AR226-3566

3M ENVIRONMENTAL LABORATORY REPORT NO. E05-0662

Amended Final Report

Analysis of PFBS, PFHS, PFOS and PFOA in Water Samples Collected at 3M Guin

Laboratory Request Number: E05-0662

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

Testing Laboratory

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Requester

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Amended Analytical Report E05-0662

Water Samples from 3M Guin November 23, 2005

1 Entroduction/Summary

The 3M Environmental Laboratory extracted and analyzed water samples from two locations within the 3M Guin facility. Samples were collected by Weston Solutions personnel on September 15, 2005. The first sampling location was a surface water sample designated as SW in the sample description. The second location was a municipal water line located in the 3M Guin plant designated as PW. Samples were returned to the 3M Environmental Laboratory for analysis of perfluorobutane sulfonate (PFBS), perfluorobexane sulfonate (PFHS), perfluoroctane sulfonate (PFOS) and perfluoroctanoate (PFOA) and analyzed under laboratory project number E05-0662 using 3M Environmental Laboratory Method ETS 8-154.1 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates in Water by Solid Phase Extractions and High Performance Liquid Chromatography/Mass Spectrometry".

The 3M Environmental Laboratory prepared sets of sample containers for the two sampling locations. Each sample set consisted of a field sample, field sample duplicate, low field spike (0.10 ng/mL) mid field field spike (1.0 ng/mL) and high field spike (10 ng/mL). Each empty container was marked with a "fill to here" line and was fortified with a surrogate spike or an appropriate matrix spike solution containing the surrogate and the target analytes prior to being sent to the field for sample collection. Additionally, a set of trip blanks was also prepared and sent to the field with the other sample collection containers.

Samples were extracted on September 22, 2005 and analyzed on September 23, 2005.

Table 1 below summarizes the sample results. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.

Table 1. ⁽¹⁾Sample Results Summary

			Concentr	ation (ng/mL)
3M LIMS ID	Sample Description	⁽²⁾ PFBS	⁽³⁾ PFHS	⁽⁴⁾ PFOS	⁽⁵⁾ PFOA
E05-0622-88564	GAL SW PC01 0 050915 (Sample)	<0.0249	0.0395	0.121	<0.0245
E05-0622-88565	GAL SW PC01 DB 050915 (Sample Duplicate)	<0.0249	0.0351	0.0908	<0.0246
Average Concents		<0.0249 NA	0.0373 12%	0.106 28%	<0.0246 NA
E05-0622-88569	GAL PW FW01 0 050915 (Sample)	<0.0249	0.029	0.0575	<0.0246
E05-0622-88570	GAL PW FW01 DB 050915 (Sample Dupilcate)	<0.0249	0.0266	0.145	<0.0246
Average Concenti		<0.0249	0.0278	0.101	<0.0246
%RSD		NA	8.60%	87%	NA

(1) Results, averages, and %RPD values rounded to three significant figures according to EPA rounding rules. Values may vary slightly from those listed in the raw data.

- (2) The analytical uncertainty for PFBS is 100±5.4% based on method accuracy and precision. See Section 3.7 for additional explanation. The PFBS LOQ was 0.0249 ng/mL.
- (3) The analytical uncertainty for PFHS is 100±7.5% based on method accuracy and precision. See Section 3.7 for additional explanation. The PFHS LOQ was 0.0247 ng/mL.
- (4) The analytical uncertainty for PFOS is 100±12% based on method accuracy and precision. See Section 3.7 for additional explanation. The PFOS LOQ was 0.0495 ng/mL.
- (5) The analytical uncertainty for PFOA is 100±10% based on method accuracy and precision. See Section 3.7 for additional explanation. The PFOA LOQ was 0.0246 ng/mL.



2.1 Sample Collection

Samples were collected in pre-rinsed NalgeneTM (low-density polyethylene) bottles prepared at the 3M Environmental Laboratory. Prior to sample collection, all bottles were spiked in the laboratory with a known volume of either a surrogate solution or an appropriate matrix spiking solution containing the analytes of interest (PFBS, PFHS, PFOS, and PFOA) and the surrogate. The "fill to here" line was marked at 450 mL. Table 2 below details the samples collected and spikes added to each bottle.

