards at six or more concentrations covering the lower to upper limit of quantitation. For all analytes except PFOSA and PFOSAA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 15% (± 20% for LLOQ) of their individual target concentrations. For PFOSA and PFOSAA, at least three-fourths of the calibration

standard's back-calculated concentrations must be within ± 20% (± 25% for LLOQ) of their individual target concentrations. A calibration standard will be considered an outlier if it is greater than two times the acceptance criteria for that standard.

Lower Limit of Quantitation

The back-calculated concentrations of at least one of the duplicate lowest points in the calibration curve must be within ± 25% of the target concentration for PFOSA and PFOSAA to qualify as the LLOQ and within ± 20% of the target concentration to qualify as the LLOQ for all other analytes. If this criterion is not met, the next level is subjected to the same test and the LLOQ raised accordingly.

Quality Control Samples

Each analytical run will include low, medium and high QC samples in duplicate. The measured concentrations of at least two-thirds of all analytical QCs must be within ± 20% of their target concentrations (± 25% for PFOSA and PFOSAA), and no two QCs at the same concentration can be outside the limit. If study samples require dilution, a dilution QC will be analyzed in triplicate for each dilution level (except for PFOSA and PFOSAA which should not be diluted with control matrix). At least two dilution QCs at each level must be within ± 20% of its target concentrations in order to accept diluted study samples at that level. The dilution QC acceptance is independent of the undiluted analytical QC acceptance.

4. RESULTS AND DISCUSSION

Serum sample results obtained using plasma curves for PFOA, PFHS, PFOSAA, PFOSA, M556, and M570 did not meet NWB SOP requirements for validation. Results may vary on average up to 26% from results obtained using human serum calibration curves at some concentration levels for PFOA, PFHS, PFOSAA, M556, and M570. For PFOSA, results may vary on average up to 43% from results obtained using human serum

calibration curves. However, in order to obtain the lower limit of quantitation less than 5 ppb required by the Sponsor, it was necessary to use plasma curves.

Each accepted run met the acceptance criteria set for the calibration curve points, for the lower limit of quantitation (LLOQ) and for the analytical quality control (QC) samples.

Run ID	PFOS Result	PFOA Result	PFHS Result	PFOSAA Result	Comments
1	Rejected	Rejected	Rejected	Rejected	All analytes rejected due to sample preparation error
2	Accepted	Accepted	Accepted	Accepted	
3	Accepted	Accepted	Accepted	Accepted	
4	Accepted	Accepted	Accepted	Accepted	
5	Accepted	Accepted	Accepted	Accepted	
6	Accepted	Accepted	Accepted	Accepted	
7	Accepted	Accepted	Accepted	Accepted	
8	Accepted	Accepted	Accepted	Accepted	
9	Accepted	Accepted	Accepted	Accepted	
10	Accepted	Accepted	Accepted	Accepted	
11	Accepted	Accepted	Accepted	Accepted	
12	Accepted	Accepted	Accepted	Accepted	LLOQ raised for PFOS
13	Accepted	Accepted	Accepted	Accepted	
14	Accepted	Accepted	Accepted	Rejected	Low QCs failed for PFOSAA.
15	N/A	N/A	N/A	N/A	
16	N/A	N/A	N/A	Accepted	
17	Accepted	Accepted	Accepted	Accepted	

N/A = not analyzed

Run ID	PFOSA Result	M556 Result	M570 Result	Comments
1	Rejected	Rejected	Rejected	All analytes rejected due to sample preparation error
2	Accepted	Accepted	Accepted	
3	Accepted	Accepted	Accepted	
4	Accepted	Accepted	Accepted	
5	Accepted	Accepted	Accepted	

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Run ID	PFOSA Result	M556 Result	M570 Result	Comments
6	Accepted	Accepted	Accepted	
7	Accepted	Accepted	Accepted	
8	Accepted	Accepted	Accepted	
9	Accepted	Accepted	Accepted	
10	Accepted	Accepted	Accepted	
11	Rejected	Accepted	Accepted	Standard curve failed for PFOSA. Reinjection of Run 11 for PFOSAA only.
12	Accepted	Accepted	Accepted	
13	Accepted	Accepted	Accepted	
14	Accepted	Accepted	Rejected	Low QCs failed for M570
15	Rejected	N/A	N/A	Standard curve failed for PFOSA
16	N/A	N/A	Accepted	
17	Accepted	Accepted	Accepted	

N/A = not analyzed

Because of the limited sample volume available, several samples in this study were analyzed at a dilution factor of 2. High QC samples, diluted at a factor of 2, were also analyzed in triplicate.

METH00040 did not meet the validation acceptance criteria for analysis of QC samples containing PFOSA or PFOSAA at concentrations above the ULOQ (analyzed after dilution). Samples containing PFOSA or PFOSAA at concentrations less than the ULOQ and analyzed after diluting with control matrix were not evaluated for accuracy and precision during method validation.

PFOSA and PFOSAA accuracy and precision for the diluted high QC samples from 3 separate runs was determined to assess the validity of diluting samples with Chinese plasma when samples have PFOSA or PFOSAA concentrations less than the ULOQ. (Refer to PFOSAA results in Runs 12, 13, and 16 and PFOSA results in Runs 12, 13, and 14.) Both intra- and inter-assay precision were <15% and intra- and inter-assay accuracy deviated <15% from target (data not shown). Therefore, the accuracy and precision obtained after diluting samples with Chinese plasma when samples have PFOSA or

PFOSAA concentrations less than the ULOQ is acceptable and PFOSA and PFOSAA results from diluted samples will be reported.

The lack of dilution data prior to analysis of diluted of high QC samples did not affect the quality of the assay or integrity of the PFOSA and PFOSAA data.

Sample 230 was analyzed on Run 1 which had to be rejected for all analytes due to sample preparation error. There is insufficient sample for reanalysis.

SOP Deviations

Samples 2193 to 2363 and 2366 to 2500 were analyzed for PFOSA in Run 11 and Run 15. The standard curves did not meet the acceptance criteria in either run and therefore, PFOSA results were rejected. There was insufficient sample to reanalyze. Since the runs failed, no standard or QC data from the runs is included in this report.

All subject PFOSA sample results obtained in Runs 11 and 15 were less than the LLOQ. The standard curve failed due to scatter and less than three-fourths of the standards were within the acceptance criteria. However, the mean of the low QC results in the two runs ranged from 109.8 to 114.3% of target and the LLOQ samples for run 15 deviated less than ± 5% of target. Since there is insufficient sample volume to repeat the analyses, the client has requested that the <LLOQ results be reported as conditional information.

The criteria specified in the analytical plan for rejection of calibration standards was not followed for the M570 curve in run 12. Standard 1 , replicate 1 was deactivated as an outlier even though the % deviation from target was less than 2 times the acceptance criteria for that standard prior to deactivation.

As a general practice, both LLOQs are deactivated if both LLOQ results are outside of the specified acceptance criteria. For this run, when both LLOQs were deactivated, the calculated concentration of Std 1, replicate 1, deviated 61% from target and replicate 2 was 11% from target. This indicated that Std 1, repl 1 was a true outlier and Std 1, repl 2 was originally not fitting the curve because Std 1, repl 1 was skewing the curve. When

Std 1, repl 2 is reactivated, it has a deviation of 2% from target. Therefore, the regression will be accepted with only Std 1, repl 1 deactivated as an outlier. Since the regression is scientifically sound and acceptance criteria for the run are met, this deviation does not effect the quality of the data or integrity of the study.

5. REFERENCES

5.1. 3M Company Protocol EPI-0011. "Identification of Fluorochemicals in Sera of Children in the United States." March 16, 2000.

5.2. C. Sakashita. "Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS." Assay Revalidation Report. NWB Study NWBS00-040. NWB Report NWBR00-108. January 24, 2001.

5.3. C. Sakashita. "Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS." Assay Revalidation Addendum Report. NWB Study NWBS00-040. NWB Report NWBR00-122. November 20, 2001.

6. DATA RETENTION

The raw data and final report for this study will be stored in the NWB Archives, 1121 East 3900 South, Salt Lake City, UT 84124 per regulations and contract agreement. After submission of the final report to the Sponsor, remaining study samples will be stored under required conditions until confirmation of Sample Disposition/Return Authorization is received from the Sponsor. 3M Company will be notified concerning final disposition of records at completion of contract obligations.

Table 1. Calibration Curve Summary for PFOS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
31-Oct-2000	2	-0.000005	0.027791	0.010909	0.9956	3.94	414
01-Nov-2000	3	-0.000005	0.027419	0.006602	0.9990	3.94	414
04-Nov-2000	4	-0.000007	0.030558	0.012087	0.9961	3.94	414
06-Nov-2000	5	-0.000017	0.038499	0.015935	0.9953	3.94	414
08-Nov-2000	6	-0.000011	0.035306	0.007489	0.9960	3.94	414
09-Nov-2000	7	-0.000010	0.039703	0.010533	0.9894	3.94	414
10-Nov-2000	8	-0.000008	0.035218	0.033595	0.9921	3.94	414
12-Nov-2000	9	-0.000003	0.020611	0.020473	0.9912	3.94	414
13-Nov-2000	10	-0.000007	0.029955	0.022348	0.9918	3.94	414
14-Nov-2000	11	0.000005	0.026248	0.022175	0.9791	3.94	414
15-Nov-2000	12	-0.000005	0.041499	0.007161	0.9829	5.17	414
16-Nov-2000	13	-0.000003	0.036442	0.026638	0.9820	3.94	414
18-Nov-2000	14	-0.000006	0.034381	0.024135	0.9970	3.94	414
25-Mar-2001	17	-0.000001	0.008025	0.004145	0.9969	3.94	414
Mean		-0.000006	0.030833	0.016016	0.9917		
S.D.		0.000005	0.008779	0.008935	0.0063		
%CV		-83.3	28.5	55.8	0.6		
n		14	14	14	14		

A, B, and C are coefficients used to define the calibration curve.

030356

Table 2. Calibration Curve Summary for PFOA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
31-Oct-2000	2	0.000001	0.018750	0.001244	0.9967	1.92	481
01-Nov-2000	3	0.000002	0.018998	0.000186	0.9980	1.92	481
04-Nov-2000	4	0.000001	0.014032	0.001606	0.9945	1.92	481
06-Nov-2000	5	0.000000	0.014340	0.002050	0.9941	1.92	481
08-Nov-2000	6	-0.000001	0.014866	0.000095	0.9971	1.92	481
09-Nov-2000	7	-0.000002	0.014932	0.002985	0.9961	1.92	481
10-Nov-2000	8	0.000001	0.015103	0.009536	0.9941	1.92	481
12-Nov-2000	9	0.000001	0.010333	0.003017	0.9961	1.92	481
13-Nov-2000	10	0.000001	0.015660	0.002794	0.9936	1.92	481
14-Nov-2000	11	0.000008	0.013586	0.006940	0.9842	1.92	481
15-Nov-2000	12	0.000002	0.015457	0.005125	0.9903	1.92	481
16-Nov-2000	13	-0.000001	0.017638	0.007935	0.9922	1.92	481
18-Nov-2000	14	0.000000	0.020492	0.010441	0.9921	1.92	481
25-Mar-2001	17	0.000000	0.006597	0.002091	0.9921	1.92	481
Mean		0.000001	0.015056	0.004003	0.9937		
S.D.		0.000002	0.003547	0.003414	0.0035		
%CV		200.0	23.6	85.3	0.4		
n		14	14	14	14		

A, B, and C are coefficients used to define the calibration curve.

030357

Table 3. Calibration Curve Summary for PFHS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
31-Oct-2000	2	-0.000009	0.031737	0.006465	0.9976	1.36	523
01-Nov-2000	3	-0.000010	0.032478	0.009418	0.9979	1.36	523
04-Nov-2000	4	-0.000008	0.031233	0.014139	0.9965	1.36	523
06-Nov-2000	5	-0.000015	0.034135	0.015590	0.9952	1.36	523
08-Nov-2000	6	-0.000015	0.035218	0.009051	0.9964	1.36	523
09-Nov-2000	7	-0.000011	0.030154	0.011565	0.9977	1.36	523
10-Nov-2000	8	-0.000008	0.028971	0.027667	0.9946	1.36	523
12-Nov-2000	9	-0.000006	0.020349	0.010603	0.9983	1.36	523
13-Nov-2000	10	-0.000008	0.030472	0.025208	0.9979	1.36	523
14-Nov-2000	11	-0.000006	0.032776	0.025491	0.9964	1.36	523
15-Nov-2000	12	-0.000008	0.030669	0.025839	0.9887	1.36	523
16-Nov-2000	13	-0.000007	0.028744	0.027330	0.9930	1.36	523
18-Nov-2000	14	-0.000008	0.032203	0.024941	0.9922	1.36	523
25-Mar-2001	17	-0.000002	0.007658	0.007909	0.9937	1.36	523
Mean		-0.000009	0.029057	0.017230	0.9954		
S.D.		0.000003	0.007075	0.008299	0.0028		
%CV		-33.3	24.3	48.2	0.3		
n		14	14	14	14		

A, B, and C are coefficients used to define the calibration curve.

Table 4. Calibration Curve Summary for PFOSAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
31-Oct-2000	2	-0.000001	0.023946	-0.003099	0.9952	1.60	501
01-Nov-2000	3	0.000002	0.022379	0.004347	0.9940	1.60	501
04-Nov-2000	4	0.000000	0.016980	0.005491	0.9840	1.60	501
06-Nov-2000	5	0.000000	0.016621	0.003312	0.9827	1.60	501
08-Nov-2000	6	0.000000	0.018859	0.003795	0.9939	1.60	501
09-Nov-2000	7	0.000002	0.014599	0.003438	0.9901	1.60	501
10-Nov-2000	8	0.000004	0.017031	0.025660	0.9812	1.60	501
12-Nov-2000	9	0.000001	0.011371	0.000468	0.9982	1.60	501
13-Nov-2000	10	0.000004	0.019123	0.007920	0.9937	1.60	501
14-Nov-2000	11	0.000016	0.014974	0.016220	0.9802	1.60	501
15-Nov-2000	12	0.000004	0.012533	0.009564	0.9810	1.60	501
16-Nov-2000	13	0.000004	0.014478	0.010593	0.9859	1.60	501
20-Nov-2000	16	0.000003	0.020670	0.021239	0.9927	1.60	501
25-Mar-2001	17	0.000001	0.005029	0.010607	0.9928	1.60	501
Mean		0.000003	0.016328	0.008540	0.9890		
S.D.		0.000004	0.004833	0.007974	0.0062		
%CV		133.3	29.6	93.4	0.6		
n		14	14	14	14		

A, B, and C are coefficients used to define the calibration curve.

000359

Table 5. Calibration Curve Summary for PFOSA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
31-Oct-2000	2	-0.000013	0.052859	0.003517	0.9949	1.00	500
01-Nov-2000	3	-0.000003	0.046250	0.010476	0.9961	1.00	500
04-Nov-2000	4	-0.000012	0.054175	0.011593	0.9918	1.00	500
06-Nov-2000	5	-0.000018	0.069706	0.019030	0.9964	1.00	500
08-Nov-2000	6	-0.000015	0.060806	0.009414	0.9958	1.00	500
09-Nov-2000	7	-0.000013	0.072938	0.011198	0.9921	1.00	500
10-Nov-2000	8	-0.000011	0.067435	0.045288	0.9914	1.00	500
12-Nov-2000	9	-0.000007	0.036838	0.013009	0.9930	1.00	500
13-Nov-2000	10	-0.000005	0.051854	0.018661	0.9911	1.00	500
15-Nov-2000	12	-0.000009	0.077880	0.003529	0.9855	1.00	500
16-Nov-2000	13	-0.000007	0.069240	0.029141	0.9812	1.00	500
18-Nov-2000	14	-0.000010	0.064050	0.019102	0.9945	1.00	500
25-Mar-2001	17	0.000000	0.015780	0.005386	0.9945	1.00	500
Mean		-0.000009	0.056909	0.015334	0.9922		
S.D.		0.000005	0.016948	0.011547	0.0044		
%CV		-55.6	29.8	75.3	0.4		
n		13	13	13	13		

A, B, and C are coefficients used to define the calibration curve.

030360

Table 6. Calibration Curve Summary for M556

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
31-Oct-2000	2	-0.000001	0.018373	0.001923	0.9972	2.50	500
01-Nov-2000	3	0.000002	0.017583	0.005331	0.9972	2.50	500
04-Nov-2000	4	0.000002	0.007944	0.003887	0.9903	2.50	500
06-Nov-2000	5	0.000001	0.007717	0.004452	0.9822	2.50	500
08-Nov-2000	6	0.000002	0.009283	0.002596	0.9949	2.50	500
09-Nov-2000	7	0.000002	0.007610	0.002860	0.9985	2.50	500
10-Nov-2000	8	0.000002	0.008464	0.004466	0.9975	2.50	500
12-Nov-2000	9	0.000002	0.006106	0.003591	0.9968	2.50	500
13-Nov-2000	10	0.000003	0.008526	0.004733	0.9962	2.50	500
14-Nov-2000	11	0.000007	0.007051	0.006423	0.9867	2.50	500
15-Nov-2000	12	0.000003	0.006747	0.003029	0.9911	2.50	500
16-Nov-2000	13	0.000002	0.006485	0.002510	0.9936	2.50	500
18-Nov-2000	14	0.000002	0.008566	0.004178	0.9942	2.50	500
25-Mar-2001	17	0.000001	0.002650	0.001081	0.9955	2.50	500
Mean		0.000002	0.008793	0.003647	0.9937		
S.D.		0.000002	0.004209	0.001423	0.0047		
%CV		100.0	47.9	39.0	0.5		
n		14	14	14	14		

A, B, and C are coefficients used to define the calibration curve.

Table 7. Calibration Curve Summary for M570

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
31-Oct-2000	2	-0.000006	0.038561	0.012040	0.9924	1.00	500
01-Nov-2000	3	-0.000001	0.036248	0.019154	0.9952	1.00	500
04-Nov-2000	4	-0.000002	0.027976	0.012726	0.9860	1.00	500
06-Nov-2000	5	-0.000002	0.026862	0.016535	0.9908	1.00	500
08-Nov-2000	6	0.000000	0.030532	0.016991	0.9961	1.00	500
09-Nov-2000	7	0.000001	0.024223	0.014409	0.9863	1.00	500
10-Nov-2000	8	0.000002	0.028172	0.063424	0.9950	1.00	500
12-Nov-2000	9	-0.000002	0.018677	0.009480	0.9951	1.00	500
13-Nov-2000	10	0.000002	0.030589	0.027413	0.9938	1.00	500
14-Nov-2000	11	0.000020	0.025065	0.030430	0.9807	1.00	500
15-Nov-2000	12	0.000003	0.021180	0.014447	0.9847	1.00	500
16-Nov-2000	13	0.000006	0.022590	0.034515	0.9802	1.00	500
20-Nov-2000	16	0.000000	0.033466	0.044312	0.9914	1.00	500
25-Mar-2001	17	0.000000	0.002606	0.003896	0.9929	1.00	500
Mean		0.000002	0.026196	0.022841	0.9900		
S.D.		0.000006	0.008809	0.015953	0.0055		
%CV		300.0	33.6	69.8	0.6		
n		14	14	14	14		

A, B, and C are coefficients used to define the calibration curve.

000362

Table 8. Back-Calculated Concentrations of Calibration Standards for PFOS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	3.94	5.17	11.3	23.6	44.2	85.4	209	332	414
31-Oct-2000	2	3.65	5.67	12.0	23.4	45.0	84.2	224	347	424
		4.10	4.90	10.7	23.3	42.3	84.5	203	345	368
01-Nov-2000	3	4.00	5.15	11.4	23.7	46.7	84.5	198	329	422
		3.94	5.04	11.4	23.9	43.4	83.6	207	351	405
04-Nov-2000	4	4.16	5.31	10.5	21.2	44.9	83.7	205	340	411
		3.70	5.05	12.6	24.1	45.7	86.9	213	332	409
06-Nov-2000	5	3.97	5.37	12.4	25.7	45.3	82.8	203	352	428
		3.83	4.96	10.7	23.2	43.5	77.3	*116	*	399
08-Nov-2000	6	3.96	5.74	11.1	25.6	44.5	87.0	215	350	436
		3.69	5.02	11.3	22.8	42.9	81.8	193	333	386
09-Nov-2000	7	4.26	5.56	12.2	26.7	46.7	89.3	227	357	433
		3.56	4.85	10.3	22.2	40.9	77.1	184	338	376
10-Nov-2000	8	4.57	4.83	11.9	24.8	47.0	86.6	212	353	418
		**7.53	4.35	11.0	22.5	41.1	87.8	207	325	390
12-Nov-2000	9	3.67	5.25	12.0	25.1	49.0	89.1	220	339	443
		4.53	4.57	10.2	23.0	42.0	79.6	195	321	392
13-Nov-2000	10	3.84	6.11	12.3	24.2	45.1	85.5	222	344	401
		3.76	4.53	11.3	24.0	40.4	75.9	208	332	413
14-Nov-2000	11	4.56	5.74	11.8	26.3	50.7	90.5	**95.5	354	401
		3.26	4.83	9.79	21.9	**28.5	64.6	210	354	391
15-Nov-2000	12	†4.93	5.81	10.8	25.9	47.0	96.6	245	374	456
		†3.23	4.86	10.1	20.9	43.6	77.6	184	294	354
16-Nov-2000	13	3.31	4.34	10.3	22.6	43.8	75.1	201	313	391
		4.83	5.52	12.0	26.2	50.4	79.5	233	330	452

* Sample deactivated due to preparation error
** Sample deactivated as an outlier
† LLOQ failed

Table 8. Back-Calculated Concentrations of Calibration Standards for PFOS (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	3.94	5.17	11.3	23.6	44.2	85.4	209	332	414
18-Nov-2000	14	3.88	5.45	11.0	23.1	44.6	83.3	206	357	388
		4.03	4.71	12.3	24.2	42.4	85.9	208	333	422
25-Mar-2001	17	3.98	4.71	11.1	25.5	44.7	81.6	211	344	423
		4.26	4.93	11.4	24.0	44.7	82.6	202	344	390
Mean		3.97	5.11	11.3	23.9	44.8	83.0	209	340	408
S.D.		0.383	0.461	0.784	1.55	2.63	6.03	13.9	16.0	24.6
%CV		9.6	9.0	6.9	6.5	5.9	7.3	6.7	4.7	6.0
%Bias		0.8	-1.2	0.0	1.3	1.4	-2.8	0.0	2.4	-1.4
n		25	28	28	28	27	28	26	27	28

Table 9. Back-Calculated Concentrations of Calibration Standards for PFOA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.92	3.36	10.6	25.0	49.0	97.1	241	385	481
31-Oct-2000	2	1.94	3.49	9.80	24.9	47.2	89.3	250	383	470
		1.88	3.36	10.7	26.6	49.5	99.2	247	427	443
01-Nov-2000	3	1.85	3.30	10.0	24.0	51.6	98.9	227	381	472
		1.94	3.64	10.5	25.7	48.7	95.3	249	406	478
04-Nov-2000	4	2.05	3.27	9.53	21.4	49.2	90.5	245	385	456
		1.80	3.49	11.0	27.9	51.0	102	253	398	478
06-Nov-2000	5	1.89	3.14	10.6	25.0	47.0	84.0	234	374	482
		1.92	3.63	10.5	27.3	55.5	92.8	*164	*	509
08-Nov-2000	6	2.03	3.22	9.85	27.5	49.6	97.8	253	382	481
		1.98	3.03	10.3	25.1	50.8	95.0	237	387	471
09-Nov-2000	7	2.17	3.06	10.4	25.8	49.2	92.7	244	394	501
		1.77	3.34	10.8	25.6	50.2	95.0	243	399	437
10-Nov-2000	8	2.17	2.75	9.73	26.1	49.5	95.4	238	386	484
		**6.15	3.22	11.2	24.5	50.9	103	255	378	467
12-Nov-2000	9	2.02	3.53	11.0	23.9	51.0	94.5	235	365	477
		**3.54	2.92	10.0	24.3	52.3	99.6	251	416	467
13-Nov-2000	10	2.22	3.19	10.8	26.1	52.2	93.0	249	412	463
		1.77	2.97	10.6	27.2	47.1	87.0	242	386	473
14-Nov-2000	11	2.30	3.92	10.6	27.2	53.6	96.8	**57.3	381	445
		1.59	2.72	9.75	24.0	**15.4	83.8	258	418	482
15-Nov-2000	12	2.28	3.16	9.24	25.1	49.5	90.5	233	339	494
		1.70	3.22	10.7	25.3	56.9	102	246	415	480
16-Nov-2000	13	1.82	3.28	10.4	27.0	51.3	96.7	233	372	466
		2.22	2.81	10.3	26.0	52.9	86.4	253	369	523

* Sample deactivated due to preparation error
** Sample deactivated as an outlier

Table 9. Back-Calculated Concentrations of Calibration Standards for PFOA (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.92	3.36	10.6	25.0	49.0	97.1	241	385	481
18-Nov-2000	14	1.77	3.29	9.90	25.1	48.7	96.5	243	404	441
		2.20	2.90	12.5	25.6	46.1	93.9	248	398	486
25-Mar-2001	17	1.85	2.67	10.4	27.1	49.4	95.5	234	395	474
		2.26	3.15	10.9	25.9	50.4	94.2	241	416	455
Mean		1.98	3.20	10.4	25.6	50.4	94.3	244	391	473
S.D.		0.199	0.298	0.634	1.40	2.47	5.07	8.09	19.6	19.7
%CV		10.1	9.3	6.1	5.5	4.9	5.4	3.3	5.0	4.2
%Bias		3.1	-4.8	-1.9	2.4	2.9	-2.9	1.2	1.6	-1.7
n		26	28	28	28	27	28	26	27	28

Table 10. Back-Calculated Concentrations of Calibration Standards for PFHS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.36	2.92	10.7	26.5	52.6	104	262	418	523
31-Oct-2000	2	1.30	3.01	11.2	26.0	49.7	98.7	268	419	530
		1.42	2.77	11.2	27.4	52.0	102	266	455	478
01-Nov-2000	3	1.36	2.73	11.2	25.8	51.1	104	238	417	513
		1.35	3.12	10.6	27.1	55.7	100	268	442	532
04-Nov-2000	4	1.43	3.08	9.86	28.5	51.2	96.4	247	432	513
		1.31	2.67	10.7	28.7	53.1	106	271	424	524
06-Nov-2000	5	1.40	2.86	11.0	26.6	51.5	87.1	252	431	515
		1.36	2.72	11.5	28.8	55.5	102	*167	*	554
08-Nov-2000	6	1.44	2.85	11.4	28.3	52.4	101	260	425	547
		1.24	3.16	10.1	25.7	53.8	96.3	247	430	516
09-Nov-2000	7	1.44	2.80	11.2	26.8	51.9	101	256	409	518
		1.26	3.05	11.0	26.4	52.3	101	251	468	515
10-Nov-2000	8	1.51	2.37	10.9	26.4	53.1	103	250	413	537
		**5.46	2.69	11.1	28.1	54.0	107	272	413	515
12-Nov-2000	9	1.35	2.88	10.6	25.4	51.4	96.8	256	391	529
		**3.07	2.97	10.9	28.3	55.3	103	270	439	530
13-Nov-2000	10	1.35	2.85	11.1	26.4	52.5	102	262	432	520
		1.41	2.73	11.3	27.9	48.8	99.6	273	434	492
14-Nov-2000	11	1.45	2.61	10.7	27.4	50.4	104	**79.3	416	488
		1.32	2.95	10.8	28.0	**23.7	101	269	469	503
15-Nov-2000	12	1.55	2.74	11.4	29.4	55.4	120	276	475	561
		1.30	2.47	10.2	25.4	51.9	91.9	237	370	482
16-Nov-2000	13	1.23	2.66	11.2	27.3	55.1	104	270	407	521
		1.59	2.64	10.8	27.3	56.3	89.6	255	420	542

* Sample deactivated due to preparation error
** Sample deactivated as an outlier

Table 10. Back-Calculated Concentrations of Calibration Standards for PFHS (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.36	2.92	10.7	26.5	52.6	104	262	418	523
18-Nov-2000	14	1.26	2.98	10.1	26.3	53.8	97.8	264	428	534
		1.48	2.61	13.3	26.6	50.3	99.7	253	407	534
25-Mar-2001	17	1.39	2.46	10.4	28.0	55.4	103	277	440	521
		1.52	2.52	10.6	27.5	55.3	103	258	427	474
Mean		1.39	2.78	10.9	27.2	52.9	101	260	427	519
S.D.		0.0963	0.208	0.631	1.09	2.07	5.94	11.2	22.9	21.7
%CV		6.9	7.5	5.8	4.0	3.9	5.9	4.3	5.4	4.2
%Bias		2.2	-4.8	1.9	2.6	0.6	-2.9	-0.8	2.2	-0.8
n		26	28	28	28	27	28	26	27	28

Table 11. Back-Calculated Concentrations of Calibration Standards for PFOSAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.60	3.10	10.6	25.6	50.6	101	251	401	501
31-Oct-2000	2	1.41	3.26	10.7	24.0	48.1	94.6	271	394	487
		1.68	3.36	10.9	25.4	48.1	106	261	433	467
01-Nov-2000	3	1.66	2.85	9.40	25.1	55.4	97.5	248	375	495
		1.54	3.50	9.99	26.9	48.8	107	261	443	481
04-Nov-2000	4	1.58	3.53	8.37	19.1	50.2	94.5	243	392	461
		1.56	3.00	12.0	28.9	54.9	107	285	429	496
06-Nov-2000	5	1.24	2.77	10.1	23.3	45.8	95.8	242	379	494
		1.94	3.56	10.4	27.7	55.2	115	*186	*	532
08-Nov-2000	6	1.52	2.88	8.73	26.6	47.4	96.4	257	432	501
		1.72	3.23	11.7	26.7	51.7	106	255	392	468
09-Nov-2000	7	1.46	2.84	9.64	24.8	45.8	90.1	260	389	449
		1.71	3.48	11.5	27.8	53.0	102	253	472	495
10-Nov-2000	8	1.98	2.13	9.82	25.8	54.7	98.0	253	403	463
		**8.58	2.70	10.6	24.6	50.3	122	289	396	489
12-Nov-2000	9	1.61	3.09	10.1	24.6	50.3	99.6	250	376	487
		**2.67	3.15	10.2	27.0	51.5	104	269	420	504
13-Nov-2000	10	1.59	3.22	11.1	24.2	50.2	98.0	275	402	424
		1.60	2.96	11.2	26.3	44.9	99.5	264	403	549
14-Nov-2000	11	1.54	3.40	9.75	28.5	59.2	105	**138	391	445
		1.53	3.39	9.65	26.4	37.1	82.5	284	447	507
15-Nov-2000	12	1.85	2.88	7.98	22.3	44.4	99.5	270	364	465
		1.40	3.33	10.9	25.3	62.0	113	284	437	483
16-Nov-2000	13	1.72	2.88	11.9	28.4	56.9	93.4	282	419	502
		**5.99	2.81	9.59	25.7	48.4	80.6	281	336	506
20-Nov-2000	16	1.52	3.14	9.31	23.8	47.1	98.1	253	413	447
		1.72	2.88	12.4	27.4	48.8	107	267	407	520

* Sample deactivated due to preparation error
** Sample deactivated as an outlier

030369

Table 11. Back-Calculated Concentrations of Calibration Standards for PFOSAA (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.60	3.10	10.6	25.6	50.6	101	251	401	501
25-Mar-2001	17	1.67	2.93	10.8	29.8	52.5	103	255	420	500
		1.71	2.54	10.5	24.2	49.1	94.5	249	429	456
Mean		1.62	3.06	10.3	25.7	50.4	100	264	407	485
S.D.		0.164	0.333	1.08	2.24	5.03	8.84	14.0	29.0	27.9
%CV		10.1	10.9	10.5	8.7	10.0	8.8	5.3	7.1	5.8
%Bias		1.3	-1.3	-2.8	0.4	-0.4	-1.0	5.2	1.5	-3.2
n		25	28	28	28	28	28	26	27	28

Table 12. Back-Calculated Concentrations of Calibration Standards for PFOSA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
31-Oct-2000	2	0.930	2.62	9.96	25.7	52.8	101	289	438	529
		1.06	2.48	9.45	24.1	47.2	100	229	362	453
01-Nov-2000	3	0.913	2.40	8.86	25.1	52.1	99.1	238	380	497
		1.10	2.56	9.78	26.7	50.4	104	255	432	491
04-Nov-2000	4	1.00	2.64	9.23	19.1	51.8	101	248	386	494
		0.998	2.33	11.5	26.1	53.7	105	241	399	525
06-Nov-2000	5	0.934	2.63	10.1	26.9	48.7	104	248	395	521
		1.03	2.60	9.67	25.7	48.4	86.1	*138	*	499
08-Nov-2000	6	0.992	2.63	8.92	28.0	49.4	104	261	413	545
		0.985	2.57	9.69	26.0	46.9	97.0	235	368	482
09-Nov-2000	7	1.04	2.71	10.4	28.9	50.5	105	271	444	521
		0.945	2.38	8.88	24.5	47.2	91.4	215	394	461
10-Nov-2000	8	1.12	2.15	10.7	27.9	52.5	103	259	424	522
		**1.67	2.04	10.1	23.6	47.1	105	252	366	467
12-Nov-2000	9	0.979	2.58	11.4	25.8	56.6	107	251	422	541
		1.00	2.53	9.14	22.1	45.8	91.7	232	385	473
13-Nov-2000	10	1.12	2.81	10.8	26.6	53.1	98.2	258	426	477
		0.850	2.32	9.74	25.9	43.7	85.7	245	390	521
15-Nov-2000	12	**1.61	3.03	8.79	26.1	50.1	114	294	416	544
		0.921	2.62	8.99	21.3	52.4	91.2	232	388	422
16-Nov-2000	13	0.788	2.06	8.94	24.4	47.6	86.3	234	385	479
		1.22	2.85	10.8	29.0	54.2	95.7	286	382	547
18-Nov-2000	14	0.965	2.39	10.0	24.9	47.2	102	240	466	438
		1.04	2.59	10.2	27.5	45.2	95.6	276	392	495

* Sample deactivated due to preparation error
** Sample deactivated as an outlier

Table 12. Back-Calculated Concentrations of Calibration Standards for PFOSA (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
25-Mar-2001	17	0.900	2.36	10.3	28.0	48.3	101	236	410	505
		1.14	2.30	10.3	25.8	48.4	95.1	241	432	479
Mean		0.999	2.51	9.87	25.6	49.7	98.8	251	404	497
S.D.		0.0978	0.232	0.781	2.28	3.14	7.01	20.0	26.7	33.7
%CV		9.8	9.2	7.9	8.9	6.3	7.1	8.0	6.6	6.8
%Bias		-0.1	0.4	-1.3	2.4	-0.6	-1.2	0.4	1.0	-0.6
n		24	26	26	26	26	26	25	25	26

Table 13. Back-Calculated Concentrations of Calibration Standards for M556

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	2.50	10.0	25.0	50.0	100	250	400	500
31-Oct-2000	2	2.33	9.67	24.3	51.9	95.2	255	388	499
		2.70	9.81	25.8	49.3	104	249	438	470
01-Nov-2000	3	2.55	9.86	25.9	55.5	107	246	403	505
		2.48	9.52	23.6	47.4	96.2	241	421	478
04-Nov-2000	4	2.38	8.49	19.4	53.2	96.1	248	388	485
		2.63	11.8	26.4	52.2	104	263	416	500
06-Nov-2000	5	2.02	8.91	22.7	46.0	86.0	235	385	492
		2.98	10.7	28.8	58.2	107	*200	*	538
08-Nov-2000	6	2.28	8.40	26.1	46.3	98.1	255	413	494
		2.75	11.0	25.7	51.4	102	250	409	481
09-Nov-2000	7	2.51	9.73	25.7	49.3	95.5	253	400	488
		2.50	9.91	25.5	51.8	100	238	432	490
10-Nov-2000	8	2.37	9.94	23.7	47.6	97.9	244	402	487
		2.63	10.3	24.0	50.9	109	271	394	500
12-Nov-2000	9	2.61	9.22	23.4	50.6	97.8	233	376	499
		2.43	9.95	26.7	51.0	107	264	421	502
13-Nov-2000	10	2.52	10.3	25.5	52.3	96.5	261	429	458
		2.43	10.4	26.0	46.8	88.5	248	403	516
14-Nov-2000	11	2.77	9.58	27.2	57.3	103	**127	392	453
		2.33	8.48	24.2	**33.4	81.4	272	439	505
15-Nov-2000	12	2.46	8.06	22.9	46.3	105	276	388	483
		2.66	10.1	23.9	57.7	104	260	427	464
16-Nov-2000	13	2.70	10.0	27.2	53.3	92.8	255	414	499
		2.32	9.10	25.3	52.2	87.3	271	359	513
18-Nov-2000	14	2.69	9.91	23.3	48.0	96.4	242	434	454
		2.25	11.4	26.6	48.1	97.0	256	420	503

* Sample deactivated due to preparation error
** Sample deactivated as an outlier

Table 13. Back-Calculated Concentrations of Calibration Standards for M556 (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	2.50	10.0	25.0	50.0	100	250	400	500
25-Mar-2001	17	2.71	10.1	27.4	51.0	99.1	247	420	507
		2.26	10.4	23.7	47.9	94.0	240	431	460
Mean		2.51	9.82	25.0	50.9	98.1	253	409	490
S.D.		0.205	0.872	1.90	3.49	6.83	11.9	20.5	20.2
%CV		8.2	8.9	7.6	6.9	7.0	4.7	5.0	4.1
%Bias		0.4	-1.8	0.0	1.8	-1.9	1.2	2.3	-2.0
n		28	28	28	27	28	26	27	28

Table 14. Back-Calculated Concentrations of Calibration Standards for M570

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
31-Oct-2000	2	0.828	2.29	10.1	23.6	48.1	93.7	262	392	486
		1.18	2.66	9.94	25.9	50.6	109	252	431	475
01-Nov-2000	3	1.07	2.22	9.01	25.2	54.1	100	239	383	485
		0.992	2.43	9.48	27.2	50.1	106	259	441	482
04-Nov-2000	4	0.974	2.74	8.15	18.7	49.4	96.4	239	389	466
		1.02	2.29	11.8	28.0	55.0	105	269	427	506
06-Nov-2000	5	0.930	2.09	9.52	23.9	44.9	97.3	234	384	486
		1.10	2.75	10.3	26.6	56.0	110	*183	*	538
08-Nov-2000	6	0.952	2.50	8.31	25.8	49.0	100	251	404	496
		1.08	2.35	10.6	25.7	51.9	107	249	404	488
09-Nov-2000	7	0.805	2.08	9.40	24.8	45.9	94.4	247	392	452
		1.23	2.66	10.6	27.0	53.8	105	246	463	499
10-Nov-2000	8	1.01	**0.653	9.16	24.3	52.0	98.0	240	403	468
		**6.51	**1.29	9.43	23.5	50.3	118	276	402	498
12-Nov-2000	9	0.964	2.40	9.29	23.1	48.8	96.7	235	375	482
		**1.93	2.89	9.84	25.7	53.4	105	264	432	510
13-Nov-2000	10	1.05	2.33	10.7	24.0	50.3	100	258	407	437
		1.03	2.13	10.9	27.1	45.7	98.2	258	401	543
14-Nov-2000	11	1.10	2.78	9.29	26.7	57.0	104	**133	399	440
		0.813	2.87	9.11	25.9	37.9	81.5	276	444	508
15-Nov-2000	12	**1.53	2.39	7.28	22.1	45.2	101	259	369	457
		1.02	2.62	10.3	25.2	62.8	112	269	439	495
16-Nov-2000	13	1.16	1.93	10.8	28.4	57.9	100	276	422	497
		1.03	1.83	9.19	26.1	52.0	83.4	275	340	495
20-Nov-2000	16	0.933	2.61	9.04	24.1	46.9	96.9	241	423	449
		1.10	2.13	12.0	26.1	48.4	107	260	404	523

* Sample deactivated due to preparation error
** Sample deactivated as an outlier

Table 14. Back-Calculated Concentrations of Calibration Standards for M570 (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
25-Mar-2001	17	1.03	1.98	9.84	29.0	52.3	102	252	424	508
		1.05	**1.64	10.0	24.8	50.4	90.5	241	419	458
Mean		1.02	2.40	9.76	25.3	50.7	101	255	408	487
S.D.		0.106	0.304	1.03	2.09	4.84	7.92	13.4	26.4	26.9
%CV		10.4	12.7	10.6	8.3	9.5	7.8	5.3	6.5	5.5
%Bias		2.0	-4.0	-2.4	1.2	1.4	1.0	2.0	2.0	-2.6
n		25	25	28	28	28	28	26	27	28

Table 15. Analytical QC Summary for PFOS

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 6.40 ppb	Medium QC 126 ppb	High QC 332 ppb	Dilution QC 335 ppb DF=2
31-Oct-2000	2	6.62	129	323	
		6.18	135	322	
01-Nov-2000	3	6.79	125	369	
		7.16	124	354	
04-Nov-2000	4	6.57	131	314	
		6.63	130	322	
06-Nov-2000	5	6.03	105	291	
		5.30	122	283	
08-Nov-2000	6	6.70	147	343	
		5.98	128	320	
09-Nov-2000	7	6.00	137	302	
		5.96	108	293	
10-Nov-2000	8	6.36	136	323	
		6.38	134	318	
12-Nov-2000	9	6.18	132	299	
		6.31	127	304	
13-Nov-2000	10	6.71	139	327	
		6.38	136	312	
14-Nov-2000	11	5.31	136	322	
		6.73	133	294	
15-Nov-2000	12	6.50	141	319	320
		5.84	103	330	323
					350

Table 15. Analytical QC Summary for PFOS (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 6.40 ppb	Medium QC 126 ppb	High QC 332 ppb	Dilution QC 335 ppb DF=2
16-Nov-2000	13	6.65	126	297	307
		5.98	129	319	312
					359
18-Nov-2000	14	5.87	130	338	335
		6.06	126	336	342
					345
25-Mar-2001	17	6.26	137	367	335
		5.95	135	349	338
					471
Mean		6.26	129	321	345
S.D.		0.430	10.1	22.1	42.6
%CV		6.9	7.8	6.9	12.3
%Theoretical		97.8	102.4	96.7	103.0
%Bias		-2.2	2.4	-3.3	3.0
n		28	28	28	12

Table 16. Analytical QC Summary for PFOA

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.81 ppb	Medium QC 145 ppb	High QC 385 ppb	Dilution QC 386 ppb DF=2
31-Oct-2000	2	4.87	156	399	
		4.90	147	380	
01-Nov-2000	3	4.78	144	396	
		4.68	144	408	
04-Nov-2000	4	5.51	149	384	
		5.09	159	383	
06-Nov-2000	5	4.45	140	342	
		5.10	147	368	
08-Nov-2000	6	4.81	154	423	
		4.18	159	388	
09-Nov-2000	7	4.22	147	369	
		4.15	130	356	
10-Nov-2000	8	4.23	154	375	
		4.66	160	385	
12-Nov-2000	9	4.72	151	382	
		5.45	150	353	
13-Nov-2000	10	5.05	159	391	
		4.80	158	353	
14-Nov-2000	11	4.19	153	371	
		4.67	155	371	
15-Nov-2000	12	4.09	158	419	396
		4.50	127	422	425
					406
16-Nov-2000	13	4.43	143	420	388
		4.65	160	421	391
					402

Table 16. Analytical QC Summary for PFOA (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.81 ppb	Medium QC 145 ppb	High QC 385 ppb	Dilution QC 386 ppb DF=2
18-Nov-2000	14	3.92	147	386	392
		3.90	151	394	416
					381
25-Mar-2001	17	4.28	163	412	404
		4.06	159	422	401
					449
Mean		4.58	151	388	404
S.D.		0.432	8.80	23.6	18.6
%CV		9.4	5.8	6.1	4.6
%Theoretical		95.2	104.1	100.8	104.7
%Bias		-4.8	4.1	0.8	4.7
n		28	28	28	12

Table 17. Analytical QC Summary for PFHS

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.48 ppb	Medium QC 156 ppb	High QC 417 ppb	Dilution QC 418 ppb DF=2
31-Oct-2000	2	4.35	160	403	
		4.60	151	400	
01-Nov-2000	3	4.34	144	420	
		4.44	146	432	
04-Nov-2000	4	4.37	163	404	
		4.40	160	409	
06-Nov-2000	5	4.10	144	364	
		4.14	150	386	
08-Nov-2000	6	3.99	157	404	
		3.89	157	413	
09-Nov-2000	7	4.09	154	371	
		4.67	134	397	
10-Nov-2000	8	3.76	162	408	
		4.00	158	404	
12-Nov-2000	9	3.99	154	391	
		4.57	166	410	
13-Nov-2000	10	4.32	171	404	
		4.15	158	435	
14-Nov-2000	11	4.08	167	385	
		3.80	155	396	
15-Nov-2000	12	4.06	164	373	367
		3.84	150	400	365
					378
16-Nov-2000	13	3.63	146	401	418
		4.12	167	396	431
					401

Table 17. Analytical QC Summary for PFHS (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.48 ppb	Medium QC 156 ppb	High QC 417 ppb	Dilution QC 418 ppb DF=2
18-Nov-2000	14	3.74	155	454	391
		3.85	148	397	401
					414
25-Mar-2001	17	3.85	153	448	390
		3.72	153	427	403
					449
Mean		4.10	155	405	401
S.D.		0.287	8.40	21.0	25.0
%CV		7.0	5.4	5.2	6.2
%Theoretical		91.5	99.4	97.1	95.9
%Bias		-8.5	-0.6	-2.9	-4.1
n		28	28	28	12

Table 18. Analytical QC Summary for PFOSAA

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.60 ppb	Medium QC 151 ppb	High QC 401 ppb	Dilution QC 401 ppb DF=2
31-Oct-2000	2	4.43	160	393	
		4.03	157	394	
01-Nov-2000	3	*5.84	150	485	
		5.21	172	488	
04-Nov-2000	4	4.26	151	391	
		4.10	162	388	
06-Nov-2000	5	3.77	136	366	
		4.18	138	356	
08-Nov-2000	6	5.22	176	443	
		4.35	157	434	
09-Nov-2000	7	3.50	153	359	
		3.93	128	409	
10-Nov-2000	8	4.23	163	415	
		4.55	181	430	
12-Nov-2000	9	4.15	153	380	
		5.55	172	389	
13-Nov-2000	10	5.24	151	415	
		5.06	172	323	
14-Nov-2000	11	3.71	168	399	
		4.52	150	371	
15-Nov-2000	12	*3.39	158	451	419
		3.70	116	427	491
					422
16-Nov-2000	13	4.10	137	419	401
		4.15	157	412	399
					424

* >± 25% deviation from theoretical

000383

Table 18. Analytical QC Summary for PFOSAA (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.60 ppb	Medium QC 151 ppb	High QC 401 ppb	Dilution QC 401 ppb DF=2
20-Nov-2000	16	3.81	164	361	411
		*3.24	155	472	414
					397
25-Mar-2001	17	4.86	173	429	426
		3.72	173	420	422
					*634
Mean		4.31	157	408	438
S.D.		0.667	15.3	39.4	66.3
%CV		15.5	9.7	9.7	15.1
%Theoretical		93.7	104.0	101.7	109.2
%Bias		-6.3	4.0	1.7	9.2
n		28	28	28	12

* >± 25% deviation from theoretical

Table 19. Analytical QC Summary for PFOSA

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb	Dilution QC 400 ppb DF=2
31-Oct-2000	2	3.74	154	423	
		4.10	158	412	
01-Nov-2000	3	4.72	151	488	
		*5.03	161	471	
04-Nov-2000	4	4.16	147	401	
		4.04	158	433	
06-Nov-2000	5	3.66	127	357	
		3.49	155	342	
08-Nov-2000	6	4.75	183	462	
		4.15	145	423	
09-Nov-2000	7	4.05	164	371	
		3.70	126	350	
10-Nov-2000	8	4.38	166	419	
		4.58	164	412	
12-Nov-2000	9	4.65	163	363	
		4.20	153	363	
13-Nov-2000	10	4.76	172	422	
		4.74	172	357	
15-Nov-2000	12	4.27	171	432	421
		3.88	*111	443	455
					475
16-Nov-2000	13	4.54	150	369	350
		3.72	149	391	360
					453

* >± 25% deviation from theoretical

000385

Table 19. Analytical QC Summary for PFOSA (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb	Dilution QC 400 ppb DF=2
18-Nov-2000	14	4.28	156	385	428
		4.23	147	417	423
					397
25-Mar-2001	17	4.50	180	450	437
		4.07	180	444	438
					*552
Mean		4.24	156	407	430
S.D.		0.399	16.6	39.4	51.3
%CV		9.4	10.6	9.7	11.9
%Theoretical		106.0	104.0	101.8	107.5
%Bias		6.0	4.0	1.8	7.5
n		26	26	26	12

* >± 25% deviation from theoretical

030386

Table 20. Analytical QC Summary for M556

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb	Dilution QC 400 ppb DF=2
31-Oct-2000	2	3.95	161	417	
		3.93	149	406	
01-Nov-2000	3	*4.81	163	449	
		4.62	171	*492	
04-Nov-2000	4	4.08	156	397	
		3.70	163	393	
06-Nov-2000	5	3.57	141	373	
		3.30	146	383	
08-Nov-2000	6	4.74	172	444	
		3.63	160	431	
09-Nov-2000	7	3.63	155	367	
		4.11	132	398	
10-Nov-2000	8	4.25	161	417	
		4.18	*182	423	
12-Nov-2000	9	3.56	158	384	
		4.00	176	404	
13-Nov-2000	10	4.49	164	421	
		4.29	175	357	
14-Nov-2000	11	3.36	163	394	
		4.51	157	384	
15-Nov-2000	12	*3.14	156	427	402
		3.56	*112	420	464
					416
16-Nov-2000	13	3.98	139	416	397
		3.81	154	407	385
					427

* >± 20% deviation from theoretical

Table 20. Analytical QC Summary for M556 (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb	Dilution QC 400 ppb DF=2
18-Nov-2000	14	3.87	150	382	416
		3.69	157	424	419
					406
25-Mar-2001	17	4.09	168	455	403
		3.52	162	434	392
					*627
Mean		3.94	157	411	430
S.D.		0.437	14.3	29.3	65.5
%CV		11.1	9.1	7.1	15.2
%Theoretical		98.5	104.7	102.8	107.5
%Bias		-1.5	4.7	2.8	7.5
n		28	28	28	12

* >± 20% deviation from theoretical

Table 21. Analytical QC Summary for M570

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb	Dilution QC 400 ppb DF=2
31-Oct-2000	2	3.70	164	410	
		3.99	160	416	
01-Nov-2000	3	4.52	152	476	
		*5.13	178	*491	
04-Nov-2000	4	3.68	147	400	
		4.06	165	385	
06-Nov-2000	5	*3.10	133	362	
		3.60	146	379	
08-Nov-2000	6	4.48	*182	446	
		3.85	161	422	
09-Nov-2000	7	*3.13	154	364	
		3.51	131	409	
10-Nov-2000	8	*2.97	165	421	
		3.37	*184	427	
12-Nov-2000	9	3.94	154	384	
		4.30	173	397	
13-Nov-2000	10	4.12	158	420	
		*4.85	176	326	
14-Nov-2000	11	3.43	166	408	
		4.32	154	383	
15-Nov-2000	12	3.29	155	452	414
		3.64	*114	429	*490
					424
16-Nov-2000	13	3.25	141	423	413
		3.65	161	415	399
					435

* >± 20% deviation from theoretical

Table 21. Analytical QC Summary for M570 (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb	Dilution QC 400 ppb DF=2
20-Nov-2000	16	*2.76	169	372	415
		3.33	151	*484	421
					391
25-Mar-2001	17	3.20	165	461	383
		*2.89	159	411	400
					*648
Mean		3.72	158	413	436
S.D.		0.596	15.6	38.1	72.1
%CV		16.0	9.9	9.2	16.5
%Theoretical		93.0	105.3	103.3	109.0
%Bias		-7.0	5.3	3.3	9.0
n		28	28	28	12

* >± 20% deviation from theoretical

Table 22. Study Sample Concentrations

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0021	11.1	2.13	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0056	27.1	5.79	3.72	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0087	18.1	3.95	1.99	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	5.15
0104	67.3	6.14	3.82	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	3.28
0109	19.4	2.65	6.12	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	6.55
0122	36.8	3.35	4.28	4.54	<LLOQ(1.00)	2.7	<LLOQ(1.00)
0123	29.0	3.37	<LLOQ(1.36)	3.03	<LLOQ(1.00)	4.62	2.37
0126	39.6	3.77	10.4	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.74
0127	42.8	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
0128	81.2	5.60	21.2	5.50	<LLOQ(1.00)	7.33	4.81
0131	68.0	4.68	28.1	5.45	<LLOQ(1.00)	6.1	1.53
0133	65.2	6.91	21.6	2.11	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0136	83.8	8.65	180	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0149	39.3	2.94	25.7	2.33	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0151	29.8	5.00	11.0	3.11	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0167	77.6	7.70	1.81	6.32	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0174	36.1	3.85	7.09	4.06	<LLOQ(1.00)	<LLOQ(2.50)	1.91

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0187	31.1	3.56	<LLOQ(1.36)	3.69	<LLOQ(1.00)	<LLOQ(2.50)	2.04
0192	35.7	6.93	6.05	6.66	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0193	90.2	6.41	71.3	3.14	<LLOQ(1.00)	3.58	<LLOQ(1.00)
0194	24.5	4.44	1.41	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0196	11.6	1.95	<LLOQ(1.36)	1.64	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0197	38.9	5.46	<LLOQ(1.36)	4.95	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0198	71.9	7.43	<LLOQ(1.36)	4.12	<LLOQ(1.00)	5.94	7.26
0199	54.8	7.37	30.0	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	2.42
0210	33.9	5.44	4.14	4.76	<LLOQ(2.00)	7.16	5.03
0216	38.2	4.79	3.12	2.42	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0217	43.6	5.16	6.86	5.34	<LLOQ(1.00)	5.59	<LLOQ(1.00)
0228	16.6	3.89	6.83	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0229	122	10.4	145	4.93	<LLOQ(1.00)	4.35	<LLOQ(1.00)
0230	No result (Insufficient sample volume)	No result (Insufficient sample volume)	No result (Insufficient sample volume)	No result (Insufficient sample volume)	No result (Insufficient sample volume)	No result (Insufficient sample volume)	No result (Insufficient sample volume)
0231	93.9	7.8	17.8	6.07	<LLOQ(1.00)	8.55	23

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0247	42.4	2.92	11.4	3.19	<LLOQ(1.00)	2.88	1.06
0250	37.7	6.11	2.51	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.67
0251	51.3	4.32	16.1	3.11	<LLOQ(1.00)	4.94	3.11
0259	38.3	5.07	11.3	2.04	<LLOQ(1.00)	5.88	<LLOQ(1.00)
0269	92.3	8.71	75.4	15.3	<LLOQ(1.00)	8.4	5.16
0270	25.7	5.09	<LLOQ(1.36)	4.54	<LLOQ(1.00)	4.42	1
0290	28.7	4.08	3.23	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.53
0296	23.3	4.28	<LLOQ(1.36)	2.62	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0308	23.7	3.56	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	3.54	1.36
0315	94.2	4.74	11.2	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.15
0329	29.6	2.66	4.60	<LLOQ(1.60)	<LLOQ(1.00)	3.21	2
0341	53.0	4.89	2.33	6.61	<LLOQ(1.00)	3.77	2.59
0342	38.3	5.88	<LLOQ(1.36)	1.98	<LLOQ(1.00)	<LLOQ(2.50)	1.28
0344	31.1	3.26	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
0356	45.8	7.85	1.50	4.37	<LLOQ(1.00)	9.71	26.6
0358	30.0	4.32	2.27	3.53	<LLOQ(1.00)	<LLOQ(2.50)	1.3
0365	26.8	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0366	41.5	4.88	<LLOQ(1.36)	3.94	<LLOQ(1.00)	<LLOQ(2.50)	1.48
0367	67.7	7.21	4.78	4.85	<LLOQ(1.00)	<LLOQ(2.50)	1.49
0370	87.4	9.35	37.4	7.33	<LLOQ(1.00)	3.82	6.63
0385	50.2	9.02	7.08	1.97	<LLOQ(1.00)	<LLOQ(2.50)	3.74
0386	96.5	6.85	74.1	3.24	<LLOQ(1.00)	3.35	8.3
0387	56.5	6.83	35.3	3.28	<LLOQ(1.00)	2.56	1.47
0388	24.2	3.46	1.94	2.09	<LLOQ(1.00)	<LLOQ(2.50)	3.69
0398	31.4	2.61	4.32	3.20	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0404	69.1	4.27	49.3	5.10	<LLOQ(1.00)	2.61	1.17
0405	77.2	6.62	24.3	4.54	<LLOQ(1.00)	3.29	10.1
0407	17.8	2.46	<LLOQ(1.36)	3.79	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0410	49.3	4.94	2.54	8.78	<LLOQ(1.00)	<LLOQ(2.50)	2.48
0411	106	5.78	33.9	8.72	<LLOQ(1.00)	<LLOQ(2.50)	3.19
0415	53.2	5.21	41.3	4.39	<LLOQ(1.00)	<LLOQ(2.50)	4.12
0420	35.7	5.49	2.61	3.26	<LLOQ(1.00)	<LLOQ(2.50)	1.74
0421	65.9	5.63	2.20	9.82	<LLOQ(1.00)	<LLOQ(2.50)	4.25
0423	184	9.20	89.3	6.71	<LLOQ(1.00)	<LLOQ(2.50)	3.29

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0425	31.9	6.79	5.36	4.17	<LLOQ(1.00)	3.21	4.8
0432	46.1	7.06	3.05	4.62	<LLOQ(1.00)	3.82	1.78
0456	34.4	8.30	3.69	3.01	<LLOQ(1.00)	<LLOQ(2.50)	3.09
0463	34.7	3.97	7.64	3.61	<LLOQ(1.00)	<LLOQ(2.50)	3.42
0465	42.5	6.90	6.21	3.28	<LLOQ(1.00)	<LLOQ(2.50)	4.35
0466	27.3	4.03	6.75	3.56	<LLOQ(1.00)	2.58	4.73
0470	34.4	4.39	3.21	3.54	<LLOQ(1.00)	<LLOQ(2.50)	2.28
0476	124	14.6	7.36	11.3	<LLOQ(1.00)	9.59	31
0478	56.7	9.05	34.8	5.59	<LLOQ(1.00)	3.46	19.7
0493	106	7.56	50.5	8.83	<LLOQ(1.00)	9.45	1.84
0495	52.0	6.14	3.11	3.50	<LLOQ(1.00)	<LLOQ(2.50)	2.83
0498	63.4	5.19	18.1	4.17	<LLOQ(1.00)	3.83	2.71
0499	27.6	5.28	2.98	4.52	<LLOQ(1.00)	2.74	2.27
0500	59.9	4.72	21.2	8.94	<LLOQ(1.00)	5.88	2
0521	31.9	11.0	6.52	5.31	<LLOQ(1.00)	<LLOQ(2.50)	3.01
0524	59.5	7.20	37.5	3.44	<LLOQ(1.00)	3.26	4.14
0526	46.9	4.29	2.07	11.2	<LLOQ(1.00)	8.22	6.39

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0527	15.4	2.52	4.78	4.18	<LLOQ(1.00)	<LLOQ(2.50)	3.38
0528	30.3	5.10	17.0	2.09	<LLOQ(1.00)	2.66	4.46
0530	98.1	8.12	150	11.5	<LLOQ(1.00)	5.91	1.62
0531	97.9	6.43	72.0	10.3	<LLOQ(1.00)	3.67	1.71
0532	38.1	6.81	11.0	1.84	<LLOQ(1.00)	<LLOQ(2.50)	1.02
0534	34.6	4.39	5.63	2.59	<LLOQ(1.00)	2.8	1.66
0541	54.0	6.88	1.70	5.55	<LLOQ(1.00)	3.24	5.05
0542	29.5	5.18	<LLOQ(1.36)	5.11	<LLOQ(1.00)	3.15	4.13
0545	36.9	8.01	7.32	2.68	<LLOQ(1.00)	2.79	3.66
0548	75.7	9.00	52.0	3.98	<LLOQ(1.00)	<LLOQ(2.50)	2.13
0572	20.4	3.00	<LLOQ(1.36)	8.92	<LLOQ(1.00)	3.25	1.75
0578	77.6	7.66	80.6	4.48	<LLOQ(1.00)	5.4	1.27
0583	70.1	5.54	14.6	12.9	<LLOQ(1.00)	6.81	5.09
0585	64.8	6.25	34.5	4.82	<LLOQ(1.00)	2.82	4.5
0586	39.5	5.73	20.5	1.82	<LLOQ(1.00)	<LLOQ(2.50)	3
0587	58.2	7.36	22.0	9.06	<LLOQ(1.00)	<LLOQ(2.50)	6.77
0588	46.0	5.77	11.6	3.59	<LLOQ(1.00)	<LLOQ(2.50)	1.7

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0589	31.0	7.57	3.71	2.84	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0590	33.4	6.37	8.80	9.81	<LLOQ(1.00)	9.8	4.02
0592	35.1	3.15	1.75	2.99	<LLOQ(1.00)	<LLOQ(2.50)	2.22
0593	165	7.94	129	2.40	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0598	25.6	5.40	1.91	2.56	<LLOQ(1.00)	4.05	4.75
0599	77.1	7.44	21.8	6.25	<LLOQ(1.00)	4.42	5.91
0604	18.0	3.54	2.85	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.5
0606	37.6	5.71	2.84	4.69	<LLOQ(1.00)	4.11	7.17
0607	48.2	5.72	2.55	10.3	<LLOQ(1.00)	7	2.82
0608	52.3	8.01	1.82	8.98	<LLOQ(1.00)	3.42	3.57
0610	29.0	5.36	7.30	4.23	<LLOQ(1.00)	3.38	11.8
0611	20.3	2.19	<LLOQ(1.36)	3.66	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0621	23.2	4.57	3.13	3.95	<LLOQ(1.00)	<LLOQ(2.50)	2.12
0627	72.7	4.65	20.0	5.02	<LLOQ(1.00)	<LLOQ(2.50)	1.51
0628	29.8	2.64	<LLOQ(1.36)	6.26	<LLOQ(1.00)	2.64	3.69
0629	37.6	4.86	<LLOQ(1.36)	8.71	<LLOQ(1.00)	2.55	<LLOQ(1.00)
0631	54.5	7.68	11.4	6.72	<LLOQ(1.00)	4.63	<LLOQ(1.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0649	28.8	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
0657	27.4	5.90	6.25	3.71	<LLOQ(1.00)	3.25	3.78
0658	19.7	2.91	<LLOQ(1.36)	2.34	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0664	55.1	3.80	3.80	4.67	<LLOQ(1.00)	<LLOQ(2.50)	1.26
0665	31.9	4.96	4.68	3.36	<LLOQ(1.00)	<LLOQ(2.50)	2.14
0666	16.3	3.17	<LLOQ(1.36)	3.19	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0667	57.8	7.41	37.1	3.95	<LLOQ(1.00)	<LLOQ(2.50)	6.96
0673	34.3	4.21	1.38	8.58	<LLOQ(1.00)	3.4	5.39
0676	25.6	4.91	1.64	4.54	<LLOQ(1.00)	<LLOQ(2.50)	3.56
0678	50.0	7.09	7.21	11.2	<LLOQ(1.00)	3.7	7.87
0679	30.2	3.16	<LLOQ(1.36)	5.15	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0680	44.6	5.80	2.35	9.12	<LLOQ(1.00)	4.86	2.92
0681	31.9	4.81	5.19	4.18	<LLOQ(1.00)	<LLOQ(2.50)	2.06
0689	40.1	5.35	<LLOQ(1.36)	3.97	<LLOQ(1.00)	2.91	6.03
0695	28.1	4.08	2.65	5.70	<LLOQ(1.00)	3.46	<LLOQ(1.00)
0697	27.8	4.29	1.56	3.65	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0700	17.8	3.90	1.83	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0711	8.76	2.02	<LLOQ(1.36)	2.23	<LLOQ(1.00)	3.63	<LLOQ(1.00)
0712	19.5	2.54	<LLOQ(1.36)	3.76	<LLOQ(1.00)	<LLOQ(2.50)	2.93
0713	32.4	4.34	<LLOQ(1.36)	2.71	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0714	23.3	2.78	1.96	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.61
0719	34.8	3.82	1.82	4.90	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0720	42.7	5.26	4.88	3.05	<LLOQ(1.00)	8.87	4.44
0721	37.8	4.97	8.24	5.34	<LLOQ(1.00)	<LLOQ(2.50)	1.19
0723	32.2	4.87	9.44	4.95	<LLOQ(1.00)	5.36	<LLOQ(1.00)
0725	26.9	5.17	1.42	8.99	<LLOQ(1.00)	2.66	<LLOQ(1.00)
0741	33.3	4.36	1.85	3.66	<LLOQ(1.00)	2.95	2.51
0748	22.0	6.00	5.04	3.67	<LLOQ(1.00)	3.76	1.81
0752	16.7	2.46	<LLOQ(1.36)	3.52	<LLOQ(1.00)	<LLOQ(2.50)	1.34
0753	37.1	4.67	1.38	2.96	<LLOQ(1.00)	<LLOQ(2.50)	5.36
0754	33.6	5.63	<LLOQ(1.36)	5.97	<LLOQ(1.00)	5.12	<LLOQ(1.00)
0763	35.7	5.05	7.10	4.60	<LLOQ(1.00)	<LLOQ(2.50)	1.13
0769	19.5	3.40	4.87	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0771	81.0	4.88	2.43	3.34	<LLOQ(1.00)	3.38	14.2

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0774	27.9	2.49	3.65	5.19	<LLOQ(1.00)	<LLOQ(2.50)	3.25
0775	41.4	9.11	8.85	5.06	<LLOQ(1.00)	7.2	14.3
0779	27.9	4.44	8.86	2.04	<LLOQ(1.00)	2.87	1.3
0783	28.8	7.14	3.50	5.92	<LLOQ(1.00)	<LLOQ(2.50)	4.58
0784	39.1	5.65	9.21	3.27	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0785	70.6	6.97	41.3	1.71	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0788	51.1	7.34	36.4	3.87	<LLOQ(1.00)	3.1	<LLOQ(1.00)
0792	54.2	5.54	4.68	3.18	<LLOQ(1.00)	<LLOQ(2.50)	3.73
0795	32.3	5.70	9.86	2.05	<LLOQ(1.00)	4.47	1.59
0796	17.6	4.61	3.11	<LLOQ(1.60)	<LLOQ(1.00)	3.93	1.42
0798	40.2	4.33	2.55	7.35	<LLOQ(1.00)	4.28	<LLOQ(1.00)
0799	54.4	3.54	3.60	4.12	<LLOQ(1.00)	3.21	8.16
0800	34.1	5.98	<LLOQ(1.36)	6.89	<LLOQ(1.00)	2.7	<LLOQ(1.00)
0813	12.2	3.84	4.31	2.20	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0814	40.7	5.47	2.39	5.76	<LLOQ(1.00)	4.44	<LLOQ(1.00)
0823	56.3	6.59	1.70	3.78	<LLOQ(1.00)	6.58	8.26
0829	41.8	4.36	13.2	5.30	<LLOQ(1.00)	3.15	1.83

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0830	32.0	6.12	3.80	10.3	<LLOQ(1.00)	5.92	18.4
0840	49.7	5.70	2.71	2.96	<LLOQ(1.00)	3.55	5.64
0841	33.1	5.68	19.5	6.56	<LLOQ(1.00)	2.82	3.54
0845	67.7	5.77	5.21	20.7	<LLOQ(1.00)	15.5	4.44
0846	39.3	4.44	2.04	7.87	<LLOQ(1.00)	5.82	<LLOQ(1.00)
0848	27.2	3.26	2.77	4.27	<LLOQ(1.00)	2.91	1.7
0849	33.4	3.76	3.24	3.86	<LLOQ(1.00)	2.75	1.55
0859	59.5	8.31	12.6	6.56	<LLOQ(1.00)	4.46	3.22
0864	74.3	4.23	13.0	2.25	<LLOQ(1.00)	4.02	9.81
0866	43.8	9.42	18.5	2.10	<LLOQ(1.00)	<LLOQ(2.50)	3.24
0867	26.7	3.86	8.01	2.01	<LLOQ(1.00)	<LLOQ(2.50)	1.98
0869	31.7	6.81	<LLOQ(1.36)	4.79	<LLOQ(1.00)	6.17	<LLOQ(1.00)
0872	58.1	6.46	3.48	9.96	<LLOQ(1.00)	<LLOQ(2.50)	7.85
0876	50.1	6.10	6.68	3.75	<LLOQ(1.00)	<LLOQ(2.50)	1.42
0880	71.7	8.95	5.35	5.84	<LLOQ(1.00)	<LLOQ(2.50)	26.7
0881	45.7	3.92	<LLOQ(1.36)	6.01	<LLOQ(1.00)	4.09	<LLOQ(1.00)
0883	42.8	5.09	18.4	6.14	<LLOQ(1.00)	<LLOQ(2.50)	2.93

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
0890	17.3	3.80	3.91	2.69	<LLOQ(1.00)	<LLOQ(2.50)	1
0906	36.3	4.47	<LLOQ(1.36)	21.7	<LLOQ(1.00)	3.81	1.55
0907	59.4	6.43	7.14	4.84	<LLOQ(1.00)	10.5	9.95
0909	36.8	4.09	<LLOQ(1.36)	4.41	<LLOQ(1.00)	<LLOQ(2.50)	1.11
0910	24.4	3.13	1.87	7.23	<LLOQ(1.00)	6.52	<LLOQ(1.00)
0916	17.6	2.85	<LLOQ(1.36)	2.11	<LLOQ(1.00)	<LLOQ(2.50)	1.08
0922	40.7	8.18	<LLOQ(1.36)	8.51	<LLOQ(1.00)	3.6	1.7
0928	74.6	6.55	43.2	3.86	<LLOQ(1.00)	<LLOQ(2.50)	4.28
0937	39.3	3.61	2.69	6.95	<LLOQ(1.00)	3.65	2.91
0938	43.9	7.20	1.67	4.06	<LLOQ(1.00)	5.07	1.54
0950	24.8	4.90	5.34	5.12	<LLOQ(1.00)	4.11	1.31
0953	30.3	8.51	1.47	5.69	<LLOQ(1.00)	4.28	3.79
0956	27.5	3.62	<LLOQ(1.36)	2.24	<LLOQ(1.00)	<LLOQ(2.50)	1.8
0970	40.4	6.62	6.18	9.69	<LLOQ(1.00)	2.66	2.95
0971	11.4	2.62	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
0986	20.1	2.57	<LLOQ(1.36)	2.46	<LLOQ(1.00)	<LLOQ(2.50)	1.51
0987	58.2	4.35	3.94	16.2	<LLOQ(1.00)	3.42	9.05

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1001	21.7	3.25	<LLOQ(1.36)	1.64	<LLOQ(1.00)	<LLOQ(2.50)	2.32
1003	18.0	3.52	2.18	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1005	26.8	5.39	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	4.31
1012	27.4	2.47	1.44	4.07	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1013	38.0	5.45	3.46	3.90	<LLOQ(1.00)	<LLOQ(2.50)	2.6
1017	16.8	3.46	<LLOQ(1.36)	7.37	<LLOQ(1.00)	3.14	2.29
1019	39.9	7.16	2.54	5.58	<LLOQ(1.00)	<LLOQ(2.50)	7.97
1021	25.6	9.81	5.11	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	3.71
1023	28.9	3.01	<LLOQ(1.36)	6.14	<LLOQ(1.00)	2.54	3.03
1029	86.4	6.18	74.2	3.18	<LLOQ(1.00)	<LLOQ(2.50)	2.1
1030	23.8	3.89	<LLOQ(1.36)	5.10	<LLOQ(1.00)	2.68	1.51
1039	325	56.1	416	3.62	<LLOQ(1.00)	4.01	7.76
1042	50.7	5.06	39.6	3.21	<LLOQ(1.00)	<LLOQ(2.50)	3.6
1044	81.4	8.05	51.9	5.79	<LLOQ(1.00)	4.25	1.92
1048	25.1	6.33	2.94	2.94	<LLOQ(1.00)	<LLOQ(2.50)	4.21
1053	66.6	4.25	35.7	3.45	<LLOQ(1.00)	<LLOQ(2.50)	2.45
1057	62.8	8.62	6.40	7.90	<LLOQ(1.00)	4.05	17.8

