

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*

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*Corrigan*  
EXHIBIT NO. *10*  
DATE *8-27-08*  
*Kelley, York & Associates*

**Exhibit  
2759**

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

3M Environmental Laboratory

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

3M L.R. No.	DuPont Smp. No.	Matrix	POAA <sup>(1)</sup> , ppb	Vol. & Semi-Vol. F, Cl, Br by AED, ppm
R2008-1	1	ground water	52	< MQL <sup>(2)</sup>
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. <sup>(3)</sup>	< 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

3d

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

3M LR No.	DuPont Smpl. No.	POAA <sup>TM</sup> mg/L	Total Fluorine mg/L	Fluoride Ion mg/L	Organic F, mg/L <sup>(1)</sup>	Absorb. Organic F, mg/L	Vol. & Semi-Vol. F by AED, ppm	Trichloroethylene mg/L	Acetone mg/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	
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	
R2148-8	MW-4-2	0.0590	4.2	0.11	4.1	0.14	0.20 < F < 2.5	670	10
R2148-9	MW-5-1	< POL <sup>TM</sup>	3.0	< 0.10	2.9	0.11	F < 0.05	130	
R2148-10	MW-5-2	< POL <sup>TM</sup>	2.8	< 0.10	2.7	0.14	0.05 < F < 0.20	140	
R2148-11	MW-6-1	< MDL <sup>TM</sup>	2.3	0.10	2.2	< 0.05	F < 0.05		
R2148-12	MW-6-2	< MDL <sup>TM</sup>	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-trichloroethane µg/L	Trichloroethene µg/L	Tetrachloroethene µg/L	Trichlorofluoromethane µg/L
R2148-1	MW-1-1	59						16
R2148-2	MW-1-2	49				140		18
R2148-3	MW-2-1					150		26
R2148-4	MW-2-2			5.5	14	520	5	26
R2148-5	MW-3-1			5.9	14	570	5.2	5.9
R2148-6	MW-3-2		5.8	37	6.3	81		
R2148-7	MW-4-1		5.9	39	6.2	86		
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 = 0.0256 mg/L (4) Total Fluorine - Fluoride Ion - Organic Fluorine

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

3M L/R No.	DuPont Sample No.	* Total Fluoride, mg/kg	POAA <sup>01</sup> , mg/kg	Sulfate, mg/kg	Sulfite, mg/kg	Nitrite, mg/kg	pH in water	pH in CaCl <sub>2</sub>	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'	39,500	13.1	100	<2	<0.10	5.8	5.2	13.1	13.6
R2382-10	SS-1 36-38'	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  
 KSH  
 fax 3/13/98

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**3M Environmental Laboratory**

**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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**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 kjh on 10/21/97
IDDJ: QAU (Archives): LIRN System: Project Manager: Sue Beach		
Others (List Recipients / Address / Phone / FAX) T. DiPasquale, 22-11E-03; 3-1891; 736-3257		Sent by: / Date 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  
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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 perfluorooctanoic 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		
R2008-1-2	water	2	1.3	52	52	0
R2008-2-1	water	2	1.3	49		
R2008-2-2	water	2	1.3	49	49	0
R2008-3-1	water	2	1.3	52		
R2008-3-2	water	2	1.3	52	52	0
R2008-4-1	water	2	1.3	57		
R2008-4-2	water	2	1.3	49	53	6
R2008-5-1	water	2	1.3	n.d.*		
R2008-5-2	water	2	1.3	n.d.*	n.a.	n.a.

\* 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/H<sub>2</sub>O. 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/H<sub>2</sub>O (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 vials with HPLC-grade ACN and milli-Q water.



### 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/H<sub>2</sub>O (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 nonextracted calibration curves using the following formula:

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

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:

$$C_p = (C_s * D)^*E \quad (5)$$

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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**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:

$$\%R = [(C_1 - C_2) / C_3] * 100\% \quad (6)$$

where,

%R = Percent recovery of POAA

C<sub>1</sub> = Concentration of POAA found in MS/MSD (µg/mL)

C<sub>2</sub> = Native concentration of POAA in sample before dilution adjustment (µg/mL)

C<sub>3</sub> = 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:

$$C_1 = 0.057 \text{ } \mu\text{g/mL};$$

$$C_2 = 0.020 \text{ } \mu\text{g/mL};$$

$$C_3 = 0.052 \text{ } \mu\text{g/mL};$$

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

*Matrix spike  
with native  
duplicate*

**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  
 Analytic: GML/gh  
 Report preparation: jph

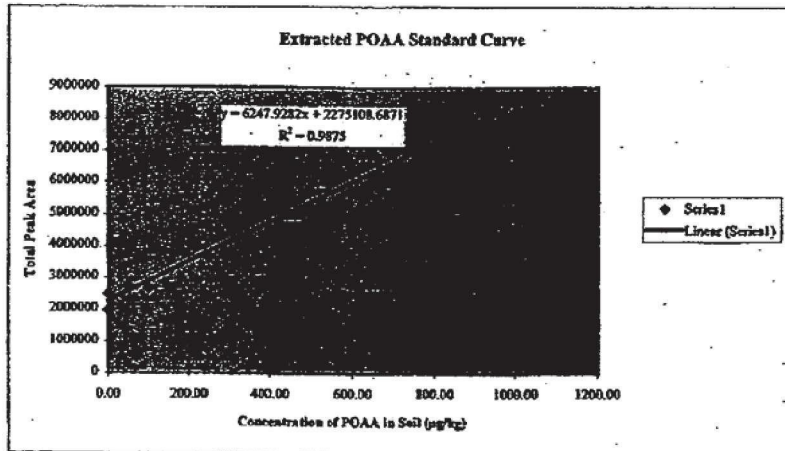
R-2008 DuPont Soil Data  
POAA Standard Curve and Data Table

DATA TABLE 1.0

Spiked Standard Conc. of POAA in Extract (µg/mL)	Volume of Extract (mL)	Spiked Amount of 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 (µg/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:

$$\begin{array}{ccccccc}
 \left| \begin{array}{c} \text{Conc. of POAA, from} \\ \text{spiked standards,} \\ \text{in the extract} \end{array} \right| & \times & \left| \begin{array}{c} \text{Volume} \\ \text{of} \\ \text{Extract} \end{array} \right| & \times & \left| \begin{array}{c} \text{Mass} \\ \text{of} \\ \text{Soil} \end{array} \right| & \times & \left| \begin{array}{c} \text{Convert} \\ \text{g to} \\ \text{kg} \end{array} \right| & = & \left| \begin{array}{c} \text{Conc. of} \\ \text{POAA in} \\ \text{Soil} \end{array} \right| \\
 \frac{\mu\text{g}}{\text{mL}} & \times & \frac{\text{mL}}{1} & \times & \frac{1}{\text{g}} & \times & \frac{1000 \text{ g}}{\text{kg}} & = & \frac{\mu\text{g}}{\text{kg}}
 \end{array}$$

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 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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3M Environmental Laboratory

**POAA Analyses in Groundwater (6/97 Samples)**

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**3M Environmental Laboratory**

**Data Transmittal Summary**  
Final

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

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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 $\mu\text{g/mL}$ (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	<PQL	<PQL
R2148-9-2 (MW-5)	23264	2	<PQL	<PQL	<PQL
R2148-10-1 (MW-5)	21873	2	<PQL	<PQL	<PQL
R2148-10-2 (MW-5)	26025	2	<PQL	<PQL	<PQL
R2148-11-1 (MW-6)	11696	2	<MDL	<MDL	<MDL
R2148-11-2 (MW-6)	11932	2	<MDL	<MDL	<MDL
R2148-12-1 (MW-6)	9132	2	<MDL	<MDL	<MDL
R2148-12-2 (MW-6)	10595	2	<MDL	<MDL	<MDL

## 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 0.503 µg/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 perfluorooctanoate were analyzed bracketing every ten samples during sample analysis.



### 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

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

Where,

$C_e$  = Concentration of ammonium perfluorooctanoate in extract (µg/mL)

P = Peak area of sample

I = Intercept of the calibration curve

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

$$C_p = C_e \times D$$

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  $C_p = 0.285$  μg/mL  $\times$  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

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

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)

$C_{oc}$  = 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:

$$C_m = 0.404 \text{ } \mu\text{g/mL};$$

$$C_b = 0.00 \text{ } \mu\text{g/mL};$$

$$C_{oc} = 0.503 \text{ } \mu\text{g/mL};$$

$$\text{therefore, } \%R = (0.404 - 0.00) / 0.503 \times 100\% = 80\%.$$

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

Sample Type	Sample ID	Recovered Concentration (µg/mL)	Expected Concentration <sup>1</sup> (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:  
<sup>1</sup> 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

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**3M Environmental Laboratory**

**POAA Analyses in Soil (6/97 Samples)**

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**Court Reporting\*Legal Videography\*Trial Services**

<p style="text-align: right;">Page 5</p> <p>1            I N D E X (cont.)</p> <p>2</p> <p>3        EXHIBITS</p> <p>4</p> <p>5    No. 42 - PFOA DuPont Meeting Talking Points    228</p> <p>6    No. 43 - Corrigan appointment announcement    229</p> <p>7    No. 44 - 7-8-03 E-mail Gebhard to Witer        241</p> <p>8    No. 45 - 3-27-03 Letter O-Grady                244</p> <p>9    No. 46 - 9-10-03 Memorandum Karnowski        247</p> <p>10   No. 47 - 3-3-04 E-mail Santoro                 252</p> <p>11   No. 48 - 4-13-04 E-mail Santoro                 256</p> <p>12   No. 49 - Pioneer Press Article                   264</p> <p>13   No. 50 - 2-17-05 E-mail Siegel                  273</p> <p>14   No. 51 - Toxic Traces Article                   275</p> <p>15   No. 52 - Environmental Working Group Letter    281</p> <p>16   No. 53 - 5-13-05 E-mail Corrigan to Oiaei      286</p> <p>17   No. 54 - Start Tribune Editorial                293</p> <p>18   No. 55 - 10-28-05 E-mail Nelson to Reed       300</p> <p>19   No. 56 - Environmental Working Group 11-18-05 302</p> <p>20   No. 57 - 12-5-05 E-mail Rageth                305</p> <p>21</p> <p>22</p> <p>23        REQUESTS FOR PRODUCTION</p> <p>24    No. 1 - 401(k) information                    22</p> <p>25            KELLEY, YORK &amp; ASSOCIATES, LTD.</p> <p>26            Court Reporting with a Difference                    (316) 267-8200    (800) 654-8684</p>	<p style="text-align: right;">Page 7</p> <p>1            THE VIDEOGRAPHER: We are on the</p> <p>2    record. Here begins tape number one in the</p> <p>3    deposition of Cheryl Corrigan in the matter</p> <p>4    of Gary Paulson, Karen Paulson, William</p> <p>5    Henry, Bradley Krank, plaintiffs versus 3M</p> <p>6    Company, case number C2-04-6309. We are on</p> <p>7    the record, and today's date is February 27,</p> <p>8    2008. The time is 9:04 a.m. The deposition</p> <p>9    is taking place at Foulston Siefkin, 1551</p> <p>10   North Waterfront Parkway, Suite 100, Wichita,</p> <p>11   Kansas 67206.</p> <p>12            Will counsel please identify</p> <p>13   yourselves and state who you represent, and</p> <p>14   the witness will be sworn.</p> <p>15            MR. BILOTT: Rob Bilott for</p> <p>16   plaintiffs. And just a correction, the</p> <p>17   defendant is 3M Company.</p> <p>18            MR. ASHLEY: Cooper Ashley for 3M</p> <p>19   Company.</p> <p>20            MR. ARMSTRONG: Jim Armstrong for</p> <p>21   the witness.</p> <p>22            SHERYL CORRIGAN,</p> <p>23   having been first duly sworn on her oath to</p> <p>24   state the truth, the whole truth, and nothing</p> <p>25   but the truth, testifies as follows:</p>
<p style="text-align: right;">Page 6</p> <p>1            I N D E X (cont.)</p> <p>2</p> <p>3        SIGNATURE OF WITNESS                        322</p> <p>4        CERTIFICATE                                    323</p> <p>5</p> <p>6</p> <p>7</p> <p>8</p> <p>9</p> <p>10</p> <p>11</p> <p>12</p> <p>13</p> <p>14</p> <p>15</p> <p>16</p> <p>17</p> <p>18</p> <p>19</p> <p>20</p> <p>21</p> <p>22            KELLEY, YORK &amp; ASSOCIATES, LTD.</p> <p>23</p> <p>24            Court Reporting with a Difference                    (316) 267-8200    (800) 654-8684</p> <p>25</p>	<p style="text-align: right;">Page 8</p> <p>1    BY MR. BILOTT:</p> <p>2    Q. Good morning.</p> <p>3    A. God morning.</p> <p>4    Q. My name is Rob Bilott. As you just heard,</p> <p>5    I'm one of the attorneys representing the</p> <p>6    plaintiffs in the case just described, which</p> <p>7    is Gary A. Paulson, et al. versus the 3M</p> <p>8    Company, a case currently pending in state</p> <p>9    court in Minnesota.</p> <p>10            Would you state your name, please.</p> <p>11   A. Sheryl Ann Corrigan.</p> <p>12   Q. And are you currently employed?</p> <p>13   A. Yes, I am.</p> <p>14   Q. And who are you employed with?</p> <p>15   A. Koch Industries.</p> <p>16   Q. And is that K-O-C-H?</p> <p>17   A. Yes, it is.</p> <p>18   Q. Is that located here -- the place of your</p> <p>19   employment located here in Wichita, Kansas?</p> <p>20   A. That's correct.</p> <p>21   Q. What is your business address?</p> <p>22   A. I'll get my business card out.</p> <p>23   Q. Okay.</p> <p>24   A. 4111 East 37th Street North, Wichita, Kansas</p> <p>25   67220.</p>

