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

Sample Analysis Report for Protocol EPI-PEuro

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QUALITY ASSURANCE STATEMENT

LABORATORY:	Northwest Bioanalytical (NWB) A Division NWT, Inc. 1121 East 3900 South Salt Lake City, UT 84124
SPONSOR:	3M Environmental Technology Services (3M) 935 Bush Avenue St. Paul, MN 55133
COMPOUND(S):	PFOS, PFOSA, PFOSAA, PFHS, POAA, M556 and M570
NWB STUDY NUMBER:	NWBS99-117
SPONSOR STUDY NUMBER:	EPI-PEuro
NWB STUDY TITLE:	Extraction and Analysis of PFOS and other Fluorochemicals in Human Serum by HPLC/Electrospray Mass Spectrometry

The clinical sample analysis 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 Inspection Report Issued To	
Inspection Date	Phase of Study	NWB Project Manager	*NWB Management
27–30 Aug 1999	Sample Receipt	30 Aug 1999	31 Aug 1999
03 Sep 1999	Analytical Plan Draft	03 Sep 1999	30 Sep 1999
08 Sep 1999	Analytical Plan	08 Sep 1999	30 Sep 1999
17–22 Sep 1999	Sample Analysis	22 Sep 1999	30 Sep 1999
01 Dec 1999	Report Draft/Raw Data	02 Dec 1999	30 Dec 1999
09 Dec 1999	Final Report	09 Dec 1999	30 Dec 1999

*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

09 December 1999
Date

Compliance Statement

The clinical sample analysis study described within this report is not included within the definition of a GLP regulated nonclinical study set forth in Title 21 of the U.S. Code of Federal Regulations Part 58. 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.) There were no known incidents that affected the quality or integrity of the project or data reported. This report represents an accurate record of the raw data.

David Vollmer, Ph.D.
NWB Project Manager

12/8/99
Date

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

12/9/99
Date

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1. INTRODUCTION

This report summarizes the analytical results from the quantitation of PFOS, PFOSA, PFOSAA, PFHS, POAA, M556 and M570 in human serum samples for 3M Environmental Technology and Services in support of Protocol EPI-PEuro [5.1]. The validated LC/MS/MS method provided a lower limit of quantitation (LLOQ) of 31.4 (PFOS), 1.00 (PFOSA), 1.45 (PFOSAA), 2.61 (PFHS) and 5.27 (POAA) ppb. All analytes exhibited a linear response to approximately 500 ppb with the exception of PFOSAA, which had an upper limit of quantitation (ULOQ) of 270 ppb [5.2]. The non-validated method for the quantitation for M556 and M570 in human serum provided a lower limit of quantitation (LLOQ) of 1.00 and 5.00 ppb, respectively, and exhibited a linear response to 500 ppb for both analytes.

The testing facility was 3M Corporate Occupational Medicine (Bldg. 220-3W-05, St. Paul, MN 55144-1000), and Jean Burris at the facility served as the Principal Investigator. The following is a list of NWB supervisory personnel involved in the completion of this work: David Vollmer, Ph.D. (NWB Project Manager); Rodger L. Foltz, Ph.D. (NWB Laboratory Director).

NWB SOPs were used in the conduct of this study and were available to study personnel in electronic and/or hard copy formats.

Date Study Initiated: September 7, 1999 Date Analyses Completed: September 20, 1999

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 validated assay used for this study is Meth98082.00 and was reported in Northwest Bioanalytical report NWBR99-005 [5.2].

Samples for this study were received at NWB on the following date:

Receipt Date	*Number of Samples Received*	*Storage Condition (except during analysis)*
August 27, 1999	18	– 20°C

Reference Material

Analyte	*Lot Number*	*Purity*	*Expiration Date*	*Source*	*Storage Conditions*
*PFOS (FC-95)	193	100%	12/31/2010	3M	Room Temperature (dry)
PFOSA	214	100%	12/31/2010	3M	Room Temperature
*PFOSAA (FC-129)	617	53.8%	12/31/2010	3M	Room Temperature
PFHS	S398-182	100%	12/31/2010	3M	– 20°C
*POAA (FC 143)	245	100%	12/31/2010	3M	Room Temperature (dry)
M556	NB 113047-80	99.89%	12/31/2010	3M	Room Temperature
M570	118506-26	99.75%	12/31/2010	3M	Room Temperature
SDS (sodium dodecylsulfate)	17H0459	99%	1/16/2001	Sigma	Room Temperature