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1059	27.9	4.09	<LLOQ(1.36)	7.44	<LLOQ(1.00)	5.33	<LLOQ(1.00)
1067	45.1	5.32	10.4	3.17	<LLOQ(1.00)	<LLOQ(2.50)	1.01
1068	19.5	3.11	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.58
1070	22.4	3.57	9.07	2.72	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1072	43.2	6.22	1.92	6.89	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1073	33.6	2.64	2.19	7.83	<LLOQ(1.00)	<LLOQ(2.50)	3.69
1082	48.2	4.66	7.91	4.62	<LLOQ(1.00)	5.99	3.6
1085	38.2	3.43	1.59	2.98	<LLOQ(1.00)	<LLOQ(2.50)	1.83
1088	31.3	6.98	1.77	2.16	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1090	50.9	5.85	12.4	4.99	<LLOQ(1.00)	<LLOQ(2.50)	6.28
1101	32.8	2.90	9.41	1.73	<LLOQ(1.00)	2.97	5.83
1104	62.6	5.63	13.3	6.31	<LLOQ(1.00)	5.8	2.25
1113	20.2	3.08	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1115	40.4	5.04	36.2	1.74	<LLOQ(1.00)	<LLOQ(2.50)	1.4
1117	46.5	3.09	<LLOQ(1.36)	6.89	<LLOQ(1.00)	<LLOQ(2.50)	1.91
1121	48.7	5.60	2.60	5.10	<LLOQ(1.00)	4.01	4.77
1122	36.5	5.40	11.9	2.96	<LLOQ(1.00)	<LLOQ(2.50)	2.5

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1124	20.7	4.35	4.70	2.13	<LLOQ(1.00)	<LLOQ(2.50)	3.15
1126	33.1	4.90	10.4	5.66	<LLOQ(1.00)	2.5	4.22
1129	52.0	6.85	4.55	4.24	<LLOQ(1.00)	<LLOQ(2.50)	5.69
1133	26.7	4.15	20.3	1.79	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1136	27.1	4.21	2.00	6.23	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1144	30.1	4.59	3.47	5.01	<LLOQ(1.00)	<LLOQ(2.50)	1.88
1147	30.1	5.51	2.39	7.87	<LLOQ(1.00)	3.29	2.15
1152	17.7	4.33	3.25	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.56
1153	31.3	4.28	8.63	2.27	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1154	22.5	3.61	2.44	4.08	<LLOQ(1.00)	<LLOQ(2.50)	2.31
1155	31.7	3.71	<LLOQ(1.36)	5.29	<LLOQ(1.00)	4.01	1.69
1157	53.0	4.38	11.5	2.51	<LLOQ(1.00)	<LLOQ(2.50)	3.28
1166	34.8	5.80	10.5	5.60	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1168	53.7	5.22	2.86	3.61	<LLOQ(1.00)	4.33	12.5
1171	55.2	5.21	2.57	9.22	<LLOQ(1.00)	6.36	17
1172	118	11.9	4.07	5.94	<LLOQ(1.00)	14.9	48
1173	43.4	11.0	9.77	4.22	<LLOQ(1.00)	<LLOQ(2.50)	6.27

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1174	39.6	5.35	17.2	8.52	<LLOQ(1.00)	4.4	1.97
1175	28.6	3.95	<LLOQ(1.36)	3.23	<LLOQ(1.00)	<LLOQ(2.50)	1.01
1177	73.8	9.51	14.3	3.66	<LLOQ(1.00)	4.76	13.4
1178	51.5	4.87	1.74	17.0	<LLOQ(1.00)	6.17	3.45
1179	49.0	6.85	18.3	6.21	<LLOQ(1.00)	5.77	7.04
11797	27.9	3.37	8.71	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11798	69.7	9.74	58.2	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11799	142	22.1	268	6.31	<LLOQ(2.00)	11.9	16.9
11800	131	7.13	82.9	5.26	<LLOQ(2.00)	<LLOQ(5.00)	10.8
11801	42.9	6.52	10.4	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11802	647	23.3	873	3.02	<LLOQ(2.00)	13.7	4.4
11803	110	8.61	80.8	15.4	<LLOQ(2.00)	6.01	2.92
11804	120	9.03	181	2.90	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11805	90.2	9.64	65.5	3.51	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11806	98.9	7.51	44.7	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11807	66.6	5.74	44.3	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11808	54.1	5.57	21.8	3.76	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
11809	418	70.0	484	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	5.52
11810	21.8	3.56	5.99	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11811	90.9	6.70	136	7.90	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11812	199	11.4	109	2.98	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11813	39.7	5.69	12.5	2.72	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11814	419	65.6	469	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	6.64
11815	47.3	7.47	8.13	10.0	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11816	88.5	19.5	19.3	6.18	<LLOQ(2.00)	7.38	35.1
11817	81.5	6.05	142	5.47	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11818	143	10.2	105	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11819	68.9	7.35	5.17	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	10.9
1182	38.1	4.26	13.0	5.36	<LLOQ(2.00)	<LLOQ(5.00)	2
11820	118	9.34	167	3.00	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11821	26.0	<LLOQ(2.88)	6.77	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11822	52.5	5.09	19.9	4.90	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11823	28.8	<LLOQ(2.88)	<LLOQ(2.41)	3.24	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11824	52.6	6.65	<LLOQ(2.41)	3.83	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
11825	92.1	20.54	20.9	7.02	<LLOQ(2.00)	8.74	35.6
11826	46.1	5.32	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11827	131	8.71	101	15.7	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11828	164	7.63	132	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11829	83.9	6.91	24.1	5.09	<LLOQ(2.00)	<LLOQ(5.00)	2.4
1183	57.7	11.6	21.8	2.39	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
11830	34.7	4.13	5.53	4.52	<LLOQ(2.00)	<LLOQ(5.00)	3.32
11831	28.1	3.05	6.04	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11832	32.0	3.19	7.86	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11833	188	18.5	228	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11834	76.6	5.97	7.85	3.23	<LLOQ(2.00)	13.5	9.14
11835	685	23.0	933	2.65	<LLOQ(2.00)	13.8	6.01
11836	55.9	6.43	<LLOQ(2.41)	3.55	<LLOQ(2.00)	<LLOQ(5.00)	11.3
11837	160	7.44	135	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11838	17.6	3.40	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11839	101	25.9	20.2	8.27	<LLOQ(2.00)	9.47	45.4
1184	37.1	5.07	1.44	1.70	<LLOQ(1.00)	<LLOQ(2.50)	2.63

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
11840	42.2	8.99	2.56	3.58	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11841	127	9.54	171	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11842	74.8	5.40	<LLOQ(2.41)	8.33	<LLOQ(2.00)	<LLOQ(5.00)	2.17
11843	146	10.8	108	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11844	29.7	3.47	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11845	196	20.2	235	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11846	88.0	6.77	141	7.35	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11847	14.9	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11848	162	21.9	274	7.77	<LLOQ(2.00)	16	21.9
11849	145	13.6	64.0	19.7	<LLOQ(2.00)	15.4	3.76
11850	613	22.5	864	<LLOQ(2.60)	<LLOQ(2.00)	13	4.31
11851	54.1	4.76	12.9	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	29.9
11852	170	7.44	135	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11853	431	67.2	490	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	7.19
11854	93.1	15.9	10.5	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	2.17
11855	103	19.6	20.8	8.05	<LLOQ(2.00)	9.46	<LLOQ(2.00)
11856	20.3	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
11857	199	20.0	241	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
11858	144	20.8	257	4.69	<LLOQ(2.00)	12.1	18.7
1186	39.3	10.2	1.89	6.23	<LLOQ(1.00)	2.79	1.14
1199	62.2	8.64	3.15	6.64	<LLOQ(1.00)	5.76	9.65
1214	44.2	5.55	33.9	2.93	<LLOQ(1.00)	2.61	4.3
1217	70.7	6.72	42.4	4.53	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1229	37.7	3.05	<LLOQ(1.36)	18.2	<LLOQ(1.00)	6.33	1.56
1232	53.5	6.70	2.97	6.98	<LLOQ(1.00)	4.46	13.8
1239	37.5	3.46	6.45	5.56	<LLOQ(1.00)	<LLOQ(2.50)	4.45
1240	67.6	10.3	3.47	14.3	<LLOQ(1.00)	4.12	6.12
1248	42.7	4.40	2.83	4.44	<LLOQ(1.00)	3.09	1.77
1254	35.4	5.44	1.38	3.26	<LLOQ(1.00)	<LLOQ(2.50)	1.81
1255	97.9	9.80	61.4	2.97	<LLOQ(1.00)	3.24	3.22
1261	47.5	6.27	2.50	2.09	<LLOQ(1.00)	3.59	3.44
1264	51.5	8.51	9.61	4.71	<LLOQ(1.00)	5.35	6.9
1269	78.1	16.4	76.5	3.62	<LLOQ(1.00)	3.34	2.48
1273	72.1	7.52	4.10	11.6	<LLOQ(1.00)	4.91	5.23

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1291	37.9	6.74	2.52	6.93	<LLOQ(1.00)	<LLOQ(2.50)	3.56
1294	119	17.5	220	6.71	<LLOQ(1.00)	13.1	16.3
1296	39.9	4.74	9.16	18.7	<LLOQ(1.00)	6.84	7.38
1299	27.7	6.57	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.9
1311	36.0	6.05	3.67	5.39	<LLOQ(1.00)	<LLOQ(2.50)	1.12
1314	51.7	5.35	18.1	7.14	<LLOQ(1.00)	5.59	1.05
1323	95.4	15.1	10.5	5.54	<LLOQ(1.00)	<LLOQ(2.50)	5.85
1337	65.4	6.41	5.46	9.47	<LLOQ(1.00)	5.09	3.93
1339	27.8	7.17	<LLOQ(1.36)	2.26	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1356	24.3	3.15	1.58	3.27	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1358	37.1	3.96	1.60	9.17	<LLOQ(1.00)	6.09	1.31
1364	33.3	5.10	3.19	6.95	<LLOQ(1.00)	5.4	<LLOQ(1.00)
1368	61.7	13.3	8.18	3.93	<LLOQ(1.00)	5.84	19.6
1372	34.8	5.88	<LLOQ(1.36)	6.66	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1387	35.0	6.76	3.26	4.53	<LLOQ(1.00)	2.96	3.67
1393	134	7.71	88.2	7.42	<LLOQ(1.00)	4.44	2.52
1395	110	11.6	129	9.89	<LLOQ(1.00)	2.6	<LLOQ(1.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1398	37.6	8.55	4.44	5.02	<LLOQ(1.00)	<LLOQ(2.50)	3.69
1400	32.5	5.00	1.49	3.17	<LLOQ(1.00)	<LLOQ(2.50)	1.25
1407	24.3	2.84	2.57	5.69	<LLOQ(1.00)	<LLOQ(2.50)	1.54
1408	34.7	3.96	4.19	4.69	<LLOQ(1.00)	2.73	1.84
1409	34.0	3.92	1.58	3.88	<LLOQ(1.00)	3.48	1.06
1410	21.3	3.41	<LLOQ(1.36)	3.40	<LLOQ(1.00)	2.74	1.42
1411	31.5	4.76	2.52	4.26	<LLOQ(1.00)	<LLOQ(2.50)	3.85
1412	35.1	4.58	7.03	14.1	<LLOQ(1.00)	5.44	4.53
1418	36.2	6.24	<LLOQ(1.36)	10.4	<LLOQ(1.00)	5.77	<LLOQ(1.00)
1420	34.5	4.71	6.13	5.18	<LLOQ(1.00)	2.77	<LLOQ(1.00)
1423	22.7	5.08	5.63	1.82	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1424	29.3	2.69	1.47	2.47	<LLOQ(1.00)	<LLOQ(2.50)	1.52
1427	17.6	3.34	<LLOQ(1.36)	4.95	<LLOQ(1.00)	<LLOQ(2.50)	1.98
1431	27.6	5.68	5.81	4.45	<LLOQ(1.00)	4.54	1.42
1433	20.3	2.88	<LLOQ(1.36)	1.89	<LLOQ(1.00)	<LLOQ(2.50)	1.24
1435	19.0	3.48	4.19	2.60	<LLOQ(1.00)	<LLOQ(2.50)	1.72
1438	56.9	5.83	22.4	2.91	<LLOQ(1.00)	2.72	2.46

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1439	20.1	4.77	9.34	3.57	<LLOQ(1.00)	5.46	2.87
1448	64.8	5.35	21.8	9.13	<LLOQ(1.00)	4.43	<LLOQ(1.00)
1455	16.7	3.23	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1461	41.3	6.21	19.2	2.71	<LLOQ(1.00)	3.34	2.98
1467	18.2	3.30	5.74	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1468	16.6	2.66	3.46	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1470	36.2	8.05	5.69	4.97	<LLOQ(1.00)	4.3	4.88
1472	32.4	5.55	1.77	1.71	<LLOQ(1.00)	2.88	1.23
1482	59.4	4.88	17.2	18.3	<LLOQ(1.00)	6.53	3.52
1483	54.6	5.24	8.66	23.8	<LLOQ(1.00)	12	4.1
1491	26.2	3.36	3.73	2.62	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1493	37.6	4.49	10.6	<LLOQ(1.60)	<LLOQ(1.00)	4.27	22.7
1494	16.8	3.01	<LLOQ(1.36)	2.77	<LLOQ(1.00)	2.79	2.28
1496	54.9	4.53	38.3	1.95	<LLOQ(1.00)	4.95	<LLOQ(1.00)
1498	41.7	4.42	<LLOQ(1.36)	5.31	<LLOQ(1.00)	3.7	1.13
1503	24.6	2.40	2.73	2.39	<LLOQ(1.00)	<LLOQ(2.50)	1.68
1510	32.5	3.21	13.7	<LLOQ(1.60)	<LLOQ(1.00)	3.24	17.1

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1512	26.4	2.63	12.1	1.73	<LLOQ(1.00)	<LLOQ(2.50)	4.33
1513	34.1	4.37	5.13	3.27	<LLOQ(1.00)	<LLOQ(2.50)	1.9
1517	62.8	5.76	58.8	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1524	28.9	4.82	4.30	1.66	<LLOQ(1.00)	6.23	15.2
1529	26.5	4.56	2.73	3.83	<LLOQ(1.00)	6.06	<LLOQ(1.00)
1537	10.4	<LLOQ(1.92)	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1538	35.9	2.36	11.2	4.33	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1542	17.2	2.14	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1545	28.9	3.89	2.28	6.05	<LLOQ(1.00)	2.5	<LLOQ(1.00)
1558	50.8	7.92	13.8	10.4	<LLOQ(1.00)	4.44	<LLOQ(1.00)
1559	61.0	10.5	14.2	8.56	<LLOQ(1.00)	6.92	1.61
1574	25.0	4.75	6.47	2.17	<LLOQ(1.00)	<LLOQ(2.50)	2
1582	26.0	3.20	<LLOQ(1.36)	1.89	<LLOQ(1.00)	3.07	<LLOQ(1.00)
1590	39.3	6.61	4.27	5.56	<LLOQ(1.00)	8.75	<LLOQ(1.00)
1597	31.5	9.93	3.34	2.8	<LLOQ(1.00)	2.74	1.49
1603	25.4	5.53	<LLOQ(1.36)	5.99	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1611	37.3	13.0	1.52	6.68	<LLOQ(1.00)	5.62	5.75

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1616	30.0	4.67	3.79	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1618	26.1	3.08	5.35	4.69	<LLOQ(1.00)	2.77	<LLOQ(1.00)
1620	21.8	3.19	1.87	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1624	45.4	5.54	41.5	1.60	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1626	25.0	3.15	1.55	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1630	13.6	3.13	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1634	22.7	2.56	7.25	1.73	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1637	35.7	3.08	23.3	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1640	11.9	2.46	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1643	17.5	2.33	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1646	15.8	2.34	<LLOQ(1.36)	2.26	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1647	18.8	3.79	<LLOQ(1.36)	2.81	<LLOQ(1.00)	3.75	<LLOQ(1.00)
1652	34.7	6.21	5.59	5.50	<LLOQ(1.00)	4.14	<LLOQ(1.00)
1653	44.2	7.80	4.54	7.02	<LLOQ(1.00)	<LLOQ(2.50)	3.91
1663	11.5	2.15	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1665	15.0	<LLOQ(1.92)	<LLOQ(1.36)	6.09	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1668	20.5	2.9	2.29	2.15	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1669	33.9	6.64	3.25	6.72	<LLOQ(1.00)	2.87	2.33
1677	27.6	2.58	3.31	2.38	<LLOQ(1.00)	<LLOQ(2.50)	1.32
1678	15.9	2.46	<LLOQ(1.36)	3.13	<LLOQ(1.00)	<LLOQ(2.50)	1.74
1679	28.1	4.27	3.52	2.39	<LLOQ(1.00)	<LLOQ(2.50)	2.39
1680	23.0	2.69	<LLOQ(1.36)	3.74	<LLOQ(1.00)	<LLOQ(2.50)	6.08
1690	38.1	4.97	<LLOQ(1.36)	12.8	<LLOQ(1.00)	<LLOQ(2.50)	1.01
1692	42.6	8.11	8.96	11.2	<LLOQ(1.00)	5.49	1.57
1700	22.4	2.14	<LLOQ(1.36)	6.01	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1707	42.4	3.56	4.17	5.07	<LLOQ(1.00)	4.96	3.77
1709	42.5	4.74	38.5	3.71	<LLOQ(1.00)	4.06	3.43
1716	33.3	4.57	<LLOQ(1.36)	6.99	<LLOQ(1.00)	<LLOQ(2.50)	3.47
1719	21.2	4.55	2.25	1.93	<LLOQ(1.00)	<LLOQ(2.50)	1.21
1721	26.1	4.96	1.93	1.67	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1722	28.6	3.25	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.82
1735	27.5	5.88	1.97	7.19	<LLOQ(1.00)	3.66	2.74
1748	27.2	3.44	3.14	2.25	<LLOQ(1.00)	<LLOQ(2.50)	2.65
1766	43.6	5.28	7.07	5.68	<LLOQ(1.00)	<LLOQ(2.50)	2.97

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1778	25.3	5.66	1.65	1.70	<LLOQ(1.00)	2.62	6.74
1781	32.4	4.02	1.67	3.58	<LLOQ(1.00)	<LLOQ(2.50)	6.82
1788	49.7	5.74	19.6	5.97	<LLOQ(1.00)	5.25	1.12
1806	67.7	7.17	94.2	4.75	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1809	12.7	1.95	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1816	33.9	5.93	<LLOQ(1.36)	11.6	<LLOQ(1.00)	9.82	<LLOQ(1.00)
1817	29.3	4.40	12.4	3.54	<LLOQ(1.00)	<LLOQ(2.50)	1.56
1819	29.3	4.12	7.59	2.90	<LLOQ(1.00)	<LLOQ(2.50)	2.62
1822	48.8	6.01	17.8	9.63	<LLOQ(1.00)	<LLOQ(2.50)	2.01
1825	20.5	4.76	2.65	2.44	<LLOQ(1.00)	2.78	2.04
1830	19.9	4.29	2.79	1.62	<LLOQ(1.00)	9.88	2.47
1834	28.7	5.75	15.7	3.68	<LLOQ(1.00)	<LLOQ(2.50)	3.06
1837	71.8	8.95	4.69	12.9	<LLOQ(1.00)	19.1	38.1
1842	32.2	5.37	9.45	1.69	<LLOQ(1.00)	7.29	3.89
1857	21.3	5.70	<LLOQ(1.36)	5.07	<LLOQ(1.00)	4.54	<LLOQ(1.00)
1875	28.3	4.91	1.58	2.18	<LLOQ(1.00)	<LLOQ(2.50)	1.88
1876	25.0	5.60	8.53	2.24	<LLOQ(1.00)	2.79	1.72

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1886	52.6	9.49	2.01	19.6	<LLOQ(1.00)	9.53	<LLOQ(1.00)
1887	16.9	3.22	<LLOQ(1.36)	4.81	<LLOQ(1.00)	<LLOQ(2.50)	1.38
1888	40.5	8.11	2.39	5.31	<LLOQ(1.00)	6.41	1.22
1898	18.3	5.26	3.40	2.22	<LLOQ(1.00)	<LLOQ(2.50)	1.03
1909	149	16.1	170	3.81	<LLOQ(1.00)	2.76	1.28
1911	35.3	6.28	2.44	2.40	<LLOQ(1.00)	4.56	1.15
1912	46.2	7.32	3.17	6.16	<LLOQ(1.00)	2.65	1.58
1913	43.9	9.06	3.52	2.93	<LLOQ(1.00)	<LLOQ(2.50)	5.23
1934	51.2	6.99	4.44	4.90	<LLOQ(1.00)	<LLOQ(2.50)	13.1
1942	50.1	8.54	4.83	3.22	<LLOQ(1.00)	3.02	1.95
1953	74.3	7.94	18.9	5.18	<LLOQ(1.00)	2.76	4.27
1954	55.5	5.09	1.64	6.62	<LLOQ(1.00)	4.45	3.76
1956	30.6	5.05	10.7	3.20	<LLOQ(1.00)	<LLOQ(2.50)	4.77
1959	23.8	4.26	3.42	3.87	<LLOQ(1.00)	<LLOQ(2.50)	2.37
1961	31.0	5.54	12.3	2.95	<LLOQ(1.00)	3.06	2.27
1962	16.2	4.12	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
1965	75.2	6.65	87.9	5.62	<LLOQ(1.00)	4.22	2.13

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

030418

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
1982	18.2	4.15	1.51	3.93	<LLOQ(1.00)	<LLOQ(2.50)	2.25
1996	27.1	3.82	2.43	3.43	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2000	93.7	10.1	7.12	8.19	<LLOQ(1.00)	9.14	17.9
2002	96.8	7.17	90.8	2.93	<LLOQ(1.00)	<LLOQ(2.50)	2.45
2003	52.2	4.87	3.72	8.86	<LLOQ(1.00)	3.48	3.08
2022	34.7	3.94	4.98	1.80	<LLOQ(1.00)	<LLOQ(2.50)	2.63
2038	70.4	8.89	15.8	2.35	<LLOQ(1.00)	<LLOQ(2.50)	5.81
2041	22.8	5.69	7.12	1.88	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2050	41.5	4.61	2.83	2.64	<LLOQ(1.00)	3.28	8.4
2052	25.8	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
2054	20.7	3.06	2.36	3.50	<LLOQ(1.00)	2.84	<LLOQ(1.00)
2056	45.6	4.31	15.4	5.44	<LLOQ(1.00)	<LLOQ(2.50)	1.49
2074	48.5	5.54	2.21	4.60	<LLOQ(1.00)	2.77	3.03
2075	38.8	5.30	3.75	2.19	<LLOQ(1.00)	2.93	5.4
2076	39.4	6.65	8.06	6.49	<LLOQ(1.00)	3.48	4.24
2078	86.8	19.5	20.4	8.81	<LLOQ(1.00)	10.2	42.5
2079	59.9	6.63	4.84	2.40	<LLOQ(1.00)	5.37	14.8

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

030419

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
2082	86.6	6.87	84.1	9.70	<LLOQ(1.00)	<LLOQ(2.50)	1.97
2087	34.4	8.42	1.55	7.75	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2089	21.0	4.61	2.16	4.61	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2099	47.1	5.29	4.23	5.82	<LLOQ(1.00)	<LLOQ(2.50)	2.09
2104	55.3	6.83	47.6	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.36
2112	80.5	10.7	62.2	5.79	<LLOQ(1.00)	3.32	1.64
2114	42.8	4.48	3.39	4.62	<LLOQ(1.00)	<LLOQ(2.50)	3.76
2115	28.0	5.83	5.04	5.3	<LLOQ(1.00)	2.75	4.04
2121	55.1	4.10	19.2	4.52	<LLOQ(1.00)	3.38	9.68
2124	42.5	6.95	15.9	4.33	<LLOQ(1.00)	<LLOQ(2.50)	4.14
2127	62.1	8.96	27.9	3.43	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2140	26.1	3.67	2.81	2.28	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2141	29.7	6.77	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2144	37.0	4.06	2.65	6.25	<LLOQ(1.00)	6.14	1.1
2150	39.2	4.24	9.88	6.59	<LLOQ(1.00)	9.89	2.36
2151	23.9	3.37	1.64	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2156	46.6	7.95	13.6	3.94	<LLOQ(1.00)	<LLOQ(2.50)	3.82

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
2158	36.4	6.93	6.10	4.02	<LLOQ(1.00)	2.55	2.48
2162	48.1	5.10	14.1	1.81	<LLOQ(1.00)	<LLOQ(2.50)	2.12
2164	36.2	5.08	4.51	2.58	<LLOQ(1.00)	<LLOQ(2.50)	3.35
2168	515	20.2	712	4.04	<LLOQ(1.00)	16.4	7
2171	65.5	7.09	37.3	5.69	<LLOQ(1.00)	<LLOQ(2.50)	2.8
2173	49.5	5.23	18.2	4.68	<LLOQ(1.00)	5.08	8.34
2174	63.5	14.0	5.92	5.23	<LLOQ(1.00)	3.92	17.7
2176	73.2	8.55	59.2	2.68	<LLOQ(1.00)	3.49	3.47
2179	123	9.96	107	7.47	<LLOQ(1.00)	4.01	9.76
2181	116	11.5	55.4	18.1	<LLOQ(1.00)	13.4	4.82
2184	53.3	10.5	5.07	3.94	<LLOQ(1.00)	6.68	7.53
2186	50.2	8.36	3.11	7.97	<LLOQ(1.00)	3.88	5.6
2191	40.2	4.52	<LLOQ(1.36)	3.37	<LLOQ(1.00)	2.97	1.08
2193	40.1	6.62	31.9	6.45	<LLOQ(1.00)**	4.54	6.58
2200	41.5	5.66	12.1	4.73	<LLOQ(1.00)**	<LLOQ(2.50)	1.42
2220	42.4	5.61	8.01	7.73	<LLOQ(1.00)**	<LLOQ(2.50)	6.69
2227	69.9	7.47	104	<LLOQ(1.60)	<LLOQ(1.00)**	5.59	6.75

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
2230	66.6	7.92	84.2	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	1.36
2234	36.1	5.77	14.8	2.95	<LLOQ(1.00)**	<LLOQ(2.50)	2.32
2236	39.8	5.97	25.4	2.92	<LLOQ(1.00)**	<LLOQ(2.50)	2.93
2239	17.2	2.77	12.5	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	<LLOQ(1.00)
2244	26.2	2.91	3.44	3.69	<LLOQ(1.00)**	<LLOQ(2.50)	<LLOQ(1.00)
2246	34.7	5.06	4.20	2.96	<LLOQ(1.00)**	<LLOQ(2.50)	1.85
2248	60.8	6.72	29.6	3.21	<LLOQ(1.00)**	<LLOQ(2.50)	2.55
2260	6.67	<LLOQ(1.92)	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	<LLOQ(1.00)
2265	44.8	5.09	10.4	4.18	<LLOQ(1.00)**	<LLOQ(2.50)	1.29
2273	28.3	3.45	1.79	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	3.59
2298	17.7	2.22	<LLOQ(1.36)	3.16	<LLOQ(1.00)**	<LLOQ(2.50)	2.48
2307	43.9	5.52	4.73	8.03	<LLOQ(1.00)**	2.94	1.2
2323	25.5	3.65	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	4.55
2327	24.3	5.47	3.79	2.22	<LLOQ(1.00)**	5.33	<LLOQ(1.00)
2335	26.5	3.18	3.79	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	<LLOQ(1.00)
2341	43.4	7.21	2.25	5.43	<LLOQ(1.00)**	4.53	10.6
2348	34.7	4.45	<LLOQ(1.36)	5.20	<LLOQ(1.00)**	7.59	6.4

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
2349	40.8	3.86	4.49	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	5.22
2350	76.4	8.86	20.3	13.1	<LLOQ(1.00)**	6.83	16.8
2351	36.9	3.79	1.76	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	<LLOQ(1.00)
2353	34.0	3.86	4.51	2.19	<LLOQ(1.00)**	<LLOQ(2.50)	1.45
2355	36.2	4.72	1.62	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	1.33
2358	61.8	10.3	1.79	2.93	<LLOQ(1.00)**	4.72	13.4
2360	29.9	7.43	<LLOQ(1.36)	5.02	<LLOQ(1.00)**	4.53	1.05
2363	29.7	3.52	2.65	6.43	<LLOQ(1.00)**	3.67	3.12
2365	42.4	3.56	7.62	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	3.33
2366	65.7	5.24	7.92	4.39	<LLOQ(1.00)**	<LLOQ(2.50)	4.88
2368	42.8	5.39	10.5	2.50	<LLOQ(1.00)**	3.77	3.54
2376	43.2	4.20	4.50	3.41	<LLOQ(1.00)**	<LLOQ(2.50)	4.58
2386	11.2	<LLOQ(1.92)	<LLOQ(1.36)	5.19	<LLOQ(1.00)**	<LLOQ(2.50)	<LLOQ(1.00)
2395	61.8	4.45	4.20	10.3	<LLOQ(1.00)**	13.4	19.5
2397	77.2	7.81	27.2	7.78	<LLOQ(1.00)**	5.09	2.89
2400	62.4	7.37	10.5	3.39	<LLOQ(1.00)**	2.55	10.4
2406	29.1	3.76	<LLOQ(1.36)	<LLOQ(1.60)	<LLOQ(1.00)**	3.62	3.5

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
2431	50.6	4.80	5.57	2.82	<LLOQ(1.00)**	9.74	9.19
2433	27.7	5.50	2.93	5.46	<LLOQ(1.00)**	3.47	<LLOQ(1.00)
2436	33.0	9.32	3.45	2.95	<LLOQ(1.00)**	4.67	2.81
2438	61.9	7.05	3.34	11.2	<LLOQ(1.00)**	4.89	7.48
2440	23.6	6.95	1.66	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	<LLOQ(1.00)
2474	42.9	3.60	1.79	9.55	<LLOQ(1.00)**	3.25	1.35
2491	40.1	5.82	10.3	2.44	<LLOQ(1.00)**	<LLOQ(2.50)	1.49
2493	34.4	7.11	5.99	6.14	<LLOQ(1.00)**	4.34	2
2495	20.8	3.08	2.53	<LLOQ(1.60)	<LLOQ(1.00)**	2.58	8.11
2496	28.0	5.40	6.77	5.36	<LLOQ(1.00)**	3.15	5.24
2499	28.0	2.42	73.9	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	<LLOQ(1.00)
2500	26.3	3.02	10.2	<LLOQ(1.60)	<LLOQ(1.00)**	<LLOQ(2.50)	<LLOQ(1.00)
2514	36.9	4.05	4.29	4.66	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2516	54.1	6.95	2.08	10.1	<LLOQ(1.00)	3.68	2.41
2517	51.6	11.3	73.4	3.02	<LLOQ(1.00)	4.49	<LLOQ(1.00)
2522	30.2	3.27	1.39	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2524	38.6	3.68	2.02	4.65	<LLOQ(1.00)	4.69	3.9

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
2526	37.0	4.49	3.65	4.26	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2528	122	5.87	2.90	4.63	<LLOQ(1.00)	12.3	34.4
2531	27.3	2.75	1.99	1.96	<LLOQ(1.00)	<LLOQ(2.50)	4.19
2567	29.0	4.44	<LLOQ(1.36)	2.21	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2570	42.7	3.71	<LLOQ(1.36)	3.73	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2575	52.1	5.92	32.6	8.19	<LLOQ(2.00)	7.53	<LLOQ(2.00)
2577	21.6	<LLOQ(1.92)	2.17	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2584	217	34.2	497	8.82	<LLOQ(1.00)	6.27	2.92
2585	58.5	6.27	1.66	9.68	<LLOQ(1.00)	10.9	22.2
2590	45.0	5.52	2.12	2.13	<LLOQ(1.00)	<LLOQ(2.50)	10.8
2593	32.9	6.98	<LLOQ(1.36)	2.10	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
2597	43.8	5.67	15.2	3.85	<LLOQ(1.00)	<LLOQ(2.50)	3.16
9433	38.2	9.14	4.97	5.78	<LLOQ(2.00)	5.43	<LLOQ(2.00)
9434	45.7	4.55	11.7	3.71	<LLOQ(2.00)	<LLOQ(5.00)	2.23
9435	35.7	6.29	25.5	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9436	35.1	6.78	<LLOQ(2.41)	4.01	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9437	14.7	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
9438	47.7	8.15	5.81	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9439	40.5	6.54	6.65	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	10.3
9440	15.0	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9441	48.0	5.05	<LLOQ(2.41)	4.23	<LLOQ(2.00)	<LLOQ(5.00)	2.37
9442	32.3	4.41	4.22	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9443	51.6	7.49	<LLOQ(2.41)	7.41	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9444	42.1	6.64	9.06	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	5.59
9445	98.9	6.35	88.7	3.61	<LLOQ(2.00)	<LLOQ(5.00)	4.88
9446	25.2	3.73	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9447	39.8	<LLOQ(2.88)	33.0	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9448	20.4	3.31	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9449	45.1	5.66	<LLOQ(2.41)	4.60	<LLOQ(2.00)	<LLOQ(5.00)	6.54
9450	29.8	3.18	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	4.1
9451	40.1	3.69	<LLOQ(2.41)	5.18	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9452	43.5	4.96	22.3	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	2.95
9453	30.5	3.54	4.48	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9454	40.2	3.95	<LLOQ(2.41)	4.81	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
9455	25.8	4.74	3.84	15.8	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9456	66.1	14.94	20.5	5.70	<LLOQ(2.00)	7.24	7.38
9457	35.8	<LLOQ(2.88)	2.49	5.49	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9458	31.6	3.39	3.58	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	5.13
9459	62.8	6.12	3.35	3.32	<LLOQ(2.00)	<LLOQ(5.00)	2.08
9460	18.8	4.09	17.1	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9461	29.2	4.08	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9462	40.9	<LLOQ(2.88)	23.0	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9463	28.0	3.71	3.4	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9464	17.2	<LLOQ(2.88)	2.98	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9465	16.5	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9466	50.0	7.50	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	3.67
9467	49.7	5.98	<LLOQ(2.41)	2.71	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9468	49.1	7.10	<LLOQ(2.41)	4.24	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9469	80.5	18.64	38.1	10.5	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9470	28.9	5.32	7.78	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9471	55.6	11.5	40.2	10.8	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
9472	54.9	3.93	9.60	3.56	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9473	34.7	4.96	12.2	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9474	43.3	4.95	6.56	2.74	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9475	57.2	7.03	4.70	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9476	40.0	<LLOQ(2.88)	10.2	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9477	22.4	2.97	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9478	33.6	5.15	<LLOQ(2.41)	3.89	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9479	14.1	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9480	42.2	5.99	2.58	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9481	33.8	5.37	<LLOQ(2.41)	6.08	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9482	33.9	<LLOQ(2.88)	12.39	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	2.79
9483	24.7	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9484	10.2	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9485	37.4	4.45	6.54	4.76	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9486	32.7	4.23	<LLOQ(2.41)	4.41	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9487	46.7	6.81	6.40	3.59	<LLOQ(2.00)	5.26	3.12
9488	22.0	4.36	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample No.	PFOS Concentration	PFOA Concentration*	PFHS Concentration*	PFOSAA Concentration*	PFOSA Concentration*	M556 Concentration*	M570 Concentration*
9489	27.0	3.04	<LLOQ(2.41)	6.87	<LLOQ(2.00)	<LLOQ(5.00)	11.3
9490	21.2	3.88	12.2	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9491	60.9	6.41	37.6	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	2.45
9492	30.3	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9493	31.6	5.43	<LLOQ(2.41)	3.83	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9494	20.3	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9495	41.4	7.57	4.56	9.31	<LLOQ(2.00)	<LLOQ(5.00)	8.68
9496	33.1	4.76	3.10	3.04	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9497	97.9	14.64	114	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9498	49.7	2.93	67.3	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9499	29.3	3.76	8.67	2.61	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9500	30.3	<LLOQ(2.88)	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9501	35.6	4.70	<LLOQ(2.41)	5.47	<LLOQ(2.00)	<LLOQ(5.00)	3.02
9502	17.8	<LLOQ(2.88)	<LLOQ(2.41)	2.74	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9503	29.3	3.22	<LLOQ(2.41)	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)
9504	34.3	5.14	10.4	<LLOQ(2.60)	<LLOQ(2.00)	<LLOQ(5.00)	2.62
9505	27.0	4.75	4.54	5.37	<LLOQ(2.00)	<LLOQ(5.00)	<LLOQ(2.00)

* Serum sample results obtained using plasma curves may vary from results obtained from serum curves (see Results and Discussion).

** Results are from a rejected run (see Results and Discussion).

The concentrations of any diluted samples have been corrected for the persistent level of analyte in the dilution matrix.

The method acceptance criteria states the concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error of any individual result may exceed the QC acceptance criteria.

Table 23. Repeat Analysis Table for PFOS

Sample No.	Treatment	Time	Original Conc. ppb	Original Run Number	Reason for Reassay	Reassay Conc. ppb	Reassay Run Number	Reported Conc. ppb	Reason for Reported Conc.
2168	Ped	0h	>ULOQ(414)	10	1	515	14	515	1

Table 24. Repeat Analysis Table for PFHS

Sample No.	Treatment	Time	Original Conc. ppb	Original Run Number	Reason for Reassay	Reassay Conc. ppb	Reassay Run Number	Reported Conc. ppb	Reason for Reported Conc.
2168	Ped	0h	>ULOQ(523)	10	1	712	14	712	1

REASONS FOR REASSAY:
1). Greater than the ULOQ.

REASONS FOR REPORTED CONC:
1). Original result outside of quantitation range. Reassay results reported.

Figure 1. Representative Calibration Curve for PFOS

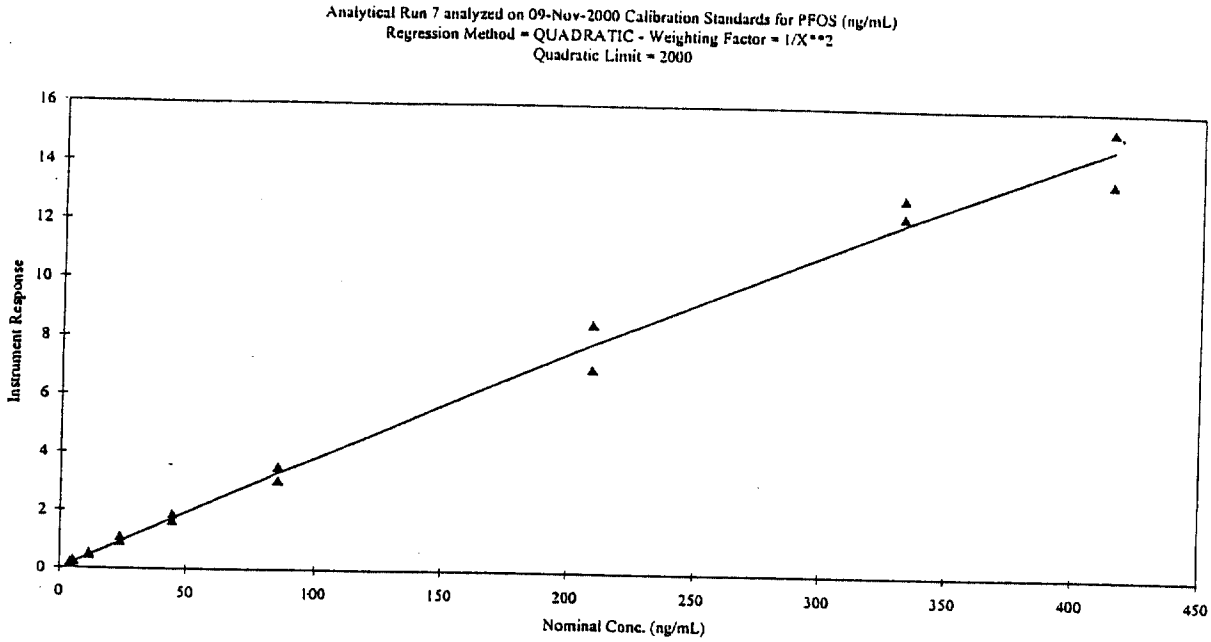

Figure 2. Representative Calibration Curve for PFOA

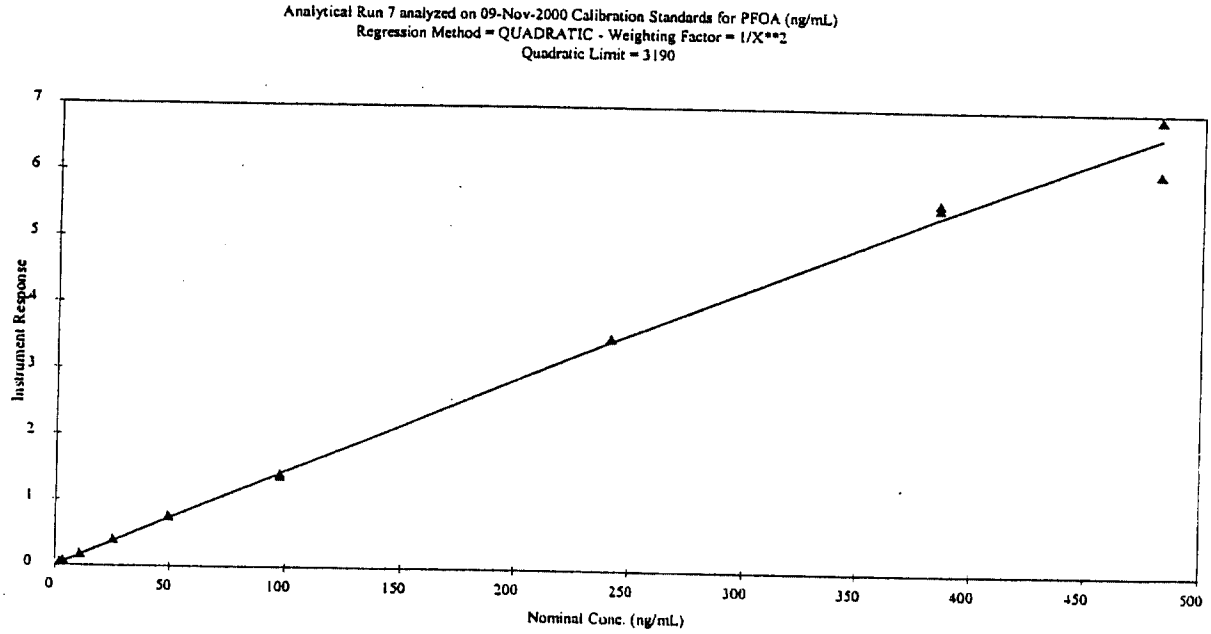

Figure 3. Representative Calibration Curve for PFHS

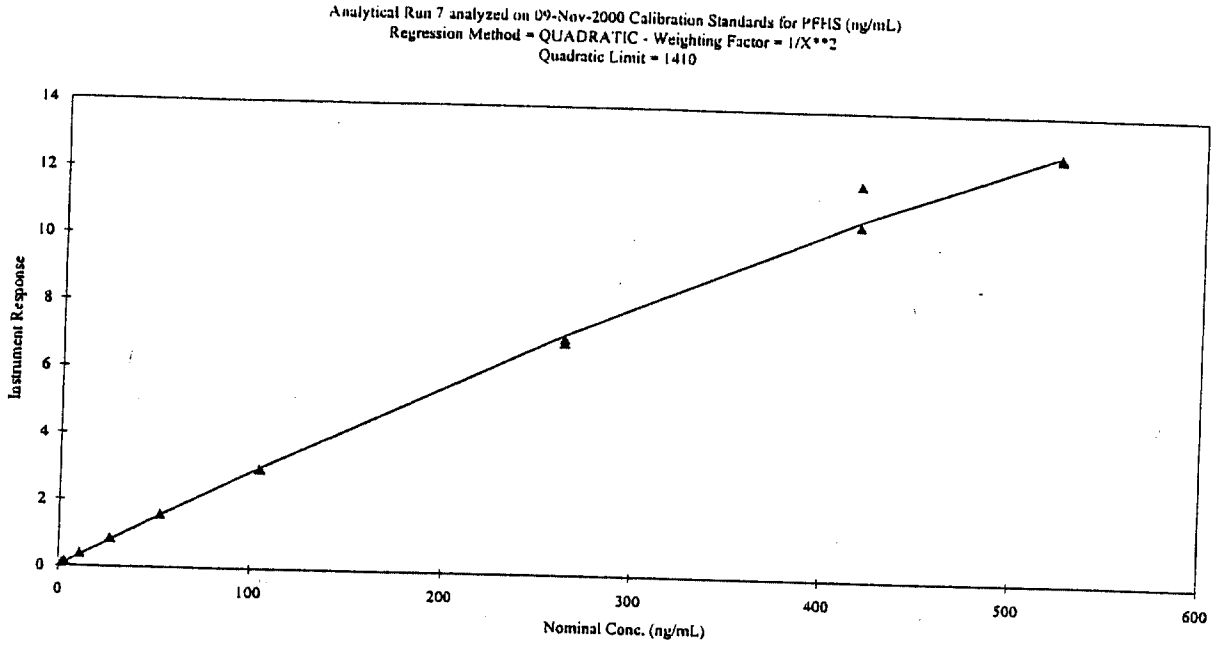

Figure 4. Representative Calibration Curve for PFOSAA

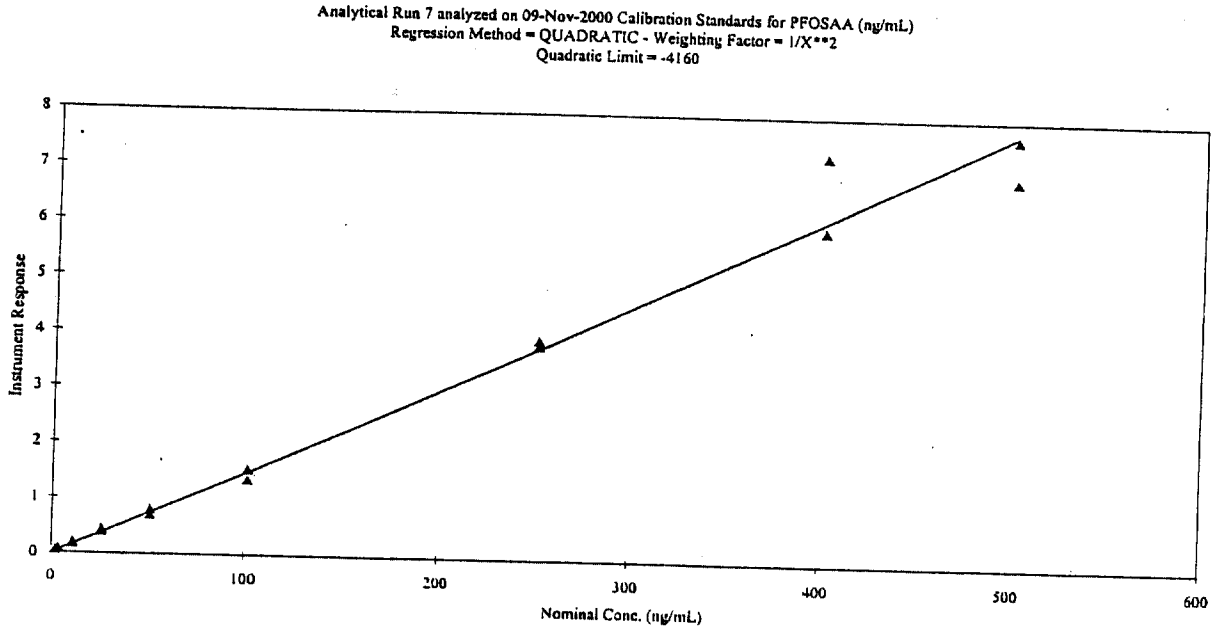

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Figure 5. Representative Calibration Curve for PFOSA

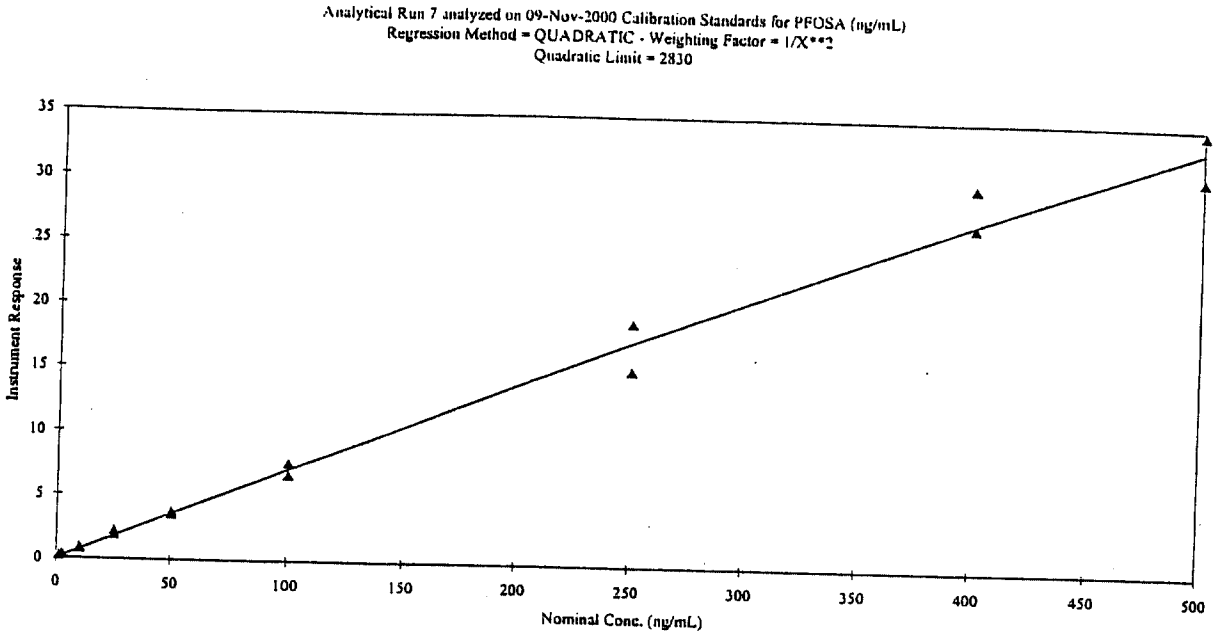


Figure 6. Representative Calibration Curve for M556

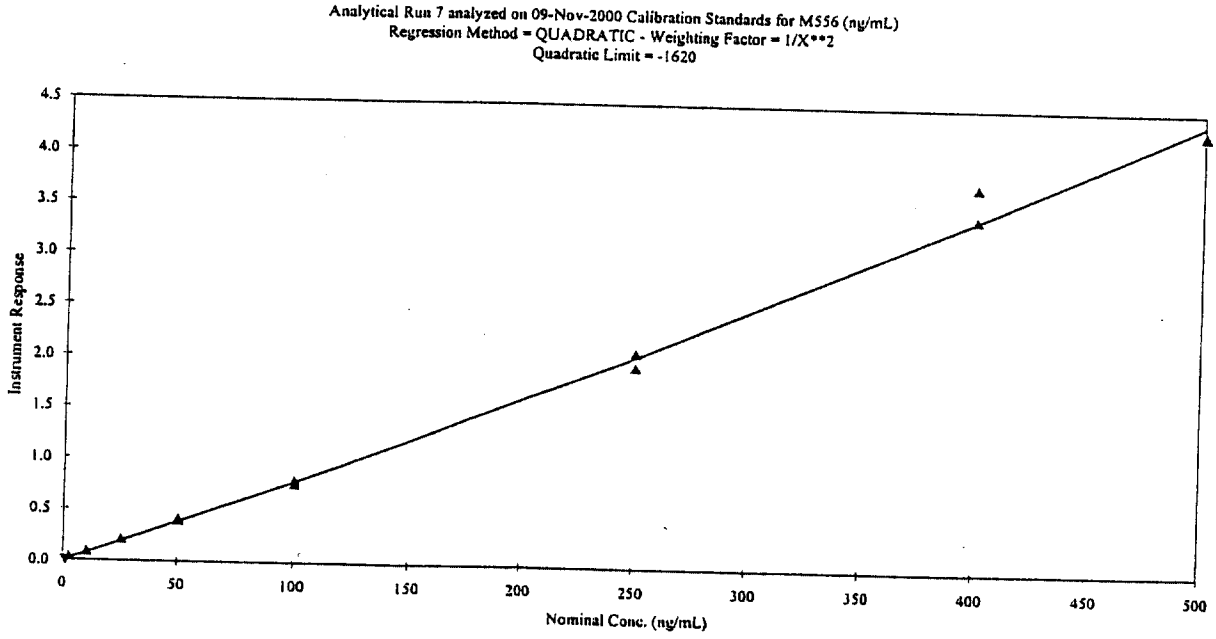


Figure 7. Representative Calibration Curve for M570

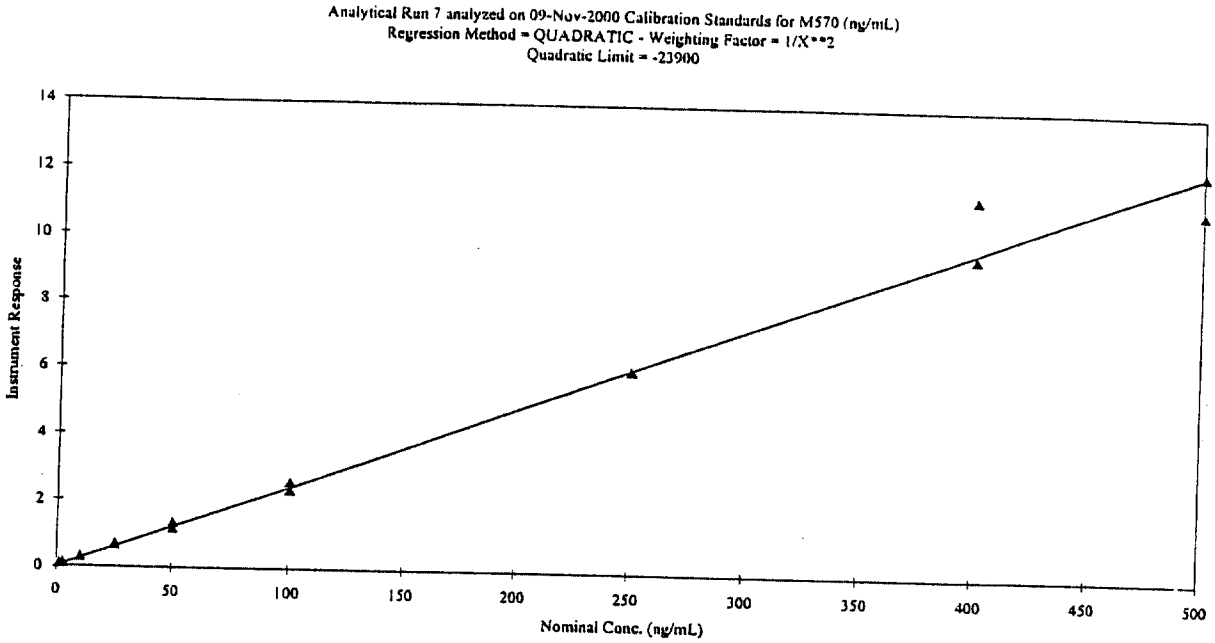


Figure 8. Human Serum Blank for PFOS

PFOS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:54

	Current	Method
Noise Thres.	5.0	5.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

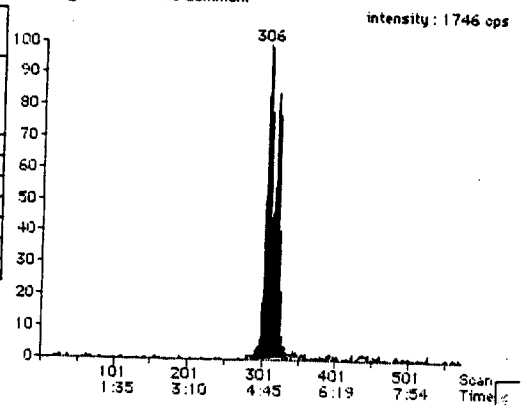
s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

499.0->80.0

Area	26722
Height	1744
Start Time	4:30
End Time	5:17
Integration Width	0:46.3
Retention Time	4:49
Integration Type	A - BV



THPFOS
use as Internal Standard

Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	

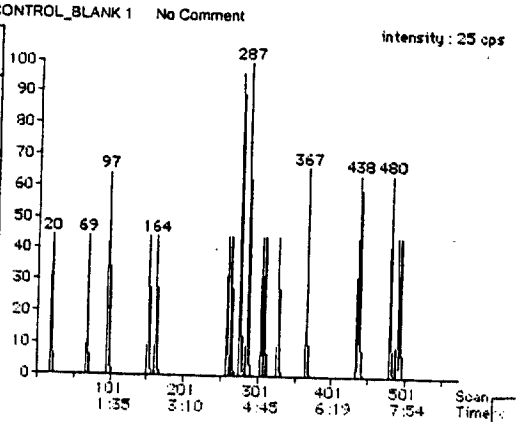


Figure 9. Human Serum Blank for PFOA

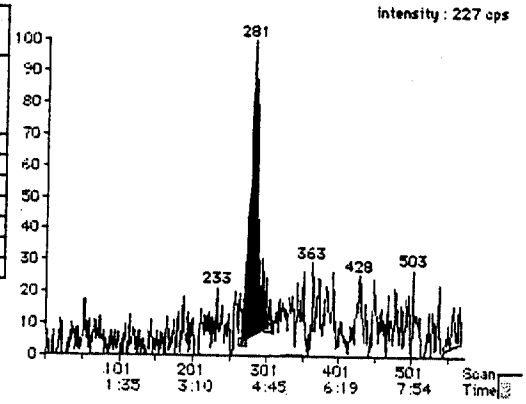
PFOA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:27

	Current	Method
Noise Thres.	1.0	1.0
Quant Thres.	0.9	0.9
Min. Width	3	3
Mult. Width	9	9
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period
1: 8:59 MRM, 571 scans
413.0->169.0

Area	2855
Height	213
Start Time	4:13
End Time	4:46
Integration Width	0:32.1
Retention Time	4:28
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period
1: 8:59 MRM, 571 scans
427.0->407.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	

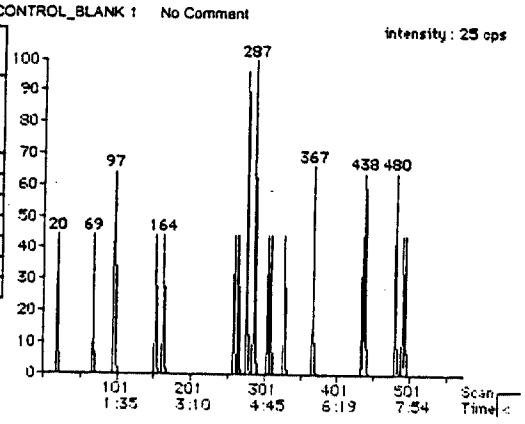


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Figure 10. Human Serum Blank for PFHS

PFHS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 3:37

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	4	4
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

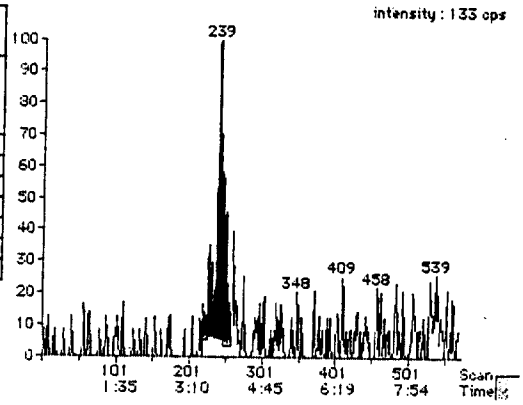
s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

399.0->80.0

Area	1410
Height	128
Start Time	3:29
End Time	3:59
Integration Width	0:30.3
Retention Time	3:46
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

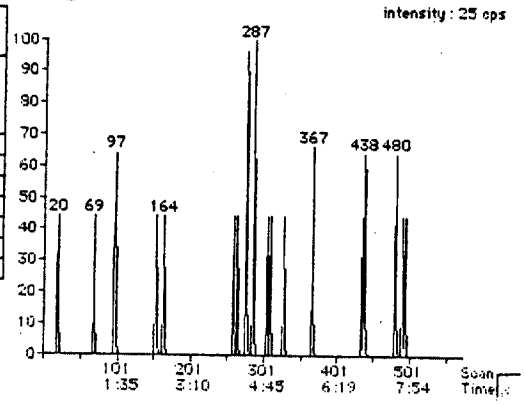
s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	



000437

Figure 11. Human Serum Blank for PFOSAA

PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:56

	Current	Method
Noise Thres.	4.0	4.0
Quant Thres.	1.5	1.5
Min. Width	3	3
Mult. Width	13	13
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

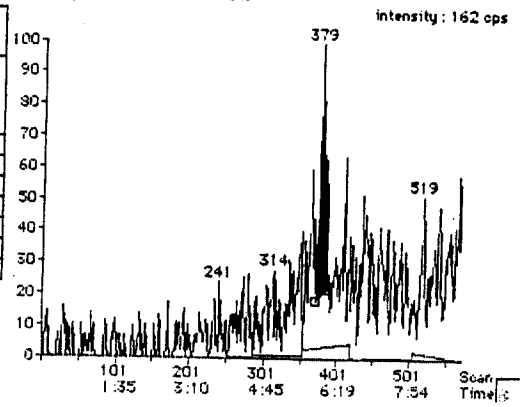
s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

584.1->419.1

Area	1020
Height	129
Start Time	5:51
End Time	6:06
Integration Width	0:15.1
Retention Time	5:58
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	

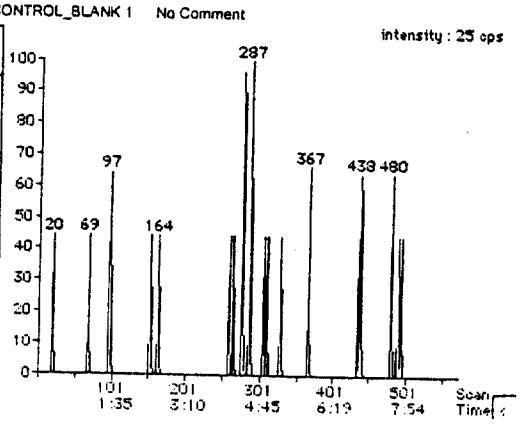


Figure 12. Human Serum Blank for PFOSA

PFOSA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:35

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.3	0.3
Min. Width	5	5
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

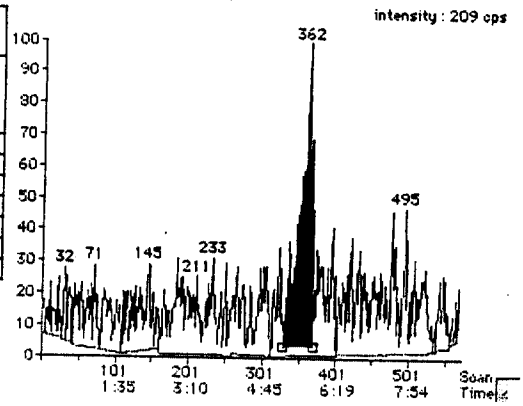
s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

498.0->78.0

Area	3039
Height	202
Start Time	5:11
End Time	5:50
Integration Width	0:38.8
Retention Time	5:42
Integration Type	A - VV



THPFOS
use as Internal Standard

Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

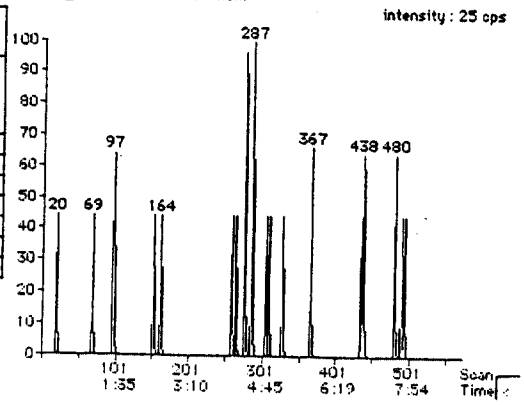
s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	



030439

Figure 13. Human Serum Blank for M556

M556

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5:25

	Current	Method
Noise Thres.	0.4	0.4
Quant Thres.	0.1	0.1
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

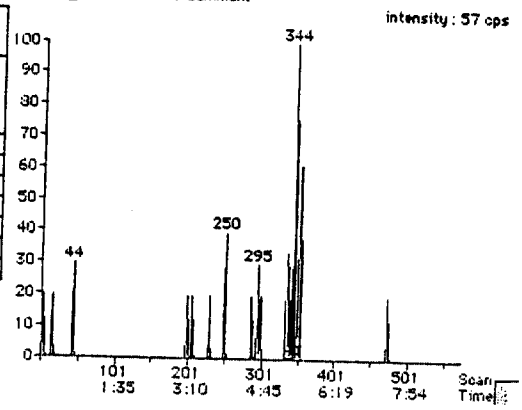
s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

556.0->498.0

Area	155
Height	55
Start Time	5:20
End Time	5:27
Integration Width	0:07.6
Retention Time	5:25
Integration Type	A - BB



THPFOS

use as Internal Standard

Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	

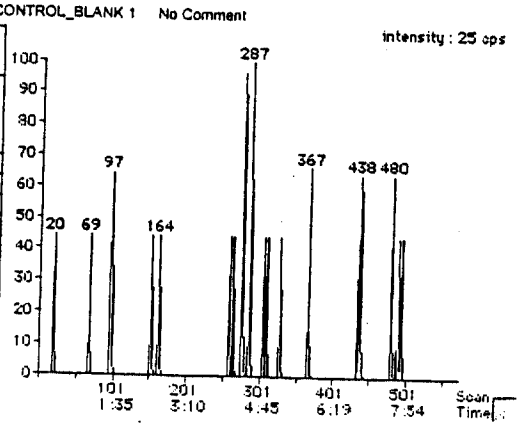


Figure 14. Human Serum Blank for M570

M570
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:38

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	5	5
Mult. Width	15	15
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

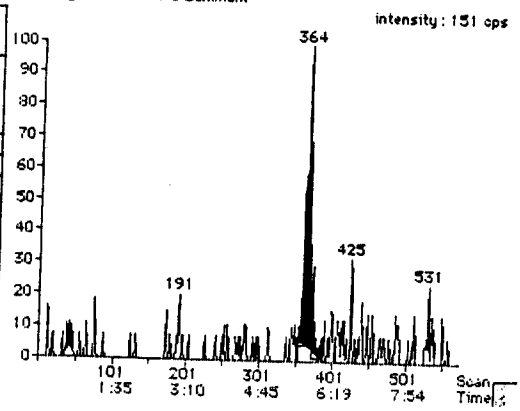
s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

570.0->419.1

Area	1074
Height	144
Start Time	5:37
End Time	5:58
Integration Width	0:18.9
Retention Time	5:44
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-012 7 012 S00088 CONTROL_BLANK 1 No Comment

Thu, Nov 9, 2000 9:08 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	

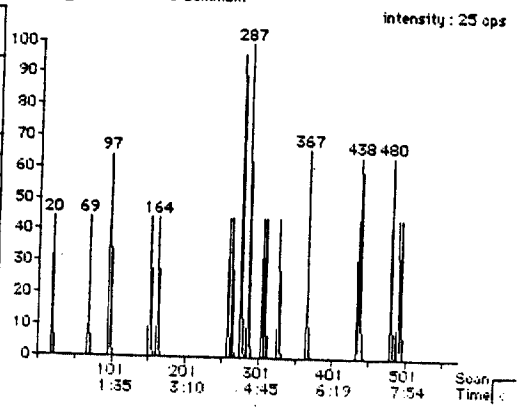


Figure 15. Human Serum Blank with Internal Standard (QC0) for PFOS

PFOS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:54

	Current	Method
Noise Thres.	5.0	5.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

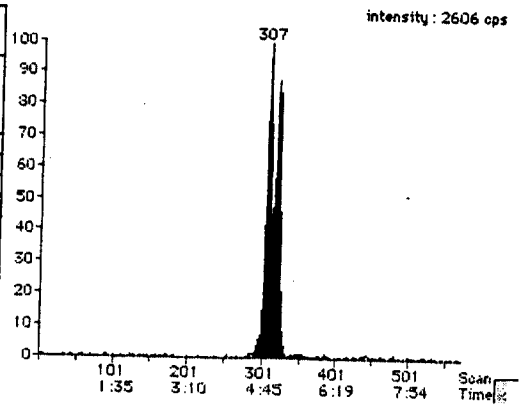
s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

499.0->80.0

Area	39395
Height	2605
Start Time	4:34
End Time	5:32
Integration Width	0:57.7
Retention Time	4:50
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	273196
Height	35433
Start Time	4:12
End Time	4:46
Integration Width	0:33.1
Retention Time	4:26
Integration Type	A - BB

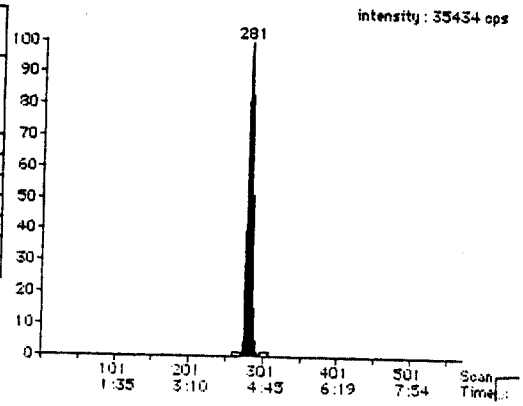


Figure 16. Human Serum Blank with Internal Standard (QC0) for PFOA

PFOA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:27

	Current	Method
Noise Thres.	1.0	1.0
Quant Thres.	0.9	0.9
Min. Width	3	3
Mult. Width	9	9
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

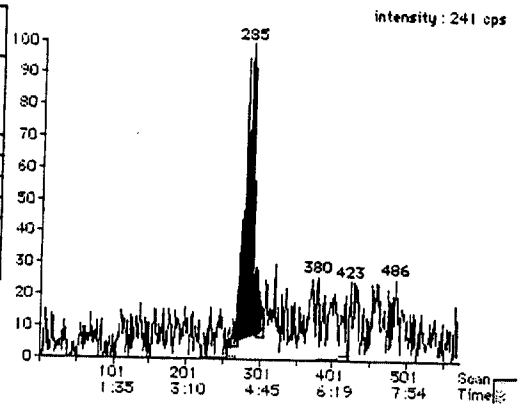
s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

413.0->169.0

Area	3134
Height	227
Start Time	4:12
End Time	4:45
Integration Width	0:33.1
Retention Time	4:29
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	273196
Height	35433
Start Time	4:12
End Time	4:46
Integration Width	0:33.1
Retention Time	4:26
Integration Type	A - BB

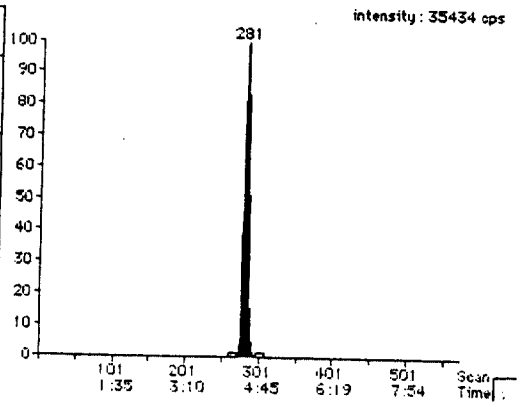


Figure 17. Human Serum Blank with Internal Standard (QC0) for PFHS

PFHS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 3:37

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	4	4
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