2 (Pages 5 to 8)

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



**3M Environmental Laboratory**

**Data Transmittal Summary**  
Final

Lab Request #: R2382	3M Study #:	Contract Laboratory #
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: <del>water</del> 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
DDJ: QAU (Archives): LIRN System: Project Manager: Sue Beach		
Others (List Recipients / Address / Phone / FAX) T. DiPasquale, 22-11E-03; 3-1891; 736-3257		Sent by: / Date 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  
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 (ESI/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/H<sub>2</sub>O (1:1)  
Flow rate: 60 µL/min  
Injection volume: 15 µL  
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/gb  
Report preparation: fjb/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

Calculations

R2382\_103097

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R-2382- DuPont Soil Data

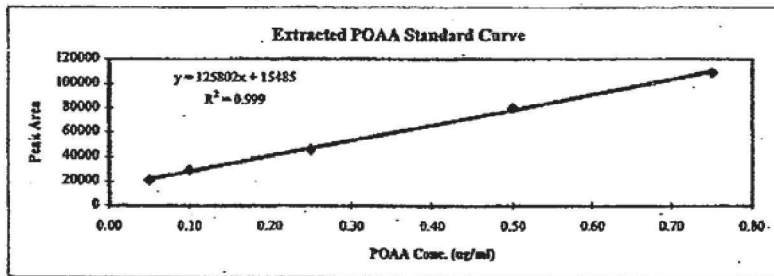
Figure 1

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		21000
0.10	25000	3000	28000		29000
0.25	40000	4000	44000		46000
0.50	71000	9000	80000		80000
0.75	96000	14000	110000		109000

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



Curve Equation:  $\frac{Y\text{-intercept}}{15000} \quad \frac{\text{Slope (m)}}{126000} \quad \frac{X\text{-intercept}}{-0.119}$

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

Soil 1 Curve ug/ml

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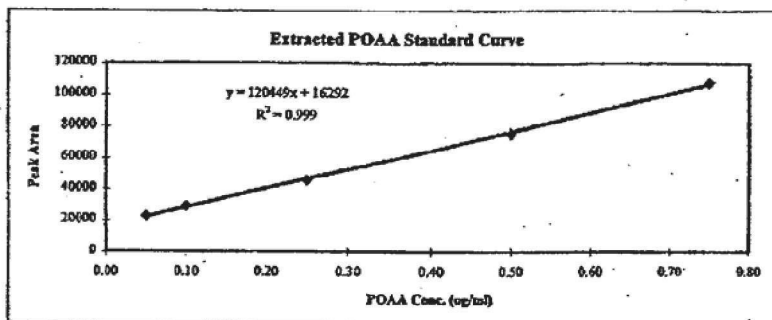
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R-2382- DuPont Soil Data

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



Curve Equation:  $\frac{Y\text{-intercept}}{16000} \quad \frac{\text{Slope (m)}}{120000} \quad \frac{X\text{-intercept}}{-0.133}$

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

11.04 Curve ugml

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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:

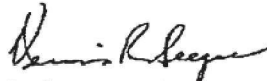
Solvent delay: 2.2 min.  
Electron multiplier: 2053 volts  
Scan range: 35 to 260 amu  
Scans per second: 2.17  
Scan threshold: 100

*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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Page 9	Page 11
<p>1 Q. And what is your date of birth, ma'am? 2 A. August 3rd, 1963. 3 Q. And your home address? 4 A. 202 North Broadview, Wichita, Kansas 67208. 5 Q. And are you taking any medications of any 6 kind today that could affect your memory in 7 any way? 8 A. No, I am not. 9 Q. Have you ever had your deposition taken 10 before? 11 A. Yes, I have. 12 Q. How many times? 13 A. Once. 14 Q. When was that? 15 A. 1986 or 7, I believe. 16 Q. And with whom, if anyone, were you employed 17 at the time? 18 A. State of Minnesota. 19 Q. With any particular agency? 20 A. The Minnesota Pollution Control Agency. 21 Q. And is it okay for purposes of today's if we 22 refer to that agency, the Minnesota Pollution 23 Control Agency, as MPCA? 24 A. Yes. 25 Q. Other than the deposition you just mentioned</p>	<p>1 responses because your responses are being 2 recorded not only by videotape, but also in a 3 written transcript. All right? 4 A. Yes. 5 Q. And, also, please make sure to keep your 6 voice up to the extent you can today so we 7 can make sure we've got a clear video record 8 and that the court reporter can hear you 9 clearly, all right? 10 A. Yes. 11 Q. If at any point in time you don't understand 12 one of my questions or want clarification in 13 any way, please let me know, and I'll go back 14 and try to rephrase it or clarify it for you. 15 Otherwise, I'm going to assume that you've 16 heard it, understood it, and gave me your 17 best response, all right? 18 A. Yes. 19 Q. And if at any point in time you feel like you 20 need to take a break for any reason, let me 21 know. Otherwise, we'll try to take breaks at 22 reasonable times today, all right? 23 A. Very good. 24 Q. Are you represented by counsel today? 25 A. Yes, I am.</p>
Page 10	Page 12
<p>1 back in 1986 while you were employed with the 2 MPCA, have you ever participated in any other 3 depositions of any kind? 4 A. No, I have not. 5 Q. Have you ever provided testimony at any 6 trial? 7 A. No, I have not. 8 Q. Have you ever provided testimony in any court 9 proceeding of any kind other than the 10 deposition you just mentioned? 11 A. No. 12 Q. Since it's been a while since your last 13 deposition, I'm going to go over some of the 14 ground rules on how the deposition process 15 will work today, all right? 16 A. Yes. 17 Q. Today I'm going to be asking you a number of 18 questions, many of which may concern events 19 or activities that occurred a long time ago. 20 So I ask that you please not guess or 21 speculate when providing answers, but please 22 provide me your best recollection of the 23 facts, all right? 24 A. (Witness nods head.) 25 Q. And please make sure to verbalize all of your</p>	<p>1 Q. And who is that counsel? 2 A. Jim Armstrong. 3 Q. And he's the attorney sitting to your right, 4 correct? 5 A. That's correct. 6 Q. At any point prior to today, have you 7 requested any counsel from the 3M Company in 8 connection with your deposition today? 9 A. No, I have not. 10 Q. Have you discussed this deposition with any 11 counsel for 3M prior to today? 12 A. I received a phone call from John Allison in, 13 I believe, the November 2007 time frame, 14 alerting me to the effect that I would be -- 15 there was a request for me to be deposed by 16 plaintiff's counsel. And that is the only 17 contact that I've had with 3M regarding this 18 proceeding. 19 Q. Was anyone else on the telephone during that 20 call between you and Mr. Allison? 21 A. No. 22 Q. How long did that call last? 23 A. Perhaps 5 to 10 minutes. 24 Q. At the time you received that call from 25 Mr. Allison, were you employed with your</p>

3 (Pages 9 to 12)

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**Appendix A: Report of Laboratory Analysis**

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3MA00230161

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-Nitropropene	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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3MA00230162

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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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	-	-	-	-	-	-
Alyl 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-Dichloropropene	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	68	-	-	-	-
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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3MA00230164

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	-	-	-	-	-
1,1,1,2-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 Environmental Laboratory

Volatiles by AED (5/97 Samples)

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**3M Environmental Laboratory**

**Data Transmittal Summary**  
 Preliminary (Final) (circle one)

Lab Request #: R2008	3M Study #: 3048/3058 Biogenenvir	Contract Laboratory #
Date Received:		
Sponsor or Client: Representative Name Company Name DuPont Company Address Phone		
Project Lead: Name / Phone KHansen / 80018 Group Leader: Name / Phone JDJohnson / 85294		
Analyte(s) or Test Method #: P0AA Sample Matrix: Water, Soil Analysis Dates: 060997 - 062697 Analyst(s): SEMiller Author: SEMiller Data Reviewed by:		
Project Lead (or designee): James D. Johnson (or designee):		
Internal		Sent by: / Date
JDI: 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  
kjhsansen@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 ☉ Ranney Well FC143 1:48 pm
R2008-2	1	DuPont Wash. Works ☉ Ranney Well FC143 1:48 pm
R2008-3	1	DuPont Wash. Works ☉ Ranney Well FC143 1:50 pm
R2008-4	1	DuPont Wash. Works ☉ Ranney Well FC143 1:50 pm
R2008-5	1	DI Water

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  $10 \pm 0.5$  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

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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1 current employer? 2 A. Yes. 3 Q. Had you asked that anyone from 3M contact 4 you? 5 A. No. 6 Q. What did Mr. Allison tell you during that 7 phone call? 8 A. Mr. Allison, as I said earlier, alerted me to 9 the fact that it was known to him that 10 plaintiff's counsel had an interest in 11 deposing me. 12 Q. And was there any discussion about why? 13 A. Not really. The only discussion that we had 14 was about the upcoming potential deposition 15 and also the time frame, because his 16 understanding was that it was going to -- 17 that the request was for later in November, 18 and it was very -- the time frame was 19 unworkable for me from my scheduling 20 perspective. 21 Q. Was there any discussion about the substance 22 of the case in which you were being requested 23 to provide the deposition? 24 A. No. 25 Q. During the call with Mr. Allison, did	1 related to then what were my options. And he 2 responded that that was really my concern, 3 that he couldn't provide any kind of legal 4 advice to me. And my response was, I will 5 then hire my own attorney. And that was 6 basically the end of the conversation. 7 Q. Did you ask for any recommendations from 8 Mr. Allison for your own counsel? 9 A. I did not. 10 Q. And then you retained Mr. Armstrong, correct? 11 A. Yes, I did. 12 Q. All right. Any further discussions with any 13 counsel for 3M other than the discussion you 14 just described with Mr. Allison? 15 A. No. 16 Q. Have you had any discussions with any 17 employee of 3M -- let me -- we'll come back 18 to that. 19 (Deposition Exhibit No. 1 was marked 20 for identification.) 21 Q. Would you prefer that I refer to you as Ms., 22 Mrs.? 23 A. It doesn't matter to me. 24 Q. Okay. Ms. Corrigan, I'm going to hand you 25 what's been marked as Exhibit 1 in your
Page 14	Page 16
1 Mr. Allison offer counsel? 2 A. He did not. 3 Q. Did you request that 3M provide counsel for 4 the deposition? 5 A. I did. 6 Q. What was the response? 7 A. The response was that because I was no longer 8 an employee with 3M it would be better for me 9 to be represented by my own counsel. 10 Q. So you did ask Mr. Allison for 3M to provide 11 counsel to you for the deposition, correct? 12 A. I did. 13 Q. Did you have any further discussion with 14 Mr. Allison about the position 3M was taking 15 in that regard? 16 MR. ARMSTRONG: In regard to 17 counsel? 18 BY MR. BILOTT: 19 Q. Yes. 20 A. Can you explain your question more fully? 21 Q. After you asked for counsel and Mr. Allison 22 told you that 3M would not be offering 23 counsel, did you have any discussion with him 24 about why? 25 A. The discussion that we had about counsel was	1 deposition, and ask you to take a look at 2 that and tell me if you recall seeing this 3 document before. 4 Let me restate the question. 5 Ms. Corrigan, do you recall ever seeing a 6 written notice for your deposition? 7 A. Yes, I do. 8 Q. All right. And you recognize this as that 9 notice? 10 A. I'm going through it now. It looks to be 11 that notice, yes. 12 Q. Do you recall the written notice for your 13 deposition also included a request for you to 14 search for certain categories of documents 15 that you might have in your possession? 16 A. Yes. 17 Q. And did you do that? 18 A. Yes, I did. 19 Q. And did you find any documents responsive to 20 any of the requests for production? 21 A. I did. 22 Q. What did you find? 23 A. I found a copy of a recusal letter that I had 24 composed while commissioner for the Minnesota 25 Pollution Control Agency.

