

**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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QUALITY ASSURANCE STATEMENT

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COMPOUND(S): PFOS and Related Compounds

NWB STUDY NUMBER: NWBS00-145

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NWB STUDY TITLE: Quantitative Determination of PFOS and Related
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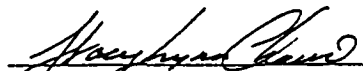
The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, Northwest Bioanalytical conducts all studies within the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (Title 21 CFR Part 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.

<u>Date of Inspection</u>	<u>Phase of Study</u>	<u>Date Report Sent to NWB Project Manager</u>	<u>Date Report Sent to NWB Management*</u>
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.

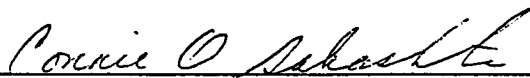
As can reasonably be established, the methods and procedures described and the results incorporated into this final report accurately reflect the raw data.


Stacy Lynn Chain, B.A.
NWB Quality Assurance Compliance Auditor

01-10-02
Date

COMPLIANCE STATEMENT

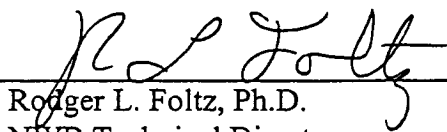
The clinical study described in this report is not included within the definition of a GLP regulated nonclinical study. However, to the best of our knowledge, this project was conducted in accordance with the guidelines of the U.S. FDA Good Laboratory Practice Regulations for Nonclinical Laboratory Studies (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, B.S.
NWB Project Manager

1-10-02

Date



Rodger L. Foltz, Ph.D.
NWB Technical Director

1-10-02

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-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:

<i>Receipt Date</i>	<i>Number of Samples Received</i>	<i>Storage Condition (except during analysis)</i>
23-Oct-2001	262	-20 °C

Reference Material

<i>Analyte</i>	<i>Lot Number</i>	<i>Purity</i>	<i>Expiration Date</i>	<i>Source</i>	<i>Storage Conditions</i>
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:

<i>Analyte</i>	<i>Correction Factor</i>
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.

Analyte	QC Concentrations (ppb)*		
	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 $\pm 15\%$ ($\pm 20\%$ 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 $\pm 20\%$ ($\pm 25\%$ 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 $\pm 25\%$ of the target concentration for PFOSA and PFOSAA to qualify as the LLOQ and within $\pm 20\%$ 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 $\pm 20\%$ of their target concentrations ($\pm 25\%$ 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 $\pm 20\%$ 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 PFOSQuadratic 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 PFOAQuadratic 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 PFHSQuadratic 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.

Table 4. Calibration Curve Summary for PFOSAAQuadratic 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.

Table 5. Calibration Curve Summary for PFOSAQuadratic 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 M556Quadratic 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 M570Quadratic 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 PFOSQuadratic 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 PFOAQuadratic 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 PFHSQuadratic 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)

Table 11. Back-Calculated Concentrations of Calibration Standards for PFOSAAQuadratic 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 PFOSAQuadratic 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 M556Quadratic 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 M570Quadratic 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

* > $\pm 20\%$ 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

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

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

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 $\pm 20\%$ ($\pm 25\%$ 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 $\pm 20\%$ ($\pm 25\%$ 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 $\pm 20\%$ ($\pm 25\%$ 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 $\pm 20\%$ ($\pm 25\%$ 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 $\pm 20\%$ ($\pm 25\%$ 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 $\pm 20\%$ ($\pm 25\%$ 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 $\pm 20\%$ ($\pm 25\%$ 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.

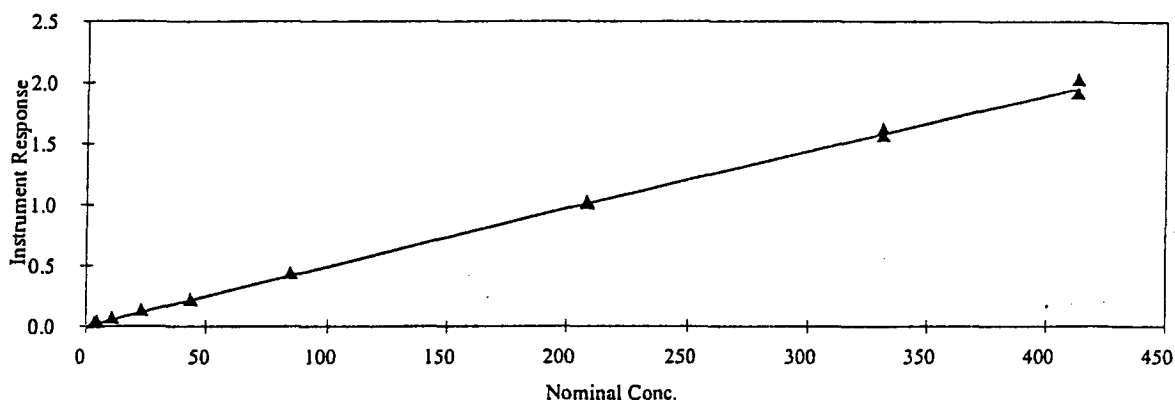
Sample Name	PFOS	PFOSA*	PFOSAA*	PFOA*	PFHS*	M556*	M570*
GH11319	34.1	<LLOQ (1.00)	<LLOQ (1.50)	4.24	2.55	<LLOQ (2.50)	1.29
GH11320	21.6	<LLOQ (1.00)	<LLOQ (1.50)	3.91	2.97	<LLOQ (2.50)	<LLOQ (1.00)
GH11321	43.5	<LLOQ (1.00)	6.67	6.96	2.19	<LLOQ (2.50)	1.04
GH11322	104	<LLOQ (1.00)	9.85	15.4	3.73	2.75	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 $\pm 20\%$ ($\pm 25\%$ 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

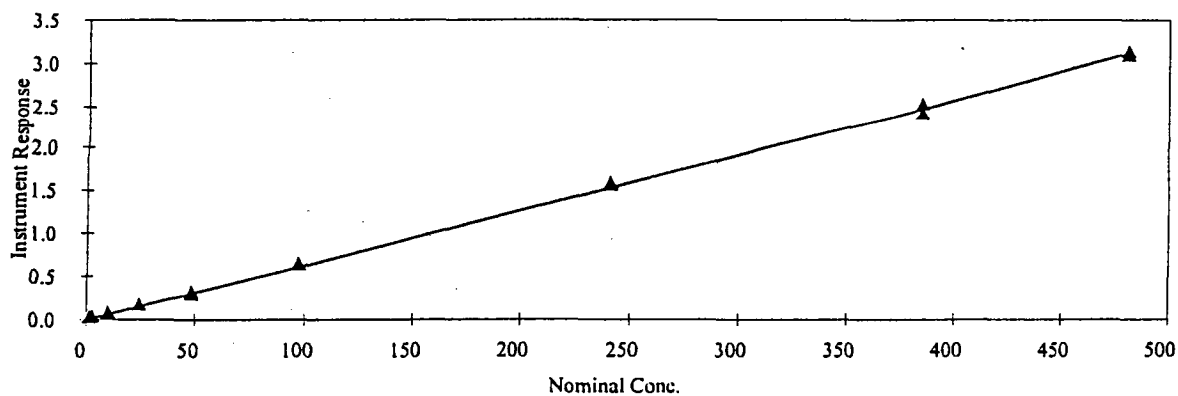
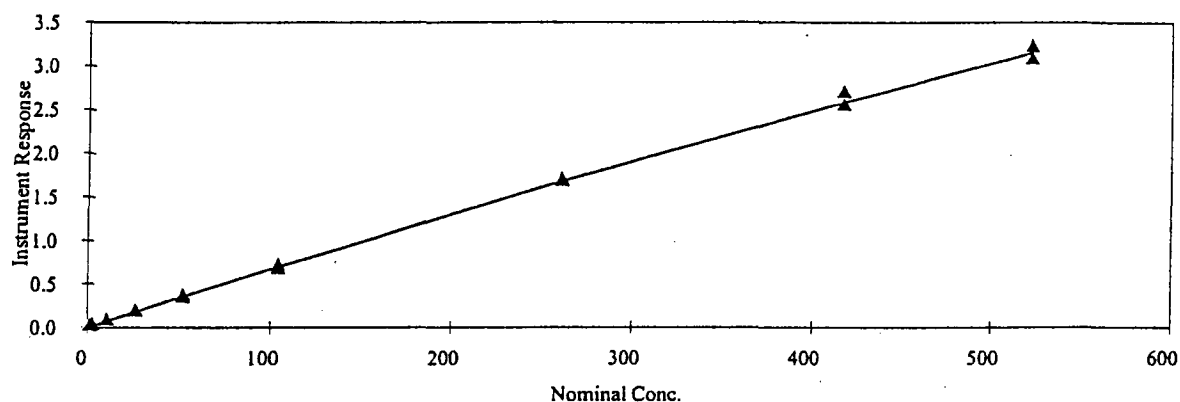


Figure 3. Representative Calibration Curve for PFHS

Analytical Run 6 analyzed on 15-Nov-2001 Calibration Standards for PFHS (ppb)
Regression Method = QUADRATIC - Weighting Factor = $1/X^{**2}$
Quadratic Limit = 2200

**Figure 4. Representative Calibration Curve for PFOSAA**

Analytical Run 6 analyzed on 15-Nov-2001 Calibration Standards for PFOSAA (ppb)
Regression Method = QUADRATIC - Weighting Factor = $1/X^{**2}$
Quadratic Limit = -3640

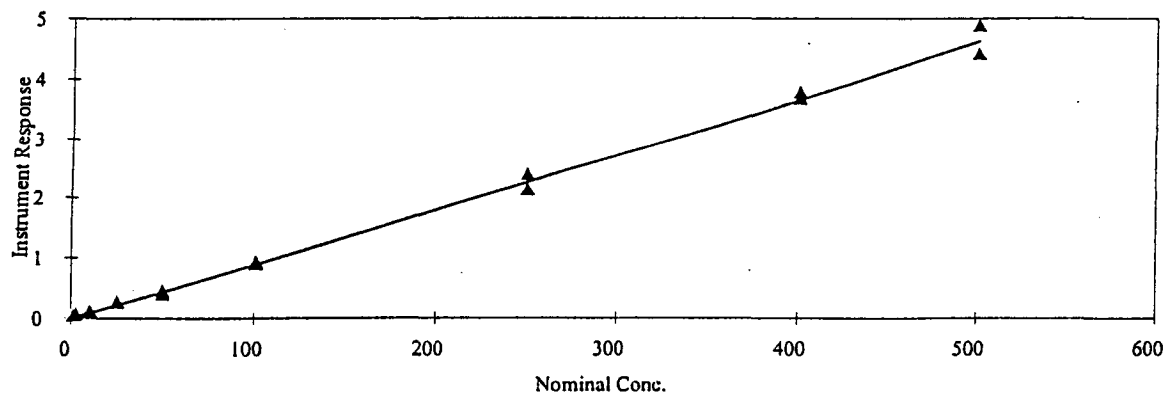
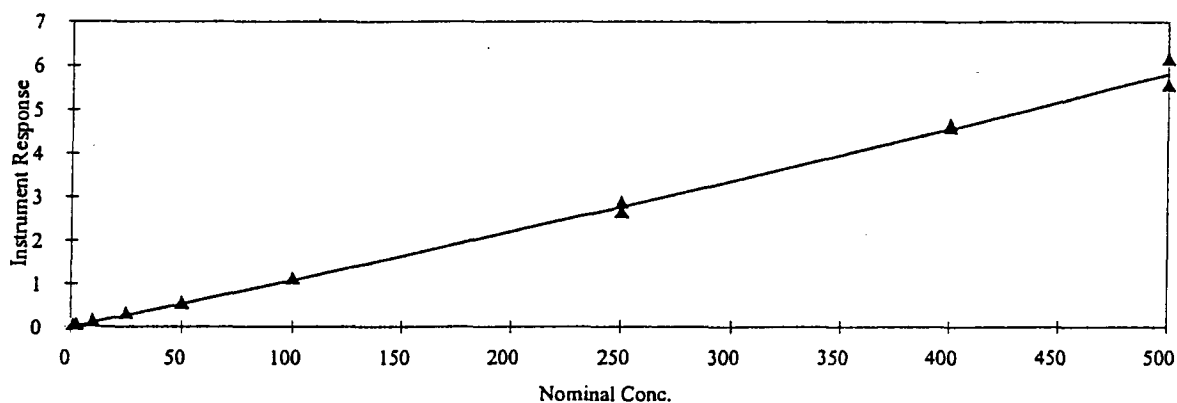


Figure 5. Representative Calibration Curve for PFOSA

Analytical Run 6 analyzed on 15-Nov-2001 Calibration Standards for PFOSA (ppb)
Regression Method = QUADRATIC - Weighting Factor = $1/X^{**2}$
Quadratic Limit = -2180

**Figure 6. Representative Calibration Curve for M556**

Analytical Run 6 analyzed on 15-Nov-2001 Calibration Standards for M556 (ppb)
Regression Method = QUADRATIC - Weighting Factor = $1/X^{**2}$
Quadratic Limit = -2310

