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3M ENVIRONMENTAL LABORATORY
REPORT NO. E06-0302

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Final Report

Analysis of PFOA and PFOS in Aqueous Samples
from 3M Cordova Wells 1, 2, 3, 4, 5, and Drinking Water
Distribution

Laboratory Request Number: E06-0302

Method Requirement: 3M Method ETS-8-154.1 (modified)

Report Date - 08/16/06

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The testing reported herein meet the requirements of ISO/IEC 17025-1999 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Certificate #2052-01. Testing that complies with this International Standard also operate in accordance with ISO 9001/ISO 9002 (1994).

Certificate #2052-01

3M Environmental Laboratory
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3M Principal Analytical Investigator: Susan Wolf
Report Author: Susan Wolf

Analytical Report E06-0302

Report Date: August 16, 2006

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed water samples collected by Weston Solutions personnel on August 2, 2006, from Cordova wells 1, 2, 3, 4, 5, and Drinking Water Distribution. Samples were returned to the 3M Environmental Laboratory for analysis of perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) under laboratory project number E06-0302. Analysis was completed following 3M Environmental Laboratory method ETS-8-154.1 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates in Water by Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry".

The 3M Environmental Laboratory prepared sets of sample containers for six sampling locations. Each sample set consisted of a field sample, field sample duplicate, low field spike, mid field spike, and high field spike. Well 3 had an additional mid-high field matrix spike prepared. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 450 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing all analytes prior to being sent to the field for sample collection.

Samples were prepared on August 3, 2006 and analyzed on August 3 and 4, 2006.

Table 1 lists the sampling locations and corresponding well ID # that will be referenced throughout the report. Table 2 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.

Table 1. Sampling Locations

Sampling Location	Field Sampling Code ⁽¹⁾
Building 11; Well 1	CIL GW PW11
Building 12; Well 2	CIL GW PW12
Building 13; Well 3	CIL GW PW13
Building 24; Well 4	CIL GW PW24
Building 37; Well 5	CIL GW PW37
Bldg 1; DW Distribution	CIL PW PWL1

(1) See field sampling raw data file.



The testing reported herein meets the requirements of ISO/IEC 17025-1999 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Certificate #2052-01. Testing that complies with this International Standard also operates in accordance with ISO 9001/ISO 9002 (1994).

Certificate #2052-01

Table 2. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOA Concentration (ng/mL)
E06-0302-068	Well 1; Sample 060802	0.386	0.181
E06-0302-069	Well 1; Sample Duplicate 060802	0.462	0.250
Average Well 1		0.424	0.216
%RPD Sample/Sample Dup Well 1		18 ⁽²⁾	32 ⁽²⁾
E06-0302-073	Well 2; Sample 060802	2.72	0.692
E06-0302-074	Well 2; Sample Duplicate 060802	3.10	0.777
Average Well 2		2.91	0.735
%RPD Sample/Sample Dup Well 2		13	11
E06-0302-078	Well 3; Sample 060802	11.7	4.59
E06-0302-079	Well 3; Sample Duplicate 060802	11.9	4.68
Average Well 3		11.8	4.64
%RPD Sample/Sample Dup Well 3		1.8	1.9
E06-0302-083	Well 4; Sample 060802	1.71	0.436
E06-0302-084	Well 4; Sample Duplicate 060802	1.73	0.398
Average Well 4		1.72	0.417
%RPD Sample/Sample Dup Well 4		1.0	9.1
E06-0302-088	Well 5; Sample 060802	0.152	0.601
E06-0302-089	Well 5; Sample Duplicate 060802	0.166	0.562
Average Well 5		0.159	0.591
%RPD Sample/Sample Dup Well 5		8.8	3.3
E06-0302-093	DW Distribution; Sample 060802	3.10	1.30
E06-0302-094	DW Distribution; Sample Duplicate 060802	3.13	1.29
Average DW Distribution		3.11	1.30
%RPD Sample/Sample Dup DW Distribution		1.1	1.1

N/A = Not Applicable

- (1) The analytical method uncertainties are as follows: PFOA is 100% ± 17%, PFOS is 100% ± 24%. Refer to Sections 3 and 4 for data acceptance criteria and discussion.
- (2) The Relative Percent Difference between the Sample/Sample Duplicate exceeds 15%.

2. Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental Laboratory method ETS-8-154.1 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates in Water by Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry".

2.2 Sample Collection

Samples were collected in Nalgene™ (low-density polyethylene) bottles prepared at the 3M Environmental Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. Collected sample bottles were returned to the laboratory at ambient conditions on August 3, 2006.

2.3 Sample Preparation

All samples, calibration standards, and associated quality control samples were extracted using a modified procedure of ETS-8-154.1. Briefly, 40 mL of sample were loaded onto a pre-conditioned Waters C18 solid phase extraction (SPE) cartridge (Sep-Pak, 1.0 g, 6 cc) using a vacuum manifold. The loaded SPE cartridges were then eluted with 5 mL of methanol. This extraction procedure concentrates the samples by a factor of eight. (Initial volume = 40 mL, final volume = 5 mL). Lab control spikes extracted in the same manner cross-validate all the method modifications/deviations from ETS-8-154.1. See Section 3.6 for additional information.

Modifications from ETS-8-154.1 that were used for this analysis:

- Samples were not extracted in duplicate as samples were collected in duplicate in the field.
- Extraction columns were not rinsed with 40% methanol after sample loading.
- After loading the sample onto the column, and just prior to eluting the column with methanol, vacuum was applied for approximately 5 minutes to remove as much sample as possible.

2.4 Analysis

All samples and quality control samples were analyzed for PFOA and PFOS using high performance liquid chromatography/ tandem mass spectrometry (HPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Table 3. Instrument Parameters.

Instrument Name	ETSGinger
Analytical Method followed	ETS-8-154.1
Liquid Chromatograph	Agilent 1100
Guard column	Prism RP (2 mm X 50 mm), 5 μ m
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ m
Injection Volume	5 μ L
Mass Spectrometer	Applied Biosystems API 5000
Ion Source	Turbo Spray
Electrode	Z-spray
Polarity	Negative
Software	Analyst 1.4.1

Table 4. Liquid Chromatography Gradient Program.**ETS Ginger**

Step Number	Total Time (min)	Flow Rate ($\mu\text{L}/\text{min}$)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0	300	60.0	40.0
1	1.0	300	60.0	40.0
2	11.0	300	10.0	90.0
3	13.5	300	10.0	90.0
4	14.0	300	60.0	40.0
5	17.0	300	60.0	40.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Dwell Time (msec) ETS/Ginger
PFOA	413/369	150
	413/219	150
	413/169	150
PFOS	499/99	150
	499/80	150
	499/130	150

3. Data Analysis**3.1 Calibration**

Calibration standards were prepared by spiking known amounts of stock solutions containing the analytes of interest into 40 mL of ASTM type I water. Each spiked water standard was then extracted in the same manner as the collected samples. A total of twelve spiked standards ranging from 0.025 ng/mL to 25 ng/mL (nominal) were prepared. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of $100\pm 25\%$ ($100\pm 30\%$ for the lowest curve point) were met. The correlation coefficients (r) were greater than 0.998 for each analyte.

3.2 Limit of Quantitation (LOQ)

The LOQ for this analysis, as defined in method ETS-8-154.1, is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and which the area counts are at least twice those of the appropriate blanks. The nominal LOQ for PFOA, and PFOS was 0.025ng/mL.

3.3 System Suitability

The 1.0 ng/mL extracted-calibration standard was analyzed five times at the beginning and end of the analytical sequence to demonstrate overall system suitability. All compounds met the acceptance

criteria of less than 5% relative standard deviation (RSD) for peak area and less than 2% RSD for retention time for the opening and closing system suitability injections.

3.4 Continuing Calibration:

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All CCVs met method criteria.

3.5 Blanks

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank result is reviewed and used to evaluate method performance to determine the LOQ for each analyte.

The trip blank (E06-0302-098) contained trace amounts of both PFOA and PFOS. However, the amounts detected were at least a factor of 3x less than the sample with the lowest reportable value for either PFOA or PFOS. Samples have not been corrected for the trip blank concentration.

