

February 1, 2005

**Data Validation for PFOS/PFOA
Oakdale Project
(MDH Sample Numbers 200501211-200501219)**

Sample receipt: Samples were received in good condition. Samples were delivered directly from the field but were not on ice. Samples were stored at 4°C $\pm$ 2°C until analysis.

Holding Times: Samples were collected January 25, 2005 and were delivered to the laboratory on January 25, 2005. Analysis of the samples took place on January 27, 2005. The analysis date was within 14 days of sample collection. No data were qualified.

LC/MS/MS Parameters:

HPLC System	Agilent 1100
Mobile Phase	Ammonium hydroxide in water, methanol
Sample Prep	All unknown, calibration, and QC samples were diluted by a factor of two with methanol prior to injection.
Mass Spec	Micromass Quattro Micro
Ionization Mode	Electrospray
Polarity	Negative
Transitions Monitored	PFOS (499>80; 499>99) PFOA (413>369; 413>169)

Method Detection Limit: MDLs of 0.100 ppb for PFOS and of 0.097 ppb for PFOA were determined by the analysis of 12 replicate samples over three days.

Instrument Calibration

Initial Calibration of Method: The PFOS/PFOA method utilizes a six point calibration curve. A new curve is analyzed for each batch of samples. Each calibration level must be accurate within $\pm$ 20% of the true value, except the lowest calibration point must be within $\pm$ 30% of the true value.

The six-point calibration curve spans a concentration range from 0.25-ug/L to 25-ug/L. Using linear regression with 1/x weighting, the correlation coefficient (r) for PFOS and PFOA were 0.999 and 0.999 respectively. A calibration curve is acceptable if $r \geq 0.990$ ($r^2 > 0.980$).

Continuing Calibrations:

Continuing calibration checks are run after a maximum of ten field samples. Two levels of continuing calibration (0.5 ppb and 10 ppb) are analyzed to verify the curve. Continuing calibration checks must be within 30% accuracy (70% – 130%) of the true value. Two sets of continuing calibration check standards were analyzed with this batch.

Continuing Calibration Results:

Analyte	Spike Amt	% Recovery	Spike Amt	% Recovery
PFOS	0.5 ppb	95	10 ppb	117
PFOA	0.5 ppb	111	10 ppb	99
PFOS	0.5 ppb	79	10 ppb	123
PFOA	0.5 ppb	97	10 ppb	100

Blanks

Method Blank: PFOS and PFOA were not detected in the method blank at $> \frac{1}{2}$ of the reporting limits. No data were qualified.

Instrument Blanks: Instrument blanks were run after every sample. PFOS and PFOA were not detected in the instrument blanks at $> \frac{1}{2}$ of the reporting limits. No carryover was observed.

Trip Blank: PFOS and PFOA (200501218) were not detected in the trip blank at $> \frac{1}{2}$ of the reporting limits. No data were qualified.

Surrogates: No surrogates were used for this method.

Internal Standards: No internal standards were used for this method.

Matrix Spikes/Matrix Spike Duplicates: A 500 uL aliquot of each sample was spiked with 10 ppb of PFOS and PFOA after receipt in the laboratory. The recovery must be within $\pm 30\%$. All matrix spikes were within the acceptable range. PFOS sample spike recoveries ranged from 105% to 116%. PFOA sample spike recoveries ranged from 88% to 113%.

Sample 200501215 (Field ID 105009 – Well #7) was used to generate accuracy and precision data. The spike recoveries for PFOS were 111% and 115% with an RPD of 3.8 %. The spike recoveries for PFOA were 97% and 99% with an RPD of 2.7 %. The recoveries met the QC criteria range of 70% to 130% with an RPD limit of less than 30%.

Laboratory Control Samples/Laboratory Control Sample Duplicates: Laboratory control waters were spiked with PFOS and PFOA for laboratory control samples at a concentration of 8 ppb. The accuracy of the LCS/LCSD samples must be within $\pm 20\%$ and the RPD within 30% to accept the sample batch. The PFOS spike recoveries were 108% and 108% respectively with an RPD of 0.1%. The PFOA spike recoveries were 85% and 88% respectively with an RPD of 3.4%.

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Method 553: PFOS/PFOA

Collected: 1/25/05

Analyzed: 1/27/05

Site: Oakdale

PFOS							Oakdale Well #
Sample ID	Field ID	Dataset	Measured Value	After Spike	Spike (%) Recovery	Reported Value (ppb)	
200501211	105005, E01	012705sa	ND	10.651	106.5	<0.5	1
200501212	105006, E02	012705sa	ND	11.131	111.3	<0.5	2
200501213	105007, E03	012705sa	ND	11.561	115.6	<0.5	3
200501214	105008, E04	012705sa	1.125	12.174	110.5	1.1	5
200501215 (duplicate)	105009, E06	012705sa	0.187 0.117	11.270 11.629	110.8 115.1	<0.5	7
200501216	105010, E07	012705sa	0.936	11.982	110.5	0.9	8
200501217	105011, E08	012705sa	0.613	12.136	115.2	0.6	9
200501218	trip blank	012705sa	ND	10.602	106.0	<0.5	Whisp. Oaks
200501219	105012, E01	012705sa	ND	10.486	104.9	<0.5	

PFOA							Oakdale Well #
Sample ID	Field ID	Dataset	Measured Value	After Spike	Spike (%) Recovery	Reported Value (ppb)	
200501211	105005, E01	012705sa	ND	11.291	112.9	<1.0	1
200501212	105006, E02	012705sa	ND	11.036	110.4	<1.0	2
200501213	105007, E03	012705sa	ND	10.737	107.4	<1.0	3
200501214	105008, E04	012705sa	0.799	11.091	102.9	<1.0	5
200501215 (duplicate)	105009, E06	012705sa	0.211 0.235	9.891 10.176	96.8 99.4	<1.0	7
200501216	105010, E07	012705sa	0.628	9.699	90.7	<1.0	8
200501217	105011, E08	012705sa	0.737	10.247	95.1	<1.0	9
200501218	trip blank	012705sa	ND	8.789	87.9	<1.0	Whisp. Oaks
200501219	105012, E01	012705sa	ND	8.859	88.6	<1.0	

Note:

1. All unknown samples are spiked with 10 ppb of PFOS and PFOA, and the measured value of a certain sample is accepted only if the spike recovery is within 70% to 130%.
2. One sample (200501215) was analyzed twice (laboratory duplicate) and each aliquot was spiked and analyzed.
3. The report level is 0.5 ppb for PFOS and 1.0 ppb for PFOA.
4. ND: Not Detected