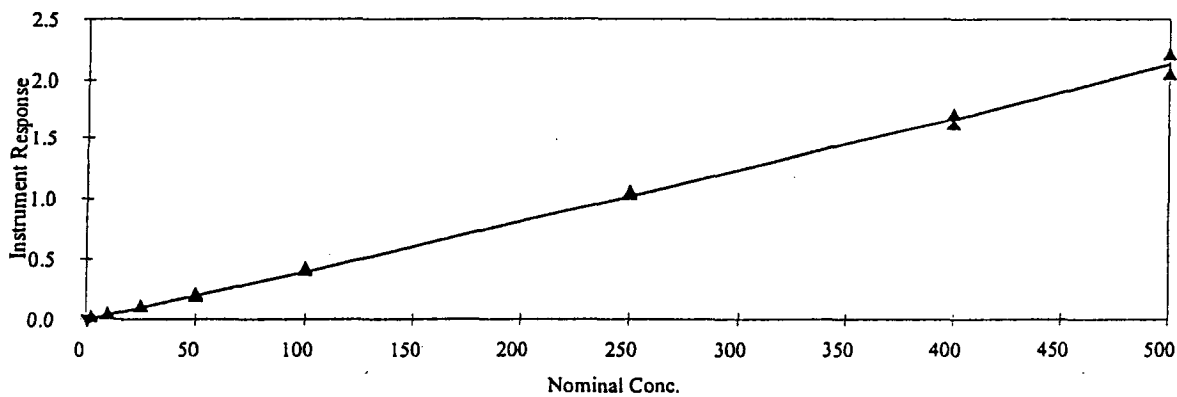


Figure 7. Representative Calibration Curve for M570

Analytical Run 6 analyzed on 15-Nov-2001 Calibration Standards for M570 (ppb)
Regression Method = QUADRATIC - Weighting Factor = $1/X^{**2}$
Quadratic Limit = -12500

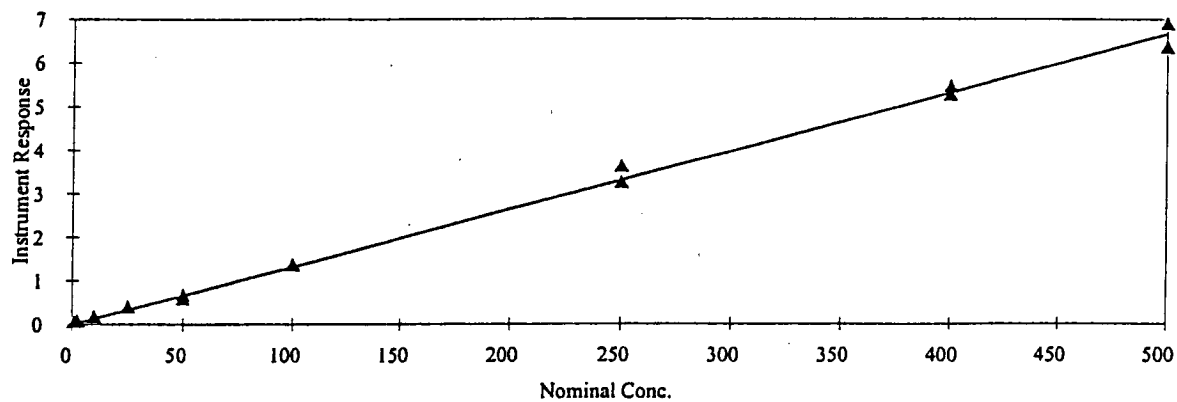
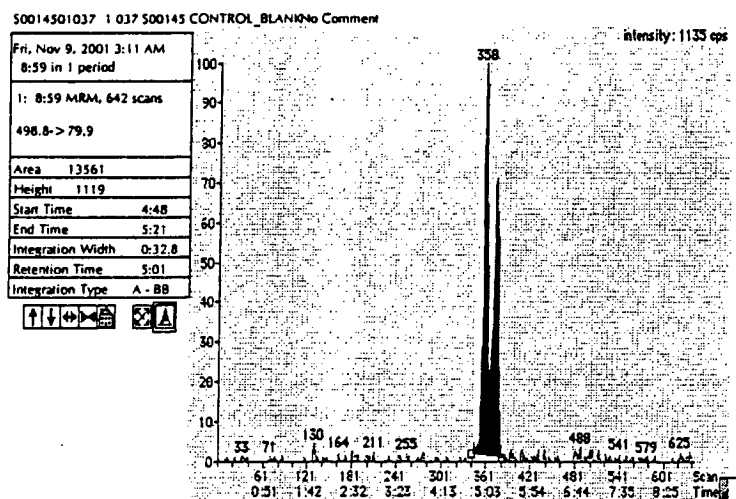


Figure 8. Human Plasma Blank for PFOS

PFOS	Current Method
Internal Standard: THPFOS	Noise Thre: 4.0 4.0
Use Area	Quant Thre: 1.0 1.0
Absolute Retention Time	Min. Wid: 5 5
Expected RT 5:06	Mult. Wid: 20 20
	Base. Wid: 100 100
	RT Win. (secs): 20 20
	Smooth: 1 1



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
	Base. Wid: 100 100
Expected RT 4:31	RT Win. (secs): 25 25
	Smooth: 1 1

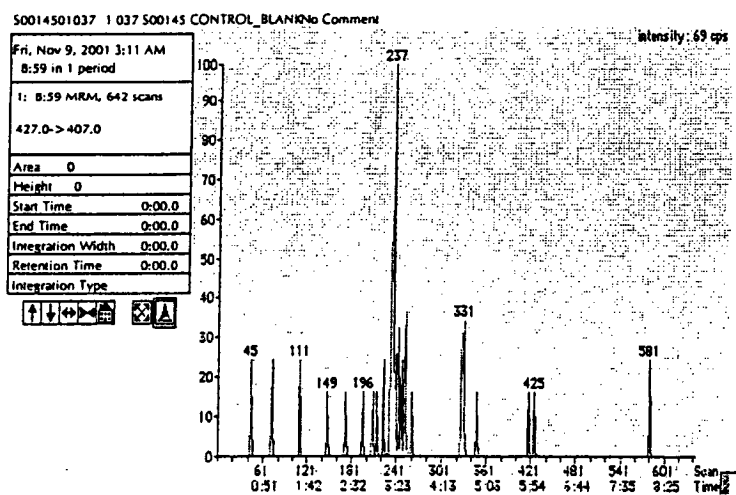


Figure 9. Human Plasma Blank for PFOA

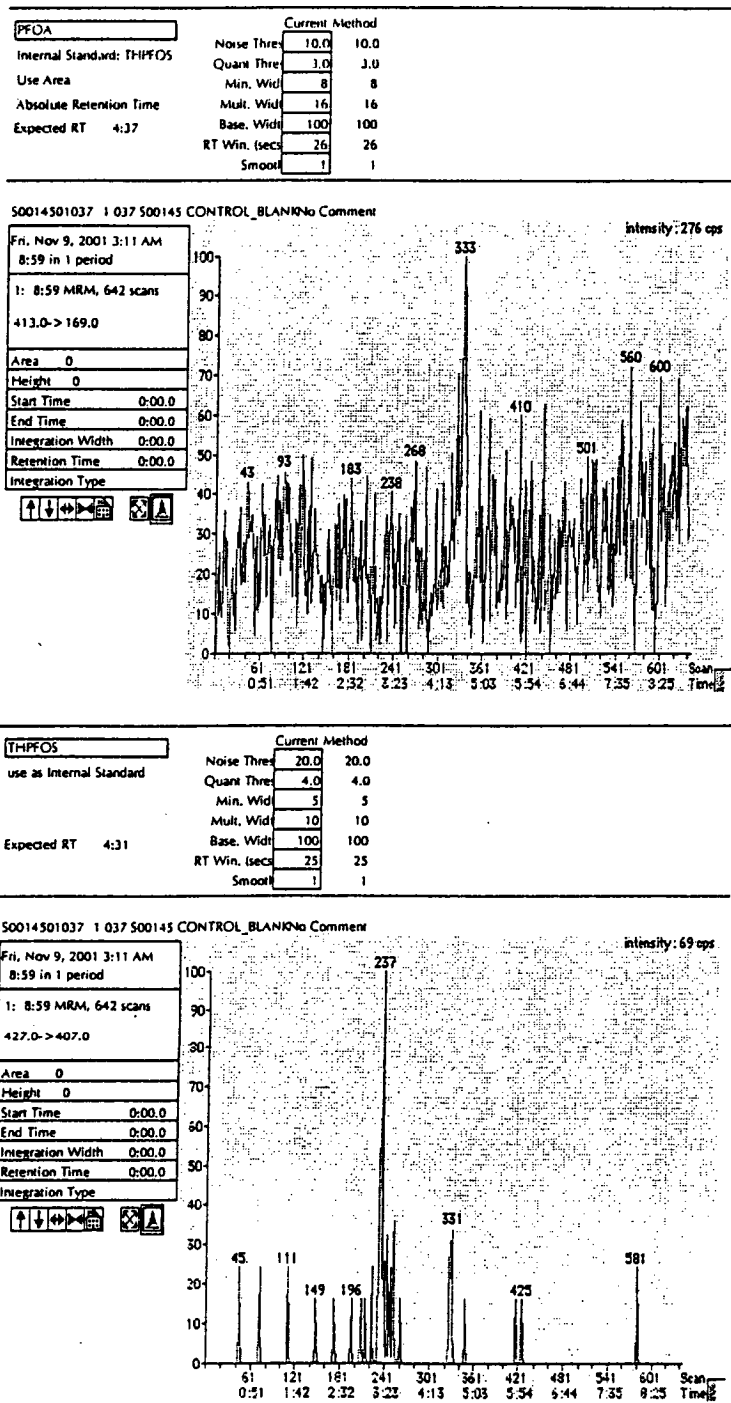


Figure 10. Human Plasma Blank for PFHS

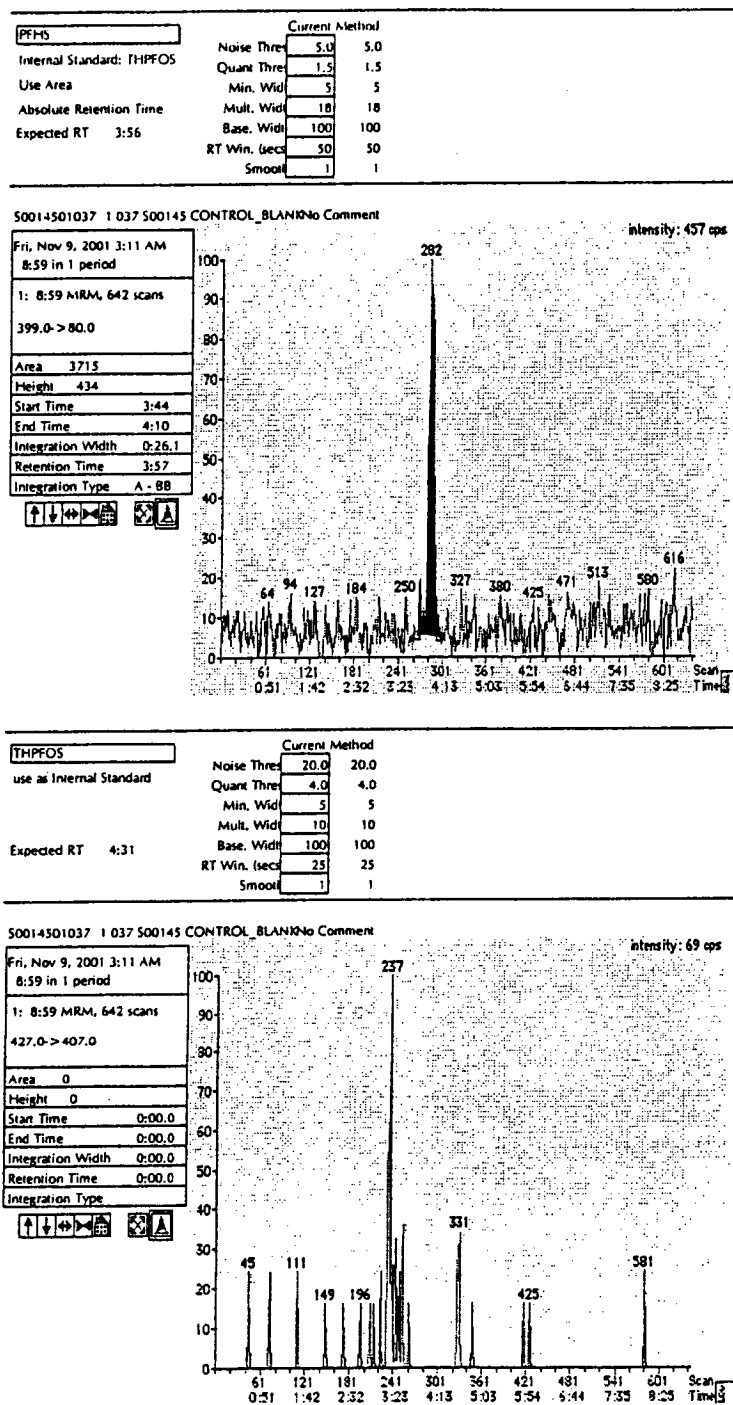
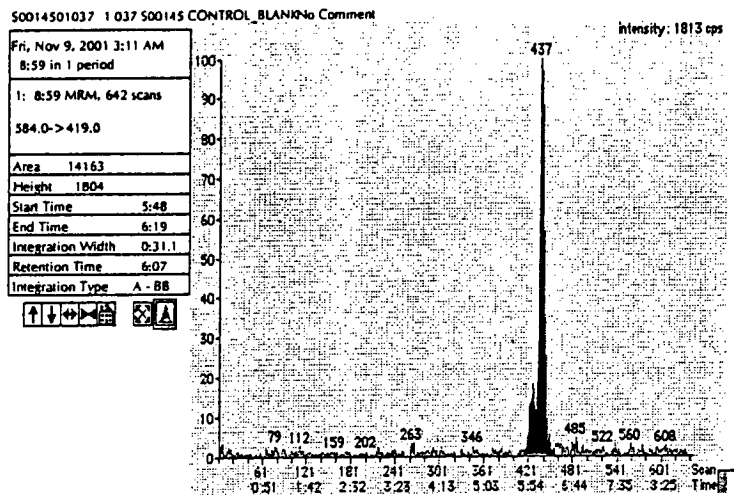