			F	inal Spike	Concentrati	on (ng/mL)	
3M LIMS ID	Location	Description	PFOA [1,2 **C]	PFBS	PFHS	PFOS	PFOA
88564	GAL SW PC01	Sample	5.00	NA	NA	NA	NA
88565	GAL SW PC01	Sample Duplicate	5.00	NA	NA	NA	NA
88566	GAL SW PC01	Low Field Matrix Spike	0.0998	0.0998	0.0987	0.0992	0.0984
88567	GAL SW PC01	Mid Field Matrix Spike	0.998	899.0	0.987	0.992	0.984
88568	GAL SW PC01	High Field Matrix Spike	9.98	9.98	9.87	9.92	9.84
88569	GAL PW FW01	Sample	5.00	NA	NA	NA	NA
88570	GAL PW FW01	Sample Duplicate	5.00	NA	NA	NA	NA
88571	GAL PW FW01	Low Field Matrix Spike	0.0998	0.0998	0.0987	0.0992	0.0984
88572	GAL PW FW01	Mid Field Matrix Spike	0.998	0.998	0.987	0.992	0.984
88573	GAL PW FW01	High Field Matrix Spike	9.98	9.98	9.57	9,92	9.84
88574	Trip Blank	Sample	5.00	NA	NA	NA	NA
88575	Trip Blank	Low Field Matrix Spike	0.0998	0.0998	0.0987	0.0992	0.0984
88576	Trip Blank	Mid Field Matrix Spike	0.998	0.998	0.987	0.992	0.984
86577	Trip Blank	High Field Matrix Spike	9.98	9.98	9.87	9.92	9.84

Table 2. Sample Collection and Spike Information.

2.2 Extraction

All samples, calibration standards, and associated quality control samples were extracted using a modified procedure of ETS-8-154.1 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates in Water by Solid Phase Extractions and High Performance Liquid Chromatography/Mass Spectrometry". 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).

Samples were extracted on September 22, 2005 and analyzed on September 23, 2005.

2.3 Analysis

All sample and quality control extracts were analyzed for PFBS, PFHS, PFOS, PFOA, and PFOA [1,2 ¹³C] 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	ETSOlie
Liquid Chromatograph	Agilent 1100
Guard column	Betasil C18 2X100, 5 µm
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 µm
Injection Volume	5 µL
Mass Spectrometer	Applied Biosystems API 4000 Q trap
Electrode	Z-spгау
Ion Source	Turbo Spray
Polarity	Negative
Software	Analyst 1.4.1

Table 4. Liquid Chromatography Gradlent Program

Step Number	Total Time (min)	Flow Rate (µL/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0	300	80.0	20.0
1	1.0	300	80.0	20.0
2	14.5	300	10.0	90.0
3	15.5	300	10.0	90.0
4	16.5	300	80.0	20.0
5	20.0	300	80.0	20.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Dwell Time (msec)
*PFBS	299/99	100
	299/80	100
PFHS	399/130	100
	399/99	100
	399/80	100
*PFOS	499/130	100
	499/99	100
	499/80	100
PFOA	413/369	100
	413/219	100
	413/169	100
PFOA [1,2 13C]	415/370	100

"All transitions were summed to produce a "total ion chromatogram" (TIC). The TICs were used for quantitation.

3 Data Analysis

3.1 Calibration

Calibration standards were prepared by spiking known amounts of stock solutions containing the target analytes plus the surrogate 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 ten 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 was 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 extracted calibration standard used to generate the final calibration curve met the method calibration accuracy requirement of ±25%. Coefficients of determination (r²) were greater than 0.999 for all analytes.

