



**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

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ASH010474

**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 LR No. | DuPont<br>Smpl. No. | Matrix       | POAA <sup>(1)</sup> ,<br>ppb | Vol. & Semi-<br>Vol. F, Cl,<br>Br by AED,<br>ppm |
|-----------|---------------------|--------------|------------------------------|--------------------------------------------------|
| R2008-1   | 1                   | ground water | 52                           | < MCL <sup>(3)</sup>                             |
| R2008-2   | 2                   | ground water | 49                           | < MCL                                            |
| R2008-3   | 3                   | ground water | 52                           | < MCL                                            |
| R2008-4   | 4                   | ground water | 53                           | < MCL                                            |
| R2008-5   | 5                   | DI blank     | n.d. <sup>(2)</sup>          | < MCL                                            |
| R2008-6   | none                | soil         | 364                          | < MCL                                            |

(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

Reg Smith - Fluoride 120,000 ppm  
Industrial Soil 4,700 ppm  
Resid. Soil 4,700 ppm  
Tap Water 2.2 ppm

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

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| 3M LR No. | DuPont Smpl. No. | POAA <sup>(1)</sup> mg/L | Total Fluorine mg/L | Fluoride Ion mg/L | Organic F, mg/L <sup>(4)</sup> | Adsorb. Organic F, mg/L | Vol. & Semi-Vol. F by AED, ppm | Trichloroethene 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                  |              |
| R2148-2   | MW-1-2           | 5.32                     | 16                  | 0.20              | 16                             | 4.2                     | 0.20 < [F] < 2.5               | 730                  | 10           |
| R2148-3   | MW-2-1           | 0.234                    | 3.3                 | 0.16              | 3.1                            | 0.28                    | 0.20 < [F] < 2.5               | 1600                 |              |
| R2148-4   | MW-2-2           | 0.234                    | 3.5                 | 0.16              | 3.3                            | 0.46                    | 0.20 < [F] < 2.5               | 1500                 |              |
| R2148-5   | MW-3-1           | 0.487                    | 4.2                 | 0.14              | 4.1                            | 1.1                     | 0.20 < [F] < 2.5               | 2300                 |              |
| R2148-6   | MW-3-2           | 0.477                    | 3.3                 | 0.14              | 3.2                            | 0.76                    | 0.20 < [F] < 2.5               | 2400                 |              |
| R2148-7   | MW-4-1           | 0.0842                   | 4.0                 | 0.11              | 3.9                            | 0.19                    | 0.20 < [F] < 2.5               | 760                  |              |
| 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>(2)</sup>     | 3.0                 | < 0.10            | 2.9                            | 0.11                    | [F] < 0.05                     | 130                  |              |
| R2148-10  | MW-5-2           | < POL <sup>(2)</sup>     | 2.8                 | < 0.10            | 2.7                            | 0.14                    | 0.05 < [F] < 0.20              | 140                  |              |
| R2148-11  | MW-6-1           | < MDL <sup>(3)</sup>     | 2.3                 | 0.10              | 2.2                            | < 0.05                  | [F] < 0.05                     |                      |              |
| R2148-12  | MW-6-2           | < MDL <sup>(3)</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                      |                             |                 |                            |                      |                        |                             |
| R2148-2   | MW-1-2           | 49                      |                             |                 |                            |                      |                        |                             |
| R2148-3   | MW-2-1           |                         |                             |                 |                            | 140                  |                        | 16                          |
| R2148-4   | MW-2-2           |                         |                             |                 |                            | 150                  |                        | 18                          |
| R2148-5   | MW-3-1           |                         |                             |                 |                            | 520                  | 5                      | 26                          |
| R2148-6   | MW-3-2           |                         |                             |                 |                            | 570                  | 5.2                    | 26                          |
| R2148-7   | MW-4-1           |                         | 5.8                         | 37              | 6.3                        | 81                   |                        | 5.9                         |
| R2148-8   | MW-4-2           |                         | 5.9                         | 39              | 6.2                        | 66                   |                        |                             |
| R2148-9   | MW-5-1           |                         |                             | 13              |                            |                      |                        |                             |
| R2148-10  | MW-5-2           |                         |                             | 16              | 5.8                        |                      |                        |                             |
| 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. = 0.0265 mg/L (4) Total Fluorine - Fluoride Ion = Organic Fluorine