Figure 11. Human Plasma Blank for PFOSAA

PFOSAA	Current Method
Internal Standard: THPFOS	Noise Thresh 5.0 5.0
Use Area	Quant Thresh 1.3 1.3
Absolute Retention Time	Min. Width 6 6
Expected RT 6:03	Mult. Width 17 17
	Base. Width 100 100
	RT Win. (secs) 20 20
	Smooth 1 1



THPFOS	Current Method
use as Internal Standard	Noise Thresh 20.0 20.0
	Quant Thresh 4.0 4.0
	Min. Width 5 5
	Mult. Width 10 10
	Base. Width 100 100
Expected RT 4:31	RT Win. (secs) 25 25
	Smooth 1 1

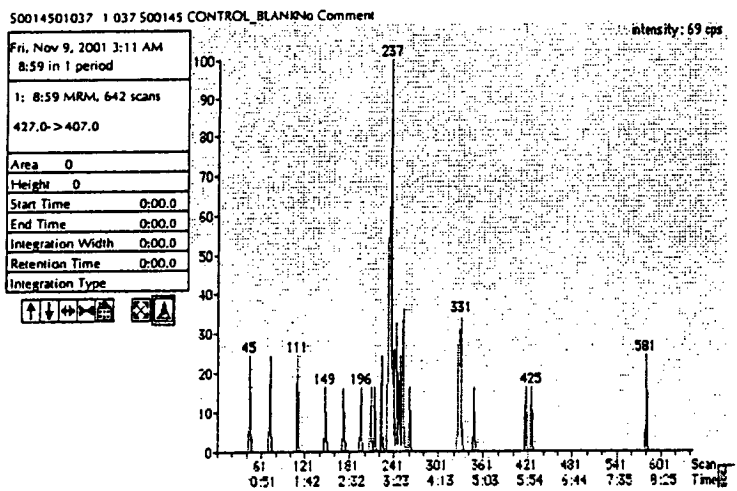


Figure 12. Human Plasma Blank for PFOSA

PFOSA		Current Method	
Internal Standard: THPFOS	Noise Thre	7.0	7.0
Use Area	Quant Thre	1.5	1.5
Absolute Retention Time	Min. Wid	5	5
Expected RT 5:51	Multi. Wid	17	17
	Base. Wid	100	100
	RT Win. (sec)	20	20
	Smooth	1	1

S0014501037 1 037 S00145 CONTROL_BLANK-No Comment

Fri, Nov 9, 2001 3:11 AM
8:59 in 1 period1: 8:59 MRM, 642 scans
497.9 > 77.9

Area 0

Height 0

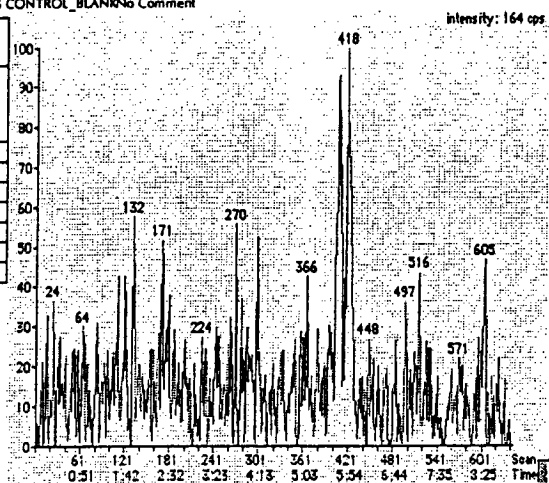
Start Time 0:00.0

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 Thre	20.0
Quant Thre	4.0
Min. Wid	5
Multi. Wid	10
Base. Wid	100
RT Win. (sec)	25
Smooth	1

S0014501037 1 037 S00145 CONTROL_BLANK-No Comment

Fri, Nov 9, 2001 3:11 AM
8:59 in 1 period1: 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

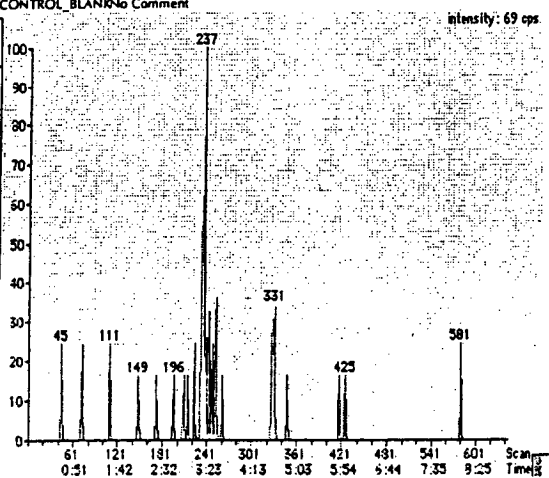


Figure 13. Human Plasma Blank for M556

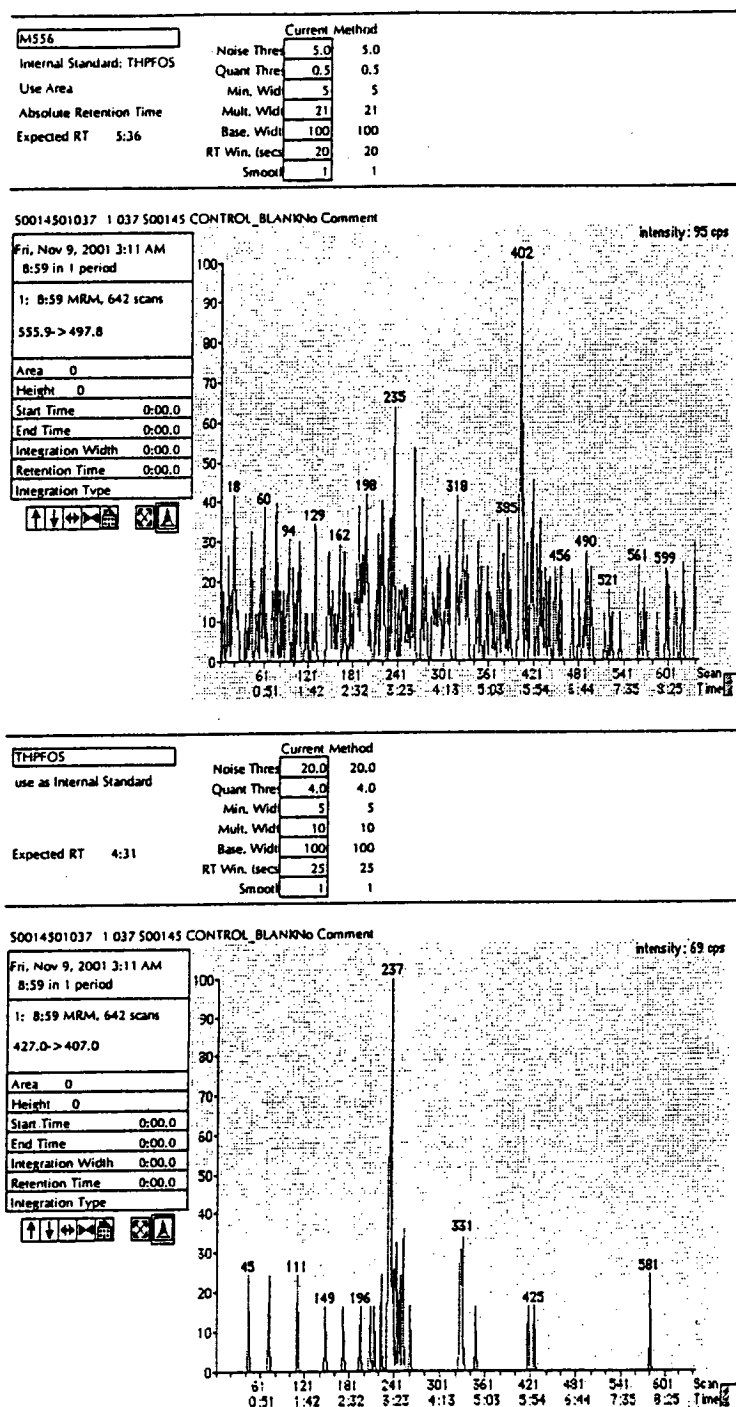
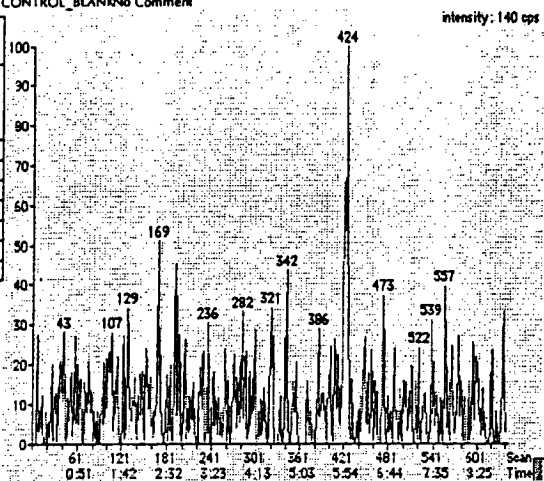


Figure 14. Human Plasma Blank for M570

M570	Current Method
Internal Standard: THPFOS	Noise Thres 5.0 5.0
Use Area	Quant Thres 1.0 1.0
Absolute Retention Time	Min. Wid 5 5
Expected RT 5:54	Mult. Wid 20 20
	Base. Wid 100 100
	RT Win. (secs) 20 20
	Smooth 1 1

50014501037 1 037 500145 CONTROL_BLANK No Comment

Fri, Nov 9, 2001 3:11 AM
8:59 in 1 period1: 8:59 MRM, 642 scans
570.0 > 419.0Area 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

50014501037 1 037 500145 CONTROL_BLANK No Comment

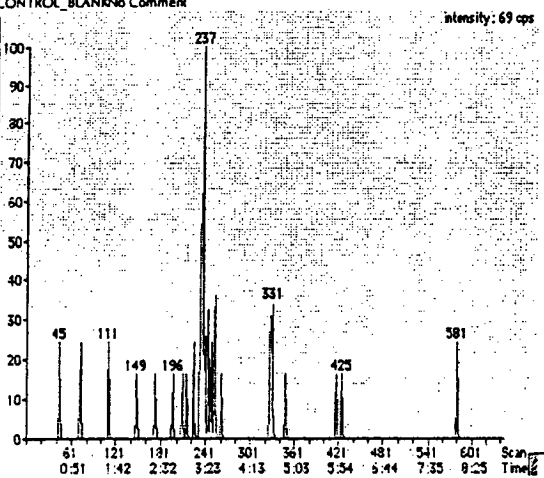
Fri, Nov 9, 2001 3:11 AM
8:59 in 1 period1: 8:59 MRM, 642 scans
427.0 > 407.0Area 0
Height 0
Start Time 0:00.0
End Time 0:00.0
Integration Width 0:00.0
Retention Time 0:00.0
Integration Type