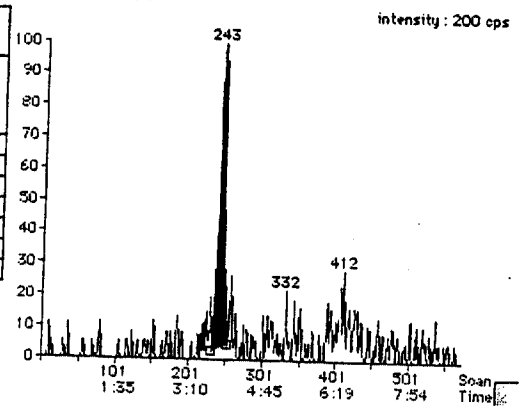
s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

399.0->80.0

Area	1769
Height	193
Start Time	3:39
End Time	3:59
Integration Width	0:19.9
Retention Time	3:50
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	273198
Height	35433
Start Time	4:12
End Time	4:46
Integration Width	0:33.1
Retention Time	4:28
Integration Type	A - BB

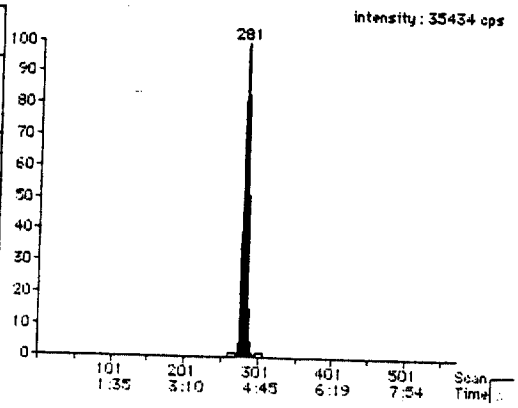


Figure 18. Human Serum Blank with Internal Standard (QC0) for PFOSAA

PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:56

	Current	Method
Noise Thres.	4.0	4.0
Quant Thres.	1.5	1.5
Min. Width	3	3
Mult. Width	13	13
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

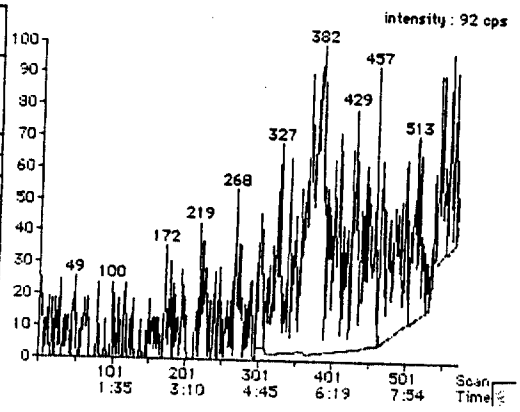
s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

584.1->419.1

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	273196
Height	35433
Start Time	4:12
End Time	4:46
Integration Width	0:33.1
Retention Time	4:26
Integration Type	A - BB

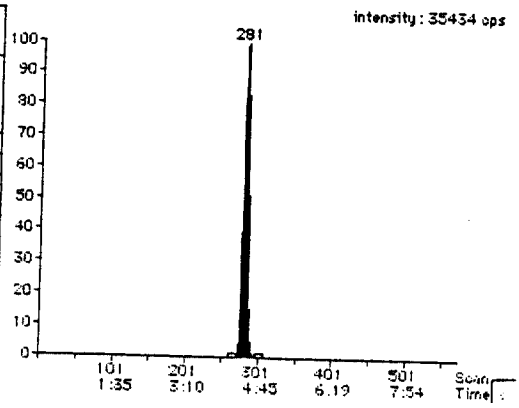


Figure 19. Human Serum Blank with Internal Standard (QC0) for PFOSA

PFOSA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:35

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.3	0.3
Min. Width	5	5
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

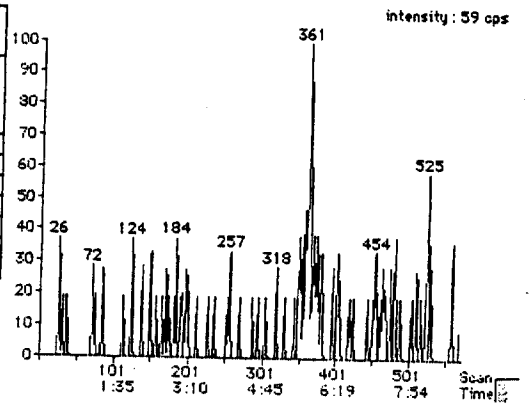
s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

498.0->78.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	273196
Height	35433
Start Time	4:12
End Time	4:48
Integration Width	0:33.1
Retention Time	4:26
Integration Type	A - BB

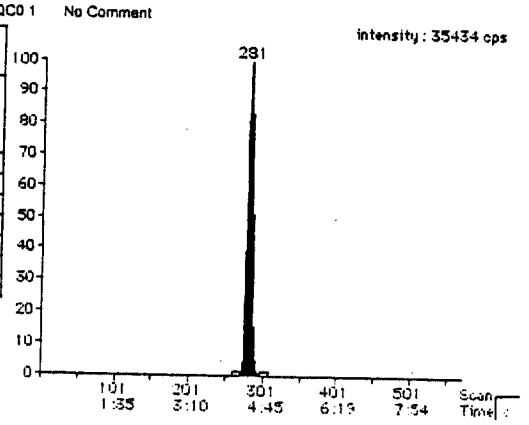
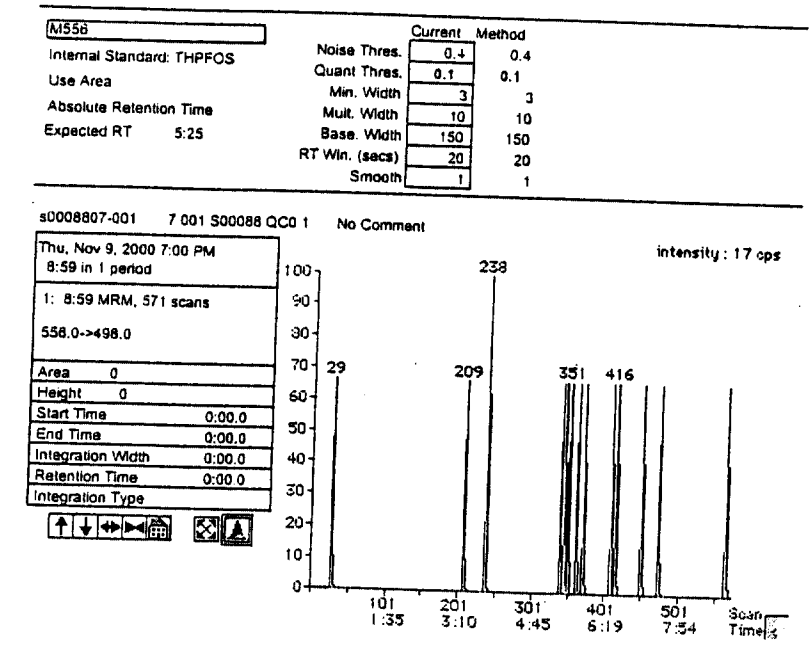


Figure 20. Human Serum Blank with Internal Standard (QC0) for M556



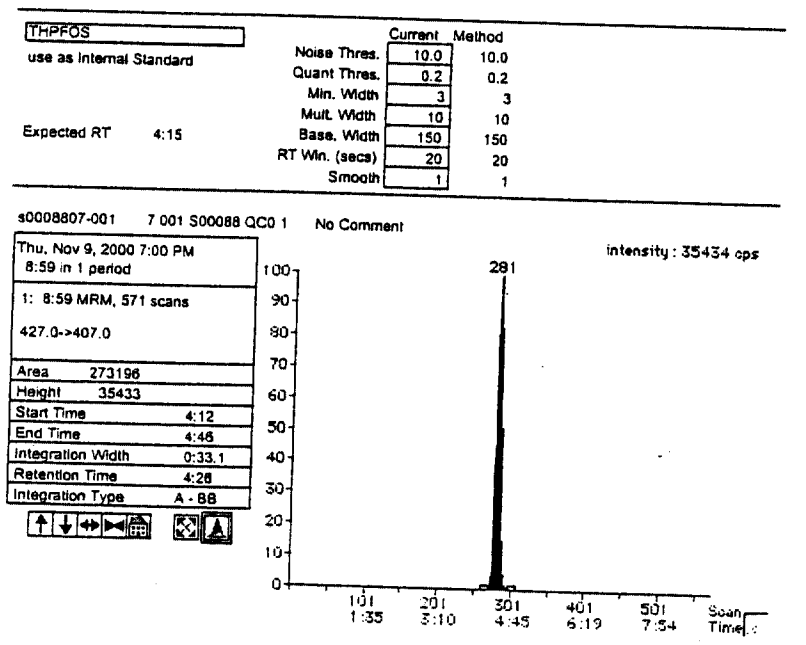


Figure 21. Human Serum Blank with Internal Standard (QC0) for M570

M570
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:38

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	5	5
Mult. Width	15	15
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

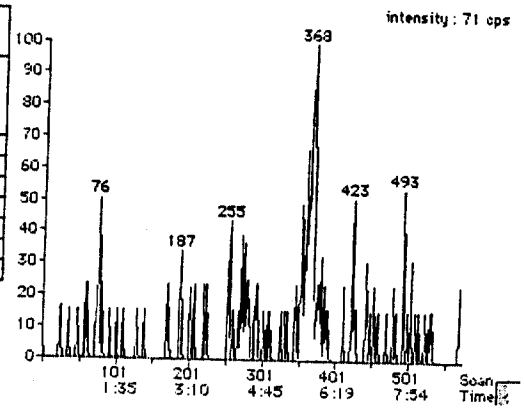
s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

570.0->419.1

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-001 7 001 S00088 QC0 1 No Comment

Thu, Nov 9, 2000 7:00 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	273196
Height	35433
Start Time	4:12
End Time	4:46
Integration Width	0:33.1
Retention Time	4:26
Integration Type	A - BB

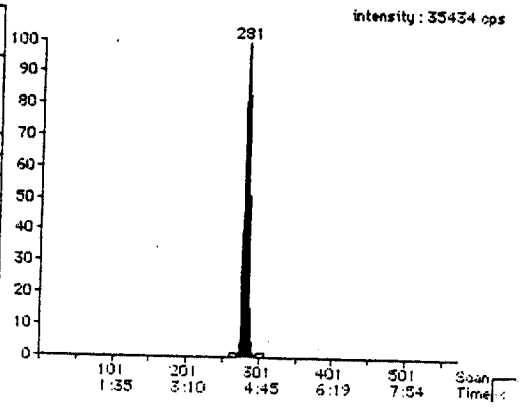


Figure 22. Low Standard (3.94 ppb) for PFOS

PFOS

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 4:54

	Current	Method
Noise Thres.	5.0	5.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

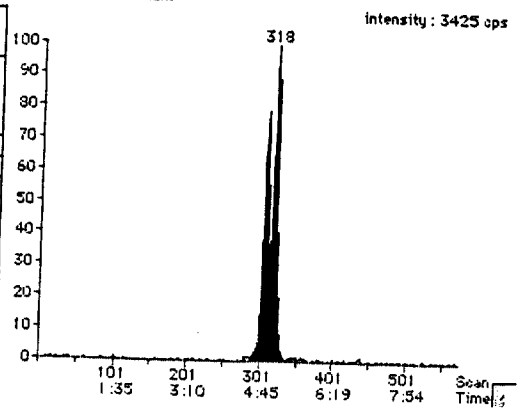
s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

499.0->80.0

Area	48519
Height	3423
Start Time	4:31
End Time	5:28
Integration Width	0:56.7
Retention Time	5:01
Integration Type	A - BV



THPFOS

use as Internal Standard

Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	270289
Height	36113
Start Time	4:12
End Time	4:47
Integration Width	0:35.0
Retention Time	4:25
Integration Type	A - BB

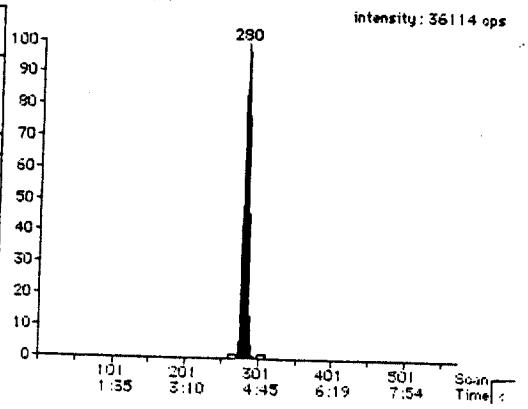


Figure 23. Low Standard (1.92 ppb) for PFOA

PFOA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:27

	Current	Method
Noise Thres.	1.0	1.0
Quant Thres.	0.9	0.9
Min. Width	3	3
Mult. Width	9	9
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

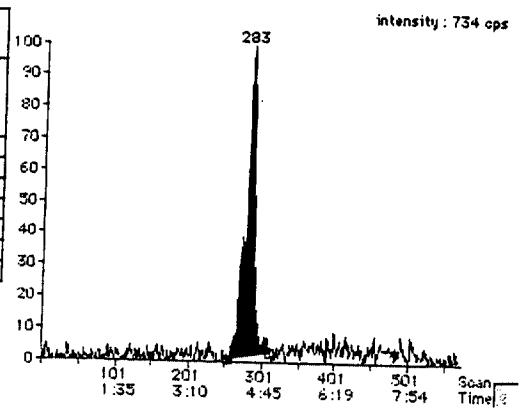
s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

413.0->169.0

Area	9583
Height	722
Start Time	4:02
End Time	4:55
Integration Width	0:52.9
Retention Time	4:28
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	270289
Height	36113
Start Time	4:12
End Time	4:47
Integration Width	0:35.0
Retention Time	4:25
Integration Type	A - BB

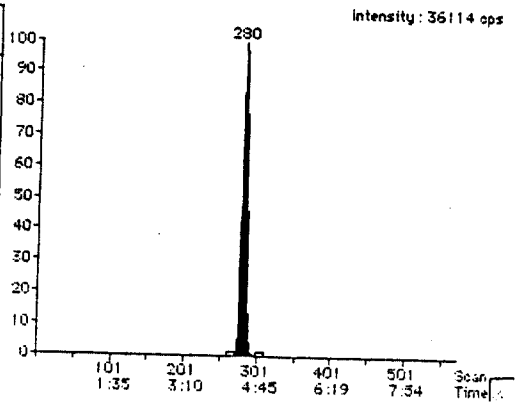


Figure 24. Low Standard (1.36 ppb) for PFHS

PFHS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 3:37

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	4	4
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

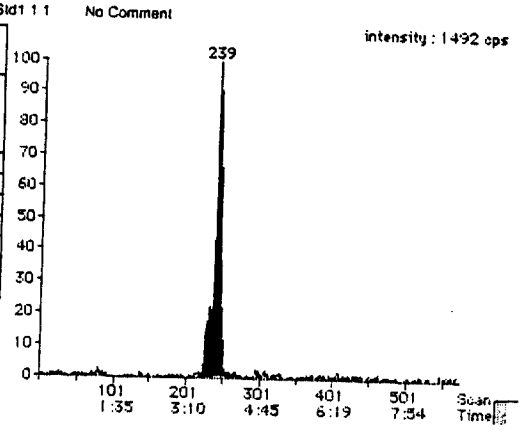
s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

399.0->80.0

Area	14877
Height	1486
Start Time	3:25
End Time	4:01
Integration Width	0:35.9
Retention Time	3:46
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	270289
Height	36113
Start Time	4:12
End Time	4:47
Integration Width	0:35.0
Retention Time	4:25
Integration Type	A - BB

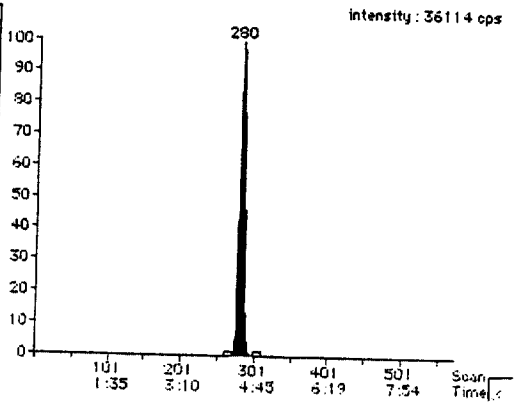


Figure 25. Low Standard (1.60 ppb) for PFOSAA

PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:56

	Current	Method
Noise Thres.	4.0	4.0
Quant Thres.	1.5	1.5
Min. Width	3	3
Mult. Width	13	13
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

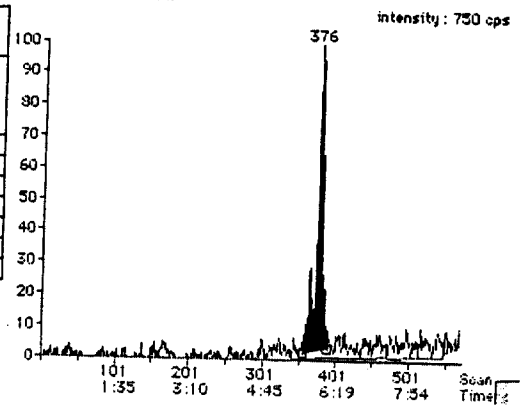
s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

584.1->419.1

Area	8884
Height	727
Start Time	5:37
End Time	6:07
Integration Width	0:30.3
Retention Time	5:56
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	270289
Height	36113
Start Time	4:12
End Time	4:47
Integration Width	0:35.0
Retention Time	4:25
Integration Type	A - BB

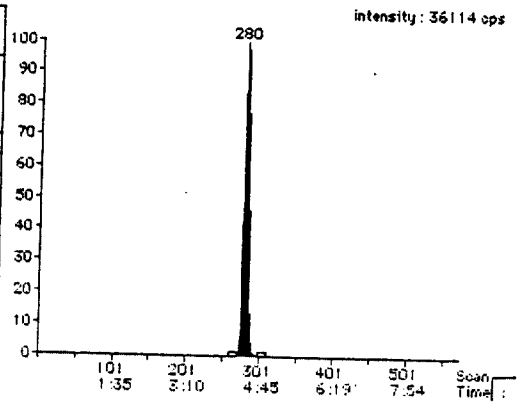


Figure 26. Low Standard (1.00 ppb) for PFOSA

PFOSA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:35

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.3	0.3
Min. Width	5	5
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

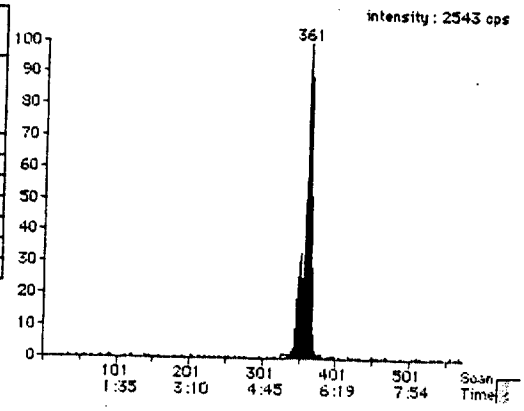
s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

498.0->78.0

Area	23612
Height	2540
Start Time	5:15
End Time	5:55
Integration Width	0:39.7
Retention Time	5:41
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-002 7 002 S00088 Std1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	270289
Height	36113
Start Time	4:12
End Time	4:47
Integration Width	0:35.0
Retention Time	4:25
Integration Type	A - BB

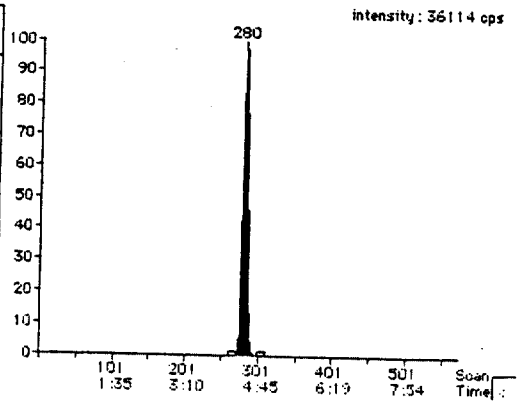


Figure 27. Low Standard (2.50 ppb) for M556

M556

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5.42

	Current	Method
Noise Thres.	0.4	0.4
Quant Thres.	0.1	0.1
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

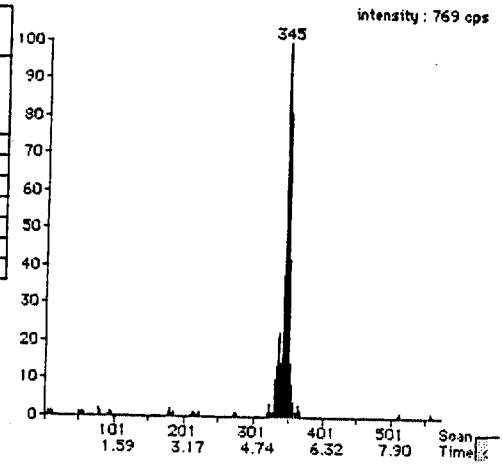
s0008807-003 7 003 S00088 Std2 1 1 No Comment

Thu, Nov 9, 2000 7:23 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

558.0->498.0

Area	5991
Height	769
Start Time	5.15
End Time	5.63
Integration Width	0.47
Retention Time	5.44
Integration Type	A - BB



THPFOS

use as Internal Standard

Expected RT 4.25

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-003 7 003 S00088 Std2 1 1 No Comment

Thu, Nov 9, 2000 7:23 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	272919
Height	35652
Start Time	4.19
End Time	4.77
Integration Width	0.58
Retention Time	4.41
Integration Type	A - BB

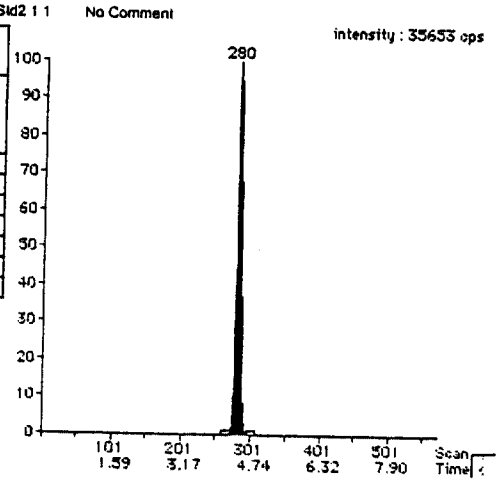


Figure 28. Low Standard (1.00 ppb) for M570

M570

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5:38

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	5	5
Mult. Width	15	15
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

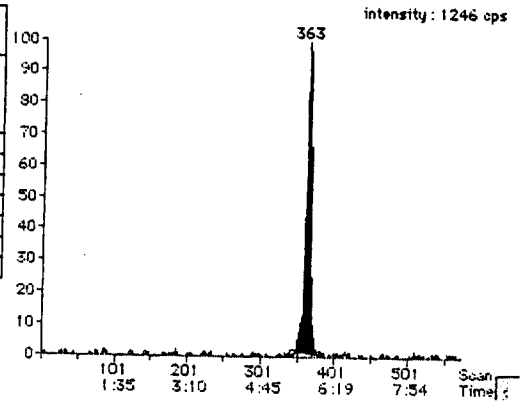
s0008807-002 7 002 S00088 Sld1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

570.0->419.1

Area	9166
Height	1236
Start Time	5:28
End Time	5:56
Integration Width	0:27.4
Retention Time	5:43
Integration Type	A - BB



THPFOS

use as internal Standard

Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-002 7 002 S00088 Sld1 1 1 No Comment

Thu, Nov 9, 2000 7:11 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	270289
Height	36113
Start Time	4:12
End Time	4:47
Integration Width	0:35.0
Retention Time	4:25
Integration Type	A - BB

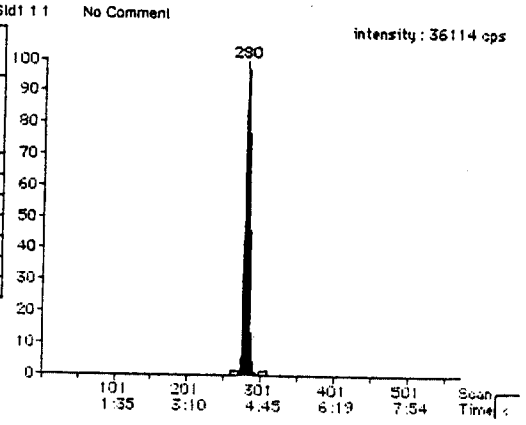


Figure 29. High Standard (414 ppb) for PFOS

PFOS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:54

	Current	Method
Noise Thres.	5.0	5.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

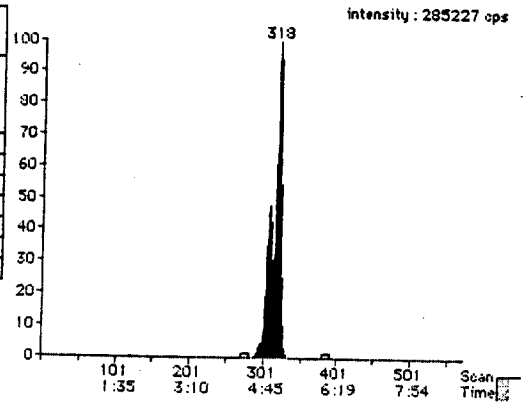
s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

499.0->80.0

Area	3453475
Height	285223
Start Time	4:22
End Time	8:04
Integration Width	1:42
Retention Time	5:01
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	225323
Height	28708
Start Time	4:12
End Time	4:46
Integration Width	0:34.0
Retention Time	4:26
Integration Type	A - BB

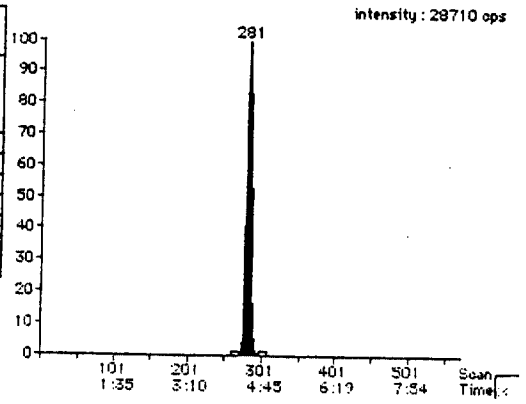


Figure 30. High Standard (481 ppb) for PFOA

PFOA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:27

	Current	Method
Noise Thres.	1.0	1.0
Quant Thres.	0.9	0.9
Min. Width	3	3
Mult. Width	9	9
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

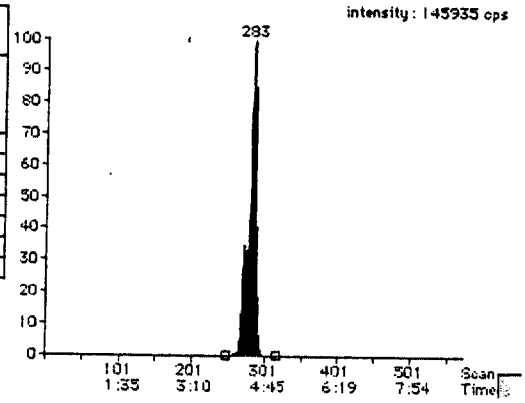
s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

413.0->169.0

Area	1553455
Height	145923
Start Time	3:55
End Time	4:59
Integration Width	1:03
Retention Time	4:28
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	225323
Height	28708
Start Time	4:12
End Time	4:46
Integration Width	0:34.0
Retention Time	4:26
Integration Type	A - BB

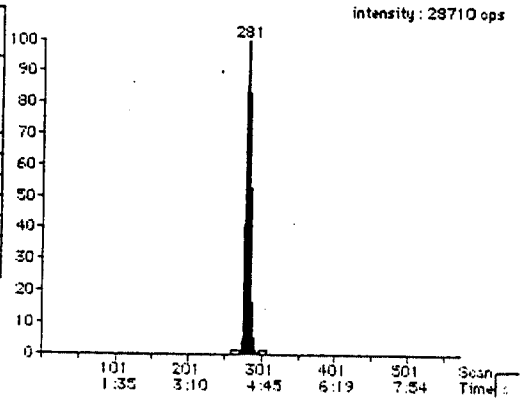


Figure 31. High Standard (523 ppb) for PFHS

PFHS

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 3:37

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	4	4
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

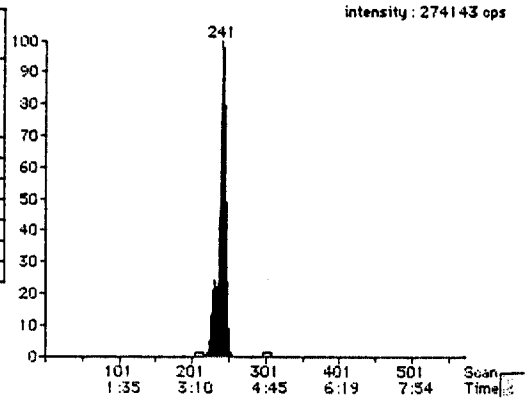
s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

399.0->80.0

Area	2879090
Height	274134
Start Time	3:20
End Time	4:46
Integration Width	1:25
Retention Time	3:48
Integration Type	A - BV



THPFOS

use as Internal Standard

Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	225323
Height	28708
Start Time	4:12
End Time	4:46
Integration Width	0:34.0
Retention Time	4:26
Integration Type	A - BB

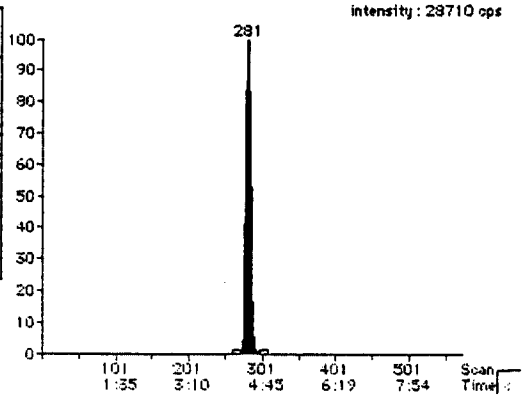


Figure 32. High Standard (501 ppb) for PFOSAA

PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:56

	Current	Method
Noise Thres.	4.0	4.0
Quant Thres.	1.5	1.5
Min. Width	3	3
Mult. Width	13	13
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

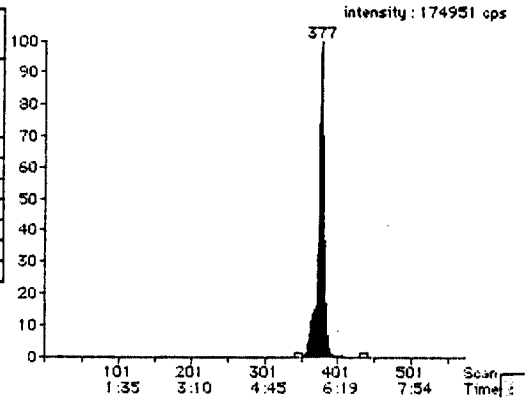
s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

584.1->419.1

Area	1556445
Height	174924
Start Time	5:29
End Time	6:52
Integration Width	1:23
Retention Time	5:56
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	225323
Height	28708
Start Time	4:12
End Time	4:46
Integration Width	0:34.0
Retention Time	4:26
Integration Type	A - BB

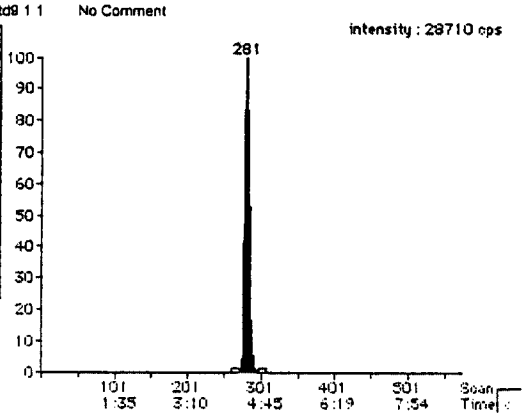


Figure 33. High Standard (500 ppb) for PFOSA

PFOSA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:35

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.3	0.3
Min. Width	5	5
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

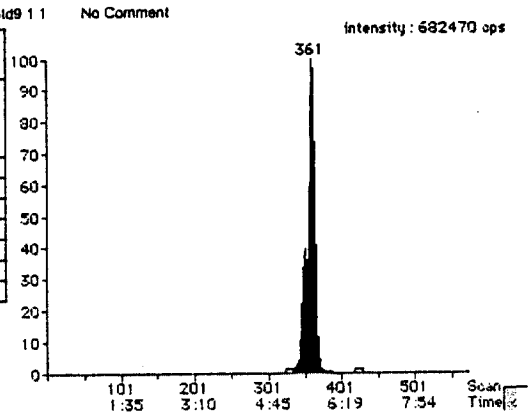
s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

498.0->78.0

Area	7778824
Height	682348
Start Time	5:12
End Time	6:39
Integration Width	1:27
Retention Time	5:41
Integration Type	A - BV



THPFOS
use as Internal Standard

Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	225323
Height	28708
Start Time	4:12
End Time	4:46
Integration Width	0:34.0
Retention Time	4:26
Integration Type	A - BB

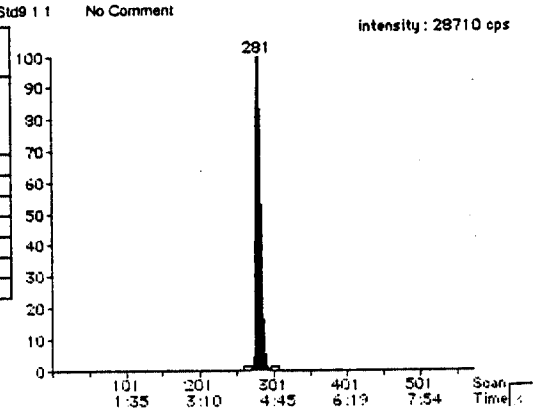


Figure 34. High Standard (500 ppb) for M556

M556
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:25

	Current	Method
Noise Thres.	0.4	0.4
Quant Thres.	0.1	0.1
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

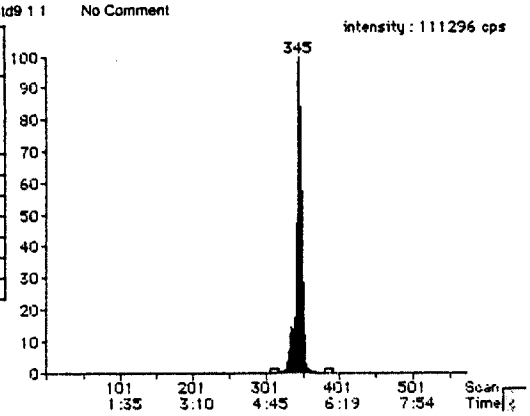
s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

556.0->498.0

Area	964595
Height	111295
Start Time	4:55
End Time	6:04
Integration Width	1:09
Retention Time	5:26
Integration Type	A - BV



THPFOS
use as Internal Standard

Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

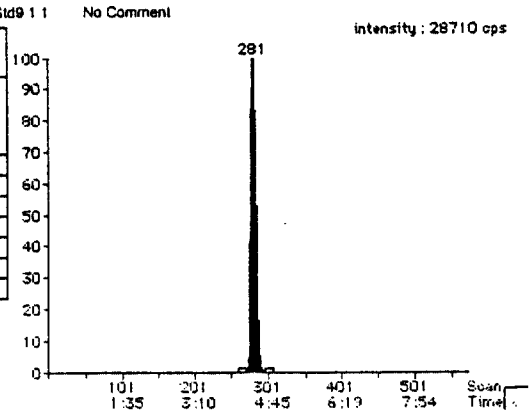
s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	225323
Height	28708
Start Time	4:12
End Time	4:46
Integration Width	0:34.0
Retention Time	4:26
Integration Type	A - BB



060461

Figure 35. High Standard (500 ppb) for M570

M570
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:38

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	5	5
Mult. Width	15	15
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

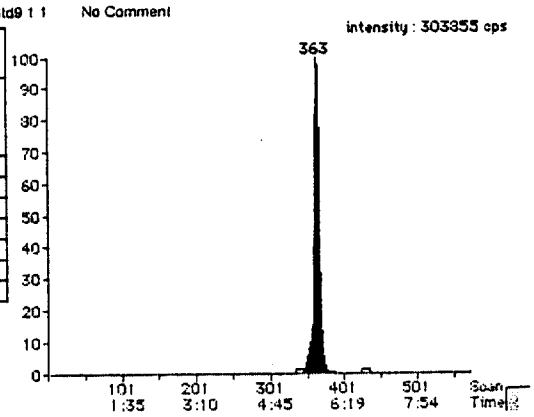
s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

570.0->419.1

Area	2494791
Height	303847
Start Time	5:21
End Time	6:46
Integration Width	1:24
Retention Time	5:43
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4:15

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

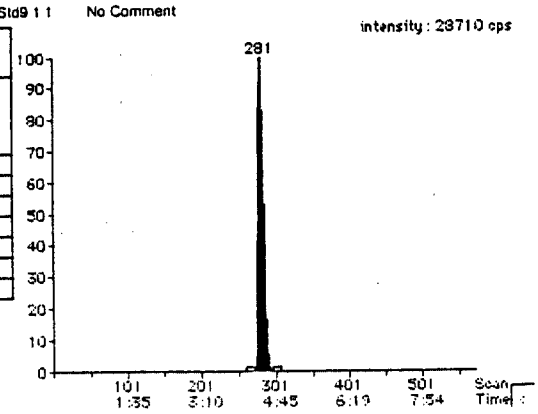
s0008807-010 7 010 S00088 Std9 1 1 No Comment

Thu, Nov 9, 2000 8:45 PM
8:59 in 1 period

1: 8:59 MRM, 571 scans

427.0->407.0

Area	225323
Height	28708
Start Time	4:12
End Time	4:48
Integration Width	0:34.0
Retention Time	4:26
Integration Type	A - BB



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Study No. NWBS00-128
Report No. NWBR01-066

Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS

Sample Analysis Report for Protocol EPI-0013

Northwest Bioanalytical (NWB)
A Division of NWT Inc.
1121 East 3900 South
Salt Lake City, UT 84124

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PREPARED FOR: 3M Company
Corporate Occupational Medicine, Medical Department
220-3W-05
St. Paul, MN 55144

AUTHOR:
Connie O. Sakashita DATE: 1-9-02
Connie O. Sakashita, B.S., NWB Project Manager

APPROVED FOR RELEASE BY:
R L Foltz DATE: 1-9-02
Rodger L. Foltz, Ph.D., NWB Technical Director

CONTAIN NO CBI

QUALITY ASSURANCE STATEMENT

LABORATORY:	Northwest Bioanalytical (NWB) A Division of NWT, Inc. 1121 East 3900 South Salt Lake City, UT 84124
SPONSOR:	3M Company Corporate Occupational Medicine, Medical Department 220-3W-05 St. Paul, MN 55144
COMPOUND(S):	PFOS, PFOA (POAA), PFHS, PFOSA, PFOSAA, M556, M570
NWB STUDY NUMBER:	NWBS00-128
SPONSOR STUDY NUMBER:	EPI-0013
NWB STUDY TITLE:	Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS

The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all projects in accordance with the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.) The following inspections were performed by the NWB QAU per SOP.

Inspection and Reporting Statement.

Inspection Date	Phase of Study	Date Inspection Report Issued To: NWB Project Manager	Date Inspection Report Issued To: [1]NWB Management
28 Nov 2000	Analytical Plan	28 Nov 2000	30 Nov 2000
20-23 Mar 2001	Sample Analysis	23 Mar 2001	31 Mar 2001
05-06 Apr 2001	Sample Receipt	06 Apr 2001	30 Apr 2001
02-04 May 2001	QC Preparation	04 May 2001	31 May 2001
31 Dec 2001 – 07 Jan 2002	Report Draft/ Raw Data	07 Jan 2002	31 Jan 2002
09 Jan 2002	Final Report	09 Jan 2002	31 Jan 2002

[1]Reports to NWB Management are issued monthly.

As can reasonably be established, the methods and procedures described and the results incorporated into this final report accurately reflect the raw data.

Sheryl A. Mathews

Sheryl A. Mathews, A.A.S.
NWB QAU Compliance Auditor

09 Jan. 2002
Date

000464

COMPLIANCE STATEMENT

The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, to the best of our knowledge, this project was conducted in accordance with the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58) and according to the methods and procedures described within this report. In addition, the study followed the guidelines of the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.) Any known incidents that may have affected the quality or integrity of the project or reported data is included in this report. This report represents an accurate record of the raw data.

Connie O. Sakashita

Connie O. Sakashita, B.S.
NWB Project Manager

1-9-02
Date

R L Foltz

Rodger L. Foltz, Ph.D.
NWB Technical Director

1-9-02
Date

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Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS

Sample Analysis for Protocol EPI-0013

1. INTRODUCTION

This report summarizes the analytical results from the quantitation of PFOS, PFHS, PFOA, PFOSAA, PFOSA, M556, and M570 in human serum samples for 3M Company in support of Protocol EPI-0013 [5.1]. The LC/MS/MS method for the analytes exhibited a quadratic response and had target LLOQ and ULOQ values of 1.00 ppb (2.50 ppb for M556) and 500 ppb for the analytes.

The testing facility was 3M Company (Corporate Occupational Medicine, Medical Department, 220-3W-05, St. Paul, MN 55144), and the Study Director was Jeffrey H. Mandel, M.D. The Study Contacts at 3M Company were Jean Burris, M.P.H, R.N., and James Lundberg, Ph.D. The following is a list of NWB supervisory personnel involved in the completion of this work: Connie O. Sakashita, B.S. (NWB Project Manager); Tonya Felix, M.A. (NWB Acting Project Manager); Patrick Bennett, M.S., M.B.A. (NWB Laboratory Director); Rodger L. Foltz, Ph.D. (NWB Technical Director). Laboratory personnel involved in the completion of this study included Suzanne Newman, B.S. (NWB Research Scientist), Toni Peacock, B.S. (NWB Assistant Scientist) and Emily Yardimci, B.S. (NWB Associate Research Scientist).

NWB SOPs and guidelines were used in the conduct of this study and were available to study personnel in electronic and hard copy formats.

Date Study Initiated: 15-Nov-2000 Date Analyses Completed: 11-May-2001

The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all studies within the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good

Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424). Any changes to or deviations from the original protocol (Analytical Plan) were documented through approved protocol amendments or deviation memos and are retained within the raw data.

2. METHODOLOGY

The assay used for this study is reported in Northwest Bioanalytical reports NWBR00-108 [5.2] and NWBR00-122 [5.3]. Chinese human plasma was used for the calibration curves and quality control samples. Serum sample results obtained using plasma curves are discussed in NWBR00-122.

Samples for this study were received at NWB on the following dates:

Receipt Date	*Number of Samples Received*	*Storage Condition (except during analysis)*
22-Nov-2000	100	-20 °C
21-Feb-2001	108	-20 °C
06-Mar-2001	330	-20 °C
15-Mar-2001	16	-20 °C
05-Apr-2001	125	-20 °C

Reference Material

Analyte	*Lot Number*	*Purity*	*Expiration Date*	*Source*	*Storage Conditions*
PFOS (FC-95)	193	100%	31-Dec-2010	3M	Room temperature*
PFHS	SE-036	100%	01-Jan-2010	3M	-20 °C
PFOA (FC-143)	245	100%	31-Dec-2010	3M	Room temperature*
PFOSAA (FC-129)	617	53.8%	31-Dec-2010	3M	Room temperature
PFOSA	214	100%	31-Dec-2010	3M	Room temperature
M556	NB113047-8D	99.89%	31-Dec-2010	3M	Room temperature
M570	118506-26	99.75%	31-Dec-2010	3M	Room temperature
THPFOS	59909	90.0%	31-Dec-2010	3M	Room temperature

*Stored dry

Eight or more calibration standards were prepared on the day of each run by adding 100 µL of blank human plasma and 400 µL of 50 mM ammonium acetate in water (unadjusted pH ~6.9) to 13 x 100 mm polypropylene tubes. After a brief vortex mixing, 10.0 µL of the appropriate spiking solution was added.

The absolute purity of PFOS, PFOSA, PFOA, and PFHS reference material was not available prior to the conduct of this study. Therefore, the reference material purity was assumed to be 100%. 3M contracted with Centre Analytical Laboratories, Inc., in State College, Pennsylvania to determine the absolute concentration of PFOS, PFHS, and PFOA in NWB stock solutions used to prepare the analytical standards and controls used for this studies. All arrangements for purity determinations and the transfer of NWB solutions to Centre Analytical Laboratories, Inc., were performed by 3M. Based on Centre Analytical Laboratories' results, the concentrations of plasma calibrator and quality control samples were corrected using the following factors:

Analyte	*Correction Factor*
PFOS	0.836
PFHS	0.909
PFOA	0.855

The final calibration standard concentrations in human plasma for each analyte can be found in Tables 8 – 14. *NOTE:* Due to the use of different matrix pools with different persistent levels, there are several standard concentrations in these tables. However, only eight (for M556) or nine (all other analytes) standards are used for a single curve. The target calibration curve range is 1.00 ppb (2.50 ppb M556) to 500 ppb prior to correction for purity.

In addition, blank plasma samples, both with and without internal standard (designated as QC0s and Blanks, respectively), were assayed in each analytical run.

Analytical QCs were prepared in human plasma on October 27, 2000, March 8, 2001, April 5, 2001 and May 2, 2001 and frozen in a -20 °C freezer. For each run, analytical QC levels were assayed in duplicate. In addition, for each dilution level, dilution QCs

were run in triplicate in any analytical run that contained diluted subject samples. The QC concentrations are shown below:

Analyte	QC Concentrations* All concentrations are expressed as ppb.			
	Low	Medium	High	Dilution DF=5
PFOS	5.75, 6.40	126	331, 332	348
PFHS	4.48	156, 157	417, 418	
PFOA	4.32, 4.81	145	385	
PFOSAA	4.30, 4.60	150, 151	400, 401	
PFOSA	4.00	150	400	
M556	4.00	150	400	
M570	4.00	150	400	

* The target QC concentrations are 4.00 ppb, 150 ppb and 400 ppb prior to correction for purity. Each analyte has different QC concentrations based upon the persistent levels of the analyte in the human plasma used.

The internal standard (THPFOS) was added to all plasma and serum samples (except Blanks) for a final concentration of approximately 200 ppb.

The analytical method consisted of a liquid:liquid extraction procedure followed by evaporation and reconstitution of the extract residue with 20 mM ammonium acetate in water: 20 mM ammonium acetate in methanol (30:70, v/v). The samples were analyzed by liquid chromatography/tandem mass spectrometry using an API 3000. The instrument was operated in the multiple reaction monitoring (MRM) mode under optimized conditions for PFOS, PFHS, PFOA, PFOSAA, PFOSA, M556 and M570 detection of the negative ions formed by TurboIonSpray™ ionization.

3. SAMPLE ANALYSIS

PFOS, PFHS, PFOA, PFOSAA, PFOSA, M556, M570, and THPFOS chromatographic peaks were integrated using the MacQuan software (version 1.6) with a smooth factor of one. Quantitation was based upon quadratic regression analysis of weighted ($1/x^2$) calibration curves using area ratio vs. concentration utilizing Watson® DMLIMS software (version 6.1.1.04). The samples were injected in a systematic order.

For sample 11689, the measured concentration of PFOS was corrected for the persistent level of analyte present in the dilution matrix. The concentration is reported as a whole number and has not been rounded to 3 significant figures.

3.1. Acceptance Criteria

For an analytical run to be accepted, it must have met the acceptance criteria listed below which are consistent with regulatory and industry recommendations.

Calibration Curve

Each run will include calibration standards in duplicate at six or more concentrations covering the lower to upper limit of quantitation. For all analytes except PFOSA and PFOSAA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 15% (± 20% for LLOQ) of their individual target concentrations. For PFOSA and PFOSAA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 20% (± 25% for LLOQ) of their individual target concentrations. A calibration standard will be considered an outlier if it is greater than two times the acceptance criteria for that standard.

Lower Limit of Quantitation

The back-calculated concentrations of at least one of the duplicate lowest points in the calibration curve must be within ± 25% of the target concentration for PFOSA and PFOSAA to qualify as the LLOQ and within ± 20% of the target concentration to qualify as the LLOQ for all other analytes. If this criterion is not met, the next level is subjected to the same test and the LLOQ raised accordingly.

Quality Control Samples

Each analytical run will include low, medium and high QC samples in duplicate. The measured concentrations of at least two-thirds of all analytical QCs must be within ± 20% of their target concentrations (± 25% for PFOSA and PFOSAA), and no two QCs at the same concentration can be outside the limit. If study samples require

dilution, a dilution QC will be analyzed in triplicate for each dilution level (except for PFOSA and PFOSAA which should not be diluted with control matrix). At least two dilution QCs at each level must be within ± 20% of its target concentrations in order to accept diluted study samples at that level. The dilution QC acceptance is independent of the undiluted analytical QC acceptance.

4. RESULTS AND DISCUSSION

Serum sample results obtained using plasma curves for PFHS, PFOA, PFOSAA, PFOSA, M556, and M570 did not meet NWB SOP requirements for validation. Results may vary on average up to 26% from results obtained using human serum calibration curves at some concentration levels for PFHS, PFOA, PFOSAA, M556, and M570. For PFOSA, results may vary on average up to 43% from results obtained using human serum calibration curves. These data are reported in Assay Revalidation Addendum Report NWBR00-122 [5.3]. However, in order to obtain the lower limit of quantitation of less than 5 ppb required by the sponsor, it was necessary to use plasma calibration curves.

Each accepted run met the acceptance criteria set for the calibration curve points, for the lower limit of quantitation (LLOQ) and for the analytical quality control (QC) samples.

Run No.	Analyte	Regression Status	Extraction Date	Assay Date	LLOQ	ULOQ	Comment
1	PFOS	Accepted	27-Nov-2000	27-Nov-2000	3.94	414	
1	PFOSA	Accepted	27-Nov-2000	27-Nov-2000	1.00	500	
1	PFOSAA	Accepted	27-Nov-2000	27-Nov-2000	1.60	501	
1	PFOA	Accepted	27-Nov-2000	27-Nov-2000	1.92	481	
1	PFHS	Accepted	27-Nov-2000	27-Nov-2000	1.36	523	
1	M556	Accepted	27-Nov-2000	27-Nov-2000	2.50	500	
1	M570	Accepted	27-Nov-2000	27-Nov-2000	1.00	500	
2	PFOS	Accepted	27-Nov-2000	28-Nov-2000	3.94	414	
2	PFOSA	Accepted	27-Nov-2000	28-Nov-2000	1.00	500	
2	PFOSAA	Accepted	27-Nov-2000	28-Nov-2000	1.60	501	
2	PFOA	Accepted	27-Nov-2000	28-Nov-2000	1.92	481	
2	PFHS	Accepted	27-Nov-2000	28-Nov-2000	1.36	523	

Run No.	Analyte	Regression Status	Extraction Date	Assay Date	LLOQ	ULOQ	Comment
2	M556	Accepted	27-Nov-2000	28-Nov-2000	2.50	500	
2	M570	Accepted	27-Nov-2000	28-Nov-2000	1.00	500	
3	PFOS	-	-	-			Run aborted due to power outage (repeated in run 6)
3	PFOSA	-	-	-			Run aborted due to power outage (repeated in run 6)
3	PFOSAA	-	-	-			Run aborted due to power outage (repeated in run 6)
3	PFOA	-	-	-			Run aborted due to power outage (repeated in run 6)
3	PFHS	-	-	-			Run aborted due to power outage (repeated in run 6)
3	M556	-	-	-			Run aborted due to power outage (repeated in run 6)
3	M570	-	-	-			Run aborted due to power outage (repeated in run 6)
4	PFOS	Accepted	21-Mar-2001	23-Mar-2001	3.94	414	
4	PFOSA	Accepted	21-Mar-2001	23-Mar-2001	1.00	500	
4	PFOSAA	Accepted	21-Mar-2001	23-Mar-2001	1.60	501	
4	PFOA	Accepted	21-Mar-2001	23-Mar-2001	1.92	481	
4	PFHS	Accepted	21-Mar-2001	23-Mar-2001	1.36	523	
4	M556	Rejected	21-Mar-2001	23-Mar-2001	2.50	500	QCs failed
4	M570	Rejected	21-Mar-2001	23-Mar-2001	1.00	500	QCs failed
5	PFOS	Accepted	22-Mar-2001	24-Mar-2001	3.94	414	
5	PFOSA	Accepted	22-Mar-2001	24-Mar-2001	1.00	500	
5	PFOSAA	Accepted	22-Mar-2001	24-Mar-2001	1.60	501	
5	PFOA	Accepted	22-Mar-2001	24-Mar-2001	1.92	481	
5	PFHS	Accepted	22-Mar-2001	24-Mar-2001	1.36	523	
5	M556	Accepted	22-Mar-2001	24-Mar-2001	2.50	500	
5	M570	Accepted	22-Mar-2001	24-Mar-2001	1.00	500	
6	PFOS	Accepted	26-Mar-2001	26-Mar-2001	4.27	414	
6	PFOSA	Accepted	26-Mar-2001	26-Mar-2001	1.40	500	
6	PFOSAA	Accepted	26-Mar-2001	26-Mar-2001	2.80	502	
6	PFOA	Accepted	26-Mar-2001	26-Mar-2001	2.11	482	
6	PFHS	Accepted	26-Mar-2001	26-Mar-2001	2.09	523	

Run No.	Analyte	Regression Status	Extraction Date	Assay Date	LLOQ	ULOQ	Comment
6	M556	Accepted	26-Mar-2001	26-Mar-2001	3.20	501	
6	M570	Accepted	26-Mar-2001	26-Mar-2001	1.80	501	
7	PFOS	Accepted	26-Mar-2001	26-Mar-2001	4.27	414	
7	PFOSA	Accepted	26-Mar-2001	26-Mar-2001	1.40	500	
7	PFOSAA	Accepted	26-Mar-2001	26-Mar-2001	2.80	502	
7	PFOA	Accepted	26-Mar-2001	26-Mar-2001	2.11	482	
7	PFHS	Accepted	26-Mar-2001	26-Mar-2001	2.09	523	
7	M556	Accepted	26-Mar-2001	26-Mar-2001	3.20	501	
7	M570	Accepted	26-Mar-2001	26-Mar-2001	1.80	501	
8	PFOS	Accepted	28-Mar-2001	28-Mar-2001	4.27	414	
8	PFOSA	Accepted	28-Mar-2001	28-Mar-2001	1.40	500	
8	PFOSAA	Accepted	28-Mar-2001	28-Mar-2001	2.80	502	
8	PFOA	Accepted	28-Mar-2001	28-Mar-2001	2.11	482	
8	PFHS	Accepted	28-Mar-2001	28-Mar-2001	2.09	523	
8	M556	Accepted	28-Mar-2001	28-Mar-2001	3.20	501	
8	M570	Accepted	28-Mar-2001	28-Mar-2001	1.80	501	
9	PFOS	Accepted	28-Mar-2001	28-Mar-2001	4.27	414	
9	PFOSA	Accepted	28-Mar-2001	28-Mar-2001	1.40	500	
9	PFOSAA	Accepted	28-Mar-2001	28-Mar-2001	2.80	502	
9	PFOA	Accepted	28-Mar-2001	28-Mar-2001	2.11	482	
9	PFHS	Accepted	28-Mar-2001	28-Mar-2001	2.09	523	
9	M556	Rejected	28-Mar-2001	28-Mar-2001	3.20	501	Low QCs failed
9	M570	Rejected	28-Mar-2001	28-Mar-2001	1.80	501	Curve and QC fail
10	PFOS	Accepted	30-Mar-2001	30-Mar-2001	4.27	414	
10	PFOSA	Accepted	30-Mar-2001	30-Mar-2001	1.40	500	
10	PFOSAA	Accepted	30-Mar-2001	30-Mar-2001	2.80	502	
10	PFOA	Accepted	30-Mar-2001	30-Mar-2001	2.11	482	
10	PFHS	Accepted	30-Mar-2001	30-Mar-2001	2.09	523	
10	M556	Accepted	30-Mar-2001	30-Mar-2001	3.20	501	
10	M570	Accepted	30-Mar-2001	30-Mar-2001	1.80	501	
11	PFOS	Accepted	30-Mar-2001	30-Mar-2001	4.27	414	
11	PFOSA	Accepted	30-Mar-2001	30-Mar-2001	1.40	500	
11	PFOSAA	Rejected	30-Mar-2001	30-Mar-2001	2.80	502	Curve fails

Run No.	Analyte	Regression Status	Extraction Date	Assay Date	LLOQ	ULOQ	Comment
11	PFOA	Rejected	30-Mar-2001	30-Mar-2001	2.11	482	Curve fails
11	PFHS	Accepted	30-Mar-2001	30-Mar-2001	2.09	523	
11	M556	Accepted	30-Mar-2001	30-Mar-2001	3.20	501	
11	M570	Rejected	30-Mar-2001	30-Mar-2001	1.80	501	Curve fails
12	M556	Accepted	28-Mar-2001	01-Apr-2001	3.20	501	Run 12 was a reinjection of run 9 for M556 and M570 only
12	M570	Accepted	28-Mar-2001	01-Apr-2001	1.80	501	Run 12 was a reinjection of run 9 for M556 and M570 only
13	PFOS	Accepted	02-Apr-2001	02-Apr-2001	4.27	414	
13	PFOSA	Accepted	02-Apr-2001	02-Apr-2001	1.40	500	
13	PFOSAA	Accepted	02-Apr-2001	02-Apr-2001	2.80	502	
13	PFOA	Accepted	02-Apr-2001	02-Apr-2001	2.11	482	
13	PFHS	Accepted	02-Apr-2001	02-Apr-2001	2.09	523	
13	M556	Rejected	02-Apr-2001	02-Apr-2001	3.20	501	Low QCs failed
13	M570	Accepted	02-Apr-2001	02-Apr-2001	1.80	501	
14	PFOS	Accepted	04-Apr-2001	07-Apr-2001	4.27	414	
14	PFOSA	Accepted	04-Apr-2001	07-Apr-2001	1.40	500	
14	PFOSAA	Accepted	04-Apr-2001	07-Apr-2001	2.80	502	
14	PFOA	Accepted	04-Apr-2001	07-Apr-2001	2.11	482	
14	PFHS	Accepted	04-Apr-2001	07-Apr-2001	2.09	523	
14	M556	Accepted	04-Apr-2001	07-Apr-2001	3.20	501	
14	M570	Accepted	04-Apr-2001	07-Apr-2001	1.80	501	
15	PFOS	Accepted	06-Apr-2001	09-Apr-2001	4.27	414	
15	PFOSA	Accepted	06-Apr-2001	09-Apr-2001	1.40	500	
15	PFOSAA	Accepted	06-Apr-2001	09-Apr-2001	2.80	502	
15	PFOA	Accepted	06-Apr-2001	09-Apr-2001	2.11	482	
15	PFHS	Accepted	06-Apr-2001	09-Apr-2001	2.09	523	
15	M556	Accepted	06-Apr-2001	09-Apr-2001	3.20	501	
15	M570	Accepted	06-Apr-2001	09-Apr-2001	1.80	501	
16	PFOS	Accepted	09-Apr-2001	11-Apr-2001	4.27	414	
16	PFOSA	Accepted	09-Apr-2001	11-Apr-2001	1.40	500	
16	PFOSAA	Rejected	09-Apr-2001	11-Apr-2001	2.80	502	Curve fails

Run No.	Analyte	Regression Status	Extraction Date	Assay Date	LLOQ	ULOQ	Comment
16	PFOA	Rejected	09-Apr-2001	11-Apr-2001	2.11	482	Curve fails
16	PFHS	Accepted	09-Apr-2001	11-Apr-2001	2.09	523	
16	M556	Rejected	09-Apr-2001	11-Apr-2001	3.20	501	Curve fails
16	M570	Rejected	09-Apr-2001	11-Apr-2001	1.80	501	Curve fails
17	PFOS	Accepted	10-Apr-2001	12-Apr-2001	4.27	414	
17	PFOSA	Accepted	10-Apr-2001	12-Apr-2001	1.40	500	
17	PFOSAA	Accepted	10-Apr-2001	12-Apr-2001	2.80	502	
17	PFOA	Accepted	10-Apr-2001	12-Apr-2001	2.11	482	
17	PFHS	Accepted	10-Apr-2001	12-Apr-2001	2.09	523	
17	M556	Accepted	10-Apr-2001	12-Apr-2001	3.20	501	
17	M570	Accepted	10-Apr-2001	12-Apr-2001	1.80	501	
18	PFOS	Accepted	19-Apr-2001	23-Apr-2001	4.27	414	
18	PFOSA	Accepted	19-Apr-2001	23-Apr-2001	1.40	500	
18	PFOSAA	Accepted	19-Apr-2001	23-Apr-2001	2.80	502	
18	PFOA	Accepted	19-Apr-2001	23-Apr-2001	2.11	482	
18	PFHS	Accepted	19-Apr-2001	23-Apr-2001	2.09	523	
18	M556	Accepted	19-Apr-2001	23-Apr-2001	3.20	501	
18	M570	Rejected	19-Apr-2001	23-Apr-2001	1.80	501	Curve and Low QC failed
19	PFOS	Rejected	09-Apr-2001	14-Apr-2001	4.02	414	Reinjection of run 16: Analyte results not needed
19	PFOSA	Rejected	09-Apr-2001	14-Apr-2001	1.30	500	Reinjection of run 16: Analyte results not needed
19	PFOSAA	Accepted	09-Apr-2001	14-Apr-2001	2.80	502	Reinjection of run 16
19	PFOA	Accepted	09-Apr-2001	14-Apr-2001	2.11	482	Reinjection of run 16
19	PFHS	Rejected	09-Apr-2001	14-Apr-2001	2.09	523	Reinjection of run 16: Analyte results not needed
19	M556	Accepted	09-Apr-2001	14-Apr-2001	3.20	501	Reinjection of run 16
19	M570	Accepted	09-Apr-2001	14-Apr-2001	1.80	501	Reinjection of run 16
20	PFOS	Accepted	06-Apr-2001	13-Apr-2001	4.27	414	Pre-study assay evaluation (new Chinese plasma pool and QCs)
20	PFOSA	Accepted	06-Apr-2001	13-Apr-2001	1.40	500	Pre-study assay evaluation (new Chinese plasma pool and QCs)

Run No.	Analyte	Regression Status	Extraction Date	Assay Date	LLOQ	ULOQ	Comment
20	PFOSAA	Accepted	06-Apr-2001	13-Apr-2001	2.80	502	Pre-study assay evaluation (new Chinese plasma pool and QCs)
20	PFOA	Accepted	06-Apr-2001	13-Apr-2001	2.10	482	Pre-study assay evaluation (new Chinese plasma pool and QCs)
20	PFHS	Accepted	06-Apr-2001	13-Apr-2001	2.09	523	Pre-study assay evaluation (new Chinese plasma pool and QCs)
20	M556	Rejected	06-Apr-2001	13-Apr-2001	1.60	501	Pre-study assay evaluation (new Chinese plasma pool and QCs); Low QC fail.
20	M570	Accepted	06-Apr-2001	13-Apr-2001	1.90	501	Pre-study assay evaluation (new Chinese plasma pool and QCs)
21	PFOSAA	Accepted	17-Apr-2001	22-Apr-2001	2.80	502	Run was analyzed for PFOSAA, PFOA and M570 only
21	PFOA	Accepted	17-Apr-2001	22-Apr-2001	2.11	482	Run was analyzed for PFOSAA, PFOA and M570 only
21	M570	Accepted	17-Apr-2001	22-Apr-2001	1.80	501	Run was analyzed for PFOSAA, PFOA and M570 only
22	M570	Accepted	19-Apr-2001	24-Apr-2001	1.80	501	Reinjection of run 18 for M570 only
23	PFOS	Accepted	02-May-2001	05-May-2001	3.86	414	Pre-study assay evaluation of new low QC
23	PFOSA	Accepted	02-May-2001	05-May-2001	1.50	501	Pre-study assay evaluation of new low QC
23	PFOSAA	Accepted	02-May-2001	05-May-2001	2.40	501	Pre-study assay evaluation of new low QC
23	PFOA	Accepted	02-May-2001	05-May-2001	2.31	482	Pre-study assay evaluation of new low QC
23	PFHS	Accepted	02-May-2001	05-May-2001	2.50	523	Pre-study assay evaluation of new low QC
23	M556	Accepted	02-May-2001	05-May-2001	3.80	501	Pre-study assay evaluation of new low QC
23	M570	Accepted	02-May-2001	05-May-2001	2.30	501	Pre-study assay evaluation of new low QC
24	PFOSAA	Rejected	10-May-2001	10-May-2001	1.50	501	Sample preparation error
24	PFOA	Rejected	10-May-2001	10-May-2001	25.4	482	Sample preparation error

Run No.	Analyte	Regression Status	Extraction Date	Assay Date	LLOQ	ULOQ	Comment
24	PFHS	Rejected	10-May-2001	10-May-2001	2.50	523	Sample preparation error
24	M570	Rejected	10-May-2001	10-May-2001	2.30	501	Sample preparation error
25	PFOSAA	Accepted	11-May-2001	11-May-2001	1.30	500	
25	PFOA	Accepted	11-May-2001	11-May-2001	1.44	481	
25	PFHS	Rejected	11-May-2001	11-May-2001	1.36	522	Analyte results not needed
25	M570	Accepted	11-May-2001	11-May-2001	1.00	500	

In run 4 for PFOSA and PFOSAA, one of the medium QC samples gave extremely high results. These results appear to skew the overall statistics for these analytes. Without this "outlier", the overall %CV was 9.2% for PFOSA and 12.3% for PFOSAA. The overall %Bias was 2.0% for PFOSA and 5.3% for PFOSAA.

In run 8 for PFOA, one of the low QC samples gave an extremely low result of 0.161 ppb. This result appears to skew the overall statistics for the analyte. Without this "outlier", the overall %CV was 14.1% and the %Bias was –10.0%/

Any known circumstances that may have affected the quality or integrity of the data are included in this report.

5. REFERENCES

5.1. 3M Company Protocol EPI-0013. "Identification of Fluorochemicals in Sera of American Red Cross Blood Donors in the United States." September 29, 2000.

5.2. C. Sakashita. "Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS." Assay Revalidation Report. NWB Study NWBS00-040. NWB Report NWBR00-108. January 24, 2001.

5.3. C. Sakashita. "Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS." Assay Revalidation Addendum Report. NWB Study NWBS00-040. NWB Report NWBR00-122. November 20, 2001.

6. DATA RETENTION

The raw data and final report for this study will be stored in the NWB Archives, 1121 East 3900 South, Salt Lake City, UT 84124 per regulations and contract agreement. After submission of the final report to the Sponsor, remaining study samples will be stored under required conditions until confirmation of Sample Disposition/Return Authorization is received from the Sponsor. 3M Company will be notified concerning final disposition of records at completion of contract obligations.

Table 1. Calibration Curve Summary for PFOS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
27-Nov-2000	1	-0.000031	0.052622	0.012630	0.9958	3.94	414
28-Nov-2000	2	-0.000027	0.049661	0.017690	0.9926	3.94	414
23-Mar-2001	4	0.000002	0.039134	0.020796	0.9869	3.94	414
24-Mar-2001	5	-0.000002	0.009354	0.001128	0.9985	3.94	414
26-Mar-2001	6	-0.000001	0.010776	-0.003903	0.9867	4.27	414
26-Mar-2001	7	0.000003	0.035721	0.024340	0.9943	4.27	414
28-Mar-2001	8	0.000000	0.037629	0.025011	0.9931	4.27	414
28-Mar-2001	9	0.000000	0.010093	0.007090	0.9895	4.27	414
30-Mar-2001	10	0.000000	0.011733	0.002809	0.9947	4.27	414
30-Mar-2001	11	0.000001	0.047343	0.024473	0.9733	4.27	414
02-Apr-2001	13	-0.000003	0.045861	0.035036	0.9973	4.27	414
07-Apr-2001	14	-0.000010	0.039827	0.014677	0.9925	4.27	414
09-Apr-2001	15	0.000004	0.045781	0.046887	0.9919	4.27	414
11-Apr-2001	16	-0.000006	0.058787	-0.001384	0.9942	4.27	414
12-Apr-2001	17	-0.000009	0.055527	0.020155	0.9976	4.27	414
23-Apr-2001	18	-0.000002	0.017703	0.000222	0.9935	4.27	414
Mean		-0.000005	0.035472	0.015479	0.9920		
S.D.		0.000010	0.017605	0.014172	0.0060		
%CV		-200.0	49.6	91.6	0.6		
n		16	16	16	16		

A, B, and C are coefficients used to define the calibration curve.

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Table 2. Calibration Curve Summary for PFHS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
27-Nov-2000	1	-0.000013	0.030678	0.025452	0.9949	1.36	523
28-Nov-2000	2	-0.000013	0.031308	0.026252	0.9950	1.36	523
23-Mar-2001	4	-0.000002	0.021861	0.020425	0.9959	1.36	523
24-Mar-2001	5	-0.000002	0.007620	0.006999	0.9966	1.36	523
26-Mar-2001	6	-0.000002	0.008120	-0.002637	0.9953	2.09	523
26-Mar-2001	7	-0.000004	0.023053	0.006759	0.9902	2.09	523
28-Mar-2001	8	-0.000003	0.025470	0.002418	0.9892	2.09	523
28-Mar-2001	9	-0.000002	0.008701	0.002381	0.9972	2.09	523
30-Mar-2001	10	-0.000001	0.008569	0.003686	0.9836	2.09	523
30-Mar-2001	11	0.000000	0.028707	0.027904	0.9813	2.09	523
02-Apr-2001	13	-0.000004	0.021870	0.016367	0.9858	2.09	523
07-Apr-2001	14	-0.000006	0.021837	0.012060	0.9915	2.09	523
09-Apr-2001	15	-0.000002	0.022158	0.030931	0.9901	2.09	523
11-Apr-2001	16	-0.000007	0.029426	0.013375	0.9881	2.09	523
12-Apr-2001	17	-0.000007	0.026000	0.009029	0.9970	2.09	523
23-Apr-2001	18	-0.000004	0.011306	0.008022	0.9875	2.09	523
Mean		-0.000004	0.020418	0.013089	0.9912		
S.D.		0.000004	0.008650	0.010370	0.0050		
%CV		-100.0	42.4	79.2	0.5		
n		16	16	16	16		

A, B, and C are coefficients used to define the calibration curve.

Table 3. Calibration Curve Summary for PFOA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
27-Nov-2000	1	-0.000001	0.015479	0.005741	0.9933	1.92	481
28-Nov-2000	2	-0.000002	0.016334	0.015052	0.9877	1.92	481
23-Mar-2001	4	0.000001	0.008393	0.007706	0.9842	1.92	481
24-Mar-2001	5	-0.000001	0.007290	0.002187	0.9979	1.92	481
26-Mar-2001	6	0.000000	0.007392	-0.000487	0.9898	2.11	482
26-Mar-2001	7	0.000003	0.008327	0.004956	0.9865	2.11	482
28-Mar-2001	8	0.000002	0.008675	0.015973	0.9825	2.11	482
28-Mar-2001	9	0.000000	0.007316	0.005861	0.9940	2.11	482
30-Mar-2001	10	0.000000	0.007744	0.003135	0.9964	2.11	482
02-Apr-2001	13	-0.000001	0.008171	0.003088	0.9901	2.11	482
07-Apr-2001	14	-0.000003	0.009779	0.000405	0.9926	2.11	482
09-Apr-2001	15	0.000002	0.007753	0.019137	0.9872	2.11	482
12-Apr-2001	17	0.000000	0.007833	0.004324	0.9945	2.11	482
14-Apr-2001	19	-0.000001	0.007768	0.001433	0.9954	2.11	482
22-Apr-2001	21	0.000000	0.015981	-0.001298	0.9878	2.11	482
23-Apr-2001	18	-0.000002	0.016857	0.000933	0.9905	2.11	482
11-May-2001	25	-0.000001	0.012990	-0.002765	0.9962	1.44	481
Mean		0.000000	0.010240	0.005022	0.9910		
S.D.		0.000002	0.003645	0.006254	0.0045		
%CV		NCL	35.6	124.5	0.5		
n		17	17	17	17		

A, B, and C are coefficients used to define the calibration curve.
NCL = not calculable

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Table 4. Calibration Curve Summary for PFOSAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
27-Nov-2000	1	0.000000	0.013246	0.007247	0.9804	1.60	501
28-Nov-2000	2	0.000005	0.012198	0.010441	0.9797	1.60	501
23-Mar-2001	4	0.000001	0.007894	0.004964	0.9685	1.60	501
24-Mar-2001	5	0.000001	0.006730	0.010546	0.9935	1.60	501
26-Mar-2001	6	0.000002	0.008322	-0.001161	0.9836	2.80	502
26-Mar-2001	7	0.000001	0.007111	-0.001354	0.9760	2.80	502
28-Mar-2001	8	0.000001	0.005138	-0.004421	0.9798	2.80	502
28-Mar-2001	9	0.000002	0.006742	0.011207	0.9666	2.80	502
30-Mar-2001	10	0.000003	0.007717	0.008093	0.9848	2.80	502
02-Apr-2001	13	0.000008	0.024032	0.015403	0.9884	2.80	502
07-Apr-2001	14	0.000001	0.033688	0.011384	0.9867	2.80	502
09-Apr-2001	15	0.000014	0.029651	0.017800	0.9846	2.80	502
12-Apr-2001	17	0.000006	0.025674	0.007502	0.9944	2.80	502
14-Apr-2001	19	0.000012	0.026710	-0.009422	0.9813	2.80	502
22-Apr-2001	21	0.000004	0.007075	-0.004642	0.9735	2.80	502
23-Apr-2001	18	0.000004	0.007908	0.000878	0.9744	2.80	502
11-May-2001	25	0.000002	0.012005	0.000362	0.9937	1.30	500
Mean		0.000004	0.014226	0.004990	0.9818		
S.D.		0.000004	0.009564	0.007661	0.0083		
%CV		100.0	67.2	153.5	0.8		
n		17	17	17	17		

A, B, and C are coefficients used to define the calibration curve.