* Analyte names used throughout this report do not include their FC identifier; for example, PFOS is synonymous with PFOS (FC-95)

Nine calibration standards were prepared fresh daily by adding the appropriate spiking standard solution into 0.200 mL of human serum. The final calibration standard concentrations in human serum for each analyte were as follows:

Analyte	*Calibration Standard Concentrations (ppb)*
PFOS	31.4, 35.4, 40.4, 55.4, 80.4, 130, 280, 430, 530
PFOSA	1.00, 5.00, 10.0, 25.0, 50.0, 100, 250, 400, 500
PFOSAA	1.45, 3.60, 6.29, 14.4, 27.8, 54.7, 135, 216, 270
PFHS	2.61, 6.61, 11.6, 26.6, 51.6, 102, 252, 402, 502
POAA	5.27, 9.27, 14.3, 29.3, 54.3, 104, 254, 404, 504
M556	1.00, 5.00, 10.0, 25.0, 50.0, 100, 250, 400, 500
M570	1.00, 5.00, 10.0, 25.0, 50.0, 100, 250, 400, 500

Analytical QCs were prepared in human serum on September 13, 1999 and frozen in a -20°C freezer. Analytical QCs at the following levels were assayed in duplicate in each analytical run:

Analyte	*QC Concentrations (ppb)*
PFOS	Low (50.4) and High (380)
PFOSA	Low (20.0) and High (350)
PFOSAA	Low (11.7) and High (189)
PFHS	Low (21.6) and High (352)
POAA	Low (24.3) and High (354)
M556	Low (20.0) and High (350)
M570	Low (20.0) and High (350)

After the addition of HPLC-grade water and sodium dodecyl sulfate (SDS, internal standard) to 0.200 mL of human 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.

3. SAMPLE ANALYSIS

The prepared samples, calibration standards and QCs were injected into the PE Sciex API 3000 LC/MS/MS system in a systematic order.

Chromatographic peak integration was performed using MacQuan software (version 1.6). Quadratic regression analysis (weighted 1/x) all analytes was performed using the peak area ratio vs. concentration utilizing Watson® DMLIMS software (version 5.4.10.2).

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 duplicate calibration standards at 7 or more concentrations covering the lower to upper limit of quantitation. At least two-thirds of the calibration standard's back-calculated concentrations must be within ± 15% (± 20% for LLOQ) of their individual target concentrations. If a calibration standard is not within the acceptance criteria, it is deactivated. This process starts from the highest calibration standard down to the lowest 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 is analyzed in duplicate for each dilution level. At least one dilution QC at each level must be within ± 20% in order to accept study sample results at that dilution level. The dilution QC acceptance was independent of the undiluted analytical QC acceptance.

4. RESULTS AND DISCUSSION

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	PFOSA Result	PFOSAA Result	PFHS Result	POAA Result	M556 Result	M570 Result	Comments
1	accepted	accepted	accepted	accepted	accepted	accepted	accepted	

There were no known circumstances that may have affected the quality or integrity of the data.

In conclusion, based on the quality control and calibration curve data, it is our opinion that the data submitted to 3M Environmental Technology and Services for PFOS, PFOSA, PFOSAA, PFHS, POAA, M556 and M570 are accurate for all of the EPI-PEuro study samples. Please note that the results for analytes M556 and M570 should be considered unvalidated data.

5. REFERENCES

5.1. 3M Protocol EPI-PEuro, "Analysis of Fluorochemicals in Pooled Sera from Europe," August 24, 1999.

5.2. D. Vollmer, "Quantitative Determination of PFOS, PFOSA, PFOSAA, N-MeFOSE-OH, N-EtFOSE-OH, POAA and PFHS in Human Serum by LC/MS/MS," NWB Study Number NWBS98-082, NWB Report Number NWBR99-005, May 13, 1999.

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 Environmental Technology and Services and 3M Corporate Occupational Medicine will be notified concerning final disposition of records at completion of contract obligations.