4 (Pages 13 to 16)

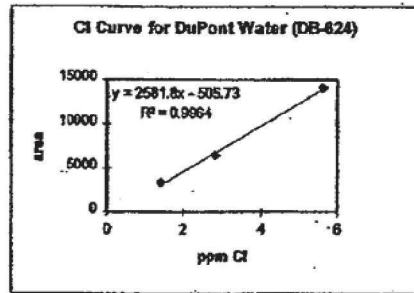
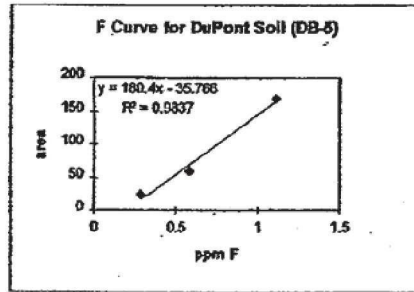
2100 3rd Avenue North, Suite 960\***Birmingham, Al 35203\***  
1-800-888-DEPO

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:





## 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

3M Environmental Laboratory

Volatiles by AED (6/97 Samples)

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3MA00230173

**3M Environmental Laboratory**

**Data Transmittal Summary**  
 Preliminary Final (circle one)

Lab Request #: R2148	3M Study #: 3048 Bigenenvir	Contract Laboratory #
Date Received: 070297		
Sponsor or Client: Representative Name Company Name DuPont Company Address Phone		
Project Lead: Name/Phone KHansen/86418 Group Leader: Name/Phone JDJohnson/85294		
Analyte(s) or Test Method #: Fluorine, Carbon, Hydrogen, Chlorine, Bromine 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
IDI: 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

**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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3MA00230175

## 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  $4 \pm 0.1$  grams of sodium chloride. The vials were reweighed 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.

3M Environmental Laboratory, Lab Request R2148

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3MA00230176

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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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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3MAD0230178

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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Column: DB-624

Lab Request #	C496*	H486*	C1479*	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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3MA00230160

MERRILL LEGAL SOLUTIONS  
Court Reporting\*Legal Videography\*Trail Services

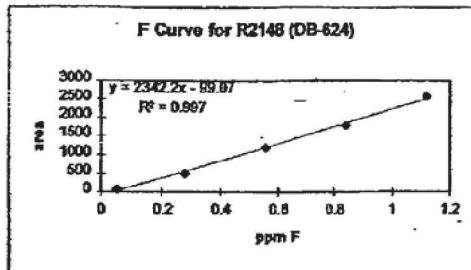
Page 17	Page 19
<p>1 Q. And prior to the beginning of the deposition, 2 your counsel handed us a copy of that. I'll 3 go ahead and mark that. 4 (Deposition Exhibit No. 2 was marked 5 for identification.) 6 Q. Ms. Corrigan, I'm going to hand you what's 7 been marked as Exhibit No. 2, and ask you to 8 look at that and tell me if you can identify 9 that is the document that you just referred 10 to that you found? 11 A. Yes, it is. 12 Q. Is this the only document that you identified 13 as being responsive to these requests for 14 production of documents? 15 A. Yes. 16 Q. What did you do to look for documents 17 requested here? 18 A. I looked through my personal files and 19 through any electronic correspondence files. 20 I looked through phone messages. And that's 21 the extent of my search. 22 Q. With respect to hard copy paper files, at the 23 time that you received this request for you 24 to look for documents, did you have any hard 25 copy paper files that related in any way to</p>	<p>1 I had with the agency, this particular 2 letter. 3 Q. The thank you notes that you just mentioned, 4 were any of those from any employees of 3M? 5 A. No. Let me be specific. They were thank you 6 notes that were given to me as part of my 7 service with the agency, congratulatory notes 8 as I left, from various groups that I had 9 interacted with. 10 Q. You mentioned that your files with the MPCA 11 were left with the MPCA when you left, 12 correct? 13 A. Correct. 14 Q. Why was Exhibit No. 2, why was a copy of that 15 put in your personal file when you left? 16 A. I put it in my personal file. 17 Q. Why? 18 A. Because I anticipated that with the 19 connection that I had to the MPCA, as well as 20 previous connections to 3M, that potentially 21 there would be a need to have this with me. 22 And it also, as you can read, pertains 23 specifically to me. 24 Q. You mentioned you also searched through 25 electronic files; is that correct?</p>
Page 18	Page 20
<p>1 your prior -- to your employment prior to 2 working for, is it, Koch Industries? 3 A. Koch Industries. I'm sorry, the question 4 again was? 5 Q. Did you have any files relating to any of 6 your prior employment before working for Koch 7 Industries? 8 A. The employment that I had prior to working 9 for Koch Industries was as the Commissioner 10 of the MPCA. And those files, as I left the 11 agency, I left them for my successor as they 12 belonged to the State of Minnesota. Prior to 13 that I worked for 3M in a number of different 14 capacities, and each time I changed roles 15 with 3M, I left the files for those 16 particular roles with the successor for those 17 roles. So the answer to your question is I 18 do not have any files in my possession. 19 Q. Where did you find the document that's marked 20 at Exhibit 2? 21 A. In my personal file. 22 Q. And by your personal file, what kind of 23 information is in your personal file? 24 A. My personal files contained thank you notes, 25 congratulatory notes on the appointment that</p>	<p>1 A. Yes. 2 Q. And do you have a personal computer? 3 A. I do. 4 Q. Was your personal computer ever used for 5 sending e-mails relating to your work while 6 at the MPCA? 7 A. I can't think of an occasion where I did send 8 e-mails from my personal computer, as I had a 9 BlackBerry while I was at the MPCA. 10 Q. And was your BlackBerry issued by the MPCA? 11 A. Yes. 12 Q. And did the messages that you sent or 13 received through that BlackBerry, or from 14 that BlackBerry, get routed through the 15 MPCA's computer system? 16 A. Yes, they did. 17 Q. Do you recall ever sending or receiving any 18 e-mail messages relating to your work at MPCA 19 through your home computer? 20 A. I don't remember. 21 Q. Do you still possess that home computer that 22 was in your possession while you were working 23 at the MPCA? 24 A. No, I do not. 25 Q. When did you purchase the current computer</p>

5 (Pages 17 to 20)

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

**STANDARD CURVES:**

Example of a standard curve from the analysis (all standard curves had R<sup>2</sup> 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**

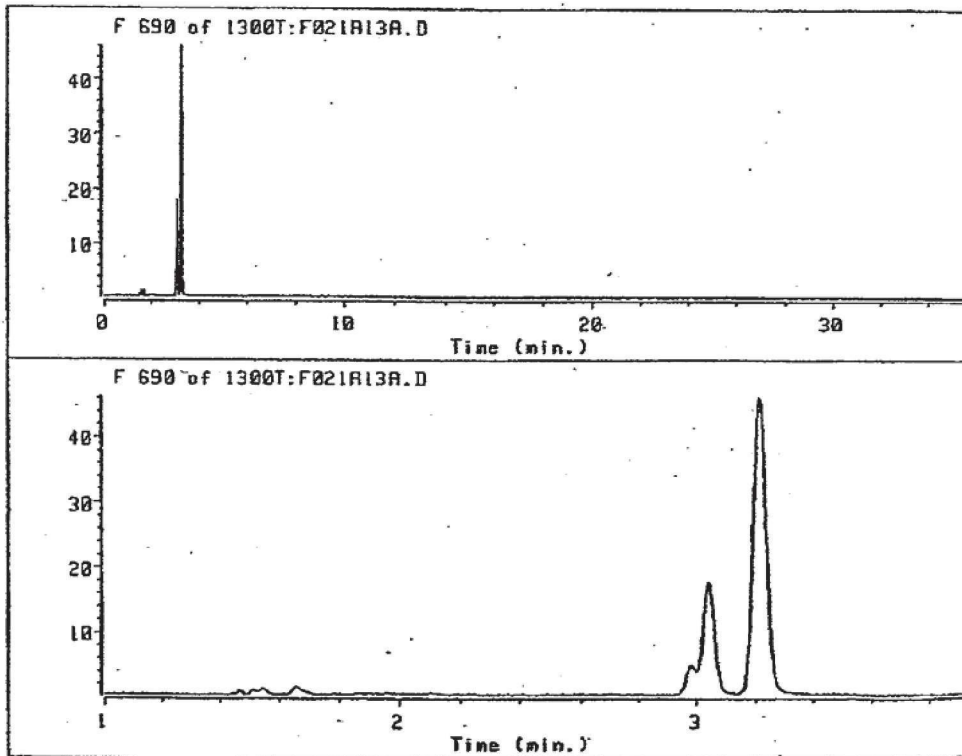
Appendix A: Chromatogram of sample 6 (MW-3 2 of 2)  
F690  
DB-624

SEMiller  
110797

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



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3MA00230182



3M Environmental Laboratory

**Total Fluoride in Soil (6/97 Samples)**

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3MA00230183

**3M Environmental Laboratory**

**Data Transmittal Summary**  
Preliminary / Final (circle one)

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

Fluoride:	Average (mg/Kg):	21300	20100	61200	78300	106300	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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3MA00230185

### 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<sub>2</sub>O

#### 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<sub>2</sub>O. 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<sub>2</sub>O.