Table 5. Calibration Curve Summary for PFOSA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
27-Nov-2000	1	-0.000041	0.096146	0.018081	0.9920	1.00	500
28-Nov-2000	2	-0.000025	0.087183	0.015131	0.9931	1.00	500
23-Mar-2001	4	0.000002	0.034802	0.013699	0.9847	1.00	500
24-Mar-2001	5	-0.000002	0.018868	0.003711	0.9985	1.00	500
26-Mar-2001	6	-0.000001	0.023192	-0.012070	0.9931	1.40	500
26-Mar-2001	7	-0.000002	0.034174	0.000108	0.9956	1.40	500
28-Mar-2001	8	0.000001	0.032420	0.015115	0.9965	1.40	500
28-Mar-2001	9	0.000000	0.022395	0.000709	0.9848	1.40	500
30-Mar-2001	10	-0.000003	0.023907	0.000482	0.9934	1.40	500
30-Mar-2001	11	0.000018	0.108434	0.040511	0.9882	1.40	500
02-Apr-2001	13	-0.000008	0.108295	0.054874	0.9885	1.40	500
07-Apr-2001	14	-0.000011	0.097511	0.020730	0.9819	1.40	500
09-Apr-2001	15	-0.000006	0.119455	0.031195	0.9923	1.40	500
11-Apr-2001	16	0.000034	0.124021	-0.030446	0.9870	1.40	500
12-Apr-2001	17	-0.000010	0.129002	0.003170	0.9944	1.40	500
23-Apr-2001	18	0.000007	0.040292	0.010493	0.9936	1.40	500
Mean		-0.000003	0.068756	0.011593	0.9911		
S.D.		0.000016	0.042828	0.020139	0.0047		
%CV		-533.3	62.3	173.7	0.5		
n		16	16	16	16		

A, B, and C are coefficients used to define the calibration curve.

Table 6. Calibration Curve Summary for M556

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
27-Nov-2000	1	0.000001	0.008245	0.002637	0.9851	2.50	500
28-Nov-2000	2	0.000003	0.007109	0.006480	0.9931	2.50	500
24-Mar-2001	5	0.000001	0.002937	0.003249	0.9943	2.50	500
26-Mar-2001	6	0.000001	0.003973	-0.002024	0.9809	3.20	501
26-Mar-2001	7	0.000001	0.006232	-0.005748	0.9928	3.20	501
28-Mar-2001	8	0.000001	0.004865	-0.004285	0.9923	3.20	501
30-Mar-2001	10	0.000001	0.002628	0.000476	0.9939	3.20	501
30-Mar-2001	11	0.000003	0.004105	0.002505	0.9789	3.20	501
01-Apr-2001	12	0.000001	0.002392	0.001473	0.9825	3.20	501
07-Apr-2001	14	0.000001	0.007457	0.002028	0.9924	3.20	501
09-Apr-2001	15	0.000003	0.006599	0.008776	0.9891	3.20	501
12-Apr-2001	17	0.000002	0.006819	-0.003271	0.9913	3.20	501
14-Apr-2001	19	0.000003	0.006308	-0.001438	0.9886	3.20	501
23-Apr-2001	18	0.000002	0.003128	0.001358	0.9870	3.20	501
Mean		0.000002	0.005200	0.000873	0.9887		
S.D.		0.000001	0.001993	0.004007	0.0051		
%CV		50.0	38.3	459.0	0.5		
n		14	14	14	14		

A, B, and C are coefficients used to define the calibration curve.

Table 7. Calibration Curve Summary for M570

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
27-Nov-2000	1	-0.000003	0.022236	0.026662	0.9910	1.00	500
28-Nov-2000	2	0.000002	0.021764	0.021002	0.9902	1.00	500
24-Mar-2001	5	0.000000	0.003326	0.003631	0.9937	1.00	500
26-Mar-2001	6	0.000002	0.003649	-0.000239	0.9851	1.80	501
26-Mar-2001	7	0.000001	0.017625	0.009909	0.9880	1.80	501
28-Mar-2001	8	0.000002	0.013605	0.006783	0.9911	1.80	501
30-Mar-2001	10	0.000001	0.003548	0.001744	0.9859	1.80	501
01-Apr-2001	12	0.000001	0.003371	0.002595	0.9779	1.80	501
02-Apr-2001	13	0.000007	0.027857	0.035353	0.9890	1.80	501
07-Apr-2001	14	-0.000001	0.034478	0.018645	0.9854	1.80	501
09-Apr-2001	15	0.000011	0.032868	0.046246	0.9863	1.80	501
12-Apr-2001	17	0.000004	0.030324	0.016921	0.9936	1.80	501
14-Apr-2001	19	0.000009	0.030213	0.008691	0.9817	1.80	501
22-Apr-2001	21	0.000001	0.003114	-0.002037	0.9880	1.80	501
24-Apr-2001	22	0.000018	0.038537	0.040536	0.9873	1.80	501
11-May-2001	25	0.000000	0.004063	-0.001174	0.9955	1.00	500
Mean		0.000003	0.018161	0.014704	0.9881		
S.D.		0.000005	0.013225	0.015506	0.0045		
%CV		166.7	72.8	105.5	0.5		
n		16	16	16	16		

A, B, and C are coefficients used to define the calibration curve.

Table 8. Back-Calculated Concentrations of Calibration Standards for PFOS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	3.94	4.27	5.17	5.50	11.3	11.7	23.6	24.0	44.2	44.5	85.4	85.5	209	332	414
27-Nov-2000	1	4.04		5.05		11.1		24.1		48.3		80.9		234	307	432
		3.98		5.02		11.4		*44.7		**18.9		77.2		209	321	417
28-Nov-2000	2	4.05		4.91		10.9		25.4		49.5		84.5		234	299	450
		4.00		5.24		10.6		*42.1		**18.1		74.2		204	319	416
23-Mar-2001	4	4.01		5.70		10.6		26.0		41.2		81.4		232	293	419
		3.42		5.60		11.3		20.3		48.5		89.2		186	380	399
24-Mar-2001	5	3.99		5.05		11.6		23.2		46.8		82.0		209	351	421
		3.94		5.07		11.6		24.2		44.0		81.3		205	314	415
26-Mar-2001	6		4.13		4.95		11.4		22.1		42.0		73.8	191	319	419
			4.46		5.64		13.7		23.7		53.9		81.8	223	368	399
26-Mar-2001	7		4.75		5.64		11.4		25.7		46.6		80.7	214	365	392
			3.85		5.34		11.4		23.8		41.9		86.0	218	304	422
28-Mar-2001	8		4.25		5.23		12.4		24.8		52.8		86.1	203	332	437
			4.54		5.26		11.0		24.3		40.5		80.3	190	340	406
28-Mar-2001	9		5.01		5.55		11.3		26.7		49.0		89.0	207	335	415
			4.07		4.86		10.0		23.0		46.2		76.3	224	314	415
30-Mar-2001	10		4.55		4.89		13.0		24.7		45.4		81.5	199	345	391
			4.54		5.10		10.8		23.8		44.1		86.6	221	342	413

* Sample deactivated as an outlier (not included in summary statistics)
** Sample deactivated due to preparation error

000490

Table 8. Back-Calculated Concentrations of Calibration Standards for PFOS (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	3.94	4.27	5.17	5.50	11.3	11.7	23.6	24.0	44.2	44.5	85.4	85.5	209	332	414
30-Mar-2001	11		5.45		4.97		10.6		20.5		44.9		81.5	204	311	397
			3.63		5.49		11.1		24.2		52.8		106	169	381	428
02-Apr-2001	13		4.16		5.28		11.1		24.8		44.9		85.4	213	351	426
			4.59		5.54		11.5		22.9		46.0		86.2	208	337	374
07-Apr-2001	14		4.24		5.75		12.2		26.9		46.3		88.3	225	373	409
			4.47		4.96		11.2		21.3		42.0		81.9	195	329	380
09-Apr-2001	15		5.00		5.11		10.5		25.9		47.1		90.8	223	359	407
			4.03		5.46		10.5		23.5		45.0		80.4	206	320	394
11-Apr-2001	16		4.66		5.37		11.1		22.8		47.4		72.9	224	328	395
			3.92		5.69		11.6		24.5		47.7		87.9	209	342	417
12-Apr-2001	17		4.45		5.20		11.3		25.4		44.2		86.4	196	328	435
			***2.26		5.38		12.5		23.3		45.9		82.6	207	341	405
23-Apr-2001	18		4.61		5.25		11.2		25.3		44.9		76.0	201	314	377
			4.33		5.25		11.3		22.9		48.1		90.0	230	364	426
Mean		3.93	4.42	5.21	5.30	11.1	11.4	23.9	24.0	46.4	46.2	81.3	84.1	210	335	411
S.D.		0.208	0.411	0.290	0.263	0.407	0.830	2.01	1.57	3.18	3.39	4.48	6.80	15.1	23.8	17.8
%CV		5.3	9.3	5.6	5.0	3.7	7.3	8.4	6.5	6.9	7.3	5.5	8.1	7.2	7.1	4.3
%Bias		-0.3	3.5	0.8	-3.6	-1.8	-2.6	1.3	0.0	5.0	3.8	-4.8	-1.6	0.5	0.9	-0.7
n		8	23	8	24	8	24	6	24	6	24	8	24	32	32	32

*** Sample deactivated due to unacceptable internal standard response

000491

Table 9. Back-Calculated Concentrations of Calibration Standards for PFHS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.36	2.09	2.92	3.65	10.7	11.5	26.5	27.1	52.6	53.2	104	105	262	418	523
27-Nov-2000	1	1.39		2.66		10.8		27.4		54.5		98.9		278	373	508
		1.46		2.54		11.4		*54.5		**26.8		106		271	415	564
28-Nov-2000	2	1.42		2.78		10.7		28.0		57.6		101		274	386	536
		1.42		2.46		10.9		*50.7		**25.0		102		257	421	534
23-Mar-2001	4	1.41		3.13		10.6		28.1		52.5		98.5		283	405	527
		1.31		2.71		10.8		28.5		48.1		104		237	449	512
24-Mar-2001	5	1.33		2.76		11.1		26.5		55.2		99.5		261	432	528
		1.48		2.58		11.3		27.3		53.8		101		255	389	545
26-Mar-2001	6		2.08		3.70		11.0		26.9		50.8		98.2	247	409	525
			2.12		3.64		10.7		28.7		62.9		102	271	441	516
26-Mar-2001	7		2.42		3.23		11.6		27.3		52.6		90.7	242	398	472
			1.94		3.39		12.4		29.4		54.0		108	294	418	594
28-Mar-2001	8		2.20		2.96		12.3		30.9		60.1		94.6	236	437	533
			2.30		3.21		11.3		27.6		52.9		104	243	415	545
28-Mar-2001	9		2.22		3.66		11.6		26.4		52.3		105	255	455	496
			1.99		3.49		12.0		28.8		55.5		96.2	254	412	544
30-Mar-2001	10		2.11		2.83		13.2		31.4		51.1		107	223	465	550
			2.27		3.71		10.3		28.8		58.8		91.3	239	374	565

* Sample deactivated as an outlier (not included in summary statistics)
** Sample deactivated due to preparation error

Table 9. Back-Calculated Concentrations of Calibration Standards for PFHS (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.36	2.09	2.92	3.65	10.7	11.5	26.5	27.1	52.6	53.2	104	105	262	418	523
30-Mar-2001	11		2.62		2.90		12.0		24.6		53.2		99.0	249	406	502
			1.89		3.36		11.1		28.0		66.3		114	230	468	537
02-Apr-2001	13		1.77		2.88		10.9		27.4		52.9		107	255	439	506
			2.68		3.61		11.7		27.2		57.4		108	266	445	485
07-Apr-2001	14		1.97		2.94		11.4		26.7		51.5		98.0	270	431	497
			2.43		3.59		11.9		30.9		54.6		103	268	422	523
09-Apr-2001	15		2.60		3.30		11.0		28.0		56.4		110	264	449	514
			1.75		3.56		10.7		27.2		55.9		99.4	263	411	498
11-Apr-2001	16		2.28		2.97		11.3		26.6		53.8		85.9	251	433	482
			2.23		3.20		12.7		29.3		58.5		106	288	445	514
12-Apr-2001	17		2.12		3.87		10.9		28.5		53.0		103	248	419	552
			***1.93		3.29		12.0		28.3		55.8		98.0	253	425	519
23-Apr-2001	18		2.54		3.15		10.7		27.1		50.2		114	251	427	502
			1.94		3.33		11.0		26.4		63.9		107	282	398	522
Mean		1.40	2.19	2.70	3.32	11.0	11.5	27.6	28.0	53.6	55.6	101	102	258	422	523
S.D.		0.0585	0.266	0.205	0.305	0.288	0.717	0.715	1.60	3.19	4.29	2.59	7.17	17.1	24.2	26.4
%CV		4.2	12.1	7.6	9.2	2.6	6.2	2.6	5.7	6.0	7.7	2.6	7.0	6.6	5.7	5.0
%Bias		2.9	4.8	-7.5	-9.0	2.8	0.0	4.2	3.3	1.9	4.5	-2.9	-2.9	-1.5	1.0	0.0
n		8	23	8	24	8	24	6	24	6	24	8	24	32	32	32

*** Sample deactivated due to unacceptable internal standard response

090493

Table 10. Back-Calculated Concentrations of Calibration Standards for PFOA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.92	3.36	10.6	25.0	49.0	97.1	241	385	481
27-Nov-2000	1	1.88	3.04	9.65	25.3	51.6	87.9	252	356	457
		2.13	3.14	11.5	*53.4	**22.5	103	261	387	503
28-Nov-2000	2	1.77	2.97	10.2	28.0	52.8	93.6	244	390	478
		2.37	2.78	10.9	*46.6	**19.9	94.2	247	372	483
23-Mar-2001	4	1.54	2.87	12.5	27.4	50.2	90.7	256	383	492
		2.40	3.40	10.7	20.7	48.5	99.6	222	400	465
24-Mar-2001	5	1.89	3.05	10.4	24.4	51.5	92.7	243	399	477
		2.05	3.38	10.6	26.2	51.4	94.9	236	380	480
Mean		2.00	3.08	10.8	25.3	51.0	94.6	245	384	480
S.D.		0.295	0.222	0.869	2.63	1.48	4.79	14.1	14.4	12.9
%CV		14.8	7.2	8.0	10.4	2.9	5.1	5.8	3.8	2.7
%Bias		4.2	-8.3	1.9	1.2	4.1	-2.6	1.7	-0.3	-0.2
n		8	8	8	6	6	8	34	10	10

* Sample deactivated as an outlier (not included in summary statistics)
** Sample deactivated due to sample preparation error

000494

Table 10. Back-Calculated Concentrations of Calibration Standards for PFOA (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	2.11	3.56	10.8	25.2	49.2	97.3	241	386	482
26-Mar-2001	6	2.49	3.57	9.76	24.0	49.0	92.5	244	355	501
		1.87	3.25	10.3	26.8	59.1	93.8	255	396	466
26-Mar-2001	7	*4.06	3.37	11.5	23.1	46.7	87.7	234	387	440
		2.45	2.72	10.2	29.1	52.2	103	275	385	501
28-Mar-2001	8	2.06	*1.68	7.94	27.8	55.8	90.6	249	372	477
		2.44	2.73	13.2	26.0	46.7	93.2	245	400	480
28-Mar-2001	9	2.40	3.26	11.4	25.5	48.8	99.5	242	392	465
		2.09	3.07	10.0	23.9	55.2	96.2	251	392	470
30-Mar-2001	10	2.03	3.10	11.4	25.7	52.4	95.3	236	389	474
		2.30	3.69	10.1	24.8	49.1	94.9	256	386	479
02-Apr-2001	13	1.83	3.48	10.2	25.1	49.5	95.0	250	419	489
		*3.48	4.55	10.5	23.8	50.1	94.9	238	390	437
07-Apr-2001	14	2.06	3.54	10.4	25.1	45.5	85.6	240	390	459
		*4.56	*5.97	13.2	23.9	52.7	95.5	238	408	498
09-Apr-2001	15	1.84	*0.842	7.81	25.3	45.7	98.2	228	389	431
		2.30	4.05	10.9	27.1	53.9	101	261	407	498
12-Apr-2001	17	2.13	2.95	11.0	26.1	47.2	102	225	380	489
		***1.13	4.03	11.3	24.6	49.9	91.7	246	408	472

* Sample deactivated as an outlier (not included in summary statistics)
*** Sample deactivated due to unacceptable internal standard response

000495

Table 10. Back-Calculated Concentrations of Calibration Standards for PFOA (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	2.11	3.56	10.8	25.2	49.2	97.3	241	386	482
14-Apr-2001	19	2.13	3.41	10.4	26.2	47.9	94.7	225	382	506
		*6.30	*6.57	12.2	23.8	49.9	93.1	275	364	474
22-Apr-2001	21	1.71	3.35	10.4	24.8	51.8	95.2	226	384	466
		2.62	3.45	10.7	28.4	48.9	99.2	213	435	489
23-Apr-2001	18	2.40	3.82	10.7	27.5	49.8	80.4	262	388	483
		1.85	3.21	10.9	23.4	54.2	92.6	255	381	465
Mean		2.16	3.43	10.7	25.5	50.5	94.4	245	391	475
S.D.		0.266	0.449	1.24	1.64	3.42	5.09	14.1	16.8	20.1
%CV		12.3	13.1	11.6	6.4	6.8	5.4	5.8	4.3	4.2
%Bias		2.4	-3.7	-0.9	1.2	2.6	-3.0	1.7	1.3	-1.5
n		19	20	24	24	24	24	34	24	24

* Sample deactivated as an outlier (not included in summary statistics)

Table 10. Back-Calculated Concentrations of Calibration Standards for PFOA (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.44	2.88	10.1	24.5	48.5	96.6	241	385	481
11-May-2001	25	1.46	2.95	9.39	25.2	45.5	90.0	245	399	482
		1.33	3.14	11.0	26.3	45.6	94.6	242	371	487
Mean		1.40	3.05	10.2	25.8	45.6	92.3	245	384	480
S.D.		ISD	ISD	ISD	ISD	ISD	ISD	14.1	14.4	12.9
%CV		ISD	ISD	ISD	ISD	ISD	ISD	5.8	3.8	2.7
%Bias		-2.8	5.9	1.0	5.3	-6.0	-4.5	1.7	-0.3	-0.2
n		2	2	2	2	2	2	34	10	10

ISD = insufficient data points for statistical calculations

000497

Table 11. Back-Calculated Concentrations of Calibration Standards for PFOSAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.60	3.10	10.6	25.6	50.6	101	251	401	501
27-Nov-2000	1	1.81	3.48	10.3	26.6	61.0	97.6	303	395	538
		1.39	2.80	9.64	*50.8	**17.1	77.2	252	369	467
28-Nov-2000	2	1.45	2.36	8.59	24.5	51.0	92.0	282	364	491
		1.77	3.84	12.0	*58.2	**22.6	101	293	399	491
23-Mar-2001	4	*7.76	4.18	10.6	24.9	46.5	94.5	301	321	525
		1.32	3.23	10.3	17.3	59.0	108	243	459	460
24-Mar-2001	5	1.62	2.75	11.0	25.6	55.3	103	272	413	511
		1.80	2.59	10.3	26.8	49.2	97.0	255	369	484
Mean		1.59	3.15	10.3	24.3	53.7	96.3	275	386	496
S.D.		0.207	0.641	0.987	3.54	5.72	9.20	23.3	40.7	27.2
%CV		13.0	20.3	9.6	14.6	10.7	9.6	8.5	10.5	5.5
%Bias		-0.6	1.6	-2.8	-5.1	6.1	-4.7	9.6	-3.7	-1.0
n		7	8	8	6	6	8	8	8	8

* Sample deactivated as an outlier (not included in summary statistics)
** Sample deactivated due to sample preparation error

Table 11. Back-Calculated Concentrations of Calibration Standards for PFOSAA (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.30	2.80	4.30	10.3	11.8	25.3	26.8	50.3	51.8	100	102	250	252	400	402	500	502
26-Mar-2001	6		2.68	3.87		12.0		23.8		48.1		86.7		250		358		505
			2.99	4.57		*19.7		26.7		65.1		99.3		287		461		463
26-Mar-2001	7		3.63	4.22		11.3		28.1		59.3		90.3		292		463		446
			2.44	3.48		10.4		26.7		45.8		108		282		380		459
28-Mar-2001	8		3.28	2.92		10.6		24.9		58.9		97.5		242		378		503
			3.05	3.75		14.6		27.2		51.7		101		269		439		481
28-Mar-2001	9		3.68	4.64		11.8		33.2		59.2		110		257		380		516
			2.43	3.02		9.06		21.9		52.8		88.1		302		386		473
30-Mar-2001	10		2.98	3.21		14.6		28.0		48.4		103		253		417		459
			3.21	3.82		10.6		25.9		47.1		111		271		419		495
02-Apr-2001	13		2.32	5.07		11.0		27.2		54.9		101		280		433		513
			3.13	4.16		10.7		24.3		54.1		97.1		262		388		443
07-Apr-2001	14		2.68	3.97		12.1		29.8		49.7		107		266		422		454
			3.29	3.72		12.1		20.8		54.2		107		244		451		468
09-Apr-2001	15		2.87	3.34		9.14		26.5		49.8		107		268		410		435
			3.18	4.62		10.8		26.9		56.0		111		286		421		490
12-Apr-2001	17		3.20	3.67		11.9		29.5		50.8		102		240		381		503
			***0.932	3.88		12.0		26.8		51.6		105		254		420		510

* Sample deactivated as an outlier (not included in summary statistics)
*** Sample deactivated due to unacceptable internal standard response

000499

Table 11. Back-Calculated Concentrations of Calibration Standards for PFOSAA (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.30	2.80	4.30	10.3	11.8	25.3	26.8	50.3	51.8	100	102	250	252	400	402	500	502
14-Apr-2001	19		2.32	4.35		10.7		25.5		54.0		113		254		425		510
			3.30	4.45		11.3		23.9		57.1		87.2		310		340		478
22-Apr-2001	21		2.32	2.75		10.1		26.7		55.7		103		217		393		492
			3.73	4.79		13.0		26.5		54.8		112		238		453		500
23-Apr-2001	18		3.26	3.04		12.7		31.8		53.2		99.2		289		413		467
			3.18	3.81		8.77		22.2		51.4		110		290		403		459
11-May-2001	25	1.45	2.84		10.6		28.3		52.3		102		266		428		495	
		1.24	2.33		10.3		23.2		48.1		94.5		257		386		472	
Mean		1.35	2.97	3.88	10.5	11.4	25.8	26.5	50.2	53.5	98.3	102	262	267	407	410	484	480
S.D.		ISD	0.437	0.634	ISD	1.50	ISD	2.90	ISD	4.48	ISD	8.01	ISD	22.9	ISD	32.1	ISD	24.6
%CV		ISD	14.7	16.3	ISD	13.2	ISD	10.9	ISD	8.4	ISD	7.9	ISD	8.6	ISD	7.8	ISD	5.1
%Bias		3.8	6.1	-9.8	1.9	-3.4	2.0	-1.1	-0.2	3.3	-1.7	0.0	4.8	6.0	1.8	2.0	-3.2	-4.4
n		2	25	24	2	23	2	24	2	24	2	24	2	24	2	24	2	24

ISD = insufficient data points for statistical calculations

000500

Table 12. Back-Calculated Concentrations of Calibration Standards for PFOSA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	1.40	2.50	2.90	10.0	10.4	25.0	25.4	50.0	50.4	100	250	400	500
27-Nov-2000	1	1.10		2.57		9.38		27.4		52.7		97.7	287	378	569
		0.893		2.46		10.0		*46.1		**18.1		87.8	242	349	497
28-Nov-2000	2	1.08		2.40		9.06		26.8		50.2		97.9	283	354	560
		0.906		2.70		10.2		*48.6		**19.1		93.3	256	377	480
23-Mar-2001	4	0.984		2.94		9.66		26.3		50.7		93.2	285	370	522
		0.906		2.80		10.6		17.0		49.6		106	240	431	458
24-Mar-2001	5	0.935		2.47		10.1		24.6		51.2		98.1	255	406	517
		1.08		2.40		10.1		26.3		49.2		98.4	247	382	497
26-Mar-2001	6		1.38		2.93		9.65		24.1		43.9	94.7	245	382	491
			1.38		3.10		*16.2		26.6		58.7	101	260	453	471
26-Mar-2001	7		*2.12		3.01		10.3		26.6		50.7	88.9	251	423	461
			1.41		2.79		10.0		26.1		46.4	110	273	377	518
28-Mar-2001	8		*0.353		3.21		11.1		25.2		54.8	99.8	240	409	527
			1.36		2.74		10.1		25.5		45.6	96.8	247	401	479
28-Mar-2001	9		1.53		3.37		10.2		30.2		51.2	106	254	388	500
			1.25		2.65		8.72		21.7		49.8	89.4	304	387	471
30-Mar-2001	10		1.39		2.47		12.1		27.1		48.9	92.0	238	409	488
			1.51		2.83		9.54		26.4		48.9	104	261	416	492
30-Mar-2001	11		*2.96		2.91		11.1		23.8		50.3	99.9	259	383	448
			1.39		2.95		9.56		24.4		58.4	*154	203	476	524

* Sample deactivated as an outlier (not included in summary statistics)
** Sample deactivated due to sample preparation error

000501

Table 12. Back-Calculated Concentrations of Calibration Standards for PFOSA (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	1.40	2.50	2.90	10.0	10.4	25.0	25.4	50.0	50.4	100	250	400	500
02-Apr-2001	13		1.06		2.93		10.2		26.1		52.2	99.3	262	429	517
			1.66		3.32		9.31		24.0		51.2	97.3	249	393	452
07-Apr-2001	14		1.14		3.23		10.9		29.4		52.0	109	285	426	502
			1.66		2.58		10.1		18.9		48.0	97.7	227	399	457
09-Apr-2001	15		*2.32		2.77		9.77		28.9		51.8	112	281	429	518
			1.40		3.11		9.74		23.9		46.7	92.5	232	364	472
11-Apr-2001	16		1.59		2.67		9.79		22.9		55.6	88.9	285	346	473
			1.23		3.16		9.03		26.6		54.6	111	241	416	533
12-Apr-2001	17		1.47		2.55		10.5		29.5		47.5	103	223	386	536
			***0.557		2.86		11.0		25.2		47.4	102	247	403	501
23-Apr-2001	18		1.44		3.00		9.87		28.1		49.5	83.9	256	405	481
			1.33		3.01		9.81		23.4		50.8	115	269	389	503
Mean		0.986	1.40	2.59	2.92	9.89	10.1	24.7	25.6	50.6	50.6	98.9	256	398	497
S.D.		0.0884	0.158	0.201	0.244	0.493	0.758	3.90	2.63	1.26	3.78	7.71	21.8	29.0	30.3
%CV		9.0	11.3	7.8	8.4	5.0	7.5	15.8	10.3	2.5	7.5	7.8	8.5	7.3	6.1
%Bias		-1.4	0.0	3.6	0.7	-1.1	-2.9	-1.2	0.8	1.2	0.4	-1.1	2.4	-0.5	-0.6
n		8	19	8	24	8	23	6	24	6	24	31	32	32	32

* Sample deactivated as an outlier (not included in summary statistics)
*** Sample deactivated due to unacceptable internal standard response

030502

Table 13. Back-Calculated Concentrations of Calibration Standards for M556

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	2.50	3.20	10.0	10.7	25.0	25.7	50.0	50.7	100	101	250	251	400	401	500	501
27-Nov-2000	1	2.81		10.2		27.5		56.0		98.1		287		421		555	
		2.23		8.65		*45.7		**17.3		84.1		235		360		451	
28-Nov-2000	2	2.20		9.06		24.5		48.3		91.1		269		378		499	
		2.83		10.7		*53.6		**21.2		106		274		404		486	
24-Mar-2001	5	2.05		9.42		24.5		52.5		104		256		407		504	
		2.98		10.0		24.7		49.8		99.5		248		384		499	
26-Mar-2001	6		3.16		9.86		23.1		43.9		86.7		234		361		497
			3.08		13.9		25.7		60.8		97.4		271		460		496
26-Mar-2001	7		3.15		10.5		25.7		55.1		85.8		258		445		468
			3.15		12.1		26.0		45.1		101		263		381		506
28-Mar-2001	8		3.13		9.57		29.3		53.8		91.5		234		373		508
			*4.76		12.2		25.6		47.5		96.3		255		440		504
30-Mar-2001	10		2.75		12.4		27.1		49.3		96.9		245		407		485
			3.55		10.4		24.0		47.4		101		265		416		496
30-Mar-2001	11		3.36		12.2		23.8		52.0		102		247		364		445
			3.00		10.2		23.2		58.2		*145		209		487		538
01-Apr-2001	12		3.90		10.7		29.4		56.4		102		263		398		498
			2.63		8.84		23.4		52.9		80.0		289		383		490

* Sample deactivated as an outlier (not included in summary statistics)
** Sample deactivated due to sample preparation error

000503

Table 13. Back-Calculated Concentrations of Calibration Standards for M556 (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	2.50	3.20	10.0	10.7	25.0	25.7	50.0	50.7	100	101	250	251	400	401	500	501
07-Apr-2001	14		3.47		9.97		28.4		49.5		99.9		258		425		478
			2.95		11.4		20.9		54.3		104		239		427		477
09-Apr-2001	15		2.68		8.84		27.5		49.7		108		254		421		459
			3.93		9.80		24.8		52.2		103		266		416		489
12-Apr-2001	17		2.67		10.5		25.8		46.2		97.3		226		368		490
			3.68		11.4		26.2		54.6		105		255		441		523
14-Apr-2001	19		3.21		9.15		25.9		51.3		106		225		408		530
			3.30		11.0		23.8		55.8		96.2		303		348		488
23-Apr-2001	18		2.89		10.2		25.8		51.1		81.9		253		402		461
			3.65		9.66		23.7		53.8		115		295		419		488
Mean		2.52	3.20	9.67	10.7	25.3	25.4	51.7	51.9	97.1	97.9	262	255	392	409	499	492
S.D.		0.400	0.387	0.765	1.29	1.47	2.12	3.38	4.29	8.23	8.74	18.9	22.7	22.3	34.9	33.6	22.4
%CV		15.9	12.1	7.9	12.1	5.8	8.3	6.5	8.3	8.5	8.9	7.2	8.9	5.7	8.5	6.7	4.6
%Bias		0.8	0.0	-3.3	0.0	1.2	-1.2	3.4	2.4	-2.9	-3.1	4.8	1.6	-2.0	2.0	-0.2	-1.8
n		6	21	6	22	4	22	4	22	6	21	6	22	6	22	6	22

050504

Table 14. Back-Calculated Concentrations of Calibration Standards for M570

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	1.80	2.50	3.30	10.0	10.8	25.0	25.8	50.0	50.8	100	101	250	251	400	401	500	501
27-Nov-2000	1	1.05		2.47		9.76		26.4		58.7		94.4		275		390		530	
		1.01		2.09		10.1		*50.6		**18.6		87.5		266		374		472	
28-Nov-2000	2	0.991		2.16		8.82		23.2		52.0		88.8		269		374		480	
		*1.56		2.90		11.4		*58.7		**21.2		101		274		407		507	
24-Mar-2001	5	0.927		2.07		10.3		26.0		55.4		96.9		263		414		507	
		1.15		2.40		9.59		26.1		50.6		97.2		241		382		492	
26-Mar-2001	6		1.60		2.96		10.5		24.6		48.6		90.0		238		354		489
			2.08		3.37		*18.0		28.3		*69.4		108		296		461		474
26-Mar-2001	7		2.04		3.40		9.97		25.7		56.3		89.3		274		462		464
			1.49		3.55		10.2		27.4		44.2		105		262		385		469
28-Mar-2001	8		1.89		3.04		9.96		25.8		60.0		101		228		405		503
			1.90		2.84		12.3		25.4		48.3		94.2		262		435		473
30-Mar-2001	10		1.87		2.55		12.8		28.6		48.7		98.3		234		413		468
			2.08		2.73		9.87		26.2		47.6		109		279		410		500
01-Apr-2001	12		2.15		3.70		10.3		32.2		56.2		103		254		387		508
			1.60		2.53		8.98		22.4		53.2		86.3		297		370		496
02-Apr-2001	13		1.53		2.87		10.3		27.1		54.5		98.1		258		426		522
			2.25		3.19		10.3		24.4		54.2		101		266		382		450

* Sample deactivated as an outlier (not included in summary statistics)
** Sample deactivated due to sample preparation error

000505

Table 14. Back-Calculated Concentrations of Calibration Standards for M570 (Continued)

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	1.80	2.50	3.30	10.0	10.8	25.0	25.8	50.0	50.8	100	101	250	251	400	401	500	501
07-Apr-2001	14		1.65		3.24		10.9		30.4		52.3		109		263		436		475
			2.11		2.80		10.8		19.4		52.0		102		231		426		466
09-Apr-2001	15		1.98		2.39		8.68		26.3		48.4		107		255		403		448
			1.86		3.60		10.1		27.6		54.8		107		279		424		491
12-Apr-2001	17		2.03		2.78		11.5		28.1		50.5		103		229		387		517
			***0.564		2.92		11.3		24.7		51.2		104		248		416		501
14-Apr-2001	19		1.46		3.44		9.54		25.8		52.4		112		240		418		521
			2.18		3.19		10.2		24.0		56.6		88.5		310		340		477
22-Apr-2001	21		1.94		2.56		10.9		25.2		54.0		106		214		395		502
			*3.02		3.40		12.4		27.2		48.9		101		214		448		518
24-Apr-2001	22		1.68		3.12		10.1		22.5		53.7		119		268		390		508
			2.15		2.76		10.4		29.0		52.8		86.3		263		406		470
11-May-2001	25	1.03		2.64		10.3		26.8		50.8		99.5		257		435		466	
		1.01		2.12		9.85		24.6		46.9		98.6		267		370		508	
Mean		1.02	1.89	2.36	3.04	10.0	10.5	25.5	26.2	52.4	52.1	95.5	101	264	257	393	407	495	488
S.D.		0.0677	0.246	0.302	0.371	0.736	1.01	1.36	2.72	4.12	3.64	4.94	8.50	10.9	25.4	23.2	30.6	21.7	22.4
%CV		6.6	13.0	12.8	12.2	7.4	9.6	5.3	10.4	7.9	7.0	5.2	8.4	4.1	9.9	5.9	7.5	4.4	4.6
%Bias		2.0	5.0	-5.6	-7.9	0.0	-2.8	2.0	1.6	4.8	2.6	-4.5	0.0	5.6	2.4	-1.8	1.5	-1.0	-2.6
n		7	22	8	24	8	23	6	24	6	23	8	24	8	24	8	24	8	24

* Sample deactivated as an outlier (not included in summary statistics)
*** Sample deactivated due unacceptable internal standard response

000506

Table 15. Analytical QC Summary for PFOS

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 6.40 ppb	Low QC 5.75 ppb	Medium QC 126 ppb	Medium QC 126 ppb	High QC 332 ppb	High QC 332 ppb	High QC 331 ppb	High QC 348 ppb DF=5
	Preparation Date	27-Oct-2000	05-Apr-2001	27-Oct-2000	05-Apr-2001	27-Oct-2000	08-Mar-2001	05-Apr-2001	08-Mar-2001
27-Nov-2000	1	5.91		121		320			
		6.13		127		372			
28-Nov-2000	2	6.49		121		347			
		5.82		125		351			
23-Mar-2001	4	*4.60		135		354			
		6.50		*192		333			
24-Mar-2001	5	6.36		134		343			
		6.49		131		362			
26-Mar-2001	6	5.88		**74.2			304		
		6.49		119			344		
26-Mar-2001	7	6.70		139			326		
		5.88		123			323		
28-Mar-2001	8	5.58		117			348		
		6.29		133			323		
28-Mar-2001	9	5.65		141			357		
		5.14		140			344		

* >±20% deviation from theoretical
** Sample deactivated due to unacceptable internal standard response

000507

Table 15. Analytical QC Summary for PFOS (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 6.40 ppb	Low QC 5.75 ppb	Medium QC 126 ppb	Medium QC 126 ppb	High QC 332 ppb	High QC 332 ppb	High QC 331 ppb	High QC 348 ppb DF=5
	Preparation Date	27-Oct-2000	05-Apr-2001	27-Oct-2000	05-Apr-2001	27-Oct-2000	08-Mar-2001	05-Apr-2001	08-Mar-2001
30-Mar-2001	10	6.08		127			324		
		5.74		138			332		
30-Mar-2001	11	6.60		126			328		
		5.82		118			**149		
02-Apr-2001	13	6.47		130			346		
		6.65		137			330		
07-Apr-2001	14	6.21		123			373		
		6.22		125			330		
09-Apr-2001	15	5.78		132			332		
		5.84		135			357		
11-Apr-2001	16	6.99		130			339		
		6.51		122			345		
12-Apr-2001	17	6.25		137			357		353
		6.20		141			352		357
									365

** Sample deactivated due to unacceptable internal standard response

Table 15. Analytical QC Summary for PFOS (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 6.40 ppb	Low QC 5.75 ppb	Medium QC 126 ppb	Medium QC 126 ppb	High QC 332 ppb	High QC 332 ppb	High QC 331 ppb	High QC 348 ppb DF=5
	Preparation Date	27-Oct-2000	05-Apr-2001	27-Oct-2000	05-Apr-2001	27-Oct-2000	08-Mar-2001	05-Apr-2001	08-Mar-2001
23-Apr-2001	18		6.57		138			296	
			6.84		130			330	
Mean		6.11	6.71	132	134	348	339	313	358
S.D.		0.492	ISD	13.7	ISD	16.3	15.8	ISD	6.11
%CV		8.1	ISD	10.4	ISD	4.7	4.7	ISD	1.7
%Theoretical		95.5	116.7	104.8	106.3	104.8	102.1	94.6	102.9
%Bias		-4.5	16.7	4.8	6.3	4.8	2.1	-5.4	2.9
n		30	2	29	2	8	21	2	3

ISD = insufficient data points for statistical calculation

650569

Table 16. Analytical QC Summary for PFHS

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.48 ppb 27-Oct-2000	Low QC 4.48 ppb 05-Apr-2001	Medium QC 156 ppb 27-Oct-2000	Medium QC 157 ppb 05-Apr-2001	High QC 417 ppb 27-Oct-2000	High QC 417 ppb 08-Mar-2001	High QC 418 ppb 05-Apr-2001
27-Nov-2000	1	3.61		161		382		
		4.28		148		443		
28-Nov-2000	2	3.70		157		411		
		3.86		153		445		
23-Mar-2001	4	4.29		155		428		
		4.06		171		406		
24-Mar-2001	5	3.96		164		414		
		4.02		161		460		
26-Mar-2001	6	4.85		**128			373	
		5.07		157			396	
26-Mar-2001	7	4.71		157			411	
		4.95		135			407	
28-Mar-2001	8	5.31		158			420	
		4.35		158			383	
28-Mar-2001	9	4.32		158			392	
		3.92		158			415	

** Sample deactivated due to unacceptable internal standard response

000510

Table 16. Analytical QC Summary for PFHS (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.48 ppb 27-Oct-2000	Low QC 4.48 ppb 05-Apr-2001	Medium QC 156 ppb 27-Oct-2000	Medium QC 157 ppb 05-Apr-2001	High QC 417 ppb 27-Oct-2000	High QC 417 ppb 08-Mar-2001	High QC 418 ppb 05-Apr-2001
30-Mar-2001	10	*3.48		136			413	
		4.29		146			345	
30-Mar-2001	11	4.08		151			391	
		4.06		154			**200	
02-Apr-2001	13	4.20		158			428	
		4.38		158			436	
07-Apr-2001	14	4.87		153			408	
		4.25		149			378	
09-Apr-2001	15	3.92		156			444	
		3.68		152			401	
11-Apr-2001	16	4.85		155			387	
		3.62		155			383	
12-Apr-2001	17	4.14		157			397	
		4.43		153			404	

* >±20% deviation from theoretical
** Sample deactivated due to unacceptable internal standard response

000511

Table 16. Analytical QC Summary for PFHS (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.48 ppb 27-Oct-2000	Low QC 4.48 ppb 05-Apr-2001	Medium QC 156 ppb 27-Oct-2000	Medium QC 157 ppb 05-Apr-2001	High QC 417 ppb 27-Oct-2000	High QC 417 ppb 08-Mar-2001	High QC 418 ppb 05-Apr-2001
23-Apr-2001	18		3.87		166			397
			4.05		145			389
Mean		4.25	3.96	155	156	424	401	393
S.D.		0.467	ISD	7.17	ISD	25.3	22.6	ISD
%CV		11.0	ISD	4.6	ISD	6.0	5.6	ISD
%Theoretical		94.9	88.4	99.4	99.4	101.7	96.2	94.0
%Bias		-5.1	-11.6	-0.6	-0.6	1.7	-3.8	-6.0
n		30	2	29	2	8	21	2

ISD = insufficient data points for statistical calculation

Table 17. Analytical QC Summary for PFOA

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.81 ppb 27-Oct-2000	Low QC 4.32 ppb 05-Apr-2001	Low QC 4.32 ppb 02-May-2001	Medium QC 145 ppb 27-Oct-2000	Medium QC 145 ppb 05-Apr-2001	High QC 385 ppb 27-Oct-2000	High QC 385 ppb 08-Mar-2001	High QC 385 ppb 05-Apr-2001
27-Nov-2000	1	4.55			154		403		
		4.30			160		429		
28-Nov-2000	2	4.35			151		416		
		3.92			162		439		
23-Mar-2001	4	*2.72			159		412		
		4.85			152		381		
24-Mar-2001	5	4.09			143		390		
		*3.69			151		377		
26-Mar-2001	6	4.12			**124			393	
		4.81			152			395	
26-Mar-2001	7	5.47			146			348	
		4.93			140			384	
28-Mar-2001	8	4.63			151			404	
		*0.161			156			375	
28-Mar-2001	9	4.33			158			404	
		3.89			157			433	

* >±20% deviation from theoretical
** Sample deactivated due to unacceptable internal standard response

Table 17. Analytical QC Summary for PFOA (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.81 ppb 27-Oct-2000	Low QC 4.32 ppb 05-Apr-2001	Low QC 4.32 ppb 02-May-2001	Medium QC 145 ppb 27-Oct-2000	Medium QC 145 ppb 05-Apr-2001	High QC 385 ppb 27-Oct-2000	High QC 385 ppb 08-Mar-2001	High QC 385 ppb 05-Apr-2001
30-Mar-2001	10	4.29			151			393	
		4.14			155			382	
02-Apr-2001	13	4.56			145			381	
		4.15			139			336	
07-Apr-2001	14	*5.97			145			381	
		4.77			136			361	
09-Apr-2001	15	4.45			159			406	
		*3.61			147			351	
12-Apr-2001	17	4.17			151			372	
		4.07			158			375	
14-Apr-2001	19	*3.79			131			377	
		5.04			*109			401	
22-Apr-2001	21	4.03			143			401	
		3.90			170			361	
23-Apr-2001	18		3.82			141			370
			4.67			151			414

* >±20% deviation from theoretical

030514

Table 17. Analytical QC Summary for PFOA (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.81 ppb	Low QC 4.32 ppb	Low QC 4.32 ppb	Medium QC 145 ppb	Medium QC 145 ppb	High QC 385 ppb	High QC 385 ppb	High QC 385 ppb
	Preparation Date	27-Oct-2000	05-Apr-2001	02-May-2001	27-Oct-2000	05-Apr-2001	27-Oct-2000	08-Mar-2001	05-Apr-2001
11-May-2001	25			4.16		150			386
				4.65		148			398
Mean		4.19	4.25	4.41	149	148	406	382	392
S.D.		0.970	ISD	ISD	11.5	4.51	22.3	22.5	18.6
%CV		23.2	ISD	ISD	7.7	3.0	5.5	5.9	4.7
%Theoretical		87.1	98.4	102.1	102.8	102.1	105.5	99.2	101.8
%Bias		-12.9	-1.6	2.1	2.8	2.1	5.5	-0.8	1.8
n		30	2	2	29	4	8	22	4

ISD = insufficient data points for statistical calculation

Table 18. Analytical QC Summary for PFOSAA

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.60 ppb 27-Oct-2000	Low QC 4.30 ppb 05-Apr-2001	Low QC 4.30 ppb 02-May-2001	Medium QC 151 ppb 27-Oct-2000	Medium QC 150 ppb 05-Apr-2001	High QC 401 ppb 27-Oct-2000	High QC 401 ppb 08-Mar-2001	High QC 400 ppb 05-Apr-2001
27-Nov-2000	1	4.22			141		398		
		*3.38			167		425		
28-Nov-2000	2	4.50			150		378		
		3.66			165		368		
23-Mar-2001	4	*2.38			172		463		
		4.11			*374		456		
24-Mar-2001	5	4.22			157		422		
		4.11			157		389		
26-Mar-2001	6	4.55			**71.5			348	
		4.98			130			445	
26-Mar-2001	7	4.85			146			365	
		4.26			135			384	
28-Mar-2001	8	*6.56			145			415	
		4.31			142			383	
28-Mar-2001	9	4.16			183			440	
		4.41			175			415	

* >±25% deviation from theoretical
** Sample deactivated due to unacceptable internal standard response

Table 18. Analytical QC Summary for PFOSAA (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.60 ppb	Low QC 4.30 ppb	Low QC 4.30 ppb	Medium QC 151 ppb	Medium QC 150 ppb	High QC 401 ppb	High QC 401 ppb	High QC 400 ppb
	Preparation Date	27-Oct-2000	05-Apr-2001	02-May-2001	27-Oct-2000	05-Apr-2001	27-Oct-2000	08-Mar-2001	05-Apr-2001
30-Mar-2001	10	5.47			149			380	
		4.96			170			376	
02-Apr-2001	13	*6.00			154			412	
		5.38			153			360	
07-Apr-2001	14	5.41			166			452	
		4.70			154			439	
09-Apr-2001	15	4.61			170			426	
		3.71			165			380	
12-Apr-2001	17	5.23			178			465	
		5.04			*189			448	
14-Apr-2001	19	4.60			160			420	
		5.43			123			429	
22-Apr-2001	21	*6.60			149			432	
		4.64			*218			413	
23-Apr-2001	18		4.41			167			398
			5.33			169			427

* >±25% deviation from theoretical

030517

Table 18. Analytical QC Summary for PFOSAA (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.60 ppb 27-Oct-2000	Low QC 4.30 ppb 05-Apr-2001	Low QC 4.30 ppb 02-May-2001	Medium QC 151 ppb 27-Oct-2000	Medium QC 150 ppb 05-Apr-2001	High QC 401 ppb 27-Oct-2000	High QC 401 ppb 08-Mar-2001	High QC 400 ppb 05-Apr-2001
11-May-2001	25			4.34		143			424
				3.95		145			407
Mean		4.68	4.87	4.15	167	156	412	410	414
S.D.		0.882	ISD	ISD	44.2	13.9	35.1	33.2	13.8
%CV		18.8	ISD	ISD	26.5	8.9	8.5	8.1	3.3
%Theoretical		101.7	113.3	96.5	110.6	104.0	102.7	102.2	103.5
%Bias		1.7	13.3	-3.5	10.6	4.0	2.7	2.2	3.5
n		30	2	2	29	4	8	22	4

ISD = insufficient data points for statistical calculations

000518

Table 19. Analytical QC Summary for PFOSA

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.00 ppb 27-Oct-2000	Low QC 4.00 ppb 05-Apr-2001	Medium QC 150 ppb 27-Oct-2000	Medium QC 150 ppb 05-Apr-2001	High QC 400 ppb 27-Oct-2000	High QC 400 ppb 08-Mar-2001	High QC 400 ppb 05-Apr-2001
27-Nov-2000	1	4.39		147		399		
		4.12		162		466		
28-Nov-2000	2	4.86		146		416		
		3.97		158		386		
23-Mar-2001	4	*2.33		180		434		
		4.47		*328		417		
24-Mar-2001	5	3.94		156		423		
		4.21		158		399		
26-Mar-2001	6	3.85		**72.6			311	
		3.85		133			457	
26-Mar-2001	7	4.80		141			369	
		3.98		133			396	
28-Mar-2001	8	4.16		146			431	
		3.58		155			406	
28-Mar-2001	9	3.84		173			447	
		3.82		157			408	

* >±25% deviation from theoretical
** Sample deactivated due to unacceptable internal standard response

Table 19. Analytical QC Summary for PFOSA (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.00 ppb 27-Oct-2000	Low QC 4.00 ppb 05-Apr-2001	Medium QC 150 ppb 27-Oct-2000	Medium QC 150 ppb 05-Apr-2001	High QC 400 ppb 27-Oct-2000	High QC 400 ppb 08-Mar-2001	High QC 400 ppb 05-Apr-2001
30-Mar-2001	10	4.16		144			400	
		3.96		161			368	
30-Mar-2001	11	*5.14		138			392	
		4.08		124			**170	
02-Apr-2001	13	4.21		148			431	
		4.39		159			361	
07-Apr-2001	14	4.79		150			456	
		4.46		147			424	
09-Apr-2001	15	4.19		157			423	
		3.92		157			422	
11-Apr-2001	16	*5.84		165			404	
		4.95		127			425	
12-Apr-2001	17	4.53		155			437	
		4.38		170			403	

* >±25% deviation from theoretical
** Sample deactivated due to unacceptable internal standard response

Table 19. Analytical QC Summary for PFOSA (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Low QC 4.00 ppb	Medium QC 150 ppb	Medium QC 150 ppb	High QC 400 ppb	High QC 400 ppb	High QC 400 ppb
	Preparation Date	27-Oct-2000	05-Apr-2001	27-Oct-2000	05-Apr-2001	27-Oct-2000	08-Mar-2001	05-Apr-2001
23-Apr-2001	18		4.95		178			392
			4.73		170			436
Mean		4.24	4.84	158	174	418	408	414
S.D.		0.596	ISD	35.3	ISD	24.9	34.9	ISD
%CV		14.1	ISD	22.3	ISD	6.0	8.6	ISD
%Theoretical		106.0	121.0	105.3	116.0	104.5	102.0	103.5
%Bias		6.0	21.0	5.3	16.0	4.5	2.0	3.5
n		30	2	29	2	8	21	2

ISD = insufficient data points for statistical calculations

Table 20. Analytical QC Summary for M556

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Low QC 4.00 ppb	Medium QC 150 ppb	Medium QC 150 ppb	High QC 400 ppb	High QC 400 ppb	High QC 400 ppb
	Preparation Date	27-Oct-2000	05-Apr-2001	27-Oct-2000	05-Apr-2001	27-Oct-2000	08-Mar-2001	05-Apr-2001
27-Nov-2000	1	4.09		133		423		
		3.23		161		439		
28-Nov-2000	2	4.52		144		393		
		3.60		166		389		
24-Mar-2001	5	4.77		160			402	
		3.60		156			401	
26-Mar-2001	6	3.52		**79.4			367	
		4.07		137			428	
26-Mar-2001	7	4.45		137			354	
		*2.73		124			404	
28-Mar-2001	8	*6.68		164			426	
		4.15		166			388	
30-Mar-2001	10	*4.85		149			382	
		4.06		171			385	
30-Mar-2001	11	***7.47		146			369	
		4.28		126			**173	
01-Apr-2001	12	*5.69		162			396	
		4.35		158			388	

* >±20% deviation from theoretical
** Sample deactivated due to unacceptable internal standard response
*** Sample deactivated due to carryover

050522

Table 20. Analytical QC Summary for M556 (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.00 ppb 27-Oct-2000	Low QC 4.00 ppb 05-Apr-2001	Medium QC 150 ppb 27-Oct-2000	Medium QC 150 ppb 05-Apr-2001	High QC 400 ppb 27-Oct-2000	High QC 400 ppb 08-Mar-2001	High QC 400 ppb 05-Apr-2001
07-Apr-2001	14	4.73		152		407		
		3.51		145		387		
09-Apr-2001	15	4.02		171			434	
		*3.14		164			395	
12-Apr-2001	17	4.40		164			395	
		4.29		162			387	
14-Apr-2001	19	3.75		155			396	
		4.71		*115			406	
23-Apr-2001	18		4.51		180			381
			4.11		160			395
Mean		4.21	4.31	152	170	406	395	388
S.D.		0.818	ISD	15.5	ISD	21.0	20.3	ISD
%CV		19.4	ISD	10.2	ISD	5.2	5.1	ISD
%Theoretical		105.3	107.8	101.3	113.3	101.5	98.8	97.0
%Bias		5.3	7.8	1.3	13.3	1.5	-1.3	-3.0
n		25	2	25	2	6	19	2

* >±20% deviation from theoretical
ISD = insufficient data points for statistical calculations

030523

Table 21. Analytical QC Summary for M570

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Low QC 4.00 ppb	Low QC 4.00 ppb	Medium QC 150 ppb	Medium QC 150 ppb	High QC 400 ppb	High QC 400 ppb	High QC 400 ppb
	Preparation Date	27-Oct-2000	05-Apr-2001	02-May-2001	27-Oct-2000	05-Apr-2001	27-Oct-2000	08-Mar-2001	05-Apr-2001
27-Nov-2000	1	3.61			149		388		
		*3.07			163		451		
28-Nov-2000	2	4.10			151		383		
		3.59			169		394		
24-Mar-2001	5	3.81			168		415		
		3.89			165		412		
26-Mar-2001	6	3.63			**74.2			338	
		4.00			140			447	
26-Mar-2001	7	4.75			150			344	
		3.26			133			403	
28-Mar-2001	8	*5.01			156			433	
		3.25			161			396	
30-Mar-2001	10	4.64			150			359	
		3.54			180			379	
01-Apr-2001	12	3.78			166			411	
		3.60			169			364	

* >±20% deviation from theoretical
** Sample deactivated due to unacceptable internal standard response

050524

Table 21. Analytical QC Summary for M570 (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.00 ppb 27-Oct-2000	Low QC 4.00 ppb 05-Apr-2001	Low QC 4.00 ppb 02-May-2001	Medium QC 150 ppb 27-Oct-2000	Medium QC 150 ppb 05-Apr-2001	High QC 400 ppb 27-Oct-2000	High QC 400 ppb 08-Mar-2001	High QC 400 ppb 05-Apr-2001
02-Apr-2001	13	4.55			156			405	
		3.99			162			362	
07-Apr-2001	14	4.80			159			446	
		4.37			156			423	
09-Apr-2001	15	3.91			168			380	
		*2.69			163			375	
12-Apr-2001	17	4.38			172			447	
		4.28			*185			446	
14-Apr-2001	19	4.17			158			413	
		4.26			120			423	
22-Apr-2001	21	*5.54			146			447	
		3.89			*207			413	
24-Apr-2001	22		*3.11			178			373
			4.18			168			393

* >±20% deviation from theoretical

000525

Table 21. Analytical QC Summary for M570 (Continued)

All concentrations are expressed as ppb.

Run Date	Run Number Preparation Date	Low QC 4.00 ppb 27-Oct-2000	Low QC 4.00 ppb 05-Apr-2001	Low QC 4.00 ppb 02-May-2001	Medium QC 150 ppb 27-Oct-2000	Medium QC 150 ppb 05-Apr-2001	High QC 400 ppb 27-Oct-2000	High QC 400 ppb 08-Mar-2001	High QC 400 ppb 05-Apr-2001
11-May-2001	25			4.51		143			417
				*2.83		149			408
Mean		4.01	3.65	3.67	160	160	407	402	398
S.D.		0.621	ISD	ISD	16.6	16.3	25.0	35.4	19.2
%CV		15.5	ISD	ISD	10.4	10.2	6.1	8.8	4.8
%Theoretical		100.3	91.3	91.8	106.7	106.7	101.8	100.5	99.5
%Bias		0.3	-8.8	-8.3	6.7	6.7	1.8	0.5	-0.5
n		28	2	2	27	4	6	22	4

* >±20% deviation from theoretical
ISD = insufficient data points for statistical calculations

Table 22. Study Sample Concentrations

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10956	54.1	3.10	3.95	3.68	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10957	51.3	5.45	6.64	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.10
10958	80.5	12.4	22.2	7.59	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10959	69.3	3.01	13.9	4.13	<LLOQ(1.40)	<LLOQ(3.20)	2.26
10960	37.3	2.95	5.13	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10961	48.4	2.79	2.77	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	6.18
10962	55.7	<LLOQ(2.09)	4.44	4.21	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10963	42.0	<LLOQ(2.09)	6.58	3.55	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10964	114	<LLOQ(2.09)	7.07	9.05	<LLOQ(1.40)	<LLOQ(3.20)	4.97
10965	45.0	8.00	3.90	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.55
10966	29.2	<LLOQ(2.09)	<LLOQ(2.11)	2.81	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10967	31.7	<LLOQ(2.09)	<LLOQ(2.11)	4.58	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10968	42.8	9.26	5.17	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	4.72
10969	37.9	2.96	<LLOQ(2.11)	4.23	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10970	46.7	<LLOQ(2.09)	3.35	3.08	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10971	13.4	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10972	24.0	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10973	34.1	3.68	5.11	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10974	51.8	<LLOQ(2.09)	3.37	3.46	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10975	35.8	5.73	3.04	3.43	<LLOQ(1.40)	<LLOQ(3.20)	2.03
10976	44.0	2.62	7.32	3.43	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10977	99.7	5.98	6.94	8.67	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10978	48.7	<LLOQ(2.09)	3.23	4.47	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10979	44.2	<LLOQ(2.09)	3.16	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10980	57.7	3.66	4.86	4.33	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10981	25.8	4.98	2.59	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10982	46.4	7.28	2.20	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.13
10983	38.6	<LLOQ(2.09)	<LLOQ(2.11)	3.90	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10984	75.1	<LLOQ(2.09)	17.4	3.44	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10985	41.9	6.97	2.22	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.86
10986	57.7	3.26	5.04	5.09	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10987	14.0	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.31
10988	39.2	2.17	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10989	68.9	2.53	5.28	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.16
10990	38.6	<LLOQ(2.09)	4.42	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10991	36.6	2.11	<LLOQ(2.11)	3.45	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10992	19.4	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10993	18.9	<LLOQ(2.09)	2.29	3.62	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10994	44.6	2.18	2.51	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10995	61.6	2.26	4.38	4.11	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10996	39.2	<LLOQ(2.09)	3.69	5.76	<LLOQ(1.40)	<LLOQ(3.20)	5.11
10997	25.6	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10998	70.6	<LLOQ(2.09)	6.92	5.16	<LLOQ(1.40)	<LLOQ(3.20)	2.99
10999	101	8.84	8.54	4.69	<LLOQ(1.40)	<LLOQ(3.20)	16.4
11000	42.3	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11001	16.3	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11002	127	3.45	9.90	6.22	<LLOQ(1.40)	<LLOQ(3.20)	3.99
11003	65.2	<LLOQ(2.09)	2.84	3.69	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11004	40.1	<LLOQ(2.09)	3.43	3.63	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11005	25.8	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.17
11006	39.1	2.17	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.58
11007	36.8	<LLOQ(2.09)	9.12	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11008	26.2	<LLOQ(2.09)	4.76	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11009	19.5	<LLOQ(2.09)	<LLOQ(2.11)	3.01	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11010	40.9	2.18	2.78	3.95	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11011	33.6	<LLOQ(2.09)	5.50	6.00	<LLOQ(1.40)	12.7	5.18
11012	23.0	2.26	3.73	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.33
11013	47.8	7.39	8.05	4.93	<LLOQ(1.40)	<LLOQ(3.20)	1.88
11014	38.7	<LLOQ(2.09)	4.25	6.10	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11015	105	2.29	10.2	6.49	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11016	42.2	8.70	7.65	4.12	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11017	27.7	<LLOQ(2.09)	3.55	3.34	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11018	19.0	2.20	3.75	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11019	84.9	2.67	17.0	3.96	<LLOQ(1.40)	<LLOQ(3.20)	2.00
11020	33.3	2.59	3.80	4.63	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11021	19.4	<LLOQ(2.09)	2.47	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11022	15.1	3.58	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11023	50.3	3.59	3.83	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.53
11024	57.5	<LLOQ(2.09)	5.91	7.77	<LLOQ(1.40)	<LLOQ(3.20)	2.45
11025	57.5	<LLOQ(2.09)	7.68	7.48	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11026	48.8	2.74	3.97	3.49	<LLOQ(1.40)	<LLOQ(3.20)	2.20
11027	48.7	5.70	6.74	4.95	<LLOQ(1.40)	<LLOQ(3.20)	2.26

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11028	45.2	2.13	6.43	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11029	21.9	<LLOQ(2.09)	3.61	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11030	36.7	<LLOQ(2.09)	6.17	3.00	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11031	54.5	8.04	4.69	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11032	39.8	<LLOQ(2.09)	3.95	3.25	<LLOQ(1.40)	<LLOQ(3.20)	5.51
11033	63.2	4.36	6.34	3.25	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11034	26.4	9.84	4.68	4.83	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11035	38.6	<LLOQ(2.09)	9.98	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.84
11036	26.4	<LLOQ(2.09)	2.95	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.57
11037	17.3	<LLOQ(2.09)	<LLOQ(2.11)	4.44	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11038	41.1	2.23	3.17	7.37	<LLOQ(1.40)	<LLOQ(3.20)	5.93
11039	60.7	2.85	7.45	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11040	44.7	<LLOQ(2.09)	4.35	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11041	42.8	3.07	10.1	3.19	<LLOQ(1.40)	<LLOQ(3.20)	2.04
11042	55.7	<LLOQ(2.09)	3.84	3.85	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11043	27.1	<LLOQ(2.09)	2.39	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11044	34.2	<LLOQ(2.09)	5.38	2.84	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11045	49.0	2.16	6.15	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11046	56.9	2.11	5.98	4.34	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11047	73.8	3.75	7.45	3.84	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11048	70.8	<LLOQ(2.09)	8.77	27.6	<LLOQ(1.40)	5.88	7.82
11049	33.5	<LLOQ(2.09)	4.71	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11050	40.4	<LLOQ(2.09)	4.11	6.39	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11051	26.7	<LLOQ(2.09)	<LLOQ(2.11)	3.11	<LLOQ(1.40)	<LLOQ(3.20)	2.51
11052	44.3	<LLOQ(2.09)	3.41	8.78	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11053	44.8	<LLOQ(2.09)	8.70	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.96
11054	49.6	3.03	5.27	2.99	<LLOQ(1.40)	<LLOQ(3.20)	2.02
11055	27.5	2.39	3.32	3.13	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11056	27.0	<LLOQ(2.09)	6.01	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11057	51.9	<LLOQ(2.09)	7.40	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11058	53.2	<LLOQ(2.09)	5.78	6.47	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11059	48.7	<LLOQ(2.09)	6.82	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.38
11060	36.4	<LLOQ(2.09)	4.67	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	4.14
11061	18.3	<LLOQ(2.09)	2.34	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11062	58.1	<LLOQ(2.09)	20.2	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.93
11063	25.8	<LLOQ(2.09)	5.08	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11064	56.0	2.96	9.41	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.29
11065	56.5	2.91	3.86	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11066	30.1	2.27	3.69	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11067	31.5	8.26	8.37	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11068	6.63	<LLOQ(2.09)	<LLOQ(2.11)	3.23	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11069	27.6	<LLOQ(2.09)	5.53	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.89
11070	205	2.63	34.1	5.28	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11071	27.6	2.73	4.60	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.13
11072	49.4	3.74	14.1	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.35
11073	47.6	<LLOQ(2.09)	4.78	3.35	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11074	29.7	<LLOQ(2.09)	3.59	3.67	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11075	69.7	<LLOQ(2.09)	6.18	5.54	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11076	27.4	<LLOQ(2.09)	4.63	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11077	42.6	2.42	6.26	4.38	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11078	46.9	<LLOQ(2.09)	6.02	3.88	<LLOQ(1.40)	<LLOQ(3.20)	1.89
11079	32.3	5.27	5.79	3.44	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11080	43.4	<LLOQ(2.09)	8.73	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.46
10556	14.1	3.88	4.18	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.01

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10557	22.5	2.42	5.84	2.81	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10558	33.1	10.4	8.55	3.51	<LLOQ(1.40)	<LLOQ(3.20)	4.26
10559	35.4	12.6	3.55	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10560	16.9	<LLOQ(2.09)	3.37	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.47
10561	40.0	2.38	10.5	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.40
10562	25.3	2.09	4.54	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.60
10563	56.0	3.49	7.83	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10564	18.1	<LLOQ(2.09)	5.37	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10565	47.2	6.23	8.44	3.48	<LLOQ(1.40)	<LLOQ(3.20)	2.57
10566	29.5	3.55	10.0	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.64
10567	23.3	2.37	6.88	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.88
10568	13.6	<LLOQ(2.09)	2.21	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10569	18.5	2.41	3.43	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10570	19.9	<LLOQ(2.09)	3.53	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10571	20.7	2.81	3.77	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10572	26.0	5.16	4.30	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10573	30.6	2.10	6.29	4.07	<LLOQ(1.40)	<LLOQ(3.20)	2.31
10574	21.1	<LLOQ(2.09)	3.19	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10575	<LLOQ(4.27)	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10576	20.3	3.13	4.17	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10577	23.6	2.54	4.60	2.92	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10578	63.7	5.97	9.31	5.14	<LLOQ(1.40)	<LLOQ(3.20)	2.37
10579	37.4	2.28	7.36	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.16
10580	44.1	<LLOQ(2.09)	6.25	3.24	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10581	9.83	<LLOQ(2.09)	5.84	2.92	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10582	25.7	2.10	4.11	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10583	39.2	2.56	9.18	3.26	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10584	13.5	<LLOQ(2.09)	4.81	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.19
10585	37.6	3.67	7.23	4.84	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10586	32.1	2.71	3.87	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.89
10587	11.0	8.05	3.50	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10588	18.1	<LLOQ(2.09)	6.97	3.10	<LLOQ(1.40)	<LLOQ(3.20)	3.41
10589	37.2	4.96	6.73	4.40	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10590	52.6	8.80	11.8	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.84
10591	38.2	4.18	6.75	3.29	<LLOQ(1.40)	<LLOQ(3.20)	4.29
10592	51.7	11.2	13.6	2.98	<LLOQ(1.40)	<LLOQ(3.20)	19.0

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10593	30.9	6.75	6.20	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10594	74.2	10.2	13.9	6.71	<LLOQ(1.40)	<LLOQ(3.20)	2.11
10595	37.1	2.56	4.59	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10596	21.8	2.39	3.91	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.40
10597	14.3	<LLOQ(2.09)	3.36	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10598	29.9	2.89	9.69	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10599	18.0	2.51	4.10	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10600	47.7	3.16	7.19	3.58	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10601	43.5	<LLOQ(2.09)	3.04	3.13	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10602	20.8	3.20	5.09	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10603	23.5	<LLOQ(2.09)	5.23	3.35	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10604	10.2	<LLOQ(2.09)	2.67	3.24	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10605	25.2	<LLOQ(2.09)	3.49	3.01	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10606	27.6	<LLOQ(1.36)	7.16	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	3.78
10607	36.7	3.62	7.89	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	2.05
10608	30.8	1.72	8.13	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	1.93
10609	29.7	2.76	6.53	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10610	65.8	1.87	13.7	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10611	22.2	<LLOQ(1.36)	5.82	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10612	32.0	11.4	7.23	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10613	67.6	1.49	10.7	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10614	57.6	5.36	10.1	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	3.92
10615	36.0	<LLOQ(1.36)	4.01	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10616	27.7	<LLOQ(1.36)	5.76	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10617	25.8	3.31	5.24	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10619	22.7	2.16	4.40	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10620	54.3	6.80	9.52	2.42	<LLOQ(1.00)	<LLOQ(3.20)	13.0
10621	14.0	<LLOQ(1.36)	2.47	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10622	35.5	<LLOQ(1.36)	6.63	2.59	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10623	11.7	<LLOQ(1.36)	4.13	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10624	25.3	3.70	5.89	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10625	35.2	<LLOQ(1.36)	4.17	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	2.39
10626	41.7	<LLOQ(1.36)	7.28	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	3.36
10627	31.8	<LLOQ(1.36)	6.28	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	1.88
10628	25.4	2.84	6.87	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	4.42
10629	36.3	<LLOQ(1.36)	4.55	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10630	30.3	1.99	4.65	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10631	54.1	1.51	8.91	2.28	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10632	41.4	1.47	5.23	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	3.03
10633	87.2	<LLOQ(1.36)	5.48	2.35	<LLOQ(1.00)	<LLOQ(3.20)	2.65
10634	30.6	<LLOQ(1.36)	7.36	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10635	36.8	1.79	5.27	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10636	26.3	1.84	3.48	3.32	<LLOQ(1.00)	<LLOQ(3.20)	2.67
10637	12.7	<LLOQ(1.36)	3.52	1.61	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10638	45.2	2.80	8.02	1.62	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10639	44.7	<LLOQ(1.36)	9.13	4.68	<LLOQ(1.00)	<LLOQ(3.20)	2.12
10640	19.3	2.36	4.73	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10641	19.8	<LLOQ(1.36)	3.07	1.78	<LLOQ(1.00)	<LLOQ(3.20)	1.83
10642	80.9	2.08	5.93	2.99	<LLOQ(1.00)	<LLOQ(3.20)	3.65
10643	40.5	2.29	7.21	2.45	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10644	23.9	3.45	7.29	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	2.41
10645	26.5	<LLOQ(1.36)	5.04	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10646	48.7	1.60	10.5	3.00	<LLOQ(1.00)	<LLOQ(3.20)	3.48
10647	26.7	1.40	7.51	3.34	<LLOQ(1.00)	<LLOQ(3.20)	6.35

