3M Environmental Laboratory

Results from Analyses of Soil and Groundwater Samples From the E. I. DuPont de Nemours and Company Facility in Parkersburg, West Virginia

STUDY COMPLETED: October 27, 1997

FINAL REPORT COMPLETED: November 13, 1997

Report Susan A. Beach

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3MA00230129

Corrigan
EXHIBIT NO. 10
DATE 2-27-08
Kelley, York & Associates

Exhibit
2759
State of Minnesota v. 3M Co.,
Court File No. 27-CV-10-28862

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Summary of Results
DuPont Washington Works Samples
Sample Dates 5/8/97 (groundwater), 5/30/97 (soil)

3M LR No.	DuPont Smpl. No.	Matrix	POAA[1], ppb	Vol. & Semi Vol. F, Cl, Br by AED, ppm
R2008-1	1	ground water	52	< MQL[2]
R2008-2	2	ground water	49	< MQL
R2008-3	3	ground water	52	< MQL
R2008-4	4	ground water	53	< MQL
R2008-5	5	DI blank	n.d.[2]	< MQL
R2008-6	none	soil	364	< MQL

(1) POAA = Perfluorooctanoic acid anion

(2) limit of detection/limit of quantitation = 650 ppt.

(3) Minimum quantitation limits:
Br = 1.176 ppm in water, 1.187, 1.220 ppm in soil
Cl = 1.408 ppm in water, 1.253, 1.461 ppm in soil
F = 0.280 ppm in water, 0.282 ppm in soil

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3M Environmental Laboratory

Summary of Results
DuPont Washington Works Groundwater Samples
Sample Date 6/26/97

3M LR No.	DuPont Smpl. No.	POAA[1] mg/L	Total Fluorine mg/L	Fluoride Ion mg/L	Organic F, mg/L[4]	Adsorb. Organic F, mg/L	Vol. & Semi-Vol. F by AED, ppm	Trichlorotri-fluoroethane µg/L	Acetone µg/L
R2148-1	MW-1-1	5.64	8.0	0.20	7.8	4.5	0.20 < [F] < 2.5	820	10
R2148-2	MW-1-2	5.32	16	0.20	16	4.2	0.20 < [F] < 2.5	730	10
R2148-3	MW-2-1	0.234	3.3	0.16	3.1	0.28	0.20 < [F] < 2.5	1600	--
R2148-4	MW-2-2	0.234	3.5	0.16	3.3	0.46	0.20 < [F] < 2.5	1500	--
R2148-5	MW-3-1	0.487	4.2	0.14	4.1	1.1	0.20 < [F] < 2.5	2300	--
R2148-6	MW-3-2	0.477	3.3	0.14	3.2	0.76	0.20 < [F] < 2.5	2400	--
R2148-7	MW-4-1	0.0842	4.0	0.11	3.9	0.19	0.20 < [F] < 2.5	760	10
R2148-8	MW-4-2	0.0590	4.2	0.11	4.1	0.14	0.20 < [F] < 2.5	670	--
R2148-9	MW-5-1	< PQL[2]	3.0	<0.10	2.9	0.11	[F] < 0.05	130	--
R2148-10	MW-5-2	< PQL[2]	2.8	<0.10	2.7	0.14	0.05 < [F] < 0.20	140	--
R2148-11	MW-6-1	< MDL[3]	2.3	0.10	2.2	<0.05	[F] < 0.05	--	--
R2148-12	MW-6-2	< MDL[3]	4.2	0.10	4.1	<0.05	[F] < 0.05	--	--

3M LR No.	DuPont Smpl. No.	tert-Butyl Alcohol µg/L	cis-1,2-Dichloroethene µg/L	Chloroform µg/L	1,1,1-trichloro-ethane µg/L	Trichloro-ethene µg/L	Tetrachloro-ethene µg/L	Trichlorofluoro-methane µg/L
R2148-1	MW-1-1	59	--	--	--	--	--	--
R2148-2	MW-1-2	49	--	--	--	140	--	16
R2148-3	MW-2-1	--	--	--	--	150	--	18
R2148-4	MW-2-2	--	--	5.5	14	520	5	26
R2148-5	MW-3-1	--	--	5.9	14	570	5.2	26
R2148-6	MW-3-2	--	5.8	37	6.3	81	--	5.9
R2148-7	MW-4-1	--	5.9	39	6.2	66	--	--
R2148-8	MW-4-2	--	--	13	--	--	--	--
R2148-9	MW-5-1	--	--	16	5.8	--	--	--
R2148-10	MW-5-2	--	--	--	--	--	--	--
R2148-11	MW-6-1	--	--	--	--	--	--	--
R2148-12	MW-6-2	--	--	--	--	--	--	--

(1) POAA = Perfluorooctanoic acid anion (2) Practical quant. limit = 0.0510 mg/L (3) Limit of detect. = 0.0255 mg/L (4) Total Fluorine - Fluoride Ion = Organic Fluorine

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3M Environmental Laboratory

Summary of Results
DuPont Washington Works Soil Samples
Sample Date 6/23/97

3M LR No.	DuPont Sample No.	*Total Fluoride, mg/kg	POAA[1], mg/kg	Sulfate, mg/kg	Sulfite, mg/kg	Nitrite, mg/kg	pH in water	pH in $CaCl_2$	CEC, meq/100 g	Moisture, %
R2382-1	SS-1 0-2'	21,300	0.119	98	<2	0.41	7.7	7.2	15.8	12.3
R2382-2	SS-1 4-6'	20,100	0.17	99	<2	0.41	7.7	7.3	18.4	12.7
R2382-3	SS-1 8-10'	61,200	614	73	<2	0.36	7.3	7.0	17.5	15.5
R2382-4	SS-1 12-14'	78,300	207	54	<2	0.14	6.7	6.3	17.5	18.9
R2382-5	SS-1 16-18'	106,300	219	43	<2	<0.10	5.7	5.3	18.4	18.3
R2382-6	SS-1 20-22'	82,700	39.8	70	<2	<0.10	6.0	5.5	19.3	19.2
R2382-7	SS-1 24-26'	59,100	24.6	220	<2	<0.10	5.8	5.3	17.5	20.0
R2382-8	SS-1 28-30'	37,600	29.3	150	<2	0.11	6.8	6.3	11.4	18.1
R2382-9	SS-1 32-34'	33,500	13.1	100	<2	<0.10	5.8	5.2	13.1	13.6
R2382-10	SS-1 36-68'	41,200	6.78	63	<2	<0.10	6.1	5.4	9.6	17.9
R2382-11	SS-1 38-40'	30,300	2.11	46	<2	<0.10	6.8	6.2	6.3	22.2

(1) POAA = Perfluorooctanoic acid anion

* See revised values Fax 3/13/97 KSH

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Project Description

Three coolers were received from E.I. DuPont de Nemours and Company ("DuPont"), each containing samples for analyses by the 3M Environmental Laboratory. Each cooler-group was assigned an unique project number (Lab Request Number). Each sample was also given an unique number which was a sub-set of the project number. The project numbers are as follows:

R2008, samples 1-6
Four groundwaters, one DI water blank and one soil sample, with sample dates 5/8/97 (waters) and 5/30/97 (soil).

R2148, samples 1-12
Twelve groundwater samples, with a sample date of 6/26/97

R2382, samples 1-11
Eleven soil samples, with a sample date of 6/23/97

Samples were stored at 4°C, in the dark, until analyzed.

Different groups within the 3M Environmental laboratory were responsible for various analyses. Attached are the summary reports for analyses of: POAA, Total, free, organic and adsorbable fluoride, volatiles and semi-volatiles, nitrate, sulfate, and sulfide, and soil pH, percent moisture and cation exchange capacity.

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3M Environmental Laboratory

POAA Analyses in Groundwater and Soil (5/97 Samples)

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3M Environmental Laboratory

Data Transmittal Summary

Final

Lab Request #:	3M Study #:	Contract Laboratory #
R2008		

Date Received:

Sponsor or Client: Representative Name
Company Name DuPont
Company Address
Phone

Project Lead: Kris Hansen (8-6018)
Group Leader: Jim Johnson (8-5294)

Analyte(s) or Test Method #: POAA
Sample Matrix: water and soil
Analysis Dates: 9/97-11/97 Analyst(s): GML, JJ, kjh
Author: kjh

Data Reviewed by:

Project Lead (or designee):
James D. Johnson (or designee):

Internal	Sent by: / Date
JDJ:	kjh on 10/21/97
QAU (Archives):	
LIRN System:	
Project Manager: Sue Beach	

Others *(List Recipients / Address / Phone / FAX)*	Sent by: / Date
T. DiPasquale, 22-11E-03; 3-1891; 736-3257	kjh on 11/10/97

A copy of the report including this form and the client cover page is to be given to QAU, LIRN and to the Group Leader.

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3M Environmental Laboratory- Advanced Method Development Team
Kris Hansen - Sr. Analytical Chemist
Advanced Method Development Team
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Report - Analytical Study
Determination of POAA in Soil and Water

1.0 Summary

One soil sample and five water samples were submitted by DuPont for quantitative analysis of perfluorooctanoic acid anion (POAA). The soil sample was assigned number R2008-6; the water samples were given numbers R2008-1 through R2008-5. Analysis of the samples by negative ion electrospray mass spectrometry (ES/MS) determined that perfluorooctanoate acid anion is present in all samples except R2008-5. Specific results are listed in Table 1.

Table 1. Concentration of POAA in R2008 samples.

Sample #	Matrix	Dilution Factor	Extractability	Corrected concentration (ppb)	Average (ppb)	Std. Dev.
R2008-6	soil	n.a.	n.a.	0.364 mg/kg	n.a.	n.a.
R2008-1-1	water	2	1.3	52	52	0
R2008-1-2	water	2	1.3	52		
R2008-2-1	water	2	1.3	49	49	0
R2008-2-2	water	2	1.3	49		
R2008-3-1	water	2	1.3	52	52	0
R2008-3-2	water	2	1.3	52		
R2008-4-1	water	2	1.3	57	53	6
R2008-4-2	water	2	1.3	49		
R2008-5-1	water	2	1.3	n.d.*	n.a.	n.a.
R2008-5-2	water	2	1.3	n.d.*		

* limit of detection/limit of quantitation is 650 ppt.

2.0 TEST MATERIALS

One soil and five water samples were received from DuPont on 06/10/97. The samples were stored at 4°C until extraction; extracts were stored at 4°C until analysis.

3.0 EXPERIMENTAL-OVERVIEW AND METHODS

3.1.1 Sample, soil

Because no uncontaminated soil was available for blank analysis, the method of standard addition was used to determine the concentration of POAA in the soil received from DuPont. An eight point standard curve was prepared by spiking 2 gram samples of the soil with some amount of POAA solution between 500 ppt and 1.0 ppm. The soil was mixed with approximately 1 gram of diatomaceous earth and loaded into a 10 mL stainless steel extraction cartridge. The spiked samples were extracted using high pressure solvent extraction (HPSE) with methanol; the extracts were dried with nitrogen and reconstituted with ACN/water (1:1). After analysis by negative ion ES/MS, the data was subjected to linear regression and the resulting prediction equation was used to determine the concentration of analyte

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in the unspiked material (see Figure 1, attached). Four unspiked soil samples were also extracted to confirm that the method reproducibility was better than 10%.

The method of standard addition assumes there are no interferences in the analysis and that the extraction efficiency of the analyte from the matrix is not dependent upon analyte concentration. The first assumption is addressed by the selectivity of the both the extraction and the analysis; the latter has been verified in another study that focuses on a similar matrix.

3.1.2 Sample, water

For method development, two series of samples were prepared for analysis by ES/MS. In series A, the target analyte was extracted from the samples with an ion pairing reagent and analyzed; in Series B, each sample was diluted (1:1) with acetonitrile (ACN). Both Series A and B consisted of 2 aliquots of water from two of the submitted samples (R2008-1 and R2008-3). The recovery of POAA resulting from Series A and Series B analysis were in close agreement.

All five water samples were prepared, in duplicate, for analysis using the Series B protocol. The samples were analyzed by ES/MS between two unextracted curves of POAA in ACN/H2O. The reproducibility of the curves was within 15%.

3.2 Calibration and controls, water

A set of controls, including a milli-Q water blank, milli-Q water spiked with POAA, and four samples of matrix spiked with POAA, was prepared along with each sample series. The controls were used to evaluate extraction efficiency of the POAA from water and subsequently determine an accurate extractability factor for final concentration calculations. A POAA standard curve from 50 ppt to 1.0 ppm in ACN/H2O (1:1) was prepared; all extracts and prepared samples were analyzed by negative ion ES/MS and quantitated relative to a standard curve. The unextracted standard curve was plotted according to linear regression with a coefficient of determination (r^2) equal to 0.999.

Two-1 mL aliquots of sample R2008-1 were spiked with POAA. These samples were designated the matrix spike (MS) and the matrix spike duplicate (MSD) and were prepared for analysis by the same procedure as the samples. The final concentration of POAA in the MS and MSD was expected to be 52 ppb. The concentration of POAA recovered from the samples was evaluated relative to the standard curve.

3.3 Extraction specifics, soil

The soil samples were extracted with the ISCO 3560 Accelerated Extraction System, with ISCO 100DX high pressure syringe pumps according to the following conditions:

Extraction solvent:	methanol, HPLC grade
Extraction pressure:	2500 psi
Extraction temperature:	70° C
Restrictor temperature:	70° C
Static extraction time-1:	40 minutes
Dynamic extraction volume-1:	15 mL
Static extraction time-2:	2 minutes
Dynamic extraction time-2:	2 minutes
Restrictor flow rate:	2.5 mL/min

Samples were reconstituted in glass autovials with HPLC-grade ACN and milli-Q water.

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3.4 ES/MS Analysis specifics, soil and water

Negative ion ES/MS analysis was performed on a Micromass Platform II atmospheric pressure ionization mass spectrometer running Mass Lynx 2.1. A Hewlett Packard 1100 was used for the autosampler and HPLC system.

Mobile phase: ACN/H2O (1:1)
Flow rate: 60 µL/min
Injection volume: 15µL
Cone Voltage = -20
Capillary voltage = -2.56
Source Temperature = 80°C
Analyzer Vacuum Pressure = 0.000079 mBar

Quantitative results were based on the instrumental response generated by monitoring a single ion characteristic of the analyte. This type of monitoring minimizes interference by other ions in solution and increases system sensitivity to the target analyte.

4.0 DATA ANALYSIS

4.1 Sample, soil

By the method of standard addition, the soil was determined to contain 0.364 mg POAA/kg. This value was calculated using the prediction equation resulting from linear regression analysis of the eight point extracted curve. The coefficient of determination for the curve is 0.990. Calculations used to determine the concentration of POAA in the soil are shown in Appendix A.

4.2 Samples, water

The concentration of POAA in each water sample was determined by comparison of detected peak areas resulting from analysis of the samples to the average of the two unextracted calibration curves using the following formula:

$$C_s = (P - I) / S \qquad (4)$$

Where,

C_s = Concentration of POAA in sample (µg/mL)
P = Peak area of sample (response)
I = Intercept of the calibration curve (response)
S = Slope of the calibration curve (response / concentration)

The concentration determined to be in the extract was converted to the concentration in the water samples according to the following equation:

$$C_p = (C_s * D)*E \qquad (5)$$

Where,

C_p = Concentration of POAA in actual sample (µg/mL)
C_s = Concentration of POAA in prepared sample (µg/mL)
D = Dilution Factor
E = Extractability

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2759.0011

4.3 Determination of percent recovery and extractability, water

Matrix spike samples were analyzed to determine the recovery of POAA from the water. POAA recovery and the related extractability value are calculated as follows:

$$\%R = [(C_f - C_n) / C_s]*100\% \quad (6)$$

where,

%R = Percent recovery of POAA
C_f = Concentration of POAA found in MS/MSD (µg/mL)
C_n = Native concentration of POAA in sample before dilution adjustment (µg/mL)
C_s = Concentration of POAA spiked in MS sample (µg/mL).

As an example, the percent recovery for the R2008-1-1, MS sample is calculated as follows:

C_f = 0.057 µg/mL;
C_n = 0.020 µg/mL;
C_s = 0.052 µg/mL;

therefore,

$$\%R = (0.057-0.020)/0.052 \times 100\% = 74\%.$$

The extractability is equal to 100% divided by %R.

The percent recoveries of POAA in the MS and MSD samples and the corresponding extractability factors are presented in Table 2.

Table 2 % recovery and extractability determined from analysis of MS and MSD.

Sample	Recovered Concentration (µg/mL)	Spiked Concentration (µg/mL)	Native Concentration (µg/mL)	% Recovery	Extractability
MS	0.057	0.052	0.02	74	1.3
MSD	0.059	0.052	0.02	75	1.3
			Average	75	1.3

5.0 CONCLUSION

High pressure solvent extraction, ES/MS analysis and linear regression analysis were used to determine that 0.364 mg/kg of POAA is present in the soil sample received from DuPont. Water samples R2008-1 through R2008-4 also contain about 50 ppb POAA. No POAA was detected in R2008-5.

6.0 MAINTENANCE OF RAW DATA AND RECORDS

Hard copies of these data are filed in the AMDT archive.

Sample preparation: GML/JJ
Analysis: GML/kjh
Report preparation: kjh

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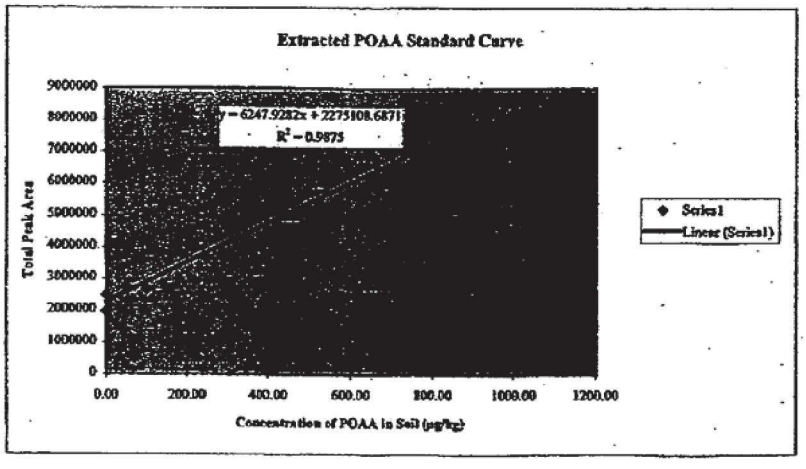
R-2008 DuPont Soil Data
POAA Standard Curve and Data Table

DATA TABLE 1.0

Spiked Standard Conc. of POAA in Extract (µg/mL)	Volume of Extract (mL)	Spiked Amount of of POAA in Extract (µg)	Mass of Soil (g)	Conc. of POAA in Soil (µg/kg)
Blank	2.0	0.00	2.0259	0.00
0.0005	2.0	0.001	2.0032	0.50
0.005	2.0	0.01	2.0004	5.00
0.050	2.0	0.10	2.0319	49.22
0.100	2.0	0.20	2.0075	99.63
0.250	2.0	0.50	2.0083	248.97
0.500	2.0	1.00	2.0248	493.88
0.750	2.0	1.50	2.0043	748.39
1.000	2.0	2.00	2.0097	995.17

Conc. of POAA in Soil (ng/kg)	Peak Area of Extract-a	Peak Area of Extract-b	Total Peak Area (a +b)
0.00	1822272	141494	1963766
0.50	2076435	396700	2473135
5.00	1967476	475027	2442503
49.22	2129047	623624	2752671
99.63	2457197	225468	2682665
248.97	3249144	395568	3644712
493.88	5136166	666830	5802996
748.39	5879632	786883	6666515
995.17	7704024	842187	8546211

FIGURE 1.0



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R-2008 DuPont Soil Data
POAA Standard Curve and Data Table

CALCULATIONS

In order to plot the Total Peak Area versus the Concentration of POAA in Soil, the following conversion calculations were performed:

Conc. of POAA, from spiked standards, in the extract	X	Volume of Extract	X	Mass of Soil	X	Convert g to kg	=	Conc. of POAA in Soil
$\frac{\mu g}{mL}$	X	$\frac{mL}{1}$	X	$\frac{1}{g}$	X	$\frac{1000\ g}{kg}$	=	$\frac{\mu g}{kg}$

To calculate total Peak Area, the area integrated for peak "a" for the initial extraction of the soil and the area integrated for peak "b" for the second extraction of the same soil are summed.

Using the Method of Standard Addition determine indigent analyte concentration by solving for for the x-intercept:
where y = 0, and equation of the slope of the line of Peak Area vs. Conc. of POAA in Soil is:

$$y = 6248\,x + 2275109$$

$$x = -2275109 / 6248$$

$$x = -364$$

concentration of indigent analyte POAA in soil determined to be 364 µg/kg

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POAA Analyses in Groundwater (6/97 Samples)

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3M Environmental Laboratory

Data Transmittal Summary
Final

Lab Request #:	3M Study #:	Contract Laboratory #
R2148		

Date Received:

Sponsor or Client: Representative Name
Company Name DuPont
Company Address
Phone

Project Lead: Kris Hansen (8-6018)
Group Leader: Jim Johnson (8-5294)

Analyte(s) or Test Method #: POAA
Sample Matrix: water
Analysis Dates: 9/97-11/97 Analyst(s): LAC
Author: LAC, kjh
Data Reviewed by: PAR
Project Lead (or designee):kjh
James D. Johnson (or designee):

Internal	Sent by: / Date
JDJ:	
QAU (Archives):	
LIRN System:	
Project Manager: Sue Beach	

Others *(List Recipients / Address / Phone / FAX)*	Sent by: / Date
T. DiPasquale, 22-11E-03; 3-1891; 736-3257	kjh on 11/10/97

A copy of the report including this form and the client cover page is to be given to QAU, LIRN and to the Group Leader.

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3M Environmental Laboratory - Advanced Method Development Team

Contact: Kris Hansen - Senior Analytical Chemist
Building 2-3E-09
778–6018

Final Report - Lab Request R2148
Electrospray Mass Spectrometry Analysis
of DuPont Water Samples Prepared 8/26/97

1.0 SUMMARY

Twelve samples from DuPont were submitted to the 3M Environmental Laboratory for the analysis of Surfactants. These samples were numbered R2148-1 through R2148-12 (MW-1 through MW-6) and analyzed with an Electrospray Mass Spectrometer. This analysis determined that perfluorooctanoic acid anion is present in samples 1 through 10. Results are listed in table 1.

Table 1 Sample Results

Sample #	Ion Count Area	Dilution Factor	Concentration µg/mL (ppm)	Average	Std. Dev.
R2148-1-1 (MW-1)	160473	20	5.71		
R2148-1-2 (MW-1)	157432	20	5.58	5.64	0.0872
R2148-2-1 (MW-1)	148341	20	5.21		
R2148-2-2 (MW-1)	153530	20	5.42	5.32	0.149
R2148-3-1 (MW-2)	79430	2	0.242		
R2148-3-2 (MW-2)	75610	2	0.226	0.234	0.0110
R2148-4-1 (MW-2)	80126	2	0.245		
R2148-4-2 (MW-2)	74915	2	0.224	0.234	0.0149
R2148-5-1 (MW-3)	135558	2	0.469		
R2148-5-2 (MW-3)	143990	2	0.504	0.487	0.0242
R2148-6-1 (MW-3)	135283	2	0.468		
R2148-6-2 (MW-3)	139707	2	0.486	0.477	0.0127
R2148-7-1 (MW-4)	43324	2	0.0954		
R2148-7-2 (MW-4)	37814	2	0.0731	0.0842	0.0158
R2148-8-1 (MW-4)	35756	2	0.0647		
R2148-8-2 (MW-4)	32916	2	0.0532	0.0590	0.00814
R2148-9-1 (MW-5)	24116	2	<PQL		
R2148-9-2 (MW-5)	23264	2	<PQL	<PQL	<PQL
R2148-10-1 (MW-5)	21873	2	<PQL		
R2148-10-2 (MW-5)	26025	2	<PQL	<PQL	<PQL
R2148-11-1 (MW-6)	11696	2	<MDL		
R2148-11-2 (MW-6)	11932	2	<MDL	<MDL	<MDL
R2148-12-1 (MW-6)	9132	2	<MDL		
R2148-12-2 (MW-6)	10595	2	<MDL	<MDL	<MDL

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2.0 TEST MATERIALS

Twelve water samples were received from DuPont on 07/02/97 (MW-1-1, MW-1-2, MW-2-1, MW-2-2, MW-3-1, MW-3-2, MW-4-1, MW-4-2, MW-5-1, MW-5-2, MW-6-1, and MW-6-2). These samples were checked-in as R2148-1 through 12 and were analyzed for surfactants. The samples were stored at 4°C until preparation and analysis.

3.0 EXPERIMENTAL-OVERVIEW AND METHODS

3.1 Investigative Samples

One half mL was removed from each sample and diluted with 0.5 mL of acetonitrile (ACN, TN-A-1504) for a final sample solvent composition of 1:1 ACN:Water. These samples were vortex mixed and ready for analysis by electrospray mass spectrometry (ES/MS).

3.2 Matrix Spike Samples

Matrix spike (MS) and matrix spike duplicate (MSD) samples were each prepared diluting 0.5 mL from sample R2148-12-1 with 0.5 mL of ACN. The MS and MSD samples were each spiked with 0.005 mL of a 101.1 µg/mL (ppm) ammonium perfluorooctanoate standard solution (W397-741) for final concentrations of 0.503µg/mL.

3.3 Calibration

Ammonium Perfluorooctanoate calibration standards, ranging in concentration from 0.0500-1.01 µg/mL, were analyzed bracketing the samples. The calibration curve was developed by plotting the mean of two standard peak areas of ammonium perfluorooctanoate versus the concentration of ammonium perfluorooctanoate standards using linear regression.

3.4 Instrumentation

The following instrumental conditions were used to analyze these samples:

Micromass Platform Electrospray Mass Spectrometer
Hewlett Packard 1100 Pump and Autosampler
MassLynx 2.1 software
Cone Voltage = -14
Skimmer Lens Offset = 3
Source Temperature = 80°C
Analyzer Vacuum Pressure = 0.000079 mBar
Injection/sample: 1
Injection size: 10 µL
Flow Rate: 0.080 mL/min

3.5 Continuing Calibration Standards

Continuing calibration standards at 0.253 ppm ammonium perfluoroctanoate were analyzed bracketing every ten samples during sample analysis.

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3.6 Detection Limits

The method detection limit (MDL) is equal to approximately 3 times the baseline noise and half the practical quantitation limit (PQL). The PQL corresponds to the lowest point on the calibration curve. The PQL is 0.0510 µg/mL; the method detection limit is 0.0255 µg/mL.

4.0 DATA ANALYSIS

4.1 Calibration Curve

Average peak areas from the initial curve were plotted against the concentration of ammonium perfluorooctanoate in the calibration standards. The standard curve was linear ($R^2 \geq 0.99$).

4.2 Continuing Calibration Standard

Continuing calibration standards were analyzed before and after every 10 samples. The continuing calibration standards remained within 20% of the initial standard. This meets the criteria used to determine if the calibration curve has maintained linearity. The relative percent difference is calculated using the following equation:

Equation 1

$$\%D = \frac{R_i - R_c}{R_i} \times 100\%$$

where,

%D = relative percent difference
R_i = area 0.253 ppm calibration standard from the initial calibration
R_c = area 0.253 ppm calibration standard from the continuing calibration

4.3 Investigative Samples

4.3.1 Calculations

Concentrations of ammonium perfluorooctanoate were determined by comparison of detected peak areas to the calibration curve using the following formula:

Equation 2

$$Ce = \frac{P - I}{S}$$

Where,

C_e = Concentration of ammonium perfluorooctanoate in extract (µg/mL)
P = Peak area of sample
I = Intercept of the calibration curve

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S = Slope of the calibration curve (mL/µg)

The concentration of ammonium perfluorooctanoate in the extract was converted to the concentration in the water samples by using the following equation:

Equation 3

$$Cp = Ce \times D$$

Where,

C_p = Concentration of ammonium perfluorooctanoate in water sample (µg/mL)
C_e = Concentration of ammonium perfluorooctanoate in extract (µg/mL)
D = Dilution Factor

As an example, ammonium perfluorooctanoate anion was detected in sample R2148-1, where P = 160473, S = 493135 mL/µg, I = 19797; therefore, using Equation (2), C_e = ((160473-19797)/493135) = 0.285 µg/mL. To determine the concentration in water, using Equation (3), C_e = 0.285 µg/mL and D = 20; thus C_f = 0.285 µg/mL x 20 = 5.71 µg/mL (ppm).