3.2 Limit of Quantitation (LOQ)

The LOQ for this analysis, as defined in ETS-8-154.1, is the lowest non-zero calibration standard in the curve in which the area counts are twice those of the method blank(s). The LOQs for PFBS, PFHS, PFOS, PFOA, and PFOA [1,2¹³C] were 0.0249ng/mL, 0.0247 ng/mL, 0.0495, 0.0246, and 0.0249 ng/mL, respectively. Area count comparison for the method blanks and the lowest calibration standard will be provided in Section 3.5.1.

3.3 System Suitability

The 10 ng/mL extracted-calibration standard was analyzed in triplicate at the beginning and end of the analytical sequence to demonstrate overall system suitability. All analytes met method acceptance criteria of less than 5% relative standard deviation (RSD) for peak area and less than 2% RSD for retention time except for PFOA[1,2¹³C]. The initial percent RSD for PFOA[1,2¹³C] area counts was 6.7%. As PFOA[1,2¹³C] is a surrogate and not a target analyte, the data was accepted. Closing percent RSD for area counts for this analyte met method criteria (4.8%).

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 was still in control. The final CCV standard analyzed produced recoveries exceeding 125% for all analytes. This standard was disabled during initial calibration as it did not meet accuracy requirements. System suitability standards analyzed immediately after this non-compliant CCV produced recoveries within 100:±25% demonstrating that the instrument was still in control. Samples were not reanalyzed.

3.5 Blanks

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below.

3.5.1 Method Blanks

Several method blanks were prepared by loading 40 mL of ASTM Type I water onto a C18 SPE cartridge and eluting with 5 mL of methanol using the same extraction procedure as the samples. Method blanks were prepared to evaluate the levels of background contamination in the overall extraction process (reagent water, glassware, SPE cartridges, etc.) Table 6 lists the area counts for the method blanks and the LOQ standard associated with the given data set. For this data set, four of the eight method blanks were spiked PFOA [1,2¹³C] surrogate solution to achieve a final concentration of approximately 5 ng/mL. Surrogate recoveries ranged from 106-152%.

Table 6. Method Blank Area Counts

			Area	Counts	
Sample iD	PFBS	PFHS	PFOS	PFOA	PFOA(1,2 "C)
MB-050922-1	7005	@39372	37570	5949	836
MB-050922-2	5390	5721	26516	3460	521
MB-050922-3	7687	9579	48281	^Ø 13361	761
MB-050922-4	4950	4434	26430	5015	372
MB-050922-5	7968	6355	24397	4875	⁽¹⁾ 1635963
MB-050922-8	^Ø 47106	5153	26032	6388	(1)1962006
MB-050922-7	7670	6434	23076	3782	⁽¹⁾ 2241749
MB-050922-8	10151	6872	30147	2221	⁽¹⁾ 1606083
2" area counts of highest Method blank	20302	19158	96562	12776	1672
LOQ Standard Area Counts	89239	74737	214922	21924	18683
LOQ Standard Conc. (ng/ml.)	0.0249	0.0247	0.0495	0.0246	0.0249

(1) Method blank spiked with 5.0 ng/mL(nominal) PFOA [1,2 ¹³C] surrogate. Area counts not used for LOQ comparision.

(2) Determined to be a statistical outlier using Doon's Q-test. Data point excluded for LOQ comparison.

Sample ID	Theoretical Surrogate Concentration (ng/mL)	Calculated Concentration (ng/mL)	Percent Recovery
MB-050922-5	5.06	5.47	108
MB-050922-6	5.06	6.68	132
MB-050922-7	5.06	7.70	152
MB-050922-8	5.06	5.36	106
	·	Average	124
		%RSD	17.5

Table 7. ^(I)Surrogate Recoveries of Spiked Method Blanks.

(1) Table displays rounded values for all concentration and percent recovery values (3 significant figures). Values may vary slightly from the values in the raw data.

(2) The same solution used to prepare the spiked method blanks was used to spike the sample/sample duplicate collection bottles.

3.5.2 Solvent Blanks

Several methanol solvent blanks were analyzed to assess system contamination and/or instrument carryover. Analyte peak area counts in all blank samples were less than half the area counts of the calibration standard used to establish the LOQ.