#### 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.

Analyst / Date

Daniel Howman / 10-1-97

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3MA00230186

#### 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  $y = 0.0067x - 0.1061$ . 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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3MA00230187

**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

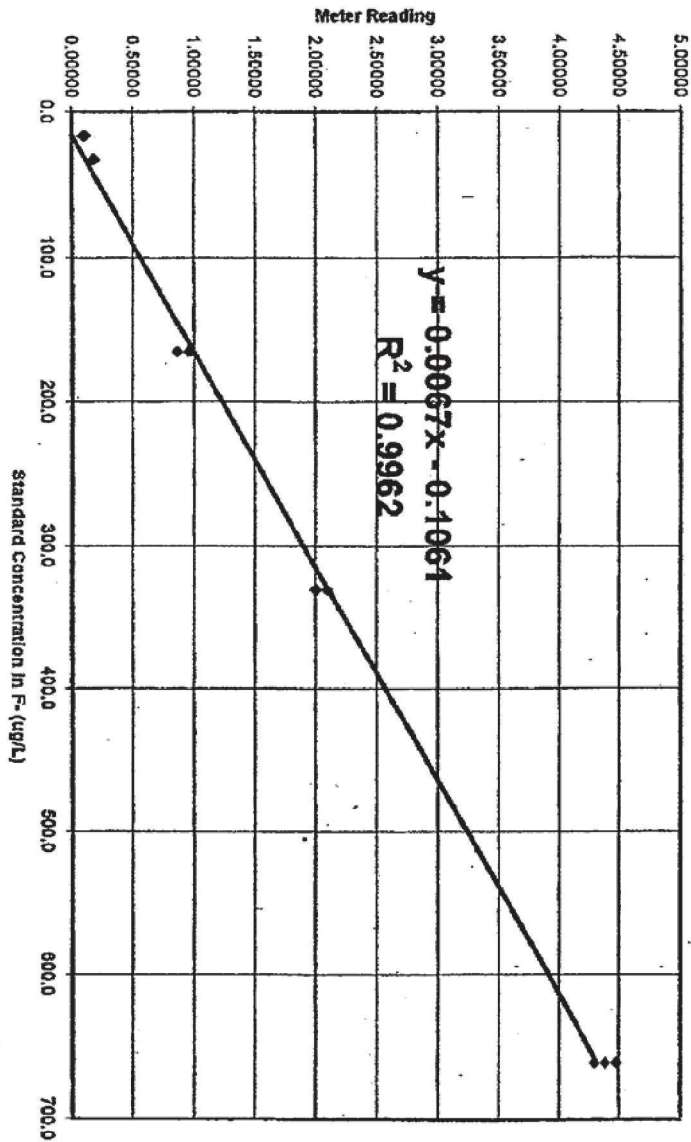
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3MA00230188



Standard Curve POAA



Analyt / Date  
Daniel Howman / 10-1-97

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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
Analytic: DRH 8-25-97								
QC 4.33PPM ERA CHECK	2					2.062	95.25	
BLANK-1	1	0.01	3.0			0.03853		
BLANK-2	1	0.01	3.0			0.03891		
BLANK-3	1	0.01	3.0			0.02618		
25PPM W397-919	1	0.01	3.0	18.3	18.5	0.06097	110.69	
25PPM W397-919	1	0.01	3.0	16.5	18.5	0.05608	89.96	
25PPM W397-919	1	0.01	3.0	12.8	18.5	0.04268	77.20	
25PPM W397-919	1	0.01	3.0	13.9	18.5	0.04858	84.20	
QC 1.0PPM CHECK	1					0.9665	96.65	
50PPM W397-920	1	0.01	3.0	36.5	33.1	0.1015	92.22	Due to low meter readings and low recovery, decided to rerun using 20uL as sample volumes
50PPM W397-920	1	0.01	3.0	28.2	33.1	0.08735	79.29	
50PPM W397-920	1	0.01	3.0	21.7	33.1	0.07238	65.71	
50PPM W397-921	1	0.01	3.0	111.7	165.3	0.3724	67.61	
250PPM W397-921	1	0.01	3.0	128.4	165.3	0.4278	77.69	
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			0.07783		
BLANK-3	1	0.02	3.0			0.02894		
25PPM W397-919	1	0.02	3.0	13.7	16.5	0.06156	82.03	
25PPM W397-919	1	0.02	3.0	17.2	16.5	0.1149	104.25	
25PPM W397-919	1	0.02	3.0	15.9	16.5	0.1067	88.86	
50PPM W397-920	1	0.02	3.0	29.5	33.1	0.1764	80.04	
50PPM W397-920	1	0.02	3.0	28.6	33.1	0.1804	88.50	
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	0.9765	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	90.86	
500PPM W397-922	1	0.02	3.0	314.8	330.5	2.096	95.19	
1000PPM W397-923	1	0.02	3.0	646.2	661.0	4.201	97.61	
1000PPM W397-923	1	0.02	3.0	672.1	661.0	4.481	101.08	
1000PPM W397-923	1	0.02	3.0	658.5	661.0	4.390	99.83	
QC 1.0PPM CHECK	1					0.9648	96.48	
Notebook Reference: Carlin-SA-2, p.20-21								

Analyst / Date  
Daniel Howman / 10-1-97

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**MERRILL LEGAL SOLUTIONS**  
**Court Reporting\*Legal Videography\*Trail Services**

Page 21	Page 23
<p>1 you're using now? Well, let me restate the 2 question. 3 How many computers do you personally 4 have now? 5 A. Two. 6 Q. All right. When did you purchase those 7 computers? 8 A. We have a laptop computer that we purchased 9 in late 2007, I recall, I believe it was in 10 the October time frame, but I could be wrong, 11 give or take a month on that one. And we 12 have a regular computer that we purchased in 13 August of 2006 when we moved to Wichita. 14 Q. And did you have any personal computer prior 15 to August of 2006? 16 A. Yes, we did. 17 Q. And what happened to that computer? 18 A. We got rid of it. 19 Q. What did you do to search your current 20 computers for any information that might be 21 responsive to the request for information in 22 Exhibit 1? 23 A. I looked through the electronic files that we 24 would have, in particular any e-mail. I 25 looked through documents, but my</p>	<p>1 correct? 2 A. Correct. 3 Q. I'd like to refer you to Page 7 of Exhibit 1 4 for a moment, and particularly Paragraph 5 5 where there is a request for, quote, any and 6 all documents, communications and slash or 7 ESI in your possession, custody or control 8 relating in any way to any compensation or 9 other financial benefit received, accrued or 10 earned. And then parens, whether or not 11 received, close parens, by deponent from 3M 12 since the cessation of deponent's formal 13 employment relationship with 3M, close quote. 14 Do you see that? 15 A. Uh-huh, yes, I do. 16 Q. And I think you mentioned earlier that you 17 had worked at one point for the 3M Company, 18 correct? 19 A. That's correct. 20 Q. And did you, during that employment, acquire 21 any stock in the 3M Company? 22 A. Yes, I did. 23 Q. And do you still own stock in the 3M Company? 24 A. Yes, I do. 25 Q. Do you have any documents relating to the</p>
Page 22	Page 24
<p>1 communications with MPCA relative to personal 2 matters are friendship based, and anything 3 else, and so those e-mails that I would have 4 received from folks would have been deleted 5 immediately. 6 Q. You also mentioned phone messages when I 7 asked you what kind of information you had 8 reviewed, correct? 9 A. Uh-huh. 10 Q. Again, just make sure you verbalize your 11 response. 12 A. Yes, yes. I'm sorry. 13 Q. Were you referring to messages on your home 14 phone or someplace else? 15 A. My home phone, yes. 16 Q. And do you save those messages? 17 A. Not typically. 18 Q. Are there messages saved on your home phone? 19 A. Not as a common practice. 20 Q. So what did you do to review your phone 21 messages? 22 A. I checked our phone messages. 23 Q. Okay. And there was nothing on there 24 relating in any way to anything requested in 25 the request for production in Exhibit 1,</p>	<p>1 value of that stock or any dividends from 2 that stock? 3 A. I do. 4 Q. Looking at Paragraph 5, did you have some 5 belief that those documents were not related 6 to Paragraph 5? 7 A. Let me give you my thinking on this, and 8 certainly let me be open about this. 9 The stock that I own from 3M came from 10 two separate sources. One was a gift that I 11 received from my parents 20 some years ago 12 when I was married. I've never made a trade 13 on that stock since I received it. 14 Q. Okay. 15 A. The other source of 3M stock is contained in 16 my 401(k) account, which still resides within 17 the 3M benefits organization. Again, I've 18 never made a trade on that, nor have I 19 manipulated that stock. I attempted last 20 night, and I apologize for this, to download 21 a statement from my 401(k), but it has been 22 so long since I had been to that account, I 23 forgot my password, and I locked my account. 24 I can't receive a password except via mail, 25 which they promised to do within two business</p>

6 (Pages 21 to 24)

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

Project: R2382									
Soil Samples from Dupont Washington									
Calculations for Total Fluoride in Soil									
Calculated F- (mg/L) = (Meter Reading + Intercept) / Slope									
Calculated F- (ppm/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- (ppm/Kg)	Statistics (mg/Kg)	% Recovery	Comments
Analytic: DRH 9-30-97									
GC 4.3PPM ERA CHECK	2			2.078				98.0	
BLANK-1	1		3.0	0.07719					
BLANK-2	1		3.0	0.05811					
BLANK-3	1		3.0	0.05168					
R2382-1-1		0.0203	3.0	0.8647	138	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	138	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		
GC 1.0PPM CHECK	1			0.9698				97.0	
R2382-3-1		0.0204	3.0	2.485	387	58,900	AVE 61200		
R2382-3-2		0.0226	3.0	2.983	451	61,500	STD 4200		
R2382-3-3		0.0202	3.0	2.341	440	65,300	CV 7		
R2382-4-1		0.0235	3.0	3.658	563	71,900	AVE 78300		
R2382-4-2		0.0227	3.0	3.860	598	79,000	STD 6000		
R2382-4-3		0.0225	3.0	4.109	629	89,800	CV 8		
R2382-4-4 w/10ul spike of 18,000ppm PDA		0.023	3.0	8.189	940	122,600		51.4	
R2382-5-1		0.0228	3.0	5.349	814	107,100	AVE 103300		
R2382-5-2		0.023	3.0	5.408	827	107,900	STD 2100		
R2382-5-3		0.0226	3.0	5.136	782	103,900	CV 2		
GC 1.0PPM CHECK	1			0.8856				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	3.714	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	2.598	402	60,000	STD 4500		
R2382-7-3		0.021	3.0	2.815	378	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		
GC 1.0PPM CHECK	1			0.9777				97.8	
R2382-9-1		0.0200	3.0	1.485	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	218	31,900	CV 4		
GC 1.0PPM CHECK	1			0.9737				97.4	
Analytic: DRH 10-1-97									
GC 4.3PPM 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	0.09990					
R2382-10-1		0.0203	3.0	1.665	264	39,100	AVE 41200		
R2382-10-2		0.0201	3.0	1.741	278	41,100	STD 2200		
R2382-10-3		0.0208	3.0	1.909	301	43,400	CV 3		
R2382-11-1		0.0218	3.0	1.288	208	28,500	AVE 30300		
R2382-11-2		0.0223	3.0	1.471	235	31,700	STD 1800		
R2382-11-3		0.022	3.0	1.402	225	30,700	CV 8		
GC 1.0PPM CHECK	1			0.9420				94.2	

Notebook Reference: Carbon-SA-2, p.21-23

Analyst / Date  
Daniel Howman / 10-1-97

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3M Environmental Laboratory

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

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**3M Environmental Laboratory**

**Data Transmittal Summary**  
Preliminary / Final (circle one)

Lab Request #: R-2148	3M Study #: Bigenenvir	Contract Laboratory #
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): James D. Johnson (or designee): Pat Rethwill 10/03/97		
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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**3M Environmental Laboratory - Advanced Method Development Team**

Contact: Kris Hansen - Sr. Analytical Chemist  
Building 02-3E-09  
778-6018  
kjhaesen @ 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	<sup>Prox</sup> Fluoride Ion (µg/mL F-)	<sup>Combust</sup> Total Fluorine (µg/mL F-)	Total Fluorine - Fluoride Ion (µg/mL)	<sup>Carbon</sup> 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 3M Environmental Laboratory, Lab Request R2148

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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  $\approx 0.9950$ .

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

A standard curve for total fluorine was generated by combusting 0.1 mL aliquots of 2.0, 5.0, 20, 50, and 100  $\mu\text{g/mL}$  POAA standard (POAA is 66.1% fluoride) in the Dohrmann DX2000 Modified Organic Halide Analyzer. The off-gasses were collected in 3 mL of 1:1 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:  $Y = 0.0341x + 0.0157$  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  $\mu\text{g/mL}$  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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**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

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**Total Fluorine Standard Curve**  
**Ammonium Perfluorooctanoate in Milli-Q water**

Sample ID	Quantity Sample (mL)	Orion Meter Reading ( $\mu\text{g/mL F-}$ ) <sup>1</sup>	Spiked $\mu\text{g/mL F-}$ 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

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

**Total Fluorine Sample Analysis**

Sample ID	Meter * Reading	Quantity of Sample (mL)	Total $\mu\text{g/mL F-}$ 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  $\mu\text{g/mL}$ , <0.05 to 4.5  $\mu\text{g/mL}$  F- for adsorbable organic fluorine analysis, and 2.3 to 16  $\mu\text{g/mL}$  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.