4.4 Matrix Spike Samples

Matrix spike samples were analyzed to determine the recovery of ammonium perfluorooctanoate. Recovery was calculated using the following equation:

Equation 4

$$\%R = \frac{C_m - C_b}{C_{mc}} \times 100\%$$

where,

%R = Percent recovery of ammonium perfluorooctanoate
C_m = Detected concentration of ammonium perfluorooctanoate in MS sample (µg/mL)
C_b = Average background concentration of ammonium perfluorooctanoate in sample (µg/mL)
C_{mc} = Expected concentration of ammonium perfluorooctanoate in MS sample (µg/mL).

As an example, the percent recovery for the R2148-12-1, MS sample is calculated as follows:

C_m = 0.404 µg/mL;
C_b = 0.00 µg/mL;
C_{mc} = 0.503 µg/mL;
therefore, %R = (0.404-0.00)/0.503 x 100% = 80%.

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The percent recoveries of ammonium perfluorooctanoate in the MS and MSD samples are presented in Table 2.

Table 2
Matrix Spike Results

Sample Type	Sample ID	Recovered Concentration (µg/mL)	Expected Concentration[1] (µg/mL)	% Recovery
DuPont Water	R2148-12-1, MS	0.404	0.503	80
	R2148-12-1, MSD	0.401	0.503	80
			Average	80

Notes:
[1] Recovered concentration is equal to the concentration detected in the spiked sample minus the average concentration detected in associated unspiked samples.

5.0 CONCLUSION

The results of ES/MS analysis determined that the ammonium perfluorooctanoate anion is present in DuPont water samples R2148-1 through R2148-10 at average concentrations of 5.64 ppm, 5.32 ppm, 0.234 ppm, 0.234 ppm, 0.487 ppm, 0.477 ppm, 0.0842 ppm, and 0.0590 ppm respectively. The results have been presented in table 1.

6.0 MAINTENANCE OF RAW DATA AND RECORDS

Hard copies of these data are filed in the AMDT archive.

7.0 APPENDICES

The appendices are not included with these data. They are filed in the AMDT archive.

7.1 Extraction Logbook
7.2 Instrument Runlog
7.3 Curve and Chromatograms
7.4 Results

LAC 8/29/97

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3M Environmental Laboratory

POAA Analyses in Soil (6/97 Samples)

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THE VIDEOGRAPHER: We are on the record. Here begins tape number one in the deposition of Cheryl Corrigan in the matter of Gary Paulson, Karen Paulson, William Henry, Bradley Krank, plaintiffs versus 3M Company, case number C2-04-6309. We are on the record, and today's date is February 27, 2008. The time is 9:04 a.m. The deposition is taking place at Foulston Siefkin, 1551 North Waterfront Parkway, Suite 100, Wichita, Kansas 67206.

Will counsel please identify yourselves and state who you represent, and the witness will be sworn.

MR. BILOTT: Rob Bilott for plaintiffs. And just a correction, the defendant is 3M Company.

MR. ASHLEY: Cooper Ashley for 3M Company.

MR. ARMSTRONG: Jim Armstrong for the witness.

SHERYL CORRIGAN,

having been first duly sworn on her oath to state the truth, the whole truth, and nothing but the truth, testifies as follows:

BY MR. BILOTT:

Q. Good morning.

A. God morning.

Q. My name is Rob Bilott. As you just heard, I'm one of the attorneys representing the plaintiffs in the case just described, which is Gary A. Paulson, et al. versus the 3M Company, a case currently pending in state court in Minnesota.

Would you state your name, please.

A. Sheryl Ann Corrigan.

Q. And are you currently employed?

A. Yes, I am.

Q. And who are you employed with?

A. Koch Industries.

Q. And is that K-O-C-H?

A. Yes, it is.

Q. Is that located here -- the place of your employment located here in Wichita, Kansas?

A. That's correct.

Q. What is your business address?

A. I'll get my business card out.

Q. Okay.

A. 4111 East 37th Street North, Wichita, Kansas 67220.

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3M Environmental Laboratory

Data Transmittal Summary

Final

Lab Request #:	3M Study #:	Contract Laboratory #
R2382		

Date Received:

Sponsor or Client: Representative Name
Company Name DuPont
Company Address
Phone

Project Lead: Kris Hansen (8-6018)
Group Leader: Jim Johnson (8-5294)

Analyte(s) or Test Method #: POAA
Sample Matrix: ~~water~~ Soil
Analysis Dates: 9/97-11/97 Analyst(s): GML, JJ, kjh
Author: kjh

Data Reviewed by: MEE
Project Lead (or designee):kjh
James D. Johnson (or designee):

Internal — Sent by: / Date

JDJ:
QAU (Archives):
LIRN System:
Project Manager: Sue Beach

Others *(List Recipients / Address / Phone / FAX)* — Sent by: / Date

T. DiPasquale, 22-11E-03; 3-1891; 736-3257 — kjh on 11/10/97

A copy of the report including this form and the client cover page is to be given to QAU, LIRN and to the Group Leader.

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3MA00230151

3M Environmental Laboratory- Advanced Method Development Team
Kris Hansen - Sr. Analytical Chemist
Advanced Method Development Team
Building 2-3E-09
612-778-6018
kjhansen@mmm.com

Report - Analytical Study
Determination of POAA in Soil and Water
Lab request- R2382

1.0 Summary

Eleven soil samples were submitted by DuPont for quantitative analysis of perfluorooctanoic acid anion (POAA). The soil samples were assigned number R2382-1 through -11. Extraction of the soils using high pressure solvent extraction (HPSE) followed by analysis of the extracts by negative ion electrospray mass spectrometry (ES/MS) determined that POAA is present in all samples. The concentration of POAA in R2382-1 was determined using the method of standard additions. All other soils were evaluated relative to the curve generated in the standard additions analysis. Specific results are listed in Table 1.

Table 1. Concentration of POAA in R2382 samples.

Sample #	mg POAA/kg soil
R2382-1	0.119
R2382-2	0.170
R2382-3	748
R2382-4	272
R2382-5	280
R2382-6	52.8
R2382-7	37.3
R2382-8	39.7
R2382-9	18.0
R2382-10	12.7
R2382-11	2.27

* limit of detection/limit of quantitation is 0.100 mg (100 ppb).

2.0 TEST MATERIALS

Eleven soil samples were received from DuPont on 06/10/97. The samples were stored at 4°C until extraction; extracts were stored at 4°C until analysis.

3.0 EXPERIMENTAL-OVERVIEW AND METHODS

3.1.1 Sample, soil

Because no uncontaminated soil was available for blank analysis, the method of standard addition was used to determine the concentration of POAA in the soil received from DuPont. A five point standard curve was prepared by spiking 2 gram samples of the soil (R2382-1) with some amount of POAA solution between 500 ppt and 1.0 ppm. Two-gram samples of the ten remaining soils, and the spiked soils were each mixed with approximately 1 gram of diatomaceous earth and loaded into a 10 mL stainless steel extraction cartridge. The samples were extracted using high pressure solvent extraction (HPSE) with methanol. Each sample was extracted into an 'a' (primary extract) and 'b' (secondary extract) vial. The

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with nitrogen and reconstituted with acetonitrile (ACN), filtered, and diluted with water (1:1). After analysis by negative ion ES/MS, the data from the spiked samples was subjected to linear regression and the resulting prediction equation was used to determine the concentration of analyte in the unspiked sample R2382-1 (see Figure 1, attached). The remaining soils were evaluated relative to this curve. Soil from samples R2382-2 through -11 was prepared in the same way. For most samples, dilutions of the extracts in 'a' and 'b' vials were necessary. It was also necessary to dilute and reanalyze four samples on 11/04/97. The POAA concentrations of these samples were determined by the same method, using a standard curve generated that day (see Figure 2, attached).

The method of standard addition assumes there are no interferences in the analysis and that the extraction efficiency of the analyte from the matrix is not dependent upon analyte concentration. The first assumption is addressed by the selectivity of both the extraction and the analysis; the latter has been verified in another study that focuses on a similar matrix.

3.1 Extraction specifics

The soil samples were extracted with the ISCO 3560 Accelerated Extraction System equipped with ISCO 100DX high pressure syringe pumps according to the following conditions:

Extraction solvent:	methanol, HPLC grade
Extraction pressure:	2500 psi
Extraction temperature:	70° C
Restrictor temperature:	70° C
Static extraction time-1:	40 minutes
Dynamic extraction volume-1:	15 mL
Static extraction time-2:	2 minutes
Dynamic extraction time-2:	2 minutes
Restrictor flow rate:	2.5 mL/min

Samples were reconstituted in glass autovials with HPLC-grade ACN and milli-Q water.

3.4 ES/MS Analysis specifics

Negative ion ES/MS analysis was performed on a Micromass Platform II atmospheric pressure ionization mass spectrometer running Mass Lynx 2.1 operating system. A Hewlett Packard 1100 was used for the autosampler and HPLC system.

Mobile phase: ACN/H2O (1:1)
Flow rate: 60 µL/min
Injection volume: 15µL
Cone Voltage = -20
Capillary voltage = -2.56
Source Temperature = 80°C
Analyzer Vacuum Pressure = 0.000079 mBar

Quantitative results were based on the instrumental response generated by monitoring a single ion characteristic of the analyte. This type of monitoring minimizes interference by other ions in the extract and increases system sensitivity to the target analyte.

4.0 DATA ANALYSIS

By the method of standard addition, sample R2382-1 was determined to contain 0.119mg/kg of POAA. This value was calculated using the prediction equation resulting from linear regression analysis of the five-point extracted curve. The coefficient of determination for the curve is 0.999. Calculations used to determine the concentration of POAA in the soil are shown in Appendix A.

The concentration of POAA in samples R2382-2 through R2382-11 was determined by evaluation of ES/MS response relative to the curve generated for sample R2382-1. Calculations are detailed in Appendix A.

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5.0 CONCLUSION

High pressure solvent extraction, ES/MS analysis, and linear regression analysis were used to determine that between 0.119 and 614 mg/kg of POAA is present in the eleven soil samples received from DuPont.

6.0 MAINTENANCE OF RAW DATA AND RECORDS

Hard copies of these data are filed in the AMDT archive.

Sample preparation : GML/JJ
Analysis: GML/JJ/kjh
Report preparation: kjh/JJ

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R-2382- DuPont Soil Data
Appendix A

R-2382 POAA Determination Calculations

For Determination of "indigent" amount in Standard Additions Curve for R2382-1

Step 1: From Plot of Peak Area vs Spiked POAA Standard Concentration determine equation of the linear regression by least squares analysis, for $y = mx + b$

Example: $y = 126000\,x + 15000$

Step 2: Solve linear equation for x, where $y = 0$, for the x-intercept of the line.

Example: $0 = 126000\,x + 15000$ $x = -15000/126000$ $x = -.119$

Step 3: Indigent amount will equal absolute value of x.

For Soil R-2382 Soils 2-11

Step 1: (Peak Area - Y intercept)/ Response = Diluted Conc. of POAA (ug/ml)
Peak Area, Intercept, and response from std.additions curve calculated in ug/ml

Example: (31000 - 15000)/126000 = 0.127 ug/ml

Step 2: (Diluted Conc. of POAA ug/ml * Dilution Factor) + Indigent Conc. ug/mL= Adjusted Conc. of POAA ug/ml

Example: (0.127 ug/ml * 50) + .119 ug/ml = 6.47 ug/ml

Step 3: Adjusted Conc. (ug/ml) * 2ml extract/mass (g) * 1000 g/kg * 1 mg/1000 ug = Total Conc. of POAA (mg/kg)

Example: 6.47 ug/ml * 2ml extract/ 1.9999 g * 1000 g/kg * 1mg/ 1000 ug = 6.47 mg/kg POAA

Step 4: Add Total Conc. of POAA determined for Peaks "a" and "b" = Total determined POAA (mg/kg)

Example: 6.47 mg/kg + 0.43 mg/kg = 6.90 mg/kg

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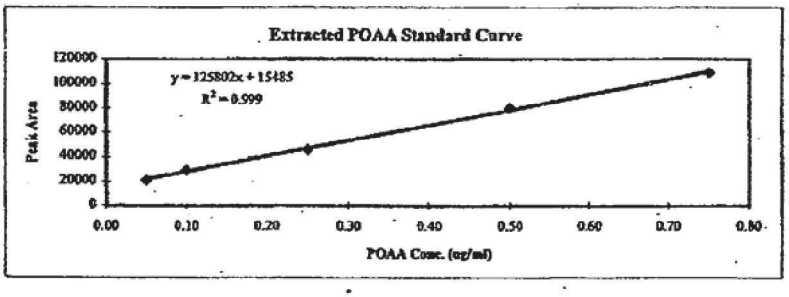
R-2382- DuPont Soil Data

Figure 1

R-2382-1 Soil- Standard Addition Curve (ug/ml extract)

Conc. of POAA Spiked into Soil (ug/ml)	File D102897B Peak Area of Extracts-a	File D102897B Peak Area of Extracts-b	File D102897B Total Peak Area (a+b)	File D102897D Total Peak Area (a+b)	Average of Initial & Final Curve
0.05	20000	1000	21000	[redacted]	21000
0.10	25000	3000	28000		29000
0.25	40000	4000	44000		46000
0.50	71000	9000	80000		86000
0.75	96000	14000	110000		109000

	File D102897D Peak-a	File D102897D Peak-b		
0.05	20000	1000	[redacted]	21000
0.10	27000	3000		30000
0.25	44000	4000		48000
0.50	71000	9000		80000
0.75	94000	14000		108000



	Y-intercept	Slope (m)	X-intercept
Curve Equation:	15000	126000	-0.119

Indigent POAA analyte (Absolute value of X-intercept): 0.119 ug/ml

Soil 1 Curve ugml R2382_103097 9:00 PM11/4/97

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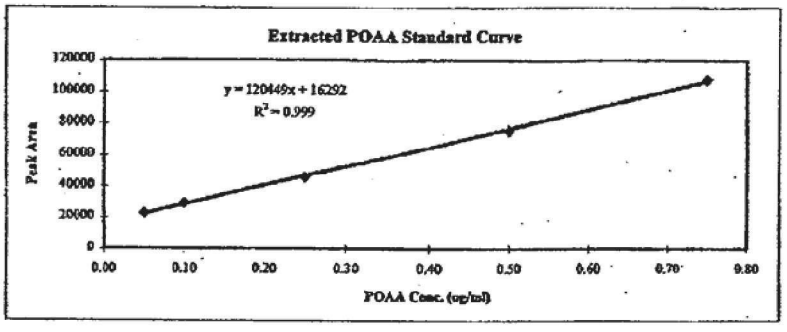
R-2382- DuPont Soil Data

Figure 2

R-2382-1 Soil- Standard Addition Curve Analyzed by ESMS on 11/04/97

Conc. of POAA Spiked into Soil (ug/ml)	File D110497B Peak Area of Extracts-a	File D110497B Peak Area of Extracts-b	File D110497B Total Peak Area (a+b)
0.05	21000	1700	22700
0.10	26000	3200	29200
0.25	41000	4400	45400
0.50	67000	8200	75200
0.75	94000	13700	107700



	Y-intercept	Slope (m)	X-intercept
Curve Equation:	16000	120000	-0.133

Indigent POAA analyte (Absolute value of X-intercept): 0.133

11.04 Curve ugml R2382_103097 8:58 PM 11/4/97

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3M Environmental Laboratory

Volatiles and Semi-volatiles in Groundwater by Purge and Trap Concentration with GC/MS Analyses (6/97 Samples)

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ANALYTICAL SUMMARY

Department: 3048 | *Contract Lab:* Pace-I

Lab Request: R2148 | *Project Lead:* Dennis R. Seeger

Project Description: DuPont Water | *Sample Matrix:* Water

Summary Prepared by: Dennis Seeger, Pace-I | *Date:* 10/7/97

Analytical Tests Requested

Twelve water samples were submitted for identification and quantitation of volatile organic sample components by purge and trap sample concentration with gas chromatography/mass spectrometry (GC/MS) analysis.

Analytical Results

The results of the GC/MS analyses are reported in *Appendix A*. After an initial analysis of the undiluted samples, appropriate dilutions were analyzed for quantitative determinations of trichlorotrifluoroethane and trichloroethene. Where the calculated concentrations were below the practical quantitation limit (J footnote), the reported values should be considered as estimates.

Analytical Summary

Gas Chromatography Methods

Summary

The samples were analyzed using the GC/MS instrument and sample concentrator conditions listed below.

Procedure

GC/MS Parameters (Instrument ID "Alphie")

Sample Concentrator: Tekmar model 2000 sample concentrator and model 2050 vial autosampler.

Trap:	Carbopack B/Carboxen 1000 & 1001 (Vocarb 3000)
Purge time:	11 min.
Purge gas flow:	40 mL/min.
Desorb time:	0.5 min.
Desorb temp.:	270 °C
Desorb flow:	30 mL/min

GC column: Restek RTx-624, 60 m x 0.32 mm I.D., 1.8 µm film thickness.

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GC conditions and oven temperature program:

Initial temp.:	40°C; 2.0 min. hold
Oven temp. ramp:	12°C/min. to 220°C; 1.0 min. hold
Injection port temp.:	250°C
Interface temp.:	250°C
Purge B:	Initial value ON
Head pressure:	19.6 psig
Split flow:	30 mL/min.

Mass spectrometer:

Solvent delay:	2.2 min.
Electron multiplier:	2053 volts
Scan range:	35 to 260 amu
Scans per second:	2.17
Scan threshold:	100

Instrument Calibration

Prior to sample analyses, the analysis of 50 ng of bromofluorobenzene (BFB) demonstrated the accuracy and resolution of the mass spectrometer. A calibration check standard containing each of the target analytes at the midpoint concentration of the most recent five level calibration curve was analyzed to demonstrate acceptable instrument response for target analyte quantitation. A blank water sample water sample was analyzed to demonstrate analytical system cleanliness. All quality control analyses satisfied the criteria specified for analyzing samples by EPA method 8260.

Closing

This analytical summary and associated analytical results have been reviewed and are approved for release.

Dennis R. Seeger, Project Manager
(612) 778-6093

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Q. And what is your date of birth, ma'am?
A. August 3rd, 1963.
Q. And your home address?
A. 202 North Broadview, Wichita, Kansas 67208.
Q. And are you taking any medications of any kind today that could affect your memory in any way?
A. No, I am not.
Q. Have you ever had your deposition taken before?
A. Yes, I have.
Q. How many times?
A. Once.
Q. When was that?
A. 1986 or 7, I believe.
Q. And with whom, if anyone, were you employed at the time?
A. State of Minnesota.
Q. With any particular agency?
A. The Minnesota Pollution Control Agency.
Q. And is it okay for purposes of today's if we refer to that agency, the Minnesota Pollution Control Agency, as MPCA?
A. Yes.
Q. Other than the deposition you just mentioned

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back in 1986 while you were employed with the MPCA, have you ever participated in any other depositions of any kind?
A. No, I have not.
Q. Have you ever provided testimony at any trial?
A. No, I have not.
Q. Have you ever provided testimony in any court proceeding of any kind other than the deposition you just mentioned?
A. No.
Q. Since it's been a while since your last deposition, I'm going to go over some of the ground rules on how the deposition process will work today, all right?
A. Yes.
Q. Today I'm going to be asking you a number of questions, many of which may concern events or activities that occurred a long time ago. So I ask that you please not guess or speculate when providing answers, but please provide me your best recollection of the facts, all right?
A. (Witness nods head.)
Q. And please make sure to verbalize all of your

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responses because your responses are being recorded not only by videotape, but also in a written transcript. All right?
A. Yes.
Q. And, also, please make sure to keep your voice up to the extent you can today so we can make sure we've got a clear video record and that the court reporter can hear you clearly, all right?
A. Yes.
Q. If at any point in time you don't understand one of my questions or want clarification in any way, please let me know, and I'll go back and try to rephrase it or clarify it for you. Otherwise, I'm going to assume that you've heard it, understood it, and gave me your best response, all right?
A. Yes.
Q. And if at any point in time you feel like you need to take a break for any reason, let me know. Otherwise, we'll try to take breaks at reasonable times today, all right?
A. Very good.
Q. Are you represented by counsel today?
A. Yes, I am.

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Q. And who is that counsel?
A. Jim Armstrong.
Q. And he's the attorney sitting to your right, correct?
A. That's correct.
Q. At any point prior to today, have you requested any counsel from the 3M Company in connection with your deposition today?
A. No, I have not.
Q. Have you discussed this deposition with any counsel for 3M prior to today?
A. I received a phone call from John Allison in, I believe, the November 2007 time frame, alerting me to the effect that I would be -- there was a request for me to be deposed by plaintiff's counsel. And that is the only contact that I've had with 3M regarding this proceeding.
Q. Was anyone else on the telephone during that call between you and Mr. Allison?
A. No.
Q. How long did that call last?
A. Perhaps 5 to 10 minutes.
Q. At the time you received that call from Mr. Allison, were you employed with your

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Appendix A: Report of Laboratory Analysis

3MA00230161

CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

3M_MN00043340

2759.0035

Method 8260 Results for R2148

Compound	PRL (ug/L)	Sample Concentrations (ug/L) R2148-1	R2148-2	R2148-3	R2148-4	R2148-5	R2148-6
Dichlorodifluoromethane	10	-	-	-	-	-	-
Chloromethane	10	-	-	-	-	-	-
Vinyl Chloride	10	-	-	-	-	-	-
Bromomethane	10	-	-	-	-	-	-
Chloroethane	10	-	-	-	-	-	-
Trichlorofluoromethane	10	-	-	18	18	26	26
Ethyl Ether	5	-	-	-	-	-	-
Trichlorotrifluoroethane	5	820	730	1600	1500	2300	2400
Acrolein	40	-	-	-	-	-	-
1,1-Dichloroethene	5	-	-	-	3.7 J	-	-
Acetone	10	-	10	-	-	7.8 J	8.0 J
Isopropyl Alcohol	60	-	-	-	-	-	-
Carbon Disulfide	5	-	-	-	-	-	-
Allyl Chloride	5	-	-	-	-	-	-
Methylene Chloride	5	2.7 J	2.9 J	-	-	2.3 J	2.7 J
tert-Butyl Alcohol	20	59	49	-	-	-	-
tert-Methyl Butyl Ether	5	-	-	-	-	-	-
trans-1,2-Dichloroethene	5	-	-	-	-	2.9 J	3.0 J
Acrylonitrile	40	-	-	-	-	-	-
Isopropyl Ether	5	-	-	-	-	-	-
1,1-Dichloroethane	5	-	-	-	-	-	-
2,2-Dichloropropane	5	-	-	-	-	-	-
Ethyl Acetate	10	-	-	-	-	-	-
cis-1,2-Dichloroethene	5	-	-	-	-	3.3 J	3.5 J
2-Butanone	10	-	-	-	-	-	-
2-Butanol	60	-	-	-	-	-	-
Bromochloromethane	5	-	-	-	-	-	-
Tetrahydrofuran	10	-	-	-	-	-	-
Chloroform	5	-	-	-	-	5.5	5.9
1,1,1-Trichloroethane	5	-	-	-	-	14	14
Carbon Tetrachloride	5	-	-	-	-	-	-
1,1-Dichloropropene	5	-	-	-	-	-	-
Isobutanol	100	-	-	-	-	-	-
Benzene	5	-	-	-	-	-	-
1,2-Dichloroethane	5	-	-	-	-	-	-
n-Butanol	100	-	-	-	-	-	-
Trichloroethene	5	1.8 J	1.5 J	140	150	520	570
1,2-Dichloropropane	5	-	-	-	-	-	-
Dibromomethane	5	-	-	-	-	-	-
Bromodichloromethane	5	-	-	-	-	-	-
2-Chloroethyl Vinyl Ether	10	-	-	-	-	-	-
2-Nitropropane	10	-	-	-	-	-	-
cis-1,3-Dichloropropene	5	-	-	-	-	-	-
4-Methyl-2-pentanone	10	-	-	-	-	-	-
Toluene	5	-	-	-	-	-	-
4-Methyl-2-Pentanol	60	-	-	-	-	-	-

PRL – Practical Quantitation Limit

J – The concentration is below the practical quantitation limit.

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3M_MN00043341

2759.0036

Method 8260 Results for R2148

Compound	PRL (ug/L)	Sample Concentrations (ug/L) R2148-1	R2148-2	R2148-3	R2148-4	R2148-5	R2148-6
trans-1,3-Dichloropropene	5	-	-	-	-	-	-
1,1,2-Trichloroethane	5	-	-	-	-	-	-
Tetrachloroethene	5	-	-	-	1.1 J	5	5.2
1,3-Dichloropropane	5	-	-	-	-	-	-
2-Hexanone	10	-	-	-	-	-	-
Dibromochloromethane	5	-	-	-	-	-	-
1,2-Dibromoethane	5	-	-	-	-	-	-
Chlorobenzene	5	-	-	-	-	-	-
Ethylbenzene	5	-	-	-	-	-	-
1,1,1,2-Tetrachloroethane	5	-	-	-	-	-	-
m & p-Xylene	5	-	-	-	-	-	-
o-Xylene	5	3.6 J	1.7 J	-	1.8 J	-	-
Styrene	5	-	-	-	-	-	-
Bromoform	5	-	-	-	-	-	-
Isopropyl benzene	5	-	-	-	-	-	-
Cyclohexanone	60	-	-	-	-	-	-
1,1,2,2-Tetrachloroethane	5	-	-	-	-	-	-
Bromobenzene	5	-	-	-	-	-	-
n-Propyl benzene	5	-	-	-	-	-	-
1,2,3-Trichloropropane	5	-	-	-	-	-	-
2-Chlorotoluene	5	-	-	-	-	-	-
1,3,5-Trimethylbenzene	5	-	-	-	-	-	-
4-Chlorotoluene	5	-	-	-	-	-	-
tert-Butyl benzene	5	-	-	-	-	-	-
1,2,4-Trimethylbenzene	5	-	-	-	-	-	-
sec-Butylbenzene	5	-	-	-	-	-	-
p-Isopropyltoluene	5	-	-	-	-	-	-
1,3-Dichlorobenzene	5	-	-	-	-	-	-
1,4-Dichlorobenzene	5	-	-	-	-	-	-
n-Butyl benzene	5	-	-	-	-	-	-
1,2-Dichlorobenzene	5	-	-	-	-	-	-
1,2-Dibromo-3-Chloropropane	5	-	-	-	-	-	-
1,2,4-Trichlorobenzene	5	-	-	-	-	-	-
Hexachlorobutadiene	5	-	-	-	-	-	-
Naphthalene	5	-	-	-	-	-	-
1,2,3-Trichlorobenzene	5	-	-	-	-	-	-

PRL – Practical Quantitation Limit
J – The concentration is below the practical quantitation limit.

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3M_MN00043342

2759.0037

Method 8260 Results for R2148

Compound	PRL (ug/L)	Sample Concentrations (ug/L) R2148-7	R2148-8	R2148-9	R2148-10	R2148-11	R2148-12
Dichlorodifluoromethane	10	-	-	-	-	-	-
Chloromethane	10	-	-	-	-	-	-
Vinyl Chloride	10	-	-	-	-	-	-
Bromomethane	10	-	-	-	-	-	-
Chloroethane	10	-	-	-	-	-	-
Trichlorofluoromethane	10	5.9	-	-	-	-	-
Ethyl Ether	5	-	-	-	-	-	-
Trichlorotrifluoroethane	5	760	670	130	140	3.2 J	1.8 J
Acrolein	40	-	-	-	-	-	-
1,1-Dichloroethene	5	-	-	-	-	-	-
Acetone	10	6.7 J	10	-	-	-	5.6 J
Isopropyl Alcohol	60	-	-	-	-	-	-
Carbon Disulfide	5	-	-	-	-	-	-
Allyl Chloride	5	-	-	-	-	-	-
Methylene Chloride	5	-	-	-	-	-	-
tert-Butyl Alcohol	20	-	-	-	-	-	-
tert-Methyl Butyl Ether	5	-	-	-	-	-	-
trans-1,2-Dichloroethene	5	-	-	-	-	-	-
Acrylonitrile	40	-	-	-	-	-	-
Isopropyl Ether	5	-	-	-	-	-	-
1,1-Dichloroethane	5	-	-	-	-	-	-
2,2-Dichloropropane	5	-	-	-	-	-	-
Ethyl Acetate	10	-	-	-	-	-	-
cis-1,2-Dichloroethene	5	5.8	5.9	2.7 J	2.6 J	-	-
2-Butanone	10	-	-	-	-	-	-
2-Butanol	60	-	-	-	-	-	-
Bromochloromethane	5	-	-	-	-	-	-
Tetrahydrofuran	10	-	-	-	-	-	-
Chloroform	5	37	39	13	16	-	-
1,1,1-Trichloroethane	5	6.3	6.2	-	5.8	-	-
Carbon Tetrachloride	5	-	-	-	-	-	-
1,1-Dichloropropene	5	-	-	-	-	-	-
Isobutanol	100	-	-	-	-	-	-
Benzene	5	-	-	-	-	-	-
1,2-Dichloroethane	5	-	-	-	-	-	-
n-Butanol	100	-	-	-	-	-	-
Trichloroethene	5	81	66	-	-	-	-
1,2-Dichloropropane	5	-	-	-	-	-	-
Dibromomethane	5	-	-	-	-	-	-
Bromodichloromethane	5	-	-	-	-	-	-
2-Chloroethyl Vinyl Ether	10	-	-	-	-	-	-
2-Nitropropane	10	-	-	-	-	-	-
cis-1,3-Dichloropropene	5	-	-	-	-	-	-
4-Methyl-2-pentanone	10	-	-	-	-	-	-
Toluene	5	-	-	-	-	-	-
4-Methyl-2-Pentanol	60	-	-	-	-	-	-

PRL – Practical Quantitation Limit
J – The concentration is below the practical quantitation limit.