3.5.3 Field/Trip Blanks

Prior to sample collection, one sample container was filled with 450 mL of ASTIM Type I water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank. The trip blank serves as an additional method blank that accounts for any storage conditions and/or holding time issues that the samples may experience. The target analytes were not detected above the stated LOQ in the trip blank.

3.6 Lab Control Spikes (LCSs)

Low (0. 25 ng/mL nominal concentration) and high (7.5 ng/mL nominal concentration) 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. Table 8 summarizes the LCS recovery results. All LCSs met method acceptance criteria of 100±25% for accuracy and <±15% relative standard deviation (RSD) for precision. The accuracy and precision of LCS data will be used to determine overall method uncertainty in Section 3.7.

Table 8. ⁽¹⁾Lab Control Spike Results.

		PFBS			PFHS	· · · ·		PFOS	
Sample ID	Spike Amount (ng/mL)	Canc. (ng/mL)	Percent Recovery	Spike Amount (ng/mL)	Conc. (ng/mL)	Percent Recovery	Spike Amount (ng/mL)	Conc. (ng/mL)	Percent Recovery
LCS-050922-1	0.249	0.242	97.2	0.247	0.235	95.1	0.248	0.224	90.3
LCS-050922-2	0.249	0.233	93.6	0.247	0.223	90.3	0.248	0.207	83.5
LCS-050922-3	0.249	0.241	96.8	0.247	0.236	95.5	0.248	0.232	93.5
LCS-050922-4	7.48	7.30	97.5	7,40	7.47	101	7.44	7.56	102
LCS-050922-5	7.48	7.28	97.3	7,40	7.10	95.9	7.44	7.54	101
LCS-050922-6	7.48	7.70	103	7.40	7.38	99.7	7.44	7.76	104
Average		97.6			96.3			95.8	
%RSD		3.10			3.93			8.43	

		PFOA			PFOA(1,2 13	<u>cı</u>
Sample ID	Spike Amount (ng/ml.)	Conc. (ng/mi_)	Percent Recovery	Spike Amount (ng/mL)	Conc. (ng/mL)	Percent Recovery
LCS-050922-1	0.246	0.266	108	0.249	0.294	118
LCS-050922-2	0.246	0.252	102	0.249	0.265	106
LCS-050922-3	0.246	0.243	98.8	0.249	0.297	119
LCS-050922-4	7.38	7.76	105	7.48	8.64	116
LCS-050922-5	7.38	7.47	101	7.46	7.47	99.9
LCS-050922-6	7,38	8.29	112	7.48	8.93	119
Average		105			113	
%RSD		4.73			7.16	

 All results, averages, and %RSD values listed to three significant figures according to EPA rounding rules. Values may vary slightly from those in the raw data.

> LCS Percent Recovery = Calculated Concentration * 100% Spike Concentration

LCS% RSD = standard deviation LCS replicates average LCS recovery * 100%

3.7 Analytical Uncertainty

Both the accuracy (percent recovery) and precision (%RSD) of the lab control spikes were used to estimate the overall method's analytical uncertainty for a given analyte. For example, the overall accuracy and precision for PFOA based on LCS results was $105\% \pm 4.73\%$. The measured precision (%RSD) is then used to determine the range of the accuracy.

Example:

105*(0.0473) = 4.966 105 + 4.966 =109.966; 105-4.966 = 100.0335

Thus, LCS accuracy results range from 100% to 110%. The absolute difference of the low and high ends of this range, when compared 100%, are then calculated.

110%-100% = 10% 100%-100% = 0%.

The most conservative (largest) absolute difference is then used as the analytical uncertainty for the given analyte. Therefore, the analytical uncertainty for PFOA is given as 100±10% for these results. The analytical uncertainty, as defined here, for PFBS, PFHS, and PFOS is 100±5.4%, 100±7.5%, and 100±12%, respectively.