Figure 15. Human Plasma Blank with Internal Standard (QC0) for PFOS

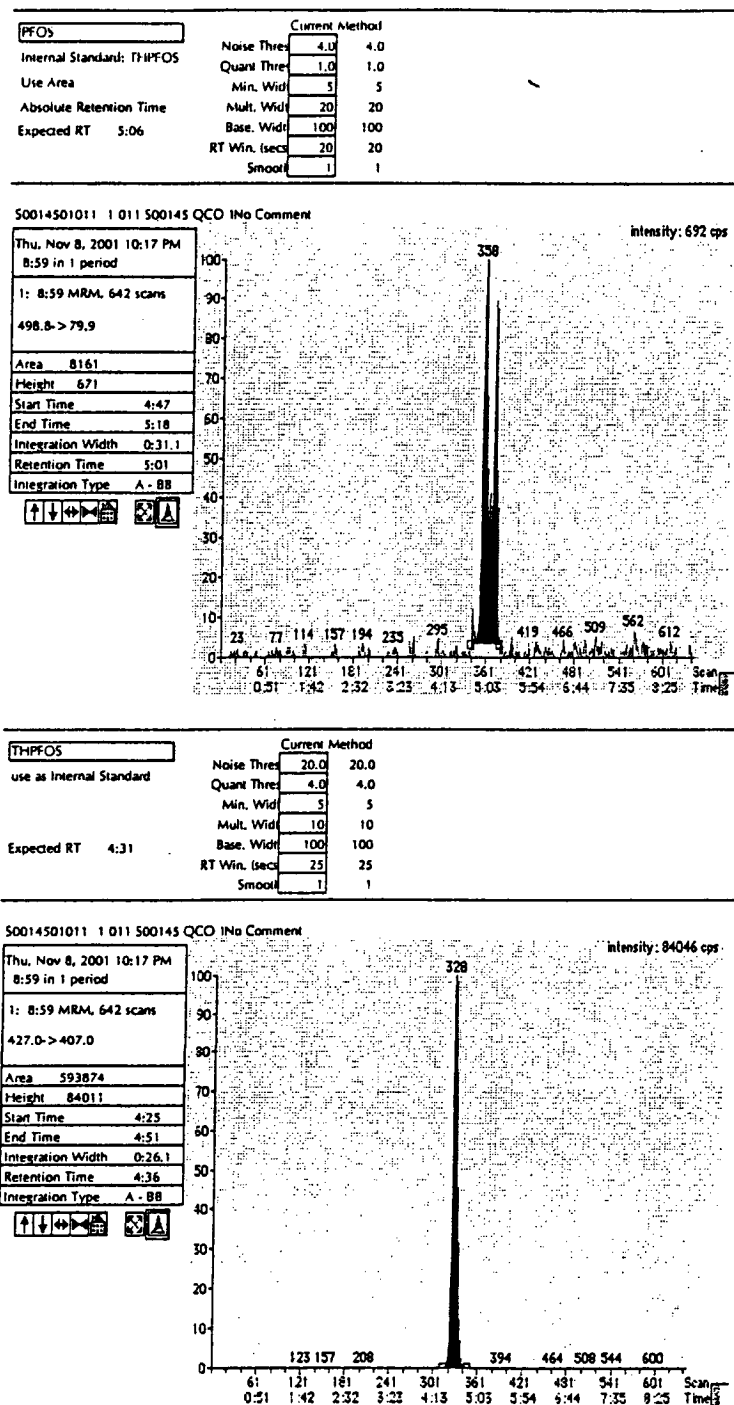


Figure 16. Human Plasma Blank with Internal Standard (QC0) for PFOA

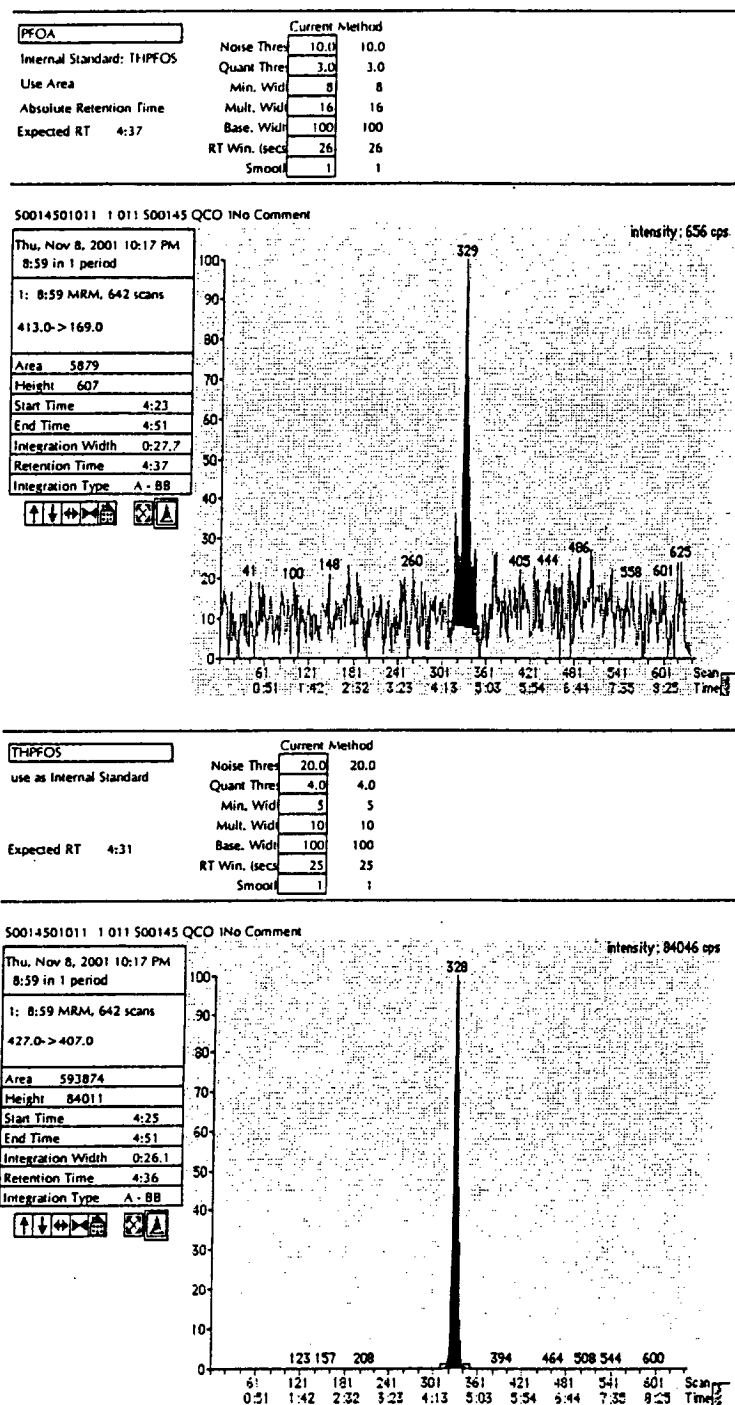


Figure 17. Human Plasma Blank with Internal Standard (QC0) for PFHS

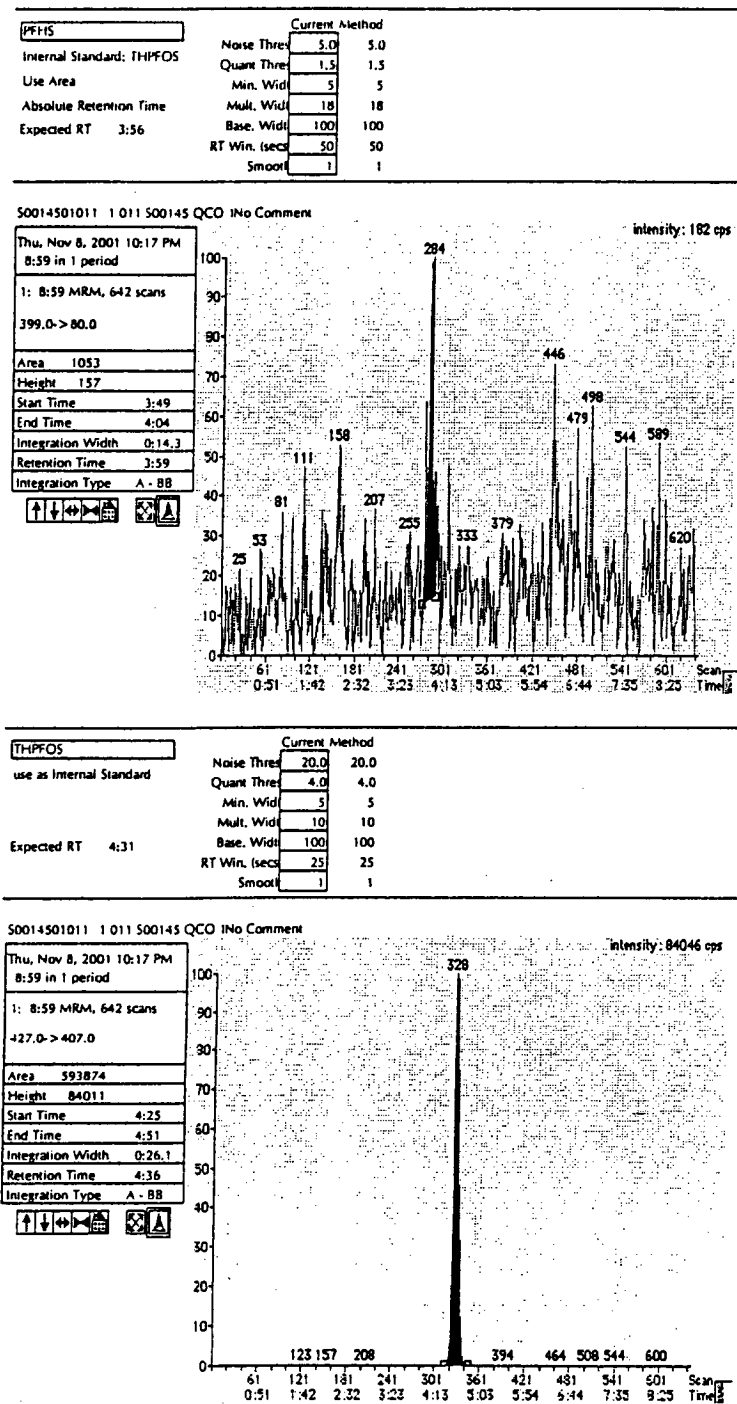


Figure 18. Human Plasma Blank with Internal Standard (QC0) for PFOSAA

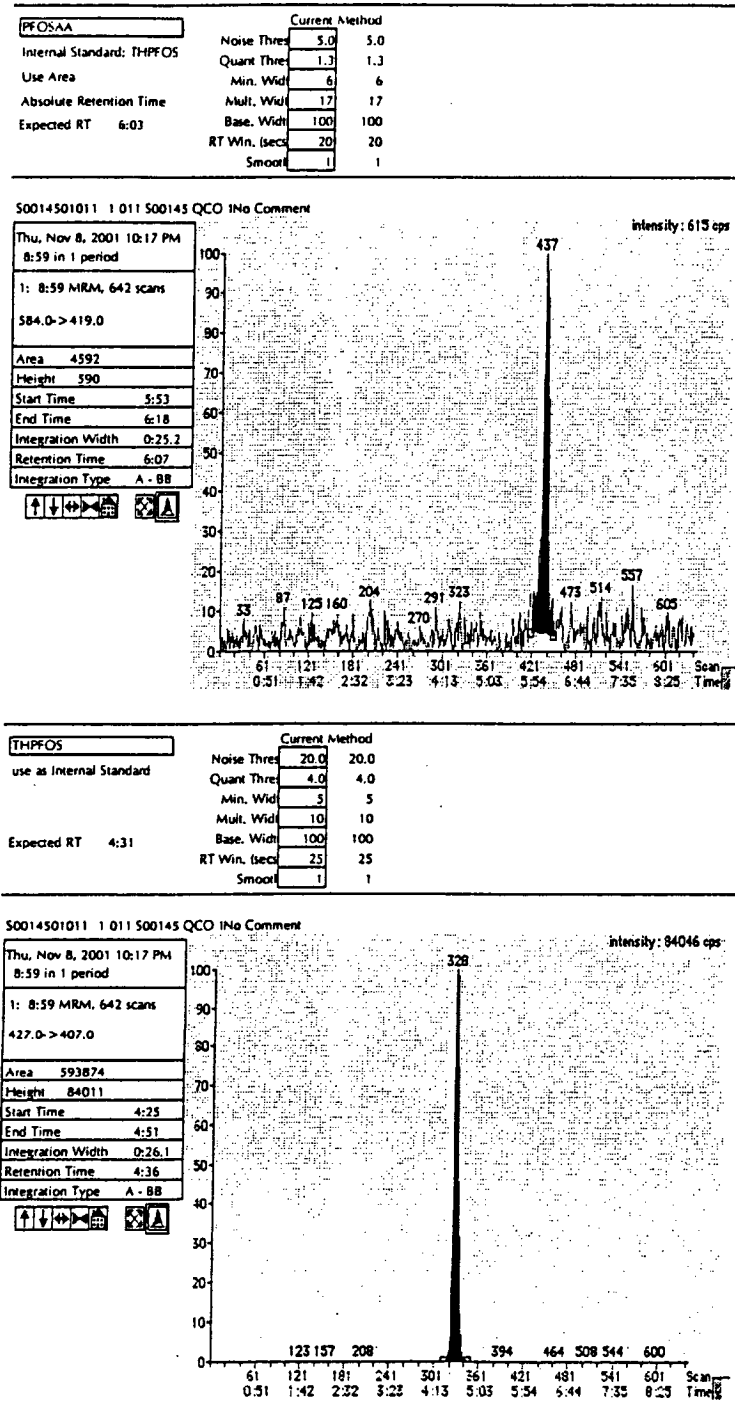


Figure 19. Human Plasma Blank with Internal Standard (QC0) for PFOSA

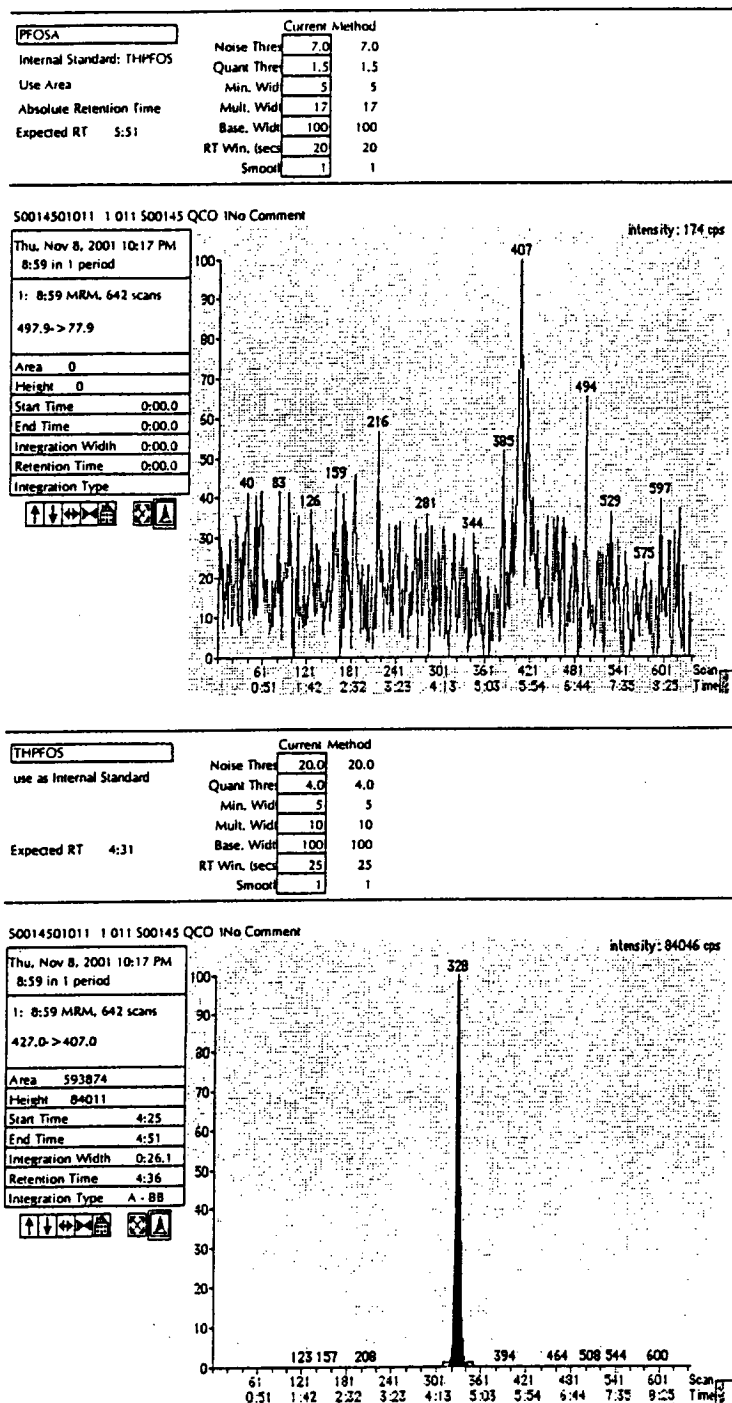


Figure 20. Human Plasma Blank with Internal Standard (QC0) for M556

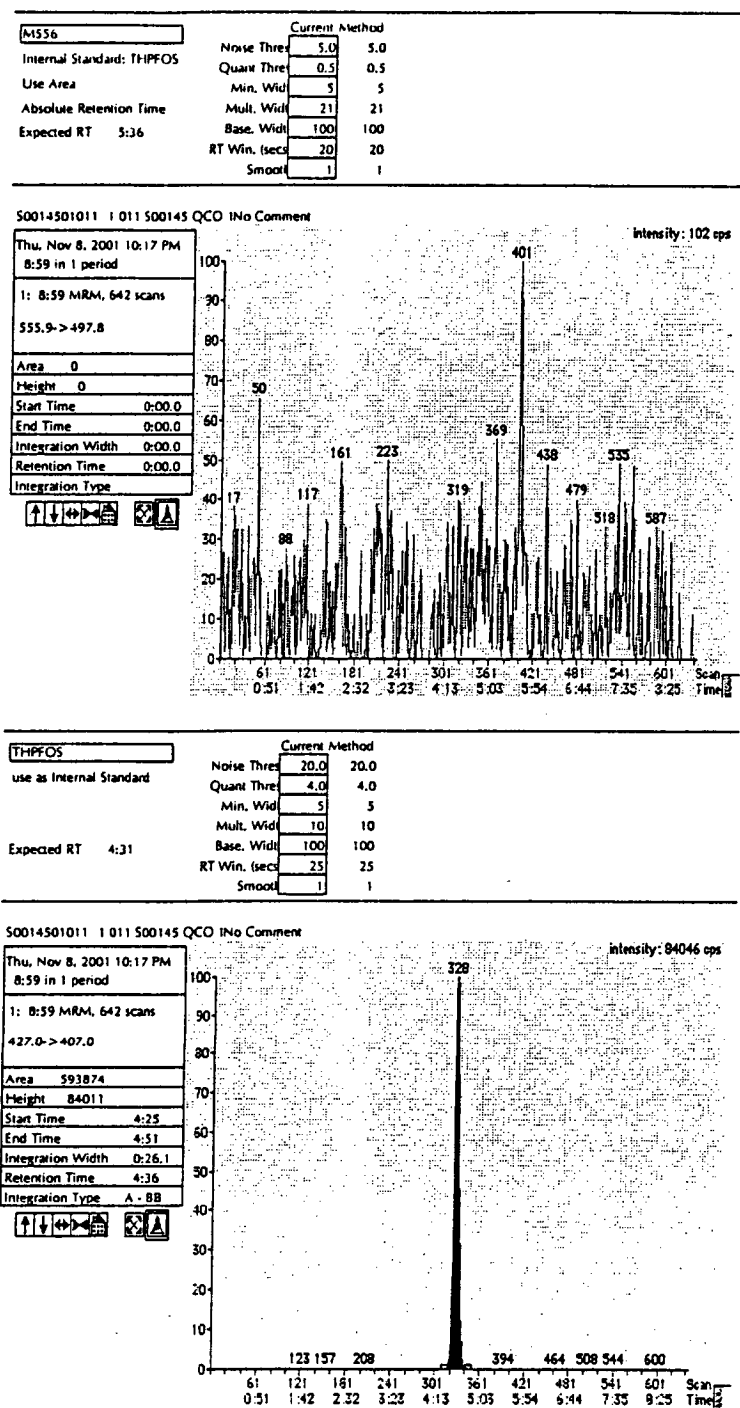


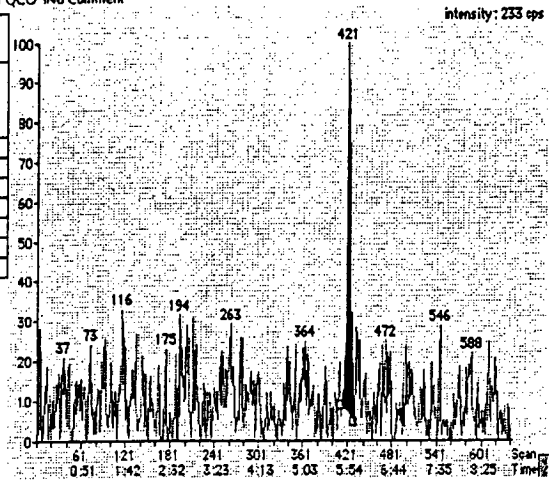
Figure 21. Human Plasma Blank with Internal Standard (QC0) for M570

M570	Current Method	
Internal Standard: THPFOS	Noise Thresh	5.0
Use Area	Quant Thresh	1.0
Absolute Retention Time	Min. Width	5
Expected RT 5:54	Mult. Width	20
	Base. Width	100
	RT Win. (secs)	20
	Smooth	1

S0014501011 1 011 S00145 QC0 INo Comment

Thu, Nov 8, 2001 10:17 PM
8:59 in 1 period1: 8:59 MRM, 642 scans
570.0 > 419.0

Area	1030
Height	217
Start Time	5:47
End Time	5:50
Integration Width	0:12.6
Retention Time	5:54
Integration Type	A - BB



THPFOS	Current Method	
use as Internal Standard	Noise Thresh	20.0
	Quant Thresh	4.0
	Min. Width	5
	Mult. Width	10
Expected RT 4:31	Base. Width	100
	RT Win. (secs)	25
	Smooth	1

S0014501011 1 011 S00145 QC0 INo Comment

Thu, Nov 8, 2001 10:17 PM
8:59 in 1 period1: 8:59 MRM, 642 scans
427.0 > 407.0

Area	593874
Height	84011
Start Time	4:25
End Time	4:51
Integration Width	0:26.1
Retention Time	4:36
Integration Type	A - BB