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3M_MN00043343

2759.0038

Method 8260 Results for R2148

Compound	PRL (ug/L)	Sample Concentrations (ug/L) R2148-7	R2148-8	R2148-9	R2148-10	R2148-11	R2148-12
trans-1,3-Dichloropropene	5	-	-	-	-	-	-
1,1,2-Trichloroethane	5	-	-	-	-	-	-
Tetrachloroethene	5	4.8 J	5.8	-	-	-	-
1,3-Dichloropropane	5	-	-	-	-	-	-
2-Hexanone	10	-	-	-	-	-	-
Dibromochloromethane	5	-	-	-	-	-	-
1,2-Dibromoethane	5	-	-	-	-	-	-
Chlorobenzene	5	-	-	-	-	-	-
Ethylbenzene	5	-	-	-	-	-	-
1,1,1,2-Tetrachloroethane	5	-	-	-	-	-	-
m & p-Xylene	5	-	-	-	-	-	-
o-Xylene	5	-	-	-	-	-	-
Styrene	5	-	-	-	-	-	-
Bromoform	5	-	-	-	-	-	-
Isopropyl benzene	5	-	-	-	-	-	-
Cyclohexanone	60	-	-	-	-	-	-
1,1,2,2-Tetrachloroethane	5	-	-	-	-	-	-
Bromobenzene	5	-	-	-	-	-	-
n-Propyl benzene	5	-	-	-	-	-	-
1,2,3-Trichloropropane	5	-	-	-	-	-	-
2-Chlorotoluene	5	-	-	-	-	-	-
1,3,5-Trimethylbenzene	5	-	-	-	-	-	-
4-Chlorotoluene	5	-	-	-	-	-	-
tert-Butyl benzene	5	-	-	-	-	-	-
1,2,4-Trimethylbenzene	5	-	-	-	-	-	-
sec-Butylbenzene	5	-	-	-	-	-	-
p-Isopropyltoluene	5	-	-	-	-	-	-
1,3-Dichlorobenzene	5	-	-	-	-	-	-
1,4-Dichlorobenzene	5	-	-	-	-	-	-
n-Butyl benzene	5	-	-	-	-	-	-
1,2-Dichlorobenzene	5	-	-	-	-	-	-
1,2-Dibromo-3-Chloropropane	5	-	-	-	-	-	-
1,2,4-Trichlorobenzene	5	-	-	-	-	-	-
Hexachlorobutadiene	5	-	-	-	-	-	-
Naphthalene	5	-	-	-	-	-	-
1,2,3-Trichlorobenzene	5	-	-	-	-	-	-

PRL – Practical Quantitation Limit
J – The concentration is below the practical quantitation limit.

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3M_MN00043344

3M Environmental Laboratory

Volatiles by AED (5/97 Samples)

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3MA00230166

CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

3M Environmental Laboratory

Data Transmittal Summary

Preliminary (Final) *(circle one)*

Lab Request #:	3M Study #:	Contract Laboratory #
R2Ø8	3Ø48/3Ø58 Bigenenvir	

Date Received:

Sponsor or Client: Representative Name
Company Name DuPont
Company Address
Phone

Project Lead: Name / Phone KHansen/86Ø18
Group Leader: Name / Phone JDJohnson/85294

Analyte(s) or Test Method #: POAA
Sample Matrix: Water, Soil
Analysis Dates: Ø6Ø997-Ø62697 Analyst(s): SEMiller
Author: SEMiller

Data Reviewed by:
Project Lead (or designee):
James D. Johnson (or designee):

Internal	Sent by: / Date
JDJ:	
QAU (Archives):	
LIRN System:	
Project Manager: Sue Beach	

Others *(List Recipients / Address / Phone / FAX)*	Sent by: / Date

A copy of the report including this form and the client cover page is to be given to QAU, LIRN and to the Group Leader.

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3MA00230167

3M Environmental Laboratory - Advanced Method Development Team

Contact: Kris Hansen
Building 02-3E-09
612-778-6018
kjhansen@mmm.com

Final Report - Lab Request R2008
DuPont Water and Soil Samples
07 November 1997

1.0 SUMMARY

Five water samples and eight soil samples from DuPont were analyzed for the presence of fluorine, chlorine, bromine, carbon, and hydrogen using headspace sampling and gas chromatography coupled with an atomic emission detector (GC/AED). The samples were analyzed on two different columns, a DB-5 and a DB-624. Standard curves were generated during each analysis. Very little was seen in the samples and the compounds that were detected existed at levels below the lowest standard.

2.0 INTRODUCTION

Five water samples and eight soil samples were received from DuPont under request #2008. The samples were to be analyzed for the presence of perfluorooctanoic acid anion (POAA). However, since it is not volatile, POAA was not detected using GC/AED. A headspace sampler was used to introduce any volatile or semi-volatile components of the samples into the GC/AED. The samples were monitored for fluorine (F690), bromine (Br478), chlorine (Cl479), hydrogen (H486), and carbon (C496).

3.0 TEST MATERIALS

The five water samples were labeled on large amber glass bottles as follows:

Lab Request #	# of Bottles	Sample Description
R2008-1	1	DuPont Wash. Works ① Ranney Well FC143 1:48 pm
R2008-2	1	DuPont Wash. Works ② Ranney Well FC143 1:48 pm
R2008-3	1	DuPont Wash. Works ③ Ranney Well FC143 1:50 pm
R2008-4	1	DuPont Wash. Works ④ Ranney Well FC143 1:50 pm
R2008-5	1	DI Water

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3M_MN00043347

2759.0042

The eight soil samples were labeled on 1L plastic containers as follows:

Lab Request #	# of Containers	Sample Description
R2008-6	8	DuPont Washington Dirt 5/30/97 11:00

All samples were refrigerated at approximately 4°C until sample preparation and analysis.

4.0 EXPERIMENTAL - OVERVIEW

Sample Preparation

The water samples were prepared by pipetting 10mL of each sample into 20mL glass headspace vials. Each sample was "salted" by adding approximately 2 to 3 grams of sodium chloride (this was done to increase the ionic strength of the solution). The soil samples were prepared by transferring 10 ± 0.5 grams of soil (weight recorded) into headspace vials. The soil samples were not salted.

The standard curves were prepared using two different standards, para-bromofluorobenzene (p-BFB) and ortho-dichlorobenzene (o-DCB). The p-BFB was prepared in acetone and the o-DCB was prepared in methanol. The standards were spiked into 10mL Milli-Q water at levels of 25µl, 50µl, and 100µl. Acetone and methanol spikes (100µl each) in 10mL Milli-Q as well as a 10mL Milli-Q blank were also analyzed.

Since all eight containers of soil were the same, three of the eight samples were used to make a standard curve. These were spiked exactly as the waters were. One soil sample was spiked with acetone and methanol, leaving four containers of soil to be treated as "samples."

Because two different columns were used and all five elements could not be monitored simultaneously, the water and soil samples were prepared four separate times. Each time a standard curve was generated. When just F690 was monitored, the standard curve was generated based on p-BFB. When all other elements were monitored, two standard curves were generated, using p-BFB and o-DCB.

Instrumentation and Operating Conditions

Headspace Sampler: Hewlett Packard 19395A
Settings: Bath Temperature 85°C
Valve/Loop Temperature 140°C
Probe in, t = 1 second
Vial Pressurized, t = 3 seconds to 13 seconds
Vent/Fill Loop, t = 14 seconds to 19 seconds
Inject into GC, t = 20 seconds to 50 seconds
Probe out, t = 51 seconds
Packed column on vent

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2759.0043

Gas Chromatograph: Hewlett Packard 5890 Series II
Column: DB-5 (J&W Scientific) 30 x .25 x .25, serial # 2633586
Oven Program: 1 min @ 60°, 10°/min to 300° for 5 min (F690)
1 min @ 40°, 10°/min to 300° for 5 min
Column: DB-624 (J&W Scientific) 30 x .32 x 1.8, serial # 5812142
Oven Program: 1 min @ 40°, 10°/min to 200° for 5 min
Injection Port: 225°C, split

Atomic Emission Detector: Hewlett Packard 5921A "Flo"
GC Block/Transfer Line Temp 275°
Cavity Block Temp 275°

5.0 DATA ANALYSIS

SAMPLE RESULTS:

Lab Request #	Column	Element	Results*
R2008-1	DB-5	F	no peaks detected
R2008-2	DB-5	F	no peaks detected
R2008-3	DB-5	F	no peaks detected
R2008-4	DB-5	F	no peaks detected
R2008-5	DB-5	F	no peaks detected
R2008-6	DB-5	F	no peaks detected

*minimum quantitation limit: 0.280 ppm F in water, 0.282 ppm F in soil

Lab Request #	Column	Elements	Results*
R2008-1	DB-5	H, C, Br, Cl	no peaks detected
R2008-2	DB-5	H, C, Br, Cl	no peaks detected
R2008-3	DB-5	H, C, Br, Cl	no peaks detected
R2008-4	DB-5	H, C, Br, Cl	no peaks detected
R2008-5	DB-5	H, C, Br, Cl	no peaks detected
R2008-6	DB-5	H, C, Br, Cl	no peaks detected

*minimum quantitation limits: 1.176 ppm Br in water, 1.220 ppm Br in soil
1.408 ppm Cl in water, 1.461 ppm Cl in soil

Lab Request #	Column	Element	Results*
R2008-1	DB-624	F	no peaks detected
R2008-2	DB-624	F	no peaks detected
R2008-3	DB-624	F	no peaks detected
R2008-4	DB-624	F	no peaks detected
R2008-5	DB-624	F	no peaks detected
R2008-6	DB-624	F	no peaks detected

*minimum quantitation limit: 0.280 ppm F in water, 0.282 ppm F in soil

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2759.0044

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current employer?
A. Yes.
Q. Had you asked that anyone from 3M contact you?
A. No.
Q. What did Mr. Allison tell you during that phone call?
A. Mr. Allison, as I said earlier, alerted me to the fact that it was known to him that plaintiff's counsel had an interest in deposing me.
Q. And was there any discussion about why?
A. Not really. The only discussion that we had was about the upcoming potential deposition and also the time frame, because his understanding was that it was going to -- that the request was for later in November, and it was very -- the time frame was unworkable for me from my scheduling perspective.
Q. Was there any discussion about the substance of the case in which you were being requested to provide the deposition?
A. No.
Q. During the call with Mr. Allison, did

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Mr. Allison offer counsel?
A. He did not.
Q. Did you request that 3M provide counsel for the deposition?
A. I did.
Q. What was the response?
A. The response was that because I was no longer an employee with 3M it would be better for me to be represented by my own counsel.
Q. So you did ask Mr. Allison for 3M to provide counsel to you for the deposition, correct?
A. I did.
Q. Did you have any further discussion with Mr. Allison about the position 3M was taking in that regard?
MR. ARMSTRONG: In regard to counsel?
BY MR. BILOTT:
Q. Yes.
A. Can you explain your question more fully?
Q. After you asked for counsel and Mr. Allison told you that 3M would not be offering counsel, did you have any discussion with him about why?
A. The discussion that we had about counsel was

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related to then what were my options. And he responded that that was really my concern, that he couldn't provide any kind of legal advice to me. And my response was, I will then hire my own attorney. And that was basically the end of the conversation.
Q. Did you ask for any recommendations from Mr. Allison for your own counsel?
A. I did not.
Q. And then you retained Mr. Armstrong, correct?
A. Yes, I did.
Q. All right. Any further discussions with any counsel for 3M other than the discussion you just described with Mr. Allison?
A. No.
Q. Have you had any discussions with any employee of 3M -- let me -- we'll come back to that.
(Deposition Exhibit No. 1 was marked for identification.)
Q. Would you prefer that I refer to you as Ms., Mrs.?
A. It doesn't matter to me.
Q. Okay. Ms. Corrigan, I'm going to hand you what's been marked as Exhibit 1 in your

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deposition, and ask you to take a look at that and tell me if you recall seeing this document before.
Let me restate the question.
Ms. Corrigan, do you recall ever seeing a written notice for your deposition?
A. Yes, I do.
Q. All right. And you recognize this as that notice?
A. I'm going through it now. It looks to be that notice, yes.
Q. Do you recall the written notice for your deposition also included a request for you to search for certain categories of documents that you might have in your possession?
A. Yes.
Q. And did you do that?
A. Yes, I did.
Q. And did you find any documents responsive to any of the requests for production?
A. I did.
Q. What did you find?
A. I found a copy of a recusal letter that I had composed while commissioner for the Minnesota Pollution Control Agency.

2100 3rd Avenue North, Suite 960*Birmingham, Al 35203*
1-800-888-DEPO

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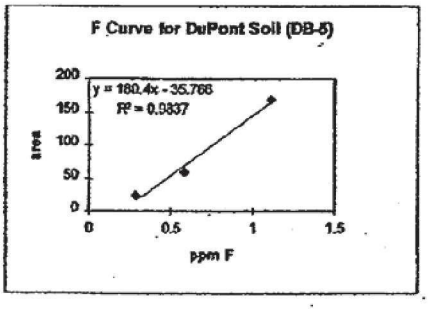
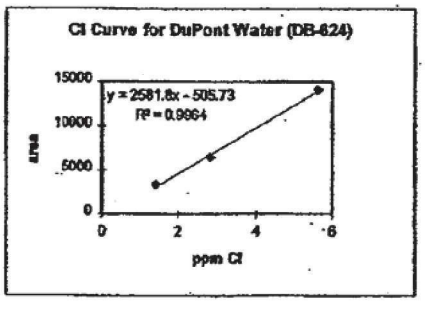
Lab Request #	Column	Elements	Results*
R2008-1	DB-624	H, C, Br, Cl	peaks detected on Cl channel (below mql), no peaks on other channels
R2008-2	DB-624	H, C, Br, Cl	peaks detected on Cl channel (below mql), no peaks on other channels
R2008-3	DB-624	H, C, Br, Cl	peaks detected on Cl channel (below mql), no peaks on other channels
R2008-4	DB-624	H, C, Br, Cl	peaks detected on Cl channel (below mql), no peaks on other channels
R2008-5	DB-624	H, C, Br, Cl	no peaks detected
R2008-6	DB-624	H, C, Br, Cl	no peaks detected

*minimum quantitation limits: 1.176 ppm Br in water, 1.187 ppm Br in soil
1.408 ppm Cl in water, 1.253 ppm Cl in soil

STANDARD CURVES:

The following are examples of standard curves taken from the analyses:





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3MA00230171

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6.0 CONCLUSION

Qualitative analysis of DuPont water and soil revealed very little was present in any of the samples. Cl-containing compounds were found using the DB-624 column in water samples R2008-1 through R2008-4. The levels of these compounds were not quantitated because they were present in levels below the lowest standard.

7.0 MAINTENANCE OF RAW DATA

Hard copies of the data are filed in the AMDT archive.

SE Miller
110797

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3M_MN00043352

2759.0047

3M Environmental Laboratory

Volatiles by AED (6/97 Samples)

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3MA00230173

CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

3M Environmental Laboratory

Data Transmittal Summary

Preliminary (Final) *(circle one)*

Lab Request #:	3M Study #:	Contract Laboratory #
R2148	3048 Bigenenvir	

Date Received: 070297

Sponsor or Client: Representative Name
Company Name DuPont
Company Address
Phone

Project Lead: Name / Phone KHansen/ 7618
Group Leader: Name / Phone JDJohnson/85294

Analyte(s) or Test Method #: Fluorine, Carbon, Hydrogen, Chlorine, Bromine (volatiles + semi-volatil
Sample Matrix: Water
Analysis Dates: 080597 - 082097 Analyst(s): SEMiller
Author: SEMiller

Data Reviewed by: PA Rethwill 100797

Project Lead (or designee):
James D. Johnson (or designee):

Internal	Sent by: / Date
JDJ:	
QAU (Archives):	
LIRN System:	
Project Manager: Sue Beach	

Others *(List Recipients / Address / Phone / FAX)*	Sent by: / Date

A copy of the report including this form and the client cover page is to be given to QAU, LIRN and to the Group Leader.

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3MA00230174

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3M Environmental Laboratory-Advanced Method Development Team

Contact: Kris Hansen
Building 02-3E-09
612-778-6018
kjhansen@mmm.com

Final Report - Lab Request R2148
DuPont Water Samples
07 November 1997

1.0 SUMMARY

Twelve water samples were received from DuPont on July 2, 1997. The samples were analyzed for the presence of fluorine, chlorine, bromine, carbon, and hydrogen using headspace sampling and gas chromatography coupled with an atomic emission detector (GC/AED). The samples were analyzed on two different columns, a DB-5 and a DB-624. Standard curves of no less than R^2=0.99 were generated during each analysis.

Column	Samples	Results Description	Levels*
DB-5	1-8	halogenated organics	0.23-1.3 ppm F, 0.29-0.86 ppm C, 0.39-2.5 ppm Cl
DB-5	9-10	low levels of halogenated organics	0.32-0.35 ppm C, F and Cl areas less than low standard response
DB-5	11-12	C only detected	C area less than low standard response
DB-624	1-8	halogenated organics	0.23-1.1 ppm F, 0.24-0.50 ppm C, 0.45-3.6 ppm Cl
DB-624	9-10	low levels of halogenated organics	0.082 ppm F (#10), F, C, and Cl areas less than low standard response
DB-624	11-12	C and F only detected	C and F areas less than low standard response

*reported as total ppm per sample

Bromine was not detected in any of the samples on either column. Hydrogen was detected in four samples on the DB-5 and in one sample on the DB-624.

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3M_MN00043355

2759.0050

2.0 INTRODUCTION

Twelve monitoring well water samples were received from DuPont under lab request R2148. The samples were to be analyzed for the presence of organofluorines. A headspace sampler was used to introduce any volatile or semi-volatile components of the samples into a GC/AED. The samples were monitored for fluorine (F690), bromine (Br478), chlorine (Cl479), carbon (C496), and hydrogen (H486).

3.0 TEST MATERIALS

The water samples were received in amber glass bottles and labeled as follows:

3M Lab Request #	Sample Description
R2148-1	MW-1 1 of 2
R2148-2	MW-1 2 of 2
R2148-3	MW-2 1 of 2
R2148-4	MW-2 2 of 2
R2148-5	MW-3 1 of 2
R2148-6	MW-3 2 of 2
R2148-7	MW-4 1 of 2
R2148-8	MW-4 2 of 2
R2148-9	MW-5 1 of 2
R2148-10	MW-5 2 of 2
R2148-11	MW-6 1 of 2
R2148-12	MW-6 2 of 2

All samples were refrigerated at approximately 4°C until sample preparation and analysis.

4.0 EXPERIMENTAL - OVERVIEW

Sample Preparation

The water samples were prepared by transferring approximately 10mL of each sample into tared headspace vials containing approximately 4 ± 0.1 grams of sodium chloride. The vials were rewieghed to get the weight of the water. This was done instead of pipetting an exact volume so that exposure to air was kept to a minimum, and the possibility of losing volatile components reduced.

Standard curves were prepared using two different standards, para-bromofluorobenzene (p-BFB) and ortho-dichlorobenzene (o-DCB). Two standard curves were necessary so calibration curves could be generated for all elements of interest: fluorine, carbon, hydrogen, and bromine from p-BFB; carbon, hydrogen, and chlorine from o-DCB. The p-BFB was prepared in acetone and the o-DCB was prepared in methanol. The standards were spiked into 10mL Milli-Q water (salted) at levels of 5µL,

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3M_MN00043356

2759.0051

25µL, 50µL, 75µL, and 100µL. Acetone and methanol spikes (100µL each) in 10mL Milli-Q as well as a 10mL Milli-Q blank were also analyzed.

The combination of two columns (the DB-5 is good for late eluting compounds and the DB-624 is used to separate compounds that elute relatively quickly) yielded a thorough analysis for each sample. Because two different columns were used and all five elements could not be monitored simultaneously, the samples were prepared four separate times. When just F690 was monitored, the standard curve was generated based on p-BFB. When all other elements were monitored, two standard curves were generated, using both p-BFB and o-DCB. The final results for fluorine, carbon, hydrogen, and bormine were based on p-BFB. The chlorine curve was based on o-DCB.

Instrumentation and Operating Conditions

Headspace Sampler: Hewlett Packard 19395A
Settings: Bath Temperature 85°C
Valve/Loop Temperature 140°C
Probe in, t = 1 second
Vial Pressurized, t = 3 seconds to 13 seconds
Vent/Fill Loop, t = 14 seconds to 19 seconds
Inject into GC, t = 20 seconds to 50 seconds
Probe out, t = 51 seconds
Packed column on vent

Gas Chromatograph: Hewlett Packard 5890 Series II
Column: DB-5 (J&W Scientific) 30m x .25mm x .25µm, serial # 2633586
Oven Program: 1 min @ 40°, 10°/min to 300° for 3 min
Column: DB-624 (J&W Scientific) 30m x .32mm x 1.8µm, serial # 5812142
Oven Program: 1 min @ 40°, 10°/min to 240° for 5 min
Injection Port: 225°C, split

Atomic Emission Detector: Hewlett Packard 5921A "Flo"

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2759.0052

5.0 DATA ANALYSIS

SAMPLE ANALYSIS:

Column: DB-5

Lab Request #	F690*
R2148-1	0.23 ppm, 1 peak < low std. response
R2148-2	0.23 ppm, 1 peak < low std. response
R2148-3	0.079 ppm, 0.55 ppm
R2148-4	0.34 ppm, 1 peak < low std. response
R2148-5	0.26 ppm, 0.57 ppm
R2148-6	0.39 ppm, 0.90 ppm
R2148-7	0.066 ppm, 0.21 ppm
R2148-8	0.075 ppm, 0.22 ppm
R2148-9	2 peaks < low std. response
R2148-10	2 peaks < low std. response
R2148-11	none detected
R2148-12	none detected

*low standard concentration: 0.056 ppm F

Column: DB-624

Lab Request #	F690*
R2148-1	0.26 ppm F, 3 peaks < low std. response
R2148-2	0.23 ppm F, 3 peaks < low std. response
R2148-3	0.075 ppm, 0.44 ppm F, 2 peaks < low std. response
R2148-4	0.074 ppm, 0.42 ppm F, 2 peaks < low std. response
R2148-5	0.086 ppm, 0.27 ppm, 0.70 ppm F, 3 peaks < low std. response
R2148-6	0.080 ppm, 0.23 ppm, 0.59 ppm F, 3 peaks < low std. response
R2148-7	0.087 ppm, 0.20 ppm F, 2 peaks < low std response
R2148-8	0.083 ppm, 0.16 ppm F, 2 peaks < low std. response
R2148-9	5 peaks < low std. response
R2148-10	0.082 ppm F, 4 peaks < low std response
R2148-11	1 peak < low std. response
R2148-12	none detected

*same low standard concentration as above

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Column: DB-5

Lab Request #	C496*	H486*	Cl479*	Br478*
R2148-1	1 peak < low std. response	none detected	0.39 ppm	none detected
R2148-2	1 peak < low std. response	none detected	0.46 ppm	none detected
R2148-3	0.43 ppm	none detected	1.2 ppm, 2 peaks < low std. response	none detected
R2148-4	0.39 ppm, 3 peaks < low std. response	1 peak < low std. response	1.0 ppm, 2 peaks < low std. response	none detected
R2148-5	0.32 ppm, 0.42 ppm, 2 peaks < low std. response	0.13 ppm, 2 peaks < low std. response	0.43 ppm, 1.2 ppm, 0.31 ppm	none detected
R2148-6	0.36 ppm, 0.50 ppm, 2 peaks < low std. response	0.078 ppm, 2 peaks < low std. response	0.54 ppm, 1.6 ppm, 0.40 ppm	none detected
R2148-7	0.29 ppm, 2 peaks < low std. response	1 peak < low std. response	0.46 ppm, 2 peaks < low std. response	none detected
R2148-8	2 peaks < low std. response	none detected	0.46 ppm, 2 peaks < low std response	none detected
R2148-9	0.32 ppm, 2 peaks < low std. response	none detected	2 peaks < low std. response	none detected
R2148-10	0.35 ppm, 2 peaks < low std. response	none detected	2 peaks < low std. response	none detected
R2148-11	1 peak < low std. response	none detected	none detected	none detected
R2148-12	1 peak < low std. response	none detected	none detected	none detected

*low standard concentrations: 0.21 ppm C, 0.060 ppm H, 0.28 ppm Cl, 0.24 ppm Br

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2759.0054

Column: DB-624

Lab Request #	C496*	H486*	Cl479*	Br478*
R2148-1	1 peak < low std. response	none detected	0.57 ppm	none detected
R2148-2	1 peak < low std. response	none detected	0.45 ppm	none detected
R2148-3	0.24 ppm, 1 peak < low std. response	none detected	1.2 ppm	none detected
R2148-4	1 peak < low std. response	none detected	1.0 ppm	none detected
R2148-5	0.34 ppm, 3 peaks < low std. response	none detected	0.40 ppm, 1.6 ppm, 0.42 ppm	none detected
R2148-6	0.50 ppm, 3 peaks < low std. response	1 peak < low std. response	0.57 ppm, 2.4 ppm, 0.58 ppm, 1 peak < low std. response	none detected
R2148-7	2 peaks < low std. response	none detected	0.45 ppm, 1 peak < low std. response	none detected
R2148-8	2 peaks < low std. response	none detected	0.66 ppm, 2 peaks < low std. response	none detected
R2148-9	1 peak < low std. response	none detected	3 peaks < low std. response	none detected
R2148-10	1 peak < low std. response	none detected	3 peaks < low std. response	none detected
R2148-11	1 peak < low std. response	none detected	none detected	none detected
R2148-12	1 peak < low std. response	none detected	none detected	none detected

*same low standard concentrations as above

All results were normalized, assuming the density of water = 1g/mL.

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2759.0055

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Q. And prior to the beginning of the deposition, your counsel handed us a copy of that. I'll go ahead and mark that.
(Deposition Exhibit No. 2 was marked for identification.)
Q. Ms. Corrigan, I'm going to hand you what's been marked as Exhibit No. 2, and ask you to look at that and tell me if you can identify that is the document that you just referred to that you found?
A. Yes, it is.
Q. Is this the only document that you identified as being responsive to these requests for production of documents?
A. Yes.
Q. What did you do to look for documents requested here?
A. I looked through my personal files and through any electronic correspondence files. I looked through phone messages. And that's the extent of my search.
Q. With respect to hard copy paper files, at the time that you received this request for you to look for documents, did you have any hard copy paper files that related in any way to

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your prior -- to your employment prior to working for, is it, Koch Industries?
A. Koch Industries. I'm sorry, the question again was?
Q. Did you have any files relating to any of your prior employment before working for Koch Industries?
A. The employment that I had prior to working for Koch Industries was as the Commissioner of the MPCA. And those files, as I left the agency, I left them for my successor as they belonged to the State of Minnesota. Prior to that I worked for 3M in a number of different capacities, and each time I changed roles with 3M, I left the files for those particular roles with the successor for those roles. So the answer to your question is I do not have any files in my possession.
Q. Where did you find the document that's marked at Exhibit 2?
A. In my personal file.
Q. And by your personal file, what kind of information is in your personal file?
A. My personal files contained thank you notes, congratulatory notes on the appointment that

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I had with the agency, this particular letter.
Q. The thank you notes that you just mentioned, were any of those from any employees of 3M?
A. No. Let me be specific. They were thank you notes that were given to me as part of my service with the agency, congratulatory notes as I left, from various groups that I had interacted with.
Q. You mentioned that your files with the MPCA were left with the MPCA when you left, correct?
A. Correct.
Q. Why was Exhibit No. 2, why was a copy of that put in your personal file when you left?
A. I put it in my personal file.
Q. Why?
A. Because I anticipated that with the connection that I had to the MPCA, as well as previous connections to 3M, that potentially there would be a need to have this with me. And it also, as you can read, pertains specifically to me.
Q. You mentioned you also searched through electronic files; is that correct?