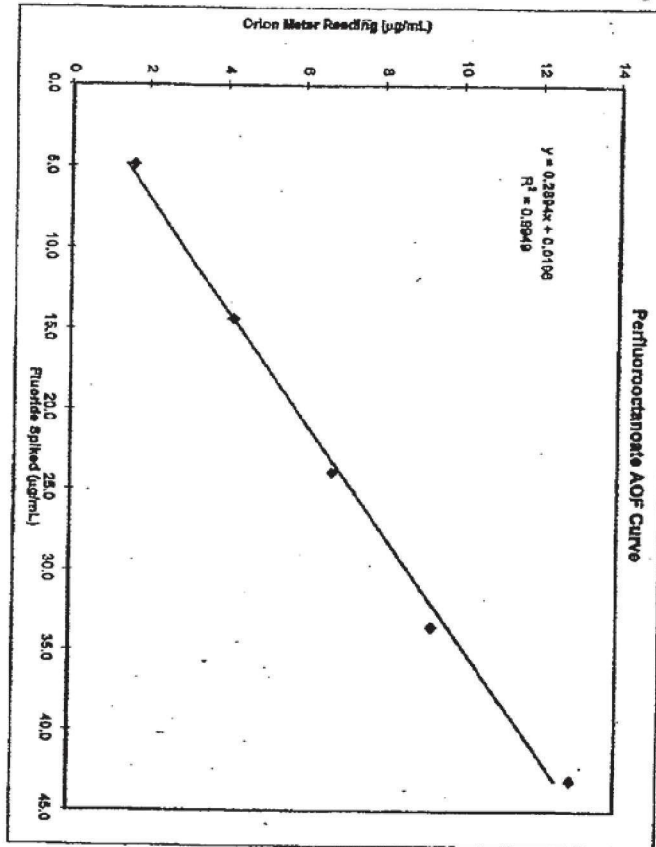
JG Schatz  
 09-30-97

3M Environmental Laboratory, Lab Request R2148

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3MA00230198

In Standard	F. Conc.	Meter Reading
4.9	14.4	1.6156
14.4	23.9	4.1827
23.9	33.6	6.7477
33.6	43.1	9.3122
43.1		12.8872



CURVE

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3MA00230199



Orion Data

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3MA00230200

DATA FOR AMMONIUM PERFLUOROCTANOATE 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					
Sid # 1 bottom	0.4538	3.0	1	100	0.01361					
Sid # 2 bottom	0.3701	3.0	1	100	0.01110					
Sid # 3 bottom	0.3843	3.0	1	100	0.0108298					
Sid # 4 bottom	0.1816	3.0	1	100	0.00544					
Sid # 5 bottom	0.1232	3.0	1	100	0.00370					
Blank top	0.0544	3.0	1	100	0.00183					
Sid # 1 top	1.1820	3.0	1	100	0.03485	1.516	0.048	0.049	100%	
Sid # 2 top	3.8128	3.0	1	100	0.1144	4.183	0.125	0.144	87%	
Sid # 3 top	6.3834	3.0	1	100	0.1915	8.748	0.202	0.239	85%	
Sid # 4 top	8.1307	3.0	1	100	0.2739	9.312	0.278	0.335	83%	
Sid # 5 top	12.784	3.0	1	100	0.3829	12.887	0.387	0.431	90%	
QC check 5.0	5.088	1.0	1	1	5.088					
FLUORIDE ION ANALYSIS										
Sample ID	Meter Read	TSAB Vol	Dilution	Sample Vol	Total Fluoride (µg/mL)					
QC check 0.50 pp	0.5018	1.0	1	1.0	0.502				100%	
R2148-1	0.1025	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	0.0807	1.0	2	1.0	0.160					
R2148-5	0.0701	1.0	2	1.0	0.140					
R2148-6	0.0666	1.0	2	1.0	0.139					
R2148-7	0.0587	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	0.083					
R2148-11	0.0481	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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<p>1 days. I am happy to provide any statement 2 from that 401(k), if you would like. 3 Q. Okay. 4 A. I in no way meant to not be responsive and 5 explained to my counsel this morning my 6 dilemma. 7 Q. Okay. We would request a copy of that when 8 it's received. 9 So I think you mentioned there's two 10 different categories of stock. 11 A. That's right. 12 Q. One would be some stock you received from 13 your parents about 20 years ago, correct? 14 A. Correct. 15 Q. And do you have an understanding as to what 16 the current value of that it is? 17 A. It obviously has varied, but it's between 20 18 and \$30,000. 19 Q. And the 401(k) account, is that -- do you 20 have a current understanding as to what the 21 current value, approximate, of that account 22 is? 23 A. My -- I recall seeing a statement from about 24 six months ago. It was a little over, if I 25 recall correctly, a little over \$100,000. of</p>	<p>1 Q. Have you been interviewed by any 2 representative of the Minnesota Attorney 3 General's Office on anything relating to 4 PFC's or 3M? 5 A. No, I have not. 6 Q. Okay. And you mentioned that for purposes of 7 your deposition today you're represented by 8 Mr. Anderson, correct? 9 MR. ARMSTRONG: Armstrong. 10 BY MR. BILOTT: 11 Q. I'm sorry. Armstrong, correct? 12 MR. ARMSTRONG: Happens all the 13 time. 14 A. That is correct. 15 Q. And did you have any meetings with him to 16 prepare for your deposition? 17 A. Yes, I did. 18 Q. How many meetings? 19 A. One. 20 Q. When was that? 21 A. Monday of this week, February 25th. 22 Q. How long did that meeting last? 23 A. Approximately two hours. 24 Q. Were you shown any documents of any kind 25 during that meeting?</p>
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<p>1 which a percentage is 3M stock and the rest 2 is other investments and mutual funds and so 3 forth. 4 Q. Do you know what percentage? 5 A. I do not. 6 Q. Do you have an approximate idea of what the 7 value of the 3M portion is? 8 A. Less than 40 percent, I would think. But, 9 again, I'm unsure of that. 10 Q. Okay. On Page 10 of Exhibit 1, Paragraph 20, 11 it refers to any documents you might have 12 relating to litigation or governmental 13 investigations or inquiries relating or 14 referring to any PFC. Do you see that? 15 A. Yes, I do. 16 Q. And is it correct that you have no documents 17 that relate in any way to any investigations 18 by any governmental agencies since you left 19 the MPCA relating to PFC's? 20 A. That is correct. 21 Q. All right. Have you ever been interviewed by 22 any representative of the United States 23 Department of Justice relating to anything to 24 do with 3M or PFC's? 25 A. No, I have not.</p>	<p>1 A. I was not. 2 Q. Did you bring any documents with you during 3 that meeting? 4 A. I did not. 5 Q. Was anyone else present during the meeting? 6 A. No. 7 Q. Was anybody participating by phone, other 8 than the two of you? 9 A. No. 10 Q. Okay. Who is paying for your counsel's time 11 today? 12 A. I am. 13 Q. Do you anticipate making any request that any 14 other entity reimburse you for whatever you 15 pay for your counsel's time? 16 A. I have not thought through that. 17 Q. After receiving the request for your 18 deposition in this case, did you have any 19 discussions with anyone at your current 20 employer about the deposition? 21 A. I did. I notified my supervisor and told him 22 that I would be involved in a deposition. 23 And I also contacted our general counsel and 24 gave him the same message. 25 Q. And did you have any discussion with them,</p>

7 (Pages 25 to 28)

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

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 Fluoride (µg/ml)	Comments	Recovery %			
QC check 6.0 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	50							
R2148-6 bottom	0.2803	3.0	1	85							
QC check 6.0 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	8.854	3.0	1	85	8.778	0.275		99%			
R2148-4 Top	11.16	3.0	1	85	11.28	0.458					
R2148-5 Top	30.15	3.0	1	80	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 6.0 ppm	4.855	1.0	1	1				99%			
MIR Q Bk Bottom	0.0585	3.0	1	100							
R2148-1 bottom	0.1833	3.0	1	5							
R2148-2 bottom	0.1910	3.0	1	5							
R2148-3 bottom	0.1423	3.0	1	10							
R2148-4 bottom	0.1128	3.0	1	10							
R2148-5 bottom	0.2289	3.0	1	50							
R2148-6 bottom	0.1881	3.0	1	50							
MIR Q Bk Top	0.0531	3.0	1	100	0.110	0.0031					
R2148-1 top	6.381	3.0	1	5	6.544	4.5081					
R2148-2 top	5.898	3.0	1	5	6.080	4.1951					
R2148-3 top	3.095	3.0	1	10	3.238	1.1113					
R2148-4 top	2.116	3.0	1	10	2.228	0.7532					
R2148-5 top	2.486	3.0	1	50	2.696	0.1849					
R2148-6 top	1.696	3.0	1	50	2.024	0.1386					
QC cell check 4.33 ppm	2.04	1.0	2	1		4.072		94%			

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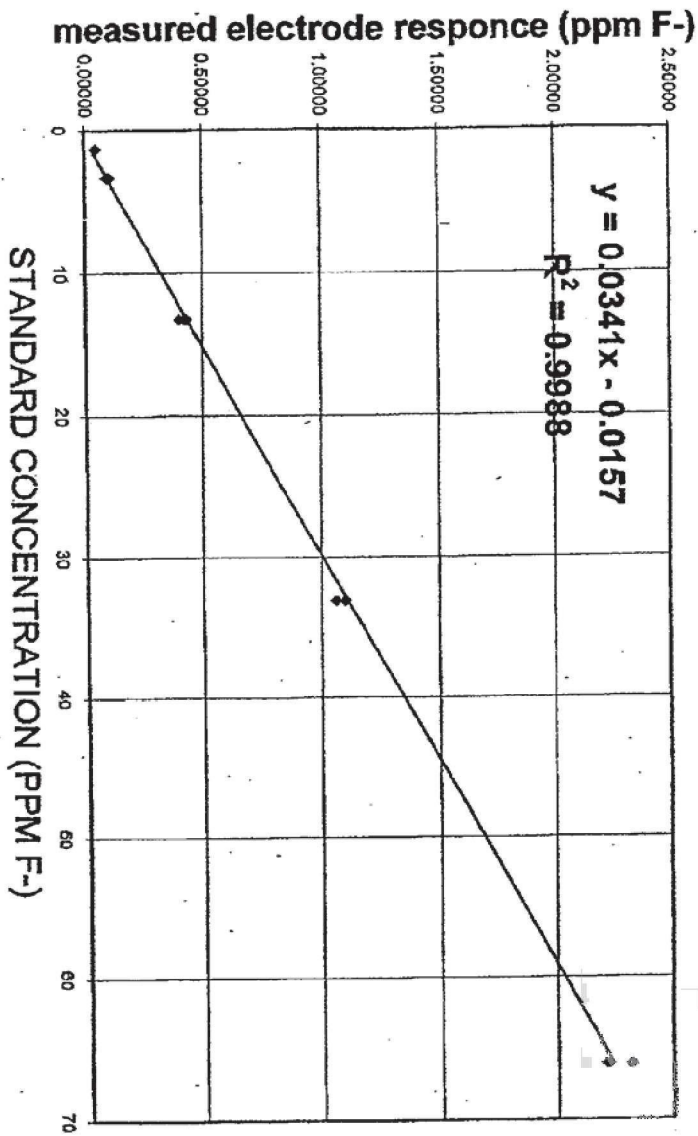