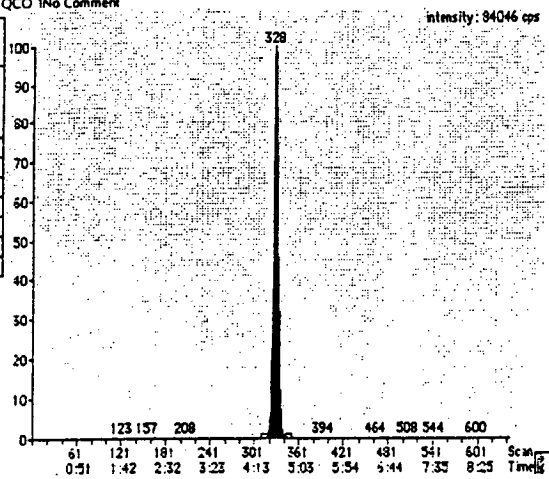


Figure 22. Low Standard for PFOS

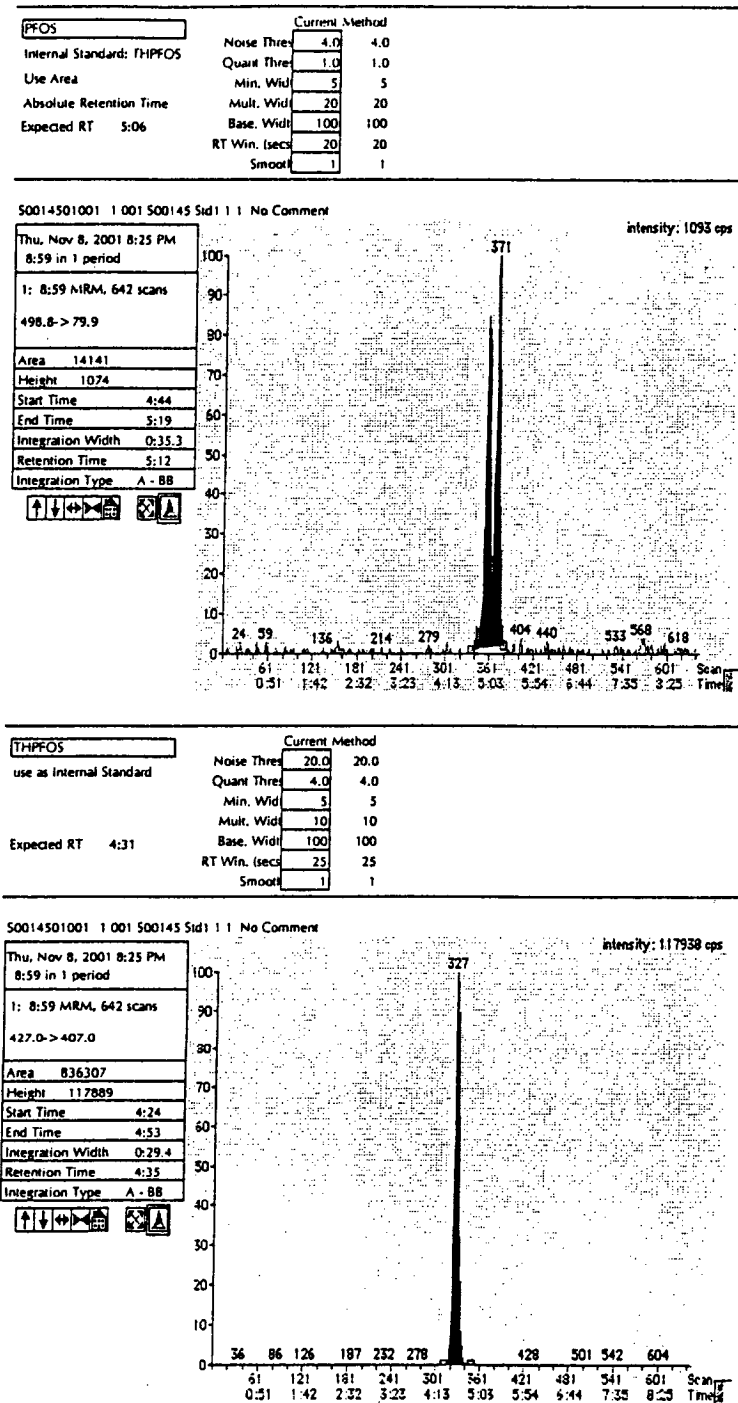


Figure 23. Low Standard for PFOA

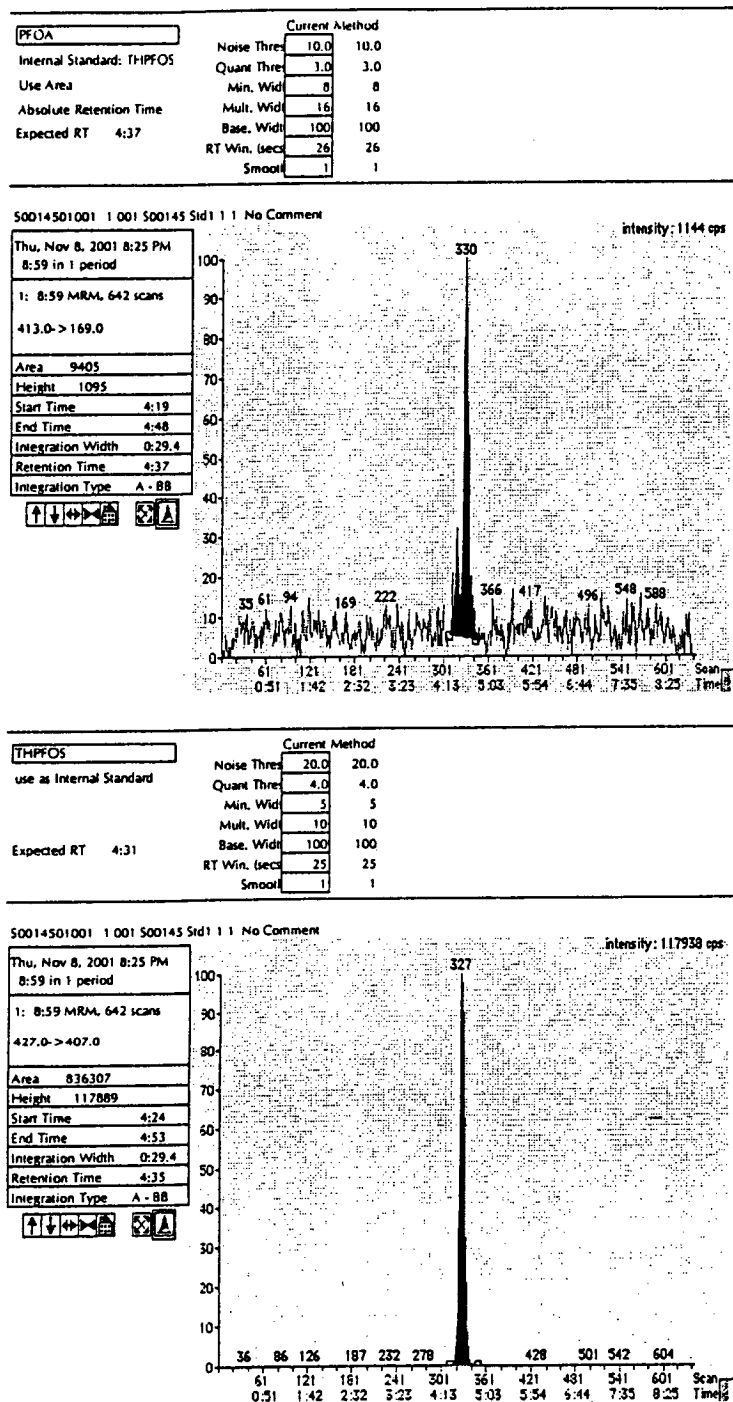


Figure 24. Low Standard for PFHS

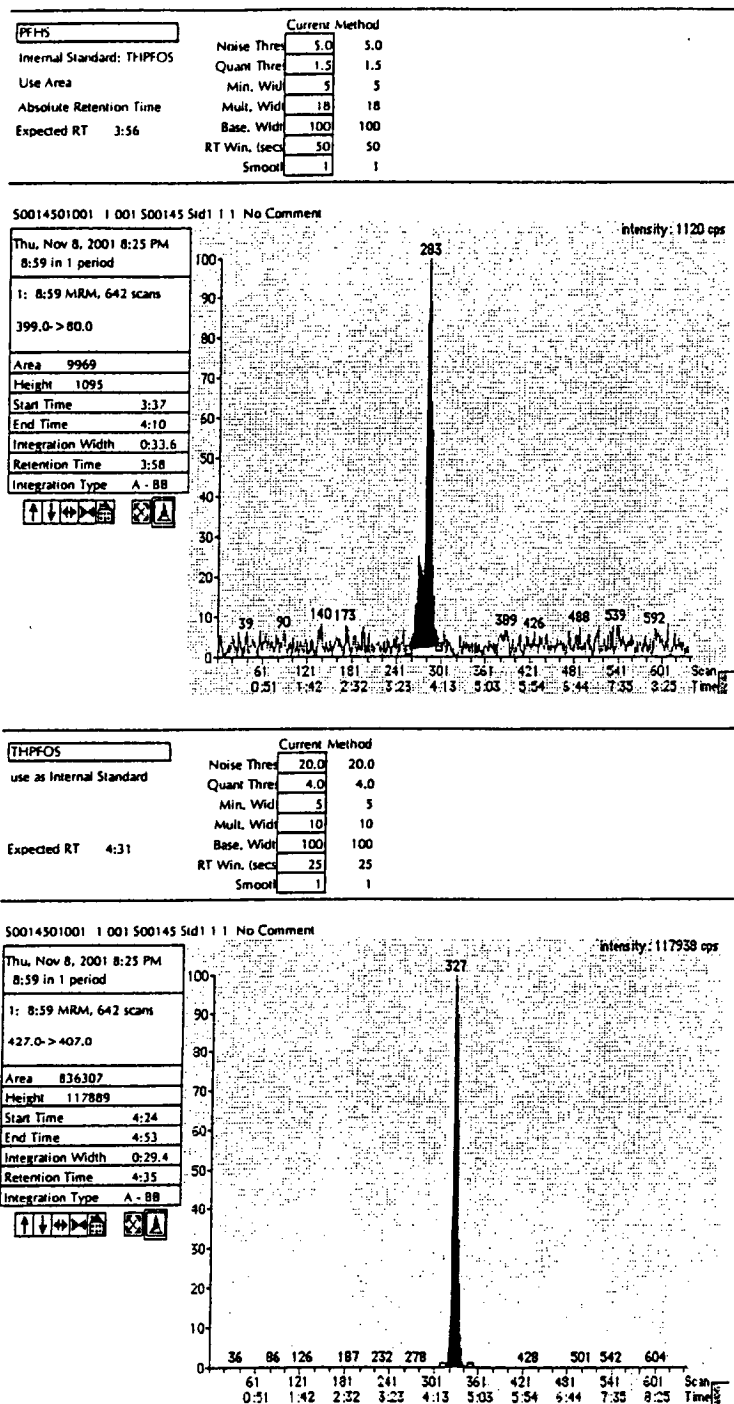


Figure 25. Low Standard for PFOSAA

PFOSAA		Current Method	
Internal Standard: THPFOS	Noise Thre	5.0	5.0
Use Area	Quant Thre	1.3	1.3
Absolute Retention Time	Min. Wid	6	6
Expected RT 6:03	Mult. Wid	17	17
	Base. Wid	100	100
	RT Win. (sec)	20	20
	Smooth	1	1

S0014S01001 1 001 S00145 Sid1 1 1 No Comment

Thu, Nov 8, 2001 8:25 PM
8:59 in 1 period

1: 8:59 MRM, 642 scans

584.0 > 419.0

Area 6942

Height 943

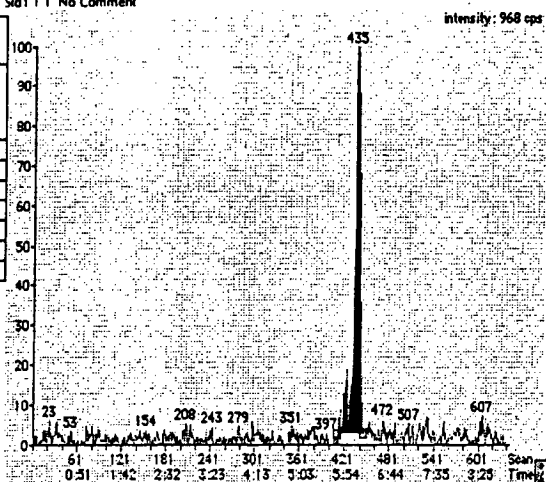
Start Time 5:47

End Time 6:15

Integration Width 0:27.7

Retention Time 6:06

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. (sec)	25	25
	Smooth	1	1

S0014S01001 1 001 S00145 Sid1 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

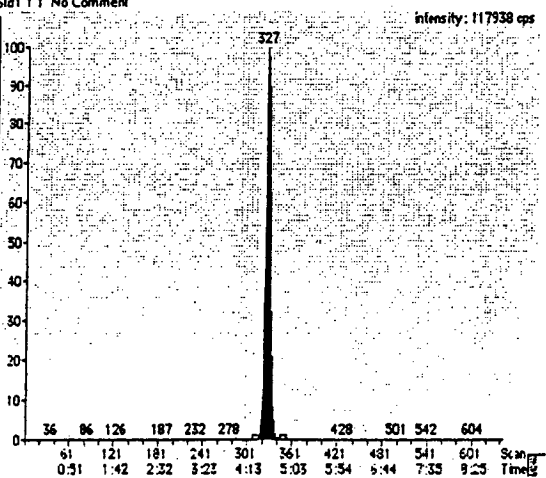


Figure 26. Low Standard for PFOSA

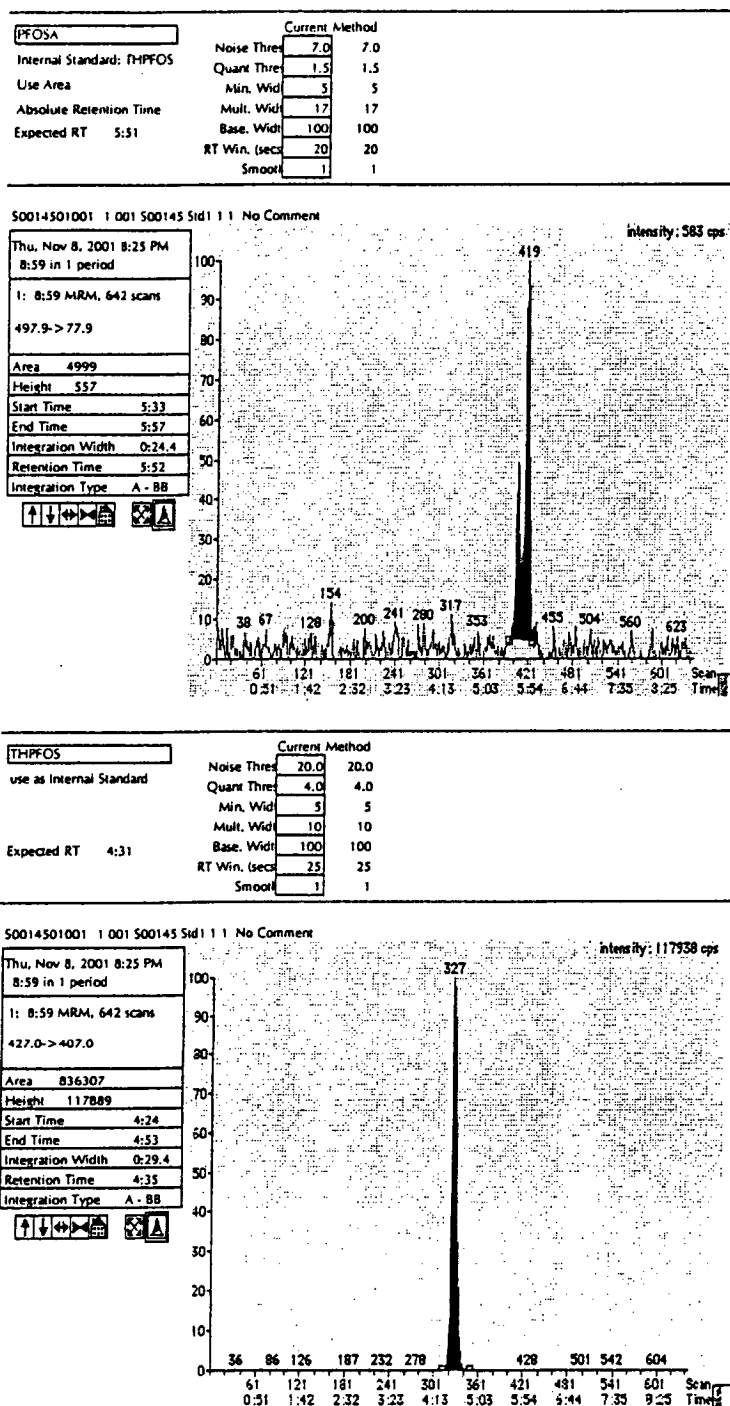
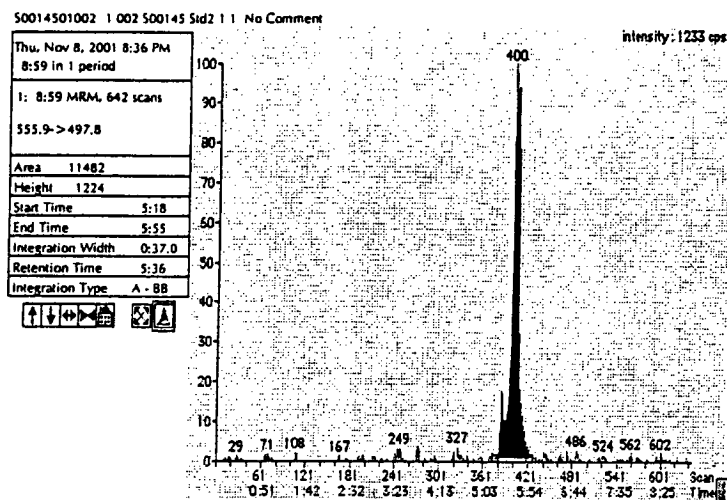