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A. Yes.
Q. And do you have a personal computer?
A. I do.
Q. Was your personal computer ever used for sending e-mails relating to your work while at the MPCA?
A. I can't think of an occasion where I did send e-mails from my personal computer, as I had a BlackBerry while I was at the MPCA.
Q. And was your BlackBerry issued by the MPCA?
A. Yes.
Q. And did the messages that you sent or received through that BlackBerry, or from that BlackBerry, get routed through the MPCA's computer system?
A. Yes, they did.
Q. Do you recall ever sending or receiving any e-mail messages relating to your work at MPCA through your home computer?
A. I don't remember.
Q. Do you still possess that home computer that was in your possession while you were working at the MPCA?
A. No, I do not.
Q. When did you purchase the current computer

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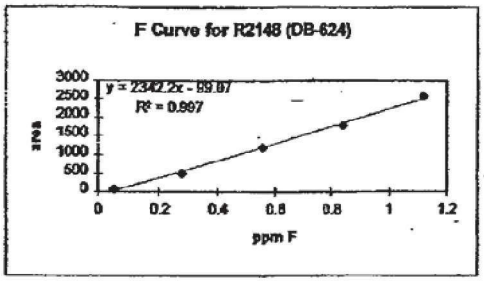
STANDARD CURVES:

Example of a standard curve from the analysis (all standard curves had R^2 of 0.99 or greater):



6.0 CONCLUSIONS

Twelve water samples were analyzed on two different columns with a GC/AED. More fluorine was detected using the DB-624 than the DB-5; in the DB-5 analysis, more carbon, hydrogen, and chlorine were detected. All of the samples evidence of at least one of the elements targeted. Many peaks were detected but not quantitated because the peak area was less than the low standard response for that element.

7.0 MAINTENANCE OF RAW DATA

Copies of all data will be filed in the AMDT archive.

8.0 APPENDICES

Appendix A: Chromatogram of sample 6 (MW-3 2 of 2)
F690
DB-624

SEMiller
110797

3MA00230181

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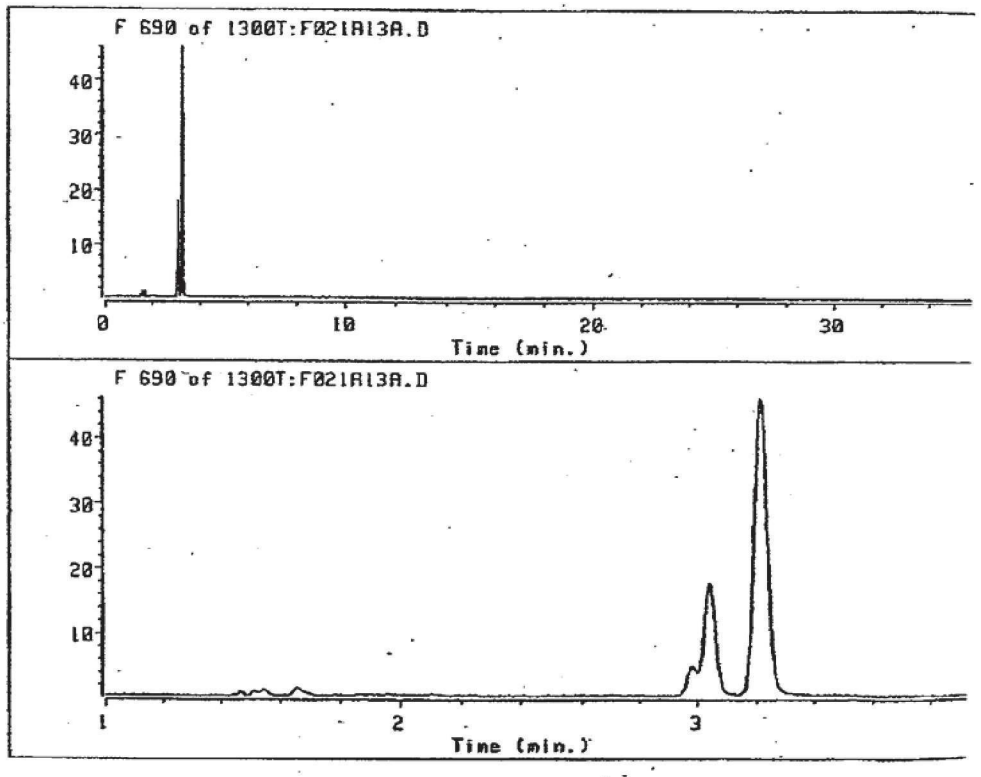
3M_MN00043362

2759.0057

Appendix A: Chromatogram of sample 6 (MW-3 2 of 2)
F690
DB-624



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2759.0058

3M Environmental Laboratory

Total Fluoride in Soil (6/97 Samples)

55 of 125

3MA00230183

3M Environmental Laboratory

Data Transmittal Summary

Preliminary / Final *(circle one)*

Lab Request #:	3M Study #:	Contract Laboratory #
R2382		

Date Received: 06/06/97

Sponsor or Client: Roger Zipfel
Dupont Washington Works Plant

Project Lead: Name / Phone Kris Hansen / (612) 778-6018

Analyte(s) or Test Method #: Total Fluoride
Sample Matrix: Soil
Analysis Dates: 9/29/97 through 10/01/97 Analyst(s): Daniel Howman
Author: Daniel Howman
Data Reviewed by: Kris Hansen

Internal	Sent by: / Date
Reviewer: James D. Johnson	DRH / 11-07-97
QAU (Archives): Rich Youngblom	
LIRN System: Denise Appleton	
Project Manager: Sue Beach	

Others *(List Recipients / Address / Phone / FAX)*	Sent by: / Date
Project Lead: Kris Hansen	DRH / 11-07-97

A copy of the report including this form and the client cover page is to be given to QAU, LIRN and to the Group Leader.

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3MA00230184

3M Environmental Laboratory - Advanced Method Development Team
Kris Hansen - Sr. Analytical Chemist
Advanced Method Development Team
Building 2-3E-09
612-778-6018
kjhansen@mmm.com

Final Report
Determination of Total Fluoride in Soil
Laboratory Request R2382

1.0 SUMMARY

Eleven soil samples from Dupont, Washington Works Plant were submitted to the Environmental Laboratory for analysis of total fluoride. The samples were submitted under Lab Request No. R2382, samples 1 through 11.

The samples were analyzed using an Orion EA 940 Expandable Ion Analyzer after combustion using a Dohrmann DX2000 Organic Halide Analyzer modified for fluoride analysis.

The following table contains a summary of the results. The Total Fluoride values are the average of three replicates of the same sample, and are given in the table along with the standard deviation of the three replicates. Total fluoride is defined as the concentration of F- measured following complete combustion of the sample.

Summary Table:
Soil Samples from Dupont Washington R2382

Fluoride:	Average (mg/Kg):	21300	20100	61,200	78300	108300	82700
	Standard Deviation:	2800	2400	4200	6000	2100	1400
Fluoride:	Average (mg/Kg):	59100	37800	33500	41200	30300	
	Standard Deviation:	4900	3600	1400	2200	1600	

2.0 TEST MATERIALS

The soil samples were sent from Dupont, Washington Works Plant, and received at the Environmental Laboratory in St. Paul on 6/6/97. The samples were logged in under Lab Request R2382, samples 1 through 11. Samples were refrigerated at 4°C until analysis.

Analyst / Date
Daniel Howman / 10-1-97

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3M_MN00043366

2759.0061

3.0 INSTRUMENTATION

A. Dohrmann DX2000 Organic Halide Analyzer modified for fluoride analysis
   OPERATING CONDITIONS
   Combustion tube temperature = 950° C
   Oxygen and Helium flow = 50 cc/minute
   Vaporization/Drying time = 240 seconds
   Bake time = 300 seconds
   Collection fluid = 3.0 mL of 1:1 TISAB/Milli-Q H_2O
B. Orion EA940 Expandable Ion Analyzer with Orion 9609BN Combination Fluoride Electrode

4.0 EXPERIMENTAL OVERVIEW: Total Fluoride Determination

4.1 Standards

A standard curve was prepared from Amonium Perfluorooctanoate (POAA) stock solution (S397-420) at the following concentrations: 25, 50, 250, 500, 1000 ppm POAA in MeOH. For each sample, 0.2mL of soil was extracted thermally with the Dohrmann DX2000 Organic Halide Analyzer. The EOX-Liquids computer program was used for the standard extraction. Standards were prepared and analyzed in triplicate. The extraction products of the standards were collected in 3 mL of 1:1 TISAB II/H_2O. The collection vial was placed so that the tip of the combustion tube was in the collection fluid. Gases released during pyrolysis bubble through the collection fluid; the F- partitions into the collection fluid.

The concentration of fluoride in the collection vial was determined by direct measurement with the Orion EA940 Expandable Ion Analyzer with Orion 9609BN Combination Fluoride Electrode. The Orion EA940 was calibrated by direct measurement with no blank correction, using standards with a concentration of 0.1, 0.5, 1.0, 1.5, 5.0 ppm F. Standards were prepared using Corning Sodium Fluoride (TN-A-0572) and diluted in 1:1 TISAB II/H_2O.

4.2 Blanks

Prior to analysis of the samples and standards, 0.1mL of Milli-Q was extracted on the Dohrmann DX2000 Halide Analyzer in the same way as the standards to insure the system was free of any fluoride contamination. Total fluoride was then measured on the Orion EA940.

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3M_MN00043367

2759.0062

4.3 Samples

For sample analysis, 0.02 gram samples of soil were extracted in triplicate on the Dohrmann DX-2000 in the same way as the standards. The EOX-Solids computer program was used for sample extraction. The concentration of fluoride extracted was determined by direct measurement with the Orion EA940.

5.0 DATA ANALYSIS: Total Fluoride determination

5.1 Standards

A standard curve was developed using the POAA standard solutions (see appendix). The fluoride content of POAA is 66.10%, thus, the concentration of fluoride in the standards was determined by multiplying the concentration of POAA by 66.10%.

Concentration of F- = (Standard concentration) * (0.661)
Concentration of F- = (25ppm POAA) * (0.661)
Concentration of F- in 25ppm POAA = 16.5ppm

These calculated values were plotted and a standard curve calculated using linear regression. The equation of the regression is y = 0.0067x - 0.1051. A linear correlation coefficient of 0.9962 was obtained for the standard range of 25 - 1000 ppm APO.

5.2 Blanks

No further analysis was done on the blanks.

5.3 Samples

The Total Fluoride in the samples is reported as the average of triplicate sample analysis using the linear regression equation to correct for extraction efficiency (see appendix).

Calculated F- (mg/L) = (Meter Reading + Intercept) / Slope
Calculated F- (mg/L) = (Meter Reading of R2382-1-1 + 0.1061) / (0.0067)
Calculated F- (mg/L) = (0.8047 + 0.1061) / (0.0067)
Calculated F- of R2382-1-1 = 136mg/L

Calculated F- (mg/Kg) = (Calculated F- (mg/L)) *
(Collection Volume) / (Sample Weight)
Calculated F- (mg/Kg) = (136mg/L) * (3ml) / (0.0203grams)
Calculated F- (mg/Kg) = 20100mg/Kg

Analysis blanks and calibration check standards were analyzed periodically to verify that the system continued to operate properly.

Analyst / Date
Daniel Howman / 10-1-97

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3M_MN00043368

2759.0063

6.0 CONCLUSIONS

Triplicate analysis of the pyrolysis products of R2382-1 to R2382-11 determined that fluorine is present in all samples. Total fluoride concentration varies from 20100ppm to 106,300ppm; data are summarized in the Summary Table (section 1.0).

The highest levels of fluoride were found in R2382-5 and R2382-6 and the lowest levels in samples R2382-1 and R2382-2.

Analyst / Date
Daniel Howman / 10-1-97

3MA00230188

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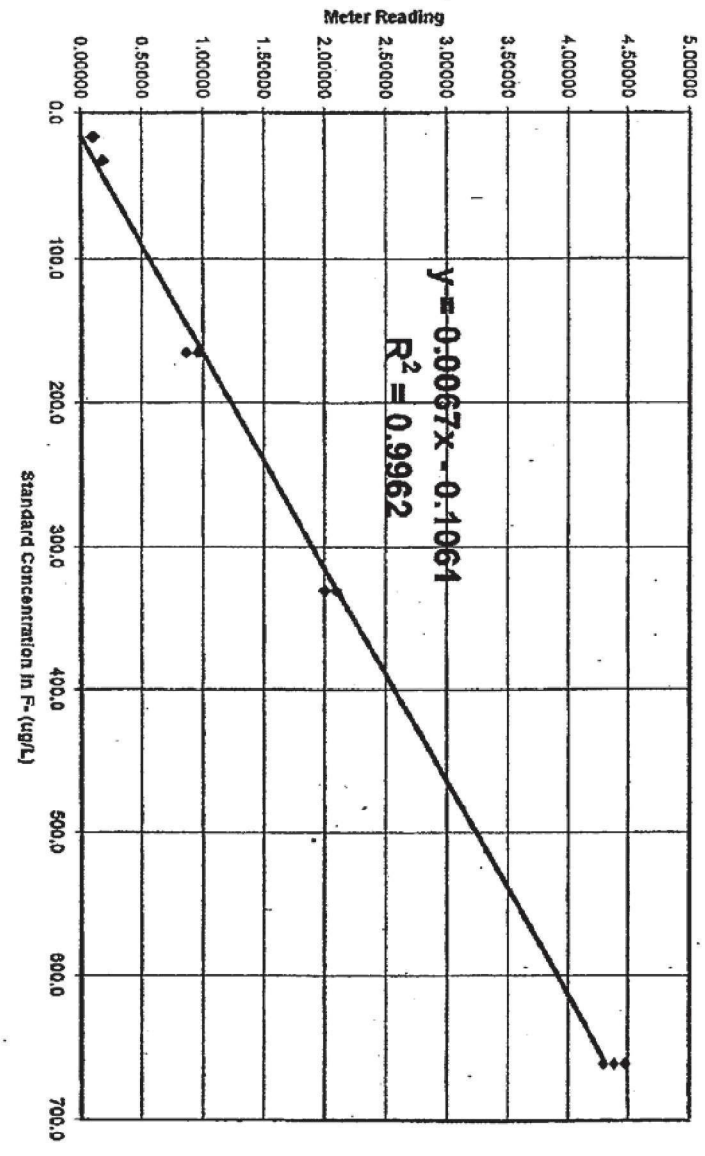
3M_MN00043369

2759.0064

Analyst / Date
Daniel Howman / 10-1-97



Standard Curve POAA

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3MA00230189

Project: R2382

Dupont Washington Soil Samples

Calculations for Standard Curve

Calculated F- (mg/L) = Meter Reading * Collection Volume / Sample Volume

Sample ID	Dilution	Sample Volume (mL)	Collection Volume (mL)	Calculated F- (mg/L)	Standard F- Concentration (mg/L)	Meter Reading	% Rec.	Comments
Analysis: DRH 9-29-97								
QC 4.33PPM ERA CHECK	2					2.062	95.25	
BLANK-1	1	0.01	3.0			0.05853		
BLANK-2	1	0.01	3.0			0.03691		
BLANK-3	1	0.01	3.0			0.02618		
25PPM W397-919	1	0.01	3.0	18.3	16.5	0.08097	110.69	
25PPM W397-919	1	0.01	3.0	16.5	16.5	0.05506	99.96	
25PPM W397-919	1	0.01	3.0	12.8	16.5	0.04256	77.26	
25PPM W397-919	1	0.01	3.0	13.9	16.5	0.04638	84.20	
QC 9.0PPM CHECK	1	0.01	3.0			0.9585	86.85	
50PPM W397-920	1	0.01	3.0	36.5	33.1	0.1016	92.22	
50PPM W397-920	1	0.01	3.0	28.2	33.1	0.08735	79.29	Due to low meter readings and low recovery, decided to
50PPM W397-920	1	0.01	3.0	21.7	33.1	0.07239	65.71	
250PPM W397-921	1	0.01	3.0	111.7	165.3	0.3724	67.51	rerun using 20uL as
250PPM W397-921	1	0.01	3.0	128.4	165.3	0.4279	77.58	sample volumes/fl
QC 1.0PPM CHECK	1					0.9845	98.45	
BLANK-1	1	0.02	3.0			0.07984		
BLANK-2	1	0.02	3.0			0.02783		
BLANK-3	1	0.02	3.0			0.02694		
25PPM W397-919	1	0.02	3.0	13.7	16.5	0.09136	82.93	
25PPM W397-919	1	0.02	3.0	17.2	16.5	0.1149	104.25	
25PPM W397-919	1	0.02	3.0	16.0	16.5	0.1067	96.86	
50PPM W397-920	1	0.02	3.0	26.5	33.1	0.1764	80.04	
50PPM W397-920	1	0.02	3.0	28.6	33.1	0.1906	86.50	
50PPM W397-920	1	0.02	3.0	26.5	33.1	0.1764	80.04	
QC 1.0PPM CHECK	1					0.9725	97.25	
250PPM W397-921	1	0.02	3.0	130.8	165.3	0.8710	79.06	
250PPM W397-921	1	0.02	3.0	146.5	165.3	0.9755	88.64	
250PPM W397-921	1	0.02	3.0	144.1	165.3	0.9607	87.20	
500PPM W397-922	1	0.02	3.0	301.8	330.5	2.012	91.32	
500PPM W397-922	1	0.02	3.0	300.6	330.5	2.004	90.96	
500PPM W397-922	1	0.02	3.0	314.4	330.5	2.096	95.13	
1000PPM W397-923	1	0.02	3.0	645.2	661.0	4.301	97.61	
1000PPM W397-923	1	0.02	3.0	672.1	661.0	4.481	101.68	
1000PPM W397-923	1	0.02	3.0	658.5	661.0	4.390	99.63	
QC 1.0PPM CHECK	1					0.9648	96.46	
Notebook Reference: Carlton-SA-2, p.20-21								

Analyst / Date
Daniel Howman / 10-1-97

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you're using now? Well, let me restate the question.
How many computers do you personally have now?
A. Two.
Q. All right. When did you purchase those computers?
A. We have a laptop computer that we purchased in late 2007, I recall, I believe it was in the October time frame, but I could be wrong, give or take a month on that one. And we have a regular computer that we purchased in August of 2006 when we moved to Wichita.
Q. And did you have any personal computer prior to August of 2006?
A. Yes, we did.
Q. And what happened to that computer?
A. We got rid of it.
Q. What did you do to search your current computers for any information that might be responsive to the request for information in Exhibit 1?
A. I looked through the electronic files that we would have, in particular any e-mail. I looked through documents, but my

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communications with MPCA relative to personal matters are friendship based, and anything else, and so those e-mails that I would have received from folks would have been deleted immediately.
Q. You also mentioned phone messages when I asked you what kind of information you had reviewed, correct?
A. Uh-huh.
Q. Again, just make sure you verbalize your response.
A. Yes, yes. I'm sorry.
Q. Were you referring to messages on your home phone or someplace else?
A. My home phone, yes.
Q. And do you save those messages?
A. Not typically.
Q. Are there messages saved on your home phone?
A. Not as a common practice.
Q. So what did you do to review your phone messages?
A. I checked our phone messages.
Q. Okay. And there was nothing on there relating in any way to anything requested in the request for production in Exhibit 1,

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correct?
A. Correct.
Q. I'd like to refer you to Page 7 of Exhibit 1 for a moment, and particularly Paragraph 5 where there is a request for, quote, any and all documents, communications and slash or ESI in your possession, custody or control relating in any way to any compensation or other financial benefit received, accrued or earned. And then parens, whether or not received, close parens, by deponent from 3M since the cessation of deponent's formal employment relationship with 3M, close quote. Do you see that?
A. Uh-huh, yes, I do.
Q. And I think you mentioned earlier that you had worked at one point for the 3M Company, correct?
A. That's correct.
Q. And did you, during that employment, acquire any stock in the 3M Company?
A. Yes, I did.
Q. And do you still own stock in the 3M Company?
A. Yes, I do.
Q. Do you have any documents relating to the

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value of that stock or any dividends from that stock?
A. I do.
Q. Looking at Paragraph 5, did you have some belief that those documents were not related to Paragraph 5?
A. Let me give you my thinking on this, and certainly let me be open about this.
The stock that I own from 3M came from two separate sources. One was a gift that I received from my parents 20 some years ago when I was married. I've never made a trade on that stock since I received it.
Q. Okay.
A. The other source of 3M stock is contained in my 401(k) account, which still resides within the 3M benefits organization. Again, I've never made a trade on that, nor have I manipulated that stock. I attempted last night, and I apologize for this, to download a statement from my 401(k), but it has been so long since I had been to that account, I forgot my password, and I locked my account. I can't receive a password except via mail, which they promised to do within two business

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Project: R2382
Soil Samples from Dupont Washington

Calculations for Total Fluoride in Soil

Calculated F- (mg/L) = (Meter Reading + Intercept) / Slope

Calculated F- (mg/Kg) = Calculated F- (mg/L) * Collection Volume / Sample Weight

Sample ID	Dilution	Sample Weight (g)	Collection Volume (mL)	Meter Reading	Calculated F- (mg/L)	Calculated F- (mg/Kg)	Statistics (mg/Kg)		% Recovery	Comments
Analysis: DRH 9-30-97										
QC 4.33PPM ERA CHECK	2			2.079					96.0	
BLANK-1	1		3.0	0.07719						
BLANK-2	1		3.0	0.05811						
BLANK-3	1		3.0	0.05165						
R2382-1-1		0.0203	3.0	0.8047	136	20,100	AVE	21300		
R2382-1-2		0.0231	3.0	0.8870	148	19,200	STD	2800		
R2382-1-3		0.023	3.0	1.155	188	24,500	CV	13		
R2382-2-1		0.0236	3.0	0.8080	136	17,300	AVE	20100		
R2382-2-2		0.0215	3.0	0.9183	153	21,300	STD	2400		
R2382-2-3		0.0219	3.0	0.9542	158	21,700	CV	12		
QC 1.0PPM CHECK	1			0.9698					97.0	
R2382-3-1		0.0204	3.0	2.485	387	56,900	AVE	61200		
R2382-3-2		0.0225	3.0	2.983	461	61,500	STD	4200		
R2382-3-3		0.0202	3.0	2.841	440	65,300	CV	7		
R2382-4-1		0.0235	3.0	3.668	563	71,900	AVE	78300		
R2382-4-2		0.0227	3.0	3.898	596	79,000	STD	6000		
R2382-4-3		0.0225	3.0	4.109	629	83,900	CV	8		
R2382-4-4 w/10mL spike of 18,000ppm PCAA		0.023	3.0	8.189	940	122,600			51.4	
R2382-5-1		0.0228	3.0	5.349	814	107,100	AVE	105300		
R2382-5-2		0.023	3.0	5.406	827	107,900	STD	2100		
R2382-5-3		0.0226	3.0	5.136	782	103,900	CV	2		
QC 1.0PPM CHECK	1			0.9856					98.6	
R2382-6-1		0.021	3.0	3.835	588	84,000	AVE	82700		
R2382-6-2		0.021	3.0	3.789	581	83,000	STD	1400		
R2382-6-3		0.0214	3.0	3.774	579	81,200	CV	2		
R2382-7-1		0.0218	3.0	2.963	461	63,400	AVE	59100		
R2382-7-2		0.0201	3.0	2.588	402	60,000	STD	4900		
R2382-7-3		0.021	3.0	2.415	376	53,800	CV	8		
R2382-8-1		0.022	3.0	1.811	286	39,000	AVE	37600		
R2382-8-2		0.0216	3.0	1.841	291	40,400	STD	3600		
R2382-8-3		0.0215	3.0	1.503	240	33,500	CV	10		
QC 1.0PPM CHECK	1			0.9777					97.8	
R2382-9-1		0.0209	3.0	1.465	237	34,100	AVE	33500		
R2382-9-2		0.021	3.0	1.509	241	34,400	STD	1400		
R2382-9-3		0.0203	3.0	1.342	216	31,900	CV	4		
QC 1.0PPM CHECK	1			0.9737					97.4	
Analysis: DRH 10-1-97										
QC 4.33PPM ERA CHECK	2			2.019					93.3	
BLANK-1	1		3.0	0.1319						
BLANK-2	1		3.0	0.08430						
BLANK-3	1		3.0	0.05910						
R2382-10-1		0.0203	3.0	1.665	264	39,100	AVE	41200		
R2382-10-2		0.0201	3.0	1.741	276	41,100	STD	2200		
R2382-10-3		0.0208	3.0	1.909	301	43,400	CV	5		
R2382-11-1		0.0218	3.0	1.288	203	28,500	AVE	30300		
R2382-11-2		0.0223	3.0	1.471	235	31,700	STD	1600		
R2382-11-3		0.022	3.0	1.402	225	30,700	CV	5		
QC 1.0PPM CHECK	1			0.9470					94.2	
Notebook Reference: Carbon-SA-2, p.21-23										

Analyst / Date
Daniel Howman / 10-1-97

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3MA00230191

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3M Environmental Laboratory

Total, Organic and Adsorbable Fluoride in Groundwater (6/97 Samples)

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3MA00230192

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3M Environmental Laboratory

Data Transmittal Summary

Preliminary / Final *(circle one)*

Lab Request #:	3M Study #:	Contract Laboratory #
R-2148	Bigenenvir	

Date Received:

Sponsor or Client: Representative Name Dale Bacon/ Robert Howell
Company Name 3M
Company Address 935 Bush Ave. , St. Paul, MN
Phone 778-4736 / 778-7540

Project Lead: Name / Phone Kris Hansen
Group Leader: Name / Phone 612-778-6018

Analyte(s) or Test Method #: Total Fluorine, Fluoride Ion, Adsorbable Organic Fluoride
Sample Matrix: Dupont Waters
Analysis Dates: 8/15/97-8/25/97 Analyst(s): Jan Schutz , Nancy Bergman
Author: Jan Schutz

Data Reviewed by:
Project Lead (or designee): Pat Rethwill 10/03/97
James D. Johnson (or designee):

Internal	Sent by: / Date
JDJ: Kris Hansen	JGS /11/07/97
QAU (Archives): Rich Youngblom	JGS /11/07/97
LIRN System: Denise Appleton	JGS /11/07/97
Project Manager: Sue Beach	JGS /11/07/97

Others *(List Recipients / Address / Phone / FAX)*	Sent by: / Date

A copy of the report including this form and the client cover page is to be given to QAU, LIRN and to the Group Leader.

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3MA00230193

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3M Environmental Laboratory - Advanced Method Development Team

Contact: Kris Hansen - Sr. Analytical Chemist
Building 02-3E-09
778-6018
kjhansen @ mmm.com

Final Report - Lab Request R2148
Total Fluoride Analysis - DuPont
30 September 1997

1.0 SUMMARY

Twelve water samples from DuPont were submitted to the 3M Environmental Laboratory for organic fluorine analysis. The samples were submitted under Lab Request R2148, samples 1 through 12. Samples were tested for fluoride ions, total fluorine, and adsorbable organic fluorine (AOF). A modified version of DIN method 38 402 H29 was used to measure the AOF.

The following table contains a summary of the results.