Orion Data

AOF ANALYSIS		AOF Calc. (µg/mL) = ((total meter reading - intercept) / slope) / sample volume					Calc. Adsorbable Organic Fluoride (top&bottom)		Comments	Recovery %
Sample ID	Meter Reading	Collected Vol (mL)	Dilution Factor	Sample Vol (mL)	Meter Reading Total (top&bottom)	Calc. Adsorbable Organic Fluoride (top&bottom)	Comments	Recovery %		
R2148-8 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	1.2556	3.0	1	50	1.5561	0.1082				
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	0.2983	3.0	1	50	0.3385	0.0221	read below low std. re-run			
R2148-7 MS Bottom	0.1134	3.0	1	100						
R2148-12 top	3.0287	3.0	1	100	3.143	0.1079				
QC cal check 4.33 ppm	2.228	1.0	2	1		4.456		103%		
R2148-11 bottom	0.0760	3.0	1	100						
R2148-12 bottom	0.0899	3.0	1	100						
R2148-11 Top	0.4608	3.0	1	100	0.5369	0.0176				
R2148-12 Top	0.4516	3.0	1	100	0.5357	0.0175				
QC check 0.050 ppm	0.4988	1.0	1	1				98%		

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Perfluorooctanoate Standard Curve

STANDARD	FACTOR	SAMPLE	COLLECT	CALC.	POAA	Actual	% REC	COMMENTS
ERA 4.33PPM STD								
QC 1.0PPM STD CHECK	2					2.200	101.6	
						0.9652	98.52	
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		0.0181		
POAA 1.0 PPM W397-795	1	0.1	3	1.16		0.0398	176	BELOW DETECTION LEVEL
POAA 1.0 PPM W397-795	1	0.1	3	0.97		0.0323	147	USE 2.0PPM STD DATA
POAA 1.0 PPM W397-795	1	0.1	3	1.40		0.0407	212	NOT INCLUDED IN CURVE
ERA 4.33PPM STD						2.162	89.89	
						0.9864	98.64	
QC 1.0PPM STD CHECK								
BLK-1	1	0.1	3			0.01599		
BLK-2	1	0.1	3			0.01466		
BLK-3	1	0.1	3			0.01481		
POAA 2.0PPM W397-801	1	0.1	3	1.473		0.04911	111	
POAA 2.0PPM W397-801	1	0.1	3	1.473		0.04911	111	
POAA 2.0PPM W397-801	1	0.1	3	1.397		0.04855	108	
POAA 5.0 PPM W397-796	1	0.1	3	2.82		0.0939	95.2	
POAA 5.0 PPM W397-796	1	0.1	3	3.22		0.1074	97.5	
POAA 5.0 PPM W397-796	1	0.1	3	2.89		0.0989	90.6	
QC 1.0 PPM CHECK								
POAA 20PPM W397-797	1	0.1	3	12.10		0.4033	91.5	
POAA 20PPM W397-797	1	0.1	3	12.80		0.4288	98.9	
POAA 20PPM W397-797	1	0.1	3	12.98		0.4321	98.1	
POAA 50PPM W397-798	1	0.1	3	31.92		1.064	98.5	
POAA 50PPM W397-798	1	0.1	3	33.12		1.104	100	
POAA 50PPM W397-798	1	0.1	3	32.97		1.089	98.8	
POAA 100PPM W397-799	1	0.1	3	68.64		2.218	101	
POAA 100PPM W397-799	1	0.1	3	69.60		2.230	105	
POAA 100PPM W397-799	1	0.1	3	66.27		2.208	100	
QC 1.0 PPM CHECK						0.9930	99.3	

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**TABLE : R2148 F. DETERMINATION: SAMPLES DUPONT INC.**

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

SEE STANDARD CURVE FOR EQUATION: Y = 0.0241X + 0.0187

SAMPLE	COLLET	Actual	CALC.	STATS	% REC
NOV 11 1987 20:57					
ERA 4.33PPM STD		2.162			99.86
QC 1.0PPM STD CHECK		0.9894			98.64
BLK-1	3	0.01598			
BLK-2	3	0.01465			
BLK-3	3	0.01491			
QC 1.0PPM CHECK		0.9743			97.43
R2148-11-1	3	0.05555	2.09	AVE 2.33	
R2148-11-2	3	0.08031	2.23	STD 0.30	
R2148-11-3	3	0.07499	2.66	CV 12.8	
R2148-12-1	3	0.1699	6.44	AVE 4.23	
R2148-12-2	3	0.1233	4.06	STD 1.14	
R2148-12-3	3	0.09247	3.17	CV 27.0	
R2148-10-1	3	0.08379	2.92	AVE 2.81	
R2148-10-2	3	0.08284	2.30	STD 0.47	
R2148-10-3	3	0.09400	3.22	CV 16.6	
QC 1.0 PPM CHECK		0.9863			98.63
R2148-8-1	3	0.1042	3.52	AVE 2.99	
R2148-8-2	3	0.07910	2.78	STD 0.48	
R2148-8-3	3	0.07288	2.60	CV 16.4	
R2148-6-1	3	0.1361	4.45	AVE 4.15	
R2148-6-2	3	0.1279	4.21	STD 0.34	
R2148-6-3	3	0.1131	3.78	CV 8.24	
QC 1.0PPM STD CHECK		0.9824			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 = 0.0241X + 0.0187

SAMPLE	COLLET	Actual	CALC.	STATS	% REC
NOV 11 1987 20:57					
ERA 4.33PPM STD		2.162			99.86
QC 1.0PPM STD CHECK		0.9894			98.64
BLK-1	3	0.01598			
BLK-2	3	0.01465			
BLK-3	3	0.01491			
QC 1.0PPM CHECK		0.9743			97.43
R2148-11-1	3	0.05555	2.09	AVE 2.33	
R2148-11-2	3	0.08031	2.23	STD 0.30	
R2148-11-3	3	0.07499	2.66	CV 12.8	
R2148-12-1	3	0.1699	6.44	AVE 4.23	
R2148-12-2	3	0.1233	4.06	STD 1.14	
R2148-12-3	3	0.09247	3.17	CV 27.0	
R2148-10-1	3	0.08379	2.92	AVE 2.61	
R2148-10-2	3	0.08284	2.30	STD 0.47	
R2148-10-3	3	0.09400	3.22	CV 16.6	
QC 1.0 PPM CHECK		0.9863			98.63
R2148-8-1	3	0.1042	3.52	AVE 2.99	
R2148-8-2	3	0.07910	2.78	STD 0.48	
R2148-8-3	3	0.07288	2.60	CV 16.4	
R2148-6-1	3	0.1361	4.45	AVE 4.15	
R2148-6-2	3	0.1279	4.21	STD 0.34	
R2148-6-3	3	0.1131	3.78	CV 8.24	
QC 1.0PPM STD CHECK		0.9824			96.24

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3MA00230205

3M Environmental Laboratory

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

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3MA00230207

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 *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

3M Ecotoxicology and Environmental Fate Laboratory

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'	79	<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

### 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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<p>1 either of them, about any of the potential  2 substance that might come up during the  3 deposition?  4 A. I did not.  5 Q. Did they ask, either of them?  6 A. My supervisor asked whether it was related to  7 my employment for 3M or whether it was  8 related to my employment as commissioner.  9 And I responded that I was unclear.  10 Q. Did they ask any other questions?  11 A. They did not.  12 Q. Did you provide any explanation to them about  13 what was going on with this deposition?  14 A. I did not.  15 Q. After you were notified that a request had  16 been made for your deposition in this case,  17 did you contact anyone at the MPCA?  18 A. I did not.  19 Q. Did you contact anyone at the Minnesota  20 Attorney General's Office?  21 A. I did.  22 Q. Who did you contact?  23 A. Bob Roach.  24 Q. And what is his position?  25 A. Assistant Attorney General.</p>	<p>1 involved in this proceeding and that I was in  2 the process of engaging an attorney.  3 Q. Do you have an understanding as to whether  4 anyone with the Minnesota Attorney General's  5 Office or the MPCA has contacted your  6 counsel?  7 MR. ARMSTRONG: Well, if she has  8 that information from somebody other than me,  9 I'll let her answer. But if her only  10 information about that is something I told  11 her, then that's privileged, and I'll  12 instruct her not to answer.  13 A. And the question was again?  14 Q. Do you have an understanding as to whether or  15 not anyone at the Minnesota Attorney  16 General's Office or MPCA has, in fact,  17 contacted your counsel?  18 MR. ARMSTRONG: And again, limit  19 your answer to anything that you learned  20 other than from me.  21 A. I do not.  22 Q. Do you know whether or not anybody at the  23 Minnesota Attorney General's Office or MPCA  24 has provided any information to your counsel?  25 MR. ARMSTRONG: Same instruction.</p>
Page 30	Page 32
<p>1 Q. With the -- in Minnesota, correct?  2 A. Correct.  3 Q. Why did you contact him?  4 A. To let him know that I had -- that I would be  5 deposed, and that they should contact my  6 attorney. At that time I don't believe I had  7 an attorney. And I said that I would make  8 sure and give my counsel Bob's information.  9 Q. Why did you contact Mr. Roach?  10 A. Because he had been the assistant AG,  11 attorney general, who had represented our  12 agency during my tenure as commissioner.  13 Q. And did Mr. Roach work with Alan Williams?  14 A. I believe they are colleagues.  15 Q. When you notified Mr. Roach that your  16 deposition was being requested, did he  17 respond in any way?  18 A. He did not.  19 Q. Did he ask any questions?  20 A. No. My phone call to him was a voice mail, I  21 believe.  22 Q. Did you receive any voice -- any return call  23 from Mr. Roach?  24 A. I did not. But I didn't expect one as the  25 call was just to let him know that I would be</p>	<p>1 A. I do not.  2 Q. You don't know?  3 A. I don't have any information from anyone  4 other than my counsel.  5 Q. Do you have information from your counsel?  6 MR. ARMSTRONG: Well, I'm going  7 to instruct her not to answer that. That's  8 privileged, because the answer to that would  9 imply one way or the other, and so I'm not  10 going to let her answer that.  11 MR. BILOTT: You're instructing  12 her not to answer?  13 MR. ARMSTRONG: I am.  14 MR. BILOTT: All right.  15 BY MR. BILOTT:  16 Q. Do you know whether or not any instructions  17 were provided by the Minnesota Attorney  18 General's Office or the MPCA to anyone  19 relating to your deposition today?  20 A. I do not know.  21 Q. Do you know whether anyone at 3M has  22 contacted your counsel?  23 MR. ARMSTRONG: Same instruction.  24 Again, I think the only way she could know  25 that, unless someone at 3M told her, is based</p>

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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:

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

### 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<sub>2</sub>

After pH in water was determined, 0.10 mL of 1.0 M CaCl<sub>2</sub> 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<sub>4</sub>OAc 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**

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

\*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. (MYTL) 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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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 order, 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 appears to be sand/silt with free flowing water in column  
Sample 11 appear to be coarse sand

An one foot core from the top of each column. When 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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FORM 5455-A	FILE
12382	ENC.
	DATE SHEET

ANALYSIS FORM		X6											
		A	B	C	D	E	F	G	H	I	J	K	L
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3													
4	2	210											
5													
6	3	200											
7													
8	4	200											
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PHONE (507) 354-8517 WATS (800) 782-3557 FAX (507) 359-2890



WE ARE AN EQUAL OPPORTUNITY EMPLOYER

<b>Report To:</b> Rochelle Robidean	<b>Date:</b> 27 August 1997
3M Environmental Lab	<b>Work Order:</b> 10-0447
935 Bush Ave., Bldg. 20	<b>Date Received:</b> 25 August 1997
St. Paul, MN 55106	

Page 1 of 4

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 ID. ....	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 Koebel*  
 Anthony R. Koebel  
 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 on any other sample unless all conditions affecting the sample are the same, including sampling by MVTL. As a mutual protection to clients, the published procedure, all reports are produced as the confidential property of clients, and authorization for publication of statements, conclusions or extracts from or reports is reserved pending our written approval.