Table 1. Calibration Curve Summary for PFOS in Human Serum

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

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
17-Sep-1999	1	-0.000003	0.007758	0.060028	0.9961	31.4	530
Mean		-0.000003	0.007758	0.060028	0.9961		
n		1	1	1	1		

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

Table 2. Calibration Curve Summary for PFOSA in Human Serum

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

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
17-Sep-1999	1	-0.000007	0.016542	0.006140	0.9960	1.00	500
Mean		-0.000007	0.016542	0.006140	0.9960		
n		1	1	1	1		

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

Table 3. Calibration Curve Summary for PFOSAA in Human Serum

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

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
17-Sep-1999	1	-0.000002	0.001907	0.002064	0.9971	1.45	270
Mean		-0.000002	0.001907	0.002064	0.9971		
n		1	1	1	1		

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

Table 4. Calibration Curve Summary for PFHS in Human Serum

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

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
17-Sep-1999	1	-0.000006	0.012069	0.031955	0.9980	6.61	502
Mean		-0.000006	0.012069	0.031955	0.9980		
n		1	1	1	1		

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

Table 5. Calibration Curve Summary for POAA in Human Serum

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

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
17-Sep-1999	1	-0.000006	0.014672	0.018212	0.9977	5.27	504
Mean		-0.000006	0.014672	0.018212	0.9977		
n		1	1	1	1		

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

Table 6. Calibration Curve Summary for M556 in Human Serum

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

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
17-Sep-1999	1	0.000000	0.000556	0.001320	0.9977	1.00	500
Mean		0.000000	0.000556	0.001320	0.9977		
n		1	1	1	1		

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

Table 7. Calibration Curve Summary for M570 in Human Serum

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

Run Date	Run Number	A	B	C	R-Squared	LLOQ	ULOQ
17-Sep-1999	1	-0.000001	0.002742	0.015241	0.9984	5.00	500
Mean		-0.000001	0.002742	0.015241	0.9984		
n		1	1	1	1		

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

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

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

Run Date	Run Number	31.4	35.4	40.4	55.4	80.4	130	280	430	530
17-Sep-1999	1	29.7	34.4	41.4	57.2	88.2	126	266	444	490
		27.5	35.9	42.2	56.2	84.3	130	278	417	581
Mean		28.6	35.2	41.8	56.7	86.3	128	272	431	536
S.D.		1.56	1.06	0.566	0.707	2.76	2.83	8.49	19.1	64.3
%CV		5.5	3.0	1.4	1.2	3.2	2.2	3.1	4.4	12.0
%Bias		-8.9	-0.6	3.5	2.3	7.3	-1.5	-2.9	0.2	1.1
n		2	2	2	2	2	2	2	2	2

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

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

Run Date	Run Number	1.00	5.00	10.0	25.0	50.0	100	250	400	500
17-Sep-1999	1	0.958	*7.04	10.3	*28.8	55.5	99.2	241	438	451
		0.852	*6.51	11.3	*30.0	51.2	88.5	259	377	542
Mean		0.905		10.8		53.4	93.9	250	408	497
S.D.		0.0750		0.707		3.04	7.57	12.7	43.1	64.3
%CV		8.3		6.5		5.7	8.1	5.1	10.6	12.9
%Bias		-9.5		8.0		6.8	-6.1	0.0	2.0	-0.6
n		2		2		2	2	2	2	2

* Sample deactivated as an outlier (> ±15% theoretical for non-LLOQ; > ±20% theoretical for LLOQ)

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

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

Run Date	Run Number	1.45	3.60	6.29	14.4	27.8	54.7	135	216	270
17-Sep-1999	1	1.16	3.60	6.37	14.7	30.9	53.5	125	217	251
		*0.720	3.53	6.41	14.9	30.6	54.9	133	214	300
Mean		1.16	3.57	6.39	14.8	30.8	54.2	129	216	276
S.D.		N.C.	0.0495	0.0283	0.141	0.212	0.990	5.66	2.12	34.6
%CV		N.C.	1.4	0.4	1.0	0.7	1.8	4.4	1.0	12.5
%Bias		-20.0	-0.8	1.6	2.8	10.8	-0.9	-4.4	0.0	2.2
n		1	2	2	2	2	2	2	2	2

* Sample deactivated as an outlier (> ±15% theoretical for non-LLOQ; > ±20% theoretical for LLOQ)