Sample ID	Sample Request	Fluoride Ion (µg/mL F-)	Total Fluorine (µg/mL F-)	Total Fluorine - Fluoride Ion (µg/mL)	Adsorbable Organic Fluorine (µg/mL F-)
MW-1-1	R2148-1	0.20	8.0	7.8	4.5
MW-1-2	R2148-2	0.20	16	16	4.2
MW-2-1	R2148-3	0.16	3.3	3.1	0.28
MW-2-2	R2148-4	0.16	3.5	3.3	0.46
MW-3-1	R2148-5	0.14	4.2	4.1	1.1
MW-3-2	R2148-6	0.14	3.3	3.2	0.76
MW-4-1	R2148-7	0.11	4.0	3.9	0.19
MW-4-2	R2148-8	0.11	4.2	4.1	0.14
MW-5-1	R2148-9	<0.10	3.0	2.9	0.11
MW-5-2	R2148-10	<0.10	2.8	2.7	0.14
MW-6-1	R2148-11	0.10	2.3	2.2	<0.05
MW-6-2	R2148-12	0.10	4.2	4.1	<0.05

2.0 INTRODUCTION

A request was made of the 3M Environmental Laboratory to determine the amount of fluoride and organic fluorine in twelve monitoring well water samples, using a modified Dohrmann Organic Halide Analyzer and the Orion EA940 Meter with a fluoride specific electrode.

3.0 TEST MATERIALS

Twelve monitoring well water samples were received from DuPont. The samples were labeled as having been collected on 6/26/97. The samples were logged in as Lab

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Request R2148, samples 1 through 12. Samples were kept refrigerated until they were analyzed.

A standard curve was prepared, from an ammonium perfluorooctanoate (POAA) standard (S397-386) for total fluoride plus organic fluorine analysis and from an ammonium perfluorooctanoate (POAA) standard (S397-383) for adsorbable organic fluorine. The Orion meter was calibrated daily with standards prepared from a Corning fluoride stock standard in 50% TISAB II/50% Milli-Q water

4.0 EXPERIMENTAL - OVERVIEW

4.1 Fluoride Ion Analysis

This analysis measured the amount of fluoride ion in the sample without combusting the sample. For measurement of fluoride ion, an Orion EA940 meter was calibrated daily, using Corning standards over the range of 0.05 - 1.5 ppm fluoride. One milliliter of unfiltered sample was diluted with one milliliter of TISAB II and analyzed on the Orion meter. A mid-range calibration standard was analyzed periodically to verify that the system continued to operate properly.

4.2 Adsorbable Organic Fluorine (AOF)

This analysis measures the amount of adsorbable organic fluorine as fluoride in the sample by passing the filtered sample through two carbon columns and then combusting the carbon. For measurement of AOF, a modified version of the German wastewater analysis method, DIN method 38 402 H29 (column method), was followed. The POAA standard curve and samples were prepared by running 100.0 mL of standard or sample (or an aliquot of sample diluted to 100 mL) through two carbon columns using a Dohrmann AD-2000 Adsorption Module. The carbon columns were burned in a modified Dohrmann DX2000 Organic Halide Analyzer, collecting the off-gases in 3.0 mL of 1:1 TISAB II/Milli-Q water and analyzing on an Orion EA940 meter with a fluoride specific electrode. The Orion meter calibrated from 0.5 - 25.0 ppm fluoride.

4.3 Total Fluorine Analysis

This analysis measured the amount of total fluorine by combusting an aliquot of unfiltered sample, collecting the off-gasses, and analyzing the collection solution for fluoride. For this measurement a modified Dohrmann Organic Halide Analyzer and an Orion EA940 meter (calibrated from 0.1 - 5.0 ppm F-) were used. An POAA standard curve was generated by burning 0.10 mL standard and collecting the off-gasses in 3.0 mL 1:1 TISAB II/Milli-Q water for analysis on the Orion EA940 meter. The water samples were analyzed following the same method as the standards.

INSTRUMENTATION

Dohrmann DX2000 Organic Halide Analyzer modified for fluoride analysis
Dohrmann Adsorption Module AD2000

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INSTRUMENTATION

Orion EA940 Expandable Ion Analyzer with Orion 9609BN Combination Fluoride Electrode
DX2000 software, version 1.00, modified for fluoride extraction
DX2000 software, version 2.00, modified for fluoride extraction (AOF analysis)
Microsoft Excel

OPERATING CONDITIONS

Combustion tube temperature = 950° C
Oxygen and Helium flow = 50 cc/minute
VPOAArization/Drying time = 240 seconds
Bake time = 300 seconds
Collection fluid = 3.0 mL 1:1 TISAB II/Milli-Q water

REAGENTS

Fluoride Standard 100 ppm, purchased from Corning (part #478170, lot #1113022)
Total Ionic Strength Adjustment Buffer (TISAB II), Orion (part #940909, lot AR1)

5.0 DATA ANALYSIS

The Orion Meter, serial # 4202, was calibrated each morning prior to any samples being run. Calibration was based on direct measurement of calibration standards made from Corning fluoride stock standard. An acceptable correlation coefficient is ≥ 0.9950.

date	analysis done	correlation coefficient (R^2)
18 - 21 August 1997	Total Fluorine Analysis / POAA standard curve	≥0.9993
18 August 1997	Fluoride ion analysis	≥0.9998
15 - 25 August 1997	AOF Analysis / POAA standard curve	≥0.9999

A standard curve for total fluorine was generated by combusting 0.1 mL aliquots of 2.0, 5.0, 20, 50, and 100 µg/mL POAA standard (POAA is 66.1% fluoride) in the Dohrmann DX2000 Modified Organic Halide Analyzer. The off-gasses were collected in 3 mL of 1:1TISAB II/ Milli-Q water and analyzed with the Orion meter. Using least squares linear regression, plotting the fluoride concentration of the standard on the x-axis, and the Orion meter response on the y-axis, the following curve was generated: $Y = 0.0341x\ 0.0157$ and $R^2 = 0.9988$.

A standard curve for adsorbable organic fluorine (AOF) was generated by pushing 100 mLs each of 5 standards containing 4.9, 14.4, 23.6, 33.5, and 43.1 µg/mL fluorine as POAA through two carbon columns. The carbon columns were combusted in the Modified Dohrmann DX2000 Organic Halide Analyzer. The effluent was collected in 3 mL of 1:1 TISAB II / Milli-Q water and analyzed with the Orion meter. Using least squares linear regression, plotting the fluoride concentration of the standard on the x axis, and the Orion meter response on the y axis, the following curve was generated: $Y = 0.2894x + 0.0196$ and $R^2 = 0.9949$.

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3M_MN00043378

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SAMPLE ANALYSIS:

Fluoride Ion Analysis

Sample #	Meter Reading	Dilution Factor	Quantity of Sample (mL)	Fluoride Ion in Sample (µg/mL)
R2148-1	0.1005	2	1.0	0.20
R2148-2	0.1017	2	1.0	0.20
R2148-3	0.0813	2	1.0	0.16
R2148-4	0.0801	2	1.0	0.16
R2148-5	0.0701	2	1.0	0.14
R2148-6	0.0696	2	1.0	0.14
R2148-7	0.0567	2	1.0	0.11
R2148-8	0.0556	2	1.0	0.11
R2148-9	0.0465	2	1.0	<0.10 *
R2148-10	0.0463	2	1.0	<0.10 *
R2148-11	0.0491	2	1.0	<0.10 *
R2148-12	0.0497	2	1.0	<0.10 *

*Method detection limit (MDL) = 0.100 ppm (lowest calibration standard x dilution factor)

AOF Standard Curve
Total Ammonium Perfluorooctanoate Standard in Columns (top + bottom)

Sample ID *		Quantity Sample (mL)	Combined Orion Meter Reading (µg/mL F-)	Spiked µg/mL F- in Sample *
POAA Standard 1	0.072 ppm	100	1.62	0.05
POAA Standard 2	0.217 ppm	100	4.18	0.14
POAA Standard 3	0.362 ppm	100	6.75	0.24
POAA Standard 4	0.507 ppm	100	9.31	0.34
POAA Standard 5	0.652 ppm	100	12.9	0.43

*POAA Standard is 66.1% Fluoride $Y = 0.2894x + 0.0196$ $R^2 = 0.9949$

AOF Sample Analysis

Sample ID	Combined Meter Reading	Quantity of Sample (mL)	Adsorbable Organic Fluorine in Sample (µg/mL)
R2148-1 (top + bottom)	6.544	5.0	4.5
R2148-2 (top + bottom)	6.063	5.0	4.2
R2148-3 (top + bottom)	6.776	85.0	0.28
R2148-4 (top + bottom)	11.30	85.0	0.46
R2148-5 (top + bottom)	3.235	10.0	1.1
R2148-6 (top + bottom)	2.229	10.0	0.76
R2148-7 (top + bottom)	2.696	50.0	0.19
R2148-8 (top + bottom)	2.024	50.0	0.14
R2148-9 (top + bottom)	1.556	50.0	0.11
R2148-10 (top + bottom)	1.937	50.0	0.13
R2148-11 (top + bottom)	0.537	100.0	<0.05*
R2148-12 (top + bottom)	0.536	100.0	<0.05*

*MDL = 0.05 µg/mL.

3M Environmental Laboratory, Lab Request R2148

3MA00230197

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3M_MN00043379

2759.0074

Total Fluorine Standard Curve
Ammonium Perfluorooctanoate in Milli-Q water

Sample ID	Quantity Sample (mL)	Orion Meter Reading (µg/mL F-)[1]	Spiked µg/mL F- in Sample *
POAA standard 1 - 2.00 ppm	0.100	0.048	1.32
POAA standard 2 - 5.00 ppm	0.100	0.100	3.31
POAA standard 3 - 20.0 ppm	0.100	0.421	13.2
POAA standard 4 - 50.0 ppm	0.100	1.089	33.1
POAA standard 5 - 100 ppm	0.100	2.248	66.1

[1]Based on the average of three replicates. $Y = 0.0341x - 0.0157$ $R^2 = 0.9988$
*ammonium perfluorooctanoate standard is 66.1 % Fluoride

Total Fluorine Sample Analysis

Sample ID		Meter * Reading	Quantity of Sample (mL)	Total µg/mL * F- in Sample
MW-1-1	R2148-1	0.2568	0.1	8.0
MW-1-2	R2148-2	0.5217	0.1	16
MW-2-1	R2148-3	0.0964	0.1	3.3
MW-2-2	R2148-4	0.1051	0.1	3.5
MW-3-1	R2148-5	0.1279	0.1	4.2
MW-3-2	R2148-6	0.0963	0.1	3.3
MW-4-1	R2148-7	0.1199	0.1	4.0
MW-4-2	R2148-8	0.1260	0.1	4.2
MW-5-1	R2148-9	0.0960	0.1	3.0
MW-5-2	R2148-10	0.0802	0.1	2.8
MW-6-1	R2148-11	0.0636	0.1	2.3
MW-6-2	R2148-12	0.1286	0.1	4.2

* Based on average of three replicates.

6.0 CONCLUSION

Fluoride ion concentrations range from <0.10 to 0.20 µg/mL, <0.05 to 4.5 µg/mL F- for adsorbable organic fluorine analysis, and 2.3 to 16 µg/mL F- for total fluorine analysis. The large difference in concentration of total fluorine for samples, R2148-1 and R2148-2 (duplicate samples) may have been caused by differences in the amount of solid particulate matter in the sample aliquots. The results for the adsorbable organic fluorine analysis may be lower than expected, due to the fact that the solid particulate matter was filtered from the AOF samples, and some polymers may not adsorb on to the charcoal columns.

7.0 MAINTENANCE OF RAW DATA

Hard copies of the data are filed in the AMDT archive.

JG Schultz
09-30-97

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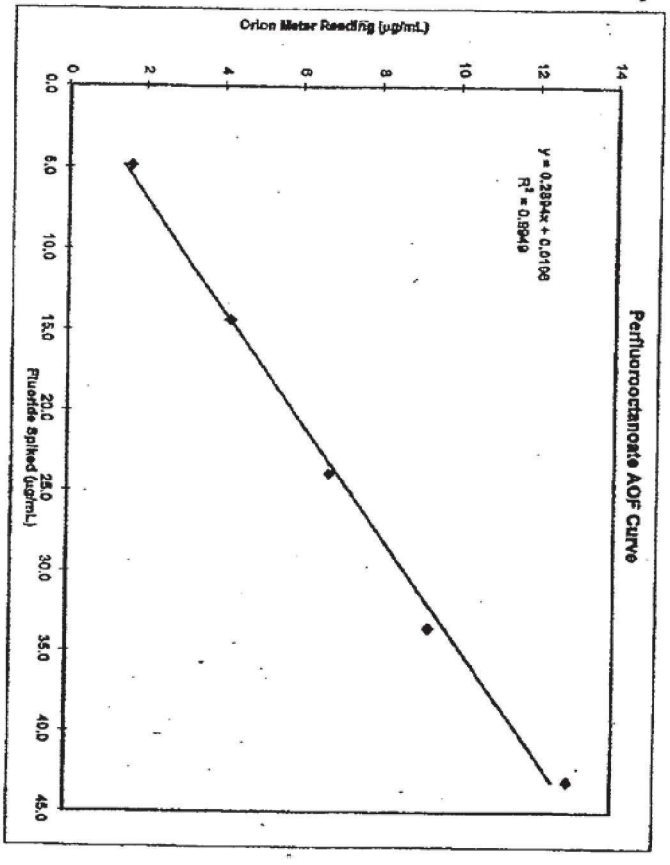
3M_MN00043380

2759.0075

F- Conc. in Standard	Meter Reading
4.9	1.8156
14.4	4.1827
23.9	6.7477
33.5	9.3122
43.1	12.8972



Curve

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3MA00230199

Orion Data

DATA FOR AMMONIUM PERFLUOROOCTANOATE AOF STANDARD CURVE

Sample ID	Actual Meter reading (ppm F-)	Collect Vol (mL)	Dilution Factor	Sample Volume (mL)	Calc AOF of column	Total of Orion read top&bottom	Calc. TOTAL AOF (Top & Bottom)	F- Conc. In Standard (µg/mL)	% Recovery
blank bottom	0.1522	3.0	1	1	0.4565				
Std # 1 bottom	0.4536	3.0	1	100	0.01361				
Std # 2 bottom	0.3701	3.0	1	100	0.01110				
Std # 3 bottom	0.3643	3.0	1	100	0.0109293				
Std # 4 bottom	0.1815	3.0	1	100	0.00544				
Std # 5 bottom	0.1232	3.0	1	100	0.00370				
blank top	0.0544	3.0	1	100	0.00163				
Std # 1 top	1.1620	3.0	1	100	0.03486	1.516	0.048	0.049	100%
Std # 2 top	3.8128	3.0	1	100	0.1144	4.183	0.125	0.144	87%
Std # 3 top	6.3834	3.0	1	100	0.1915	8.748	0.202	0.239	85%
Std # 4 top	9.1307	3.0	1	100	0.2739	9.312	0.278	0.335	83%
Std # 5 top	12.784	3.0	1	100	0.3828	12.887	0.387	0.431	90%
QC check 5.0	5.098	1.0	1	1	5.098				

FLUORIDE ION ANALYSIS

Total Fluoride

Sample ID	Meter Read	TISAB Vol	Dilution	Sample Vol	(µg/mL)		
QC check 0.50 pp	0.5018	1.0	1	1.0	0.502		100%
R2148-1	0.1005	1.0	2	1.0	0.201		
R2148-2	0.1017	1.0	2	1.0	0.203		
R2148-3	0.0813	1.0	2	1.0	0.163		
R2148-4	0.0801	1.0	2	1.0	0.160		
R2148-5	0.0701	1.0	2	1.0	0.140		
R2148-6	0.0696	1.0	2	1.0	0.139		
R2148-7	0.0567	1.0	2	1.0	0.113		
R2148-8	0.0556	1.0	2	1.0	0.111		
R2148-9	0.0465	1.0	2	1.0	0.093		
R2148-10	0.0463	1.0	2	1.0	0.093		
R2148-11	0.0461	1.0	2	1.0	0.098		
R2148-12	0.0487	1.0	2	1.0	0.099		
QC check 0.50 pp	0.5039	1.0	1	1.0	0.504		101%

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R2148.xls

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3MA00230200

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days. I am happy to provide any statement from that 401(k), if you would like.
Q. Okay.
A. I in no way meant to not be responsive and explained to my counsel this morning my dilemma.
Q. Okay. We would request a copy of that when it's received.
So I think you mentioned there's two different categories of stock.
A. That's right.
Q. One would be some stock you received from your parents about 20 years ago, correct?
A. Correct.
Q. And do you have an understanding as to what the current value of that it is?
A. It obviously has varied, but it's between 20 and $30,000.
Q. And the 401(k) account, is that -- do you have a current understanding as to what the current value, approximate, of that account is?
A. My -- I recall seeing a statement from about six months ago. It was a little over, if I recall correctly, a little over $100,000, of

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which a percentage is 3M stock and the rest is other investments and mutual funds and so forth.
Q. Do you know what percentage?
A. I do not.
Q. Do you have an approximate idea of what the value of the 3M portion is?
A. Less than 40 percent, I would think. But, again, I'm unsure of that.
Q. Okay. On Page 10 of Exhibit 1, Paragraph 20, it refers to any documents you might have relating to litigation or governmental investigations or inquiries relating or referring to any PFC. Do you see that?
A. Yes, I do.
Q. And is it correct that you have no documents that relate in any way to any investigations by any governmental agencies since you left the MPCA relating to PFC's?
A. That is correct.
Q. All right. Have you ever been interviewed by any representative of the United States Department of Justice relating to anything to do with 3M or PFC's?
A. No, I have not.

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Q. Have you been interviewed by any representative of the Minnesota Attorney General's Office on anything relating to PFC's or 3M?
A. No, I have not.
Q. Okay. And you mentioned that for purposes of your deposition today you're represented by Mr. Anderson, correct?
MR. ARMSTRONG: Armstrong.
BY MR. BILOTT:
Q. I'm sorry. Armstrong, correct?
MR. ARMSTRONG: Happens all the time.
A. That is correct.
Q. And did you have any meetings with him to prepare for your deposition?
A. Yes, I did.
Q. How many meetings?
A. One.
Q. When was that?
A. Monday of this week, February 25th.
Q. How long did that meeting last?
A. Approximately two hours.
Q. Were you shown any documents of any kind during that meeting?

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A. I was not.
Q. Did you bring any documents with you during that meeting?
A. I did not.
Q. Was anyone else present during the meeting?
A. No.
Q. Was anybody participating by phone, other than the two of you?
A. No.
Q. Okay. Who is paying for your counsel's time today?
A. I am.
Q. Do you anticipate making any request that any other entity reimburse you for whatever you pay for your counsel's time?
A. I have not thought through that.
Q. After receiving the request for your deposition in this case, did you have any discussions with anyone at your current employer about the deposition?
A. I did. I notified my supervisor and told him that I would be involved in a deposition. And I also contacted our general counsel and gave him the same message.
Q. And did you have any discussion with them,

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Orion Data

AOF ANALYSIS — **AOF Calc.** (µg/mL) = ((total meter reading - intercept)/ slope) / sample volume

Sample ID	Meter Reading	Collect Vol (mL)	Dilution Factor	Sample Vol (mL)	Meter Reading Total (top&bottom)	Calc. Adsorbable Organic Fluorine (µg/mL)	Comments	y=0.2894+0.0196 % Recovery
QC check 5.0 ppm	5.039	1.0	1	1				101%
AOF instr blank Bottom	0.0280	3.0	1	1				
R2148-1 bottom	0.3019	3.0	1	85				
R2148-2 bottom	0.2972	3.0	1	70				
R2148-3 bottom	0.1222	3.0	1	85				
R2148-4 bottom	0.1358	3.0	1	85				
R2148-5 bottom	0.2881	3.0	1	90				
R2148-6 bottom	0.2603	3.0	1	85				
QC check 5.0 ppm	4.903	1.0	1	1				98%
R2148-1 Top	124.9	3.0	1	85	125.2	5.089	Read above top std, re-run	
R2148-2 Top	102.7	3.0	1	70	103.0	5.083	Read above top std, re-run	
R2148-3 Top	6.854	3.0	1	85	6.776	0.275		99%
R2148-4 Top	11.16	3.0	1	85	11.29	0.458		
R2148-5 Top	30.15	3.0	1	90	30.44	1.168	Read above top std, re-run	
R2148-6 Top	26.09	3.0	1	85	26.37	1.030	Read above top std, re-run	
QC check 5.0 ppm	4.955	1.0	1	1				99%
Mill Q Blk Bottom	0.0586	3.0	1	100				
R2148-1 bottom	0.1633	3.0	1	5				
R2148-2 bottom	0.1910	3.0	1	5				
R2148-3 bottom	0.1423	3.0	1	10				
R2148-6 bottom	0.1128	3.0	1	10				
R2148-7 bottom	0.2298	3.0	1	50				
R2148-8 bottom	0.1881	3.0	1	50				
Mill Q blk Top	0.0531	3.0	1	100	0.110	0.0031		
R2148-1 top	6.381	3.0	1	5	6.544	4.5061		
R2148-2 top	5.899	3.0	1	5	6.090	4.1951		
R2148-3 top	3.093	3.0	1	10	3.236	1.1113		
R2148- 6 top	2.116	3.0	1	10	2.228	0.7632		
R2148- 7 top	2.466	3.0	1	50	2.696	0.1849		
R2148- 8 top	1.936	3.0	1	50	2.024	0.1386		
QC cal check 4.33 ppm	2.04	1.0	2	1		4.072		94%

R2148.xls

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3MA00230201

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3M_MN00043384

2759.0079

Orion Data

AOF ANALYSIS — AOF Calc. (µg/mL) = ((total meter reading - Intercept)/ slope) / sample volume — y=0.2884+0.0196

Sample ID	Meter Reading	Collect Vol (mL)	Dilution Factor	Sample Vol (mL)	Meter Reading Total (top&bottom)	Calc. Adsorbable Organic Fluorine (top&bottom)	Comments	% Recovery
R2148-9 bottom	0.2705	3.0	1	50				
R2148-10 bottom	0.2784	3.0	1	50				
R2148-11 bottom	0.1188	3.0	1	50				
R2148-12 bottom	0.0713	3.0	1	50				
R2148-9 top	1.2856	3.0	1	50	1.5661	0.1062		
R2148-10 top	1.6588	3.0	1	50	1.9372	0.1325		
R2148-11 top	0.3520	3.0	1	50	0.4707	0.0312	read below low std. re-run	
R2148-12 top	0.2883	3.0	1	50	0.3596	0.0221	read below low std. re-run	
R2148-7 MS Bottom	0.1134	3.0	1	100				
R2148-7 MS Top	3.0287	3.0	1	100	3.143	0.1079		103%
QC cal check 4.33 ppm	2.228	1.0	2	1		4.456		
R 2148-11 bottom	0.0760	3.0	1	100				
R 2148-12 bottom	0.0839	3.0	1	100				
R 2148-11 Top	0.4608	3.0	1	100	0.5368	0.0176		
R 2148-12 Top	0.4516	3.0	1	100	0.5357	0.0175		98%
QC check 0.050 ppm	0.4988	1.0	1	1				

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3MA00230202

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3M_MN00043385

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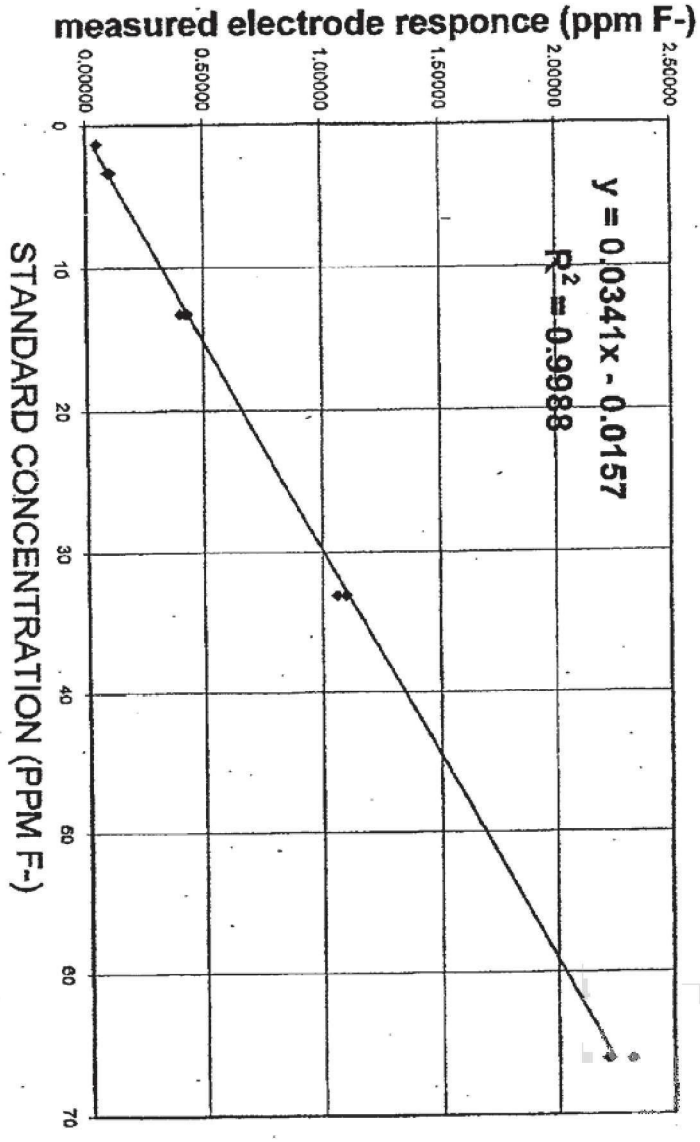

Thermally Extracted Perfluorooctanoate Curve

POAA CURVE

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3MA00230203

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Perfluorooctanoate Standard Curve

Sample ID	FACTOR	SAMPLE (mL)	COLLECT (mL)	CALC. (ppm)	POAA (ppm)	Actual (ppm)	% REC	COMMENTS
ERA 4.33PPM STD	2					2.200	101.6	
QC 1.0PPM STD CHECK						0.9652	96.52	
BLK-1	1	0.1	3	1.47		0.0491		
BLK-2	1	0.1	3	1.02		0.0340		
BLK-3	1	0.1	3	0.48		0.0161		
POAA 1.0 PPM W397-795	1	0.1	3	1.16	0.661	0.0388	176	BELOW DETECTION LEVEL
POAA 1.0 PPM W397-795	1	0.1	3	0.97	0.661	0.0323	147	USE 2.0PPM STD DATA
POAA 1.0 PPM W397-795	1	0.1	3	1.40	0.661	0.0467	212	NOT INCLUDED IN CURVE
ERA 4.33PPM STD						2.162	99.86	
QC 1.0PPM STD CHECK						0.984	98.64	
BLK-1	1	0.1	3			0.01599		
BLK-2	1	0.1	3			0.01455		
BLK-3	1	0.1	3			0.01461		
POAA 2.0PPM W397-801	1	0.1	3	1.473	1.322	0.04911	111	111
POAA 2.0PPM W397-801	1	0.1	3	1.473	1.322	0.04911	111	111
POAA 2.0PPM W397-801	1	0.1	3	1.397	1.322	0.04655	106	106
POAA 5.0 PPM W397-796	1	0.1	3	2.82	3.31	0.0939	85.2	
POAA 5.0 PPM W397-796	1	0.1	3	3.22	3.31	0.1074	97.5	
POAA 5.0 PPM W397-796	1	0.1	3	2.89	3.31	0.0998	90.6	
QC 1.0 PPM CHECK						0.9691	96.9	
POAA 20PPM W397-797	1	0.1	3	12.10	13.22	0.4033	91.5	
POAA 20PPM W397-797	1	0.1	3	12.80	13.22	0.4268	96.9	
POAA 20PPM W397-797	1	0.1	3	12.96	13.22	0.4321	98.1	
POAA 50PPM W397-798	1	0.1	3	31.82	33.05	1.064	96.8	
POAA 50PPM W397-798	1	0.1	3	33.12	33.05	1.104	100	
POAA 50PPM W397-798	1	0.1	3	32.97	33.05	1.099	99.8	
POAA 100PPM W397-799	1	0.1	3	66.54	66.10	2.218	101	
POAA 100PPM W397-799	1	0.1	3	69.60	66.10	2.320	105	
POAA 100PPM W397-799	1	0.1	3	66.27	66.10	2.209	100	
QC 1.0 PPM CHECK						0.9930	99.3	

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3MA00230204

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TABLE : R2148 F- DETERMINATION: SAMPLES DUPONT INC.