Figure 27. Low Standard for M556

M556	Current Method
Internal Standard: THPFOS	Noise Thresh 5.0 5.0
Use Area	Quant Thresh 0.5 0.5
Absolute Retention Time	Min. Width 5 5
Expected RT 5:36	Mult. Width 21 21
	Base. Width 100 100
	RT Win. (secs) 20 20
	Smooth 1 1



THPFOS	Current Method
use as Internal Standard	Noise Thresh 20.0 20.0
	Quant Thresh 4.0 4.0
	Min. Width 5 5
	Mult. Width 10 10
Expected RT 4:31	Base. Width 100 100
	RT Win. (secs) 25 25
	Smooth 1 1

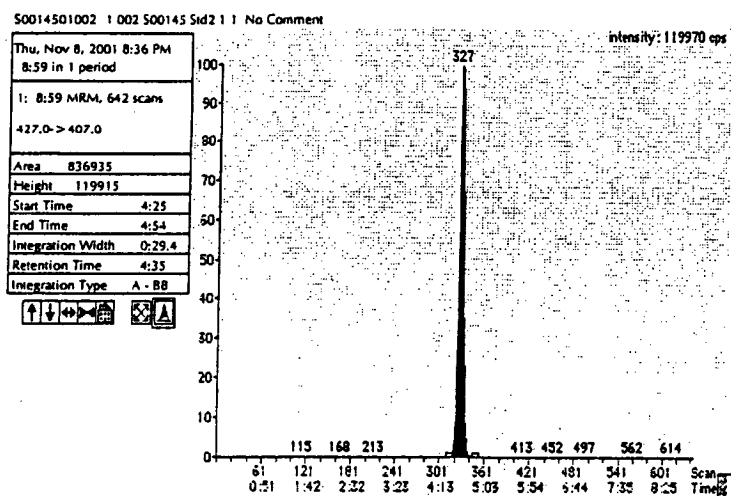


Figure 28. Low Standard for M570

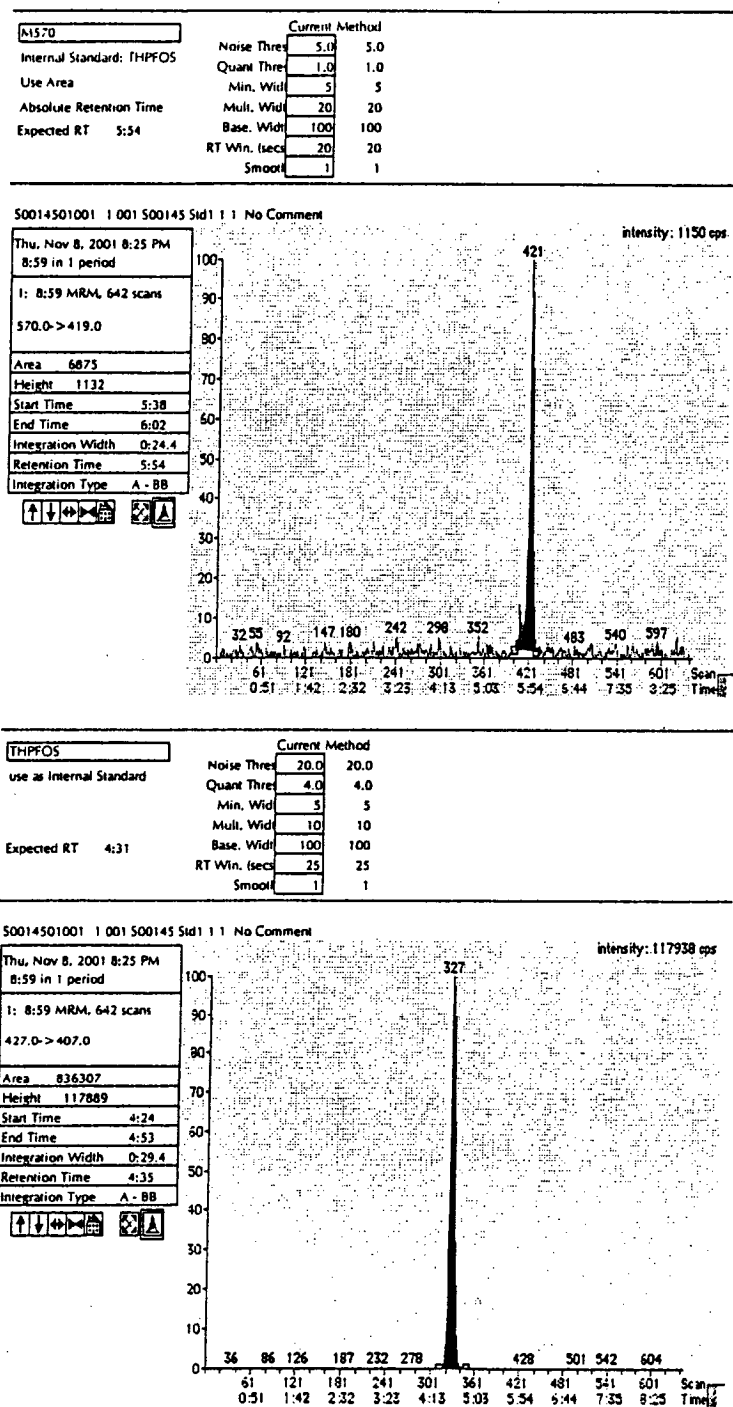


Figure 29. High Standard for PFOS

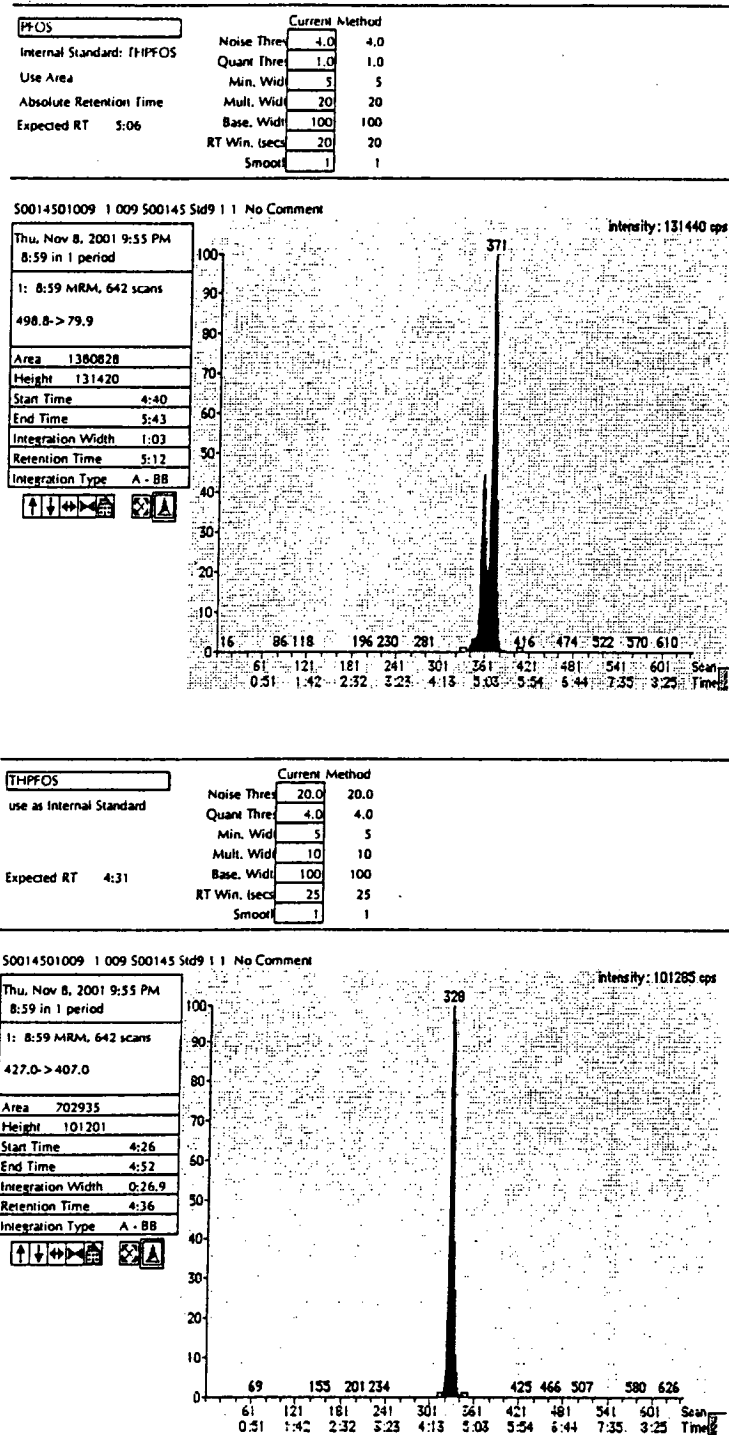


Figure 30. High Standard for PFOA

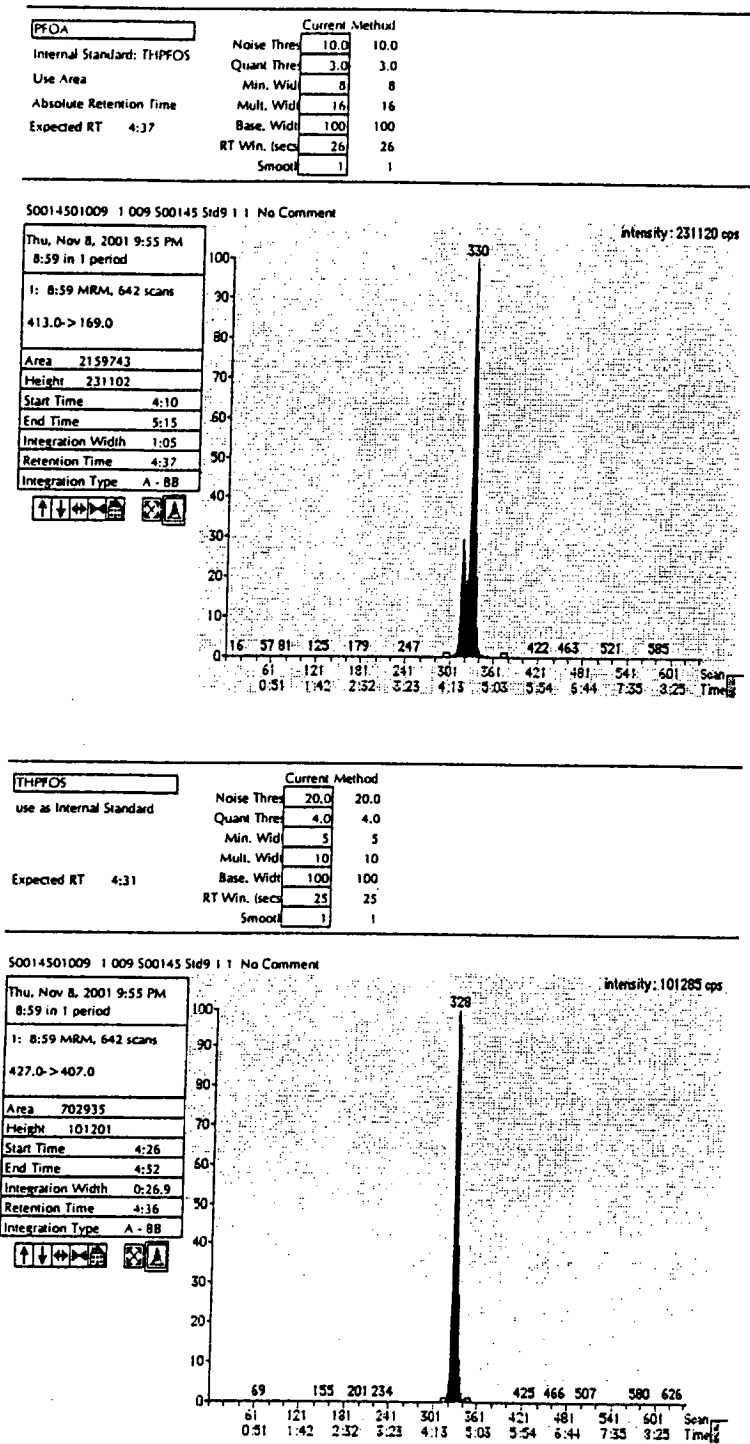