3.6 Lab Control Spikes (LCSs)

Low and high lab control spikes were prepared and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 40 mL of ASTM Type I water to produce the desired concentration. The spiked water samples were then extracted and analyzed in the same manner as the samples. Analysis of triplicate LCSs at the two specified levels cross-validates the analytical method as used here for any modifications/deviations from ETS 8-154.1. Additionally, LCS results are used to determine overall method uncertainty (see Section 3.7).

Eight LCS at three different levels were prepared with the samples. LCS-060803-1 prepared at a concentration of 0.203 ng/mL, had a recovery of 173% for PFOA. This sample results was determined to be a statistical outlier. The other two LCS prepared at the same level met method criteria and demonstrate the analysis was in control.

The following calculations were used to generate data in Table 6.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} \cdot 100\%$$

$$\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} \cdot 100\%$$

Table 6. (1) Lab Control Spike Results.

Lab ID		PFOS			PFOA		
		Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-060803-1	0.2 ppb Lab Control Spike	0.201	0.212	105%	0.203	0.352	173% ⁽²⁾
LCS-060803-2	0.2 ppb Lab Control Spike	0.201	0.204	102%	0.203	0.212	104%
LCS-060803-3	0.2 ppb Lab Control Spike	0.201	0.206	102%	0.203	0.213	105%
LCS-060803-4	2.0 ppb Lab Control Spike	2.01	2.02	101%	2.03	2.08	102%
LCS-060803-5	2.0 ppb Lab Control Spike	2.01	1.96	97.5%	2.03	2.02	100%
LCS-060803-6	2.0 ppb Lab Control Spike	2.01	1.90	94.5%	2.03	1.94	95.6%
LCS-060803-7	10 ppb Lab Control Spike	10.1	9.47	93.8%	10.2	10.1	99.0%
LCS-060803-8	10 ppb Lab Control Spike	10.1	9.88	97.8%	10.2	10.5	103%
LCS-060803-8	10 ppb Lab Control Spike	10.1	9.80	97.0%	10.2	10.5	103%
Average ± %RSD			99.0% ± 3.9%			101% ± 3.1%	

NA = Not applicable

- (1) All results and average values listed to three significant figures according to EPA rounding rules. %RSD values given to two significant figures. Due to rounding, values may vary slightly from those in the raw data.
- (2) LCS recovery did not meet method criteria of 100%±25% and was determined to be a statistical outlier.

3.7 Analytical Method Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.2. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. The expanded uncertainty is calculated by multiplying the standard deviation by factor of 2, which correspond with a confidence level of 95%.

Table 7. Analytical Method Uncertainty

Analyte	Number of data points used for determining uncertainty	Standard Deviation	Method Uncertainty
PFOA	203	8.604	100% +/- 17%
PFOS	189	11.89	100% +/- 24%

3.8 Field Matrix Spikes (FMS)

Low, mid, and high field matrix spikes were collected at each sampling point to verify that the analytical method is applicable to the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the extraction and analysis of the analytes of interest. Field matrix spikes are presented in the section 4 of this report.

$$\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration: Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} \cdot 100\%$$

Table 8. Field Matrix Spike Concentrations

Location	Description	Final Spike Concentration (ng/mL)	
		PFOA	PFOS
Well 1	Low Field Matrix Spike	0.203	0.201
	Mid Field Matrix Spike	0.508	0.503
	High Field Matrix Spike	1.02	1.01
Well 2	Low Field Matrix Spike	0.508	0.503
	Mid Field Matrix Spike	1.53	1.52
	High Field Matrix Spike	5.08	5.03
Well 3	Low Field Matrix Spike	2.48	2.46
	Mid Field Matrix Spike	5.08	5.03
	Mid-High Field Matrix Spike	10.2	10.1
	High Field Matrix Spike	15.2	15.1
Well 4	Low Field Matrix Spike	0.203	0.201
	Mid Field Matrix Spike	0.508	0.503
	High Field Matrix Spike	2.03	2.01
Well 5	Low Field Matrix Spike	0.203	0.201
	Mid Field Matrix Spike	0.508	0.503
	High Field Matrix Spike	2.03	2.01
DW Distribution	Low Field Matrix Spike	1.02	1.01
	Mid Field Matrix Spike	2.03	2.01
	High Field Matrix Spike	5.08	5.03
Trip Blank	Low Field Matrix Spike	0.203	0.201
	Mid Field Matrix Spike	2.03	2.01
	High Field Matrix Spike	10.2	10.1