3.8 Surrogates

Although not specified in the ETS 8-154.1, PFOA [1,2¹³C] was added to all samples and sample spikes as a surrogate to evaluate overall method performance. The final PFOA [1,2¹³C] concentration was 5.00 ng/mL. Surrogate recoveries are reported in the next section with sample data.

Surrogate Recovery = $\frac{\text{Calculated Sample Concentration}}{\text{Spike Concentration}} *100\%$

3.9 Field Matrix Spikes (FMS)

Low (nominal concentration of 0.1 ng/mL), mid (nominal concentration of 1 ng/mL), and high (nominal concentration 10 ng/mL) field matrix spikes were collected at each sampling point to verify that the analytical method is applicable to the collected matrix. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not interfare with the extraction and analysis of the analytes of interest. Field matrix spikes will be presented in the next section with the sample data.

FMS Recovery = <u>(Calculated Sample Concentration - Average Concentration : Field Sample & Field Sample Dup.)</u> *100% Spike Concentration

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4- Data Summary

The table below summarize the sample results, field matrix spike (FMS) recoveries, and surrogate spike recoveries for the two locations as well as the Trip Blank. Table 9 provides the average concentration and the relative percent difference (RPD) of the sample and sample duplicate. All field matrix spike recoveries were within method acceptance criteria of 100±30% except for the low level PFOS spikes (recoveries <70%). Poor reproducibility between the sample and the sample duplicate (RPD>15%) may have contributed to the low recovery. The mid and high level matrix spikes for PFOS exhibited good recovery (>86%). Therefore, the sample results for all analytes, including PFOS, are reported and considered to be accurate within 100±30%.

All samples, sample duplicates, low, mid, and high spikes exhibited sporadic PFOA [1,2¹³C] surrogate recoveries ranging from 3.83% to 93.6%. At this time, no explanation can be given for this behavior. The 3M Environmental Laboratory will be investigating this issue in depth in a future study. Because matrix spikes of the target analytes produced excellent recoveries, sample concentrations have **not** been corrected for surrogate recovery.

5 Conclusion : 1 . .

Results for the two sampling locations are presented in Table 1. Laboratory control spikes were used to determine the method accuracy and precision for the target analytes. The accuracy and precision were then used to estimate the analytical uncertainty for the results ($100\pm5.4\%$ for PFBS; $100\pm7.5\%$ for PFHS, $100\pm12\%$ for PFOS, and $100\pm10\%$ for PFOA). Field matrix spike recoveries of the target analytes demonstrated that the results for the sample matrix are accurate within $100\pm30\%$.

6 Data/Sample-Retention

All hardcopy and electronic data will be archived according to 3M Environmental Laboratory standard operating procedures. Remaining sample will be retained at the 3M Environmental Laboratory in accordance with current sample retention policies.

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Table 9. ⁽¹⁾Detailed Sample Results.