CALCULATED (PPM F-) = (meter reading + intercept) / slope)

SEE STANDARD CURVE FOR EQUATION: Y = 0.0341X + -0.0157

	SAMPLE	COLLET	Actual	CALC.		STATS		% REC
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]		[redacted]		[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]		[redacted]		[redacted]
ERA 4.33PPM STD			2.162	0.9884				99.86
QC 1.0PPM STD CHECK								98.64
BLK-1	0.1	3	0.01598					
BLK-2	0.1	3	0.01455					
BLK-3	0.1	3	0.01491					
QC 1.0PPM CHECK			0.9743					97.43
R2148-11-1	0.1	3	0.05555	2.09	AVE	2.33		
R2148-11-2	0.1	3	0.06031	2.23	STD	0.30		
R2148-11-3	0.1	3	0.07499	2.66	CV	12.8		
R2148-12-1	0.1	3	0.1699	5.44	AVE	4.23		
R2148-12-2	0.1	3	0.1233	4.08	STD	1.14		
R2148-12-3	0.1	3	0.09247	3.17	CV	27.0		
R2148-10-1	0.1	3	0.08379	2.92	AVE	2.81		
R2148-10-2	0.1	3	0.08284	2.90	STD	0.47		
R2148-10-3	0.1	3	0.09400	3.22	CV	16.6		
QC 1.0 PPM CHECK			0.9663					96.63
R2148-8-1	0.1	3	0.1042	3.52	AVE	2.96		
R2148-8-2	0.1	3	0.07910	2.78	STD	0.49		
R2148-8-3	0.1	3	0.07288	2.60	CV	16.4		
R2148-6-1	0.1	3	0.1361	4.45	AVE	4.15		
R2148-6-2	0.1	3	0.1279	4.21	STD	0.34		
R2148-6-3	0.1	3	0.1131	3.78	CV	8.24		
QC 1.0PPM STD CHECK			0.9624					96.24

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TABLE : R2148 F- DETERMINATION: SAMPLES DUPONT INC.

CALCULATED (PPM F-) = (meter reading + intercept) / slope)

SEE STANDARD CURVE FOR EQUATION: Y = 0.0341X + -0.0157

	SAMPLE	COLLET	Actual	CALC.		STATS		% REC
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]		[redacted]		[redacted]
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]		[redacted]		[redacted]
ERA 4.33PPM STD			2.162	0.9884				99.86
QC 1.0PPM STD CHECK								98.64
BLK-1	0.1	3	0.01598					
BLK-2	0.1	3	0.01455					
BLK-3	0.1	3	0.01491					
QC 1.0PPM CHECK			0.9743					97.43
R2148-11-1	0.1	3	0.05555	2.09	AVE	2.33		
R2148-11-2	0.1	3	0.06031	2.23	STD	0.30		
R2148-11-3	0.1	3	0.07499	2.66	CV	12.8		
R2148-12-1	0.1	3	0.1699	5.44	AVE	4.23		
R2148-12-2	0.1	3	0.1233	4.08	STD	1.14		
R2148-12-3	0.1	3	0.09247	3.17	CV	27.0		
R2148-10-1	0.1	3	0.08379	2.92	AVE	2.81		
R2148-10-2	0.1	3	0.08284	2.90	STD	0.47		
R2148-10-3	0.1	3	0.09400	3.22	CV	16.6		
QC 1.0 PPM CHECK			0.9663					96.63
R2148-8-1	0.1	3	0.1042	3.52	AVE	2.96		
R2148-8-2	0.1	3	0.07910	2.78	STD	0.49		
R2148-8-3	0.1	3	0.07288	2.60	CV	16.4		
R2148-6-1	0.1	3	0.1361	4.45	AVE	4.15		
R2148-6-2	0.1	3	0.1279	4.21	STD	0.34		
R2148-6-3	0.1	3	0.1131	3.78	CV	8.24		
QC 1.0PPM STD CHECK			0.9624					96.24

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3M Environmental Laboratory

Soil Properties and Nutrient Concentration Analyses
(6/97 Samples)

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3MA00230207

CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

3M Ecotoxicology and Environmental Fate Laboratory

Soil Properties and Nutrient Concentration Analyses of Samples Received From the E. I. DuPont de Nemours and Company Facility in Parkersburg, West Virginia

STUDY COMPLETED: August 27, 1997

FINAL REPORT COMPLETED: October 31, 1997

Prepared by: Susan A. Beach
Senior Environmental Biologist
3M Environmental Laboratory
Building 2-3E-09
935 Bush Avenue
St Paul, MN 55144

Lab Request No. R2382

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3MA00230208

CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

1.0 Introduction

Eleven soil samples were received from E.I. DuPont de Nemours and Company for preparation and analyses by the Ecotoxicology and Environmental Fate Testing Group of the 3M Environmental Laboratory. These samples were assigned a Lab Request number (LR No.) of R2382. The sample date was 6/23/97. Samples were numbered R2382-1 through R2382-11 as follows:

3M LR No.	DuPont COC Description
R2382-1	SS -1 0-2'
R2382-2	SS -1 4-6'
R2382-3	SS -1 8-10'
R2382-4	SS -1 12-14'
R2382-5	SS -1 16-18'
R2382-6	SS -1 20-22'
R2382-7	SS -1 24-26'
R2382-8	SS -1 28-30'
R2382-9	SS -1 32-34'
R2382-10	SS -1 36-38'
R2382-11	SS -1 38-40'

2.0 Results

A summary of the results obtained is presented below. Copies of methods, raw data sheets, and contract laboratory reports are attached to this summary report.

	DuPont Sample No.	Sulfate, mg/kg	Sulfite, mg/L	Nitrite Nitrogen, mg/kg	pH in CaCl2	pH in water	CEC, meq/100 g	% Moisture, (as-rec'd)
R2382-1	SS-1 0-2'	98	<2	0.41	7.2	7.7	15.8	12.3
R2382-2	SS-1 4-6'	99	<2	0.41	7.3	7.7	18.4	12.7
R2382-3	SS-1 8-10'	73	<2	0.38	7.0	7.3	17.5	15.5
R2382-4	SS-1 12-14'	54	<2	0.14	6.3	6.7	17.5	18.9
R2382-5	SS-1 16-18'	43	<2	<0.10	5.3	5.7	18.4	18.3
R2382-6	SS-1 20-22'	70	<2	<0.10	5.5	6.0	19.3	19.2
R2382-7	SS-1 24-26'	220	<2	<0.10	5.3	5.8	17.5	20.0
R2382-8	SS-1 28-30'	150	<2	0.11	6.3	6.8	11.4	18.1
R2382-9	SS-1 32-34'	100	<2	<0.10	5.2	5.8	13.1	13.6
R2382-10	SS-1 36-38'	63	<2	<0.10	5.4	6.1	9.6	17.9
R2382-11	SS-1 38-40'	46	<2	<0.10	6.2	6.8	6.3	22.2

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3.0 Initial Observations

3.1 Sample R2382-1
Half of the two foot column not filled. Only one foot of soil present.

3.2 Samples R2382-2 through R2382-11
Had an unusual odor, possibly hydrocarbons.

3.3 Samples R2382-1 through R2382-6
Appear to be clay/silt.

3.4 Samples R2382-7 and R2382-8
Appear to be clay/silt/sand, more silt/sand.

3.5 Sample R2382-9
Appears to be sand/silt.

3.6 Sample R2382-10
Appears to be sand/silt with free-flowing water in column.

3.7 Sample R2382-11
Appears to be coarse sand.

4.0 Sub sampling

A one-foot core from the top of each column through the center was removed. The sample was thoroughly mixed then split for inorganic and organic analyses. The remaining intact cores were refrigerated at 4°C in the dark.

5.0 Sample Preparation

Aliquots of well-mixed wet soil (as received) were prepared as necessary for soil properties testing, nutrient analyses, CEC, and total fluoride analyses. After preparation, aliquots were provided to the proper laboratory personnel for testing.

5.1 Air-Dried Soil - 2.00 mm
Soil samples were air-dried at ambient room temperature to constant weight. Soil was crushed, as necessary with a mortar and pestle, and passed through a 2.00 mm stainless steel sieve. Soil prepared this way was used for pH analyses.

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either of them, about any of the potential substance that might come up during the deposition?
A. I did not.
Q. Did they ask, either of them?
A. My supervisor asked whether it was related to my employment for 3M or whether it was related to my employment as commissioner. And I responded that I was unclear.
Q. Did they ask any other questions?
A. They did not.
Q. Did you provide any explanation to them about what was going on with this deposition?
A. I did not.
Q. After you were notified that a request had been made for your deposition in this case, did you contact anyone at the MPCA?
A. I did not.
Q. Did you contact anyone at the Minnesota Attorney General's Office?
A. I did.
Q. Who did you contact?
A. Bob Roach.
Q. And what is his position?
A. Assistant Attorney General.

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Q. With the -- in Minnesota, correct?
A. Correct.
Q. Why did you contact him?
A. To let him know that I had -- that I would be deposed, and that they should contact my attorney. At that time I don't believe I had an attorney. And I said that I would make sure and give my counsel Bob's information.
Q. Why did you contact Mr. Roach?
A. Because he had been the assistant AG, attorney general, who had represented our agency during my tenure as commissioner.
Q. And did Mr. Roach work with Alan Williams?
A. I believe they are colleagues.
Q. When you notified Mr. Roach that your deposition was being requested, did he respond in any way?
A. He did not.
Q. Did he ask any questions?
A. No. My phone call to him was a voice mail, I believe.
Q. Did you receive any voice -- any return call from Mr. Roach?
A. I did not. But I didn't expect one as the call was just to let him know that I would be

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involved in this proceeding and that I was in the process of engaging an attorney.
Q. Do you have an understanding as to whether anyone with the Minnesota Attorney General's Office or the MPCA has contacted your counsel?
MR. ARMSTRONG: Well, if she has that information from somebody other than me, I'll let her answer. But if her only information about that is something I told her, then that's privileged, and I'll instruct her not to answer.
A. And the question was again?
Q. Do you have an understanding as to whether or not anyone at the Minnesota Attorney General's Office or MPCA has, in fact, contacted your counsel?
MR. ARMSTRONG: And again, limit your answer to anything that you learned other than from me.
A. I do not.
Q. Do you know whether or not anybody at the Minnesota Attorney General's Office or MPCA has provided any information to your counsel?
MR. ARMSTRONG: Same instruction.

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A. I do not.
Q. You don't know?
A. I don't have any information from anyone other than my counsel.
Q. Do you have information from your counsel?
MR. ARMSTRONG: Well, I'm going to instruct her not to answer that. That's privileged, because the answer to that would imply one way or the other, and so I'm not going to let her answer that.
MR. BILOTT: You're instructing her not to answer?
MR. ARMSTRONG: I am.
MR. BILOTT: All right.
BY MR. BILOTT:
Q. Do you know whether or not any instructions were provided by the Minnesota Attorney General's Office or the MPCA to anyone relating to your deposition today?
A. I do not know.
Q. Do you know whether anyone at 3M has contacted your counsel?
MR. ARMSTRONG: Same instruction. Again, I think the only way she could know that, unless someone at 3M told her, is based

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5.2 Air-Dried Soil - 0.500 mm

Aliquots of air-dried 2.00 mm soil were crushed with a mortar and pestle until the entire sample passed through a 0.500 mm stainless steel sieve. Soil prepared this way was used for CEC and nutrient analyses.

5.3 Oven-Dried Soil - 0.063 mm

Aliquots of the 2.00 mm air-dried soil were finely ground with a mortar and pestle until the entire sample passed through a 0.063 mm stainless steel sieve. The samples were then oven-dried (105°C) to constant weight. Soil prepared this way was used for total fluoride analyses (results presented in a separate report by 3M AMDT Laboratory).

6.0 Analytical Methodology

6.1 Soil Water Content

Aluminum pans were oven-dried to constant weight. Twenty-three to thirty-five gram aliquots of well-mixed wet soil (as-received) were weighed in the aluminum pans. The pans and soil were then oven-dried at 105°C to constant weight. The soil water content was determined by the following equation:

$$\frac{\text{Weight of Wet Soil - Weight of Dry Soil}}{\text{Weight of Dry Soil}} \times 100$$

6.2 Soil pH

6.2.1 pH in Water

Ten grams of 2.00 mm-sieved soil and 10 mL Milli-Q® water were placed into 50 mL conical centrifuge tubes. The tubes were then capped and shaken for one hour. After shaking, the tubes were allowed to stand for one hour. A Cole-Parmer Model 5992-60 soil electrode was used to measure the pH.

6.2.2 pH in 0.01M $CaCl_2$

After pH in water was determined, 0.10 mL of 1.0 M $CaCl_2$ was added to each tube. The tubes were shaken for 30-minutes then allowed to stand for 30-minutes. A Cole-Parmer Model 5992-60 soil electrode was used to measure the pH.

6.3 Cation Exchange Capacity (CEC) by Sodium Saturation

6.3.1 Adsorption Step

Five grams of 0.500 mm-sieved soil and 132 mL of 1.0N pH 8.2 NaOAC were placed into 250 mL conical polypropylene centrifuge tubes. The

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tubes were then capped and shaken overnight at 300-400 rpm. After shaking, the tubes were centrifuged for 10 minutes at 3000 rpm. The supernatant was then decanted and discarded.

6.3.2 Washing Step
Fifty mL of 2-propanol was then added to each soil. Tubes were capped and shaken for 30-minutes. After shaking, they were centrifuged as in 6.3.1 and the supernatant discarded. This step was then repeated with another 50 mL aliquot of 2-propanol.

6.3.3 Desorption Step
One hundred mL of 1.0 N, pH 7.0 NH_4OAc was added to each sample. The tubes were stoppered and shaken over-night. After shaking, they were centrifuged for 10-minutes at 3000 rpm. The supernatants were decanted for sodium analyses. The supernatants were then submitted to the Inorganic Analysis Group of the 3M Environmental Laboratory for analysis of sodium by ICP (SW-846, Method 6010).

6.3.4 Calculation of Cation Exchange Capacity

$$\frac{0.1 \times (\text{conc. of Na, mg/L})/23}{\text{oven-dried weight of soil, g}^*} \times 100 = \text{meq / 100 g soil}$$

*Sub-samples of 0.500 mm air-dried soil were oven-dried to constant weight and the moisture content was determined. The values obtained were used to calculate the final CEC value of soil on an oven-dried weight basis.

6.4 Nutrient Analyses
Aliquots of the 0.500 mm-sieved soil were submitted to Minnesota Valley Testing Laboratories, Inc. (MVTL) for analysis of nitrite, sulfate and sulfite. The following methods were employed:

6.4.1 Nitrite Nitrogen
Methods of Soil Analysis, 2nd Edition, 33-8.

6.4.2 Sulfate
SW-846, Method 9088.

6.4.3 Sulfite
EPA Method 377.1.

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Copies of Raw Data and Contract Laboratory Reports

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ENVIRONMENTAL LABORATORY

WORKSHEET

LR RZ382
PAGE 1 OF 1
DATE 8/5/94
ANALYST 3

Initial Observations
Samples 2 thru 11 all had a non-normal soil odor, HC?

Sample 1 Half of 2' column not there. Only 1' of soil present. (Clay silt)

Sample 1-6 appear to be clay/silt
Sample 7&8 appear to be clay/silt/sand more silt/sand

Sample 9 appear to be sand/silt

Sample 10 appear to be sand/silt with free standing water in column

Sample 11 appear to be coarse sand

An one foot core from the top of each column (bury the center was removed for analysis. The rest of the intact cores were refrigerated @ 4°C in the dark until needed. The core sample was mixed then split for inorganic and organic analysis.

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ENVIRONMENTAL LABORATORY

SOIL WATER CONTENT
PERCENTAGE OF WATER IN THE SAMPLE ON A DRY-MASS BASIS

Gardner, Walter. 1986. Water Content. p. 493-544. In Arnold Klute (Ed.). Methods of Soil Analysis, Part 1. Physical and Mineralogical Methods. Agronomy Monograph No. 9 (2nd Edition).

% Water Content = $\frac{\text{Weight of Wet Soil} - \text{Weight of Dry Soil}}{\text{Weight of Dry Soil}} \times 100$

SAMPLE DESCRIPTION	As Recd Wet Soil g (air-dried)	Dry Soil g (oven-dried)	NET LOSS g	Soil-Moist %
RZ3B2-1	33.818	30.123	3.695	12.3
2	23.192	20.580	2.612	12.7
3	25.591	22.159	3.432	15.5
4	26.343	22.160	4.183	18.9
5	22.670	19.161	3.509	18.3
6	33.796	28.354	5.442	19.2
7	27.695	23.096	4.599	20.0
8	31.806	26.929	4.877	18.1
9	22.507	19.816	2.691	13.6
10	24.960	21.168	3.792	17.9
11	36.229	29.659	6.570	22.2

ANALYST: [signature]
DATE: 8/5-6/97

3MA00230215

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ENVIRONMENTAL LABORATORY

SOIL pH and LIME REQUIREMENT(LR)

McLean, E. O. 1982. Soil pH and Lime Requirement. p. 199-224. In A. L. Page, R. H. Miller & D. R. Keeney (Eds.) Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties, Agronomy Monograph No. 9 (2nd Edition).

2.00 mm air-dried soil used.

Soil - to - Millipore Milli-Q™ Water Ratio 1:1, 10 g plus 10 mL.
For LR, SMP single-buffer method used, Soil - to - SMP Buffer Ratio 1:2, 10 g plus 20 mL.

SAMPLE DESCRIPTION	pHw (pH in water)	pHs (pH in 0.01M CaCl2)	Soil-Buffer pH	LR (T/A)
R2382-1	7.7	7.2		
2	7.7	7.5		
3	7.3	7.0		
4	6.4	6.3		
5	5.7	5.3		
6	6.0	5.5		
7	5.8	5.3		
8	6.8	6.3		
9	5.8	5.2		
10	6.1	5.7		
11	6.8	6.2		

ANALYST:
DATE: 8/24/94

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3MA00230216

Form 5455-A

FILE

ENG.

DATE SHEET

12382

ANALYSIS FORM

		A
1		
2	123821	180
3		
4	2	110
5		
6	3	200
7		
8	4	200
9		
10	5	210
11		
12	6	220
13		
14	7	200
15		
16	8	130
17		
18	9	150
19		
20	10	110
21		
22	11	70
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89 % 125

3MA00230217

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ENVIRONMENTAL LABORATORY

SOIL WATER CONTENT
PERCENTAGE OF WATER IN THE SAMPLE ON A DRY-MASS BASIS

Gardner, Walter. 1986. Water Content. p. 493-544. In Arnold Klute (Ed.). Methods of Soil Analysis, Part 1. Physical and Mineralogical Methods. Agronomy Monograph No. 9 (2nd Edition).

$$\% \text{ Water Content} = \frac{\text{Weight of Wet Soil} - \text{Weight of Dry Soil}}{\text{Weight of Dry Soil}} \times 100$$

For CEC Calculation

SAMPLE DESCRIPTION	Wet Soil g (air-dried)	Dry Soil g (oven-dried)	NET LOSS g	%
RZ382-1	4.749	4.688	0.061	0.79
2	9.137	9.054	0.083	0.87
3	7.848	7.777	0.071	0.91
4	4.079	4.021	0.058	0.83
5	6.510	6.452	0.058	0.90
6	6.651	6.602	0.049	0.74
7	7.125	7.070	0.055	0.78
8	7.056	7.022	0.034	0.48
9	6.749	6.722	0.027	0.40
10	6.655	6.634	0.021	0.32
11	6.090	6.075	0.015	0.25

ANALYST:
DATE: 8/27/94

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3MA00230218

ENVIRONMENTAL LABORATORY

CATION EXCHANGE CAPACITY (CEC)
BY SODIUM SATURATION

Rhoades, J. D. 1982. Cation Exchange Capacity. p. 149-157. In A. L. Page, R. H. Miller & D. R. Keeney (Eds.) Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties, Agronomy Monograph No. 9 (2nd Edition).

Extracts prepared using 0.500 mm air-dried soil. Cation Exchange Capacity by Sodium Saturation; modification of method is as follows:

*ADSORPTION Batch equilibrium method (over-night) with 1.0 N NaOAc pH 8.2 as saturation solution.
*WASHED Two washings with 2-Propanol.
*DESORPTION Batch equilibrium method (over-night) with 1.0N NH4OAc pH 7.0 as extraction solution.

CEC EQUATION: (0.1 Na ppm/ 23 / oven-dried wt. of soil g) x 100 = meq/ 100 g soil

SAMPLE DESCRIPTION	AIR-DRIED WT. g	OVEN-DRIED WT. g	mg Na / L	meq / 100 g soil
R7382-1	5.000	4.961	180	15.9
2	5.000	4.957	210	18.4
3	5.000	4.955	200	17.5
4	5.000	4.959	200	17.5
5	5.000	4.955	210	18.4
6	5.000	4.963	220	19.3
7	5.000	4.961	200	17.5
8	5.000	4.976	130	11.4
9	5.000	4.980	150	13.1
10	5.000	4.984	110	9.6
11	5.000	4.988	72	6.3

ANALYST:
DATE: 5/27/94

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3MA00230219

MVTL LABORATORIES, Inc.
P.O. BOX 249, 1126 N. FRONT STREET
NEW ULM, MN 56073-0249
PHONE (507) 354-8517 WATS (800) 782-3557 FAX (507) 359-2890
WE ARE AN EQUAL OPPORTUNITY EMPLOYER

MEMBER ACIL

Report To: Rochelle Robideau
3M Environmental Lab
935 Bush Ave., Bldg. 20
St. Paul, MN 55106

Date: 27 August 1997
Work Order: 10-0447
Date Received: 25 August 1997

Inorganics Lab No.	S4352	S4353	S4354	S4355	S4356	S4357
Soil Lab No.	W-547	W-548	W-549	W-550	W-551	W-552
Sample I.D.	R2382-1	R2382-2	R2382-3	R2382-4	R2382-5	R2382-6
Analyte						
Nitrite Nitrogen (mg/Kg N) ..	0.41	0.41	0.36	0.14	<0.1	<0.1
Sulfate (mg/Kg)	98.4	98.6	73.3	53.9	43.4	70.4
Sulfite (mg/L)	<2	<2	<2	<2	<2	<2

Report approved by: a Koebele
Anthony R. Koebele
By and for Minnesota Valley Testing Laboratories, Inc.

MVTL guarantees the accuracy of the analysis done on the sample submitted for testing. It is not possible for MVTL to guarantee that a test result obtained on a particular sample will be the same as any other sample unless all conditions affecting the sample are the same, including sampling by MVTL. As a mutual protection to clients, the public and ourselves, all reports are submitted as the confidential property of clients, and authorization for publication of statements, conclusions or extracts from or regarding our reports is reserved pending our written approval.

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upon a communication between my client and me. And so you need to limit your answer to outside that context.
A. There's been no contact that I'm aware of outside of that context.
Q. Let me ask it this way: Without telling me whether -- what information was provided, do you know the answer to that question?
MR. ARMSTRONG: To what question?
BY MR. BILOTT:
Q. That I just asked. Whether or not there has been information provided by 3M to your counsel. Do you know the answer?
MR. ARMSTRONG: That's a yes or no question. I'll let you answer that.
A. Yes.
Q. Can you briefly summarize for me your educational background after high school, degrees you have obtained?
A. I attended the University of Minnesota Institute of Technology and received a Bachelor of Science degree in geology.
Q. When did you obtain that degree?
A. 1986, I believe.
Q. And have you obtained any other educational

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degrees beyond your BS?
A. Not a full degree, no.
Q. Did you work toward any further degrees?
A. I did.
Q. What did you work towards?
A. A Master's of Secondary Science Education.
Q. How many -- how long did you pursue that degree?
A. A year.
Q. Any other -- have you worked toward any other educational degrees?
A. No, I have not.
Q. Do you have any professional licenses or certifications of any kind?
A. Yes, I do.
Q. What are those?
A. I'm a professional geologist licensed in the State of Wisconsin.
Q. When did you first become licensed in Wisconsin in that regard?
A. I want -- I'm thinking back. 1994 or '95, I believe.
Q. And do you remain licensed as a professional geologist?
A. Yes, I do.

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Q. And have you remained so licensed since '94?
A. Yes, I have.
Q. Any other professional licenses or certifications of any kind?
A. No.
Q. Can you summarize for me your employment history beginning with your first full-time job after college?
A. My first full-time job after college was with the Minnesota Pollution Control Agency.
Q. And when did you start with MPCA?
A. 1986 or 1987.
Q. And what position did you start at MPCA at that time?
A. I was a pollution control specialist.
Q. How long did you remain in that position with MPCA?
A. I recall about a year and a half, perhaps, or a year, somewhere around that.
Q. So until late 1987 or 1988?
A. Correct.
Q. And what happened with respect to your employment at that time?
A. I stayed at the agency, but I had a different position.

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Q. And what position did you get at that point?
A. I was a Pollution Control Specialist II, and I worked in a different group within the organization.
Q. Okay. And how long did you remain in that position?
A. About a year and a half, a year, year and a half.
Q. And then what happened with respect to your employment at that point?
A. Then I took a new role at the Metropolitan Council as a water planner.
Q. And that was in approximately 1990?
A. Correct.
Q. How long did you remain in that position?
A. Again, it's quite some time ago. My recollection is for about a year, year and a half, two years, somewhere around there.
Q. And what happened with respect to your employment at that point?
A. Then I took a new role with the -- with King County in Seattle, Washington.
Q. And what did you do for King County?
A. I was a surface water planner.
Q. And that was beginning approximately 1992?

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MEMBER ACIL

Report To: Rochelle Robidoux
3M Environmental Lab
935 Bush Ave., Bldg. 20
St. Paul, MN 55106

Date: 27 August 1997
Work Order: 10-0447
Date Received: 25 August 1997

Inorganics Lab No.	S4358	S4359	S4360	S4361	S4362
Soil Lab No.	W-553	W-554	W-555	W-556	W-557
Sample I.D.	R2382-7	R2382-8	R2382-9	R2382-10	R2382-11
Analyte					
Nitrite Nitrogen (mg/Kg N) ..	<0.1	0.11	<0.1	<0.1	<0.1
Sulfate (mg/Kg)	217	153	102	63.3	46.2
Sulfite (mg/L)	<2	<2	<2	<2	<2

Report approved by:
Anthony R. Koebele
By and for Minnesota Valley Testing Laboratories, Inc.

MVTL guarantees the accuracy of the analysis done on the sample submitted for testing. It is not possible for MVTL to guarantee that a test result obtained on a particular sample will be the same as any other sample unless all conditions affecting the sample are the same, including sampling by MVTL. As a test and protection to clients, the public and ourselves, all reports are submitted as the confidential property of clients, and authorization for publication of statements, conclusions or extracts from or regarding our reports is reserved pending our written approval.

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3MA00230221

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MEMBER ACIL

Analysis of 3M Samples

Analyte	Detection Level	Method Reference
Nitrite Nitrogen	0.1 mg/Kg N	Methods of Soil Analysis, 2nd Edition, 33-8
Sulfate	40 mg/Kg on a 5 g sample	SW-846, Method 9038
Sulfite	2.00 mg/L	EPA Method 377.1
[redacted]	[redacted]	[redacted]
[redacted]	[redacted]	[redacted]

MVTL guarantees the accuracy of the analysis done on the sample submitted for testing. It is not possible for MVTL to guarantee that a test result obtained on a particular sample will be the same on any other sample unless all conditions affecting the sample are the same, including sampling by MVTL. As a mutual protection to clients, the public and ourselves, all reports are submitted as the confidential property of clients, and authorization for publication of statements, conclusions or extracts from or regarding our reports is reserved pending our written approval.

94 & 125

3MA00230222

DATE: 08/21/1997

3M ENVIRONMENTAL LABORATORY
CONTRACT LABORATORY WORK ORDER BY PARAMETER

LAB REQUEST NO. R2382

CONTRACT LAB : MVTL
PROJECT LEAD: RD HOWELL

PROJECT NUMBER : BIOENVIR
TELEPHONE :

SHIP DATE : 8/21/97
FAX : 612-778-6176

Computer Code	Test Name	Sample Numbers	Sample Available Date	Result Due Date
NO2-N	NITRITE NITROGEN - as N	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11	08/13/1997	08/27/1997
SO3	SULFITE	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11	08/13/1997	08/27/1997
SO4	SULFATE - as SO4	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11	08/13/1997	08/27/1997

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Copies of Soil Methods

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CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

3M ENVIRONMENTAL LABORATORY

PRELIMINARY PREPARATION OF SOIL SAMPLES FOR LABORATORY ANALYSIS

2.00 mm Air-Dry Soil

- Air-dry (ambient room temperature) soil samples on flat trays for 24 to 48 hours or until thoroughly dry (constant weight). If desired, samples can be oven-dried at 35°C overnight (18 ± 4 hours).*
- Pass air-dried soil (crushed and mixed via mortar and pestle) through a 2.00 mm (10 mesh) stainless steel sieve. This removes large pieces of foreign material such as stones, gravel and twigs.
- Partition the sample by the "quarter" system or by passing through a riffle sampler (sample splitter). If desired, quarters can be further divided into smaller portions.
- The soil sample is now ready for laboratory analysis. Use the 2.00 mm air-dry soil for soil reaction tests and for soluble salts analysis or store in a cool, dark room. This soil is also used for physical characteristic analysis.