4. Data Summary

This was the third sampling round for these locations. The first sampling set collected on 5/18/06 was not reported due to QC requirements. The second sampling set collected on 7/27/06 was not reported due to sampling errors. Additional information on these two sampling events can be found in the raw data file.

Tables 9 – 15 summarize the sample results and field matrix spike recoveries for the seven locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures according to EPA rounding rules. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values may vary slightly from those listed in the raw data.

Well 1

For Well 1, the RPD for both PFOA and PFOS did not meet method criteria of <15%. Additional samples from this location would need to be collected to determine if the variability is statistically relevant.

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Table 9. Well 1 Results

3M LIMS ID	Description	PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E06-0302-068	Well 1; Sample 060802	0.191	NA	0.386	NA
E06-0302-069	Well 1; Sample Duplicate 060802	0.250	NA	0.462	NA
E06-0302-070	Well 1; Field Matrix Spike Low 060802	0.475	128	0.656	NR
E06-0302-071	Well 1; Field Matrix Spike Mid 060802	0.705	95.3	0.847	84.1
E06-0302-072	Well 1; Field Matrix Spike High 060802	1.22	98.5	1.23	80.1
Average Concentration (ng/mL) ± %RPD		0.216 ng/mL ± 32%⁽¹⁾		0.424 ng/mL ± 18%⁽¹⁾	

NA = Not Applicable

NR = Not Reported = Endogenous concentration of analyte exceeded 2x the spiked level and therefore an accurate recovery cannot be calculated.

- (1) RPD exceeded method acceptance criteria of <15%. Additional samples from this location would need to be collected to determine if the variability is statistically relevant. Field matrix spike recoveries were determined using the average concentration detected in the sample and sample duplicate.

Table 10. Well 2 Results

3M LIMS ID	Description	PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E06-0302-073	Well 2; Sample 060802	0.692	NA	2.72	NA
E06-0302-074	Well 2; Sample Duplicate 060802	0.777	NA	3.10	NA
E06-0302-075	Well 2; Field Matrix Spike Low 060802	1.17	86.3	3.26	NR
E06-0302-076	Well 2; Field Matrix Spike Mid 060802	2.05	85.9	4.24	87.5
E06-0302-077	Well 2; Field Matrix Spike High 060802	5.06	85.2	6.95	80.3
Average Concentration (ng/mL) ± %RPD		0.735 ng/mL ± 11%		2.91 ng/mL ± 13%	

NA = Not Applicable

NR = Not Reported = Endogenous concentration of analyte exceeded 2x the spiked level and therefore an accurate recovery cannot be calculated.

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Table 11. Well 3 Results

3M LIMS ID	Description	PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E06-0302-078	Well 3; Sample 060802	4.59	NA	11.7	NA
E06-0302-079	Well 3; Sample Duplicate 060802	4.68	NA	11.9	NA
E06-0302-080	Well 3; Field Matrix Spike Low 060802	6.66	81.5	13.5	NR
E06-0302-081	Well 3; Field Matrix Spike Mid 060802	9.54	96.5	16.7	NR
E06-0302-082	Well 3; Field Matrix Spike Mid High 060802	NC	NA	20.9	90.1
E06-0302-102	Well 3; Field Matrix Spike High 060802	NC	NA	23.7	78.5
Average Concentration (ng/mL) ± %RPD		4.64 ng/mL ± 1.9%		11.8 ng/mL ± 1.8%	

NA = Not Applicable

NR = Not Reported = Endogenous concentration of analyte exceeded 2x the spiked level and therefore an accurate recovery cannot be calculated.