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<p>1 upon a communication between my client and 2 me. And so you need to limit your answer to 3 outside that context. 4 A. There's been no contact that I'm aware of 5 outside of that context. 6 Q. Let me ask it this way: Without telling me 7 whether -- what information was provided, do 8 you know the answer to that question? 9 MR. ARMSTRONG: To what question? 10 BY MR. BILOTT: 11 Q. That I just asked. Whether or not there has 12 been information provided by 3M to your 13 counsel. Do you know the answer? 14 MR. ARMSTRONG: That's a yes or 15 no question. I'll let you answer that. 16 A. Yes. 17 Q. Can you briefly summarize for me your 18 educational background after high school, 19 degrees you have obtained? 20 A. I attended the University of Minnesota 21 Institute of Technology and received a 22 Bachelor of Science degree in geology. 23 Q. When did you obtain that degree? 24 A. 1986, I believe. 25 Q. And have you obtained any other educational</p>	<p>1 Q. And have you remained so licensed since '94? 2 A. Yes, I have. 3 Q. Any other professional licenses or 4 certifications of any kind? 5 A. No. 6 Q. Can you summarize for me your employment 7 history beginning with your first full-time 8 job after college? 9 A. My first full-time job after college was with 10 the Minnesota Pollution Control Agency. 11 Q. And when did you start with MPCA? 12 A. 1986 or 1987. 13 Q. And what position did you start at MPCA at 14 that time? 15 A. I was a pollution control specialist. 16 Q. How long did you remain in that position with 17 MPCA? 18 A. I recall about a year and a half, perhaps, or 19 a year, somewhere around that. 20 Q. So until late 1987 or 1988? 21 A. Correct. 22 Q. And what happened with respect to your 23 employment at that time? 24 A. I stayed at the agency, but I had a different 25 position.</p>
Page 34	Page 36
<p>1 degrees beyond your BS? 2 A. Not a full degree, no. 3 Q. Did you work toward any further degrees? 4 A. I did. 5 Q. What did you work towards? 6 A. A Master's of Secondary Science Education. 7 Q. How many -- how long did you pursue that 8 degree? 9 A. A year. 10 Q. Any other -- have you worked toward any other 11 educational degrees? 12 A. No, I have not. 13 Q. Do you have any professional licenses or 14 certifications of any kind? 15 A. Yes, I do. 16 Q. What are those? 17 A. I'm a professional geologist licensed in the 18 State of Wisconsin. 19 Q. When did you first become licensed in 20 Wisconsin in that regard? 21 A. I want -- I'm thinking back. 1994 or '95, I 22 believe. 23 Q. And do you remain licensed as a professional 24 geologist? 25 A. Yes, I do.</p>	<p>1 Q. And what position did you get at that point? 2 A. I was a Pollution Control Specialist II, and 3 I worked in a different group within the 4 organization. 5 Q. Okay. And how long did you remain in that 6 position? 7 A. About a year and a half, a year, year and a 8 half. 9 Q. And then what happened with respect to your 10 employment at that point? 11 A. Then I took a new role at the Metropolitan 12 Council as a water planner. 13 Q. And that was in approximately 1990? 14 A. Correct. 15 Q. How long did you remain in that position? 16 A. Again, it's quite some time ago. My 17 recollection is for about a year, year and a 18 half, two years, somewhere around there. 19 Q. And what happened with respect to your 20 employment at that point? 21 A. Then I took a new role with the -- with King 22 County in Seattle, Washington. 23 Q. And what did you do for King County? 24 A. I was a surface water planner. 25 Q. And that was beginning approximately 1992?</p>

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<b>Report To:</b>	Rochelle Robidean	<b>Date:</b>	27 August 1997
	3M Environmental Lab	<b>Work Order:</b>	10-0447
	935 Bush Ave., Bldg. 20	<b>Date Received:</b>	25 August 1997
	St. Paul, MN 55106		

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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: *a Koebel*  
 Anthony R. Koebel  
 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 on any other sample unless all conditions affecting the sample are the same, including sampling by MVTL. As a test and procedure to ensure the public and customer, all reports are submitted on 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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## Analysis of 3M Samples

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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 data 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 matter of 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 hereon or republishing our reports is reserved pending our written approval.

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DATE: 08/21/1997

3M ENVIRONMENTAL LABORATORY  
CONTRACT LABORATORY WORK ORDER BY PARAMETER

LAB REQUEST NO. R2382

CONTRACT LAB : MVL

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
N02-N	NITRITE NITROGEN - as N	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11	08/13/1997	08/27/1997
S03	SULFITE	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11	08/13/1997	08/27/1997
S04	SULFATE - as S04	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11	08/13/1997	08/27/1997

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**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.

<u>SOIL REACTION TESTS</u>	<u>SOLUBLE SALTS ANALYSIS</u>	<u>PHYSICAL CHARACTERISTICS</u>
pH	Electrical Conductivity	Soil texture and classification
Lime Requirement	Cl <sup>-</sup> , SO <sub>4</sub>	
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).

**0.063 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 0.063 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. Gelderman, 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 RFFH)

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3MA00230225



### 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).

#### 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:

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

#### 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).

## 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 7.9 to 8.2 indicates the presence of CaCO<sub>3</sub>.

Soil pH is a measure of the activity of H<sup>+</sup> 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<sup>+</sup> 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<sup>+</sup> 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<sup>+</sup> in solution.
- The CO<sub>2</sub> content (CO<sub>2</sub> from the atmosphere or soil air) dissolves in water forming carbonic acid (H<sub>2</sub>CO<sub>3</sub>) 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<sub>2</sub> in the air, there is no effect on the pH measurement. Only in soils of very low [H<sup>+</sup>] where the pH is considerably above 7.0 and particularly in soils containing free CaCO<sub>3</sub> does the CO<sub>2</sub> concentration of the air have any appreciable measurement effect on pH.
- Errors associated with equipment standardization and liquid junction potential. The use of 0.01 M CaCl<sub>2</sub> 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<sup>+</sup> or Al<sup>3+</sup>. 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.

#### EQUIPMENT AND REAGENTS

- 1). pH meter equipped with a combination electrode ( or soil pH electrode, Cole-Parmer Model #5892-60) 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). Gyrotory shaker.
- 6). Millipore Mill-Q™ water.
- 7). Calcium chloride (CaCl<sub>2</sub>) solution, 1 M or 0.01 M.

#### pH<sub>w</sub> AND pH<sub>s</sub> 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 Mill-Q™ water to each tube.
- 4). Mix thoroughly for 5 minutes, preferably on a gyrotory 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<sub>w</sub>.
- 8). To determine the soil pH in 0.01 M CaCl<sub>2</sub>, add 0.10 mL of 1M CaCl<sub>2</sub> 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<sub>2</sub>, pH<sub>s</sub>. Alternatively, the soil pH in 0.01M CaCl<sub>2</sub> may be determined directly by substituting 0.01 M CaCl<sub>2</sub> 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<sub>w</sub> or pH<sub>s</sub>.

#### 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. 6-9. In: Recommended Chemical Soil Test Procedures for the North Central Region. North Central Regional Publication No. 221 (Revised).



**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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3MA00230230



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<p>1 A. 1992, 1993, somewhere around there. 2 Q. And how long did you remain in that position? 3 A. A year and a half. 4 Q. And what happened with respect to your 5 employment at that point? 6 A. I took a new role with Northern Environmental 7 Technologies. 8 Q. Northern? 9 A. Uh-huh. 10 Q. Where was that? 11 A. New Brighton, Minnesota. 12 Q. And that was in approximately 1994? 13 A. Yes. 14 Q. And how long did you remain in that position? 15 A. About a year, year and a half. 16 Q. And then what happened with respect to your 17 employment at that point? 18 A. Then I took a new role with 3M Company. 19 Q. And what role was that? 20 A. As a remediation -- actually, it was a senior 21 engineer with 3M that specialized in 22 remediation of contaminated sites. 23 Q. And that was in approximately 1996? 24 A. 19 -- yes, 1996 it would have been. 25 Q. And how long did you remain in that position?</p>	<p>1 Q. Environment health and safety manager for 2 which division? I'm sorry. 3 A. Industrial Mineral Products. 4 Q. Do you recall what year it was that you 5 acquired that new position? 6 A. 2001 or 2002. 7 Q. And how long did you remain in that position? 8 A. A year. 9 Q. And then what happened with respect to your 10 employment then? 11 A. Then I was appointed as the Commissioner of 12 the Minnesota Pollution Control Agency. 13 Q. And how long did you remain in that position? 14 A. Three plus years. 15 Q. Until 1996 -- I'm sorry, 2006? 16 A. Correct. 17 Q. And did you leave MPCA in, was it, June of 18 2006? 19 A. Correct. 20 Q. When did you start working for Koch? 21 A. September of 2006. 22 Q. And did you -- were you employed by anyone 23 between June and September of 2006? 24 A. No, I was not. 25 Q. When you started working for Koch, what was</p>
Page 38	Page 40
<p>1 A. In that particular position two -- between 2 two and three years, I believe. 3 Q. And then what happened with respect to your 4 employment at that point? 5 A. Then I took a new role within 3M Company as a 6 manager of Environmental Operations. 7 Q. Manager of Environmental Operations? 8 A. Uh-huh. 9 MR. ARMSTRONG: You have to 10 answer out loud. 11 A. Yes. 12 Q. Thank you. And that was in approximately 13 1998 or 1999? 14 A. Yes. 15 Q. Do you recall what year? 16 A. I don't, honestly. It might have been a 17 little bit later than that. 18 Q. And how long did you remain in that position? 19 A. For a year. 20 Q. And then what happened with respect to your 21 employment then? 22 A. Then I moved to a new role and I was the 23 environmental health and safety manager for 24 the Industrial Mineral Products Division 25 within 3M.</p>	<p>1 the position you were hired into? 2 A. Director of Environmental Health and Safety 3 Compliance. 4 Q. Is that the position you are still in? 5 A. No. 6 Q. How long did you hold the position of 7 Director of Environmental Health and Safety 8 Compliance? 9 A. Until December of 2006. 10 Q. And what happened with respect to your 11 employment then? 12 A. I took a new role as the Director of 13 Compliance. 14 Q. Is that the role you're in now? 15 A. That's correct. 16 Q. Okay. Going back up to the first position 17 you mentioned, which was with the MPCA as a 18 pollution control specialist beginning in 19 1986, all right? 20 A. Or somewhere thereabouts. 21 Q. Okay. What MPCA location did you work out 22 of? 23 A. The St. Paul office in Minnesota. 24 Q. Who did you report to, who was your direct 25 supervisor at that time?</p>

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1-800-888-DEPO

#### EQUIPMENT AND REAGENTS

- 1). 250 mL conical, polypropylene centrifuge tubes.
- 2). Automatic pipets.
- 3). Gyrotory 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  $\text{NaC}_2\text{H}_3\text{O}_2$  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 ( $\text{CH}_3\text{COOH}$ ) or sodium hydroxide ( $\text{NaOH}$ ) to bring the reaction of the solution to pH 8.2.
- 6). 1.0 N, pH 7.0  $\text{NH}_4\text{OAc}$  (Ammonium acetate), identified as Reagent #2:  
For each liter of solution, add 58 mL of glacial acetic acid ( $\text{CH}_3\text{COOH}$ ) to approximately 600 mL of Milli-Q™ water and then add 70 mL of concentrated ammonium hydroxide ( $\text{NH}_4\text{OH}$ , specific gravity 0.90). It is best to add the  $\text{NH}_4\text{OH}$  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  $\text{CH}_3\text{COOH}$  or  $\text{NH}_4\text{OH}$ . 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 gyrotory 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 gyrotory 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  $\text{NH}_4\text{OAc}$  (Reagent #2) to the sample, stopper the tube and shake it on the gyrotory 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.

#### CALCULATION

CEC EQUATION:

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

\* % 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).