SOIL REACTION TESTS	SOLUBLE SALTS ANALYSIS	PHYSICAL CHARACTERISTICS
pH	Electrical Conductivity	Soil texture and classification
Lime Requirement	Cl^-, SO_4	
Gypsum Requirement	Alkalinity	

0.500 mm Air-Dry Soil

- Obtain about 20 to 40 g of a representative portion of the 2.00 mm soil and grind in an agate mortar and pestle until the entire sample passes through a 0.500 mm (35 mesh) sieve.
- Use this soil for exchange activity tests (Cation Exchange Capacity, Base Saturation, SAR, ESP) and for nutrient analysis (N, P, S).

0.053 mm Oven-Dry Soil

- Obtain about 10 to 20 g of a representative portion of the 2.00 mm soil and finely grind in an agate ball mill until the entire sample passes through a 0.053 mm (250 mesh) sieve.
- Oven-dry (105-110°C) the above sample (placed in a tared aluminum weighing dish) overnight and report the loss in weight.
- Use this finely ground (ball milled) oven-dry soil for total elemental analysis (ICP, AA), total fluoride and TOC analysis.

SPECIAL NOTE

- All dried soil samples are placed in impermeable, polypropylene bottles. They are stored in the soil cabinet (dark and at ambient room temperature) for one year after testing.
- Disposal for all soils and their extracts is by incineration.

REFERENCES

Page, A. L., Miller R. H. & Keeney D. R. (Eds.) 1982. Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties. Agronomy Monograph No. 9 (2nd Edition).

Elk, K. and R. H. Goldsmith, 1988. Soil Sample Preparation. p. 2-4. In: Recommended Chemical Soil Test Procedures for the North Central Region. North Central Regional Publication No. 221 (Revised).

*NOTE, if nitrate analyses are to be determined, the soil should be dried within twelve hours of sampling to prevent changes in the nitrate content.

(Revised 12/94 RRR)

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3MA00230225

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3M ENVIRONMENTAL LABORATORY

SOIL WATER CONTENT
PERCENTAGE OF WATER IN THE SAMPLE ON A DRY-MASS BASIS

PRINCIPLE OF THE SOIL WATER CONTENT METHOD

The amount of water in a soil affects directly the growth of crops, microbes, and insects. The strength of the soil, which determines root penetration and the energy requirements for tillage are dependent on the water content, however, the amount of plant available water in the soil is dependent on the soil water potential. Since the water potential is more difficult to determine, the water content is used as the indicator of the state of water in the soil. (In laboratory terms, practically every type of soil analysis requires that the results be reported on a dry mass basis.)

Traditionally, the water content has been expressed as the ratio of the mass of water present in the sample to the mass of the sample after it has been dried at 105 °C to a constant mass. Thus, the water content as usually used in soil studies is a dimensionless ratio of two masses or is expressed as a percentage resulting from multiplying the dimensionless ratio by 100.

The laboratory procedure employed here is water content measurements by the gravimetric method. It involves weighing the wet sample, removing the water, and reweighing the sample to determine the amount of water removed. Water content is determined by dividing the difference between wet and dry masses by the mass of the dry sample to obtain the ratio of the water mass to the mass of the dry soil, then multiplied by 100. This is now the percentage of the water in the sample on a dry-mass or dry-weight basis.

RANGE AND SENSITIVITY

The range and sensitivity will depend on the time necessary to reach constant weight and the analytical balance used.

INTERFERENCES

Factors that may influence the results include:

- Failure of temperature control. The drying oven used must maintain a temperature in the range of 105 to 110 °C.
- Sample matrix. Organic soils may have mass losses arising from oxidation and volatilization of organic components, also stony and gravelly soils, both on a mass and volume basis, can be grossly misleading.

PRECISION AND ACCURACY

Accuracy and reproducibility of water content measurements, assuming that the weighing precision is consistent with the desired precision of the water content measurements, depend upon the drying technique and how used (whether 24 hours is adequate in obtaining a constant weight).

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EQUIPMENT AND REAGENTS

1). Analytical balance accurate to 0.001 g.

2). Oven-dried aluminum weighing dishes.

3). Drying oven with temperature control device that will maintain a temperature between 105-110 °C. Forced -air circulating ovens will dry samples more rapidly, but convection ovens are sufficient.

4). Desiccators containing active desiccant.

5) No reagents are required.

WATER CONTENT PROCEDURE

1). Obtain at a minimum 10 to 40 g representative portion of either a ball milled (air-dried) sample or as received (wet) sample.

2). Place in oven-dried aluminum weighing dish.

3). Weigh the sample to the nearest 0.001 g as soon as possible.

4). Place the sample in the drying oven and dry it to a constant weight (at a minimum 24 hours).

5). Remove the sample from the oven and place it in a desiccator until cooled to ambient room temperature.

6). Reweigh the sample to the nearest 0.001 g.

7). Calculate the water content as percentage of water in the sample on a dry-mass basis:

$$\% \text{ Water Content} = \frac{(\text{Weight of Wet Soil + Pan}) - (\text{Weight of Dry Soil + Pan})}{\text{Weight of Dry Soil}} \times 100$$

REFERENCES

Gardner, W. 1986. Water Content, p. 493-544. In: Arnold Klute (Ed.). Methods of Soil Analysis, Part 1. Physical and Mineralogical Methods. Agronomy Monograph No. 9 (2nd Edition).

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3M ENVIRONMENTAL LABORATORY

SOIL pH METHOD

PRINCIPLE OF THE SOIL pH METHOD

Soil pH is one of the most indicative measurements of the chemical properties of a soil. Whether a soil is acidic, neutral, or basic has much to do with the solubility of various compounds, the relative bonding of ions to exchange sites, and the activity of various microorganisms. Three soil pH ranges are particularly informative: a pH <4 indicates the presence of free acids generally from oxidation of sulfides; a pH <5.5 suggests the likely occurrence of exchangeable Al; and a pH from 7.8 to 8.2 indicates the presence of $CaCO_3$.

Soil pH is a measure of the activity of H^+ in the soil solution. Ionized H is in equilibrium with the adsorbed nonionized H but usually is a small fraction of it. Much of the nonionized acidity is exchangeable only at higher pH. Although other criteria are sometimes used as indices of lime needs of acid soils, the lime requirement is generally a measure of the base (lime) required to neutralize that fraction of the total acidity that must be neutralized to attain a desired soil pH that is favorable for crop growth. Hence the activity of H^+ in the soil solutions is the intensity factor (index), whereas exchange acidity and lime requirement are the capacity factors of soil acidity.

RANGE AND SENSITIVITY

The range and sensitivity of the method will depend on the pH meter used. In routine soil testing, it is only necessary to read the pH to 0.1 units.

INTERFERENCES

Factors that may influence the measured pH include:

- The nature and type of inorganic and organic constituents that contribute to soil acidity. (Hydrogen ions may dissociate from the exchange sites or may be displaced by hydrolysis.)
- The soil/solution ratio (1:1 is the most commonly used).
- The salt or electrolyte content (H^+ are displaced by the cations of salts contained in the soils, in addition, the salts also displace exchangeable Al, which upon hydrolysis increases the H^+ in solution.
- The CO_2 content (CO_2 from the atmosphere or soil air) dissolves in water forming carbonic acid (H_2CO_3) which can lower the pH. In the actual measurement of soil pH, the soil and water are shaken so they come to equilibrium with the CO_2 in the air, there is no effect on the pH measurement. Only in soils of very low [H^+] where the pH is considerably above 7.0 and particularly in soils containing free $CaCO_3$ does the CO_2 concentration of the air has any appreciable measurement effect on pH.
- Errors associated with equipment standardization and liquid junction potential.

The use of 0.01 M $CaCl_2$ is recommended to minimize differences caused by some of the above factors. This dilute salt solution masks small differences in salt contents without displacing a large fraction of the H^+ or Al^{3+}. In addition, errors due to the liquid junction potential are decreased.

PRECISION AND ACCURACY

Random variation of 0.1 to 0.2 pH units is allowable in replicate determinations and can be expected from one laboratory to another. Dehydrated and scratched electrodes will give erratic values.

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EQUIPMENT AND REAGENTS

1). pH meter equipped with a combination electrode (or soil pH electrode, Cole-Parmer Model #5992-60) and automatic temperature compensation (ATC) probe.

2). Standard buffers, pH 7.0 and pH 4.0.

3). 50 mL conical, polypropylene centrifuge tubes.

4). Automatic pipets.

5). Gyratory shaker.

6). Millipore Milli-Q™ water.

7). Calcium chloride ($CaCl_2$) solution, 1 M or 0.01 M.

pH_w AND pH_s PROCEDURES

1). Calibrate pH meter with commercially prepared buffer solutions of pH 7.0 and 4.0 according to the instrument instruction manual.

2). Weigh 10.000 g of 2.00 mm air-dried soil into a 50 mL conical centrifuge tube.

3). With automatic pipet, add 10 mL of Milli-Q™ water to each tube.

4). Mix thoroughly for 5 minutes, preferably on a gyratory shaker.
(Option: mix for one hour.)

5). Let stand for 10 minutes.
(Option: let stand for one hour.)

6). Insert the electrodes into the container. Note; the test mixture after settling will have an upper, relatively clear layer (supernatant layer) and a lower layer of opaque soil suspension. Immerse the electrode into this mixture until the pH sensitive bulb is covered by the opaque soil suspension while leaving the reference contact in the supernatant layer.

7). Allow time for the electrode to reach equilibrium (~1 to 3 min.) and record as soil pH in water, pH_w.

8). To determine the soil pH in 0.01 M $CaCl_2$, add 0.10 mL of 1M $CaCl_2$ solution to the soil water suspension.

9). Mix intermittently for 30 minutes.
(Option: mix 30 minutes, let stand 30 minutes.)

10). Insert electrodes, and record as soil pH in 0.01 M $CaCl_2$, pH_s. Alternatively, the soil pH in 0.01M $CaCl_2$ may be determined directly by substituting 0.01 M $CaCl_2$ for the water in step 2.

11) If the lime requirement is to be determined on the samples (pH < 6.9), save them for this purpose after reading the pH_w or pH_s.

REFERENCES

McLean, E. O. 1982. Soil pH and Lime Requirement. p. 199-224. In: A. L. Page, R. H. Miller & D. R. Keeney (Eds.) Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties, Agronomy Monograph No. 9 (2nd Edition).

Eckert, D. J. 1988. Recommended pH and Lime Requirement Tests. p. 6-8. In: Recommended Chemical Soil Test Procedures for the North Central Region. North Central Regional Publication No. 221 (Revised).

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3M ENVIRONMENTAL LABORATORY

CATION EXCHANGE CAPACITY (CEC) BY SODIUM SATURATION BATCH EQUILIBRIUM METHOD

PRINCIPLE OF THE CEC METHOD

Soils possess an electrostatic charge as a result of the atomic substitution in the lattices of the soil minerals and as a result of hydrolysis reactions on broken edges of the lattices and surfaces of the oxides, hydroxides, hydrous oxides and organic matter. These charges attract exchangeable ions and form the exchange complex.

The cation exchange capacity (CEC) is the measure of the quantity of cations reversibly adsorbed per unit weight of soil. It is expressed in milliequivalents per 100 grams of oven-dried soil. (An equivalent weight is that quantity that is chemically equal to one gram of hydrogen.) The principle of the method described here measures CEC by saturating the cation exchange sites in the soil with a specific cation, sodium; removal of the excess saturating solution (washing); and finally replacing the adsorbed cation, sodium with the ammonium ion (desorption) which is measured by an appropriate method (e.g. ICP).

RANGE AND SENSITIVITY

The range and sensitivity of the method are dependent on the complicating interactions between saturating, washing, and extracting solutions and the soil constituents.

INTERFERENCES

Potential errors exist in each step of the CEC method use. The three steps are saturation of the cation exchange sites with a specific cation; the removal of the excess saturating solution; and replacement of the saturating cation. Possible factors of error influencing these steps are:

- Saturation Step. Exchange sites may not be completely saturated with the saturating cation due to other cations in the saturating solution competing for adsorption sites or may be due to the saturating cation's replacing power is insufficient to replace the more strongly adsorbed cations, such as exchangeable aluminum and its hydroxy forms. (Exchangeable aluminum and its hydroxy forms are not readily exchanged with monovalent cation saturating solutions.) This effect causes an underestimate of the CEC. Another problem associated with this step could be the presence of other cations in the saturating solution (dissolution of calcium carbonate, gypsum and silicate minerals).
- Washing Step. This step has the most potential sources of errors. The adsorbed cation may be removed by hydrolysis and replaced by a hydrogen ion. It may also be replaced by cations brought into solution in the washing solvent from the dissolution of calcium carbonate, gypsum and silicates. Fine clay particles and organic matter may be lost during the decanting (these exchangers have a tendency to disperse as the excess electrolyte is removed during washing). Some of the saturating solution may be retained in the soil and later extracted as an exchangeable cation if the washing is incomplete or if the salt is retained. The majority of these errors cause the CEC to be underestimated.
- Replacement Step. Adsorbed cation could be trapped between interlayers by contraction of expandable 2:1 layer silicates (this is especially true in vermiculites and weathered micas) thus, preventing its replacement during extraction. The end result is an underestimate of the CEC. The second factor causing potential error is that nonexchangeable cations may be extracted from zeolites, feldspathoid, feldspar, and mafic minerals by the extracting solution. The error gives high CEC values.

PRECISION AND ACCURACY

Errors can be reduced by using a method of CEC determination that employs reagents of similar concentration and pH to those of the soil to be analyzed.

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A. 1992, 1993, somewhere around there.
Q. And how long did you remain in that position?
A. A year and a half.
Q. And what happened with respect to your employment at that point?
A. I took a new role with Northern Environmental Technologies.
Q. Northern?
A. Uh-huh.
Q. Where was that?
A. New Brighton, Minnesota.
Q. And that was in approximately 1994?
A. Yes.
Q. And how long did you remain in that position?
A. About a year, year and a half.
Q. And then what happened with respect to your employment at that point?
A. Then I took a new role with 3M Company.
Q. And what role was that?
A. As a remediation -- actually, it was a senior engineer with 3M that specialized in remediation of contaminated sites.
Q. And that was in approximately 1996?
A. 19 -- yes, 1996 it would have been.
Q. And how long did you remain in that position?

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A. In that particular position two -- between two and three years, I believe.
Q. And then what happened with respect to your employment at that point?
A. Then I took a new role within 3M Company as a manager of Environmental Operations.
Q. Manager of Environmental Operations?
A. Uh-huh.
MR. ARMSTRONG: You have to answer out loud.
A. Yes.
Q. Thank you. And that was in approximately 1998 or 1999?
A. Yes.
Q. Do you recall what year?
A. I don't, honestly. It might have been a little bit later than that.
Q. And how long did you remain in that position?
A. For a year.
Q. And then what happened with respect to your employment then?
A. Then I moved to a new role and I was the environmental health and safety manager for the Industrial Mineral Products Division within 3M.

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Q. Environment health and safety manager for which division? I'm sorry.
A. Industrial Mineral Products.
Q. Do you recall what year it was that you acquired that new position?
A. 2001 or 2002.
Q. And how long did you remain in that position?
A. A year.
Q. And then what happened with respect to your employment then?
A. Then I was appointed as the Commissioner of the Minnesota Pollution Control Agency.
Q. And how long did you remain in that position?
A. Three plus years.
Q. Until 1996 -- I'm sorry, 2006?
A. Correct.
Q. And did you leave MPCA in, was it, June of 2006?
A. Correct.
Q. When did you start working for Koch?
A. September of 2006.
Q. And did you -- were you employed by anyone between June and September of 2006?
A. No, I was not.
Q. When you started working for Koch, what was

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the position you were hired into?
A. Director of Environmental Health and Safety Compliance.
Q. Is that the position you are still in?
A. No.
Q. How long did you hold the position of Director of Environmental Health and Safety Compliance?
A. Until December of 2006.
Q. And what happened with respect to your employment then?
A. I took a new role as the Director of Compliance.
Q. Is that the role you're in now?
A. That's correct.
Q. Okay. Going back up to the first position you mentioned, which was with the MPCA as a pollution control specialist beginning in 1986, all right?
A. Or somewhere thereabouts.
Q. Okay. What MPCA location did you work out of?
A. The St. Paul office in Minnesota.
Q. Who did you report to, who was your direct supervisor at that time?

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EQUIPMENT AND REAGENTS

1). 250 mL conical, polypropylene centrifuge tubes.

2). Automatic pipets.

3). Gyratory shaker.

4). Programmable centrifuge.

5). 1.0 N, pH 8.2 NaOAc (Sodium acetate), identified as Reagent #1:
For each liter of solution, dissolve 82.03 g of $NaC_2H_3O_2$ in Milli-Q™ water. Measure the pH. The pH of this solution should be 8.2. If necessary, adjust the pH with either a few drop of acetic acid (CH_3COOH) or sodium hydroxide (NaOH) to bring the reaction of the solution to pH 8.2.

6). 1.0 N, pH 7.0 NH_4OAc (Ammonium acetate), identified as Reagent #2:
For each liter of solution, add 58 mL of glacial acetic acid (CH_3COOH) to approximately 600 mL of Milli-Q™ water and then add 70 mL of concentrated ammonium hydroxide (NH_4OH, specific gravity 0.90). It is best to add the NH_4OH under a fume hood through a long-stemmed glass funnel so that it is introduced into the bottom of the acid solution. Cool the solution to room temperature (~20 to 25 °C) and adjust the pH to 7.0 with either CH_3COOH or NH_4OH. Dilute the solution to volume, mix it and store until ready for use. Recheck the pH prior to using the solution.

7). Reagent grade, 2-propanol (99% isopropyl alcohol).

PROCEDURE FOR CEC BY SODIUM SATURATION

1). Weigh 5.000 g of a 0.500 mm air-dried soil sample and transfer the sample to a 250 mL conical, polypropylene centrifuge tube.

2). Add 132 mL of 1.0 N, pH 8.2 NaOAC solution (Reagent #1), stopper the tube and shake on the gyratory shaker over-night (~18 hours) at 300 to 400 rpm. This is the saturation step.

3). Remove the sample from the shaker and place it in the centrifuge. Centrifuge 10 minutes at 3000 rpm. This recommended time and speed will be sufficient; a clear supernatant will be obtained.

4). Decant the supernatant and discard the liquid. NOTE: Careful decanting is very important. Particles of soil lost during the decanting steps will effect the final CEC result; a lower CEC value is the end result of this which leads to a false interpretation: poor soil quality.

5). Washing the sample is the next step. This eliminates the excess sodium. Add 50 mL of 2-propanol to the sample, stopper the tube and shake it on the gyratory shaker for 30 minutes. Centrifuge as before. Decant the supernatant and discard the liquid. Repeat this step once more. Shaker speed should be the same as used in the saturation step. (Total wash time 60 minutes using 100 mL of 2-propanol.)

6). Add 100 mL of 1.0 N, pH 7.0 NH_4OAc (Reagent #2) to the sample, stopper the tube and shake it on the gyratory shaker over-night (~18 hours). This is the replacement step. NOTE: Make sure the identical shaker speed and time are used as in the saturation step.

7). Remove the sample from the shaker and place it in the centrifuge. Centrifuge 10 minutes at 3000 rpm and decant the supernatant into a 125 mL polypropylene bottle.

8). Determine the sodium (Na) content by available methods, e.g. ICP.

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CALCULATION

CEC EQUATION:

$$\frac{0.1 \text{ Na ppm}/23}{\text{oven-dried weight of soil (g)*}} \times 100 = \text{meq}/100\text{g soil}$$

* % soil moisture was previously determined.

REFERENCES

Chapman, H. D. 1965. Cation Exchange Capacity. p. 891-900. In: C. A. Black (Ed.) Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties, Agronomy Monograph No. 9 (1st Edition).

Rhoades, J. D. 1982. Cation Exchange Capacity. p. 149-157. In: A. L. Page, R. H. Miller & D. R. Keeney (Eds.) Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties, Agronomy Monograph No. 9 (2nd Edition).

Brown, J. R. and D. Warncke. 1988. Recommended Cation Tests and Measures of Cation Exchange Capacity. p. 15-16. In: Recommended Chemical Soil Test Procedures for the North Central Region. North Central Regional Publication No. 221 (Revised).

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3M Environmental Laboratory

Copies of Chain of Custody, Shipping Papers, Lab Requests

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3MA00230233

1mSHIPPING MEMORANDUM0m
(FOR NO-CHARGE SHIPMENTS ONLY)

1m4m

Ship to 4m 3M Environmental Technology &
4m Service Laboratory, B-2-3E-09
4m 935 Bush Avenue
4m St. Paul, Minnesota 55144-1000
4m Attn: Robert Howell

|Date 05/08/97 No. 38-97
|Routing
|F.O.B. Date Shipped
|B/L

Quantity	Product Code / Description	Weight
4m 1	Container of Water Samples	48 lbs

Special Inst 4mAbsolutely must arrive Friday AM/Time restraints.

Freight Class 4m

4m

4m

4mOriginator VERA L WIGAL Phone 304-863-4895

4mCharge (Cost Code) 8309019511001280

4mPurchase Order No.

4mValue for Customs

Ship From 4mStores, Bldg. 4

|MSDS Sheets Required?
|Hazardous Material? N
| MATERIAL CLASSIFICATION
|Corrosive Toxic
|Flammable Other

4m
4m
4m
4m
4m

** AUTHORIZATION **

0m
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|0m
|0m
|0m

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3MA00230234

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Purchase Order #

Project Name: DUPONT WASHINGTON WORKS [redacted] FOR ROBERT HOWELL

Project Manager & Phone #: Mr. [ ] Ms. [ ] Dr. [ ] R. ZIPFEL

Report Copy to: R. ZIPFEL

Requested Completion Date: PER AGREEMENT

Sampling Requirements: SDWA ☐ NPDES ☐ RCRA ☐ OTHER ✓

Sample Disposal: Dispose ☒ Return ☐

#	Date	Time	Type COMP	Type GRAB	Matrix WATER	Matrix SOIL	CLIENT SAMPLE ID (9 CHARACTERS)	# OF CONTAINERS	LAB TEST CODES / ANALYSES REQUESTED: FC143	C7	REMARKS
1	5/8/97	1:48P		X	X	-	RANNGI WELL		X	X	CONTACT DAN WEBER AT 304-863-4415 IF THERE ARE QUESTIONS
2	5/8/97	1:48P		X	X		" " " " (ditto)		X	X	
3	5/8/97	1:50P		X	X		" " " " (ditto)		X	X	
4	5/8/97	1:50P		X	X		" " " " (ditto)		X	X	
							OF WATER BLANK (NOT LABELED)		X	X	

SHADED AREA—FOR LAB USE ONLY: Lab 1 # / Lab 2 # / Quote # / Kit Request # / Project # / No. of Samples / Page of / COO Rev / Login / LIMS Ver / Ack Gen / LAB ID / REMARKS

107 of 1

Sampled By & Title (Please sign and print name): B. DEEM [signature] — Date/Time: 5/8/97 1:48-1:50 pm

Relinquished By (Please sign and print name): OVERNIGHT DELIVERY — Date/Time

Received By (Please sign and print name): R. CRESSMAN [signature] 3M — Date/Time: 5/9/97 10:00

Relinquished By (Please sign and print name) — Date/Time

Received By (Please sign and print name) — Date/Time

Relinquished By (Please sign and print name) — Date/Time

Received By (Please sign and print name) — Date/Time

Shipped Via: UPS BUS Fed-Ex Hand Other______ — Shipping #

Work Authorized By (Please sign and print name) — Remarks

HAZWRAP/NESSA: Y N — QC Level: 1 2 3 Other — COC Rec. — ICE — Ret Req — TEMP — Cust Seal — Ph

Instructions and Agreement Provisions on Reverse Side

DISTRIBUTION: ORIGINAL - LAB, Yellow - LAB, Pink - Client
REV 11/92 FORM 340

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1mSHIPPING MEMORANDUM0m
(FOR NO-CHARGE SHIPMENTS ONLY)

1m4m 0m

Ship to 4m 3M Environmental Technology & | Date 06/02/97 No. 38-9732010m
4m Service Laboratory, B-2-3E-09 | Routing 0m
4m 935 Bush Avenue | F.O.B. 0m
4m St Paul, Minesota 55144-1000 | Date Shipped 0m
4m Attn: Robert Howell | B/L 0m

4m Quantity	Product Code / Description	Weight 0m
4m 1	Container/Soil sample	30 lbs 0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m

Special Inst 4mShip Overnight 6/2--Must be delivered Tues. AM 0m
Freight Class 4m 0m
4m 0m
4m 0m
4mOriginator VERA L WIGAL Phone 304-863-4895 0m
4mCharge (Cost Code) 8309019511001280 | MSDS Sheets Required? 0m
4mPurchase Order No. | Hazardous Material? N 0m
4mValue for Customs | MATERIAL CLASSIFICATION 0m
Ship From 4mStores, Bldg. 4 | Corrosive Toxic 0m
4m | Flammable Other 0m
4m 0m
4m ** AUTHORIZATION ** |0m
4m |0m
4m |0m
4m |0m
4m

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3MA00230236

ENGINEERING DUPONT COMPUTATION SHEET

TITLE OF PROJECT OR STUDY: DUPONT WASHINGTON WORKS — PROJECT OR STUDY NO.

SUBJECT: PARKERSBURG, WV — WORK NO.

COMPUTED — DATE — 19

"CHAIN OF CUSTODY"

6 8 SAMPLE BOTTLES OF SOIL TAKEN ON 5/30/97
AT 11:00 AM BY BARBARA DEEM AND DAN WEBER.

FOR USE BY ROBERT HEWELL, 3M

Dan Weber

304-863-4415

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USE STANDARDS FOR MINIMUM ESSENTIAL INSTALLATIONS

3MA00230237

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3M Environmental Laboratory
Request Form

Lab Request No. R2008 | Date Received 6/6/97 | Page 1 of 1

Address: 3M Environmental Laboratory, 935 Bush Ave. Building 2-3E-09, St. Paul, MN 55106
Attn: Sample Custodian, Phone (612) 778-6750, Fax (612) 778-6176

Requester: Name DLB/RDJ | Phone No. | Location | Date Needed ☐ Routine | Past Date | Project Level 3 D S | Proposed Completion Date 12-31-97

Project Description (40 Characters Max): Dupont Soil and Groundwater

Is this a TSCA (40) project? (New book entries/ transactions) MUST BE CHECKED ☐ Yes ☒ No | Dept. No. (Mnth) | Sub. Acct. | Cost/Job/Project No. See comments | Budget | Planning Source CO | Quality Review | Data Shipped

Purpose of Testing: ☐ RCRA ☐ TSCA ☐ CWA ☐ CAA ☐ SDWA ☐ MSDS ☒ Customer Service | Class/Priority | Data Shipped

Billing Comments/Special Handling: Comments: Dupont Washington Works samples for FC-143 analysis. Split charge 50:50 between 3058 & 3048 BIOENVIRO.