NC = Not Calculated = Sample concentration was outside of the range of calibration.

Table 12. Well 4 Results

3M LIMS ID	Description	PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E06-0302-083	Well 4; Sample 060802	0.436	NA	1.71	NA
E06-0302-084	Well 4; Sample Duplicate 060802	0.398	NA	1.73	NA
E06-0302-085	Well 4; Field Matrix Spike Low 060802	0.604	NR	1.89	NR
E06-0302-086	Well 4; Field Matrix Spike Mid 060802	0.887	92.5	2.04	NR
E06-0302-087	Well 4; Field Matrix Spike High 060802	2.18	86.8	3.35	80.8
Average Concentration (ng/mL) ± %RPD		0.417 ng/mL ± 9.1%		1.72 ng/mL ± 1.0%	

NA = Not Applicable

NR = Not Reported = Endogenous concentration of analyte exceeded 2x the spiked level and therefore an accurate recovery cannot be calculated.

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Table 13. Well 5 Results

3M LIMS ID	Description	PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E06-0302-088	Well 5; Sample 060802	0.601	NA	0.152	NA
E06-0302-089	Well 5; Sample Duplicate 060802	0.582	NA	0.166	NA
E06-0302-090	Well 5; Field Matrix Spike Low 060802	0.797	NR	0.357	98.6
E06-0302-091	Well 5; Field Matrix Spike Mid 060802	1.05	90.9	0.576	82.9
E06-0302-092	Well 5; Field Matrix Spike High 060802	2.16	77.2	1.94	88.3
Average Concentration (ng/mL) ± %RPD		0.591 ng/mL ± 3.3%		0.159 ng/mL ± 8.8%	

NA = Not Applicable

NR = Not Reported = Endogenous concentration of analyte exceeded 2x the spiked level and therefore an accurate recovery cannot be calculated.

Table 14. Drinking Water (DW) Distribution Results

3M LIMS ID	Description	PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E06-0302-093	DW Distribution; Sample 060802	1.30	NA	3.10	NA
E06-0302-094	DW Distribution; Sample Duplicate 060802	1.29	NA	3.13	NA
E06-0302-095	DW Distribution; Field Matrix Spike Low 060802	2.10	79.0	3.82	NR
E06-0302-096	DW Distribution; Field Matrix Spike Mid 060802	3.04	86.0	4.83	85.4
E06-0302-097	DW Distribution; Field Matrix Spike High 060802	5.69	86.6	7.44	86.0
Average Concentration (ng/mL) ± %RPD		1.30 ng/mL ± 1.1%		3.11 ng/mL ± 1.1%	

N/A = Not Applicable

NR = Not Reported = Endogenous concentration of analyte exceeded 2x the spiked level and therefore an accurate recovery cannot be calculated.

3M ENVIRONMENTAL LABORATORY
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3M LIMS ID	Description	PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E06-0302-098	Trip Blank 060802	0.0615	NA	0.0498	NA
E06-0302-099	Trip Blank Spike Low 060802	0.248	92.0	0.231	90.2
E06-0302-100	Trip Blank Spike Mid 060802	1.98	94.3	1.92	92.9
E06-0302-101	Trip Blank Spike High 060802	10.2	99.0	9.85	97.1

N/A = Not Applicable

(1) The trip blank amounts detected were at least a factor of 3x less than the sample with the lowest reportable value for either PFOA or PFOS. Trip blank spikes recoveries were corrected for trip blank concentration.

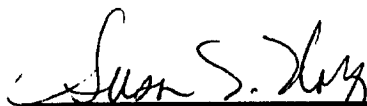
5 Conclusion

Laboratory control spikes are used to determine the analytical method accuracy and precision for both analytes and are used to determine the method uncertainty for each analyte. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix. Analysis was successfully completed following 3M Environmental Laboratory method ETS-8-154.1 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates in Water by Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry". Analytical results are reported in Table 2 of this report.

6 Data/Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Signatures


Susan T. Wolf, 3M Senior Chemist

8-16-06
Date


William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Manager

8-16-06
Date

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.


Quality Assurance Representative

08/16/06
Date