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10648	16.7	<LLOQ(1.36)	3.49	4.28	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10649	39.0	1.58	10.1	4.64	<LLOQ(1.00)	<LLOQ(3.20)	2.95
10650	14.3	<LLOQ(1.36)	2.86	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10651	28.6	1.36	5.69	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	1.84
10652	32.7	2.12	11.4	2.81	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10653	11.7	<LLOQ(1.36)	2.13	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	2.51
10654	28.5	2.19	11.4	2.43	<LLOQ(1.00)	<LLOQ(3.20)	9.66
10655	15.9	2.64	4.26	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	2.83
10656	33.9	2.23	2.14	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(3.20)	<LLOQ(1.80)
10657	48.1	3.02	3.46	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10658	45.7	3.79	5.80	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10659	18.7	2.94	4.57	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10660	26.1	6.70	4.58	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10661	28.6	2.68	5.56	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	4.30
10662	44.5	<LLOQ(2.09)	6.28	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10663	26.9	2.85	2.80	5.09	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10664	31.4	<LLOQ(2.09)	3.28	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10665	44.9	2.57	5.48	4.40	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10666	29.9	<LLOQ(2.09)	4.65	4.52	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10667	40.8	<LLOQ(2.09)	4.41	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10668	19.8	<LLOQ(2.09)	4.30	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10328	24.3	<LLOQ(1.36)	4.90	2.59	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10329	50.6	3.86	7.92	4.83	<LLOQ(1.00)	<LLOQ(2.50)	1.95
10330	47.7	3.50	18.7	5.56	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10331	31.7	8.44	5.39	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10332	30.8	5.15	4.08	1.87	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10333	31.9	1.65	4.05	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.76
10334	24.6	<LLOQ(1.36)	3.23	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10335	19.3	<LLOQ(1.36)	3.20	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10336	42.5	<LLOQ(1.36)	3.62	2.22	<LLOQ(1.00)	<LLOQ(2.50)	1.21
10337	32.1	1.52	5.76	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	6.28
10338	19.3	<LLOQ(1.36)	2.46	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	3.92
10339	28.4	<LLOQ(1.36)	4.59	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10340	39.2	<LLOQ(1.36)	5.02	4.66	<LLOQ(1.00)	<LLOQ(2.50)	1.35
10341	14.8	<LLOQ(1.36)	3.84	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10342	23.1	4.89	8.59	2.17	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10343	29.6	<LLOQ(1.36)	6.45	3.42	<LLOQ(1.00)	<LLOQ(2.50)	1.50
10344	44.9	<LLOQ(1.36)	3.03	4.92	<LLOQ(1.00)	<LLOQ(2.50)	2.51
10345	45.6	2.07	5.74	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10346	39.8	2.44	4.38	1.83	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10347	14.7	<LLOQ(1.36)	2.28	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10348	17.7	<LLOQ(1.36)	3.58	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.83
10349	33.0	<LLOQ(1.36)	4.94	2.05	<LLOQ(1.00)	<LLOQ(2.50)	1.39
10350	30.5	2.58	2.35	4.32	<LLOQ(1.00)	<LLOQ(2.50)	5.37
10351	34.8	6.40	3.95	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	3.17
10352	18.0	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10353	23.8	2.02	6.55	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10354	31.6	3.17	4.74	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10355	95.7	1.43	10.7	2.38	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10356	26.5	<LLOQ(1.36)	3.80	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10357	22.0	<LLOQ(1.36)	4.05	1.84	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10358	43.5	4.08	4.92	3.40	<LLOQ(1.00)	<LLOQ(2.50)	1.51
10359	71.0	1.37	5.30	1.96	<LLOQ(1.00)	<LLOQ(2.50)	1.03
10360	17.0	<LLOQ(1.36)	4.90	1.79	<LLOQ(1.00)	<LLOQ(2.50)	1.28

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10361	7.70	<LLOQ(1.36)	2.53	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10362	41.7	3.02	5.90	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	3.01
10363	34.7	1.41	4.93	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.59
10364	22.8	<LLOQ(1.36)	3.92	1.83	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10365	22.6	<LLOQ(1.36)	3.07	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.85
10366	21.9	<LLOQ(1.36)	4.96	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10367	17.7	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10368	74.3	<LLOQ(1.36)	9.98	10.3	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10369	18.1	<LLOQ(1.36)	3.53	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	3.02
10370	24.7	<LLOQ(1.36)	3.98	2.75	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10371	71.8	7.12	7.18	5.41	<LLOQ(1.00)	<LLOQ(2.50)	3.89
10372	59.5	12.0	5.03	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.62
10373	52.8	3.87	20.0	1.79	<LLOQ(1.00)	<LLOQ(2.50)	10.6
10374	29.7	<LLOQ(1.36)	4.85	1.94	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10375	49.2	10.8	4.40	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.20
10376	21.1	<LLOQ(1.36)	3.54	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.62
10377	88.4	6.67	15.8	7.53	<LLOQ(1.00)	<LLOQ(2.50)	2.31
10378	100	1.37	5.57	4.93	<LLOQ(1.00)	<LLOQ(2.50)	1.52

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10379	54.6	<LLOQ(1.36)	3.88	3.27	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10380	31.3	1.49	4.22	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.72
10381	34.8	<LLOQ(1.36)	3.92	2.56	<LLOQ(1.00)	<LLOQ(2.50)	1.69
10382	35.8	1.72	2.81	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.70
10383	15.6	<LLOQ(1.36)	2.80	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.84
10384	24.7	<LLOQ(1.36)	2.56	2.34	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10385	28.3	2.03	4.89	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.40
10386	41.4	5.75	3.30	8.31	<LLOQ(1.00)	<LLOQ(2.50)	1.33
10387	11.7	<LLOQ(1.36)	2.43	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10388	79.1	<LLOQ(2.09)	5.93	4.38	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10389	28.4	1.60	4.29	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.29
10390	15.8	<LLOQ(1.36)	3.09	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.42
10391	48.4	2.45	7.41	3.27	<LLOQ(1.00)	<LLOQ(2.50)	3.07
10392	55.5	1.61	5.34	2.54	<LLOQ(1.00)	<LLOQ(2.50)	4.03
10393	20.9	1.41	2.91	1.98	<LLOQ(1.00)	<LLOQ(2.50)	3.12
10394	21.7	1.65	2.18	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10395	40.0	4.73	4.92	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.41
10396	36.3	1.81	5.66	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.32

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10397	28.2	6.51	4.57	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.62
10398	36.9	5.10	5.59	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	4.39
10399	38.9	5.63	3.38	2.00	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10400	27.5	<LLOQ(1.36)	4.57	1.72	<LLOQ(1.00)	<LLOQ(2.50)	1.34
10401	32.0	2.79	5.63	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10402	25.2	<LLOQ(1.36)	<LLOQ(1.92)	2.12	<LLOQ(1.00)	<LLOQ(2.50)	4.05
10403	34.3	3.92	2.97	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10404	30.6	3.56	2.78	4.71	<LLOQ(1.00)	<LLOQ(2.50)	1.34
10405	36.5	1.69	7.05	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10406	24.1	6.87	6.27	1.78	<LLOQ(1.00)	<LLOQ(2.50)	2.03
10407	27.0	<LLOQ(1.36)	4.62	2.44	<LLOQ(1.00)	<LLOQ(2.50)	6.76
10408	80.4	1.90	10.6	2.83	<LLOQ(1.00)	<LLOQ(2.50)	1.81
10409	28.8	1.61	3.49	1.75	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10410	88.8	15.2	13.8	3.43	<LLOQ(1.00)	<LLOQ(2.50)	4.60
10411	32.3	<LLOQ(1.36)	3.26	1.68	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10412	53.5	1.87	8.98	7.34	<LLOQ(1.00)	<LLOQ(2.50)	3.45
10413	16.8	<LLOQ(1.36)	2.53	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10414	27.0	<LLOQ(1.36)	3.17	2.57	<LLOQ(1.00)	<LLOQ(2.50)	1.42

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10415	207	2.09	14.9	7.76	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10416	17.2	<LLOQ(1.36)	<LLOQ(1.92)	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10417	37.7	4.54	4.28	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10418	29.1	<LLOQ(1.36)	4.43	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10419	30.6	<LLOQ(1.36)	2.82	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10420	56.6	2.70	11.5	2.58	<LLOQ(1.00)	<LLOQ(2.50)	5.07
10421	33.6	1.83	4.61	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.63
10422	14.4	<LLOQ(1.36)	2.40	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.78
10423	36.7	<LLOQ(1.36)	5.76	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10424	38.8	1.39	3.38	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	3.20
10425	30.8	<LLOQ(1.36)	3.59	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	7.63
10426	115	<LLOQ(1.36)	10.3	12.9	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10427	57.7	<LLOQ(1.36)	5.95	1.91	<LLOQ(1.00)	<LLOQ(2.50)	2.21
11883	17.8	<LLOQ(2.09)	2.40	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11884	26.1	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11885	26.5	<LLOQ(2.09)	6.13	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11886	26.6	<LLOQ(2.09)	4.07	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11887	60.1	<LLOQ(2.09)	26.7	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.93

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11888	91.8	7.34	10.1	3.64	<LLOQ(1.40)	<LLOQ(3.20)	13.3
11889	33.8	<LLOQ(2.09)	5.85	3.43	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11890	23.2	<LLOQ(2.09)	3.64	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11891	71.3	<LLOQ(2.09)	6.40	4.04	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11892	30.2	3.60	7.40	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11893	35.0	<LLOQ(2.09)	4.90	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.90
11894	22.0	<LLOQ(2.09)	4.55	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11895	12.5	<LLOQ(2.09)	4.48	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11896	34.2	<LLOQ(2.09)	4.71	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10756	166	4.51	29.0	60.1	<LLOQ(1.00)	12.9	3.75
10757	77.9	4.27	11.7	52.9	<LLOQ(1.00)	9.21	<LLOQ(1.00)
10758	133	11.6	6.25	7.18	<LLOQ(1.00)	<LLOQ(2.50)	5.21
10759	160	2.78	15.3	8.11	<LLOQ(1.00)	<LLOQ(2.50)	1.28
10760	38.1	1.71	5.39	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.55
10761	90.5	1.50	8.54	12.2	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10762	41.7	<LLOQ(1.36)	6.09	1.80	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10763	36.4	<LLOQ(1.36)	6.25	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10764	30.3	<LLOQ(1.36)	3.29	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10765	96.1	3.73	11.7	1.75	<LLOQ(1.00)	<LLOQ(2.50)	2.82
10766	40.7	7.70	6.54	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10767	34.1	<LLOQ(1.36)	6.81	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.65
10768	28.1	4.56	5.96	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10769	54.7	4.64	6.69	2.19	<LLOQ(1.00)	<LLOQ(2.50)	1.18
10770	42.7	<LLOQ(1.36)	4.14	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.59
10771	26.8	1.57	4.70	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	4.22
10772	36.1	2.00	4.34	3.44	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10773	66.6	15.6	5.78	2.89	<LLOQ(1.00)	<LLOQ(2.50)	2.77
10774	77.2	2.25	8.75	8.08	<LLOQ(1.00)	<LLOQ(2.50)	1.86
10775	46.8	<LLOQ(1.36)	9.30	8.98	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10776	159	4.24	29.8	56.6	<LLOQ(1.00)	12.2	2.45
10777	49.1	2.76	6.08	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10778	31.0	<LLOQ(1.36)	2.21	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	2.07
10779	22.7	<LLOQ(1.36)	5.12	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10780	57.3	3.76	7.72	3.56	<LLOQ(1.00)	<LLOQ(2.50)	1.73
10781	43.9	2.78	6.04	1.68	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10782	79.0	4.71	9.85	3.38	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10783	54.2	1.78	7.46	4.60	<LLOQ(1.00)	<LLOQ(2.50)	2.05
10784	37.3	2.12	5.89	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10785	31.6	1.91	4.88	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10786	38.9	5.22	4.79	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.98
10787	73.0	6.27	18.2	2.28	<LLOQ(1.00)	<LLOQ(2.50)	2.16
10788	33.8	2.19	5.34	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10789	49.3	2.00	7.30	1.95	<LLOQ(1.00)	<LLOQ(2.50)	1.61
10790	55.2	<LLOQ(1.36)	6.45	4.51	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10791	69.8	2.67	7.16	2.36	<LLOQ(1.00)	<LLOQ(2.50)	1.57
10792	22.0	1.38	4.02	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.21
10793	31.1	1.64	5.08	2.31	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10794	74.2	3.86	10.6	13.7	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10795	105	2.09	5.51	1.96	<LLOQ(1.00)	<LLOQ(2.50)	2.67
10796	40.2	19.8	8.76	2.09	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10797	52.5	4.12	4.32	1.76	<LLOQ(1.00)	<LLOQ(2.50)	4.69
10798	58.2	4.96	7.87	1.81	<LLOQ(1.00)	<LLOQ(2.50)	1.97
10799	117	1.70	13.1	5.44	<LLOQ(1.00)	<LLOQ(2.50)	1.27
10800	75.1	3.15	8.95	8.62	<LLOQ(1.00)	<LLOQ(2.50)	2.97

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10801	48.7	2.48	6.21	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.22
10802	33.3	<LLOQ(1.36)	4.47	6.91	<LLOQ(1.00)	<LLOQ(2.50)	2.16
10803	32.6	8.59	4.31	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10804	68.5	<LLOQ(1.36)	17.8	3.71	<LLOQ(1.00)	<LLOQ(2.50)	3.03
10805	45.5	4.35	6.93	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10806	110	11.5	21.8	3.35	<LLOQ(1.00)	<LLOQ(2.50)	5.59
10807	33.9	3.52	7.08	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	1.68
10808	40.3	1.86	2.70	<LLOQ(1.60)	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10809	29.8	1.79	4.72	3.58	<LLOQ(1.00)	<LLOQ(2.50)	<LLOQ(1.00)
10810	42.9	<LLOQ(2.09)	3.54	3.58	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10811	32.6	<LLOQ(2.09)	4.45	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.99
10812	69.9	3.77	7.50	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.46
10813	27.4	<LLOQ(2.09)	2.85	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.95
10814	38.4	2.82	6.57	6.45	<LLOQ(1.40)	<LLOQ(3.20)	3.66
10815	55.1	<LLOQ(2.09)	8.35	4.05	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10816	50.1	4.39	2.60	3.04	<LLOQ(1.40)	<LLOQ(3.20)	5.68
10817	106	4.58	7.17	3.25	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10818	67.6	9.17	11.4	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.34

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10819	37.1	<LLOQ(2.09)	4.65	3.21	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10820	71.0	2.96	3.88	4.70	<LLOQ(1.40)	<LLOQ(3.20)	2.09
10821	45.3	<LLOQ(2.09)	4.17	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10822	54.4	10.7	7.46	15.1	<LLOQ(1.40)	<LLOQ(3.20)	4.37
10823	35.9	<LLOQ(2.09)	7.14	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.51
10824	109	<LLOQ(2.09)	6.50	9.12	<LLOQ(1.40)	3.91	4.18
10825	72.7	14.1	6.90	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10826	30.6	2.47	3.42	3.76	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10827	59.4	5.37	8.35	6.62	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10828	143	3.33	13.4	8.59	<LLOQ(1.40)	<LLOQ(3.20)	4.03
10829	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received
10830	34.3	4.97	5.75	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	8.31
10831	75.8	9.32	13.5	4.28	<LLOQ(1.40)	<LLOQ(3.20)	5.26
10832	44.5	4.10	4.87	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10833	68.9	3.29	10.7	3.51	<LLOQ(1.40)	<LLOQ(3.20)	10.8
10834	49.8	5.53	3.62	5.10	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10835	71.3	4.02	10.7	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	4.47
10836	35.5	3.82	4.66	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10837	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received
10838	36.5	7.12	8.60	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.75
10839	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received
10840	34.1	2.50	6.97	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.83
10841	68.2	22.4	5.68	4.33	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10842	154	12.1	18.2	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10843	41.8	2.34	4.37	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	8.20
10844	70.7	9.59	14.9	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	6.61
10845	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received
10846	65.8	18.7	13.3	2.85	<LLOQ(1.40)	<LLOQ(3.20)	2.83
10847	92.4	12.9	9.46	4.10	<LLOQ(1.40)	<LLOQ(3.20)	3.57
10848	44.2	<LLOQ(2.09)	3.27	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10849	51.5	2.98	7.02	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.99
10850	40.7	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10851	70.5	4.00	6.49	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.11
10852	19.3	<LLOQ(2.09)	2.95	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10853	31.7	<LLOQ(2.09)	4.70	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.44
10854	50.5	2.46	3.97	3.65	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
10855	18.5	<LLOQ(2.09)	2.16	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.53
10856	36.3	7.00	4.66	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10857	81.9	2.48	12.0	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10858	54.7	<LLOQ(2.09)	4.82	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10859	19.5	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10860	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received	Not Received
10861	39.9	2.57	3.35	5.15	<LLOQ(1.40)	<LLOQ(3.20)	3.21
10862	37.8	4.03	4.23	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.23
10863	70.6	<LLOQ(2.09)	4.20	3.11	<LLOQ(1.40)	<LLOQ(3.20)	3.10
10864	58.3	2.34	3.80	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10865	56.8	6.25	3.85	24.7	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
10866	24.0	<LLOQ(2.09)	3.03	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.35
10867	57.4	2.28	5.32	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.95
10868	22.9	4.80	3.46	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.79
11649	17.6	<LLOQ(2.09)	3.96	3.20	<LLOQ(1.40)	3.88	<LLOQ(1.80)
11650	28.3	<LLOQ(2.09)	3.49	3.24	<LLOQ(1.40)	<LLOQ(3.20)	2.06
11651	48.8	<LLOQ(2.09)	4.07	25.1	<LLOQ(1.40)	5.12	<LLOQ(1.80)
11652	23.5	<LLOQ(2.09)	2.71	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11653	25.2	<LLOQ(2.09)	4.76	3.89	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11654	329	6.91	16.7	36.9	<LLOQ(1.40)	12.2	<LLOQ(1.80)
11655	31.2	<LLOQ(2.09)	3.77	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11656	31.5	<LLOQ(2.09)	3.13	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.96
11657	70.3	<LLOQ(2.09)	6.63	13.3	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11658	27.4	2.54	2.94	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11659	43.9	11.8	3.61	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.43
11660	17.7	<LLOQ(2.09)	3.13	3.42	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11661	46.4	<LLOQ(2.09)	3.91	4.90	<LLOQ(1.40)	<LLOQ(3.20)	2.63
11662	44.6	10.9	4.92	2.82	<LLOQ(1.40)	<LLOQ(3.20)	2.06
11663	14.0	<LLOQ(2.09)	3.77	4.98	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11664	64.1	2.48	6.96	9.91	<LLOQ(1.40)	4.21	2.03
11665	45.0	<LLOQ(2.09)	4.49	5.04	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11666	37.7	<LLOQ(2.09)	5.07	2.83	<LLOQ(1.40)	<LLOQ(3.20)	2.86
11667	9.45	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11668	29.9	<LLOQ(2.09)	3.81	5.86	<LLOQ(1.40)	<LLOQ(3.20)	1.82
11669	31.9	<LLOQ(2.09)	6.28	3.77	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11670	28.3	<LLOQ(2.09)	4.08	2.92	<LLOQ(1.40)	<LLOQ(3.20)	3.64

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11671	37.7	2.16	4.44	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11672	30.7	<LLOQ(2.09)	5.43	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11673	36.2	<LLOQ(2.09)	2.68	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.37
11674	37.5	3.91	4.44	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11675	14.2	3.99	3.22	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11676	12.0	<LLOQ(2.09)	2.33	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11677	43.4	3.22	3.60	3.39	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11678	32.2	3.83	3.57	2.96	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11679	17.0	<LLOQ(2.09)	2.56	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11680	44.5	7.24	6.79	5.66	<LLOQ(1.40)	<LLOQ(3.20)	2.65
11681	15.8	<LLOQ(2.09)	5.41	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11682	9.20	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.62
11683	40.3	8.61	6.21	4.72	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11684	42.3	<LLOQ(2.09)	4.48	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11685	24.4	<LLOQ(2.09)	2.34	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11686	14.6	4.12	5.38	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11687	36.1	2.11	4.10	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11688	17.2	<LLOQ(2.09)	<LLOQ(2.11)	4.43	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11689	1656**	9.87	4.32	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11690	25.6	<LLOQ(2.09)	2.92	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11691	24.4	2.59	3.13	3.88	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11692	14.2	<LLOQ(2.09)	2.71	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11693	27.1	2.58	3.91	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.14
11694	33.4	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11695	20.8	<LLOQ(2.09)	3.04	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11696	14.5	<LLOQ(2.09)	<LLOQ(2.11)	3.03	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11697	108	<LLOQ(2.09)	12.2	13.0	<LLOQ(1.40)	<LLOQ(3.20)	2.07
11698	124	<LLOQ(2.09)	12.0	9.58	<LLOQ(1.40)	3.58	<LLOQ(1.80)
11699	22.9	2.28	4.24	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11700	25.5	<LLOQ(2.09)	3.48	5.45	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11701	14.1	<LLOQ(2.09)	3.11	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11702	39.0	3.27	5.61	6.74	<LLOQ(1.40)	<LLOQ(3.20)	3.70
11703	12.0	<LLOQ(2.09)	<LLOQ(2.11)	2.97	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11704	78.7	6.54	12.9	15.6	<LLOQ(1.40)	<LLOQ(3.20)	3.68
11705	12.0	<LLOQ(2.09)	3.09	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

** Concentration corrected for amount of persistent PFOS in dilution matrix.

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11706	22.3	<LLOQ(2.09)	3.35	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11707	17.5	<LLOQ(2.09)	3.87	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.96
11708	14.5	<LLOQ(2.09)	2.43	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11709	43.5	<LLOQ(2.09)	5.13	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	4.82
11710	35.9	3.37	2.90	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11711	30.3	<LLOQ(2.09)	3.45	3.48	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11712	11.1	<LLOQ(2.09)	3.39	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11713	50.8	3.76	11.2	20.6	<LLOQ(1.40)	<LLOQ(3.20)	5.27
11714	18.4	<LLOQ(2.09)	3.80	6.13	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11715	36.8	<LLOQ(2.09)	4.04	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11716	12.6	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11717	13.2	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11718	17.6	2.25	5.15	2.63	<LLOQ(1.40)	<LLOQ(3.20)	1.55
11719	16.1	<LLOQ(2.09)	3.22	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	5.50
11720	25.2	<LLOQ(2.09)	2.11	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11721	37.4	2.26	4.13	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11722	8.26	<LLOQ(2.09)	2.53	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.95
11723	30.2	5.64	4.19	3.21	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11725	16.6	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11726	20.2	<LLOQ(2.09)	2.78	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11727	25.7	<LLOQ(2.09)	8.48	2.90	<LLOQ(1.40)	<LLOQ(3.20)	2.68
11729	21.6	<LLOQ(2.09)	4.08	7.58	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11731	63.4	4.77	10.2	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.10
11732	30.3	<LLOQ(2.09)	4.07	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.85
11735	20.5	<LLOQ(2.09)	3.05	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11739	54.6	9.42	5.41	5.86	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11745	26.9	<LLOQ(2.09)	4.97	4.48	<LLOQ(1.40)	<LLOQ(3.20)	2.06
11746	24.6	<LLOQ(2.09)	3.76	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11748	31.1	<LLOQ(2.09)	3.31	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11749	17.6	<LLOQ(2.09)	4.36	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.94
11750	20.8	5.48	4.37	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11751	6.60	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.85
11752	45.3	<LLOQ(2.09)	4.09	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.05
11753	29.2	3.61	2.28	2.95	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11754	37.8	<LLOQ(2.09)	3.66	7.38	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11756	36.4	4.53	8.37	3.59	<LLOQ(1.40)	<LLOQ(3.20)	6.89

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11757	34.1	6.63	3.58	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.02
11758	24.8	<LLOQ(2.09)	4.65	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.22
11759	17.9	<LLOQ(2.09)	4.20	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11761	17.2	<LLOQ(2.09)	<LLOQ(2.11)	3.20	<LLOQ(1.40)	<LLOQ(3.20)	2.59
11762	20.0	<LLOQ(2.09)	2.82	3.55	<LLOQ(1.40)	<LLOQ(3.20)	2.85
11763	28.3	<LLOQ(2.09)	6.68	4.33	<LLOQ(1.40)	<LLOQ(3.20)	2.94
11765	22.3	<LLOQ(2.09)	2.62	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	4.62
11766	49.1	<LLOQ(2.09)	4.74	7.58	<LLOQ(1.40)	<LLOQ(3.20)	2.93
11767	16.6	<LLOQ(2.09)	2.97	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11768	14.8	<LLOQ(2.09)	2.52	3.18	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11769	26.0	<LLOQ(2.09)	4.17	3.25	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11771	6.03	<LLOQ(2.09)	2.76	2.81	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11772	40.4	4.13	7.03	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.93
11773	9.26	<LLOQ(2.09)	<LLOQ(2.11)	2.87	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11156	44.3	<LLOQ(2.09)	6.33	3.11	<LLOQ(1.40)	6.60	3.84
11157	28.0	2.16	3.28	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11158	21.5	4.10	4.16	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11159	40.0	3.04	4.72	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11160	31.5	<LLOQ(2.09)	2.74	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11161	20.7	<LLOQ(2.09)	3.03	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11162	30.9	3.08	6.16	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.04
11163	56.8	2.40	4.63	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11164	76.4	4.42	9.41	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	4.60
11165	93.3	13.7	5.53	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11166	17.7	3.64	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11167	11.1	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11168	48.1	3.33	4.69	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11169	36.6	3.51	5.19	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11170	41.4	<LLOQ(2.09)	8.30	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11171	48.1	5.19	4.74	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.97
11172	50.3	15.3	5.31	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.96
11173	42.7	<LLOQ(2.09)	4.77	3.14	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11174	65.4	3.24	6.58	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.96
11175	52.6	<LLOQ(2.09)	5.50	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.37
11176	24.3	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11177	30.8	<LLOQ(2.09)	3.33	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11178	41.0	<LLOQ(2.09)	9.73	<LLOQ(2.80)	<LLOQ(1.40)	3.84	2.88
11179	35.2	<LLOQ(2.09)	3.68	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11180	28.3	<LLOQ(2.09)	3.44	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11181	92.8	12.4	4.16	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11182	50.0	25.7	14.2	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11183	37.4	5.94	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11184	48.8	9.40	5.68	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.97
11185	71.6	4.31	9.01	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11186	21.7	<LLOQ(2.09)	4.50	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11187	15.1	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11188	23.2	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11189	52.6	<LLOQ(2.09)	13.2	9.91	<LLOQ(1.40)	<LLOQ(3.20)	7.86
11190	81.9	<LLOQ(2.09)	6.91	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.99
11191	24.3	<LLOQ(2.09)	3.26	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11192	27.1	<LLOQ(2.09)	5.59	2.92	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11193	34.3	<LLOQ(2.09)	7.09	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11194	17.1	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11195	45.6	<LLOQ(2.09)	7.49	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11196	18.2	<LLOQ(2.09)	4.69	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11197	21.4	<LLOQ(2.09)	3.76	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.07
11198	38.2	11.1	3.02	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11199	28.8	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11200	26.2	<LLOQ(2.09)	2.87	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11201	24.1	4.20	3.00	<LLOQ(2.80)	2.09	<LLOQ(3.20)	<LLOQ(1.80)
11202	42.1	10.7	6.93	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11203	25.2	2.79	3.45	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11204	31.1	3.27	5.55	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11205	12.8	2.26	2.47	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11206	24.3	<LLOQ(2.09)	6.67	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.96
11207	44.2	<LLOQ(2.09)	5.25	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11208	15.9	<LLOQ(2.09)	3.61	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11209	25.7	<LLOQ(2.09)	4.69	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11210	29.3	4.41	4.32	4.64	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11211	47.5	<LLOQ(2.09)	2.81	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11212	69.6	<LLOQ(2.09)	7.25	3.34	<LLOQ(1.40)	<LLOQ(3.20)	3.30
11213	51.9	<LLOQ(2.09)	5.10	3.98	<LLOQ(1.40)	<LLOQ(3.20)	2.10

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11214	36.4	6.25	3.57	4.34	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11215	122	<LLOQ(2.09)	5.93	3.21	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11216	42.5	<LLOQ(2.09)	5.22	2.98	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11217	41.4	<LLOQ(2.09)	6.32	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11218	57.9	2.91	6.89	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.01
11219	19.4	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11220	29.8	<LLOQ(2.09)	2.94	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	3.12
11221	46.0	<LLOQ(2.09)	6.44	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11222	26.5	19.2	2.73	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11223	41.5	5.21	5.80	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11224	64.9	66.3	4.30	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11225	29.9	<LLOQ(2.09)	4.50	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	4.02
11226	18.7	<LLOQ(2.09)	5.01	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11227	23.1	2.33	4.61	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11228	41.4	<LLOQ(2.09)	6.50	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11229	45.9	3.22	5.43	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.07
11230	85.2	2.45	7.01	4.60	<LLOQ(1.40)	<LLOQ(3.20)	2.57
11231	62.9	2.51	3.53	6.36	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11232	21.6	<LLOQ(2.09)	4.87	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	4.44
11233	30.0	<LLOQ(2.09)	3.10	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.15
11234	39.0	<LLOQ(2.09)	4.81	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.58
11235	22.0	2.23	3.39	3.18	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11236	26.7	<LLOQ(2.09)	4.13	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11237	80.0	<LLOQ(2.09)	9.10	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11238	37.8	<LLOQ(2.09)	4.97	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11239	24.7	2.64	4.41	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11240	37.2	<LLOQ(2.09)	3.29	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	6.15
11241	139	5.3	20.6	3.83	<LLOQ(1.40)	<LLOQ(3.20)	2.56
11242	65.9	9.47	6.87	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.88
11243	27.0	<LLOQ(2.09)	3.01	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11244	64.9	3.73	8.74	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11245	29.7	2.22	5.21	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.49
11246	28.2	<LLOQ(2.09)	2.44	3.26	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11247	19.0	<LLOQ(2.09)	3.14	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11248	36.2	5.00	5.70	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	2.17
11249	36.3	<LLOQ(2.09)	5.58	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11250	26.1	4.07	2.32	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11251	34.6	2.19	5.07	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11252	13.1	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11253	56.8	4.36	7.39	21.2	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11254	50.8	<LLOQ(2.09)	4.81	3.65	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11256	47.8	7.07	2.64	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11257	33.8	<LLOQ(2.09)	5.55	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11258	23.1	<LLOQ(2.09)	4.33	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11259	7.63	<LLOQ(2.09)	<LLOQ(2.11)	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11260	34.7	4.14	5.12	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11261	12.6	3.80	2.53	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11262	17.7	<LLOQ(2.09)	4.07	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11263	226	<LLOQ(2.09)	52.3	7.22	<LLOQ(1.40)	<LLOQ(3.20)	1.87
11264	42.2	5.11	3.54	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11265	24.7	2.45	5.69	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11266	27.2	<LLOQ(2.09)	3.47	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)
11267	36.8	11.3	2.84	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	1.86
11268	39.7	<LLOQ(2.09)	2.47	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample ID	PFOS Concentration (ppb)	PFHS* Concentration (ppb)	PFOA* Concentration (ppb)	PFOSAA* Concentration (ppb)	PFOSA* Concentration (ppb)	M556* Concentration (ppb)	M570* Concentration (ppb)
11269	25.2	3.67	2.54	<LLOQ(2.80)	<LLOQ(1.40)	<LLOQ(3.20)	<LLOQ(1.80)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 23. Repeat Analysis Table for PFOS

Sample ID	Original Conc. ppb	Original Run Number	Reason for Reassay	Reassay Conc. ppb	Reassay Run Number	Reported Conc. ppb	Reason for Reported Conc.
11689	>ULOQ(414)	10	1	1670	17	1656*	1

* Concentration corrected for amount of persistent PFOS in dilution matrix.

REASONS FOR REASSAY:
1). Greater than the ULOQ.

REASONS FOR REPORTED CONC:
1). Original result outside of quantitation range. Reassay results reported.

Figure 1. Representative Calibration Curve for PFOS

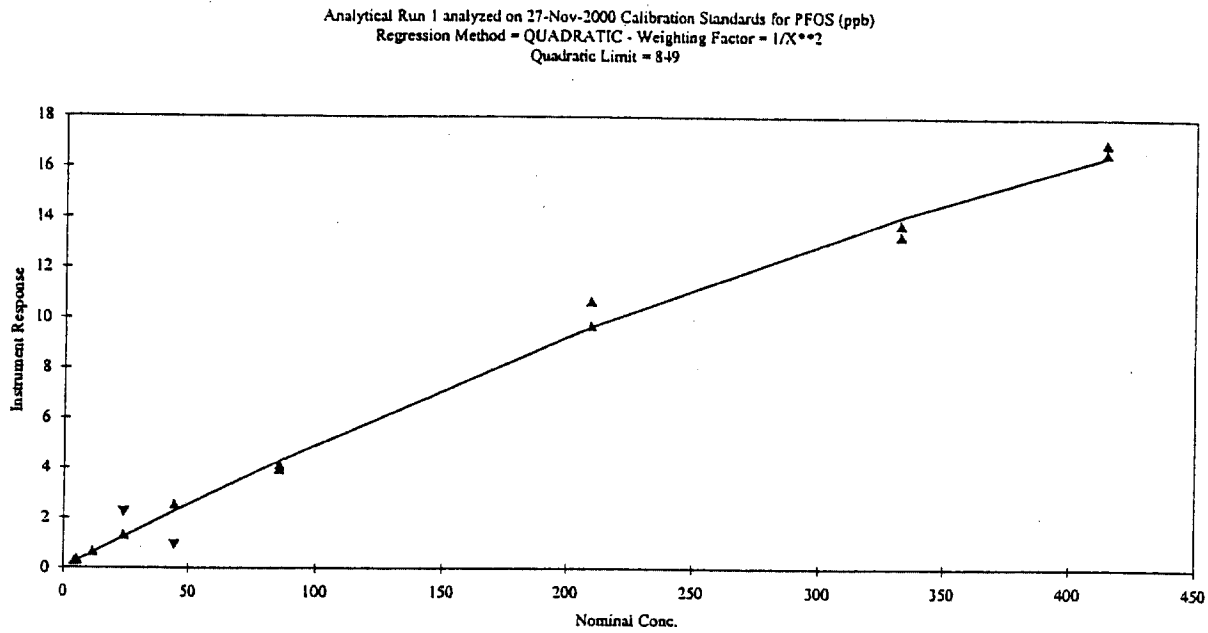
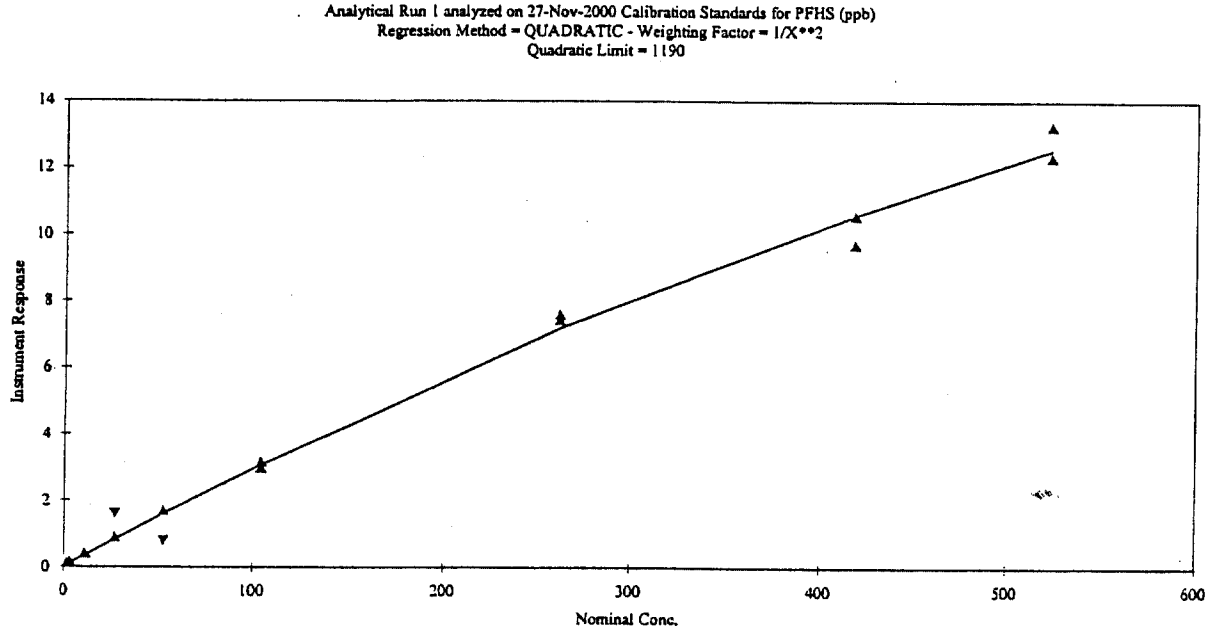


Figure 2. Representative Calibration Curve for PFHS



000567

Figure 3. Representative Calibration Curve for PFOA

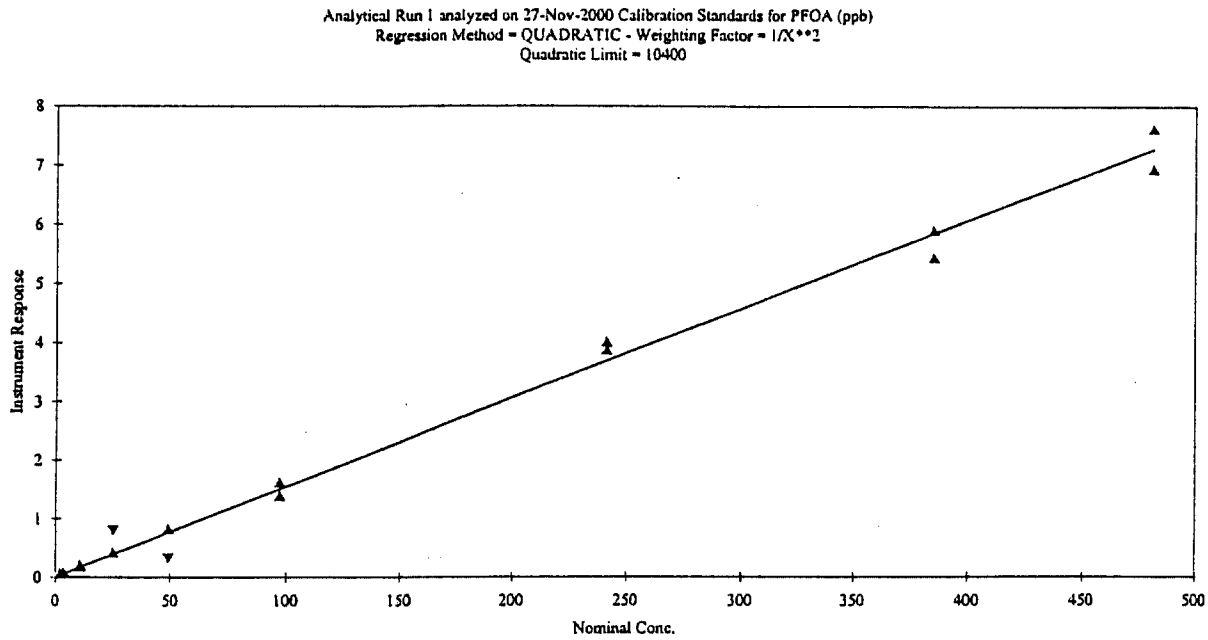


Figure 4. Representative Calibration Curve for PFOSAA

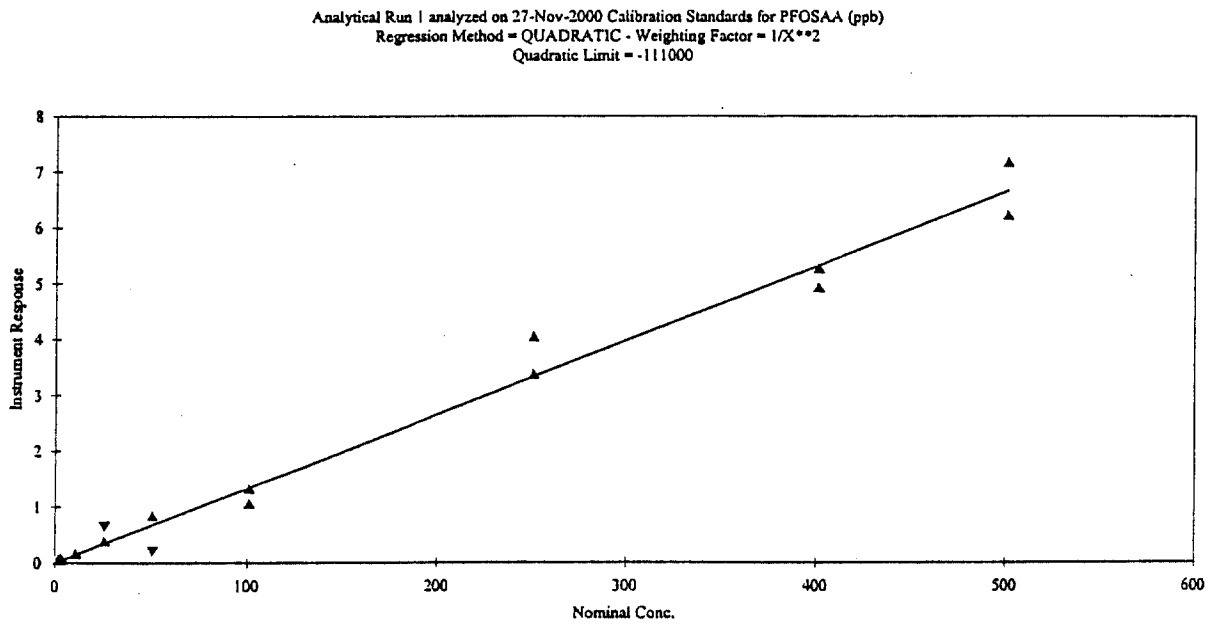


Figure 5. Representative Calibration Curve for PFOSA

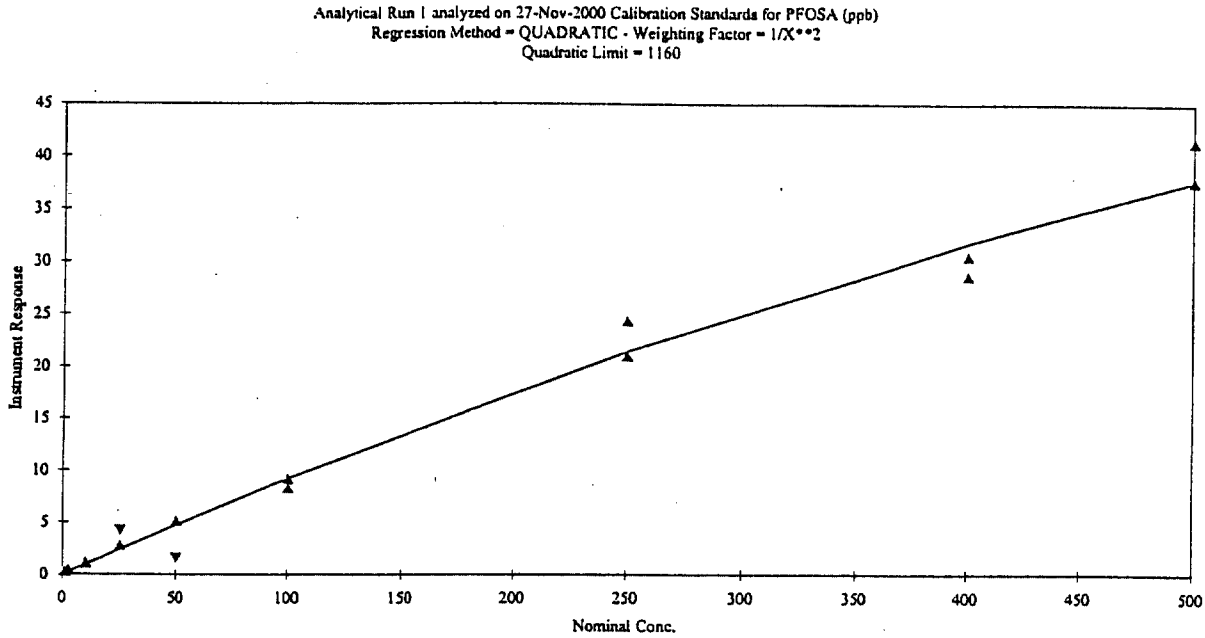
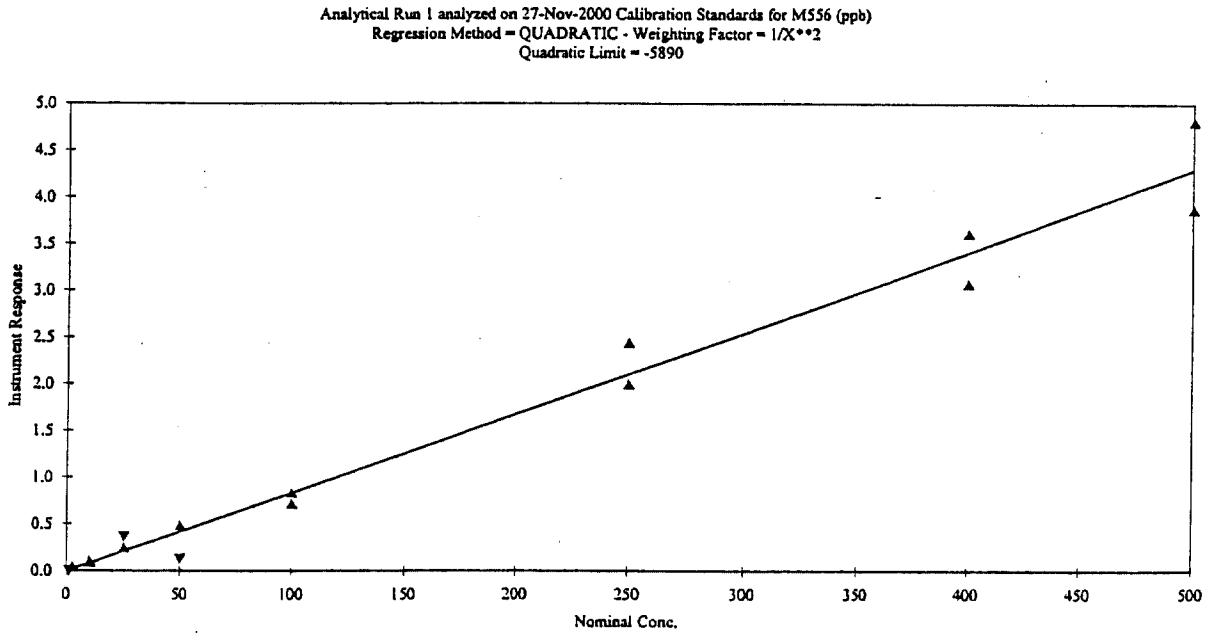
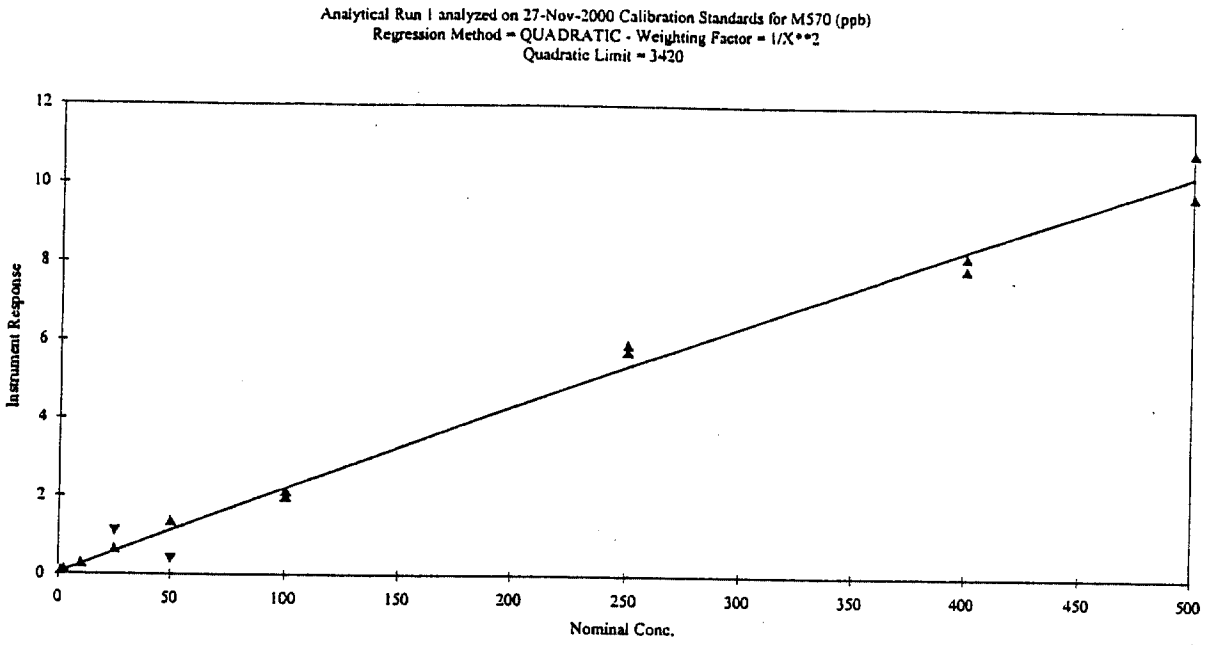


Figure 6. Representative Calibration Curve for M556



000569

Figure 7. Representative Calibration Curve for M570



000570

Figure 8. Human Plasma Blank for PFOS

PFOS		Current	Method
Internal Standard: THPFOS	Noise Thres.	5.0	5.0
Use Area	Quant Thres.	0.2	0.2
Absolute Retention Time	Min. Width	3	3
Expected RT 5.31	Mult. Width	12	12
	Base. Width	150	150
	RT Win. (secs)	20	20
	Smooth	1	1

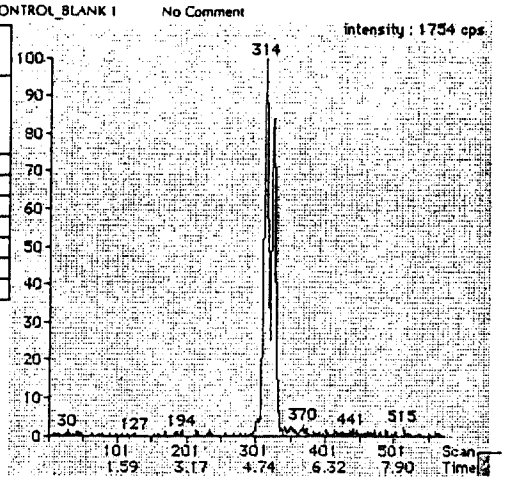
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

499.0->80.0

Area	0
Height	0
Start Time	0.00
End Time	0.00
Integration Width	0.00
Retention Time	0.00
Integration Type	



THPFOS		Current	Method
use as Internal Standard	Noise Thres.	10.0	10.0
	Quant Thres.	0.2	0.2
	Min. Width	3	3
	Mult. Width	10	10
Expected RT 4.63	Base. Width	150	150
	RT Win. (secs)	20	20
	Smooth	1	1

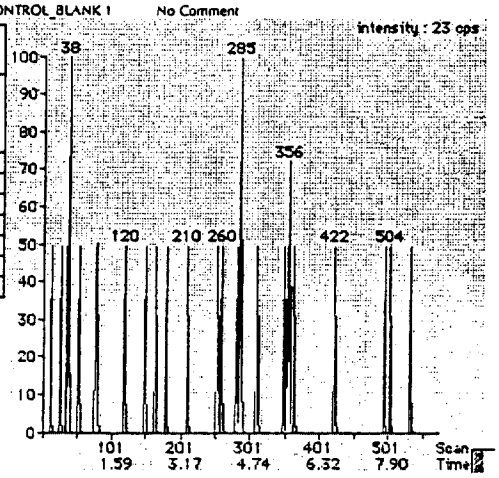
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0.00
End Time	0.00
Integration Width	0.00
Retention Time	0.00
Integration Type	



000571

Figure 9. Human Plasma Blank for PFHS

PFHS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4.00

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	4	4
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

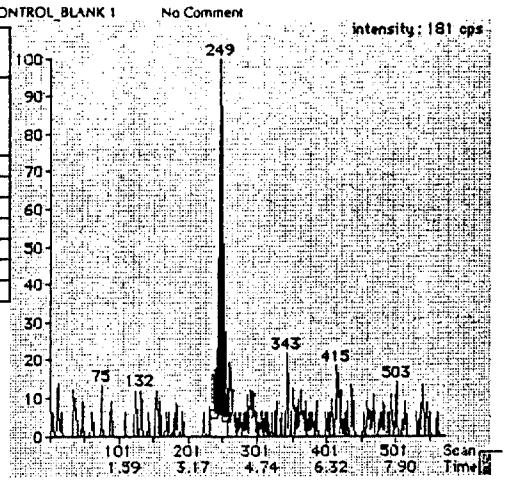
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

399.0->80.0

Area	1159
Height	171
Start Time	3.77
End Time	4.03
Integration Width	0.27
Retention Time	3.92
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0.00
End Time	0.00
Integration Width	0.00
Retention Time	0.00
Integration Type	

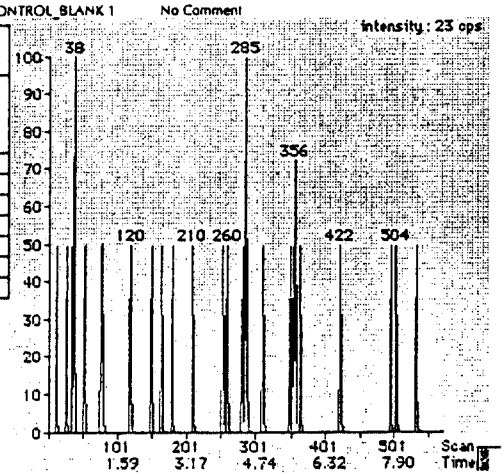


Figure 10. Human Plasma Blank for PFOA

POAA

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 4.59

	Current	Method
Noise Thres.	1.0	1.0
Quant Thres.	0.9	0.9
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

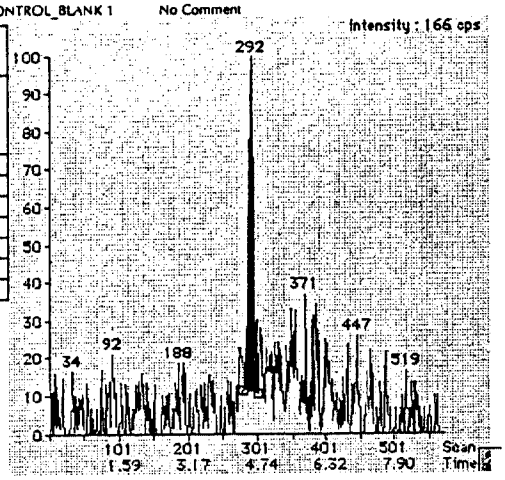
Intensity : 166 cps

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

413.0-> 169.0

Area	1104
Height	147
Start Time	4.44
End Time	4.74
Integration Width	0.30
Retention Time	4.60
Integration Type	A - BB



THPFOS

use as Internal Standard

Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

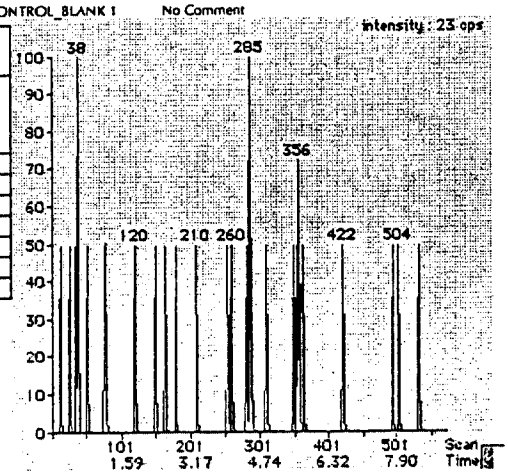
Intensity : 23 cps

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0-> 407.0

Area	0
Height	0
Start Time	0.00
End Time	0.00
Integration Width	0.00
Retention Time	0.00
Integration Type	



000573

Figure 11. Human Plasma Blank for PFOSAA

PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.93

	Current	Method
Noise Thres.	4.0	4.0
Quant Thres.	1.5	1.5
Min. Width	3	3
Mult. Width	13	13
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

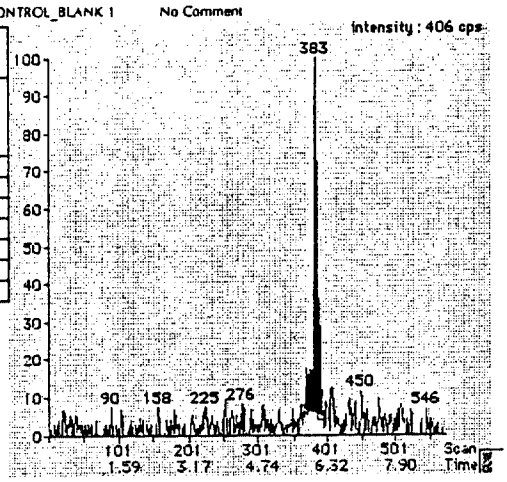
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

584.1->419.1

Area	2658
Height	383
Start Time	5.78
End Time	6.19
Integration Width	0.41
Retention Time	6.04
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

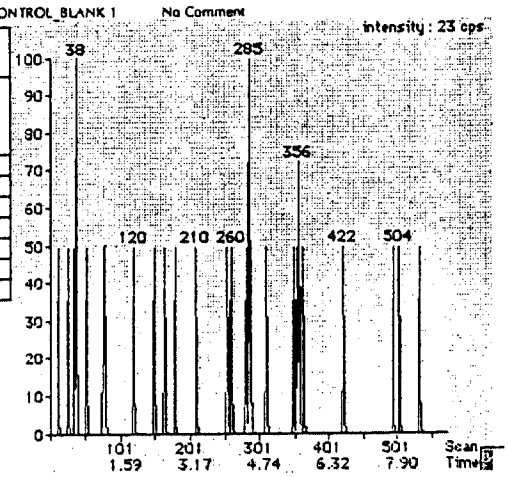
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0.00
End Time	0.00
Integration Width	0.00
Retention Time	0.00
Integration Type	



000574

Figure 12. Human Plasma Blank for PFOSA

PFOSA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.70

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	1.0	1.0
Min. Width	5	5
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

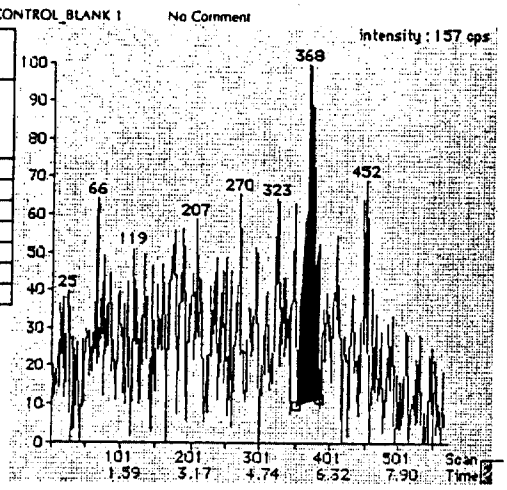
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

498.0-> 78.0

Area	2097
Height	140
Start Time	5.56
End Time	6.07
Integration Width	0.50
Retention Time	5.80
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0-> 407.0

Area	0
Height	0
Start Time	0.00
End Time	0.00
Integration Width	0.00
Retention Time	0.00
Integration Type	

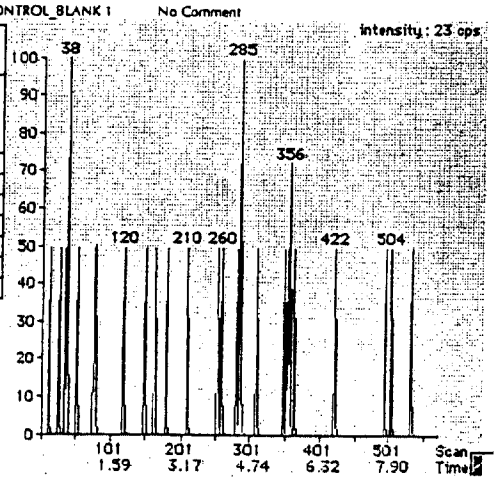


Figure 13. Human Plasma Blank for M556

M556

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5.37

	Current	Method
Noise Thres.	0.4	0.4
Quant Thres.	0.1	0.1
Min. Width	3	3
Mult. Width	11	11
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

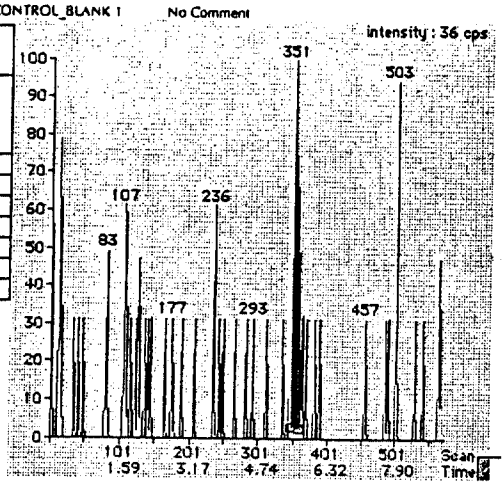
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

556.0->498.0

Area	187
Height	35
Start Time	5.50
End Time	5.66
Integration Width	0.16
Retention Time	5.53
Integration Type	A - BB



THPFOS

use as Internal Standard

Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

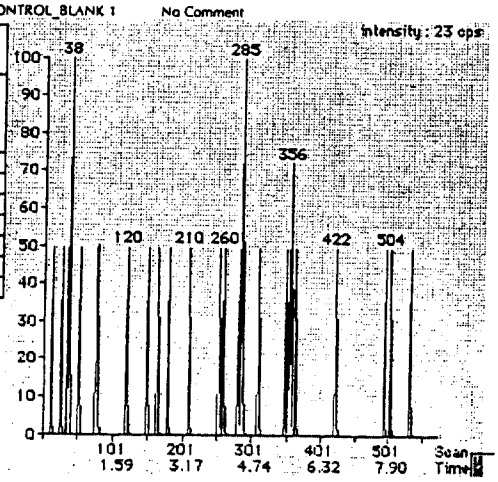
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0.00
End Time	0.00
Integration Width	0.00
Retention Time	0.00
Integration Type	



000576

Figure 14. Human Plasma Blank for M570

M570

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5.63

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	5	5
Mult. Width	15	15
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

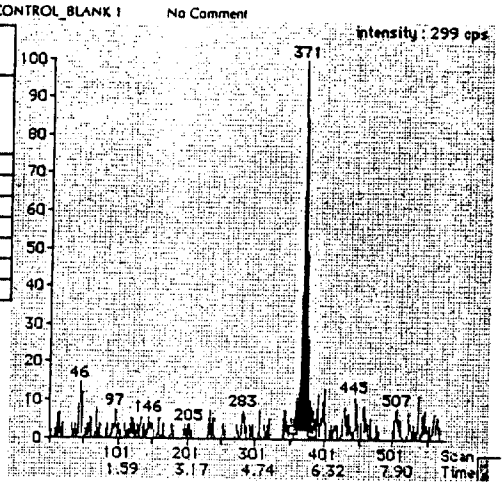
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

570.0->419.1

Area	1990
Height	292
Start Time	5.63
End Time	6.02
Integration Width	0.39
Retention Time	5.85
Integration Type	A - BB



THPFOS

use as Internal Standard

Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

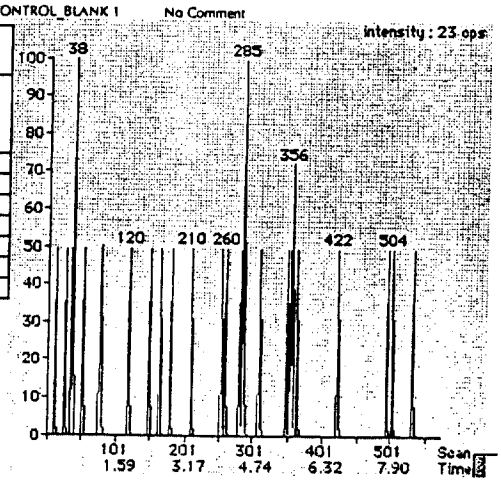
s0012801-011 1 011 S00128 CONTROL_BLANK 1 No Comment

Mon, Nov 27, 2000 7:20 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	0
Height	0
Start Time	0.00
End Time	0.00
Integration Width	0.00
Retention Time	0.00
Integration Type	



000577

Figure 15. Human Plasma Blank with Internal Standard (QC0) for PFOS

PFOS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.31

	Current	Method
Noise Thres.	5.0	5.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

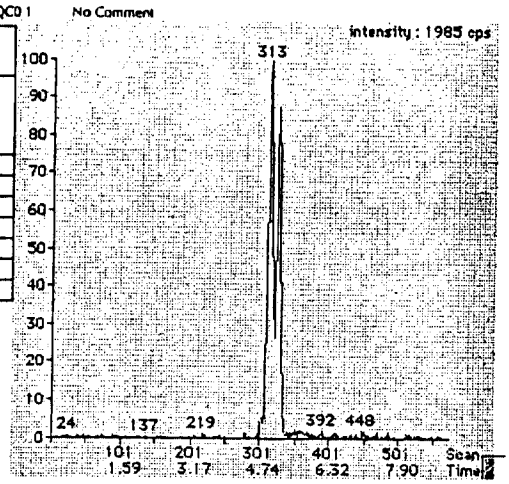
s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

499.0->80.0

Area	0
Height	0
Start Time	0.00
End Time	0.00
Integration Width	0.00
Retention Time	0.00
Integration Type	



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

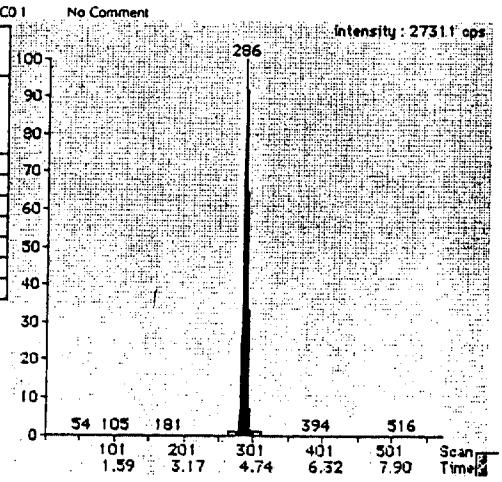
s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	185736
Height	27311
Start Time	4.30
End Time	4.84
Integration Width	0.54
Retention Time	4.51
Integration Type	A - BB



000578

Figure 16. Human Plasma Blank with Internal Standard (QC0) for PFHS

PFHS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4.00

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	4	4
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

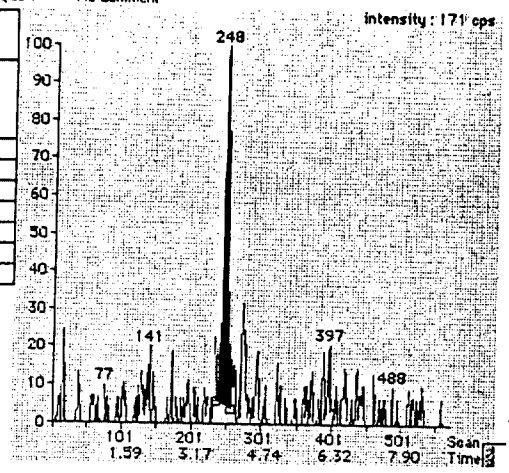
s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

399.0->80.0

Area	1155
Height	162
Start Time	3.73
End Time	4.05
Integration Width	0.32
Retention Time	3.91
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	185736
Height	27311
Start Time	4.30
End Time	4.84
Integration Width	0.54
Retention Time	4.51
Integration Type	A - BB

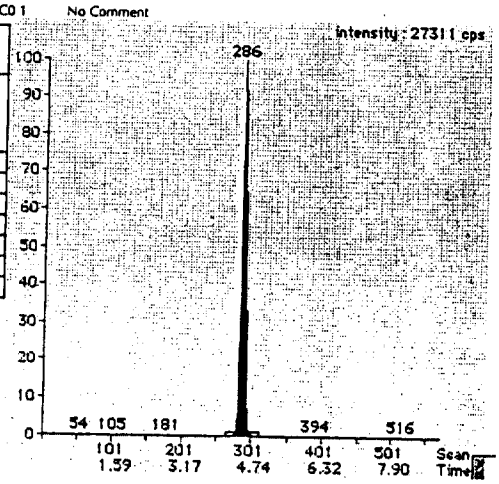
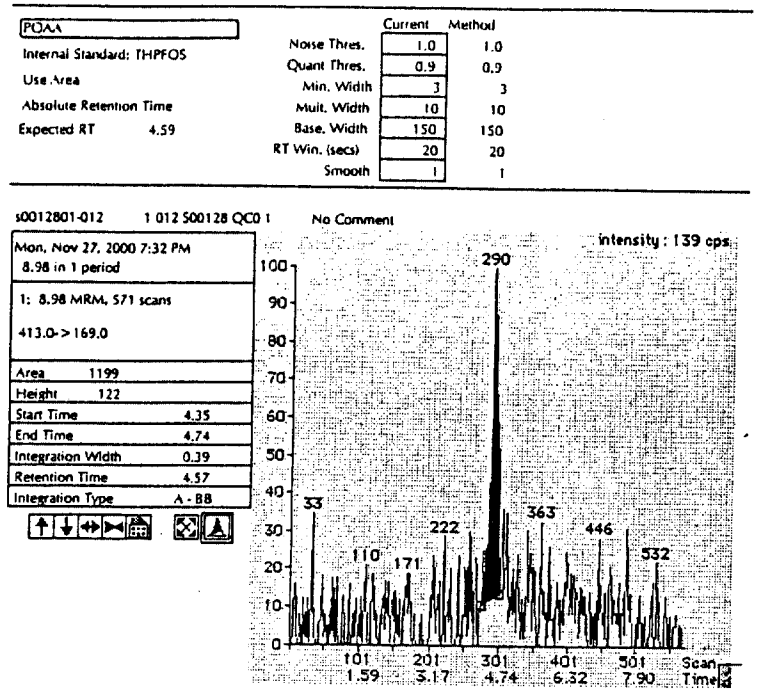
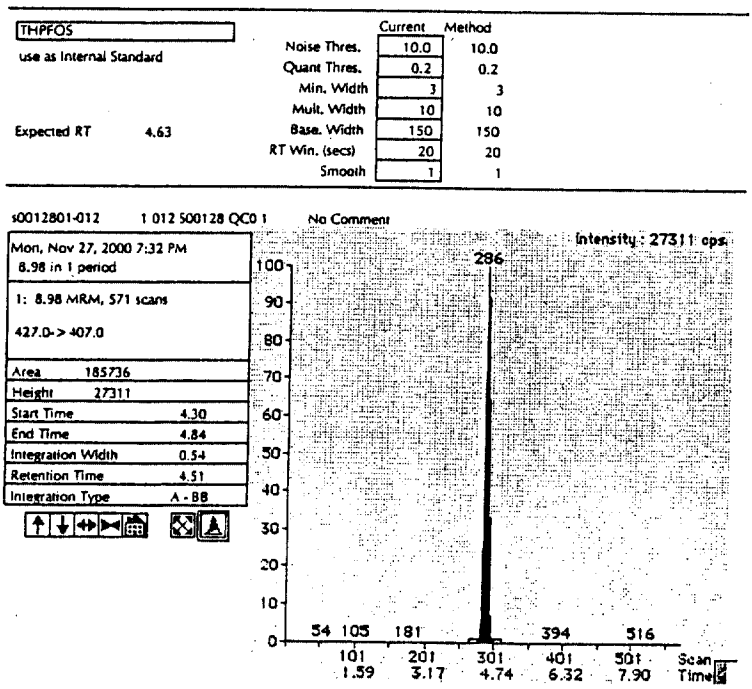


Figure 17. Human Plasma Blank with Internal Standard (QC0) for PFOA





000580

Figure 18. Human Plasma Blank with Internal Standard (QC0) for PFOSAA

PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.93

	Current	Method
Noise Thres.	4.0	4.0
Quant Thres.	1.5	1.5
Min. Width	3	3
Mult. Width	13	13
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

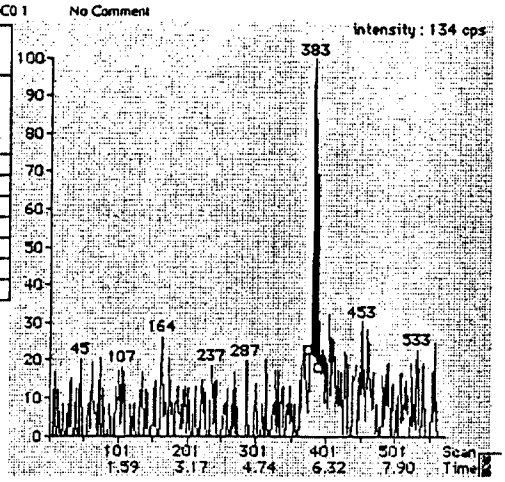
s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