3M Environmental Laboratory

**Copies of Chain of Custody, Shipping Papers,  
Lab Requests**

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3MA00230233

1mSHIPPING MEMORANDUM  
(FOR NO-CHARGE SHIPMENTS ONLY)

1m4m  
 Ship to 4m 3M Environmental Technology & | Date 05/08/97 No. 38-97:  
 4m Service Laboratory, B-2-3E-09 | Routing  
 4m 935 Bush Avenue | F.O.B.  
 4m St. Paul, Minnesota 55144-1000 | Date  
 | Shipped  
 4m Attn: Robert Howell | E/L

4m Quantity	Product Code / Description	Weight
4m 1	Container of Water Samples	48 lbs
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Special Inst 4m Absolutely must arrive Friday AM/Time restraints.

Freight Class 4m

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4m Originator VERA L WIGAL

Phone 304-863-4895

4m Charge (Cost Code) 8309019511001280

| MSDS Sheets Required?

4m Purchase Order No.

| Hazardous Material? N

4m Value for Customs

| MATERIAL CLASSIFICATION

Ship From 4m Stores, Bldg. 4

| Corrosive Toxic

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| Flammable Other

4m

\*\* AUTHORIZATION \*\*

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3MA00230234







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2 "CHAIN OF CUSTODY"  
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5 8 SAMPLE BOTTLES OF SOIL TAKEN ON 5/30/97  
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USE STANDARDS FOR MINIMUM ESSENTIAL INSTALLATIONS

3MA00230237











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Page 41	Page 43
<p>1 A. Kathy Swanda. 2 Q. During the time that you were a Pollution 3 Control Specialist I, did you have any 4 involvement of any kind with 3M? 5 A. No. 6 Q. In approximately 1988 or so, when you became 7 a Pollution Control Specialist II, did your 8 location within MPCA change? 9 A. No. 10 Q. You continued to work out of the St. Paul 11 office, correct? 12 A. Correct. 13 Q. And was your immediate supervisor the same? 14 A. No. 15 Q. Who did you report to at that point? 16 A. Wayne Anderson. 17 Q. How, if at all -- what were your particular 18 responsibilities as a Pollution Control 19 Specialist I? 20 A. I wrote and edited the 305(b) report, which 21 is a requirement under the Clean Water Act. 22 And I also collected water samples for 23 compliance purposes for wastewater treatment 24 plants. And I prepared fish tissue as well 25 as other tissue for analysis.</p>	<p>1 did you have any contact or involvement in 2 any way with a facility in Washington County, 3 Minnesota, known as the Washington County 4 Landfill? 5 A. I did not. 6 Q. During that same period of time, did you have 7 any involvement or contact in any way with a 8 facility known as the Woodbury Landfill? 9 A. I did not. 10 Q. During that point in time, that same point in 11 time between 1986 and approximately 1990, 12 while you were at MPCA, did you have any 13 involvement or contact in any way with a 14 facility known as the 3M Chemolite plant or 15 Cottage Grove plant? 16 A. I did not. 17 Q. Why did you leave MPCA to begin working for 18 the Metropolitan Council? 19 A. At the time, the Metropolitan Council was 20 engaged in an effort focused on long-term 21 water supply planning for the metro region. 22 And as a geologist I was very interested in 23 that. And it also allowed me to use some of 24 the learning that I had gleaned while at the 25 Pollution Control Agency. So to help the</p>
Page 42	Page 44
<p>1 Q. And, again, none of the water samples you 2 collected was from any 3M facility; is that 3 correct? 4 A. No. 5 Q. Did you ever collect any water samples -- 6 first of all, were these surface water 7 samples? 8 A. Correct. 9 Q. When you became a Pollution Control 10 Specialist II, how, if at all, did your 11 responsibilities change? 12 A. I moved to the, what's called the non-point 13 source pollution unit, again focused 14 primarily with surface water, but there was 15 always a groundwater component. And I 16 reviewed local water plans for consistency 17 with state rules and regulations. 18 Q. At any time while you worked for MPCA between 19 1986 and approximately 1990, the point when 20 you started working for the Metropolitan 21 Council, did you have any contact with a 22 facility in Minnesota in Washington County 23 known as the Oakdale disposal site? 24 A. I did not. 25 Q. Did you -- during that same period of time,</p>	<p>1 planning effort. 2 Q. The Metropolitan Council, this was within the 3 Twin Cities area of Minnesota? 4 A. Correct, yes. It's a regional planning 5 authority. 6 Q. Was the pay better at the Metropolitan 7 Council? 8 A. I believe so. 9 Q. Had you had any negative reviews by anyone at 10 the MPCA prior to going to the Metropolitan 11 Council? 12 A. No. 13 Q. Did you have any difficulties with any of the 14 individuals you were working with at MPCA 15 that led to you going to the Metropolitan 16 Council? 17 A. No. 18 Q. All right. While you were at the met -- 19 well, let me back up on moment. 20 Did you ever intern with 3M at any 21 point in time while you were at college? 22 A. No. 23 Q. While you were at the Metropolitan Council, 24 did you ever have any involvement or contact 25 with representatives of 3M on any issues?</p>

11 (Pages 41 to 44)

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**CH2MHILL** Analytical Services

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COG 8

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3MA00230243

Project #		Purchase Order #		Requested Analytical Method #		THIS AREA FOR LAB USE ONLY	
Project Name <i>ALBERT WASHINGTON LOGS</i>		Company Name <i>ALBERT</i>		Lab #		Page # of #	
Project Manager/Contact & Phone # <i>ANDREW HARTLEY (952) 234-6820</i>		Report Copy to:		Lab PIN		Custody Review	
Requested Completion Date: <i>8/26/92</i>		Site ID		Custody Scale		Y N	
Sample Description: <input type="checkbox"/> Degrade <input type="checkbox"/> Meth <input type="checkbox"/> QC		CLIENT SAMPLE ID (9 CHARACTER)		pH		Custody Scale Y N	
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				METHANE			
				NITRATE			
				SULFATE			
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Special Instructions		Printed on and returned		Date/TIME <i>8/26/92</i>		Shipping #	

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1m4m Ship to 4m 3M Environmental Technology & | Date 06/24/97 No. 38-973740 0m  
 4m Service Laboratory, B-2-3E-09 | Routing 0m  
 4m 935 Bush Avenue | F.O.B. 0m  
 4m St. Paul, Minnesota 55144-1000 | Date 0m  
 | Shipped 0m  
 4m Attn: Robert Howell | B/L 0m

4m Quantity	Product Code / Description	Weight	0m
4m 1	Cooler of soil samples/soil & ice	100 lbs	0m
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4m			0m
4m			0m
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4m Originator VERA L WIGAL Phone 304-863-4895 0m  
 4m Charge (Cost Code) 8309066742001280 | MSDS Sheets Required? 0m  
 4m Purchase Order No. | Hazardous Material? N 0m  
 4m Value for Customs | MATERIAL CLASSIFICATION 0m  
 Ship From 4m Stores, Bldg. 4 | Corrosive Toxic 0m  
 | Flammable Other 0m

\*\* AUTHORIZATION \*\*  
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Doc #

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 3MA00230249

Project Name: **LAKE WASHINGTON WORKS, C-8 STORY**  
 Company Name: **LAKE WASHINGTON ENVIRONMENTAL CORP.**  
 Project Manager: **ANDREW HARTLEY** Report Copy To: **SM**  
 Requested Completion Date: **MM-1** Sample Disposal:  Recycle  Other

Sampling Date	Time	Type		Matrix		CLIENT SAMPLE ID (or CHARACTERS)	LAB OC	PRESERVATIVE	PH	Lab #	Page of
		COR	LAB	W	A						
1550	11	X	X	S	S	-1-0-2					
1551	11	X	X	S	S	-1-4-6					
1610	11	X	X	S	S	-1-8-10					
1620	11	X	X	S	S	-1-12-14					
1630	11	X	X	S	S	-1-16-18					
1634	11	X	X	S	S	-1-20-22					
1645	11	X	X	S	S	-1-24-26					
1650	11	X	X	S	S	-1-28-30					

Requested Analytical Method #  
 C-8  
 C-7  
 SURFACTANTS (C.B., IRON...)  
 METHANE  
 NITRATE  
 SULFATE  
 SULFIDE

Lab # \_\_\_\_\_ Page of \_\_\_\_\_  
 Lab PM \_\_\_\_\_ Custody Review \_\_\_\_\_  
 Custody Seal Y N  
 Lab ID \_\_\_\_\_

QC Level 1 2 3 Other \_\_\_\_\_  
 Cooler Temperature 330F  
 Alternate Description \_\_\_\_\_

Shipped By: **ANDREW HARTLEY**  
 Shipped Via: **UPS** Other: \_\_\_\_\_  
 Definition: **C12497 1600**

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Page 45	Page 47
<p>1 A. I don't recall. I don't believe so. 2 Q. Why did you leave the Metropolitan Council to 3 begin working in Seattle in approximately 4 1992? 5 A. Personal reasons. My husband and I had grown 6 up in the St. Croix River Valley, which is 7 where we lived. And we thought it would be 8 interesting to live in a new place. And 9 Seattle was very inviting from that regard. 10 And, again, the position that King County had 11 open was very attractive to me from a 12 learning perspective. 13 I had spent a fair amount of time, as 14 you have seen, working in water planning. 15 And in the early '90s that particular region, 16 the Puget Sound region, was and continues, I 17 believe, to experience explosive growth and 18 changes in land use, which have considerable 19 impact on surface water resources. And the 20 position that I was -- that I took at King 21 County was engaged in trying to come up with 22 engineering -- innovative engineering ideas 23 to ameliorate the pollution concerns that 24 were the result of the explosive growth, in 25 particular, trying to save the native salmon</p>	<p>1 Q. And during the time that you were working for 2 that consulting firm, did you provide any 3 services to the 3M Company? 4 A. I did not, although I did provide services to 5 the City of Woodbury. 6 Q. And what services did you provide to 7 Woodbury? 8 A. Well field analysis. 9 Q. And this is the City of Woodbury, Minnesota, 10 correct? 11 A. Correct. 12 Q. What kind of well field analysis did you 13 provide? 14 A. Looking at their well spacing and in their 15 existing well field. 16 Q. Are you referring to their drinking water 17 well? 18 A. Correct. 19 Q. Why was the City of Woodbury, to your 20 knowledge, looking at that issue at that 21 point in time? 22 A. The City of Woodbury, at that time and in the 23 current time, is, again, undergoing quite 24 expansive growth. And every city in their 25 municipal systems have to make sure that they</p>
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<p>1 runs in that area. 2 Q. Was there an increase in pay when you went to 3 the position in Seattle? 4 A. No. 5 Q. Was it a decrease in pay? 6 A. It wasn't a decrease, but the cost of living 7 was quite substantially higher in Seattle. 8 Q. And why did you leave the position in Seattle 9 to begin working for Northern Environment 10 Technology back in Minnesota in 1994? 11 A. Again, personal and professional reasons. I 12 had had a baby while we were in Seattle, our 13 first child. And we had no family or support 14 system. And so it just became more -- it 15 became more important for us to be back in 16 the midwest after we had our son. 17 And I also had worked for government 18 for quite some time at various different 19 levels, state, regional and local level, and 20 was ready to try the private sector. 21 Q. Was there an increase in pay going to 22 Northern Environmental Technology? 23 A. Yes, but not substantial. 24 Q. And was this a private consulting firm? 25 A. It is a private consulting firm, yes.</p>	<p>1 have enough water for their planned capacity, 2 for the -- the plan that they have in their 3 comprehensive efforts, planning efforts and 4 that's why we took a look at it. 5 Q. So the City of Woodbury retained Northern 6 Environmental Technologies to do this well 7 work; is that correct? 8 A. Uh-huh. 9 Q. And what in particular was Northern 10 Environmental Technologies asked to do for 11 the City? 12 A. Just look at the spacing between their 13 municipal wells to make sure that there 14 wouldn't be any significant drawdown as a 15 result of any further growth. 16 Q. While you were at Northern Environmental 17 Technologies, did you personally work on that 18 project? 19 A. I did. 20 Q. And were, to your knowledge -- let me restate 21 that. 22 To your knowledge, was Northern 23 Environmental Technologies asked to do any 24 sampling of the water Woodbury water wells 25 for any contaminants of any kind?</p>

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