N.C. = not calculable

Table 11. Back-Calculated Concentrations of Calibration Standards for PFHS in Human Serum

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

Run Date	Run Number	2.61	6.61	11.6	26.6	51.6	102	252	402	502
17-Sep-1999	1	*0.735	5.57	10.9	27.3	58.3	101	232	404	491
		*0.571	6.57	11.7	27.7	56.4	102	243	412	523
Mean			6.07	11.3	27.5	57.4	102	238	408	507
S.D.			0.707	0.566	0.283	1.34	0.707	7.78	5.66	22.6
%CV			11.6	5.0	1.0	2.3	0.7	3.3	1.4	4.5
%Bias			-8.2	-2.6	3.4	11.2	0.0	-5.6	1.5	1.0
n			2	2	2	2	2	2	2	2

* Sample deactivated as an outlier (> ±15% theoretical for non-LLOQ; > ±20% theoretical for LLOQ)

Table 12. Back-Calculated Concentrations of Calibration Standards for POAA in Human Serum

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

Run Date	Run Number	5.27	9.27	14.3	29.3	54.3	104	254	404	504
17-Sep-1999	1	4.41	9.29	14.7	30.1	60.2	99.9	235	403	480
		4.35	9.85	15.0	31.4	59.3	103	246	421	533
Mean		4.38	9.57	14.9	30.8	59.8	101	241	412	507
S.D.		0.0424	0.396	0.212	0.919	0.636	2.19	7.78	12.7	37.5
%CV		1.0	4.1	1.4	3.0	1.1	2.2	3.2	3.1	7.4
%Bias		-16.9	3.2	4.2	5.1	10.1	-2.9	-5.1	2.0	0.6
n		2	2	2	2	2	2	2	2	2

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

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

Run Date	Run Number	1.00	5.00	10.0	25.0	50.0	100	250	400	500
17-Sep-1999	1	1.03	4.49	*7.17	21.3	50.5	103	239	394	467
		*<LLOQ	*2.56	10.1	27.7	54.9	101	248	401	544
Mean		1.03	4.49	10.1	24.5	52.7	102	244	398	506
S.D.		N.C.	N.C.	N.C.	4.53	3.11	1.41	6.36	4.95	54.4
%CV		N.C.	N.C.	N.C.	18.5	5.9	1.4	2.6	1.2	10.8
%Bias		3.0	-10.2	1.0	-2.0	5.4	2.0	-2.4	-0.5	1.2
n		1	1	1	2	2	2	2	2	2

* Sample deactivated as an outlier (> ±15% theoretical for non-LLOQ; > ±20% theoretical for LLOQ)

N.C. = not calculable

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

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

Run Date	Run Number	1.00	5.00	10.0	25.0	50.0	100	250	400	500
17-Sep-1999	1	*<LLOQ	4.82	10.4	27.9	*61.7	102	240	425	485
		*<LLOQ	4.17	9.69	26.3	54.1	96.9	242	386	517
Mean			4.50	10.0	27.1	54.1	99.5	241	406	501
S.D.			0.460	0.502	1.13	N.C.	3.61	1.41	27.6	22.6
%CV			10.2	5.0	4.2	N.C.	3.6	0.6	6.8	4.5
%Bias			-10.0	0.0	8.4	8.2	-0.5	-3.6	1.5	0.2
n			2	2	2	1	2	2	2	2

* Sample deactivated as an outlier (> ±15% theoretical for non-LLOQ; > ±20% theoretical for LLOQ)

N.C. = not calculable

Table 15. Analytical QC Summary for PFOS in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 50.4 ppb	High QC 380 ppb
17-Sep-1999	1	49.9	393
		49.4	322
		53.5	369
Mean		50.9	361
S.D.		2.24	36.1
%CV		4.4	10.0
%Theoretical		101.0	95.0
%Bias		1.0	-5.0
n		3	3

Table 16. Analytical QC Summary for PFOSA in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 20.0 ppb	High QC 350 ppb
17-Sep-1999	1	18.0	302
		20.0	*264
		18.5	280
Mean		18.8	282
S.D.		1.04	19.1
%CV		5.5	6.8
%Theoretical		94.0	80.6
%Bias		-6.0	-19.4
n		3	3