584.1->419.1

Area	627
Height	106
Start Time	5.96
End Time	6.13
Integration Width	0.17
Retention Time	6.04
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	185736
Height	27311
Start Time	4.30
End Time	4.84
Integration Width	0.54
Retention Time	4.51
Integration Type	A - BB

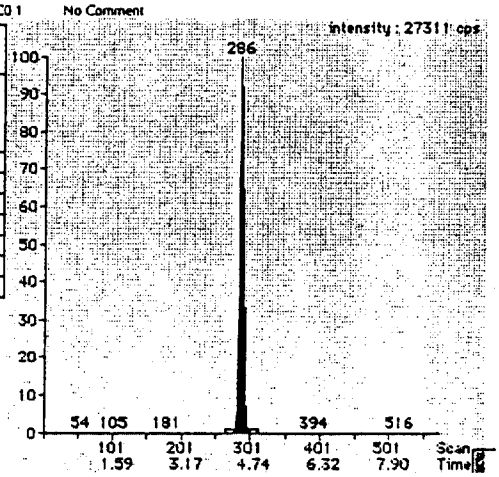


Figure 19. Human Plasma Blank with Internal Standard (QC0) for PFOSA

PFOSA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.70

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	1.0	1.0
Min. Width	5	5
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

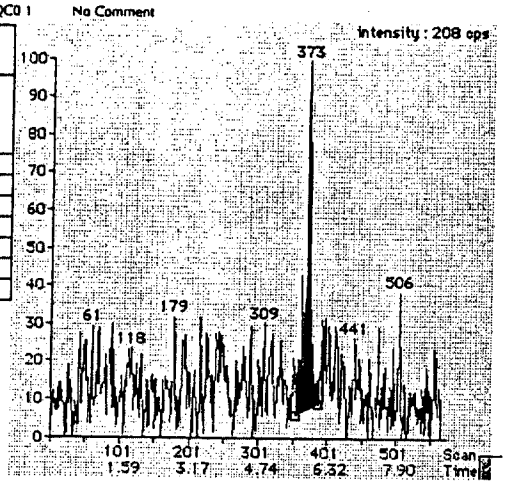
s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

498.0->78.0

Area	1451
Height	192
Start Time	5.61
End Time	6.10
Integration Width	0.49
Retention Time	5.88
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	185736
Height	27311
Start Time	4.30
End Time	4.84
Integration Width	0.54
Retention Time	4.51
Integration Type	A - BB

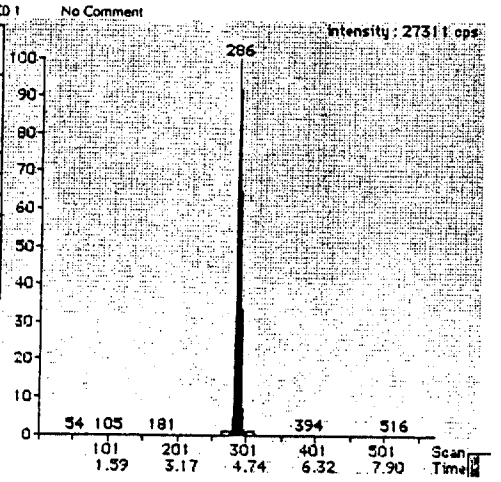


Figure 20. Human Plasma Blank with Internal Standard (QC0) for M556

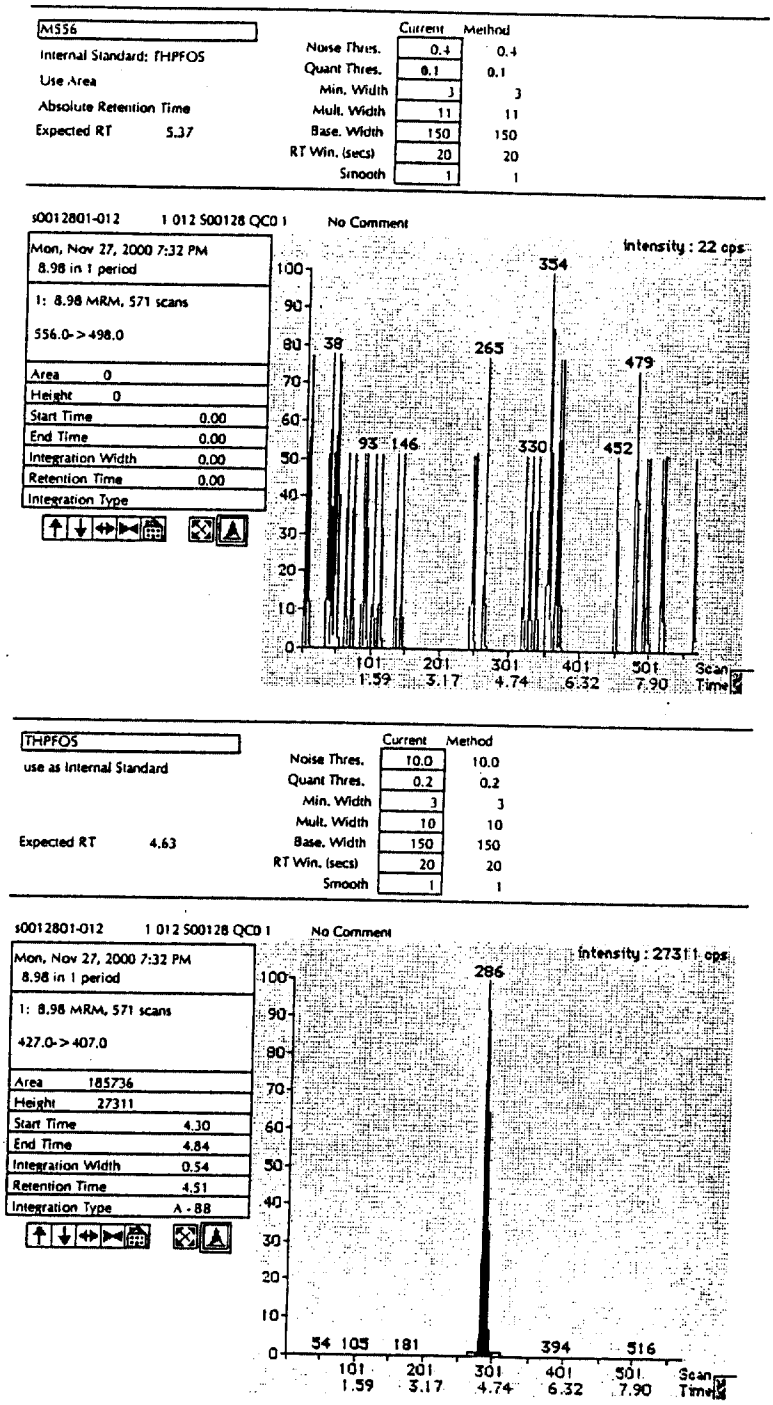


Figure 21. Human Plasma Blank with Internal Standard (QC0) for M570

M570

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5.63

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	5	5
Mult. Width	15	15
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

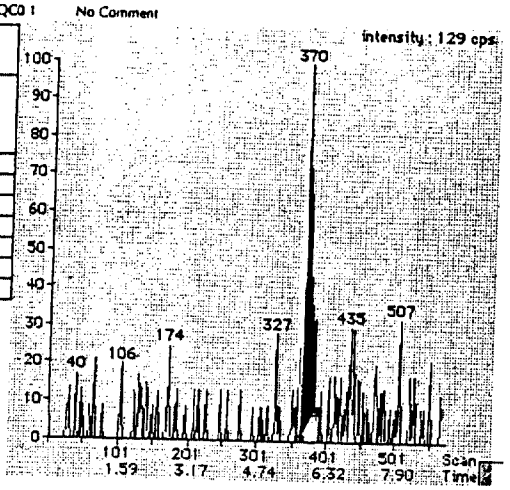
s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

570.0->419.1

Area	1046
Height	126
Start Time	5.74
End Time	6.05
Integration Width	0.32
Retention Time	5.83
Integration Type	A - BB



THPFOS

use as Internal Standard

Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-012 1 012 S00128 QC0 1 No Comment

Mon, Nov 27, 2000 7:32 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	185736
Height	27311
Start Time	4.30
End Time	4.84
Integration Width	0.54
Retention Time	4.51
Integration Type	A - BB

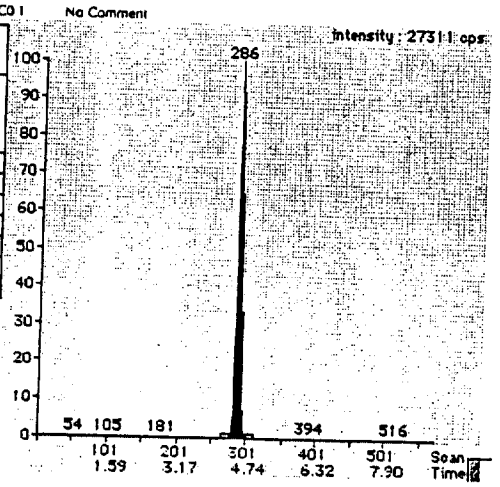


Figure 22. Low Standard for PFOS

PFOS		Current	Method
Internal Standard: THPFOS	Noise Thres.	5.0	5.0
Use Area	Quant Thres.	0.2	0.2
Absolute Retention Time	Min. Width	3	3
Expected RT 5.31	Mult. Width	12	12
	Base. Width	150	150
	RT Win. (secs)	20	20
	Smooth	1	1

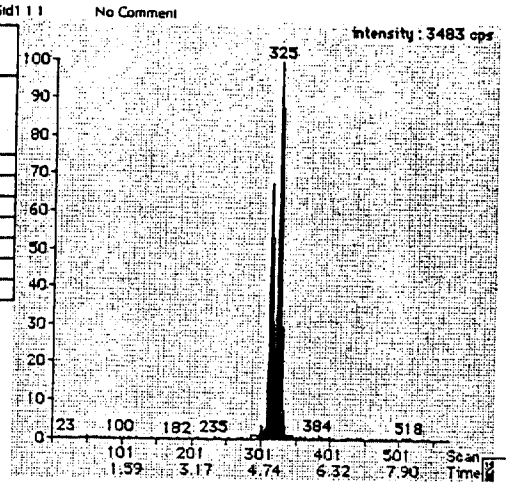
s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

499.0->80.0

Area	40256
Height	3482
Start Time	4.60
End Time	5.39
Integration Width	0.79
Retention Time	5.12
Integration Type	A - BB



THPFOS		Current	Method
use as Internal Standard	Noise Thres.	10.0	10.0
	Quant Thres.	0.2	0.2
	Min. Width	3	3
	Mult. Width	10	10
Expected RT 4.63	Base. Width	150	150
	RT Win. (secs)	20	20
	Smooth	1	1

s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	178943
Height	26839
Start Time	4.30
End Time	4.87
Integration Width	0.57
Retention Time	4.51
Integration Type	A - BB

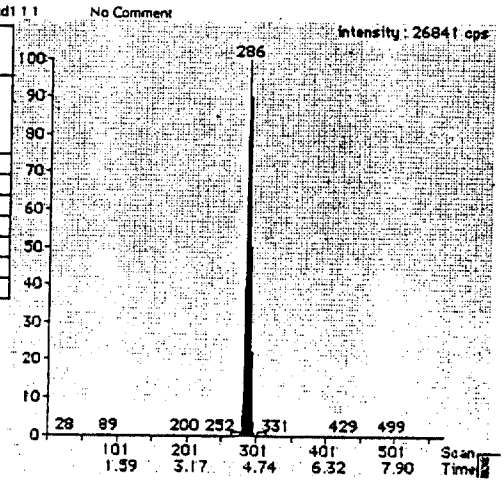


Figure 23. Low Standard for PFHS

PFHS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4.00

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	4	4
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

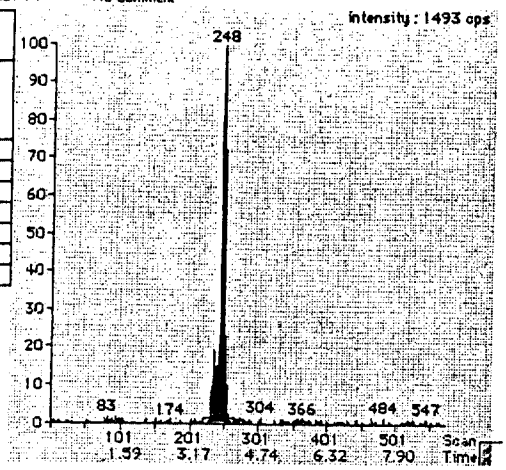
s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

399.0->80.0

Area	12158
Height	1488
Start Time	3.58
End Time	4.08
Integration Width	0.50
Retention Time	3.91
Integration Type	A - BB



THPFOS
use as Internal Standard

Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	178943
Height	26839
Start Time	4.30
End Time	4.87
Integration Width	0.57
Retention Time	4.51
Integration Type	A - BB

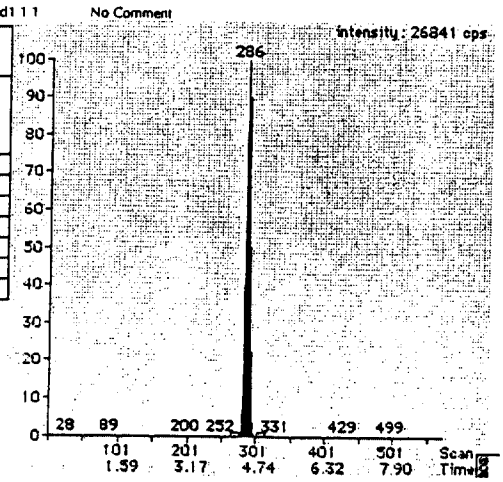


Figure 24. Low Standard for PFOA

POAA

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 4.59

	Current	Method
Noise Thres.	1.0	1.0
Quant Thres.	0.9	0.9
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

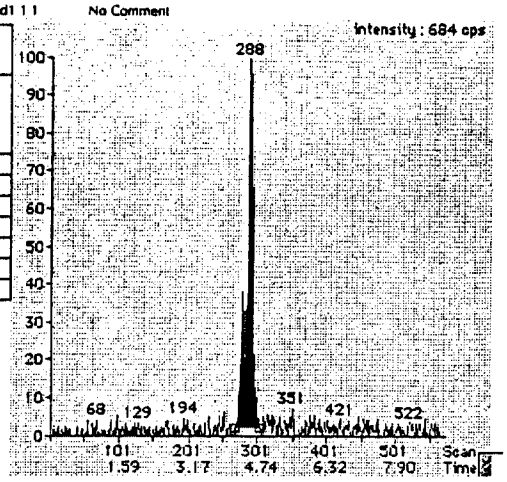
s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

413.0->169.0

Area	6248
Height	669
Start Time	4.25
End Time	4.77
Integration Width	0.52
Retention Time	4.54
Integration Type	A - BB



THPFOS

use as Internal Standard

Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	178943
Height	26839
Start Time	4.30
End Time	4.87
Integration Width	0.57
Retention Time	4.51
Integration Type	A - BB

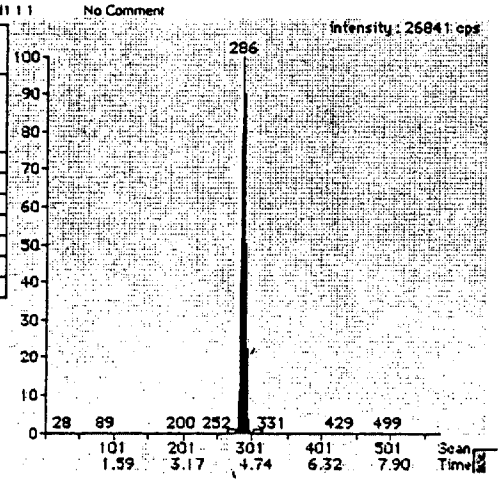


Figure 25. Low Standard for PFOSAA

PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.93

	Current	Method
Noise Thres.	4.0	4.0
Quant Thres.	1.5	1.5
Min. Width	3	3
Mult. Width	13	13
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

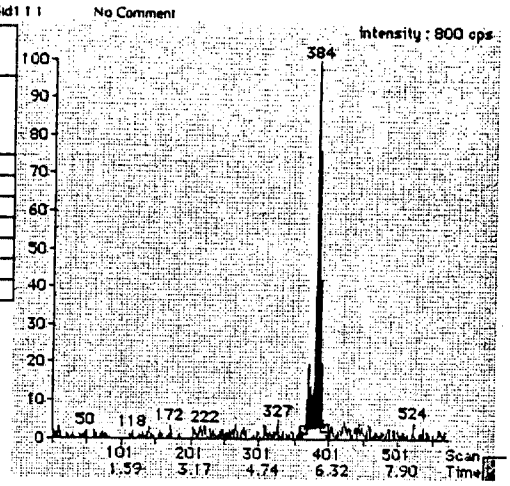
s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

584.1->419.1

Area	5578
Height	777
Start Time	5.77
End Time	6.21
Integration Width	0.44
Retention Time	6.05
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	178943
Height	26839
Start Time	4.30
End Time	4.87
Integration Width	0.57
Retention Time	4.51
Integration Type	A - BB

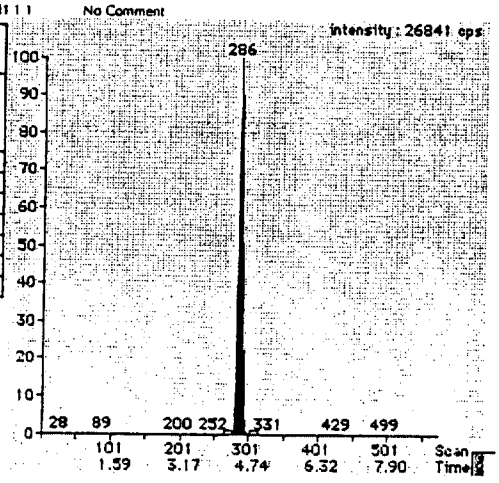


Figure 26. Low Standard for PFOSA

PFOSA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.70

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	1.0	1.0
Min. Width	5	5
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

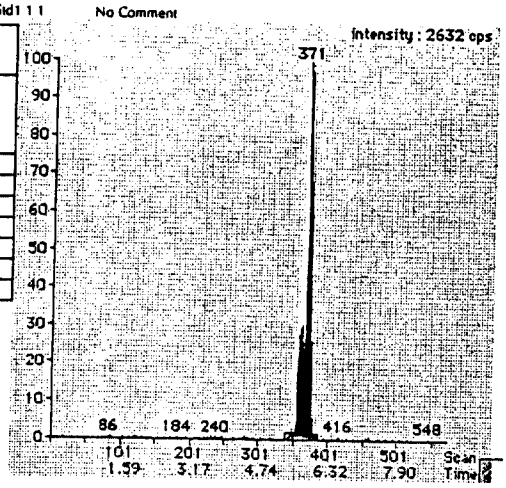
s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

498.0->78.0

Area	22235
Height	2620
Start Time	5.45
End Time	6.00
Integration Width	0.55
Retention Time	5.85
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	178943
Height	26839
Start Time	4.30
End Time	4.87
Integration Width	0.57
Retention Time	4.51
Integration Type	A - BB

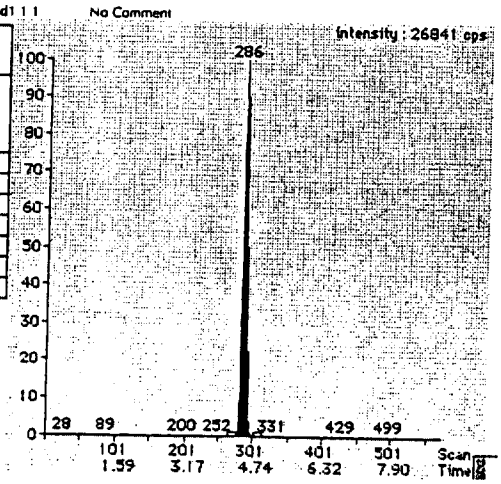


Figure 27. Low Standard for M556

M556
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.37

	Current	Method
Noise Thres.	0.4	0.4
Quant Thres.	0.1	0.1
Min. Width	3	3
Mult. Width	11	11
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

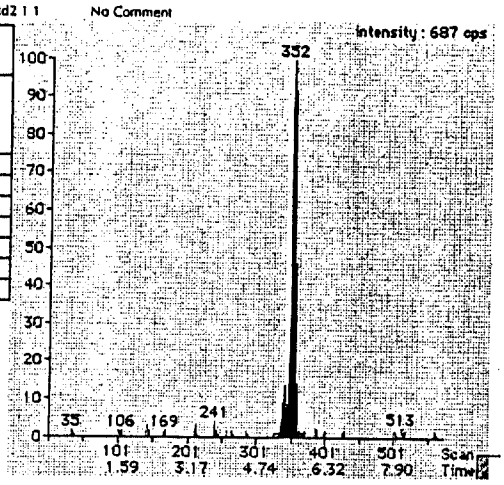
s0012801-003 1 003 S00128 Std2 1 1 No Comment

Mon, Nov 27, 2000 5:47 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

556.0->498.0

Area	4630
Height	686
Start Time	5.25
End Time	5.72
Integration Width	0.47
Retention Time	5.55
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-003 1 003 S00128 Std2 1 1 No Comment

Mon, Nov 27, 2000 5:47 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	179259
Height	27253
Start Time	4.30
End Time	4.84
Integration Width	0.54
Retention Time	4.51
Integration Type	A - BB

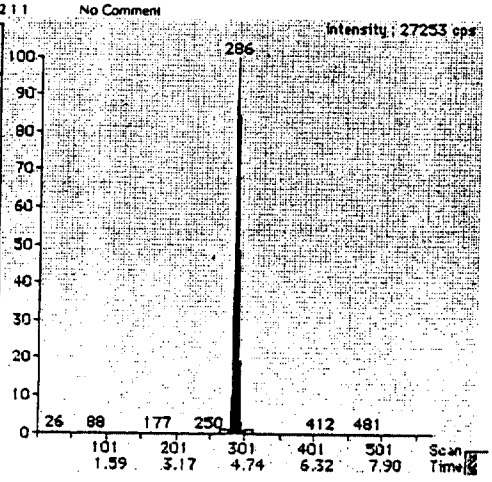


Figure 28. Low Standard for M570

M570

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5.63

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	5	5
Mult. Width	15	15
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

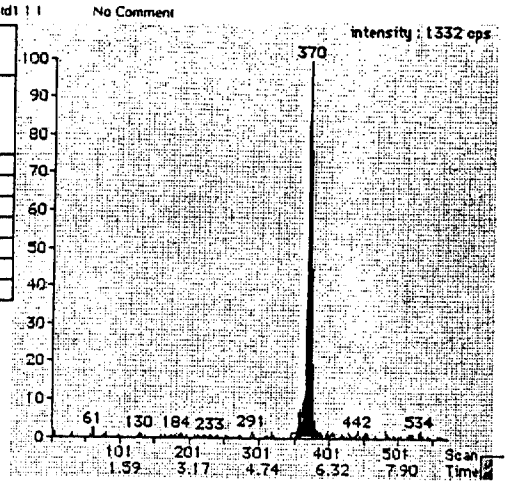
s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

570.0->419.1

Area	8966
Height	1325
Start Time	5.56
End Time	6.04
Integration Width	0.47
Retention Time	5.83
Integration Type	A - BB



THPFOS

use as Internal Standard

Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-002 1 002 S00128 Std1 1 1 No Comment

Mon, Nov 27, 2000 5:36 PM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	178943
Height	26839
Start Time	4.30
End Time	4.87
Integration Width	0.57
Retention Time	4.51
Integration Type	A - BB

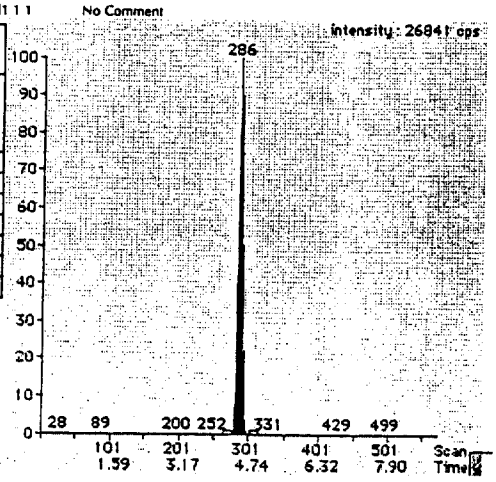


Figure 29. High Standard for PFOS

PFOS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.31

	Current	Method
Noise Thres.	5.0	5.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	12	12
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

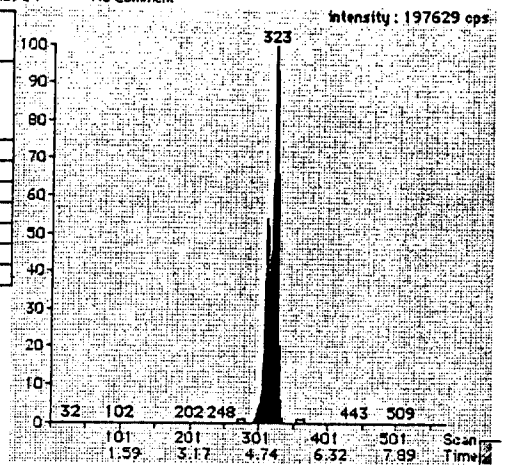
s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

499.0->80.0

Area	2662643
Height	197627
Start Time	4.37
End Time	5.70
Integration Width	1.34
Retention Time	5.09
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	160597
Height	18653
Start Time	4.27
End Time	4.73
Integration Width	0.46
Retention Time	4.51
Integration Type	A - BB

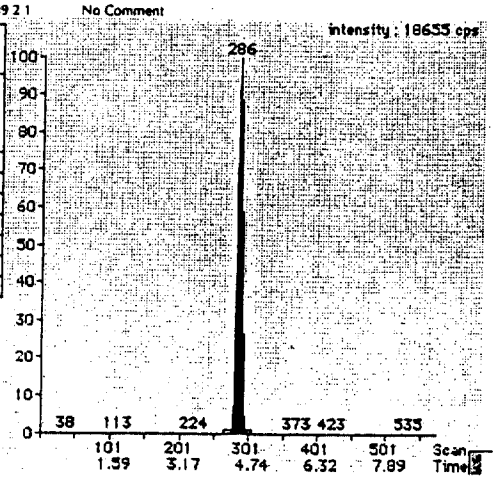


Figure 30. High Standard for PFHS

PFHS		Current	Method
Internal Standard: THPFOS	Noise Thres.	3.0	3.0
Use Area	Quant Thres.	0.5	0.5
Absolute Retention Time	Min. Width	4	4
Expected RT 4.00	Mult. Width	12	12
	Base. Width	150	150
	RT Win. (secs)	20	20
	Smooth	1	1

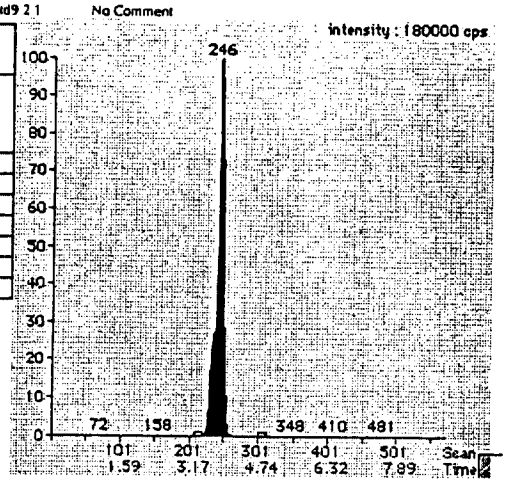
s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

399.0->80.0

Area	2124101
Height	179995
Start Time	3.39
End Time	4.82
Integration Width	1.43
Retention Time	3.88
Integration Type	A - BB



THPFOS		Current	Method
use as Internal Standard	Noise Thres.	10.0	10.0
	Quant Thres.	0.2	0.2
	Min. Width	3	3
	Mult. Width	10	10
Expected RT 4.63	Base. Width	150	150
	RT Win. (secs)	20	20
	Smooth	1	1

s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	160597
Height	18653
Start Time	4.27
End Time	4.73
Integration Width	0.46
Retention Time	4.51
Integration Type	A - BB

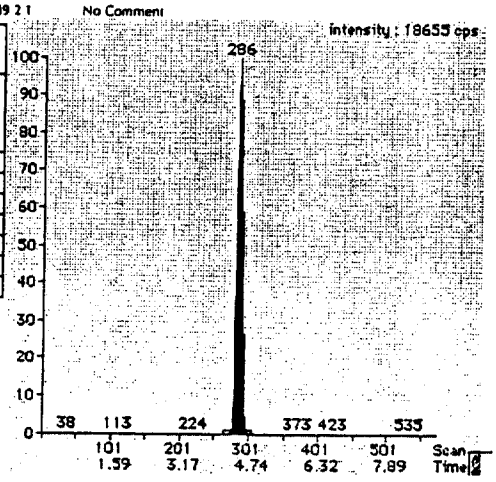


Figure 31. High Standard for PFOA

POAA		Current	Method
Internal Standard: THPFOS	Noise Thres.	1.0	1.0
Use Area	Quant Thres.	0.9	0.9
Absolute Retention Time	Min. Width	3	3
Expected RT 4.59	Mult. Width	10	10
	Base. Width	150	150
	RT Win. (secs)	20	20
	Smooth	1	1

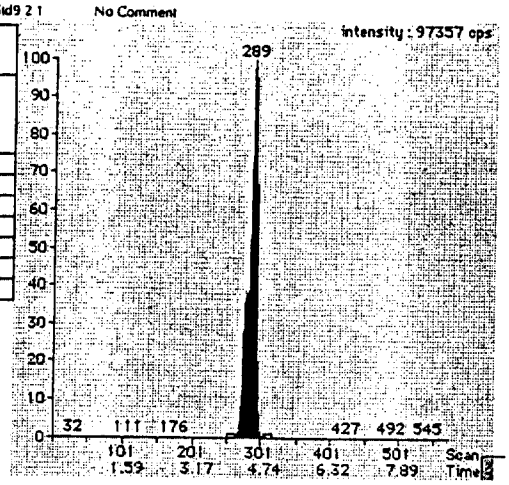
s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

413.0->169.0

Area	1221015
Height	97340
Start Time	4.07
End Time	4.89
Integration Width	0.82
Retention Time	4.55
Integration Type	A - BV



THPFOS		Current	Method
use as Internal Standard	Noise Thres.	10.0	10.0
	Quant Thres.	0.2	0.2
	Min. Width	3	3
	Mult. Width	10	10
Expected RT 4.63	Base. Width	150	150
	RT Win. (secs)	20	20
	Smooth	1	1

s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	160597
Height	18653
Start Time	4.27
End Time	4.73
Integration Width	0.46
Retention Time	4.51
Integration Type	A - BB

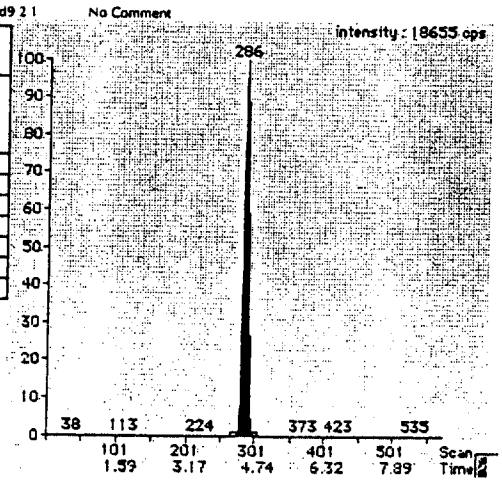


Figure 32. High Standard for PFOSAA

PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.93

	Current	Method
Noise Thres.	4.0	4.0
Quant Thres.	1.5	1.5
Min. Width	3	3
Mult. Width	13	13
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

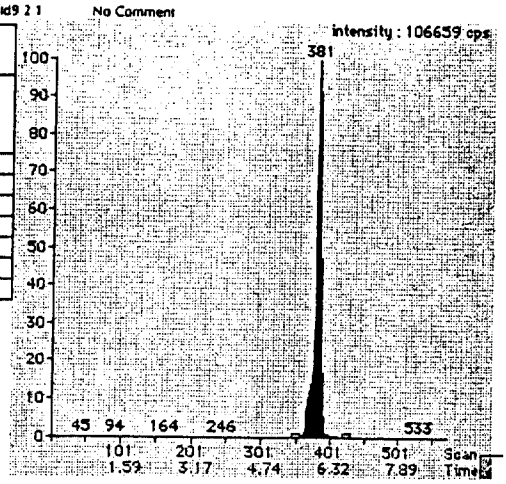
s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

584.1->419.1

Area	995787
Height	106632
Start Time	5.55
End Time	6.71
Integration Width	1.17
Retention Time	6.00
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	160597
Height	18653
Start Time	4.27
End Time	4.73
Integration Width	0.46
Retention Time	4.51
Integration Type	A - BB

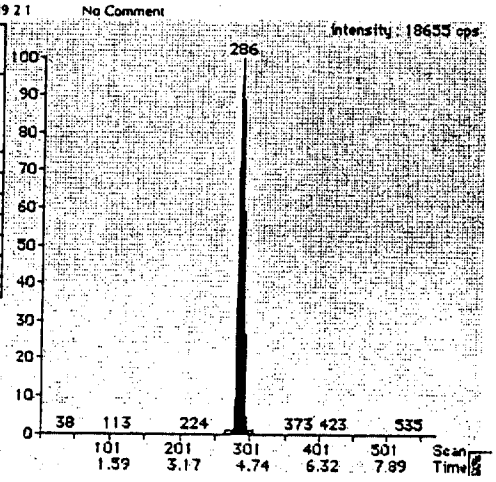


Figure 33. High Standard for PFOSA

PFOSA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5.70

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	1.0	1.0
Min. Width	5	5
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

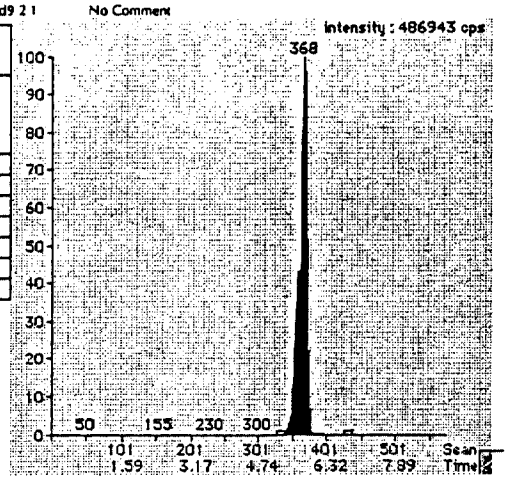
s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

498.0->78.0

Area	6035364
Height	486936
Start Time	5.26
End Time	6.79
Integration Width	1.53
Retention Time	5.80
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	160597
Height	18653
Start Time	4.27
End Time	4.73
Integration Width	0.46
Retention Time	4.51
Integration Type	A - BB

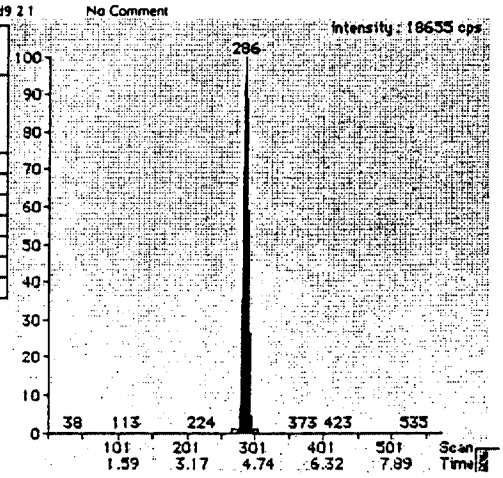


Figure 34. High Standard for M556

M556

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5.37

	Current	Method
Noise Thres.	0.4	0.4
Quant Thres.	0.1	0.1
Min. Width	3	3
Mult. Width	11	11
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

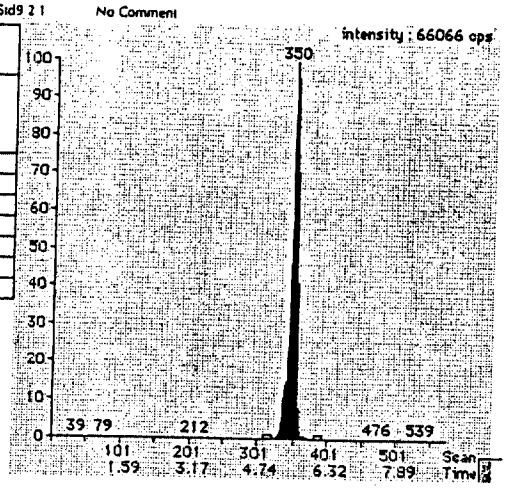
s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

556.0-> 498.0

Area	620535
Height	66065
Start Time	5.00
End Time	6.10
Integration Width	1.10
Retention Time	5.52
Integration Type	A - BV



THPFOS

use as Internal Standard

Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

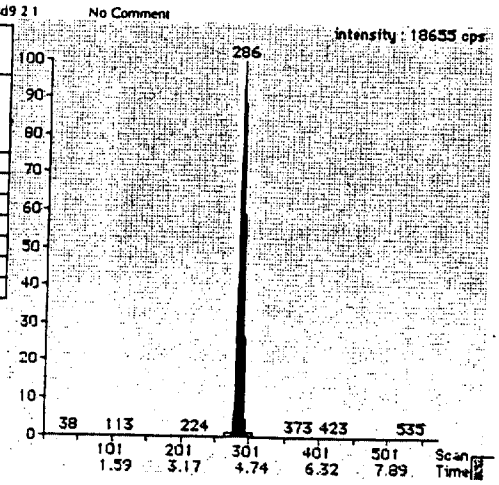
s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0-> 407.0

Area	160597
Height	18653
Start Time	4.27
End Time	4.73
Integration Width	0.46
Retention Time	4.51
Integration Type	A - BB



000597

Figure 35. High Standard for M570

M570

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5.63

	Current	Method
Noise Thres.	3.0	3.0
Quant Thres.	0.5	0.5
Min. Width	5	5
Mult. Width	15	15
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

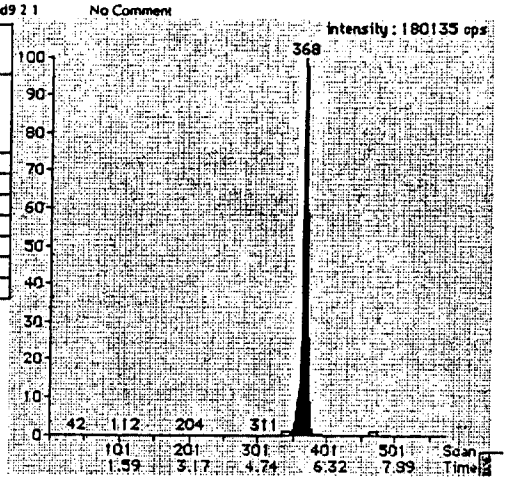
s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

570.0->419.1

Area	1573682
Height	180133
Start Time	5.37
End Time	7.39
Integration Width	2.02
Retention Time	5.80
Integration Type	A - BB



THPFOS

use as Internal Standard

Expected RT 4.63

	Current	Method
Noise Thres.	10.0	10.0
Quant Thres.	0.2	0.2
Min. Width	3	3
Mult. Width	10	10
Base. Width	150	150
RT Win. (secs)	20	20
Smooth	1	1

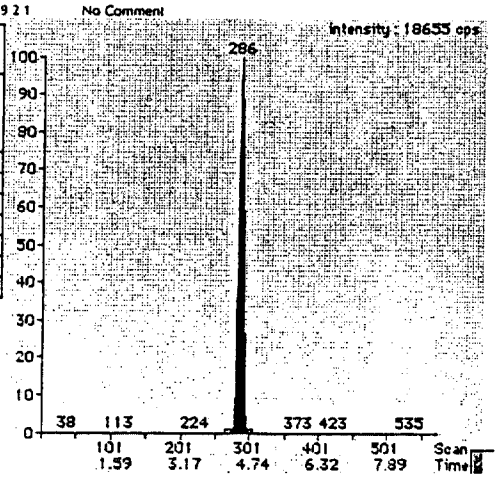
s0012801-080 1 080 S00128 Std9 2 1 No Comment

Tue, Nov 28, 2000 8:39 AM
8.98 in 1 period

1: 8.98 MRM, 571 scans

427.0->407.0

Area	160597
Height	18653
Start Time	4.27
End Time	4.73
Integration Width	0.46
Retention Time	4.51
Integration Type	A - BB



Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS

Sample Analysis Report for Protocol EPI-0016

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Northwest Bioanalytical (NWB)
A Division of NWT Inc.
1121 East 3900 South
Salt Lake City, UT 84124

PREPARED FOR: 3M Company
Corporate Occupational Medicine, Medical Department
220-3W-05
St. Paul, MN 55144

AUTHOR:
Connie O Sakashita DATE: 1-10-02
Connie O. Sakashita, B.S., NWB Project Manager

APPROVED FOR RELEASE BY:
RL Foltz DATE: 1-10-02
Rodger L. Foltz, Ph.D., NWB Technical Director

CONTAIN NO CBI

QUALITY ASSURANCE STATEMENT

LABORATORY:	Northwest Bioanalytical (NWB) A Division of NWT Inc. 1121 East 3900 South Salt Lake City, UT 84124
SPONSOR:	3M Company Corporate Occupational Medicine, Medical Department 220-3W-05 St. Paul, MN 55144
COMPOUND(S):	PFOS and Related Compounds
NWB STUDY NUMBER:	NWBS00-145
SPONSOR STUDY NUMBER:	EPI-0016
NWB STUDY TITLE:	Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS

The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all studies within the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.) The following inspections were performed by the NWB QAU per SOP.

Inspection and Reporting Statement.

Date of Inspection	Phase of Study	Date Report Sent to NWB Project Manager	Date Report Sent to NWB Management*
22-23 Oct 2001	Standards Preparation	23 Oct 2001	31 Oct 2001
22-23 Oct 2001	QC Preparation	23 Oct 2001	31 Oct 2001
24 Oct 2001	Analytical Plan	24 Oct 2001	31 Oct 2001
15-16 Nov 2001	Sample Analysis	16 Nov 2001	30 Nov 2001
07-09 Jan 2002	Report Draft/Raw Data	09 Jan 2002	31 Jan 2002
10 Jan 2002	Final Report	10 Jan 2002	31 Jan 2002

*Reports to NWB Management are issued monthly.

As can reasonably be established, the methods and procedures described and the results incorporated into this final report accurately reflect the raw data.

Stacy Lynn Chain, B.A.
NWB Quality Assurance Compliance Auditor

01-10-02
Date

000600

COMPLIANCE STATEMENT

The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, to the best of our knowledge, this project was conducted in accordance with the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58) and according to the methods and procedures described within this report. In addition, the study followed the guidelines of the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424.) Any known incidents that may have affected the quality or integrity of the project or reported data is included in this report. This report represents an accurate record of the raw data.

Connie O Sakashita

Connie O. Sakashita, B.S.
NWB Project Manager

1-10-02
Date

R L Foltz

Rodger L. Foltz, Ph.D.
NWB Technical Director

1-10-02
Date

000601

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Quantitative Determination of PFOS and Related Compounds in Human Serum by LC/MS/MS

Sample Analysis Report for Protocol EPI-0016

1. INTRODUCTION

This report summarizes the analytical results from the quantitation of PFOS, PFOA, PFHS, PFOSAA, PFOSA, M556, and M570 in human serum samples for 3M Company (3M) in support of Protocol EPI-0016 [5.1]. The LC/MS/MS method for the analytes has a targeted calibration curve range of 1.00 to 500 ppb (except for M556 which has a LLOQ of 2.50 ppb). The actual calibration curve range for each analyte varies based upon the persistent levels of the analyte in the matrix. For this study, the assay exhibited the following LLOQ and ULOQ values:

Analyte	LLOQ	ULOQ
PFOS	3.37 ppb	413 ppb
PFOA	1.44 ppb	481 ppb
PFHS	1.36 ppb	522 ppb
PFOSAA	1.50 ppb	501 ppb
PFOSA	1.00 ppb	500 ppb
M556	2.50 ppb	500 ppb
M570	1.00 ppb	500 ppb

The sponsor was 3M Company (Corporate Occupational Medicine, Medical Department, 220-3W-05, St. Paul, MN 55144), and the Study Director was Jeffrey H. Mandel, M.D., M.P.H. The Study Contact at 3M was Jean M. Burris. James K. Lundberg was the technical contact at 3M. The following is a list of NWB supervisory personnel involved in the completion of this work: Connie O. Sakashita, B.S. (NWB Project Manager); Patrick Bennett, M.S., M.B.A. (NWB Laboratory Director); Rodger L. Foltz, Ph.D. (NWB Technical Director). Laboratory personnel involved in the completion of this

study included Suzanne Newman, B.S. (NWB Research Scientist); Laura Struhs, B.S. (NWB Scientist) and Anna Akrami, B.S. (NWB Associate Research Scientist).

NWB SOPs and guidelines were used in the conduct of this study and were available to study personnel in electronic and/or hard copy formats.

Date Study Initiated: 23-Oct-2001 Date Analyses Completed: 16-Nov-2001

The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all studies within the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 58), the OECD Principles of Good Laboratory Practice and the Japanese MHW Good Laboratory Practice Standard Ordinance for Nonclinical Laboratory Studies on the Safety of Drugs (Ordinance No. 21, PAB Notification No. 424). Any changes to or deviations from the original protocol (Analytical Plan) were documented through approved protocol amendments or deviation memos and are retained within the raw data.

2. METHODOLOGY

The assay used for this study is reported in Northwest Bioanalytical reports NWBR00-108 [5.2] and NWBR00-122 [5.3]. Chinese human plasma was used for the calibration curves and quality control samples. Serum sample results obtained using plasma curves are discussed in NWBR00-122.

Samples for this study were received at NWB on the following date:

Receipt Date	*Number of Samples Received*	*Storage Condition (except during analysis)*
23-Oct-2001	262	-20 °C

000607

Reference Material

Analyte	*Lot Number*	*Purity*	*Expiration Date*	*Source*	*Storage Conditions*
PFOS	193	100%	31-Dec-2010	3M	Room temperature*
PFOA	245	100%	31-Dec-2010	3M	Room temperature*
PFHS	SE-036	100%	01-Jan-2010	3M	-20 °C
PFOSAA	617	53.8%	31-Dec-2010	3M	Room temperature
PFOSA	214	100%	31-Dec-2010	3M	Room temperature
M556	NB113047-8D	99.89%	31-Dec-2010	3M	Room temperature
M570	118506-26	99.75%	31-Dec-2010	3M	Room temperature
THPFOS	59909	90%	31-Dec-2010	3M	Room temperature

*Stored dry

The reference material purity for PFOS, PFOSA, PFHS, and PFOA was not available prior to the conduct of this study. Therefore, the reference material purity was assumed to be 100%.

3M contracted with Centre Analytical Laboratories, Inc., in State College, Pennsylvania to determine the absolute concentration of PFOS, PFOA, and PFHS in NWB stock solutions used to prepare the analytical standards and controls used for this studies. All arrangements for purity determinations and the transfer of NWB solutions to Centre Analytical Laboratories, Inc., were performed by 3M. Based on Centre Analytical Laboratories' results, the concentrations of plasma calibrator and quality control samples were corrected using the following factors:

Analyte	*Correction Factor*
PFOS	0.821
PFOA	0.961
PFHS	1.043

Eight or more calibration standards were prepared on the day of each run by adding 100 µL of blank Chinese human plasma and 400 µL of 50 mM ammonium acetate in

water (unadjusted pH ~6.9) to 13 x 100 mm polypropylene tubes. After a brief vortex mixing, 10.0 µL of the appropriate spiking solution was added. The final calibration standard concentrations in human plasma for each analyte are listed below:

Analyte	Calibration Standard Concentrations*
PFOS	3.37, 4.60, 10.8, 23.1, 43.6, 84.6, 208, 331 and 413 ppb
PFOA	1.44, 2.88, 10.1, 24.5, 48.5, 97.1, 241, 385 and 481 ppb
PFHS	1.36, 2.92, 10.7, 26.4, 52.5, 104, 261, 417 and 522 ppb
PFOSAA	1.50, 3.00, 10.5, 25.5, 50.5, 101, 251, 401 and 501 ppb
PFOSA	1.00, 2.50, 10.0, 25.0, 50.0, 100, 250, 400 and 500 ppb
M556	2.50, 10.0, 25.0, 50.0, 100, 250, 400 and 500 ppb
M570	1.00, 2.50, 10.0, 25.0, 50.0, 100, 250, 400 and 500 ppb

* The target calibration curve range is 1.00 ppb to 500 ppb (except M556 which has a LLOQ of 2.50 ppb). Each analyte has a different final curve range based upon the persistent levels of the analyte in the human plasma used.

In addition, blank human plasma samples, both with and without internal standard (designated as QC0s and Blanks, respectively), were assayed in each analytical run.

Analytical QCs were prepared in Chinese human plasma on November 5, 2001 and stored in a -20 °C freezer. For each run, analytical QC levels were assayed in duplicate.

	QC Concentrations (ppb)*		
Analyte	Low	Medium	High
PFOS	5.83	126	331
PFOA	4.32	144	384
PFHS	4.48	156	417
PFOSAA	4.50	151	401
PFOSA	4.00	150	400
M556	4.00	150	400
M570	4.00	150	400

* The target QC concentrations are 4.00 ppb, 150 ppb and 400 ppb. Each analyte has different QC concentrations based upon the persistent levels of the analyte in the human plasma used.

The internal standard (THPFOS) was added to all serum samples (except Blanks) for a final concentration of approximately 200 ppb.

The analytical method consisted of a liquid:liquid extraction procedure followed by evaporation and reconstitution of the extract residue with 20 mM ammonium acetate in water: 20 mM ammonium acetate in methanol (30:70 v/v). The samples were analyzed by liquid chromatography/tandem mass spectrometry using an API 3000. The instrument was operated in the multiple reaction monitoring (MRM) mode under optimized conditions for PFOS, PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 detection of the negative ions formed by turboionspray ionization.

3. SAMPLE ANALYSIS

PFOS, PFOA, PFHS, PFOSAA, PFOSA, M556, M570, and THPFOS chromatographic peaks were integrated using the MacQuan software (version 1.6) with a smooth factor of one. Quantitation was based upon quadratic regression analysis of weighted ($1/x^2$) calibration curves using area ratio vs. concentration utilizing Watson® DMLIMS software (version 6.1.1.04). The samples were injected in a systematic order.

3.1. Acceptance Criteria

For an analytical run to be accepted, it must have met the acceptance criteria listed below which are consistent with regulatory and industry recommendations.

Calibration Curve

Each run will include in duplicate calibration standards at six or more concentrations covering the lower to upper limit of quantitation. For all analytes except PFOSA and PFOSAA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 15% (± 20% for LLOQ) of their individual target concentrations. For PFOSA and PFOSAA, at least three-fourths of the calibration standard's back-calculated concentrations must be within ± 20% (± 25% for LLOQ) of their individual target concentrations. A calibration standard will be considered an outlier if it is greater than two times the acceptance criteria for that standard.

Lower Limit of Quantitation

The back-calculated concentrations of at least one of the duplicate lowest points in the calibration curve must be within ± 25% of the target concentration for PFOSA and PFOSAA to qualify as the LLOQ and within ± 20% of the target concentration to qualify as the LLOQ for all other analytes. If this criterion is not met, the next level is subjected to the same test and the LLOQ raised accordingly.

Quality Control Samples

Each analytical run will include low, medium and high QC samples in duplicate. The measured concentrations of at least two-thirds of all analytical QCs must be within ± 20% of their target concentrations (± 25% for PFOSA and PFOSAA), and no two QCs at the same concentration can be outside the limit. If study samples require dilution, a dilution QC will be analyzed in triplicate for each dilution level (except for PFOSA and PFOSAA which should not be diluted with control matrix). At least two dilution QCs at each level must be within ± 20% of its target concentrations in order to accept diluted study samples at that level. The dilution QC acceptance is independent of the undiluted analytical QC acceptance.

4. RESULTS AND DISCUSSION

Serum sample results obtained using plasma curves for PFOA, PFHS, PFOSAA, PFOSA, M556, and M570 did not meet NWB SOP requirements for validation. Results may vary on average up to 26% from results obtained using human serum calibration curves at some concentration levels for PFOA, PFHS, PFOSAA, M556, and M570. For PFOSA, results may vary on average up to 43% from results obtained using human serum calibration curves. These data are reported in Assay Revalidation Addendum Report NWBR00-122 [5.3]. However, in order to obtain the lower limit of quantitation of less than 5 ppb required by the sponsor, it was necessary to use plasma calibration curves.

Percent theoretical and percent bias are separate calculations determined from the mean. Therefore, due to rounding, percent theoretical plus percent bias may not equal 100.0%.

Each accepted run met the acceptance criteria set for the calibration curve points, for the lower limit of quantitation (LLOQ) and for the analytical quality control (QC) samples.

Run ID	PFOS Result	PFOA Result	PFHS Result	PFOSAA Result	Comments
1	Accepted	Accepted	Accepted	Accepted	
2	Accepted	Accepted	Accepted	Accepted	
3	Accepted	Accepted	Accepted	Accepted	
4	N/A	N/A	N/A	N/A	Run aborted, sample preparation error
5	Accepted	Accepted	Accepted	Accepted	
6	Accepted	Accepted	Accepted	Accepted	
7	Accepted	Accepted	Accepted	Accepted	
8	Accepted	Accepted	Accepted	Accepted	

N/A = not applicable

Run ID	PFOSA Result	M556 Result	M570 Result	Comments
1	Accepted	Accepted	Accepted	
2	Accepted	Accepted	Accepted	
3	Accepted	Accepted	Accepted	
4	N/A	N/A	N/A	Run aborted, sample preparation error
5	Accepted	Accepted	Accepted	
6	Accepted	Accepted	Accepted	
7	Accepted	Accepted	Accepted	
8	Accepted	Accepted	Accepted	

N/A = not applicable

Any known circumstances that may have affected the quality or integrity of the data are included in this report.

5. REFERENCES

5.1. 3M Company Protocol EPI-0016. "Identification of Fluorochemicals in Serum from and Elderly Population in the Seattle, Washington Metropolitan Area."

5.2. C. Sakashita. "Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS." Assay Revalidation Report. NWB Study NWBS00-040. NWB Report NWBR00-108. January 24, 2001.

5.3. C. Sakashita. "Quantitative Determination of PFOS, PFOSA, PFOSAA, POAA, PFHS, M556 and M570 in Human Serum by LC/MS/MS." Assay Revalidation Addendum Report. NWB Study NWBS00-040. NWB Report NWBR00-122. November 20, 2001.

6. DATA RETENTION

The raw data and final report for this study will be stored in the NWB Archives, 1121 East 3900 South, Salt Lake City, UT 84124 per regulations and contract agreement. After submission of the final report to the Sponsor, remaining study samples will be stored under required conditions until confirmation of Sample Disposition/Return Authorization is received from the Sponsor. 3M Company will be notified concerning final disposition of records at completion of contract obligations.

Table 1. Calibration Curve Summary for PFOS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
08-Nov-2001	1	0.000000	0.004611	0.001070	0.9984	3.37	413
09-Nov-2001	2	0.000000	0.004551	-0.000952	0.9958	3.37	413
13-Nov-2001	3	-0.000001	0.004805	0.001604	0.9974	3.37	413
14-Nov-2001	5	0.000000	0.005168	0.001523	0.9917	3.37	413
15-Nov-2001	6	-0.000001	0.004975	-0.001202	0.9985	3.37	413
15-Nov-2001	7	-0.000001	0.004756	-0.001763	0.9956	3.37	413
16-Nov-2001	8	0.000000	0.005736	-0.000223	0.9964	3.37	413
Mean		0.000000	0.004943	0.000008	0.9963		
S.D.		0.000001	0.000408	0.001387	0.0023		
%CV			8.3	17337.5	0.2		
n		7	7	7	7		

A, B, and C are coefficients used to define the calibration curve.

Table 2. Calibration Curve Summary for PFOA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
08-Nov-2001	1	0.000001	0.006091	0.001738	0.9965	1.44	481
09-Nov-2001	2	0.000001	0.005194	0.000808	0.9931	1.44	481
13-Nov-2001	3	0.000000	0.005054	0.000831	0.9960	1.44	481
14-Nov-2001	5	-0.000001	0.005711	0.000382	0.9966	1.44	481
15-Nov-2001	6	0.000001	0.006132	-0.000212	0.9961	1.44	481
15-Nov-2001	7	0.000001	0.006102	0.000361	0.9977	1.44	481
16-Nov-2001	8	0.000000	0.006771	-0.000296	0.9970	1.44	481
Mean		0.000000	0.005865	0.000516	0.9961		
S.D.		0.000001	0.000596	0.000696	0.0015		
%CV			10.2	134.9	0.2		
n		7	7	7	7		

A, B, and C are coefficients used to define the calibration curve.

Table 3. Calibration Curve Summary for PFHS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
08-Nov-2001	1	-0.000002	0.007829	0.000656	0.9971	1.36	522
09-Nov-2001	2	-0.000002	0.008314	-0.001142	0.9969	1.36	522
13-Nov-2001	3	-0.000001	0.005315	0.000115	0.9972	1.36	522
14-Nov-2001	5	-0.000001	0.006211	-0.000433	0.9960	1.36	522
15-Nov-2001	6	-0.000002	0.006849	-0.001305	0.9977	1.36	522
15-Nov-2001	7	-0.000002	0.006791	-0.000751	0.9978	1.36	522
16-Nov-2001	8	-0.000002	0.006953	0.000081	0.9986	1.36	522
Mean		-0.000002	0.006895	-0.000397	0.9973		
S.D.		0.000000	0.000989	0.000719	0.0008		
%CV		0.0	14.3	-181.1	0.1		
n		7	7	7	7		

A, B, and C are coefficients used to define the calibration curve.

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Table 4. Calibration Curve Summary for PFOSAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
08-Nov-2001	1	0.000002	0.004883	0.002297	0.9868	1.50	501
09-Nov-2001	2	0.000001	0.005152	0.000251	0.9916	1.50	501
13-Nov-2001	3	-0.000001	0.010855	-0.001647	0.9963	1.50	501
14-Nov-2001	5	-0.000001	0.010710	-0.000618	0.9811	1.50	501
15-Nov-2001	6	0.000001	0.008626	-0.000767	0.9955	1.50	501
15-Nov-2001	7	0.000002	0.007392	-0.001672	0.9905	1.50	501
16-Nov-2001	8	0.000003	0.011058	-0.000963	0.9960	1.50	501
Mean		0.000001	0.008382	-0.000446	0.9911		
S.D.		0.000002	0.002659	0.001376	0.0056		
%CV		200.0	31.7	-308.5	0.6		
n		7	7	7	7		

A, B, and C are coefficients used to define the calibration curve.

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Table 5. Calibration Curve Summary for PFOSA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
08-Nov-2001	1	0.000001	0.005637	0.000591	0.9955	1.00	500
09-Nov-2001	2	0.000001	0.005563	0.001213	0.9908	1.00	500
13-Nov-2001	3	-0.000002	0.014503	0.002158	0.9896	1.00	500
14-Nov-2001	5	-0.000001	0.013789	0.002219	0.9880	1.00	500
15-Nov-2001	6	0.000002	0.010415	0.000499	0.9971	1.00	500
15-Nov-2001	7	0.000002	0.010693	0.000700	0.9940	1.00	500
16-Nov-2001	8	0.000004	0.014421	0.003092	0.9962	1.00	500
Mean		0.000001	0.010717	0.001496	0.9930		
S.D.		0.000002	0.003872	0.001003	0.0036		
%CV		200.0	36.1	67.0	0.4		
n		7	7	7	7		

A, B, and C are coefficients used to define the calibration curve.

Table 6. Calibration Curve Summary for M556

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
08-Nov-2001	1	0.000001	0.006345	-0.000642	0.9952	2.50	500
09-Nov-2001	2	0.000001	0.006891	-0.001177	0.9950	2.50	500
13-Nov-2001	3	0.000000	0.004174	-0.002780	0.9942	2.50	500
14-Nov-2001	5	0.000000	0.003962	0.001886	0.9844	2.50	500
15-Nov-2001	6	0.000001	0.003844	-0.002125	0.9986	2.50	500
15-Nov-2001	7	0.000002	0.003291	-0.000626	0.9947	2.50	500
16-Nov-2001	8	0.000001	0.004646	-0.001463	0.9970	2.50	500
Mean		0.000001	0.004736	-0.000990	0.9942		
S.D.		0.000001	0.001356	0.001488	0.0046		
%CV		100.0	28.6	-150.3	0.5		
n		7	7	7	7		

A, B, and C are coefficients used to define the calibration curve.

Table 7. Calibration Curve Summary for M570

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
08-Nov-2001	1	0.000001	0.007316	0.000681	0.9969	1.00	500
09-Nov-2001	2	0.000001	0.007844	0.001194	0.9924	1.00	500
13-Nov-2001	3	-0.000003	0.015001	0.000743	0.9964	1.00	500
14-Nov-2001	5	-0.000002	0.014938	0.001637	0.9922	1.00	500
15-Nov-2001	6	0.000001	0.013060	0.001232	0.9940	1.00	500
15-Nov-2001	7	0.000001	0.011552	0.000705	0.9936	1.00	500
16-Nov-2001	8	0.000001	0.016317	0.002517	0.9979	1.00	500
Mean		0.000000	0.012290	0.001244	0.9948		
S.D.		0.000002	0.003564	0.000663	0.0023		
%CV			29.0	53.3	0.2		
n		7	7	7	7		

A, B, and C are coefficients used to define the calibration curve.

Table 8. Back-Calculated Concentrations of Calibration Standards for PFOS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	3.37	4.60	10.8	23.1	43.6	84.6	208	331	413
08-Nov-2001	1	3.44	4.54	10.9	23.0	43.8	84.8	212	326	424
		3.42	4.37	10.9	24.4	43.3	77.8	213	342	393
09-Nov-2001	2	3.58	4.49	11.1	22.9	42.2	87.3	219	336	373
		3.37	4.39	10.4	21.7	41.9	85.6	238	335	403
13-Nov-2001	3	3.57	4.80	10.8	24.9	44.1	84.8	220	338	399
		3.16	4.50	10.3	22.4	41.6	81.8	215	324	410
14-Nov-2001	5	3.07	5.33	10.1	23.8	43.6	82.5	213	317	412
		2.97	5.41	10.2	22.7	42.8	83.6	217	334	416
15-Nov-2001	6	3.46	4.77	10.8	24.1	40.7	85.5	208	338	426
		3.24	4.51	10.7	24.1	42.0	86.3	205	325	402
15-Nov-2001	7	3.06	4.58	10.4	23.1	41.5	77.2	209	314	443
		3.43	5.09	10.9	24.8	45.4	83.7	217	323	407
16-Nov-2001	8	3.63	4.32	11.6	23.8	40.6	80.8	209	328	403
		3.21	4.41	11.4	24.2	44.7	78.3	206	342	425
Mean		3.33	4.68	10.8	23.6	42.7	82.9	214	330	410
S.D.		0.211	0.357	0.440	0.947	1.47	3.27	8.28	8.98	17.0
%CV		6.3	7.6	4.1	4.0	3.4	3.9	3.9	2.7	4.1
%Bias		-1.2	1.7	0.0	2.2	-2.1	-2.0	2.9	-0.3	-0.7
n		14	14	14	14	14	14	14	14	14

Table 9. Back-Calculated Concentrations of Calibration Standards for PFOA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.44	2.88	10.1	24.5	48.5	97.1	241	385	481
08-Nov-2001	1	1.56	3.08	10.1	24.5	49.1	92.3	248	373	470
		1.31	2.73	9.56	26.2	49.4	89.7	255	403	476
09-Nov-2001	2	1.71	2.73	10.2	25.1	50.4	103	251	388	444
		1.26	2.73	9.55	22.5	47.2	97.5	266	390	472
13-Nov-2001	3	1.60	2.90	9.74	23.8	47.7	91.7	245	377	443
		1.27	2.95	9.74	26.2	50.1	96.7	252	414	488
14-Nov-2001	5	1.44	*4.47	9.53	27.5	48.7	99.6	229	380	484
		1.47	*5.21	8.81	24.9	49.4	95.7	250	401	465
15-Nov-2001	6	1.36	2.58	9.29	24.7	43.9	101	244	375	481
		1.56	2.97	11.1	25.5	48.3	99.0	247	391	475
15-Nov-2001	7	1.58	2.90	9.72	25.0	48.9	91.9	246	385	489
		1.33	2.77	9.74	26.1	50.8	95.6	238	389	470
16-Nov-2001	8	1.54	3.12	9.94	25.5	49.2	93.0	233	390	459
		1.30	2.80	9.91	25.5	48.5	93.5	233	408	496
Mean		1.45	2.86	9.78	25.2	48.7	95.7	246	390	472
S.D.		0.145	0.160	0.513	1.19	1.69	3.94	9.83	12.4	15.7
%CV		10.0	5.6	5.2	4.7	3.5	4.1	4.0	3.2	3.3
%Bias		0.7	-0.7	-3.2	2.9	0.4	-1.4	2.1	1.3	-1.9
n		14	12	14	14	14	14	14	14	14

* Sample deactivated as an outlier (not included in summary statistics)

Table 10. Back-Calculated Concentrations of Calibration Standards for PFHS

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.36	2.92	10.7	26.4	52.5	104	261	417	522
08-Nov-2001	1	1.44	2.94	11.5	29.2	53.3	99.8	264	412	540
		1.31	2.72	10.3	26.6	49.3	99.4	260	420	515
09-Nov-2001	2	1.43	2.97	11.2	27.3	53.6	111	282	448	499
		1.33	2.74	10.2	25.2	49.8	98.2	271	398	498
13-Nov-2001	3	1.40	2.95	10.1	30.1	51.5	100	262	420	509
		1.31	2.97	10.6	25.5	50.9	103	278	401	536
14-Nov-2001	5	1.39	*4.07	10.6	28.5	51.2	93.9	304	408	521
		1.33	*4.42	11.1	26.6	49.7	100	262	398	523
15-Nov-2001	6	1.28	2.93	11.2	26.9	52.7	105	263	436	533
		1.46	2.78	11.4	26.1	48.9	99.0	259	409	507
15-Nov-2001	7	1.39	2.76	10.1	26.3	52.4	106	253	394	516
		1.42	2.71	10.7	28.5	55.7	104	265	428	538
16-Nov-2001	8	1.36	3.03	10.6	26.7	49.8	103	264	421	508
		1.34	2.85	10.9	28.6	51.9	97.8	258	426	532
Mean		1.37	2.86	10.8	27.3	51.5	101	268	416	520
S.D.		0.0557	0.115	0.477	1.46	1.94	4.24	13.0	15.7	14.4
%CV		4.1	4.0	4.4	5.3	3.8	4.2	4.9	3.8	2.8
%Bias		0.7	-2.1	0.9	3.4	-1.9	-2.9	2.7	-0.2	-0.4
n		14	12	14	14	14	14	14	14	14

* Sample deactivated as an outlier (not included in summary statistics)

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Table 11. Back-Calculated Concentrations of Calibration Standards for PFOSAA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.50	3.00	10.5	25.5	50.5	101	251	401	501
08-Nov-2001	1	1.23	2.63	10.3	25.2	52.8	111	276	428	509
		1.90	2.87	10.3	26.5	51.0	86.7	251	399	448
09-Nov-2001	2	1.62	2.88	10.8	26.6	53.8	105	266	405	451
		1.51	2.63	9.67	23.7	46.8	98.0	300	416	473
13-Nov-2001	3	1.61	3.00	11.0	24.9	54.1	106	266	435	457
		1.41	2.94	9.72	24.7	49.7	95.7	243	415	488
14-Nov-2001	5	1.40	*4.70	8.98	27.6	48.3	102	207	386	515
		1.38	4.10	8.98	26.7	52.3	99.2	269	442	489
15-Nov-2001	6	1.49	3.11	10.4	28.9	42.5	103	261	411	525
		1.49	2.95	10.6	26.8	48.9	98.9	235	401	477
15-Nov-2001	7	1.49	2.67	9.62	24.0	48.9	93.3	245	361	564
		1.48	3.52	10.4	27.7	53.6	108	275	376	482
16-Nov-2001	8	1.53	2.76	10.6	28.1	53.5	101	255	411	506
		1.62	2.61	10.2	25.8	52.3	91.9	251	398	488
Mean		1.51	2.97	10.1	26.2	50.6	100	257	406	491
S.D.		0.153	0.420	0.629	1.57	3.31	6.63	21.9	22.0	31.4
%CV		10.1	14.1	6.2	6.0	6.5	6.6	8.5	5.4	6.4
%Bias		0.7	-1.0	-3.8	2.7	0.2	-1.0	2.4	1.2	-2.0
n		14	13	14	14	14	14	14	14	14

* Sample deactivated as an outlier (not included in summary statistics)

Table 12. Back-Calculated Concentrations of Calibration Standards for PFOSA

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10.0	25.0	50.0	100	250	400	500
08-Nov-2001	1	0.955	2.45	9.66	23.7	51.9	100	266	396	511
		0.992	2.91	10.0	26.0	48.4	87.5	253	413	472
09-Nov-2001	2	0.831	2.61	9.84	25.5	51.9	100	263	376	487
		1.19	2.33	9.56	23.1	48.6	98.4	298	403	479
13-Nov-2001	3	1.09	2.89	10.6	25.0	56.8	108	258	446	462
		0.875	2.37	8.69	23.2	46.8	91.4	231	423	480
14-Nov-2001	5	1.00	*4.45	8.89	27.4	48.0	98.3	208	395	496
		0.919	3.12	8.63	26.8	52.2	94.6	268	436	502
15-Nov-2001	6	1.09	2.66	10.2	25.7	49.2	100	256	400	525
		0.919	2.27	10.2	25.9	47.4	100	236	406	477
15-Nov-2001	7	1.04	2.45	8.98	25.0	50.5	89.4	236	423	527
		0.934	2.79	9.85	27.1	54.0	94.2	272	385	466
16-Nov-2001	8	1.10	2.66	9.95	27.6	50.7	94.7	249	397	510
		0.915	2.24	9.88	25.7	51.1	94.1	237	418	494
Mean		0.989	2.60	9.64	25.6	50.5	96.5	252	408	492
S.D.		0.101	0.274	0.610	1.45	2.75	5.29	22.0	19.4	20.9
%CV		10.2	10.5	6.3	5.7	5.4	5.5	8.7	4.8	4.2
%Bias		-1.1	4.0	-3.6	2.4	1.0	-3.5	0.8	2.0	-1.6
n		14	13	14	14	14	14	14	14	14

* Sample deactivated as an outlier (not included in summary statistics)

Table 13. Back-Calculated Concentrations of Calibration Standards for M556

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	2.50	10.0	25.0	50.0	100	250	400	500
08-Nov-2001	1	2.26	9.38	23.4	51.9	103	259	410	507
		2.75	10.3	27.3	50.5	87.1	255	405	472
09-Nov-2001	2	2.65	10.1	25.8	50.2	99.3	261	389	456
		2.36	9.91	23.1	46.8	97.5	291	416	494
13-Nov-2001	3	2.59	11.1	25.2	53.6	102	276	430	476
		2.43	8.53	24.1	49.7	92.5	238	404	479
14-Nov-2001	5	*4.38	8.25	30.4	51.8	107	224	396	490
		2.70	7.51	23.5	54.9	101	268	431	476
15-Nov-2001	6	2.48	10.1	24.7	45.5	100	257	407	515
		2.52	10.0	25.1	51.0	106	252	387	480
15-Nov-2001	7	2.31	9.28	25.4	50.5	88.4	251	390	539
		2.72	10.0	24.8	54.2	103	268	388	467
16-Nov-2001	8	2.61	9.97	26.5	46.2	102	245	401	496
		2.34	10.5	26.9	45.1	97.5	252	421	490
Mean		2.52	9.64	25.4	50.1	99.0	257	405	488
S.D.		0.167	0.964	1.92	3.18	6.01	16.3	14.9	21.4
%CV		6.6	10.0	7.6	6.3	6.1	6.3	3.7	4.4
%Bias		0.8	-3.6	1.6	0.2	-1.0	2.8	1.3	-2.4
n		13	14	14	14	14	14	14	14

* Sample deactivated as an outlier (not included in summary statistics)

Table 14. Back-Calculated Concentrations of Calibration Standards for M570

Quadratic weighted $1/x^2$. All concentrations are expressed as ppb.