		PF	PFBS	ЪЧ	PFHS	đ	PFOS	Цd	PFOA	PFOA [Surre	PFOA [1,2 ¹³ C] Surrogate
OI SHA THAS ID	Sample Description	Conc. (ng/mL)	FMS Recovery	Conc (ng/mL)	Recovery	Conc. (ngtinL)	Recovery	Canc. (ng/ml.)	FINS Recovery	Conc. (ng/mi.)	Percent Recovery
E05-0622-88564	GAL SW PC01 0 050015	<0.0249	¥	995010	¥	0.121	¥	<0.0246	¥	3.29	66.7
E05-0622-88586	GAL SW PC01 DB 050915	<0.0249	¥	0.0351	¥	0.0908	¥	<0.0246	¥	3.39	67.8
E05-0822-88566	GAL SW PC01 LS 050915	0.096	99.8	0.128	91.9	0.171	⁽⁴ 65.6	0.101	ŝ	0.0119	11.9
E05-0822-88567	GAL SW PC01 MS 050915	0.956	96.0	0.918	89.2	0.963	86.4	0.863	89.7	0.156	15.6
E05-0622-88568	GAL SW PC01 HS 050916	9.89	99.1	9.73	98.2	9.26	92.3	10.8	110	2.85	28.6
Average Concentration	ation	<0.0249		0.0373		0.106		<0.0246		¥	
%RPD		Ą		12		28		M		W	
E05-0622-88589	GAL PW FW01 0 050915	<0.0249	¥	0.029	¥	0.0575	AN	<0.0246	Ą	1.75	36.0
E05-0622-88570	GAL PW FW01 DB 050915	<0.0248	¥	0.0266	¥	0.145	¥	≤0.0246	¥	2.23	44.6
E05-0622-88571	GAL PW FW01 LS 060915	0.101	ĪĐ	0.118	814	0.159	⁽³⁾ 58.5	0.0961	£78	0.00608	6.09
E05-0622-88572	GAL PW FW01 MS 050815	0.875	87.7	0.864	84.7	1.05	95.7	0.933	8,48	0.138	13.8
E05-0622-88573	GAL PWFW01 HS 050915	844	94.6	9.12	92.1	8.17	91.4	9.1	92.5	1.96	19.6
Average Concentration	ation	<0.0249		0.0278		0.101		<0.0246	-	NA	
%RPD		¥		8.6		87		NA		W	
E05-0622-88574	GAL OA TRIP01 0 050915	Ŷ	¥	<0.0247	Ą	<0.0495	A	<0.0246	¥	4.68	93.6
E05-0622-88575	GAL QA TRIP01 0 050915	0.0933	93.5	0.0884	89.6	0.0753	76.0	0.109	ŧ	0.00382	3.83
E05-0622-88576	GAL QA TRIP01 0 050915	0.945	84.7	0.904	91.6	0.934	94.2	1.01	103	0.142	14.2
E05-0622-88577	E05-0622-88577 GAL QA TRIPD1 0 050915 9.91 99.3 9.29 94.1 9.48 95.6 9.51 96.8	3 .81	6.66	9.29	94.1	9.48	95.6	9.51	96.8	6.61	66.2
(1) Table dis	Table displays rounded values for all concentration and percent recovery values (3 significant figures Remnery of field matrix reflex expression matrix of criteria of f00+104. Externational reflexed matrix	centration and mathod criterie	percent recov	ery values (3 /	significant figun	es). Values m	ay vary slightly	r from the value	es in the raw data.		

Recovery of field matrix spike exceeded method of the anticipation of 100±30%. FMS recovery calculated using just the sample duplicate = 80.8%. The large %RPD (>15%) may contribute to the few spike recovery. The field matrix spike exceeded method of the field of 100±30%. FMS recovery calculated using just the sample value = 103%. The large %RPD (>15%) may contribute to the low spike recovery. ନ୍ତି C

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7 Signatur . T RESIL 2005 231 1 Date Michelle D. Malinsky, Ph.D., 3M Technical Lead, 11-23-2005 Date 11/23/2005 QAU Representative Date L. Bacon, 3M Environmental Laboratory Technical Director



8.1 Report Amendment Declaration

ENNIPONMENTAL I ADODATOD **REPORT NO. E05-0662**

3M ENVIRONMENTAL LABORATORY

Final Project Report Amendment

Project number: E05-0662

Project title: Water Samples from 3M Guin

Laboratory Project Lead: Michelle Malineky

Amendment date: November 23, 2005

Amendment number: 1

This amendment modifies the following portion of the final report:

Title on the cover page and page 2.

Section 1. Introduction/Summary changed to clarify that one sample was from a Municipal water line located in the 3M Guin Plant.

Added this amendment as an attachment, increasing the total pages to 13

Reason for the amendment:

To clarify that one sample was from a Municipal water line located in the 3M Guin Plant, instead of normal surface water.

Approved by:

lle Malinsky,

Dale-Bacon, Laboratory Management

11/23/2005 Date 11/23/2005

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Aerial Photograph of the Guin, AL facility showing the location of the two samples collected on 15 Sep 05.