* > ±20%theoretical

Table 17. Analytical QC Summary for PFOSAA in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 11.7 ppb	High QC 189 ppb
17-Sep-1999	1	11.0	186
		10.1	156
		11.0	184
Mean		10.7	175
S.D.		0.520	16.8
%CV		4.9	9.6
%Theoretical		91.5	92.6
%Bias		-8.5	-7.4
n		3	3

Table 18. Analytical QC Summary for PFHS in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 21.6 ppb	High QC 352 ppb
17-Sep-1999	1	21.7	374
		20.9	313
		22.6	356
Mean		21.7	348
S.D.		0.850	31.3
%CV		3.9	9.0
%Theoretical		100.5	98.9
%Bias		0.5	-1.1
n		3	3

Table 19. Analytical QC Summary for POAA in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 24.3 ppb	High QC 354 ppb
17-Sep-1999	1	23.5	362
		22.9	311
		25.6	357
Mean		24.0	343
S.D.		1.42	28.1
%CV		5.9	8.2
%Theoretical		98.8	96.9
%Bias		-1.2	-3.1
n		3	3

Table 20. Analytical QC Summary for M556 in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 20.0 ppb	High QC 350 ppb
17-Sep-1999	1	16.7	290
		*15.6	281
		18.5	352
Mean		16.9	308
S.D.		1.46	38.7
%CV		8.6	12.6
%Theoretical		84.5	88.0
%Bias		-15.5	-12.0
n		3	3

* > ±20%theoretical

Table 21. Analytical QC Summary for M570 in Human Serum

All concentrations are expressed as ppb.

Run Date	Run Number	Low QC 20.0 ppb	High QC 350 ppb
17-Sep-1999	1	19.7	340
		18.2	280
		19.2	342
Mean		19.0	321
S.D.		0.764	35.2
%CV		4.0	11.0
%Theoretical		95.0	91.7
%Bias		-5.0	-8.3
n		3	3

12/16/99
All verified against Excel spreadsheet
JMB

Table 22. Study Sample Concentrations

All concentrations are expressed as ppb.

Sample ID	PFOS	PFOSA	PFOSAA	PFHS	POAA	M556	M570
9545	45.6	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	8.56	<LLOQ(1.00)	<LLOQ(5.00)
9546	39.7	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	7.94	<LLOQ(1.00)	<LLOQ(5.00)
9547	32.7	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	7.74	<LLOQ(1.00)	<LLOQ(5.00)
9548	32.6	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	7.49	3.89	<LLOQ(5.00)
9549	32.0	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	7.02	<LLOQ(1.00)	<LLOQ(5.00)
9550	41.7	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	8.35	<LLOQ(1.00)	<LLOQ(5.00)
9551	<LLOQ(31.4)	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	<LLOQ(5.27)	<LLOQ(1.00)	<LLOQ(5.00)
9552	<LLOQ(31.4)	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	<LLOQ(5.27)	<LLOQ(1.00)	<LLOQ(5.00)
9553	<LLOQ(31.4)	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	<LLOQ(5.27)	<LLOQ(1.00)	<LLOQ(5.00)
9554	<LLOQ(31.4)	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	<LLOQ(5.27)	<LLOQ(1.00)	<LLOQ(5.00)
9555	<LLOQ(31.4)	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	5.46	<LLOQ(1.00)	<LLOQ(5.00)
9556	43.7	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	8.13	<LLOQ(1.00)	<LLOQ(5.00)
9557	60.6	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	10.1	<LLOQ(1.00)	<LLOQ(5.00)
9558	42.2	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	10.0	<LLOQ(1.00)	<LLOQ(5.00)
9559	64.5	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	15.7	<LLOQ(1.00)	<LLOQ(5.00)
9560	38.9	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	9.01	<LLOQ(1.00)	<LLOQ(5.00)
9561	58.8	<LLOQ(1.00)	<LLOQ(1.45)	<LLOQ(6.61)	9.61	<LLOQ(1.00)	<LLOQ(5.00)
9565	*	*	*	*	*	*	*

( ) value in parentheses is the LLOQ
* The sample received was not in extractable form and was not analyzed.

Note
estimated LLOQ values for PFOS for Belgium (per Dan Vehlner) were:

9551 20.8
9552 20.7
9553 14.2
9554 4.9
9555 22.2

6.61 is curved standard failed @ 2.61