Run Date	Run Number	1.00	2.50	10. 0	25. 0	50.0	100	250	400	500
08-Nov-2001	1	1.03	2.47	9.84	23.1	50.9	108	263	417	511
		0.987	2.42	10.2	26.3	50.2	89.3	254	397	462
09-Nov-2001	2	1.11	2.52	10.1	25.9	51.4	104	264	403	443
		0.925	2.27	9.96	23.2	45.4	95.9	299	411	487
13-Nov-2001	3	1.08	2.64	10.0	24.1	52.3	102	264	421	450
		0.896	2.58	9.38	24.0	49.6	96.1	255	425	484
14-Nov-2001	5	0.985	*4.20	9.07	28.9	47.3	103	212	399	506
		1.03	*3.85	8.56	27.0	52.2	99.8	275	424	479
15-Nov-2001	6	1.10	2.64	9.91	27.3	41.3	100	271	408	514
		0.906	2.32	10.1	26.8	48.2	102	243	394	475
15-Nov-2001	7	0.930	2.33	9.10	25.1	49.4	90.7	239	373	561
		1.07	2.72	9.95	26.7	52.6	105	276	382	470
16-Nov-2001	8	1.06	2.57	9.97	26.8	49.7	98.8	250	409	506
		0.963	2.28	10.1	25.3	51.9	92.0	244	406	489
Mean		1.01	2.48	9.73	25.8	49.5	99.0	258	405	488
S.D.		0.0736	0.155	0.499	1.69	3.13	5.60	20.7	15.2	30.3
%CV		7.3	6.3	5.1	6.6	6.3	5.7	8.0	3.8	6.2
%Bias		1.0	-0.8	-2.7	3.2	-1.0	-1.0	3.2	1.3	-2.4
n		14	12	14	14	14	14	14	14	14

* Sample deactivated as an outlier (not included in summary statistics)

Table 15. Analytical QC Summary for PFOS

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 5.83 ppb	Medium QC 126 ppb	High QC 331 ppb
Preparation Date		05-Nov-2001	05-Nov-2001	05-Nov-2001
08-Nov-2001	1	5.89	121	336
		5.75	127	349
09-Nov-2001	2	6.63	120	338
		6.50	121	333
13-Nov-2001	3	6.21	134	363
		6.28	118	335
14-Nov-2001	5	5.48	108	303
		5.95	114	322
15-Nov-2001	6	6.16	128	338
		6.20	118	347
15-Nov-2001	7	5.56	114	323
		6.11	117	355
16-Nov-2001	8	6.21	124	330
		6.17	120	323
Mean		6.08	120	335
S.D.		0.325	6.56	15.3
%CV		5.3	5.5	4.6
%Theoretical		104.3	95.2	101.2
%Bias		4.3	-4.8	1.2
n		14	14	14

Table 16. Analytical QC Summary for PFOA

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.32 ppb	Medium QC 144 ppb	High QC 384 ppb
Preparation Date		05-Nov-2001	05-Nov-2001	05-Nov-2001
08-Nov-2001	1	4.04	138	384
		3.80	141	395
09-Nov-2001	2	4.25	137	391
		3.78	138	382
13-Nov-2001	3	4.03	156	436
		4.46	141	430
14-Nov-2001	5	4.16	132	351
		4.63	135	391
15-Nov-2001	6	4.30	139	394
		4.30	136	396
15-Nov-2001	7	4.25	140	379
		4.65	139	399
16-Nov-2001	8	4.30	142	383
		4.33	131	374
Mean		4.23	139	392
S.D.		0.261	5.89	21.3
%CV		6.2	4.2	5.4
%Theoretical		97.9	96.5	102.1
%Bias		-2.1	-3.5	2.1
n		14	14	14

Table 17. Analytical QC Summary for PFHS

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.48 ppb	Medium QC 156 ppb	High QC 417 ppb
Preparation Date		05-Nov-2001	05-Nov-2001	05-Nov-2001
08-Nov-2001	1	4.50	140	404
		4.13	146	409
09-Nov-2001	2	4.55	140	401
		4.61	142	389
13-Nov-2001	3	*6.27	176	443
		4.42	144	433
14-Nov-2001	5	4.82	131	358
		4.15	143	385
15-Nov-2001	6	4.58	144	397
		4.27	129	408
15-Nov-2001	7	4.24	143	407
		4.51	149	415
16-Nov-2001	8	4.49	141	407
		4.39	144	392
Mean		4.57	144	403
S.D.		0.526	10.7	20.5
%CV		11.5	7.4	5.1
%Theoretical		102.0	92.3	96.6
%Bias		2.0	-7.7	-3.4
n		14	14	14

* > ±20% deviation from theoretical

Table 18. Analytical QC Summary for PFOSAA

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.50 ppb	Medium QC 151 ppb	High QC 401 ppb
Preparation Date		05-Nov-2001	05-Nov-2001	05-Nov-2001
08-Nov-2001	1	5.01	151	392
		4.35	149	419
09-Nov-2001	2	5.06	149	400
		4.31	145	401
13-Nov-2001	3	*3.34	150	446
		4.79	149	453
14-Nov-2001	5	3.95	139	399
		4.75	148	421
15-Nov-2001	6	5.03	159	443
		4.32	149	398
15-Nov-2001	7	4.75	139	353
		4.60	129	395
16-Nov-2001	8	4.74	150	390
		4.89	146	387
Mean		4.56	147	407
S.D.		0.478	7.07	27.0
%CV		10.5	4.8	6.6
%Theoretical		101.3	97.4	101.5
%Bias		1.3	-2.6	1.5
n		14	14	14

* > ±25% deviation from theoretical

000631

Table 19. Analytical QC Summary for PFOSA

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb
Preparation Date		05-Nov-2001	05-Nov-2001	05-Nov-2001
08-Nov-2001	1	3.84	149	398
		4.16	149	424
09-Nov-2001	2	4.17	133	393
		3.67	133	404
13-Nov-2001	3	3.09	159	446
		4.22	153	427
14-Nov-2001	5	3.37	140	411
		4.42	139	443
15-Nov-2001	6	4.73	151	412
		3.80	142	395
15-Nov-2001	7	3.82	131	346
		3.80	121	399
16-Nov-2001	8	4.15	142	347
		3.77	135	359
Mean		3.93	141	400
S.D.		0.420	10.3	31.7
%CV		10.7	7.3	7.9
%Theoretical		98.3	94.0	100.0
%Bias		-1.8	-6.0	0.0
n		14	14	14

000632

Table 20. Analytical QC Summary for M556

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb
Preparation Date		05-Nov-2001	05-Nov-2001	05-Nov-2001
08-Nov-2001	1	3.80	140	396
		4.02	145	432
09-Nov-2001	2	4.58	145	408
		4.18	143	414
13-Nov-2001	3	*3.07	151	470
		3.41	146	436
14-Nov-2001	5	3.43	148	383
		3.38	143	419
15-Nov-2001	6	*5.10	157	414
		3.95	148	416
15-Nov-2001	7	3.91	148	387
		4.16	138	419
16-Nov-2001	8	4.53	145	404
		4.53	143	387
Mean		4.00	146	413
S.D.		0.564	4.70	23.0
%CV		14.1	3.2	5.6
%Theoretical		100.0	97.3	103.3
%Bias		0.0	-2.7	3.3
n		14	14	14

* > ±20% deviation from theoretical

000633

Table 21. Analytical QC Summary for M570

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 4.00 ppb	Medium QC 150 ppb	High QC 400 ppb
Preparation Date		05-Nov-2001	05-Nov-2001	05-Nov-2001
08-Nov-2001	1	4.07	149	397
		3.86	146	433
09-Nov-2001	2	4.55	146	405
		4.20	140	404
13-Nov-2001	3	*2.95	143	451
		4.32	149	455
14-Nov-2001	5	3.83	142	388
		4.49	146	429
15-Nov-2001	6	4.49	153	432
		3.74	147	411
15-Nov-2001	7	4.02	141	367
		4.22	132	399
16-Nov-2001	8	4.25	149	403
		4.32	144	391
Mean		4.09	145	412
S.D.		0.415	5.13	25.0
%CV		10.1	3.5	6.1
%Theoretical		102.3	96.7	103.0
%Bias		2.3	-3.3	3.0
n		14	14	14

* > ±20% deviation from theoretical

Table 22. Serum Study Sample Concentrations

All concentrations are expressed as ppb.

Sample Name	PFOS	PFOSA*	PFOSAA*	PFOA*	PFHS*	M556*	M570*
11331	33.7	<LLOQ (1.00)	1.96	6.20	<LLOQ (1.36)	<LLOQ (2.50)	1.30
11332	28.8	<LLOQ (1.00)	<LLOQ (1.50)	4.73	4.50	<LLOQ (2.50)	1.37
11333	44.8	<LLOQ (1.00)	3.33	6.16	3.99	<LLOQ (2.50)	2.22
11334	12.4	<LLOQ (1.00)	1.64	2.47	<LLOQ (1.36)	<LLOQ (2.50)	1.82
11335	18.5	<LLOQ (1.00)	<LLOQ (1.50)	<LLOQ (1.44)	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11336	25.4	<LLOQ (1.00)	<LLOQ (1.50)	2.93	2.67	<LLOQ (2.50)	1.36
11337	29.8	<LLOQ (1.00)	<LLOQ (1.50)	3.56	3.23	<LLOQ (2.50)	<LLOQ (1.00)
11338	33.2	<LLOQ (1.00)	<LLOQ (1.50)	4.03	1.73	<LLOQ (2.50)	1.72
11339	32.3	<LLOQ (1.00)	<LLOQ (1.50)	4.67	2.48	<LLOQ (2.50)	1.29
11340	37.4	<LLOQ (1.00)	2.41	3.96	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11341	35.3	<LLOQ (1.00)	1.50	2.94	7.19	<LLOQ (2.50)	<LLOQ (1.00)
11342	83.2	<LLOQ (1.00)	19.0	4.69	3.20	4.79	3.25
11343	30.8	<LLOQ (1.00)	<LLOQ (1.50)	3.78	2.00	<LLOQ (2.50)	<LLOQ (1.00)
11344	61.9	<LLOQ (1.00)	10.7	7.35	3.35	3.04	2.68
11345	54.9	<LLOQ (1.00)	1.68	11.4	5.00	<LLOQ (2.50)	2.50
11346	34.6	<LLOQ (1.00)	<LLOQ (1.50)	4.66	1.92	<LLOQ (2.50)	1.18
11347	58.9	<LLOQ (1.00)	5.43	7.52	7.78	<LLOQ (2.50)	<LLOQ (1.00)
11348	57.7	<LLOQ (1.00)	2.44	5.01	5.80	<LLOQ (2.50)	1.18
11349	14.5	<LLOQ (1.00)	<LLOQ (1.50)	2.84	<LLOQ (1.36)	<LLOQ (2.50)	1.43
11350	24.5	<LLOQ (1.00)	<LLOQ (1.50)	4.12	1.77	<LLOQ (2.50)	1.38
11351	14.7	<LLOQ (1.00)	<LLOQ (1.50)	10.6	<LLOQ (1.36)	<LLOQ (2.50)	3.55
11352	13.5	<LLOQ (1.00)	4.73	2.37	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11353	22.1	<LLOQ (1.00)	2.48	3.75	5.26	<LLOQ (2.50)	<LLOQ (1.00)
11354	71.3	<LLOQ (1.00)	3.80	12.5	15.9	<LLOQ (2.50)	2.47
11355	48.3	<LLOQ (1.00)	4.88	4.01	<LLOQ (1.36)	<LLOQ (2.50)	1.13
11356	59.7	<LLOQ (1.00)	<LLOQ (1.50)	9.19	2.40	<LLOQ (2.50)	1.26
11357	18.8	<LLOQ (1.00)	2.01	4.03	2.10	<LLOQ (2.50)	1.08
11358	25.2	<LLOQ (1.00)	<LLOQ (1.50)	1.94	10.3	<LLOQ (2.50)	<LLOQ (1.00)
11359	49.7	<LLOQ (1.00)	1.93	6.32	8.53	<LLOQ (2.50)	<LLOQ (1.00)
11360	19.0	<LLOQ (1.00)	4.58	4.27	1.99	<LLOQ (2.50)	<LLOQ (1.00)
11361	16.3	<LLOQ (1.00)	<LLOQ (1.50)	2.58	1.69	<LLOQ (2.50)	<LLOQ (1.00)
11362	95.2	<LLOQ (1.00)	17.7	8.56	3.29	2.80	1.82
11363	133	<LLOQ (1.00)	16.2	16.7	<LLOQ (1.36)	2.89	1.49
11364	49.1	<LLOQ (1.00)	5.30	5.24	2.48	<LLOQ (2.50)	3.76
11365	104	<LLOQ (1.00)	1.95	9.16	2.13	<LLOQ (2.50)	2.74
11366	39.6	<LLOQ (1.00)	1.63	5.13	4.42	<LLOQ (2.50)	2.97

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Serum Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample Name	PFOS	PFOSA*	PFOSAA*	PFOA*	PFHS*	M556*	M570*
11367	13.7	<LLOQ (1.00)	<LLOQ (1.50)	2.20	2.31	<LLOQ (2.50)	<LLOQ (1.00)
11368	10.7	<LLOQ (1.00)	<LLOQ (1.50)	<LLOQ (1.44)	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11369	31.7	<LLOQ (1.00)	<LLOQ (1.50)	2.37	1.63	<LLOQ (2.50)	2.80
11370	30.1	<LLOQ (1.00)	2.13	3.64	1.45	<LLOQ (2.50)	1.05
11371	22.9	<LLOQ (1.00)	2.25	2.75	1.79	<LLOQ (2.50)	1.09
11372	27.8	<LLOQ (1.00)	<LLOQ (1.50)	3.42	1.84	<LLOQ (2.50)	1.13
11373	18.1	<LLOQ (1.00)	2.19	2.35	<LLOQ (1.36)	<LLOQ (2.50)	1.18
11374	40.0	<LLOQ (1.00)	13.8	6.85	2.98	<LLOQ (2.50)	3.25
11375	18.5	<LLOQ (1.00)	<LLOQ (1.50)	3.32	3.04	<LLOQ (2.50)	<LLOQ (1.00)
11376	21.1	<LLOQ (1.00)	<LLOQ (1.50)	1.81	1.36	<LLOQ (2.50)	1.48
11377	71.8	<LLOQ (1.00)	2.22	6.34	17.5	<LLOQ (2.50)	1.77
11378	35.3	<LLOQ (1.00)	3.55	6.19	2.21	<LLOQ (2.50)	2.45
11379	11.2	<LLOQ (1.00)	1.69	2.89	<LLOQ (1.36)	<LLOQ (2.50)	1.00
11380	60.1	<LLOQ (1.00)	<LLOQ (1.50)	4.18	2.39	<LLOQ (2.50)	<LLOQ (1.00)
11381	29.6	<LLOQ (1.00)	<LLOQ (1.50)	2.47	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11382	35.9	<LLOQ (1.00)	<LLOQ (1.50)	5.08	1.58	<LLOQ (2.50)	<LLOQ (1.00)
11383	26.1	<LLOQ (1.00)	1.81	3.70	1.59	<LLOQ (2.50)	3.01
11384	19.3	<LLOQ (1.00)	<LLOQ (1.50)	2.90	1.96	<LLOQ (2.50)	2.46
11385	30.2	<LLOQ (1.00)	<LLOQ (1.50)	4.24	2.49	<LLOQ (2.50)	<LLOQ (1.00)
11386	47.2	<LLOQ (1.00)	8.12	5.17	2.57	<LLOQ (2.50)	<LLOQ (1.00)
11387	53.5	<LLOQ (1.00)	2.28	5.04	2.65	<LLOQ (2.50)	<LLOQ (1.00)
11388	28.9	<LLOQ (1.00)	14.6	4.65	3.95	2.67	2.06
11389	38.3	<LLOQ (1.00)	1.73	3.82	2.40	<LLOQ (2.50)	1.49
11390	31.6	<LLOQ (1.00)	1.64	3.44	1.90	<LLOQ (2.50)	<LLOQ (1.00)
11391	18.6	<LLOQ (1.00)	<LLOQ (1.50)	1.86	2.72	<LLOQ (2.50)	1.27
11392	37.1	<LLOQ (1.00)	<LLOQ (1.50)	2.45	1.88	<LLOQ (2.50)	1.84
11393	25.6	<LLOQ (1.00)	<LLOQ (1.50)	3.20	1.55	<LLOQ (2.50)	<LLOQ (1.00)
11394	43.2	<LLOQ (1.00)	2.62	3.98	3.63	<LLOQ (2.50)	5.69
11395	24.2	<LLOQ (1.00)	5.16	3.47	1.87	<LLOQ (2.50)	<LLOQ (1.00)
11396	73.7	<LLOQ (1.00)	5.62	6.92	8.28	<LLOQ (2.50)	<LLOQ (1.00)
11397	41.7	<LLOQ (1.00)	<LLOQ (1.50)	4.30	2.48	<LLOQ (2.50)	1.02
11398	45.7	<LLOQ (1.00)	2.27	5.50	1.94	<LLOQ (2.50)	<LLOQ (1.00)
11399	10.7	<LLOQ (1.00)	<LLOQ (1.50)	2.44	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11400	22.1	<LLOQ (1.00)	<LLOQ (1.50)	3.00	<LLOQ (1.36)	<LLOQ (2.50)	1.31
11401	44.5	<LLOQ (1.00)	3.24	5.03	5.12	<LLOQ (2.50)	<LLOQ (1.00)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Serum Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample Name	PFOS	PFOSA*	PFOSAA*	PFOA*	PFHS*	M556*	M570*
11402	51.5	<LLOQ (1.00)	<LLOQ (1.50)	6.35	9.74	<LLOQ (2.50)	1.39
11403	21.7	<LLOQ (1.00)	2.94	4.57	1.80	<LLOQ (2.50)	2.08
11404	29.4	<LLOQ (1.00)	<LLOQ (1.50)	2.99	3.42	<LLOQ (2.50)	<LLOQ (1.00)
11405	22.3	<LLOQ (1.00)	1.74	2.19	1.43	<LLOQ (2.50)	2.13
11406	40.6	<LLOQ (1.00)	<LLOQ (1.50)	3.56	4.57	<LLOQ (2.50)	<LLOQ (1.00)
11407	40.6	<LLOQ (1.00)	<LLOQ (1.50)	7.00	7.00	<LLOQ (2.50)	1.01
11408	27.0	<LLOQ (1.00)	2.06	3.57	2.46	<LLOQ (2.50)	<LLOQ (1.00)
11409	13.8	<LLOQ (1.00)	<LLOQ (1.50)	4.17	2.22	<LLOQ (2.50)	1.13
11410	104	<LLOQ (1.00)	2.57	10.2	2.33	<LLOQ (2.50)	4.05
11411	25.1	<LLOQ (1.00)	3.33	<LLOQ (1.44)	1.71	<LLOQ (2.50)	<LLOQ (1.00)
11412	26.5	<LLOQ (1.00)	<LLOQ (1.50)	3.14	8.74	<LLOQ (2.50)	2.71
11413	34.1	<LLOQ (1.00)	<LLOQ (1.50)	4.44	1.40	<LLOQ (2.50)	<LLOQ (1.00)
11414	18.0	<LLOQ (1.00)	<LLOQ (1.50)	3.84	6.32	<LLOQ (2.50)	<LLOQ (1.00)
11415	61.3	<LLOQ (1.00)	3.79	7.53	6.43	<LLOQ (2.50)	<LLOQ (1.00)
11416	22.1	<LLOQ (1.00)	<LLOQ (1.50)	2.28	<LLOQ (1.36)	<LLOQ (2.50)	1.44
11417	58.9	<LLOQ (1.00)	<LLOQ (1.50)	5.67	17.1	<LLOQ (2.50)	1.83
11418	25.6	<LLOQ (1.00)	2.43	4.69	2.78	<LLOQ (2.50)	1.14
11419	26.3	<LLOQ (1.00)	2.23	4.30	1.57	<LLOQ (2.50)	1.62
11420	27.2	<LLOQ (1.00)	<LLOQ (1.50)	2.27	2.02	<LLOQ (2.50)	1.95
11421	61.3	<LLOQ (1.00)	10.5	8.76	6.97	<LLOQ (2.50)	3.31
11422	<LLOQ (3.37)	<LLOQ (1.00)	<LLOQ (1.50)	<LLOQ (1.44)	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11423	37.3	<LLOQ (1.00)	<LLOQ (1.50)	4.90	3.52	<LLOQ (2.50)	<LLOQ (1.00)
11424	25.4	<LLOQ (1.00)	5.31	6.62	1.92	<LLOQ (2.50)	4.74
11425	15.1	<LLOQ (1.00)	<LLOQ (1.50)	3.43	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11426	25.8	<LLOQ (1.00)	<LLOQ (1.50)	2.64	6.22	<LLOQ (2.50)	3.48
11427	41.6	<LLOQ (1.00)	<LLOQ (1.50)	8.98	2.66	<LLOQ (2.50)	6.30
11428	29.3	<LLOQ (1.00)	2.94	5.90	1.68	<LLOQ (2.50)	3.51
11429	29.7	<LLOQ (1.00)	<LLOQ (1.50)	4.44	4.74	<LLOQ (2.50)	1.63
11430	41.1	<LLOQ (1.00)	<LLOQ (1.50)	4.08	2.15	<LLOQ (2.50)	<LLOQ (1.00)
11431	26.3	<LLOQ (1.00)	<LLOQ (1.50)	2.56	1.70	<LLOQ (2.50)	3.71
11432	114	<LLOQ (1.00)	4.63	14.4	2.32	<LLOQ (2.50)	3.03
11433	48.8	<LLOQ (1.00)	<LLOQ (1.50)	9.17	7.42	<LLOQ (2.50)	6.15
11434	30.7	<LLOQ (1.00)	1.87	6.89	2.22	<LLOQ (2.50)	1.16
11435	24.3	<LLOQ (1.00)	3.14	3.72	1.59	<LLOQ (2.50)	<LLOQ (1.00)
11436	14.6	<LLOQ (1.00)	<LLOQ (1.50)	2.82	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Serum Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample Name	PFOS	PFOSA*	PFOSAA*	PFOA*	PFHS*	M556*	M570*
11437	20.2	<LLOQ (1.00)	5.89	2.63	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11438	32.8	<LLOQ (1.00)	<LLOQ (1.50)	6.47	3.22	<LLOQ (2.50)	1.55
11439	49.9	<LLOQ (1.00)	8.95	5.96	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11440	15.4	<LLOQ (1.00)	<LLOQ (1.50)	3.33	2.05	<LLOQ (2.50)	1.28
11441	20.3	<LLOQ (1.00)	<LLOQ (1.50)	4.30	2.15	<LLOQ (2.50)	<LLOQ (1.00)
11442	12.2	<LLOQ (1.00)	<LLOQ (1.50)	1.84	2.65	<LLOQ (2.50)	<LLOQ (1.00)
11443	34.6	<LLOQ (1.00)	2.26	4.54	4.25	<LLOQ (2.50)	1.70
11444	17.2	<LLOQ (1.00)	<LLOQ (1.50)	2.33	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11445	11.7	<LLOQ (1.00)	<LLOQ (1.50)	1.87	3.71	<LLOQ (2.50)	<LLOQ (1.00)
11446	29.2	<LLOQ (1.00)	<LLOQ (1.50)	3.45	2.30	<LLOQ (2.50)	<LLOQ (1.00)
11447	20.4	<LLOQ (1.00)	<LLOQ (1.50)	2.99	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11448	13.4	<LLOQ (1.00)	<LLOQ (1.50)	2.78	<LLOQ (1.36)	<LLOQ (2.50)	1.18
11449	36.7	<LLOQ (1.00)	3.76	4.05	2.69	<LLOQ (2.50)	2.86
11450	36.8	<LLOQ (1.00)	<LLOQ (1.50)	3.18	1.72	<LLOQ (2.50)	2.58
11451	50.8	<LLOQ (1.00)	<LLOQ (1.50)	4.87	1.80	<LLOQ (2.50)	<LLOQ (1.00)
11452	39.0	<LLOQ (1.00)	<LLOQ (1.50)	2.85	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11453	34.3	<LLOQ (1.00)	2.86	4.44	1.47	<LLOQ (2.50)	<LLOQ (1.00)
11454	29.0	<LLOQ (1.00)	2.18	4.19	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11455	27.6	<LLOQ (1.00)	1.62	2.48	<LLOQ (1.36)	<LLOQ (2.50)	1.16
11456	16.4	<LLOQ (1.00)	2.40	3.42	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11457	138	<LLOQ (1.00)	21.1	11.3	<LLOQ (1.36)	2.82	1.22
11458	19.0	<LLOQ (1.00)	1.57	2.76	<LLOQ (1.36)	<LLOQ (2.50)	2.86
11459	46.1	<LLOQ (1.00)	<LLOQ (1.50)	4.81	5.25	<LLOQ (2.50)	<LLOQ (1.00)
11460	51.6	<LLOQ (1.00)	2.81	6.17	1.58	<LLOQ (2.50)	1.52
11461	42.9	<LLOQ (1.00)	1.60	2.21	5.44	<LLOQ (2.50)	4.94
11462	175	<LLOQ (1.00)	2.46	9.25	17.0	<LLOQ (2.50)	1.01
11463	9.06	<LLOQ (1.00)	1.79	4.35	4.90	<LLOQ (2.50)	1.02
11464	26.7	<LLOQ (1.00)	<LLOQ (1.50)	3.02	2.74	<LLOQ (2.50)	1.71
11465	17.5	<LLOQ (1.00)	2.00	2.99	<LLOQ (1.36)	<LLOQ (2.50)	2.00
11466	33.6	<LLOQ (1.00)	<LLOQ (1.50)	4.40	4.23	<LLOQ (2.50)	1.75
11467	32.6	<LLOQ (1.00)	1.78	5.41	4.00	<LLOQ (2.50)	2.81
11468	64.7	<LLOQ (1.00)	2.77	6.84	1.59	<LLOQ (2.50)	4.23
11469	28.1	<LLOQ (1.00)	1.77	3.96	2.16	<LLOQ (2.50)	<LLOQ (1.00)
11470	22.0	<LLOQ (1.00)	<LLOQ (1.50)	1.81	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11471	61.4	<LLOQ (1.00)	6.75	9.95	1.64	<LLOQ (2.50)	2.87
11472	28.3	<LLOQ (1.00)	<LLOQ (1.50)	4.22	3.56	<LLOQ (2.50)	<LLOQ (1.00)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Serum Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample Name	PFOS	PFOSA*	PFOSAA*	PFOA*	PFHS*	M556*	M570*
11473	32.1	<LLOQ (1.00)	<LLOQ (1.50)	3.60	2.67	<LLOQ (2.50)	1.67
11474	27.9	<LLOQ (1.00)	<LLOQ (1.50)	6.01	3.66	<LLOQ (2.50)	2.38
11475	27.4	<LLOQ (1.00)	<LLOQ (1.50)	3.59	5.79	<LLOQ (2.50)	1.09
11476	18.9	<LLOQ (1.00)	<LLOQ (1.50)	3.21	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11477	38.1	<LLOQ (1.00)	1.96	6.16	2.38	<LLOQ (2.50)	1.84
11478	47.5	<LLOQ (1.00)	1.61	6.97	3.19	<LLOQ (2.50)	1.52
11479	28.4	<LLOQ (1.00)	<LLOQ (1.50)	4.44	6.50	<LLOQ (2.50)	1.99
11480	48.4	<LLOQ (1.00)	6.49	7.22	1.69	<LLOQ (2.50)	1.54
11481	26.0	<LLOQ (1.00)	<LLOQ (1.50)	4.74	3.64	<LLOQ (2.50)	1.70
11482	19.7	<LLOQ (1.00)	<LLOQ (1.50)	3.15	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11483	47.2	<LLOQ (1.00)	2.50	6.41	2.06	<LLOQ (2.50)	1.43
11484	72.5	<LLOQ (1.00)	7.66	7.20	<LLOQ (1.36)	4.09	1.82
11485	33.6	<LLOQ (1.00)	<LLOQ (1.50)	4.21	3.72	<LLOQ (2.50)	1.08
11486	31.8	<LLOQ (1.00)	<LLOQ (1.50)	3.69	1.95	<LLOQ (2.50)	<LLOQ (1.00)
11487	37.3	<LLOQ (1.00)	3.40	6.65	3.21	<LLOQ (2.50)	3.39
11488	25.2	<LLOQ (1.00)	1.68	5.40	3.78	<LLOQ (2.50)	1.43
11489	21.4	<LLOQ (1.00)	<LLOQ (1.50)	3.01	<LLOQ (1.36)	<LLOQ (2.50)	1.41
11490	29.6	<LLOQ (1.00)	2.67	7.23	<LLOQ (1.36)	<LLOQ (2.50)	3.44
11491	91.5	<LLOQ (1.00)	1.85	10.5	5.42	<LLOQ (2.50)	1.50
11492	50.2	<LLOQ (1.00)	<LLOQ (1.50)	4.16	3.12	<LLOQ (2.50)	1.67
11493	24.4	<LLOQ (1.00)	1.82	4.75	2.47	<LLOQ (2.50)	1.53
11494	47.0	<LLOQ (1.00)	4.38	5.95	2.62	<LLOQ (2.50)	2.23
11495	48.6	<LLOQ (1.00)	1.53	7.78	3.15	<LLOQ (2.50)	1.48
11496	47.3	<LLOQ (1.00)	1.82	4.62	5.50	<LLOQ (2.50)	<LLOQ (1.00)
11497	76.4	<LLOQ (1.00)	<LLOQ (1.50)	8.95	2.27	<LLOQ (2.50)	1.44
11498	34.2	<LLOQ (1.00)	2.57	3.72	2.77	<LLOQ (2.50)	1.45
11499	57.0	<LLOQ (1.00)	5.54	5.38	10.6	<LLOQ (2.50)	1.78
11500	20.5	<LLOQ (1.00)	<LLOQ (1.50)	3.96	<LLOQ (1.36)	<LLOQ (2.50)	1.60
11501	17.2	<LLOQ (1.00)	<LLOQ (1.50)	2.21	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11502	42.2	<LLOQ (1.00)	<LLOQ (1.50)	7.45	2.55	<LLOQ (2.50)	1.97
11503	56.1	<LLOQ (1.00)	1.58	6.90	40.3	<LLOQ (2.50)	6.56
11504	30.5	<LLOQ (1.00)	<LLOQ (1.50)	3.52	3.94	<LLOQ (2.50)	1.10
11505	104	<LLOQ (1.00)	2.29	7.81	3.85	<LLOQ (2.50)	2.08
11506	44.0	<LLOQ (1.00)	2.10	8.07	6.92	<LLOQ (2.50)	1.90
11507	32.1	<LLOQ (1.00)	2.31	3.89	2.21	<LLOQ (2.50)	1.01
11508	24.3	<LLOQ (1.00)	<LLOQ (1.50)	2.57	<LLOQ (1.36)	<LLOQ (2.50)	1.49

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Serum Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample Name	PFOS	PFOSA*	PFOSAA*	PFOA*	PFHS*	M556*	M570*
11509	22.9	<LLOQ (1.00)	1.59	5.53	2.23	<LLOQ (2.50)	<LLOQ (1.00)
11510	46.7	<LLOQ (1.00)	<LLOQ (1.50)	6.62	5.35	<LLOQ (2.50)	1.13
11511	161	<LLOQ (1.00)	5.99	14.2	4.61	<LLOQ (2.50)	<LLOQ (1.00)
11512	54.4	<LLOQ (1.00)	<LLOQ (1.50)	5.07	3.30	<LLOQ (2.50)	2.87
11513	14.5	<LLOQ (1.00)	3.03	4.58	8.03	<LLOQ (2.50)	1.20
11514	20.4	<LLOQ (1.00)	1.58	2.12	<LLOQ (1.36)	<LLOQ (2.50)	2.86
11515	26.5	<LLOQ (1.00)	<LLOQ (1.50)	2.97	4.86	<LLOQ (2.50)	1.54
11516	37.6	<LLOQ (1.00)	<LLOQ (1.50)	5.83	4.35	<LLOQ (2.50)	1.05
11517	49.2	<LLOQ (1.00)	<LLOQ (1.50)	9.56	2.03	<LLOQ (2.50)	2.48
11518	8.50	<LLOQ (1.00)	<LLOQ (1.50)	1.57	3.72	<LLOQ (2.50)	<LLOQ (1.00)
11519	19.5	<LLOQ (1.00)	<LLOQ (1.50)	2.10	2.35	<LLOQ (2.50)	1.53
11520	25.3	<LLOQ (1.00)	1.92	3.63	<LLOQ (1.36)	<LLOQ (2.50)	2.70
11521	18.6	<LLOQ (1.00)	1.83	2.48	1.71	<LLOQ (2.50)	<LLOQ (1.00)
11522	35.2	<LLOQ (1.00)	<LLOQ (1.50)	5.36	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11523	28.7	<LLOQ (1.00)	1.80	4.73	2.39	<LLOQ (2.50)	1.21
11524	20.1	<LLOQ (1.00)	<LLOQ (1.50)	3.48	1.37	<LLOQ (2.50)	<LLOQ (1.00)
11525	23.9	<LLOQ (1.00)	<LLOQ (1.50)	3.61	1.38	<LLOQ (2.50)	<LLOQ (1.00)
11526	49.8	<LLOQ (1.00)	2.30	5.51	6.41	<LLOQ (2.50)	<LLOQ (1.00)
11527	15.4	<LLOQ (1.00)	<LLOQ (1.50)	2.82	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11528	31.1	<LLOQ (1.00)	<LLOQ (1.50)	4.43	3.99	<LLOQ (2.50)	2.05
11529	15.6	<LLOQ (1.00)	<LLOQ (1.50)	2.15	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11530	19.3	<LLOQ (1.00)	<LLOQ (1.50)	3.18	2.71	<LLOQ (2.50)	<LLOQ (1.00)
11531	21.2	<LLOQ (1.00)	<LLOQ (1.50)	2.99	<LLOQ (1.36)	<LLOQ (2.50)	2.46
11532	40.4	<LLOQ (1.00)	1.62	4.76	2.16	<LLOQ (2.50)	2.57
11533	25.5	<LLOQ (1.00)	<LLOQ (1.50)	1.88	1.46	<LLOQ (2.50)	<LLOQ (1.00)
11534	20.5	<LLOQ (1.00)	<LLOQ (1.50)	2.20	1.38	<LLOQ (2.50)	<LLOQ (1.00)
11535	23.2	<LLOQ (1.00)	<LLOQ (1.50)	2.62	1.49	<LLOQ (2.50)	<LLOQ (1.00)
11536	38.6	<LLOQ (1.00)	1.86	6.95	9.60	<LLOQ (2.50)	1.26
11537	15.4	<LLOQ (1.00)	<LLOQ (1.50)	2.43	<LLOQ (1.36)	<LLOQ (2.50)	1.45
11538	33.4	<LLOQ (1.00)	<LLOQ (1.50)	2.38	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11539	32.9	<LLOQ (1.00)	<LLOQ (1.50)	4.40	2.28	<LLOQ (2.50)	<LLOQ (1.00)
11540	15.7	<LLOQ (1.00)	<LLOQ (1.50)	2.25	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
11541	24.4	<LLOQ (1.00)	<LLOQ (1.50)	3.69	21.8	<LLOQ (2.50)	3.10
11542	40.9	<LLOQ (1.00)	<LLOQ (1.50)	3.77	2.68	<LLOQ (2.50)	2.00
GH11281	36.2	<LLOQ (1.00)	<LLOQ (1.50)	5.91	3.10	<LLOQ (2.50)	2.13
GH11282	18.2	<LLOQ (1.00)	1.68	2.58	<LLOQ (1.36)	<LLOQ (2.50)	2.49

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Serum Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample Name	PFOS	PFOSA*	PFOSAA*	PFOA*	PFHS*	M556*	M570*
GH11283	13.8	<LLOQ (1.00)	<LLOQ (1.50)	2.29	3.37	<LLOQ (2.50)	<LLOQ (1.00)
GH11284	18.7	<LLOQ (1.00)	<LLOQ (1.50)	3.60	5.20	<LLOQ (2.50)	1.13
GH11285	21.1	<LLOQ (1.00)	<LLOQ (1.50)	3.82	1.96	<LLOQ (2.50)	2.52
GH11286	29.4	<LLOQ (1.00)	3.09	3.69	1.83	<LLOQ (2.50)	<LLOQ (1.00)
GH11287	52.0	<LLOQ (1.00)	1.74	4.55	<LLOQ (1.36)	<LLOQ (2.50)	4.24
GH11288	40.5	<LLOQ (1.00)	<LLOQ (1.50)	7.41	1.78	<LLOQ (2.50)	1.78
GH11289	37.7	<LLOQ (1.00)	1.81	6.69	2.94	<LLOQ (2.50)	2.62
GH11290	22.7	<LLOQ (1.00)	3.25	4.13	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
GH11291	41.3	<LLOQ (1.00)	1.54	4.53	8.76	<LLOQ (2.50)	1.04
GH11292	62.6	<LLOQ (1.00)	2.09	6.56	5.25	<LLOQ (2.50)	3.48
GH11293	9.58	<LLOQ (1.00)	5.06	2.04	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
GH11294	20.3	<LLOQ (1.00)	<LLOQ (1.50)	5.59	1.70	<LLOQ (2.50)	1.61
GH11295	16.3	<LLOQ (1.00)	<LLOQ (1.50)	<LLOQ (1.44)	7.23	<LLOQ (2.50)	2.82
GH11296	30.0	<LLOQ (1.00)	4.29	5.24	3.08	<LLOQ (2.50)	1.91
GH11297	21.8	<LLOQ (1.00)	1.53	3.96	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
GH11298	54.3	<LLOQ (1.00)	1.68	6.52	2.84	<LLOQ (2.50)	4.11
GH11299	15.4	<LLOQ (1.00)	<LLOQ (1.50)	2.55	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
GH11300	27.2	<LLOQ (1.00)	1.77	4.28	1.85	<LLOQ (2.50)	1.02
GH11301	27.1	<LLOQ (1.00)	2.61	6.88	3.18	<LLOQ (2.50)	2.09
GH11302	15.7	<LLOQ (1.00)	<LLOQ (1.50)	3.17	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
GH11303	30.5	<LLOQ (1.00)	1.61	3.73	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
GH11304	16.2	<LLOQ (1.00)	3.75	1.79	2.03	<LLOQ (2.50)	1.49
GH11305	11.7	<LLOQ (1.00)	2.24	3.44	<LLOQ (1.36)	<LLOQ (2.50)	1.43
GH11306	21.2	<LLOQ (1.00)	<LLOQ (1.50)	3.22	<LLOQ (1.36)	<LLOQ (2.50)	2.37
GH11307	36.1	<LLOQ (1.00)	<LLOQ (1.50)	3.92	<LLOQ (1.36)	<LLOQ (2.50)	1.40
GH11308	35.7	<LLOQ (1.00)	2.05	3.34	1.90	<LLOQ (2.50)	2.52
GH11309	21.2	<LLOQ (1.00)	<LLOQ (1.50)	3.04	10.6	<LLOQ (2.50)	<LLOQ (1.00)
GH11310	44.8	<LLOQ (1.00)	1.64	3.50	2.01	<LLOQ (2.50)	1.77
GH11311	103	<LLOQ (1.00)	7.65	15.0	6.84	<LLOQ (2.50)	2.79
GH11312	13.4	<LLOQ (1.00)	<LLOQ (1.50)	3.47	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
GH11313	27.4	<LLOQ (1.00)	<LLOQ (1.50)	4.08	2.74	<LLOQ (2.50)	2.12
GH11314	26.0	<LLOQ (1.00)	<LLOQ (1.50)	4.59	2.75	<LLOQ (2.50)	5.07
GH11315	31.8	<LLOQ (1.00)	<LLOQ (1.50)	3.81	2.11	<LLOQ (2.50)	<LLOQ (1.00)
GH11316	27.1	<LLOQ (1.00)	<LLOQ (1.50)	3.86	2.78	<LLOQ (2.50)	1.59
GH11317	39.7	<LLOQ (1.00)	2.06	5.33	3.90	<LLOQ (2.50)	1.75
GH11318	37.2	<LLOQ (1.00)	1.66	3.56	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Table 22. Serum Study Sample Concentrations (Continued)

All concentrations are expressed as ppb.

Sample Name	PFOS	PFOSA*	PFOSAA*	PFOA*	PFHS*	M556*	M570*
GH11319	34.1	<LLOQ (1.00)	<LLOQ (1.50)	4.24	2.55	<LLOQ (2.50)	1.29
GH11320	21.6	<LLOQ (1.00)	<LLOQ (1.50)	3.91	2.97	<LLOQ (2.50)	<LLOQ (1.00)
GH11321	43.5	<LLOQ (1.00)	6.67	6.96	2.19	<LLOQ (2.50)	1.04
GH11322	104	<LLOQ (1.00)	9.85	15.4	3.73	2.75	1.06
GH11323	10.9	<LLOQ (1.00)	2.94	2.12	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
GH11324	27.6	<LLOQ (1.00)	2.69	4.55	3.58	<LLOQ (2.50)	1.30
GH11325	17.7	<LLOQ (1.00)	<LLOQ (1.50)	2.96	<LLOQ (1.36)	<LLOQ (2.50)	1.16
GH11326	45.6	<LLOQ (1.00)	11.1	5.89	6.02	<LLOQ (2.50)	2.06
GH11327	69.6	<LLOQ (1.00)	2.37	8.38	2.39	<LLOQ (2.50)	<LLOQ (1.00)
GH11328	36.4	<LLOQ (1.00)	<LLOQ (1.50)	3.35	<LLOQ (1.36)	<LLOQ (2.50)	<LLOQ (1.00)
GH11329	12.8	<LLOQ (1.00)	<LLOQ (1.50)	3.22	<LLOQ (1.36)	<LLOQ (2.50)	1.53
GH11330	27.3	<LLOQ (1.00)	<LLOQ (1.50)	3.03	1.92	<LLOQ (2.50)	<LLOQ (1.00)

NOTE: The method acceptance criteria states that the measured concentration for each QC must be within ±20% (±25% for PFOSA and PFOSAA) of the target concentration. The statistical error associated with any individual result may exceed the QC acceptance criteria.

* Serum sample results for PFOA, PFHS, PFOSAA, PFOSA, M556 and M570 obtained using plasma calibration curves may vary from results obtained using serum calibration curves (see Results and Discussion section).

Figure 1. Representative Calibration Curve for PFOS

Analytical Run 6 analyzed on 15-Nov-2001 Calibration Standards for PFOS (ppb)
Regression Method = QUADRATIC - Weighting Factor = 1/X**2
Quadratic Limit = 4300

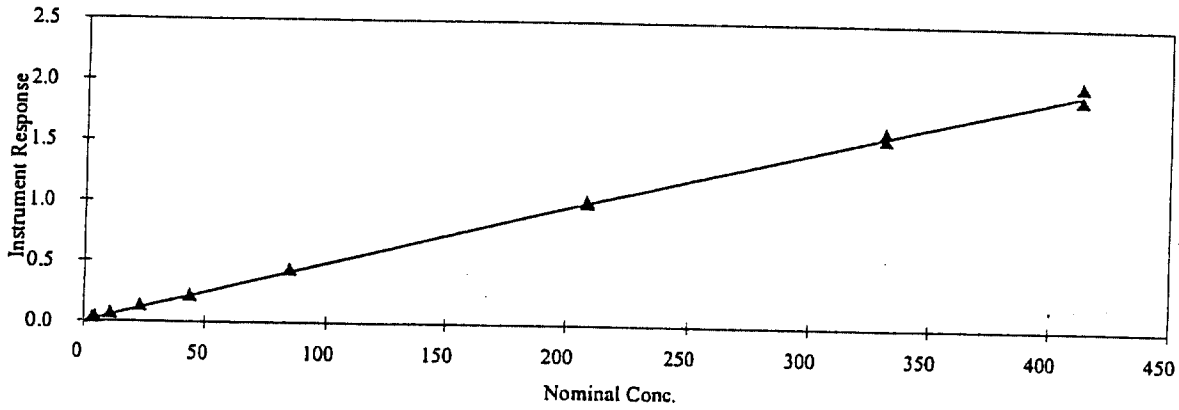


Figure 2. Representative Calibration Curve for PFOA

Analytical Run 6 analyzed on 15-Nov-2001 Calibration Standards for PFOA (ppb)
Regression Method = QUADRATIC - Weighting Factor = 1/X**2
Quadratic Limit = -4270

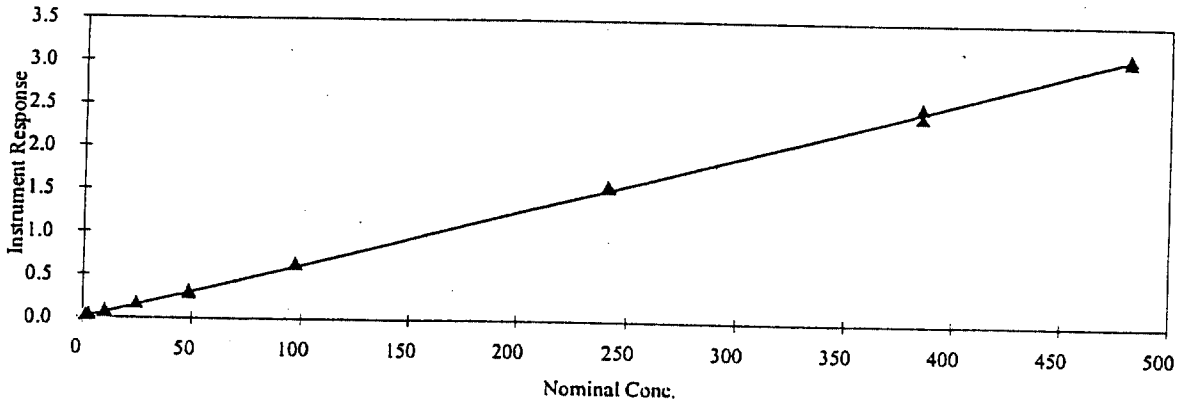


Figure 3. Representative Calibration Curve for PFHS

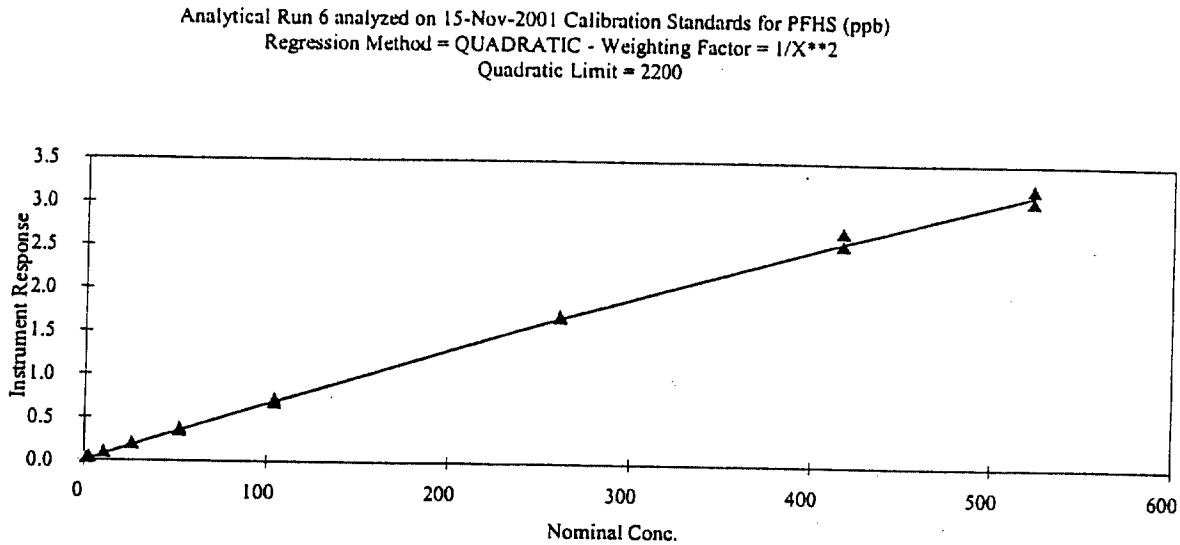
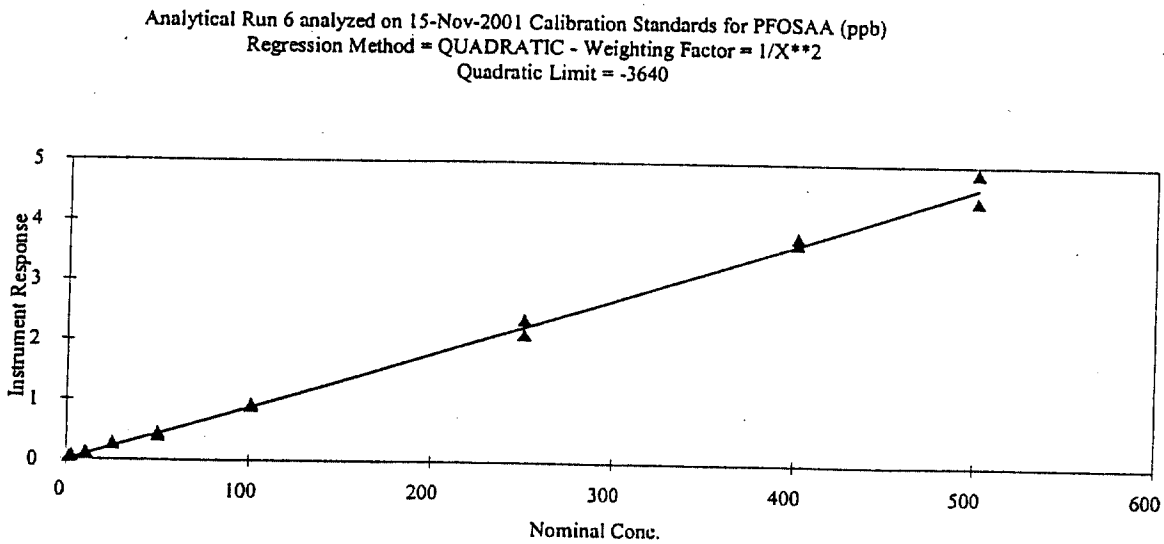


Figure 4. Representative Calibration Curve for PFOSAA



000644

Figure 5. Representative Calibration Curve for PFOSA

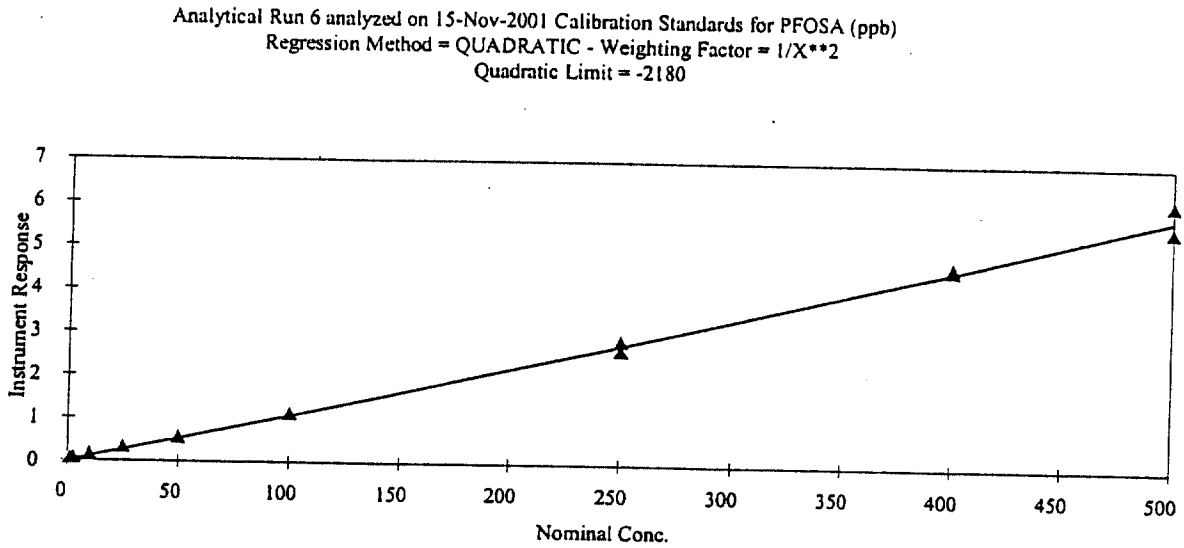


Figure 6. Representative Calibration Curve for M556

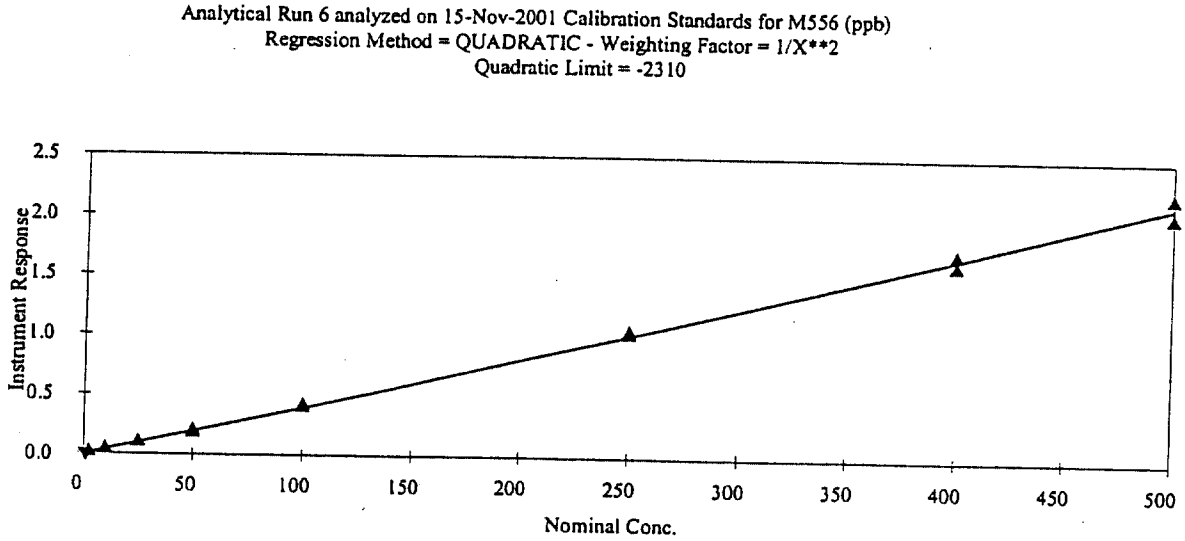


Figure 7. Representative Calibration Curve for M570

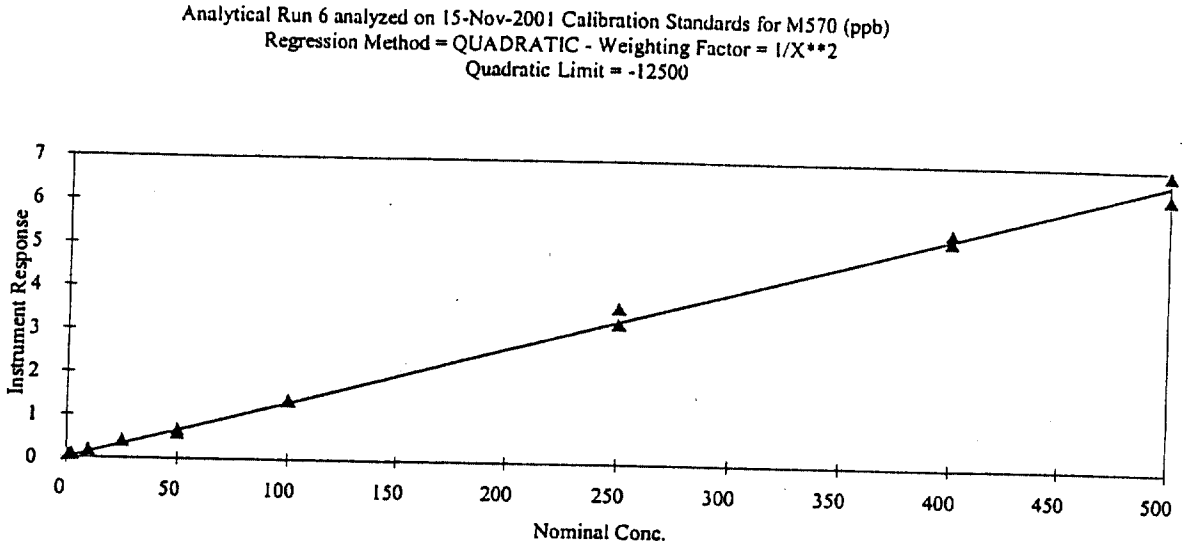


Figure 8. Human Plasma Blank for PFOS

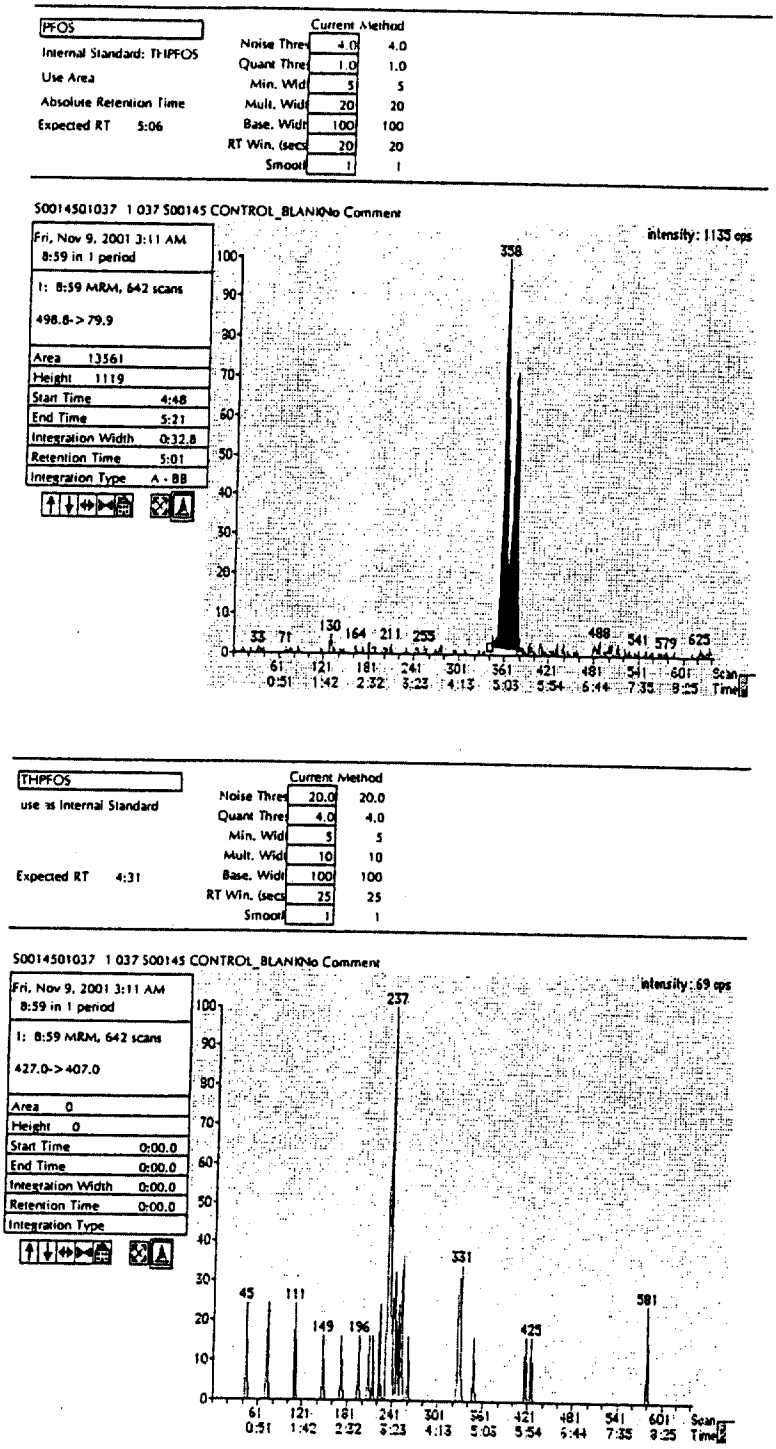


Figure 9. Human Plasma Blank for PFOA

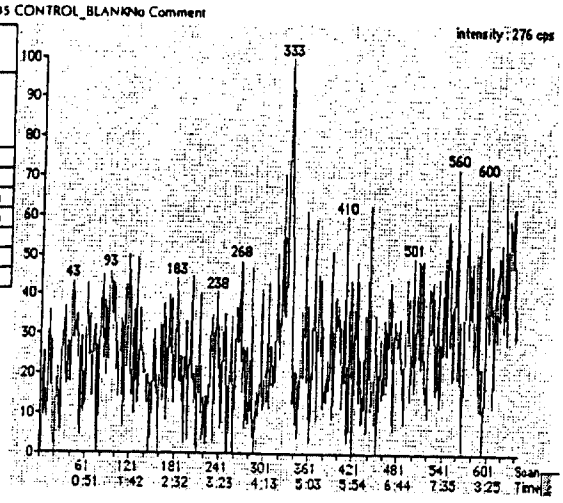
PFOA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:37

	Current	Method
Noise Thres	10.0	10.0
Quant Thres	3.0	3.0
Min. Wid	8	8
Mult. Wid	16	16
Base. Wid	100	100
RT Win. (secs	26	26
Smooth	1	1

S0014501037 1 037 S00145 CONTROL_BLANKNo Comment

Fri, Nov 9, 2001 3:11 AM
8:59 in 1 period
1: 8:59 MRM, 642 scans
413.0->169.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	



THPFOS
use as Internal Standard
Expected RT 4:31

	Current	Method
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smooth	1	1

S0014501037 1 037 S00145 CONTROL_BLANKNo Comment

Fri, Nov 9, 2001 3:11 AM
8:59 in 1 period
1: 8:59 MRM, 642 scans
427.0->407.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	

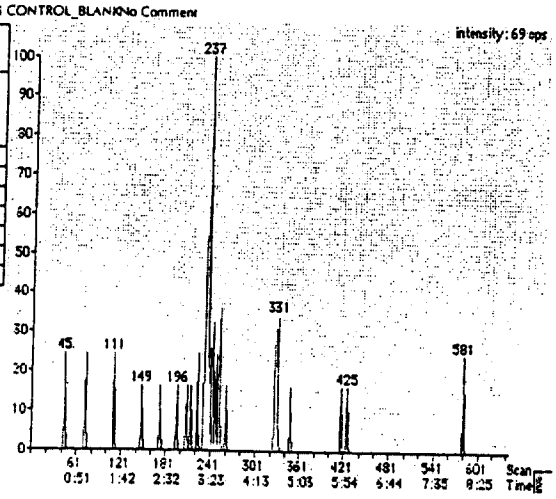
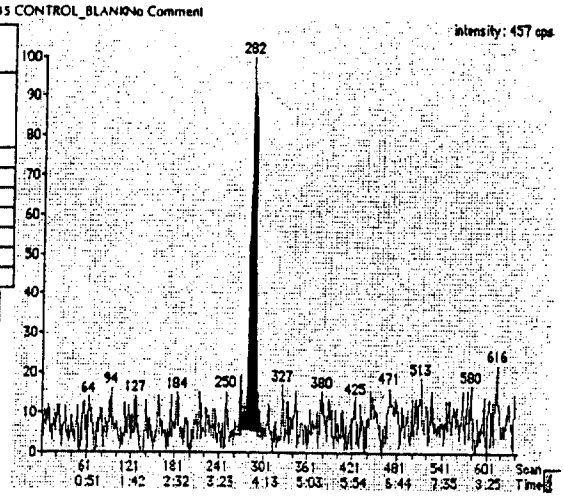


Figure 10. Human Plasma Blank for PFHS

PFHS		Current Method
Internal Standard: THPFOS	Noise Thre	5.0	5.0
Use Area	Quant Thre	1.5	1.5
Absolute Retention Time	Min. Wid	5	5
Expected RT 3:56	Mult. Wid	18	18
	Base. Wid	100	100
	RT Win. (secs	50	50
	Smooth	1	1

S0014501037 1 037 S00145 CONTROL_BLANKNo Comment

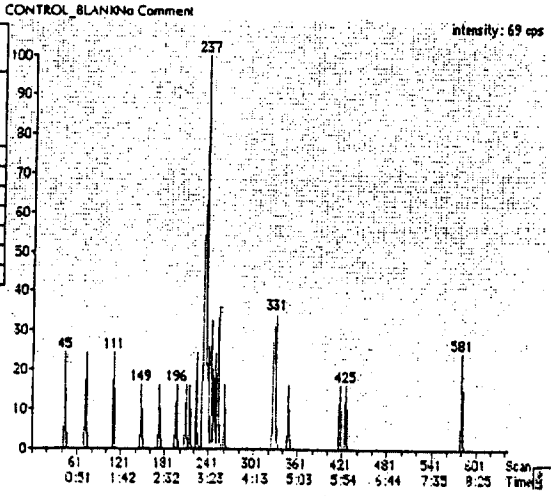
Fri, Nov 9, 2001 3:11 AM 8:59 in 1 period	
1: 8:59 MRM, 642 scans 399.0->80.0	
Area	3715
Height	434
Start Time	3:44
End Time	4:10
Integration Width	0:26.1
Retention Time	3:57
Integration Type	A - BB



THPFOS		Current Method
use as Internal Standard	Noise Thre	20.0	20.0
	Quant Thre	4.0	4.0
	Min. Wid	5	5
	Mult. Wid	10	10
Expected RT 4:31	Base. Wid	100	100
	RT Win. (secs	25	25
	Smooth	1	1

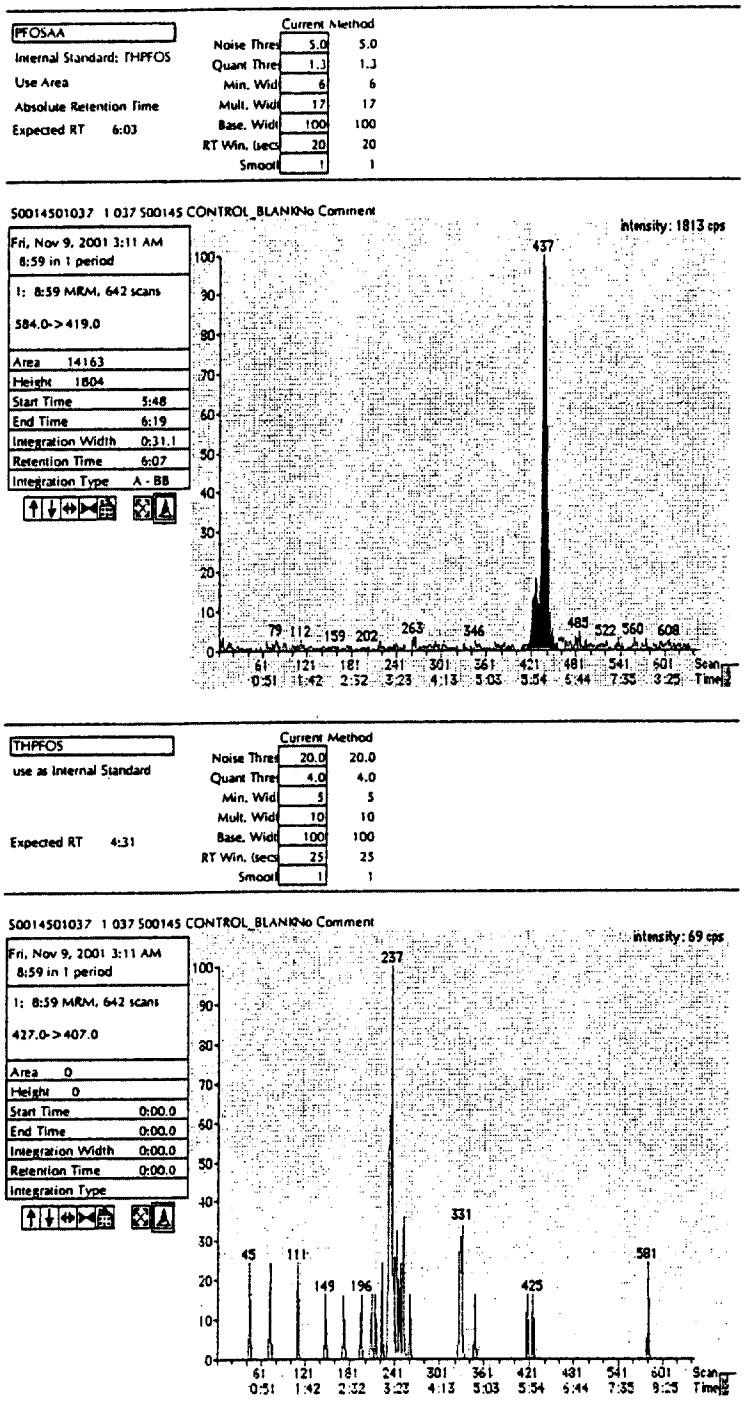
S0014501037 1 037 S00145 CONTROL_BLANKNo Comment

Fri, Nov 9, 2001 3:11 AM 8:59 in 1 period	
1: 8:59 MRM, 642 scans 427.0->407.0	
Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	