Sample No. (Range 0-999)	1	2	3	4	5
Sample Code (12 Character Max)					DI Blank
Sample Date & Time (MM/DD/YY, HH:MM)	5/8/97 1:45 pm	5/8/97 1:45	5/8/97 1:50	5/8/97 1:50	
Sample Description (12 Character Max)	DUPT. WASH Water				
3M I.D. (11 Character Stock No.)					
Class Number / Compatible Code					
CLP Species					
Class					
Analy. Date (Optional)	6/6 13-31-97				

1mSHIPPING MEMORANDUM0m
(FOR NO-CHARGE SHIPMENTS ONLY)

1m4m 0m

Ship to 4m 3M Environmental Technology & | Date 06/26/97 No. 38-9737950m
4m Service Laboratory, B-2-3E-09 | Routing 0m
4m 935 Bush Avenue | F.O.B. Date 0m
4m St. Paul, Minnesota 55144-1000 | Shipped 0m
4m Attn: Robert Howell | B/L 0m

4m Quantity	Product Code / Description	Weight 0m
4m 7	Coolers containing water samples/water & ice	40 each 0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m
4m		0m

Special Inst 4mNeed 7 labels, Overnight Air, Deliver 6/27 before noon 0m
Freight Class 4m 0m
4m 0m
4m 0m

4mOriginator VERA L WICAL Phone 304-863-4895 0m
4mCharge (Cost Code) 8309066742001280 | MSDS Sheets Required? 0m
4mPurchase Order No. | Hazardous Material? N 0m
4mValue for Customs | MATERIAL CLASSIFICATION 0m
Ship From 4mStores, Bldg. 4 | Corrosive Toxic 0m
4m | Flammable Other 0m

4m 0m
4m
4m| ** AUTHORIZATION ** |0m
4m| |0m
4m| |0m
4m| |0m

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3MA00230239

CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

CH2MHILL Analytical Services

☐ LMAO 2567 Fairborn Drive
Montgomery, AL 36116-1862
(334) 271-3444 FAX (334) 271-3428

☐ LRD 8030 Caterpillar Road
Redding, CA 96002-1412
(916) 244-6227 FAX (916) 244-4109

☐ LKW Clarkto Analytical Laboratories, Inc.
50 Bathurst, Unit 12, Waterloo, Ontario, Canada N2V 2C5
(519) 747-8976 FAX (519) 747-8906

☐ CVO 2300 NW Walnut Boulevard
Corvallis, Oregon 97330-9636
(541) 752-4271 FAX (541) 752-0276

COC # _____

Project #

Purchase Order #

Project Name: DuPONT WASHINGTON WORKS

Company Name: DuPONT

Project Manager or Contact & Phone #: (304) 992-6820 ANDREA HARTEN

Report Copy to:

Requested Completion Date:

Site ID

Sample Disposal: Return ☐ Lab ☐

Matrix: Type — Water / Soil / Air

Sampling: Date / Time

CLIENT SAMPLE ID (9 CHARACTERS)

LAB QC

Preservative

Requested Analytical Method #

| | | |

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A. Kathy Swanda.
Q. During the time that you were a Pollution Control Specialist I, did you have any involvement of any kind with 3M?
A. No.
Q. In approximately 1988 or so, when you became a Pollution Control Specialist II, did your location within MPCA change?
A. No.
Q. You continued to work out of the St. Paul office, correct?
A. Correct.
Q. And was your immediate supervisor the same?
A. No.
Q. Who did you report to at that point?
A. Wayne Anderson.
Q. How, if at all -- what were your particular responsibilities as a Pollution Control Specialist I?
A. I wrote and edited the 305(b) report, which is a requirement under the Clean Water Act. And I also collected water samples for compliance purposes for wastewater treatment plants. And I prepared fish tissue as well as other tissue for analysis.

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Q. And, again, none of the water samples you collected was from any 3M facility; is that correct?
A. No.
Q. Did you ever collect any water samples -- first of all, were these surface water samples?
A. Correct.
Q. When you became a Pollution Control Specialist II, how, if at all, did your responsibilities change?
A. I moved to the, what's called the non-point source pollution unit, again focused primarily with surface water, but there was always a groundwater component. And I reviewed local water plans for consistency with state rules and regulations.
Q. At any time while you worked for MPCA between 1986 and approximately 1990, the point when you started working for the Metropolitan Council, did you have any contact with a facility in Minnesota in Washington County known as the Oakdale disposal site?
A. I did not.
Q. Did you -- during that same period of time, did you have any contact or involvement in any way with a facility in Washington County, Minnesota, known as the Washington County Landfill?
A. I did not.
Q. During that same period of time, did you have any involvement or contact in any way with a facility known as the Woodbury Landfill?
A. I did not.
Q. During that point in time, that same point in time between 1986 and approximately 1990, while you were at MPCA, did you have any involvement or contact in any way with a facility known as the 3M Chemolite plant or Cottage Grove plant?
A. I did not.
Q. Why did you leave MPCA to begin working for the Metropolitan Council?
A. At the time, the Metropolitan Council was engaged in an effort focused on long-term water supply planning for the metro region. And as a geologist I was very interested in that. And it also allowed me to use some of the learning that I had gleaned while at the Pollution Control Agency. So to help the

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Page 44

planning effort.
Q. The Metropolitan Council, this was within the Twin Cities area of Minnesota?
A. Correct, yes. It's a regional planning authority.
Q. Was the pay better at the Metropolitan Council?
A. I believe so.
Q. Had you had any negative reviews by anyone at the MPCA prior to going to the Metropolitan Council?
A. No.
Q. Did you have any difficulties with any of the individuals you were working with at MPCA that led to you going to the Metropolitan Council?
A. No.
Q. All right. While you were at the met -- well, let me back up on moment.
Did you ever intern with 3M at any point in time while you were at college?
A. No.
Q. While you were at the Metropolitan Council, did you ever have any involvement or contact with representatives of 3M on any issues?

CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

CH2MHILL Analytical Services

LMG 2567 Feltens Drive, Montgomery, AL 36116-1662, (334) 271-1444 FAX (334) 271-3428
LRD 6680 Caterpillar Road, Redding, CA 96002-1412, (916) 244-6227 FAX (916) 244-4109
LWW Corvallis Analytical Laboratories, Inc., 40 Bertrand Unit 12, Waterloo, Ontario, Canada N2V 2C6, (519) 747-3678 FAX (519) 747-3806
CWO 2560 NW Walnut Boulevard, Corvallis, Oregon 97330-3538, (541) 752-4271 FAX (541) 752-4276

COC # _____

Project #

Purchase Order #

Project Name: DUPONT WASHINGTON WORKS

Company Name: DUPONT

Project Manager or Contact & Phone #: (302) 992-6820 ANDREW HUTTON

Report Copy To:

Requested Completion Date:

Site ID

Sample Disposal: Return / Dispose

Matrix: SOLID / AQUEOUS

Type: COMP / GRAB

Sampling: Date / Time

CLIENT SAMPLE ID (8 CHARACTERS)

LAB QC

Preservative

Requested Analytical Method #

C-8
C-7
SURFACTANTS
METHANE
NITRATE
SULFIDE
SULFATE

THIS AREA FOR LAB USE ONLY

Lab #

Page 01

Lab PM

Custody Review

Custody Seals Y N

Ice Y N

pH

QC Level 1 2 3 Other

Cooler Temperature

Alternate Description

Lab ID

Sampling: Date	Time	Type	Matrix	CLIENT SAMPLE ID (8 CHARACTERS)	LAB QC	Preservative	C-8	C-7	SURFACTANTS	METHANE	NITRATE	SULFIDE	SULFATE
6/26/07	1115	GRAB	AQUEOUS	MW-4		2	X	X	X	X	X	X	X

Sampled By & Title: KEN WESLEY

Phone (sign and print name)

Received By / Date/Time: 6/26/07 1600

Relinquished By / Date/Time

Phone (sign and print name)

Received By / Date/Time

Relinquished By / Date/Time

Phone (sign and print name)

Received By / Date/Time

Shipped Via: UPS Fed-Ex Other

Shipping #

Special Instructions:

Instructions and Agreement Provisions on Reverse Side

DISTRIBUTION: Original – LAB, Yellow – LAB, Pink – Client

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3MA00230241

CH2MHILL Analytical Services

LMG 2567 Fairlane Drive, Montgomery, AL 36116-1602, (334) 271-3444 FAX (334) 271-3428
LRD 5600 Corporate Road, Raleigh, NC 27606-3441, (919) 233-8091 FAX (919) 233-8485
LKW Corning Analytical Laboratories, Inc., 50 Bathurst, Unit 12, Waterloo, Ontario, Canada N2V 2C5, (519) 747-8979 FAX (519) 747-8906
CYG 2300 NW Walnut Boulevard, Corvallis, OR 97330, (541) 753-4217 FAX (541) 752-0276

COC # _____

Project # | Purchase Order #

Project Name: DUPONT WASHINGTON WORKS
Company Name: DUPONT
Project Manager or Contact & Phone # (301) 990-6822
Report Copy to:
Requested Completion Date: ANDREW HALTEN
Site ID
Sample Disposal: Dispose ☒ Return ☐

TURN-AROUND TIME: 10 / 4 / 2 / 1

Requested Analytical Method #

Sampling Date	Sampling Time	Type: COMP	Type: GRAB	Matrix: WATER	Matrix: AIR	CLIENT SAMPLE ID (9 CHARACTERS)	LAB QC	Preservative	C-8	C-7	SURFACTANTS	METHANE	NITRATE	SULFATE	SULFIDE
6/24/97	0830		X		MW-1			2	X	X	X	X	X	X	X

Sampled By A Title: RANDY LEE, GEOLOGIST (Please sign and print name)
Relinquished By / Date/Time: RANDY LEE 6/24/97 1600 (Please sign and print name)
Received By / Date/Time: (Please sign and print name)
Relinquished By / Date/Time: (Please sign and print name)
Received By / Date/Time: (Please sign and print name)
Shipped Via: UPS Fed-Ex Other
Shipping #

THIS AREA FOR LAB USE ONLY

Lab # | Page | of
Lab PM | Custody Review
pH | Custody Seals Y N | Ice Y N
QC Level 1 2 3 Other
Cooler Temperature
Alternate Description | Lab ID
Date/Time: 6/26/97 1600

Special Instructions:

Instructions and Agreement Provisions on Reverse Side

DISTRIBUTION: Original – LAB, Yellow – LAB, Pink – Client

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3MA00230242

CH2MHILL Analytical Services

LLNRS 2061 Fairview Lane
Montgomery, AL 36117-1622
(334) 271-1444 FAX (334) 271-0428

LLRS 5000 Chesapeake Road
Richmond, CA 94804-1412
(510) 234-3227 FAX (510) 234-1100

LLNRS Lakeview Technology, Inc.
50 Bastrop, LRN 78602-4240
(512) 321-3276 FAX (512) 321-3276

LLNRS Limited Partnership Laboratories, Inc.
50 Bastrop, LRN 78602-4240
(512) 321-3276 FAX (512) 321-3276

LLNRS Limited Partnership Laboratories, Inc.
50 Bastrop, LRN 78602-4240
(512) 321-3276 FAX (512) 321-3276

CHD 2000 NW Nimitz Boulevard
Corvallis, OR 97330-4580
(541) 752-4271 FAX (541) 752-0276

COC # _____

Project #

Purchase Order #

Project Name: Robert Washington Wells

Company Name: Robert

Project Manager or Contact & Phone #: (502) 426-6820 Andrew Hazelton

Report Copy to:

Requested Completion Date: Wks ID

Sample Disposal: Return ☑ Return ☐

Type: Matrix

Sampling: Date / Time

CONTAINER: GW / SOIL / AIR / OTHER

CLIENT SAMPLE ID (9 CHARACTERS)

LAB QC

Preservative

Requested Analytical Method #

C-8
C-7
SURFACTANTS
METHANE
NITRATE
SULFATE
SULFIDE

THIS AREA FOR LAB USE ONLY

Lab #

Page / of

Lab PM

Custody Review

pH

Ice Y N

Custody Seals Y N

QC Level 1 2 3 Other_____

Cooler Temperature

Alternate Description

Lab ID

| Date | Time | | |

CH2MHILL Analytical Services

Project #

Project Name: Pulent Washington Works

Company Name: Pulent

Project Manager Contact & Phone #: (302) 892-6028 Andrew Hatten

Report Copy to:

Requested Completion Date:

Site ID

Sample Disposal: Dispose ☒ Return ☐

Purchase Order #

FA-10-1

Requested Analytical Method #

| Sampling Date | Sampling Time | Matrix | Type | CLIENT SAMPLE ID (9 CHARACTERS) | LAB QC | Preservative | C-8 | C-7 | SURFACTANTS | NITRATE | METHANE | SULFATE | SULFIDE | | |

CH2MHILL Analytical Services

LMG 2567 Parkens Drive
Montgomery, AL 36116-1622
(334) 271-1444 FAX (334) 271-3428

LRD 5000 Centerville Road
Redding, CA 96002-1412
(916) 244-5227 FAX (916) 244-4109

LNW Garrison Analytical Laboratories, Inc.
50 Burr Oak Drive, Waterloo, Ontario, Canada N2V 2C8
(519) 740-5975 FAX (519) 747-0606

CHO 2520 NW Walnut Boulevard
Corvallis, Oregon 97330-3638
(541) 752-4271 FAX (514) 752-0276

Project #

Purchase Order #

Project Name: DuPont Washington Works

Company Name: DuPont

Project Manager or Contact & Phone #: (304) 422-6820 Andrew Hacketew

Report Copy to:

Requested Completion Date:

Site ID

Sample Disposal: Dispose ☒ Return ☐

LAB QC

Matrix: Water / Soil / Air

Type: Grab / Comp

Sampling: Date / Time

CLIENT SAMPLE ID (9 CHARACTERS)

Preservative

Requested Analytical Method #

C-8
C-7
SURFACTANTS
METHANE
NITRATE
SULFIDE
SULFATE

Lab #

Lab PM

pH

QC Level 1 2 3 Other

Cooler Temperature

Alternate Description

Lab ID

THIS AREA FOR LAB USE ONLY

COC #

Page / of

Custody Seals Y N / Ice Y N

Custody Review

Date	Time	Grab	Comp	Water	Soil	Air	Type	Matrix	CLIENT SAMPLE ID (9 CHARACTERS)	LAB QC	Preservative	Lab #	Alternate Description	Lab ID
6/24/92	1150	XX		MW-5						2	X X X X X X X			

Sampled By & Title: LOU WESLEY

Sampled By (Please sign and print name)

Date/Time: 6/24/92

Relinquished By (Please sign and print name)

Date/Time: 6/24/92 1400

Received By (Please sign and print name)

Date/Time: 6/25/92

Relinquished By (Please sign and print name)

Date/Time

Received By (Please sign and print name)

Date/Time

Shipped Via: UPS / Fed Ex / Other

Shipping #

Special Instructions:

Instructions and Agreement Provisions on Reverse Side

DISTRIBUTION: Original – LAB, Yellow – LAB, Pink – Client

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3MA00230245

3M Environmental Laboratory
Request Form

Lab Request No. R2148 | Date Received | Page 1 of 3

Address: 3M Environmental Laboratory, 935 Bush Ave., Building 2-3E-09, St. Paul, MN 55106
Attn: Sample Custodian, Phone (612) 778-6750, Fax (612) 778-6176

Name: DLB/RDH | Requestor | Phone No. | Data Needed ☐ Routine ☐ | Environmental Lab Approved Completion Date: 9/1/97

Project Code/Error (10 Characters Max.): Plymouth Wells | Location | Phone Code | Project Lead: JDJ | Planning Status | Quality Review

Is this LTGA RI/O project? (See back side for instructions): MUST BE CHECKED ☐ Yes ☐ No | Chck. No. (Main): 3048 | Sub. Acct. | Client/Job/Project No.: 81GEN | Client Priority | Data Shipped

Purpose of Testing: ☐ RCRA ☐ TSCA ☐ CWA ☐ CAA ☐ SDWA ☐ MSDS ☐ ____

Safety Concerns/Special Handling | Comments: Two samples of each.

Sample No. (Range 0-999)	1	2	3	4	5
Sample Code (12 Character Max.)	MW-1-1	MW-1-2	MW-2-1	MW-2-2	MW-3-1
Sample Date & Time (MM/DD/YY, HH:MM)	6/26/97	6/26/97	6/26/97	6/26/97	6/26/97
Sample Description (12 Character Max.)	MW-1 1 of 2	MW-1 2 of 2	MW-2 1 of 2	MW-2 2 of 2	MW-3 1 of 2
3M I.D. (11 Character Stock No.)					
Task Required: 503-SPEC					
Computer Code					
Code					
Anal. Date: 7/29/97					
Due Date: 8/29/97					

dated per Robert

Copies To:

Form 10520 - P - PINK | White - Environmental Lab Archives | Pink - Contractor | Canary - Disbursements

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3MA00230246

3M Environmental Laboratory
Request Form - Continued

Form 16690 • 11 • C • PWO

3M Environmental Laboratory
Building 2-3E-09
935 Bush Ave.
St. Paul, MN 55106

Lab Request No. R2148
Date Received
Page 2 of 2

Safety Considerations/Special Handling

Comments

Sample No. (Range 0-999)	6	7	8	9	10
Sample Code (12 Character Max)	MW-3-2	MW-4-1	MW-4-2	MW-5-1	MW-5-2
Sample Date (MM/DD/YY)	6/26/97	6/26/97	6/26/97	6/26/97	6/26/97
Sample Description (12 Character Max)	MW-3 2 of 2	MW-4 1 of 2	MW-4 2 of 2	MW-5 1 of 2	MW-5 2 of 2
3M I.D. (11 Character Stock No.)					
Tests Required					
Computer Code					
Date					
Time					
JDJ-SYEE					

White - Environmental Lab Archives
Pink - Contractor
Canary - Requestor

3MA00230247

1mSHIPPING MEMORANDUM0m
(FOR NO-CHARGE SHIPMENTS ONLY)

1m4m

Ship to 4m 3M Environmental Technology &
4m Service Laboratory, B-2-3E-09
4m 935 Bush Avenue
4m St. Paul, Minnesota 55144-1000
4m Attn: Robert Howell

Date 06/24/97 No. 38-973740

Routing

F.O.B.
Date Shipped

B/L

Quantity	Product Code / Description	Weight
4m 1	Cooler of soil samples/soil & ice	100 lbs

Special Inst 4mOvernight-EAGLE USA AIR, Deliver Before NOON

Freight Class 4m

4mOriginator VERA L WIGAL Phone 304-863-4895

4mCharge (Cost Code) 83090667420O1280

4mPurchase Order No.

4mValue for Customs

Ship From Stores, Bldg. 4

MSDS Sheets Required?

Hazardous Material? N

MATERIAL CLASSIFICATION

Corrosive Toxic

Flammable Other

** AUTHORIZATION **

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3MA00230248

CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

CH2MHILL Analytical Services

Montgomery, AL 36116-1822 (334) 271-1444 FAX (334) 271-3422
LRD 5000 Caterpillar Road Redding, CA 96003-1412 (916) 244-6227 FAX (916) 244-4109
LIMIT-LIMITED ANALYTICAL LABORATORIES, INC. 80 Bathurst, Unit 12, Waterloo, Ontario, Canada N2V 2G5 (519) 747-2876 FAX (519) 747-2906
CVO 2300 NW Walnut Boulevard Corvallis, Oregon 97330-3538 (541) 758-4271 FAX (541) 758-0276

COC # _____

Project #

Purchase Order #

Project Name: DuPont Washington Works, C-8 Study

Company Name: DuPont Environmental Comps)

Project Manager or Contact & Phone #: 304-424-3849

Report Copy to: SAME

Requested Completion Date: SEE CONTACT

Billing ID: MW-1

Sample Disposal: Return ☒ Archive ☐

Sampling: Date, Time

Type: Grab (G) / Composite (C)

Matrix: Water (W) / Soil (S) / Air (A) / Other (O)

CLIENT SAMPLE ID (9 CHARACTERS)

LAB QC

Preservative

Requested Analytical Method #

C-8
C-7
SURFACTANTS (e.g., TBOM...)
METHANE
NITRATE
SULFATE
SULFIDE

THIS AREA FOR LAB USE ONLY

Lab #

Page: 01

Lab PM

Custody Seals: Y N

Custody Review

pH

Ice: Y N

QC Level: 1 2 3 Other

Cooler Temperature: 3.3°C

Alternate Description

Lab ID

Date	Time				CLIENT SAMPLE ID	LAB QC	C-8	C-7	SURFACTANTS	METHANE	NITRATE	SULFATE	SULFIDE
11/21/97	1550	X	X	S	S-11-02-2		X	X	X	X	X	X	X
"	1559	X	X	S	S-11-01-6								
	1610	X	X	S	S-11-03-10								
	1620	X	X	S	S-11-12-14								
	1630	X	X	S	S-11-116-18								
	1639	X	X	S	S-11-20-22								
	1645	X	X	S	S-11-24-26								
↓	1650	X	X	S	S-11-28-30		↓	↓	↓	↓	↓	↓	↓

Relinquished By: Edward B. Welch / Edward B. Welch — Date/Time: 11/21/97 1600

Received By: Pat Fredette — Date/Time: 11/22/97 1420

Relinquished By: Pat Fredette — Date/Time: 11/24/97 1600

Received By: _____ — Date/Time: _____

Shipped Via: UPS / FedEx / Other _____ — Shipping #

Special Instructions:

Printed By & Title: Edward B. Welch / Project Leader — Signature: _____

Printed By & Title: Pat Fredette / Lab Tech — Signature: _____

Instructions and Agreement Provisions on Reverse Side

DISTRIBUTION: Original – LAB, Yellow – LAB, Pink – Client

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3MA00230249

CH2MHILL Analytical Services

Project #

Project Name: WASHINGTON WORKS AUGUST C-8 STUDY

Company Name: AUGUST ENVIRONMENTAL (AEES)

Project Manager: ANGELA HARTER

Phone #: (502) 412-6820

Purchase Order #

Report Copy to: SAME

Requested Completion Date: SEE CONTRACT

Site ID: MW-1

Sampling: Date 6/19/17; Time 1300

Matrix: GW

Type: COMP

Sample Disposal: Dispose; Return

CLIENT SAMPLE ID (9 CHARACTERS): SS-1-312-34

LAB QC: 1

Preservative: X

Requested Analytical Method

C-8

C-7

SURFACTANTS (e.g. TRITON...)

METHANE

NITRATE

SULFATE

SULFIDE

Page 1 of 1

COC #

THIS AREA FOR LAB USE ONLY

Lab #

Lab PM

Custody Seals Y N

Custody Review

pH

Ice Y N

QC Level 1 2 3 Other

Cooler Temperature 33°F

Alternate Description

Lab ID

Sampled By & Title: EDWARD BURGESS, GEOLOGIST

Date/Time: 6/20/17 1600

Relinquished By: PHIL BRADLEY

Date/Time: 6/20/17 1600

Received By: PHIL BRADLEY

Date/Time: 6/20/17 1600

Shipped Via: UPS Fed-Ex Other

Shipping #

Special Instructions:

Instructions and Agreement Provisions on Reverse Side

DISTRIBUTION: Original – LAB; Yellow – LAB; Pink – Client

LOW Centro Analytical Laboratories, Inc.
60 Belhaven, Unit 12, Waterloo, Ontario, Canada N2V 2C4
(519) 747-8575 FAX (519) 747-8580

CHEMTECH
2200 Marcus Avenue
Suite 100E
Lake Success, NY 11042-1019
(516) 326-1622
(631) 777-1444 FAX (631) 271-0428

Page 45

A. I don't recall. I don't believe so.
Q. Why did you leave the Metropolitan Council to begin working in Seattle in approximately 1992?
A. Personal reasons. My husband and I had grown up in the St. Croix River Valley, which is where we lived. And we thought it would be interesting to live in a new place. And Seattle was very inviting from that regard. And, again, the position that King County had open was very attractive to me from a learning perspective.
I had spent a fair amount of time, as you have seen, working in water planning. And in the early '90s that particular region, the Puget Sound region, was and continues, I believe, to experience explosive growth and changes in land use, which have considerable impact on surface water resources. And the position that I was -- that I took at King County was engaged in trying to come up with engineering -- innovative engineering ideas to ameliorate the pollution concerns that were the result of the explosive growth, in particular, trying to save the native salmon

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runs in that area.
Q. Was there an increase in pay when you went to the position in Seattle?
A. No.
Q. Was it a decrease in pay?
A. It wasn't a decrease, but the cost of living was quite substantially higher in Seattle.
Q. And why did you leave the position in Seattle to begin working for Northern Environment Technology back in Minnesota in 1994?
A. Again, personal and professional reasons. I had had a baby while we were in Seattle, our first child. And we had no family or support system. And so it just became more -- it became more important for us to be back in the midwest after we had our son.
And I also had worked for government for quite some time at various different levels, state, regional and local level, and was ready to try the private sector.
Q. Was there an increase in pay going to Northern Environmental Technology?
A. Yes, but not substantial.
Q. And was this a private consulting firm?
A. It is a private consulting firm, yes.

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Q. And during the time that you were working for that consulting firm, did you provide any services to the 3M Company?
A. I did not, although I did provide services to the City of Woodbury.
Q. And what services did you provide to Woodbury?
A. Well field analysis.
Q. And this is the City of Woodbury, Minnesota, correct?
A. Correct.
Q. What kind of well field analysis did you provide?
A. Looking at their well spacing and in their existing well field.
Q. Are you referring to their drinking water well?
A. Correct.
Q. Why was the City of Woodbury, to your knowledge, looking at that issue at that point in time?
A. The City of Woodbury, at that time and in the current time, is, again, undergoing quite expansive growth. And every city in their municipal systems have to make sure that they

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have enough water for their planned capacity, for the -- the plan that they have in their comprehensive efforts, planning efforts and that's why we took a look at it.
Q. So the City of Woodbury retained Northern Environmental Technologies to do this well work; is that correct?
A. Uh-huh.
Q. And what in particular was Northern Environmental Technologies asked to do for the City?
A. Just look at the spacing between their municipal wells to make sure that there wouldn't be any significant drawdown as a result of any further growth.
Q. While you were at Northern Environmental Technologies, did you personally work on that project?
A. I did.
Q. And were, to your knowledge -- let me restate that.
To your knowledge, was Northern Environmental Technologies asked to do any sampling of the water Woodbury water wells for any contaminants of any kind?

2100 3rd Avenue North, Suite 960*Birmingham, Al 35203*
1-800-888-DEPO

CONFIDENTIAL - SUBJECT TO A PROTECTIVE ORDER ENTERED IN HENNEPIN COUNTY DISTRICT COURT, NO. 27-CV-10-28862

3M Environmental Laboratory

Request Form

Address	3M Environmental Laboratory 935 Bush Ave. Building 2-3E-09 St. Paul, MN 55106
Attn:	Sample Custodian
Phone	(612) 778-6750
Fax	(612) 778-6176

Name: D. Bonn

Project Description (40 Characters Max.): DuPont Washington Works CB Study

Location (Requested By): Phone No. (Requester): Date Received: 8/5/94

Lab Request No.: R2582

Page: 1 of 2

Data Package Required: ☐ Routine ☐

Project Code: RDH4282

Project Lead: Environmental Lab

Requested Completion Date: 8/29/94

Purpose of Testing: ☐ RCRA ☐ TSCA ☐ CWA ☐ CAA ☐ SDWA ☐ MSDS ☐

Is this a TSCA Bird project? (See Sheet 4 for Instructions): ☐ Yes ☒ No

MUST BE CHECKED

Billing Ref. No. (Work): 3058

Billing Acct.:

Charge Code/Project No.: BIGENENVIRO

Charge Party:

Priority: Overnight

Date Shipped:

Sample Goodness/Special Handling:

Comments:

Sample No. (Range 0-999)	1	2	3	4	5
Sample Code (12 Character Max.)	MW-1 Sinkhole				
Sample Date & Time (MM/DD/YY, HH:MM)	4/23/97 1550	4/23/97 1559	4/23/97 1610	4/23/97 1620	4/23/97 1630
Sample Description (12 Character Max.)	0-2'	4-6'	8-10'	12-14'	16-18'
3M I.D. (11 Character Stock No.)					
Container Code					
Cub					
Analysis Due Date					
Oxy-Ex-Scan	8/26				
Semi-Volatiles BNA Pest	8/26				
CH3	8/13				
NO2-N	8/13				
BOD					
SO4	✓				
Soil-Prep	8/13				
CEC-NH4	8/13	8/27			
Soil-Moist	8/26	8/13			
pH-H2O	8/13	8/27			
pH-CaCl2	✓	✓			
Ext-Comb	8/26	8/27			

Copies To:

10650 • P • PWO White - Environmental Lab Archive Pink - Contractor Canary - Disbursements

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3M Environmental Laboratory
Request Form - Continued

Form 16830 - 14-C-Pink

3M Environmental Laboratory
Building 2-3E-09
935 Bush Ave.
St. Paul, MN 55106

Lab Request No. R2282
Page 2 of 2

Safety Concerns/Special Handling

Comments

Sample No. (Range 0-999)	6	7	8	9	10	11
Sample Code (12 Character Max)	MW-1 SB-01					
Sample Date (MMDDYY)	4/23/97	4/23/97	4/23/97	4/23/97	4/23/97	4/23/97
Sample Description (12 Character Max)	20-22'	24-24'	29-30'	32-34'	36-38'	38-40'
3M I.D. (11 Character Stock No.)	1459	1495	1450	1400	1477	1478
Tests Required						
Computer Code						
Chrb						
AV Date						
ID Date						

Same as Page 1

3M - Environmental Lab Address

Pink - Contractor

Canary - Requester

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