Figure 31. High Standard for PFHS

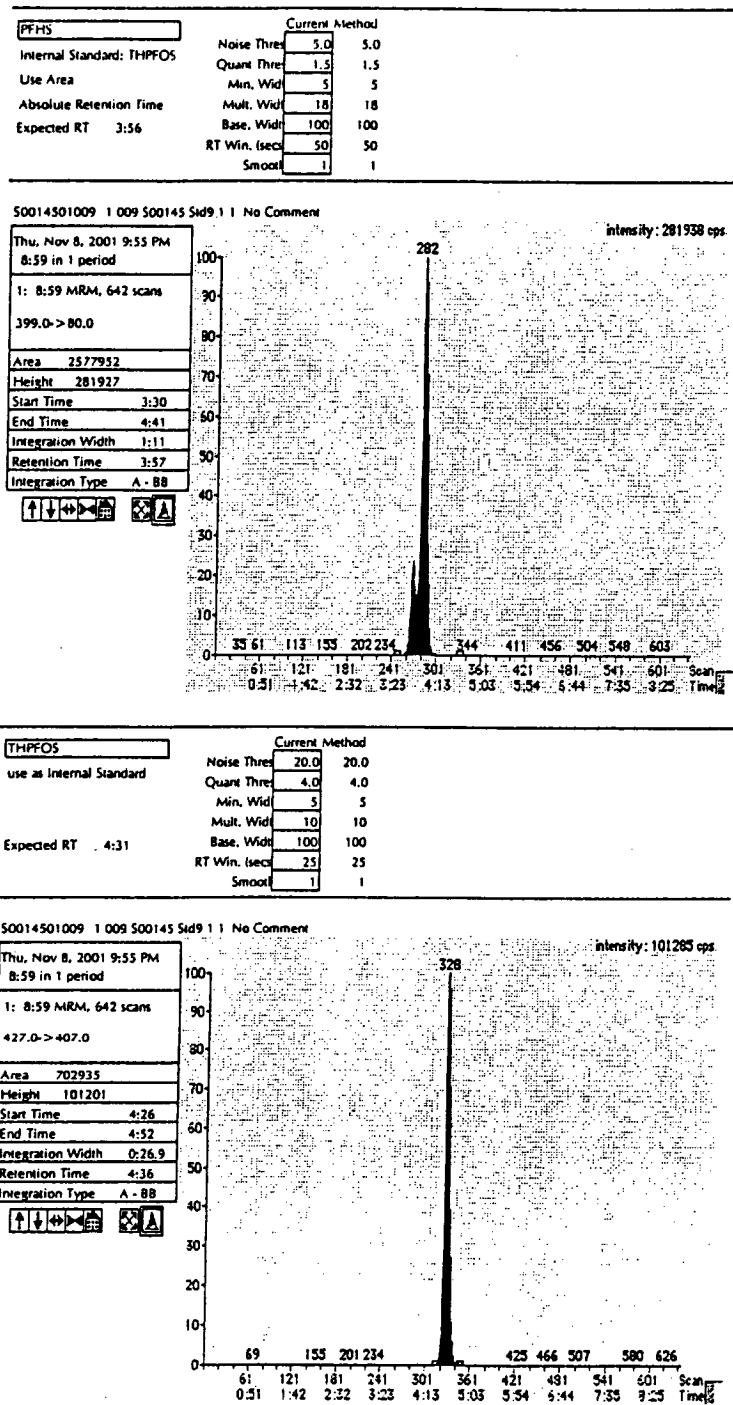
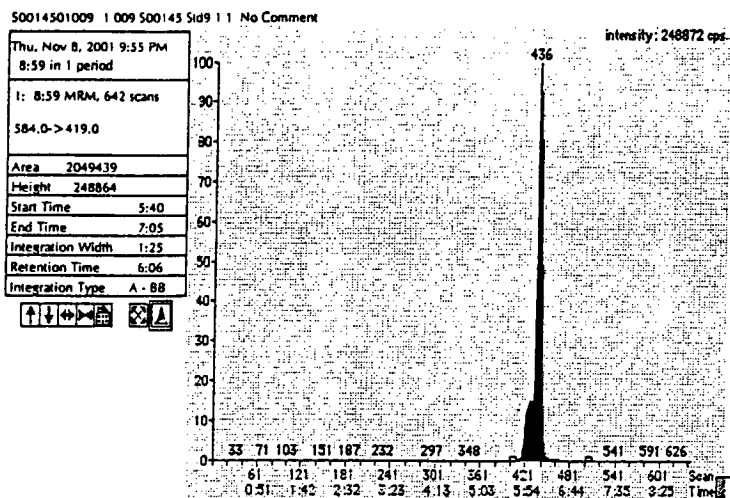


Figure 32. High Standard for PFOSAA

PFOSAA	Current Method
Internal Standard: THPFOS	Noise Thres 5.0 5.0
Use Area	Quant Thres 1.3 1.3
Absolute Retention Time	Min. Wid 6 6
Expected RT 6:03	Mult. Wid 17 17
	Base. Wid 100 100
	RT Win. (secs) 20 20
	Smooth 1 1



THPFOS	Current Method
use as Internal Standard	Noise Thres 20.0 20.0
	Quant Thres 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

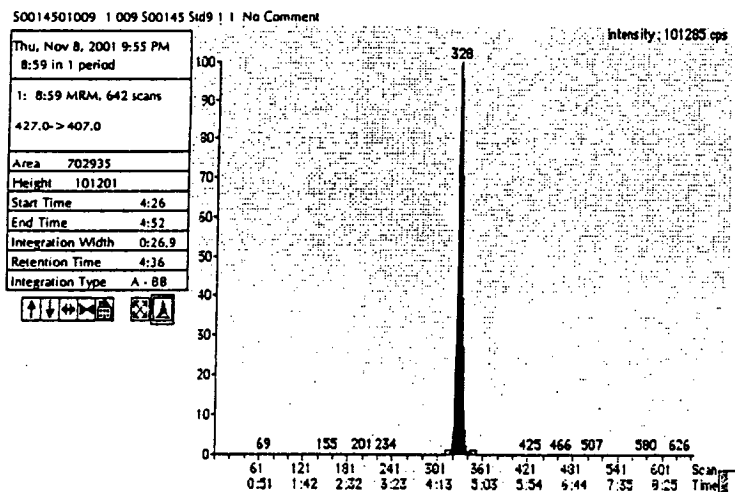


Figure 33. High Standard for PFOSA

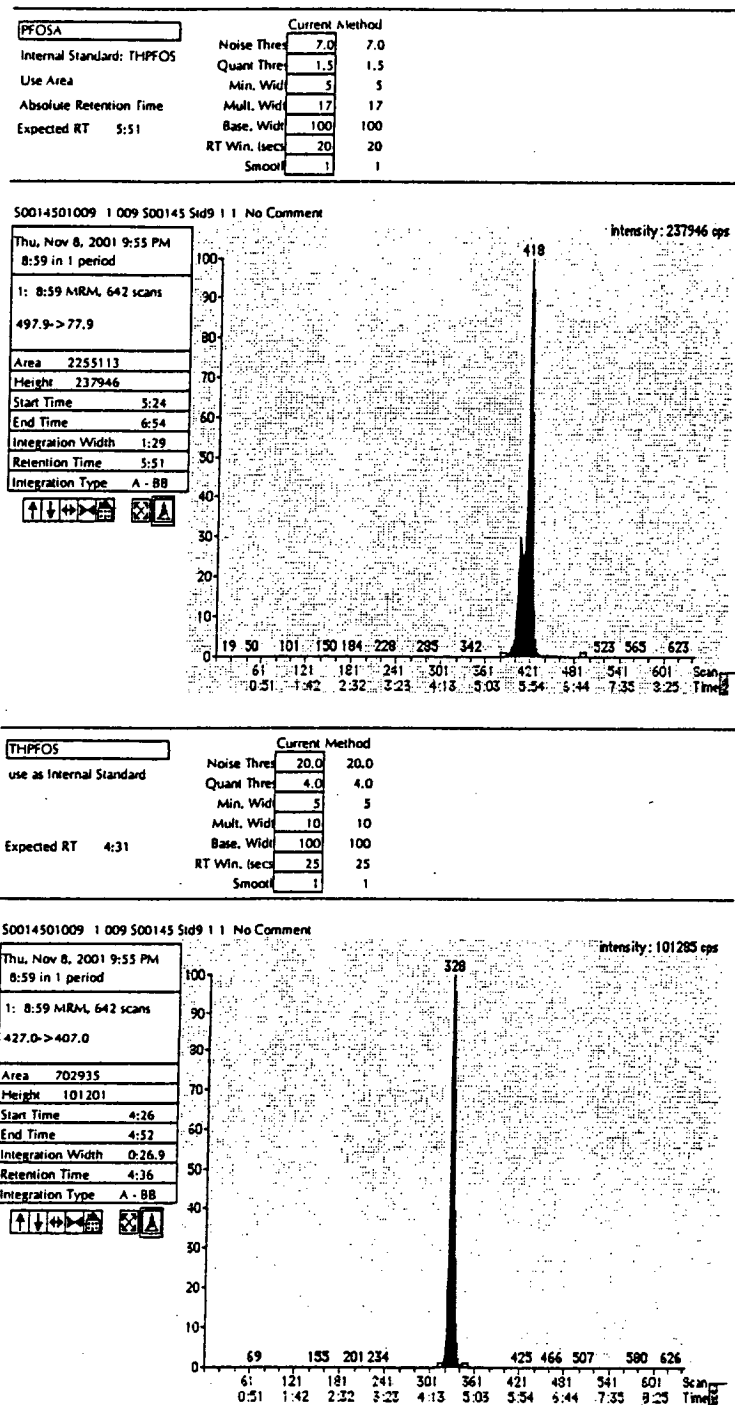


Figure 34. High Standard for M556

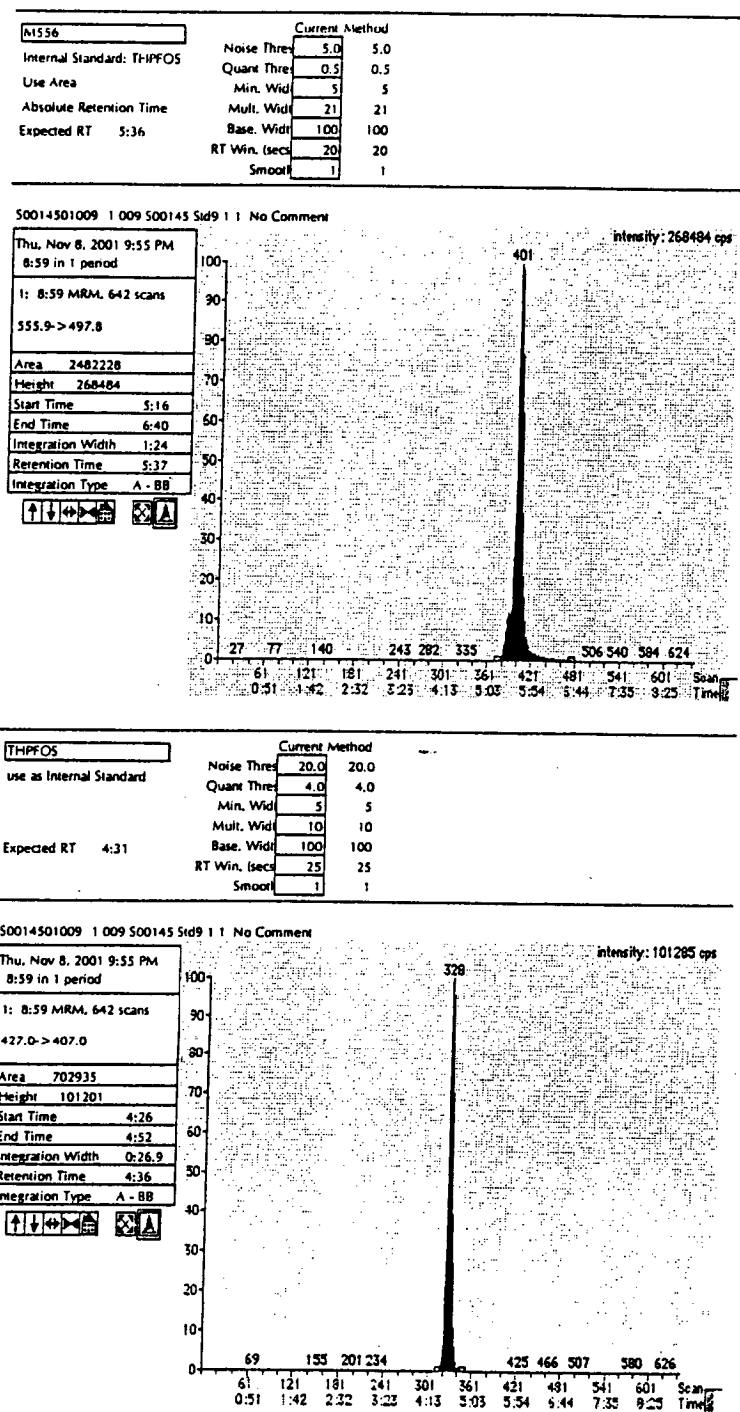


Figure 35. High Standard for M570