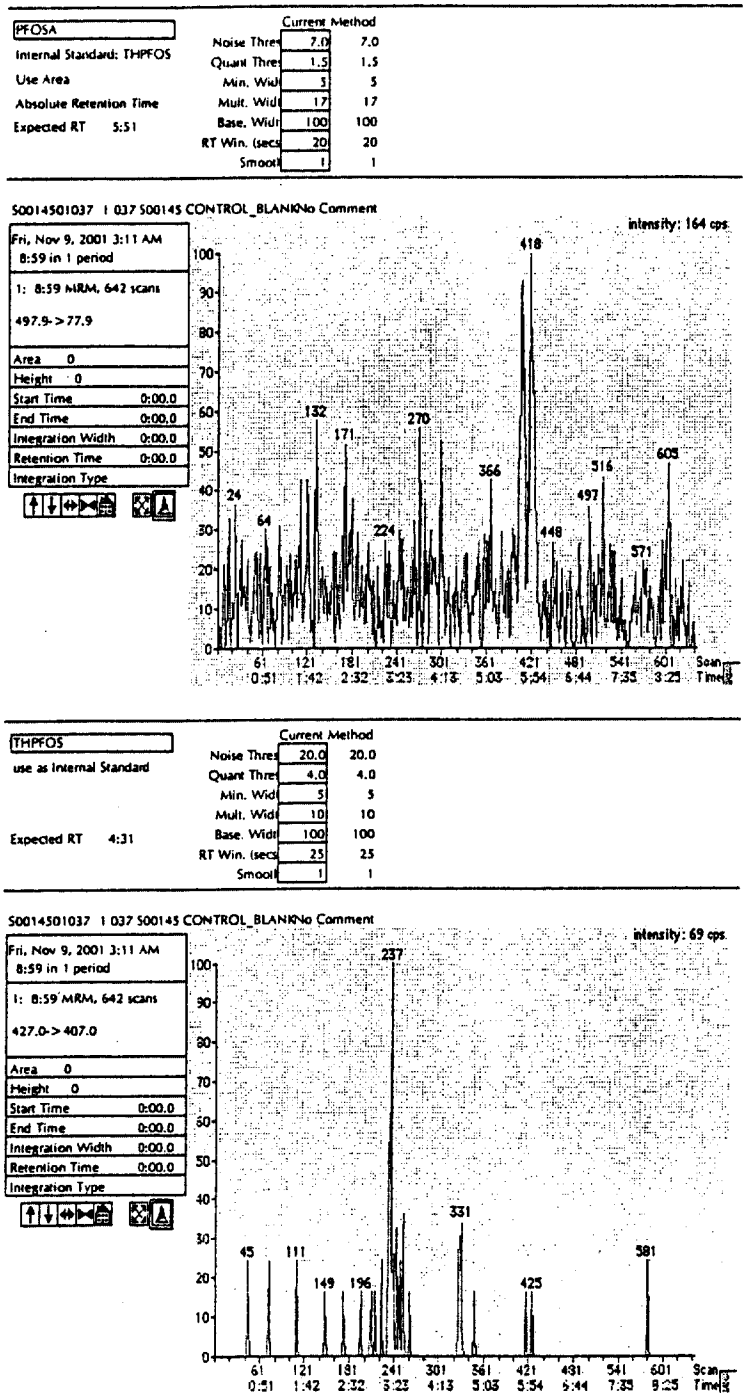
000649

Figure 11. Human Plasma Blank for PFOSAA



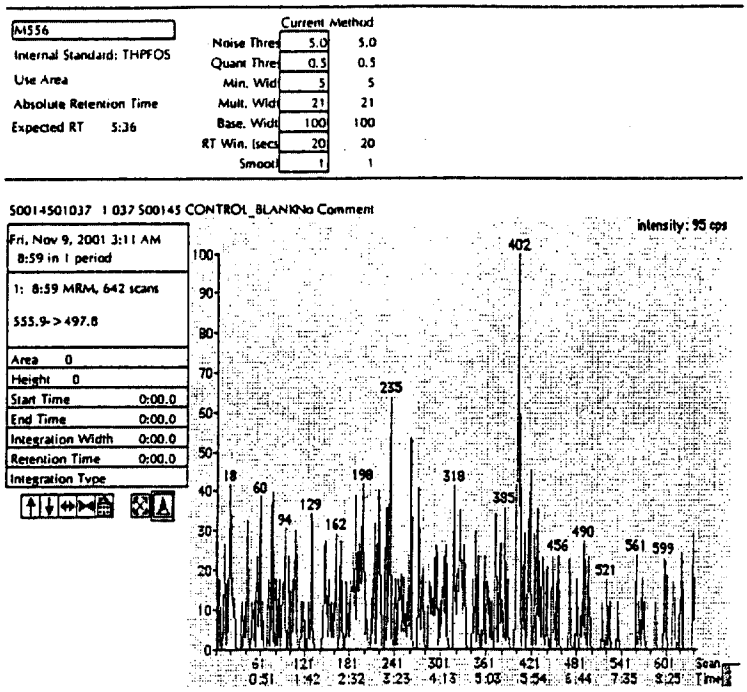
000650

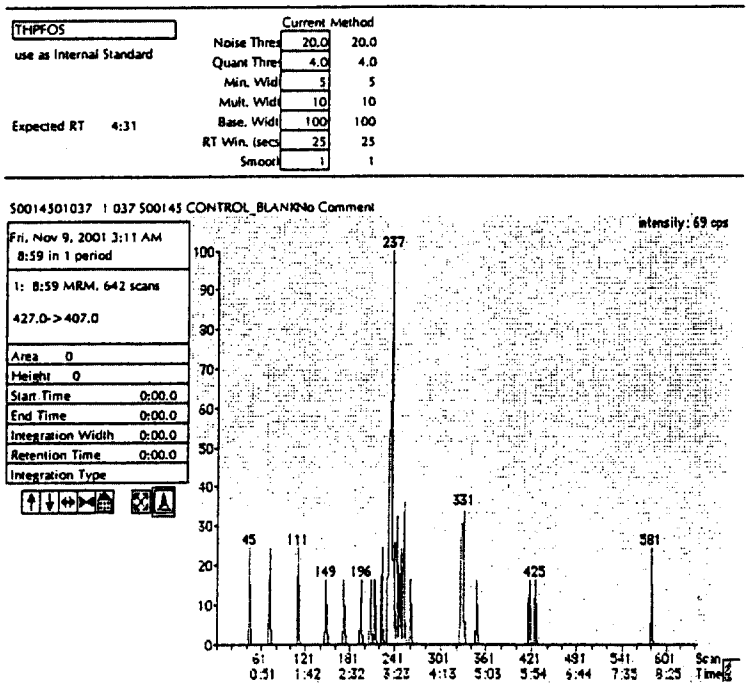
Figure 12. Human Plasma Blank for PFOSA



000651

Figure 13. Human Plasma Blank for M556





000652

Figure 14. Human Plasma Blank for M570

M570

Internal Standard: THPFOS

Use Area

Absolute Retention Time

Expected RT 5:54

	Current	Method
Noise Thres	5.0	5.0
Quant Thres	1.0	1.0
Min. Wid	5	5
Mult. Wid	20	20
Base. Wid	100	100
RT Win. (secs	20	20
Smoot	1	1

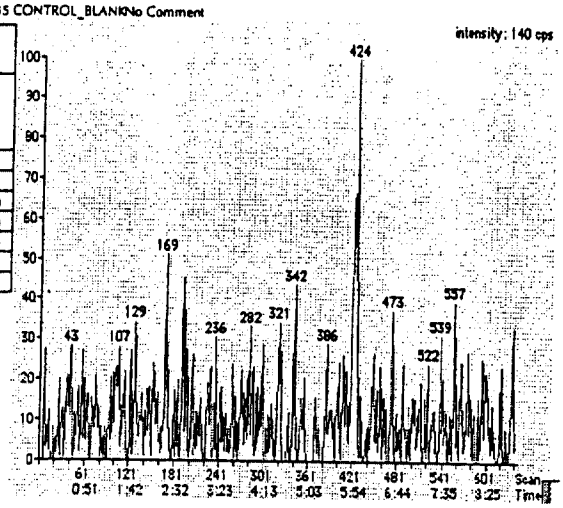
S00145010 37 1 037 S00145 CONTROL_BLANKNo Comment

Fri, Nov 9, 2001 3:11 AM
8:59 in 1 period

1: 8:59 MRM, 642 scans

570.0->419.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	



THPFOS

use as Internal Standard

Expected RT 4:31

	Current	Method
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smoot	1	1

S00145010 37 1 037 S00145 CONTROL_BLANKNo Comment

Fri, Nov 9, 2001 3:11 AM
8:59 in 1 period

1: 8:59 MRM, 642 scans

427.0->407.0

Area	0
Height	0
Start Time	0:00.0
End Time	0:00.0
Integration Width	0:00.0
Retention Time	0:00.0
Integration Type	

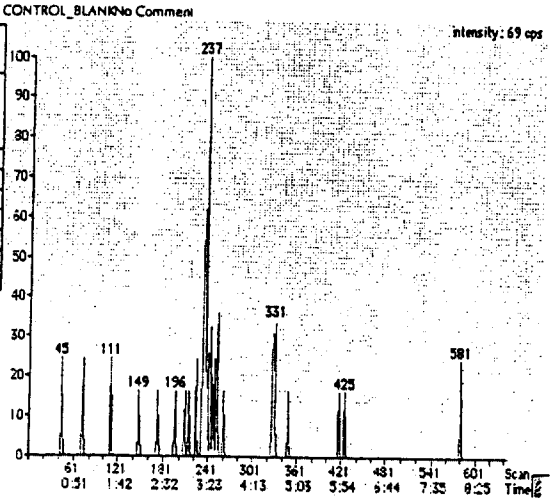


Figure 15. Human Plasma Blank with Internal Standard (QC0) for PFOS

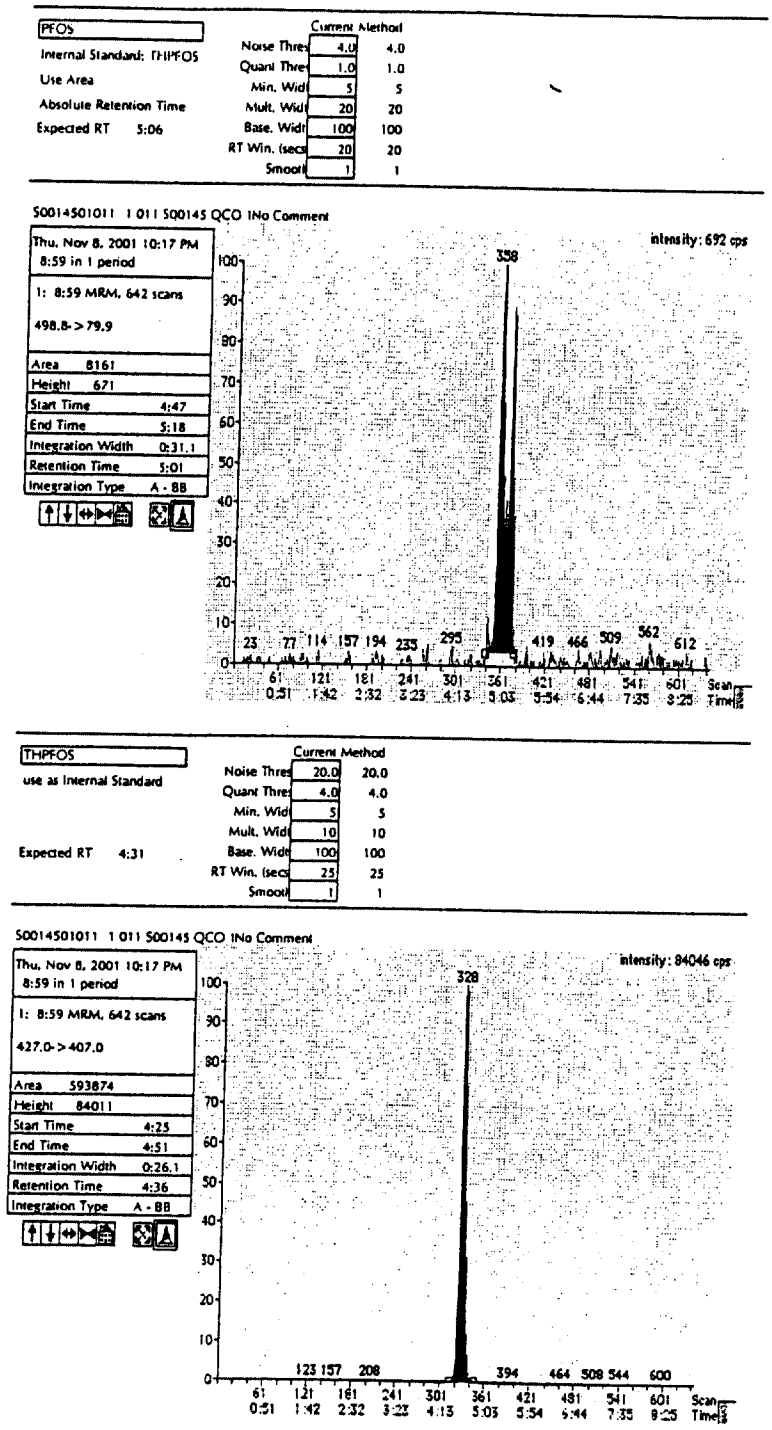


Figure 16. Human Plasma Blank with Internal Standard (QC0) for PFOA

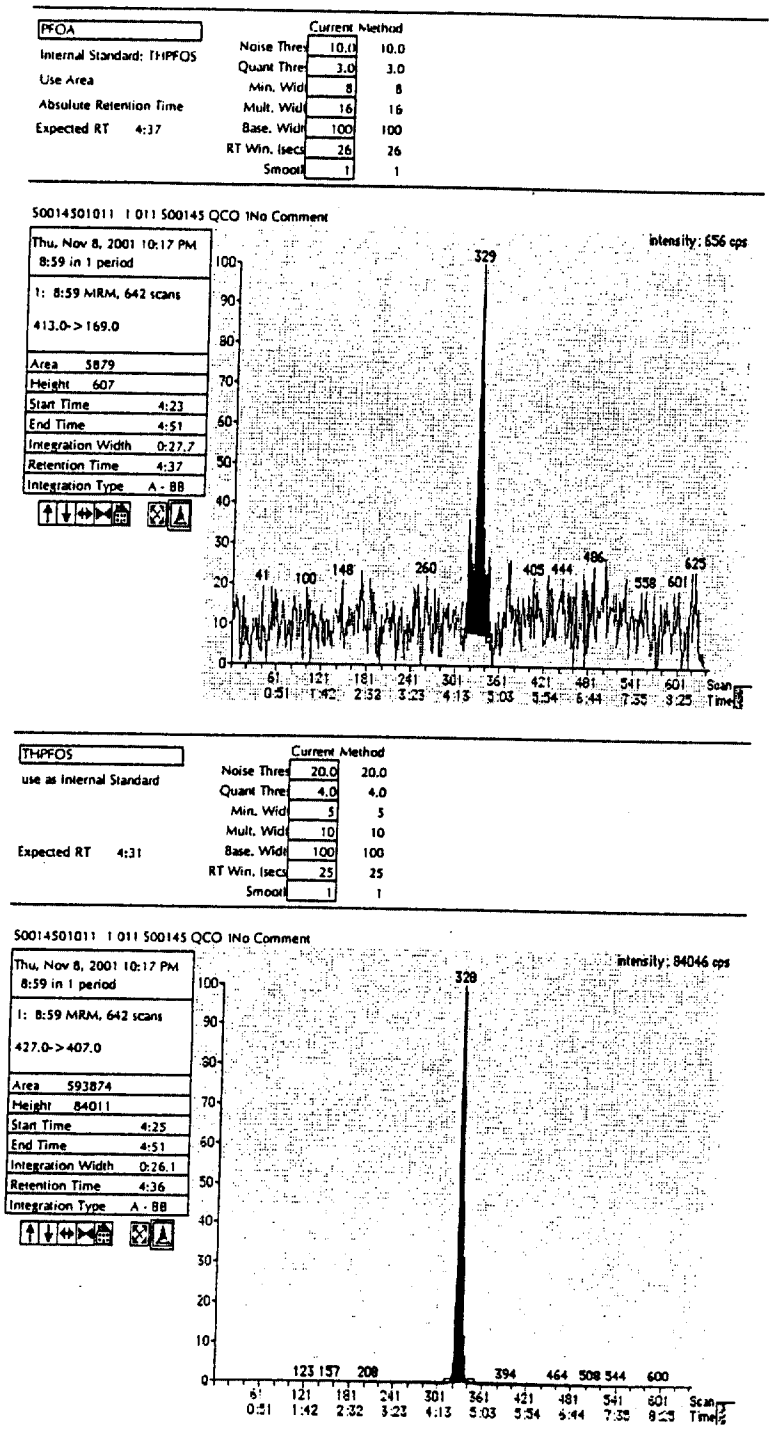


Figure 17. Human Plasma Blank with Internal Standard (QC0) for PFHS

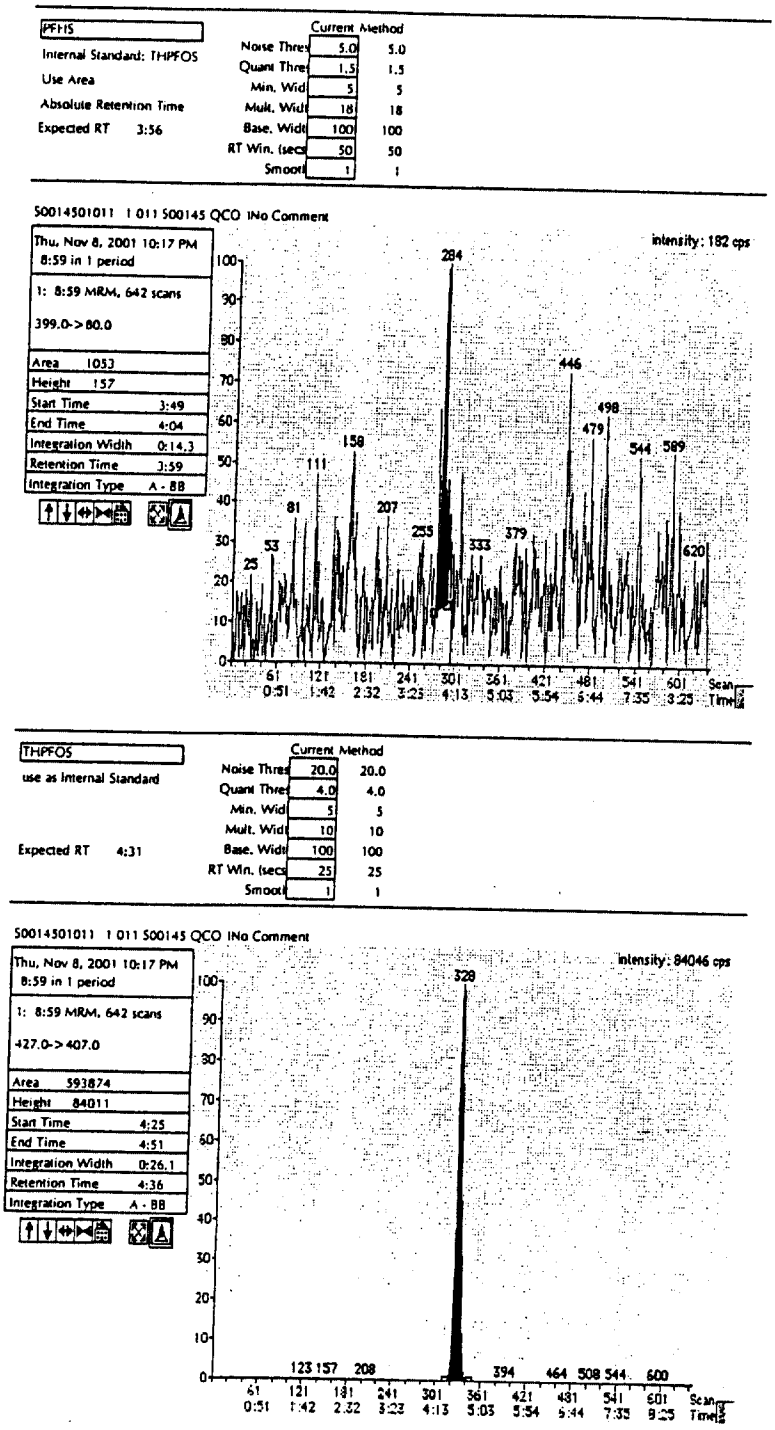


Figure 18. Human Plasma Blank with Internal Standard (QC0) for PFOSAA

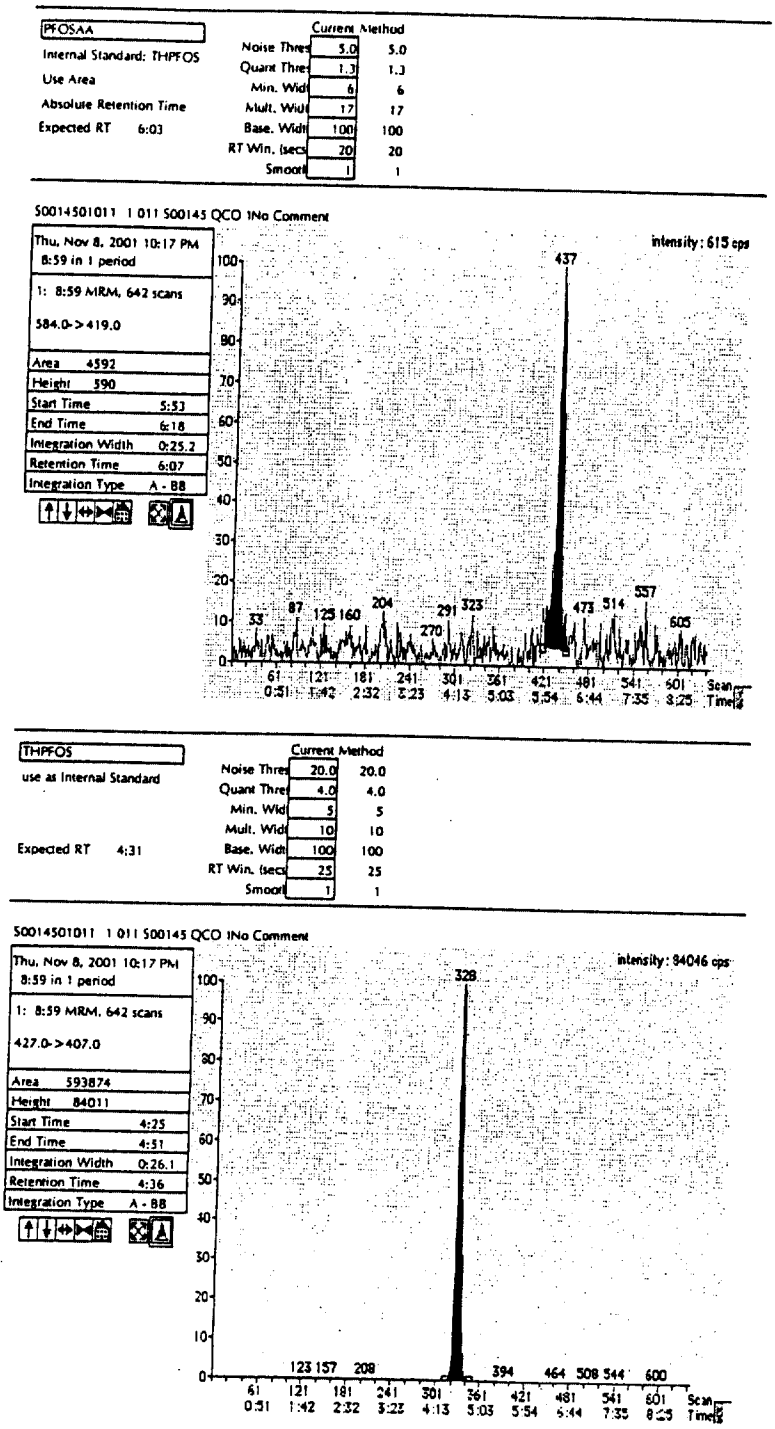


Figure 19. Human Plasma Blank with Internal Standard (QC0) for PFOSA

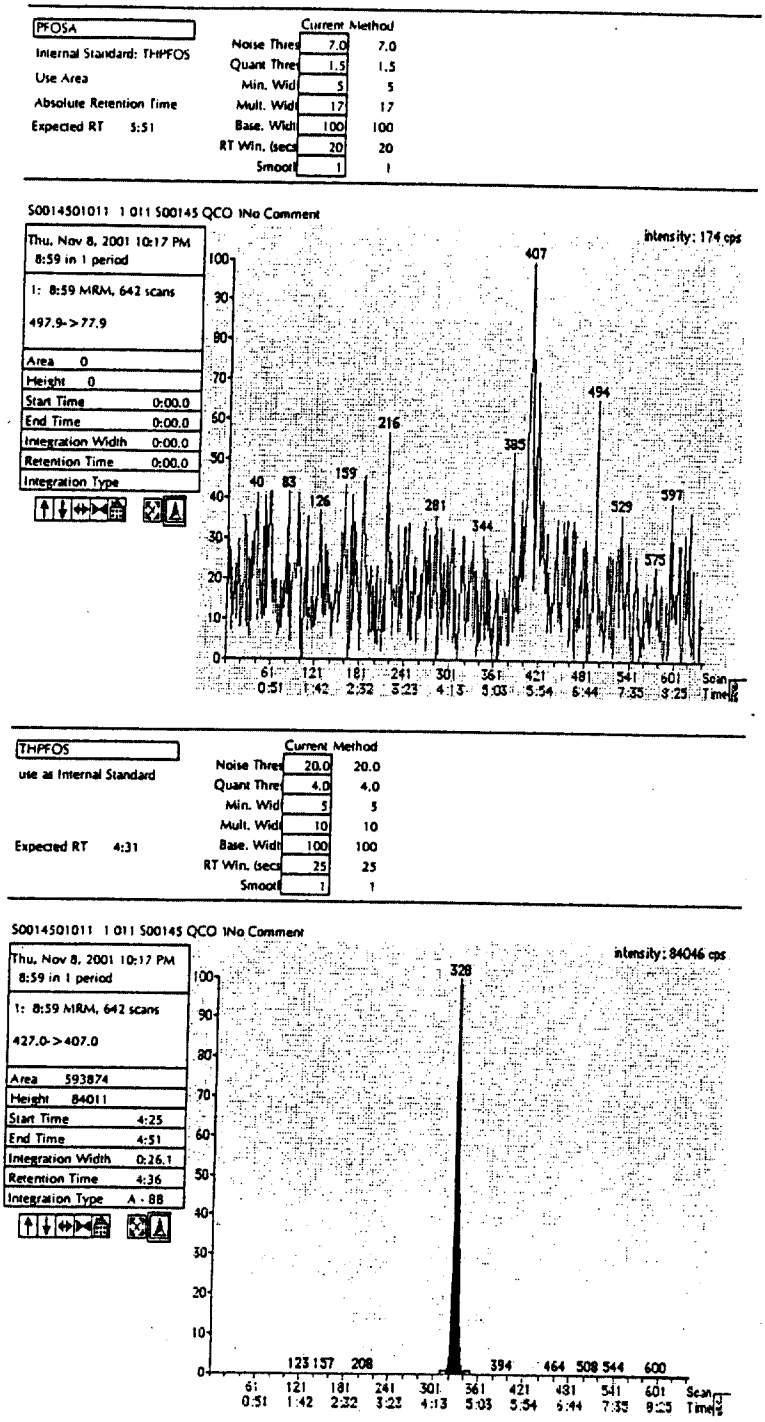


Figure 20. Human Plasma Blank with Internal Standard (QC0) for M556

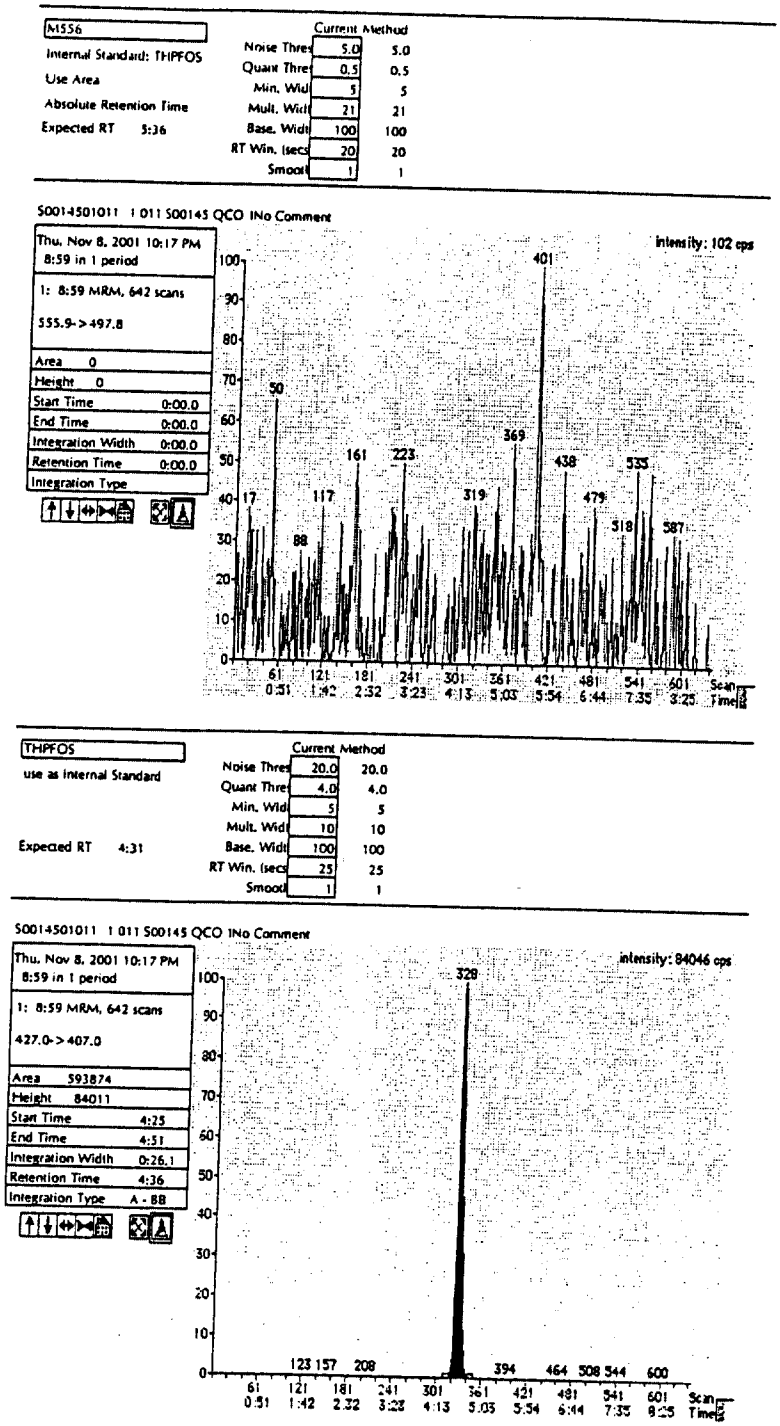


Figure 21. Human Plasma Blank with Internal Standard (QC0) for M570

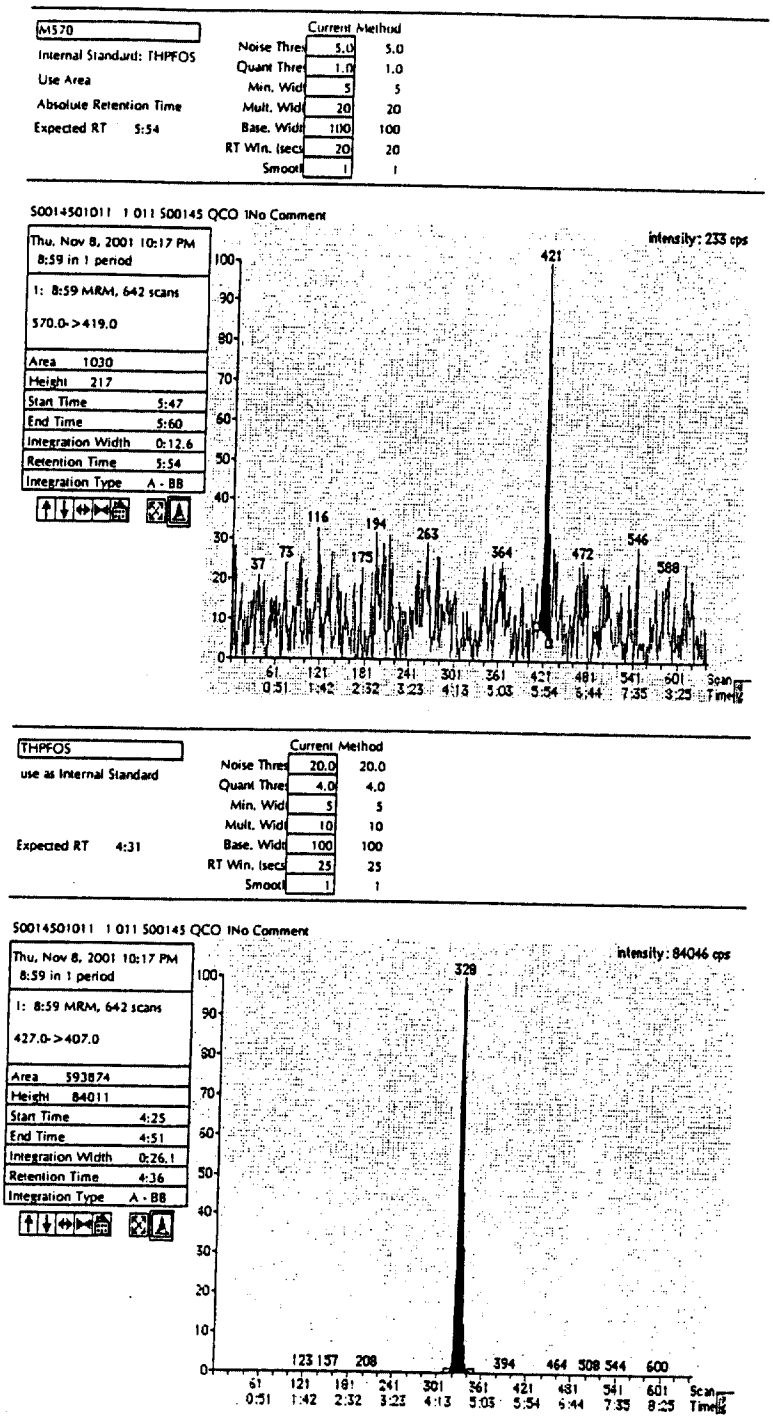


Figure 22. Low Standard for PFOS

PFOS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:06

	Current	Method
Noise Thres	4.0	4.0
Quant Thres	1.0	1.0
Min. Wid	5	5
Mult. Wid	20	20
Base. Wid	100	100
RT Win. (secs	20	20
Smooth	1	1

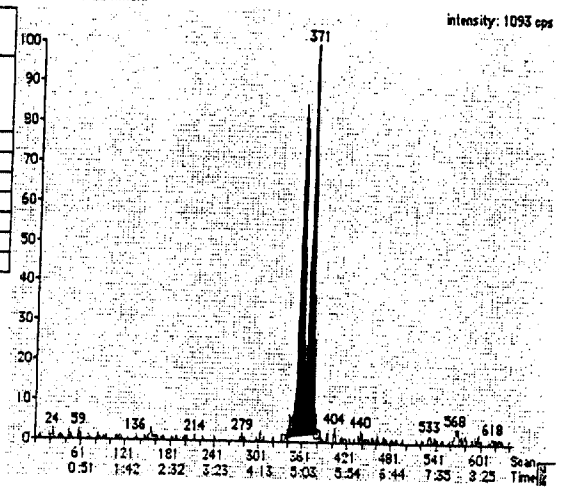
S0014501001 1 001 S00145 Std1 1 1 No Comment

Thu, Nov 8, 2001 8:25 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

498.8->79.9

Area	14141
Height	1074
Start Time	4:44
End Time	5:19
Integration Width	0:35.3
Retention Time	5:12
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:31

	Current	Method
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smooth	1	1

S0014501001 1 001 S00145 Std1 1 1 No Comment

Thu, Nov 8, 2001 8:25 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

427.0->407.0

Area	836307
Height	117889
Start Time	4:24
End Time	4:53
Integration Width	0:29.4
Retention Time	4:35
Integration Type	A - BB

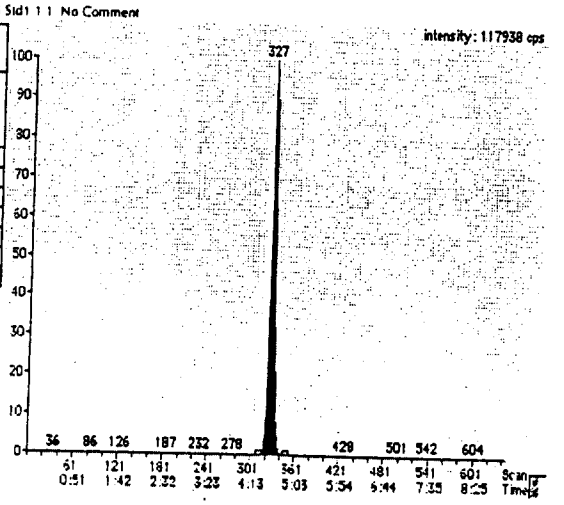


Figure 23. Low Standard for PFOA

PFOA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:37

	Current	Method
Noise Thres	10.0	10.0
Quant Thres	1.0	3.0
Min. Wid	8	8
Mult. Wid	16	16
Base. Wid	100	100
RT Win. (secs	26	26
Smooth	1	1

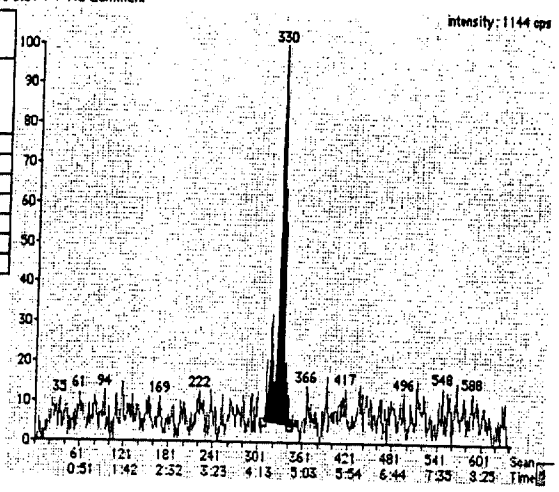
S0014501001 1 001 S00145 Std1 1 1 No Comment

Thu, Nov 8, 2001 8:25 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

413.0->169.0

Area	9405
Height	1095
Start Time	4:19
End Time	4:48
Integration Width	0:29.4
Retention Time	4:37
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:31

	Current	Method
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smooth	1	1

S0014501001 1 001 S00145 Std1 1 1 No Comment

Thu, Nov 8, 2001 8:25 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

427.0->407.0

Area	836307
Height	117889
Start Time	4:24
End Time	4:53
Integration Width	0:29.4
Retention Time	4:35
Integration Type	A - BB

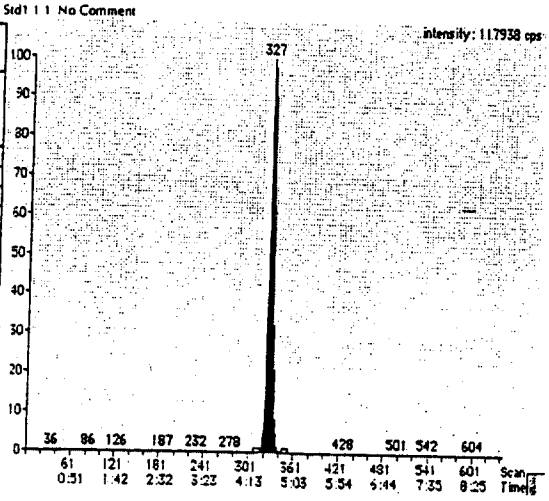


Figure 24. Low Standard for PFHS

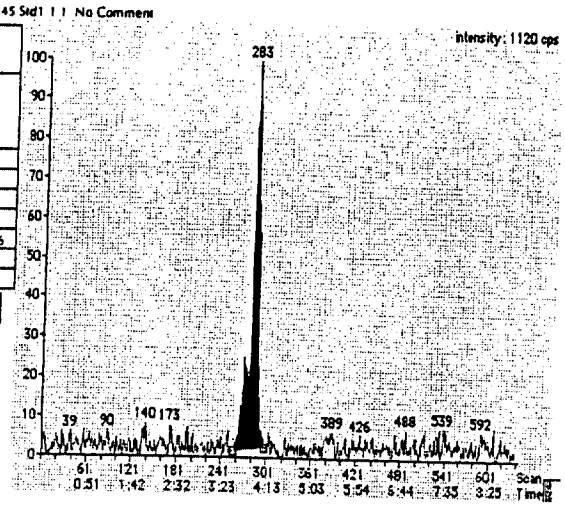
PFHS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 3:56

	Current	Method
Noise Thres	5.0	5.0
Quant Thre	1.5	1.5
Min. Wid	5	5
Mult. Wid	18	18
Base. Wid	100	100
RT Win. (secs	50	50
Smooth	1	1

S0014501001 1 001 S00145 Std1 1 1 No Comment

Thu, Nov 8, 2001 8:25 PM
8:59 in 1 period
1: 8:59 MRM, 642 scans
399.0->80.0

Area	9969
Height	1095
Start Time	3:37
End Time	4:10
Integration Width	0:33.6
Retention Time	3:58
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:31

	Current	Method
Noise Thres	20.0	20.0
Quant Thre	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smooth	1	1

S0014501001 1 001 S00145 Std1 1 1 No Comment

Thu, Nov 8, 2001 8:25 PM
8:59 in 1 period
1: 8:59 MRM, 642 scans
427.0->407.0

Area	836307
Height	117889
Start Time	4:24
End Time	4:53
Integration Width	0:29.4
Retention Time	4:35
Integration Type	A - BB

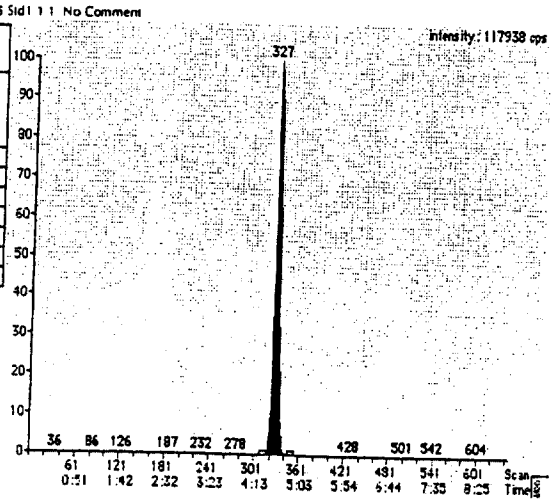
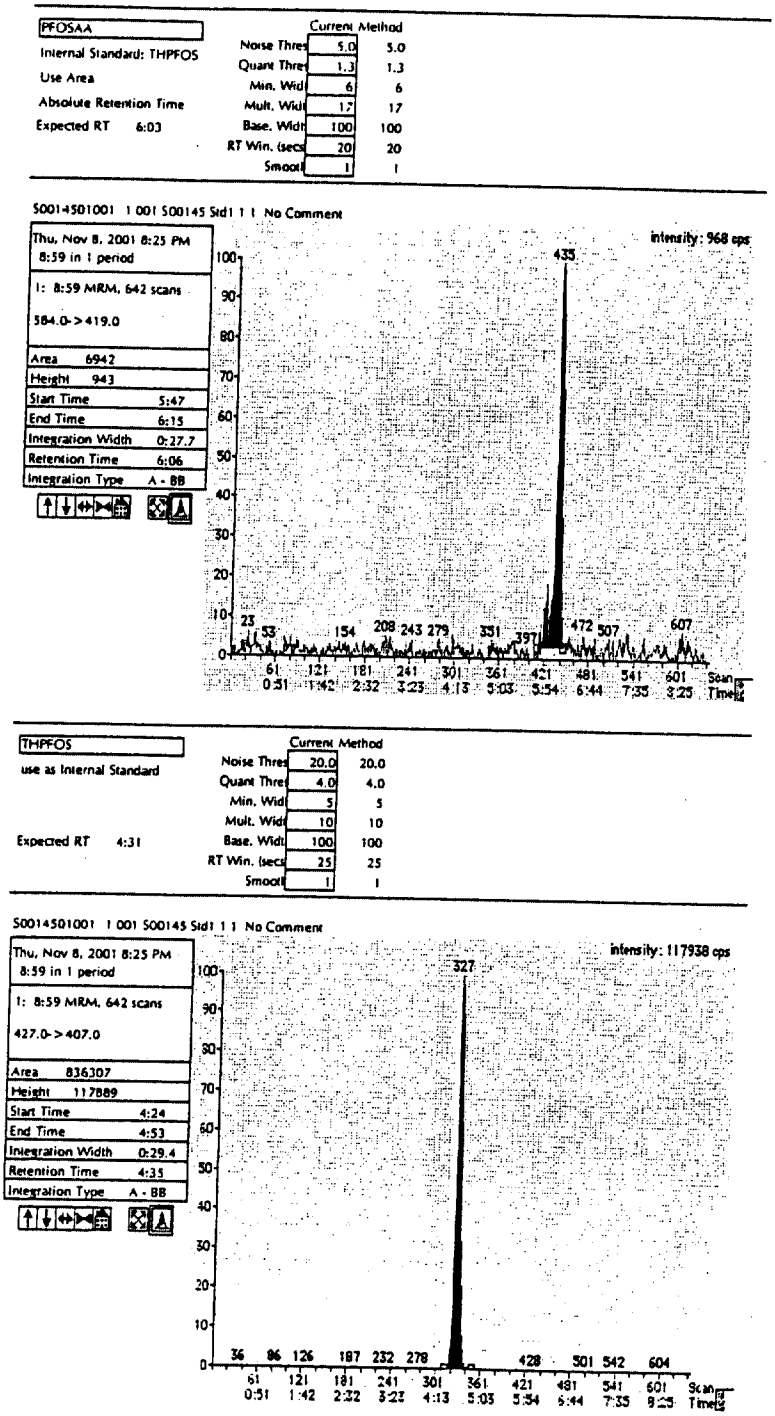
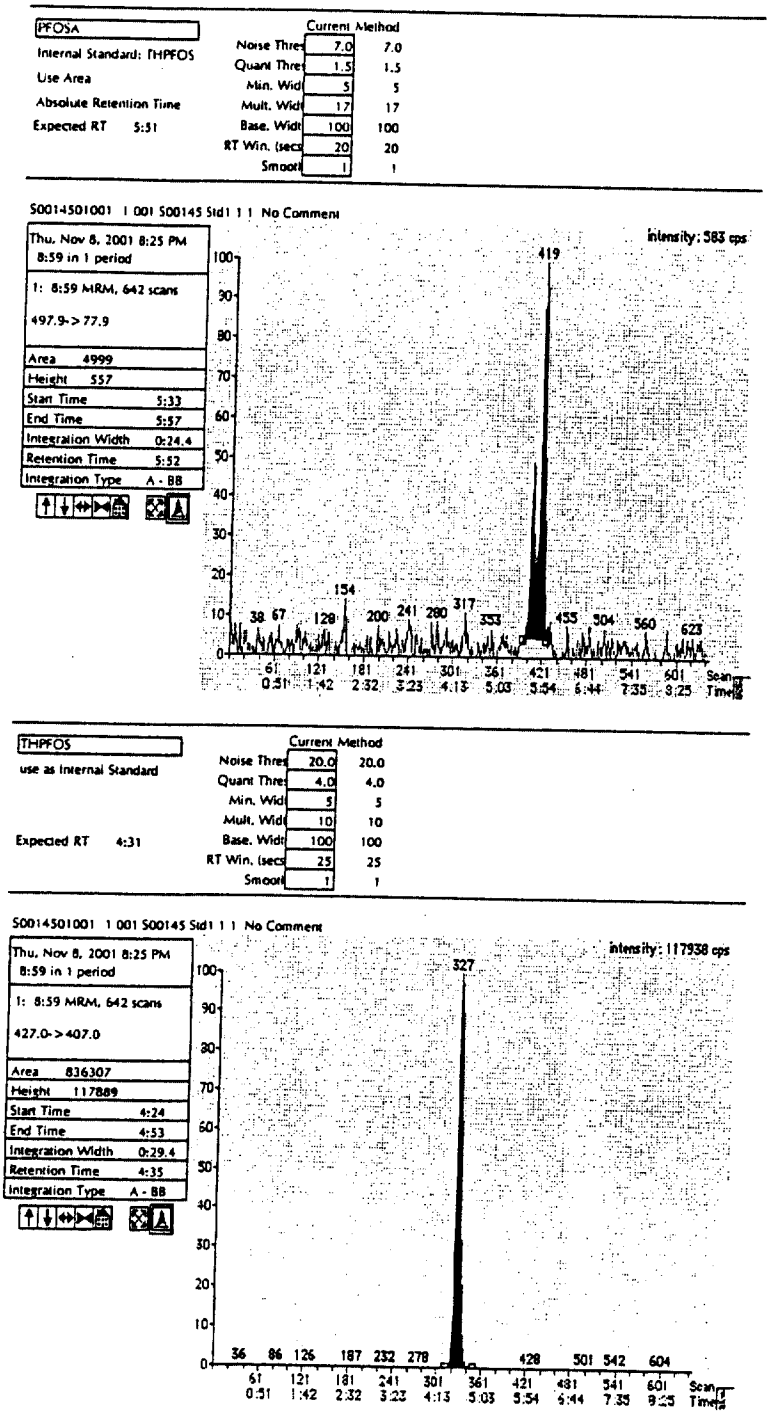


Figure 25. Low Standard for PFOSAA



000664

Figure 26. Low Standard for PFOSA



000665

Figure 27. Low Standard for M556

M556
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:36

	Current Method	
Noise Thres	5.0	5.0
Quant Thres	0.5	0.5
Min. Wid	5	5
Mult. Wid	21	21
Base. Wid	100	100
RT Win. (secs)	20	20
Smooth	1	1

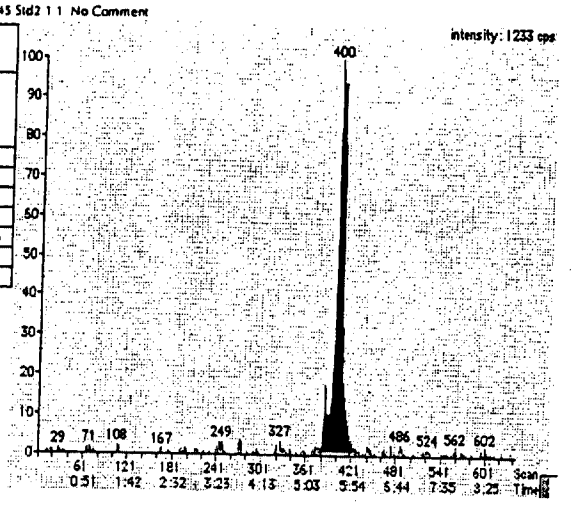
S0014501002 1 002 500145 Std2 1 1 No Comment

Thu, Nov 8, 2001 8:36 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

555.9->497.8

Area	11482
Height	1224
Start Time	5:18
End Time	5:55
Integration Width	0:37.0
Retention Time	5:36
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:31

	Current Method	
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs)	25	25
Smooth	1	1

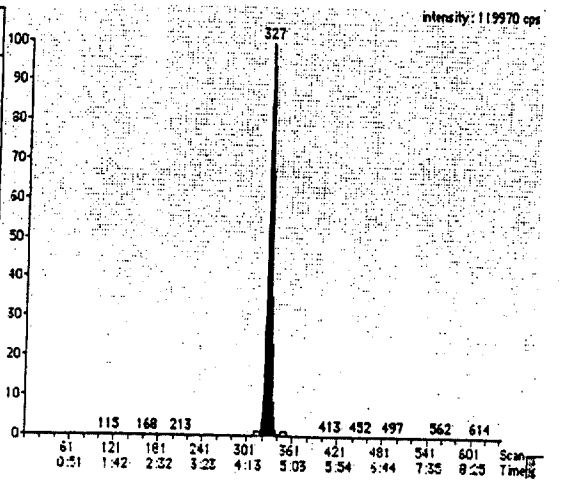
S0014501002 1 002 500145 Std2 1 1 No Comment

Thu, Nov 8, 2001 8:36 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

427.0->407.0

Area	836935
Height	119915
Start Time	4:25
End Time	4:54
Integration Width	0:29.4
Retention Time	4:35
Integration Type	A - BB



000666

Figure 28. Low Standard for M570

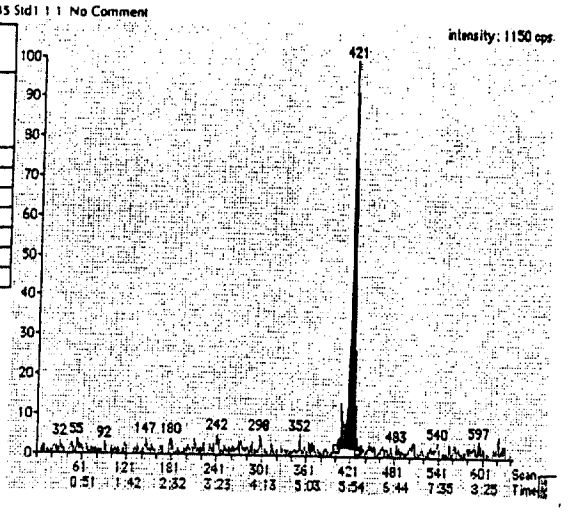
M570
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:54

	Current Method	
Noise Thres	5.0	5.0
Quant Thre	1.0	1.0
Min. Wid	5	5
Mult. Wid	20	20
Base. Wid	100	100
RT Win. (secs	20	20
Smoot	1	1

S0014501001 1 001 S00145 Std1 1 1 No Comment

Thu, Nov 8, 2001 8:25 PM
8:59 in 1 period
1: 8:59 MRM, 642 scans
570.0->419.0

Area	6875
Height	1132
Start Time	5:38
End Time	6:02
Integration Width	0:24.4
Retention Time	5:54
Integration Type	A - BB



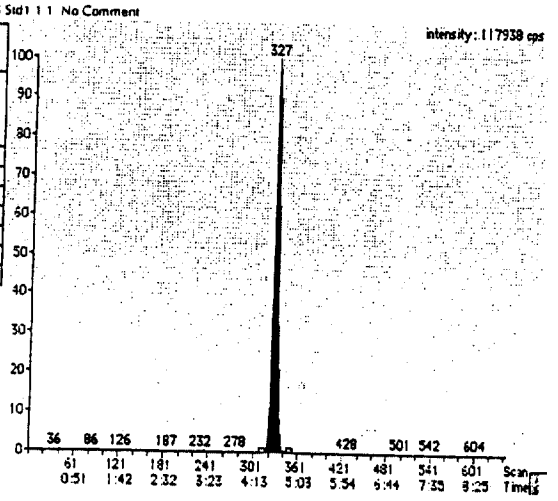
THPFOS
use as Internal Standard
Expected RT 4:31

	Current Method	
Noise Thres	20.0	20.0
Quant Thre	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smoot	1	1

S0014501001 1 001 S00145 Std1 1 1 No Comment

Thu, Nov 8, 2001 8:25 PM
8:59 in 1 period
1: 8:59 MRM, 642 scans
427.0->407.0

Area	836307
Height	117889
Start Time	4:24
End Time	4:53
Integration Width	0:29.4
Retention Time	4:35
Integration Type	A - BB



000667

Figure 29. High Standard for PFOS

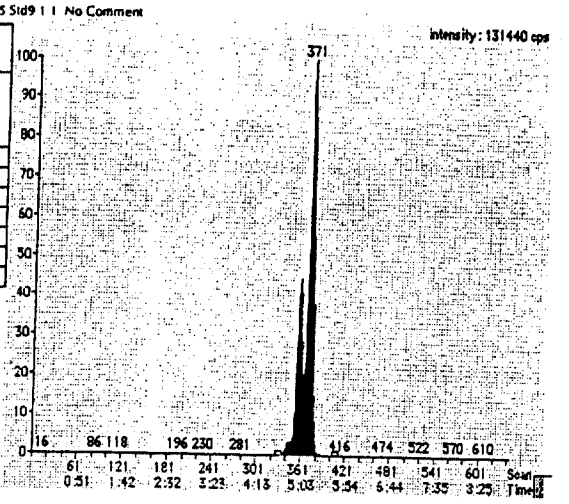
PFOS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:06

	Current	Method
Noise Thres	4.0	4.0
Quant Thres	1.0	1.0
Min. Wid	5	5
Mult. Wid	20	20
Base. Wid	100	100
RT Win. (secs	20	20
Smoot	1	1

S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period
1: 8:59 MRM, 642 scans
498.8->79.9

Area	1380828
Height	131420
Start Time	4:40
End Time	5:43
Integration Width	1:03
Retention Time	5:12
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:31

	Current	Method
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smoot	1	1

S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period
1: 8:59 MRM, 642 scans
427.0->407.0

Area	702935
Height	101201
Start Time	4:26
End Time	4:52
Integration Width	0:26.9
Retention Time	4:36
Integration Type	A - BB

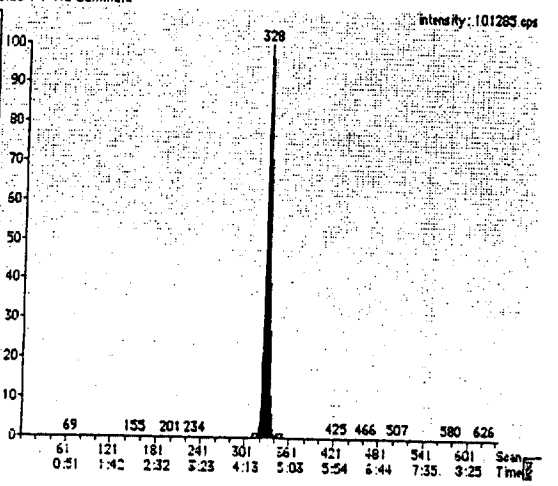


Figure 30. High Standard for PFOA

PFOA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 4:37

	Current	Method
Noise Thres	10.0	10.0
Quant Thres	3.0	3.0
Min. Wid	8	8
Mult. Wid	16	16
Base. Wid	100	100
RT Win. (secs)	26	26
Smooth	1	1

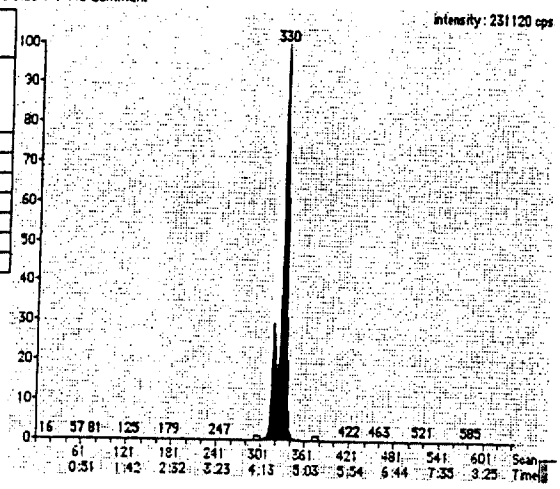
S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

413.0->169.0

Area	2159743
Height	231102
Start Time	4:10
End Time	5:15
Integration Width	1:05
Retention Time	4:37
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:31

	Current	Method
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs)	25	25
Smooth	1	1

S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

427.0->407.0

Area	702935
Height	101201
Start Time	4:26
End Time	4:52
Integration Width	0:26.9
Retention Time	4:36
Integration Type	A - BB

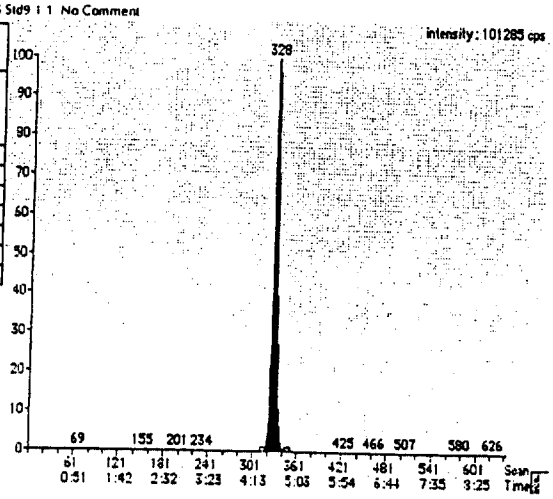


Figure 31. High Standard for PFHS

PFHS
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 3:56

	Current	Method
Noise Thres	5.0	5.0
Quant Thres	1.5	1.5
Min. Wid	5	5
Mult. Wid	18	18
Base. Wid	100	100
RT Win. (secs	50	50
Smoot	1	1

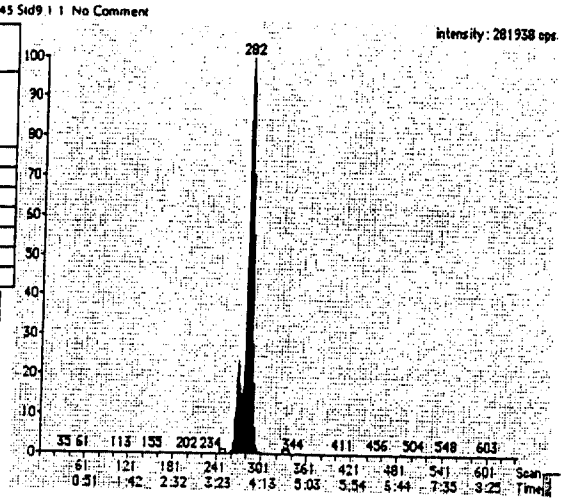
S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

399.0->80.0

Area	2577952
Height	281927
Start Time	3:30
End Time	4:41
Integration Width	1:11
Retention Time	3:57
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:31

	Current	Method
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smoot	1	1

S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

427.0->407.0

Area	702935
Height	101201
Start Time	4:26
End Time	4:52
Integration Width	0:26.9
Retention Time	4:36
Integration Type	A - BB

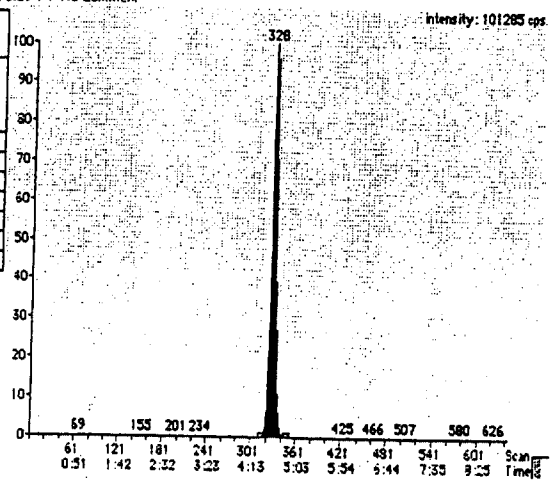


Figure 32. High Standard for PFOSAA

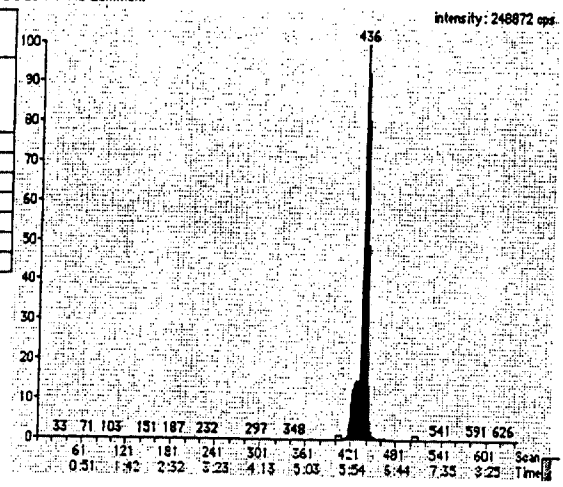
PFOSAA
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 6:03

	Current	Method
Noise Thres	5.0	5.0
Quant Thres	1.3	1.3
Min. Wid	6	6
Mult. Wid	17	17
Base. Wid	100	100
RT Win. (secs	20	20
Smoot	1	1

S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period
1: 8:59 MRM, 642 scans
584.0->419.0

Area	2049439
Height	248864
Start Time	5:40
End Time	7:05
Integration Width	1:25
Retention Time	6:06
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:31

	Current	Method
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smoot	1	1

S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period
1: 8:59 MRM, 642 scans
427.0->407.0

Area	702935
Height	101201
Start Time	4:26
End Time	4:52
Integration Width	0:26.9
Retention Time	4:36
Integration Type	A - BB

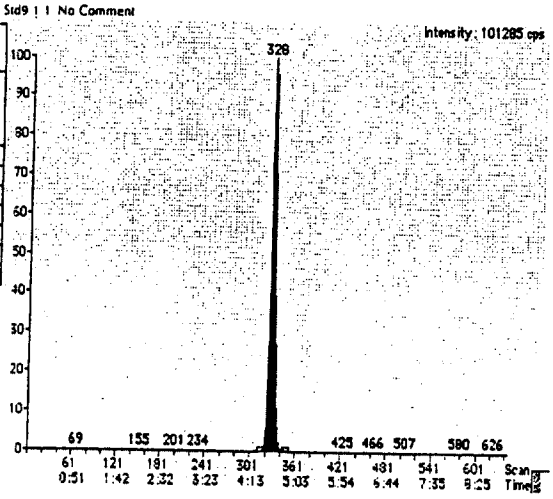


Figure 33. High Standard for PFOSA

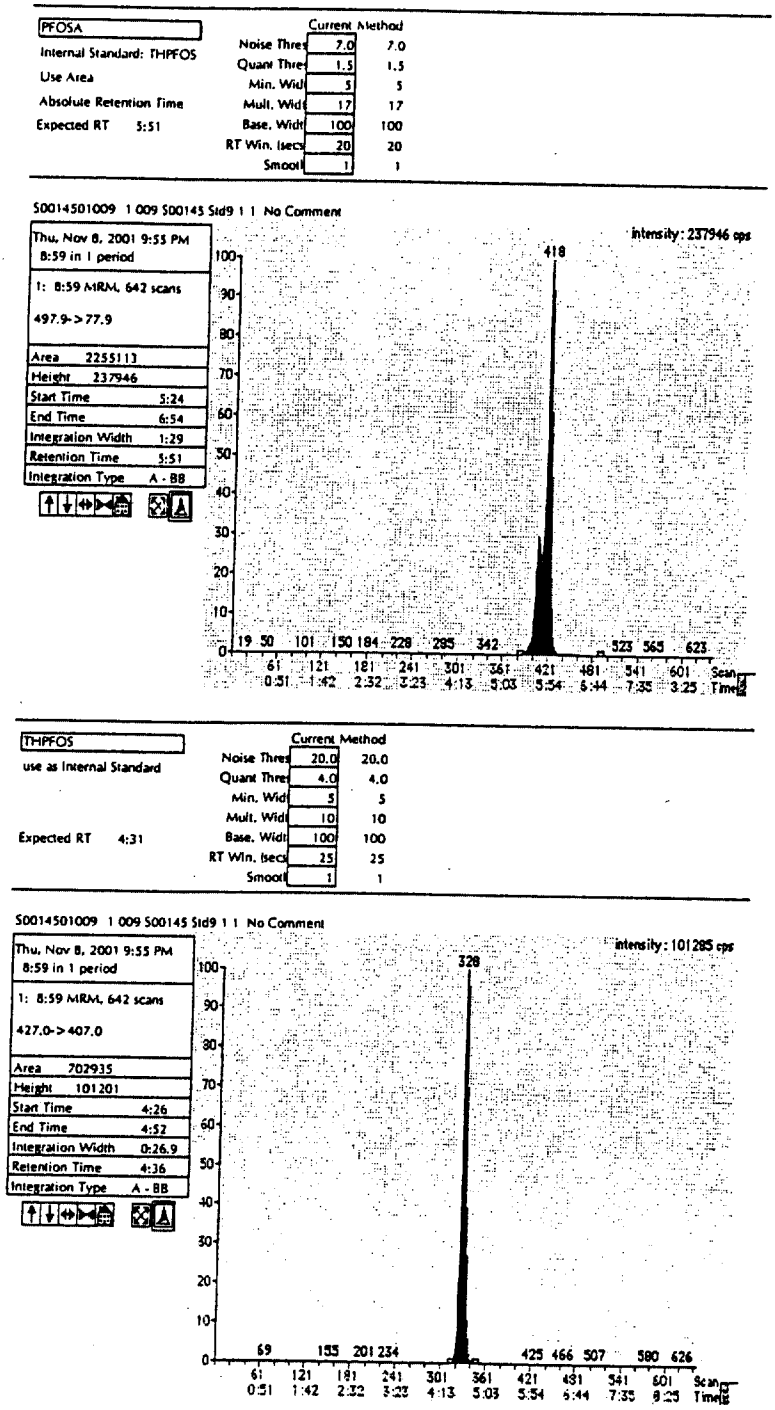


Figure 34. High Standard for M556

M556
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:36

	Current Method	
Noise Thres	5.0	5.0
Quant Thres	0.5	0.5
Min. Wid	5	5
Mult. Wid	21	21
Base. Wid	100	100
RT Win. (sec)	20	20
Smooth	1	1

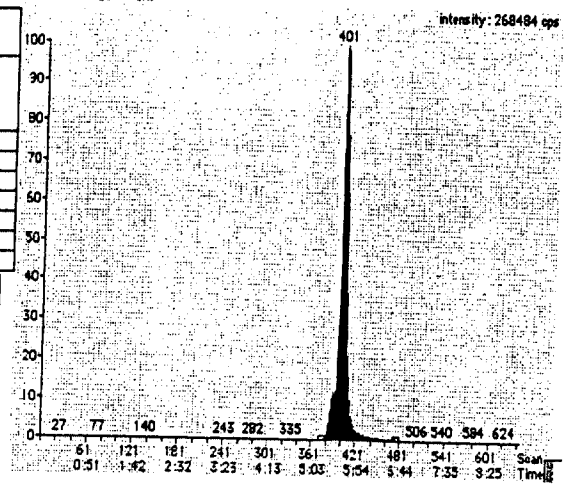
S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

555.9->497.8

Area	2482228
Height	268484
Start Time	5:16
End Time	6:40
Integration Width	1:24
Retention Time	5:37
Integration Type	A - BB



THPFOS
use as Internal Standard
Expected RT 4:31

	Current Method	
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (sec)	25	25
Smooth	1	1

S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

427.0->407.0

Area	702935
Height	101201
Start Time	4:26
End Time	4:52
Integration Width	0:26.9
Retention Time	4:36
Integration Type	A - BB

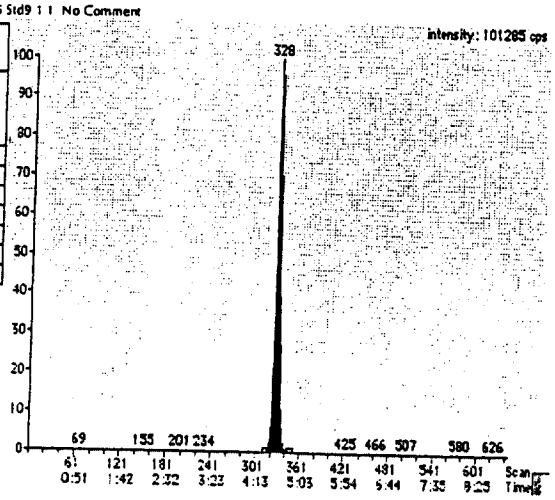


Figure 35. High Standard for M570

M570
Internal Standard: THPFOS
Use Area
Absolute Retention Time
Expected RT 5:54

	Current Method	
Noise Thres	5.0	5.0
Quant Thres	1.0	1.0
Min. Wid	5	5
Mult. Wid	20	20
Base. Wid	100	100
RT Win. (secs	20	20
Smooth	1	1

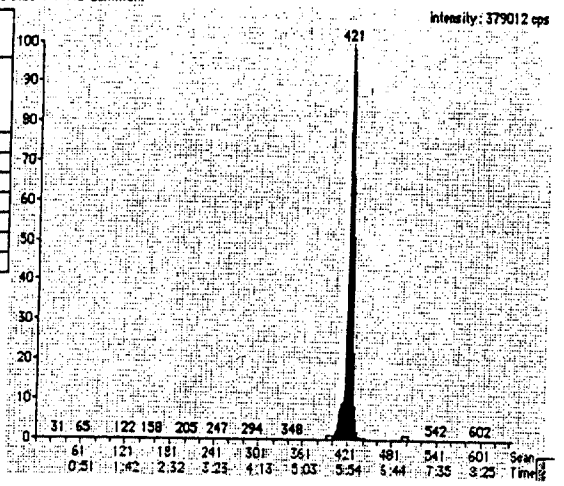
S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

570.0->419.0

Area	2874524
Height	379007
Start Time	5:34
End Time	6:59
Integration Width	1:25
Retention Time	5:54
Integration Type	A - BV



THPFOS
use as Internal Standard
Expected RT 4:31

	Current Method	
Noise Thres	20.0	20.0
Quant Thres	4.0	4.0
Min. Wid	5	5
Mult. Wid	10	10
Base. Wid	100	100
RT Win. (secs	25	25
Smooth	1	1

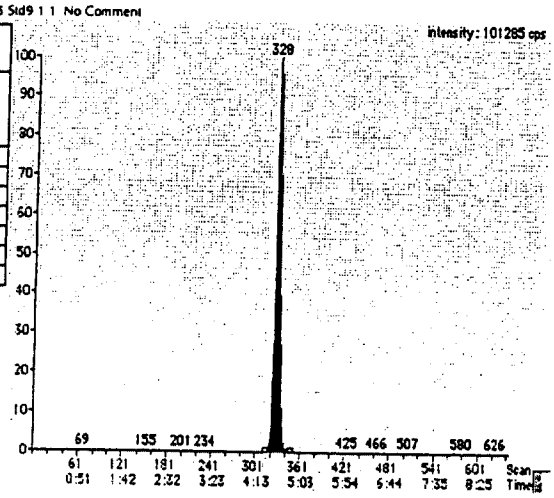
S0014501009 1 009 S00145 Std9 1 1 No Comment

Thu, Nov 8, 2001 9:55 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

427.0->407.0

Area	702935
Height	101201
Start Time	4:26
End Time	4:52
Integration Width	0:26.9
Retention Time	4:36
Integration Type	A - BB



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