## Summary of Results

## DuPont Washington Works Soil Samples

Sample Date 6/23/97

| 3M LR No. | DuPont Sample No. | Total Fluoride, mg/kg | POAA <sup>(1)</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'       | 33,500                | 13.1                        | 100            | <2             | <0.10          | 5.8         | 5.2                     | 13.1           | 13.6        |
| R2382-10  | SS-1 36-68'       | 41,200                | 6.78                        | 63             | <2             | <0.10          | 6.1         | 5.4                     | 9.6            | 17.9        |
| R2382-11  | SS-1 38-40'       | 30,300                | 2.11                        | 46             | <2             | <0.10          | 6.8         | 6.2                     | 6.3            | 22.2        |

(1) POAA = Perfluorooctanoic acid anion

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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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## Data Transmittal Summary

Final

| Project Information                                                                                                                                              |             |                                    |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|------------------------------------|
| Lab Request #:                                                                                                                                                   | 3M Study #: | Contract Laboratory #              |
| R2008                                                                                                                                                            |             |                                    |
| Date Received:                                                                                                                                                   |             |                                    |
| Sponsor or Client: Representative Name<br>Company Name DuPont<br>Company Address<br>Phone                                                                        |             |                                    |
| Project Lead: Kris Hansen (8-6018)<br>Group Leader: Jim Johnson (8-5294)                                                                                         |             |                                    |
| Analyte(s) or Test Method #: POAA<br>Sample Matrix: water and soil<br>Analysis Dates: 9/97-11/97<br>Author: kjh<br>Analyst(s): GML, JJ, kjh<br>Data Reviewed by: |             |                                    |
| Project Lead (or designee):<br>James D. Johnson (or designee):                                                                                                   |             |                                    |
| Copy to: Internal                                                                                                                                                |             |                                    |
| JDI:<br>QAU (Archives):<br>LIRN System:<br>Project Manager: Sue Beach                                                                                            |             | Sent by: / Date<br>kjh on 10/21/97 |
| Others (List Recipients / Address / Phone / FAX)<br>T. DiPasquale, 22-11E-03; 3-1891; 736-3257                                                                   |             | Sent by: / Date<br>kjh on 11/10/97 |

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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**

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#### **Report - Analytical Study Determination of POAA in Soil and Water**

##### **1.0 Summary**

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

**Table 1. Concentration of POAA in R2008 samples.**

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

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

##### **2.0 TEST MATERIALS**

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

##### **3.0 EXPERIMENTAL-OVERVIEW AND METHODS**

###### **3.1.1 Sample, soil**

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

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

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

### 3.1.2 Sample, water

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

All five water samples were prepared, in duplicate, for analysis using the Series B protocol. The samples were analyzed by ES/MS between two unextracted curves of POAA in ACN/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 autovials with HPLC-grade ACN and milli-Q water.

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### 3.4 ES/MS Analysis specifics, soil and water

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

Mobile phase: ACN/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 solution and increases system sensitivity to the target analyte.

## 4.0 DATA ANALYSIS

### 4.1 Sample, soil

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

### 4.2 Samples, water

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

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

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

#### 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:

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

where,

%R = Percent recovery of POAA

$C_t$  = Concentration of POAA found in MS/MSD ( $\mu\text{g/mL}$ )

$C_n$  = Native concentration of POAA in sample before dilution adjustment ( $\mu\text{g/mL}$ )

$C_s$  = Concentration of POAA spiked in MS sample ( $\mu\text{g/mL}$ ).

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

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

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

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

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 ( $\mu\text{g/mL}$ ) | Spiked Concentration ( $\mu\text{g/mL}$ ) | Native Concentration ( $\mu\text{g/mL}$ ) | % Recovery | Extractability |
|--------|----------------------------------------------|-------------------------------------------|-------------------------------------------|------------|----------------|
| MS     | 0.057                                        | 0.052                                     | 0.02                                      | 74         | 1.3            |
| MSD    | 0.059                                        | 0.052                                     | 0.02                                      | 75         | 1.3            |
|        |                                              |                                           | Average                                   | 75         | 1.3            |

#### 5.0 CONCLUSION

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

#### 6.0 MAINTENANCE OF RAW DATA AND RECORDS

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

Sample preparation : GML/JJ

Analysis: GML/kjh

Report preparation: kjh

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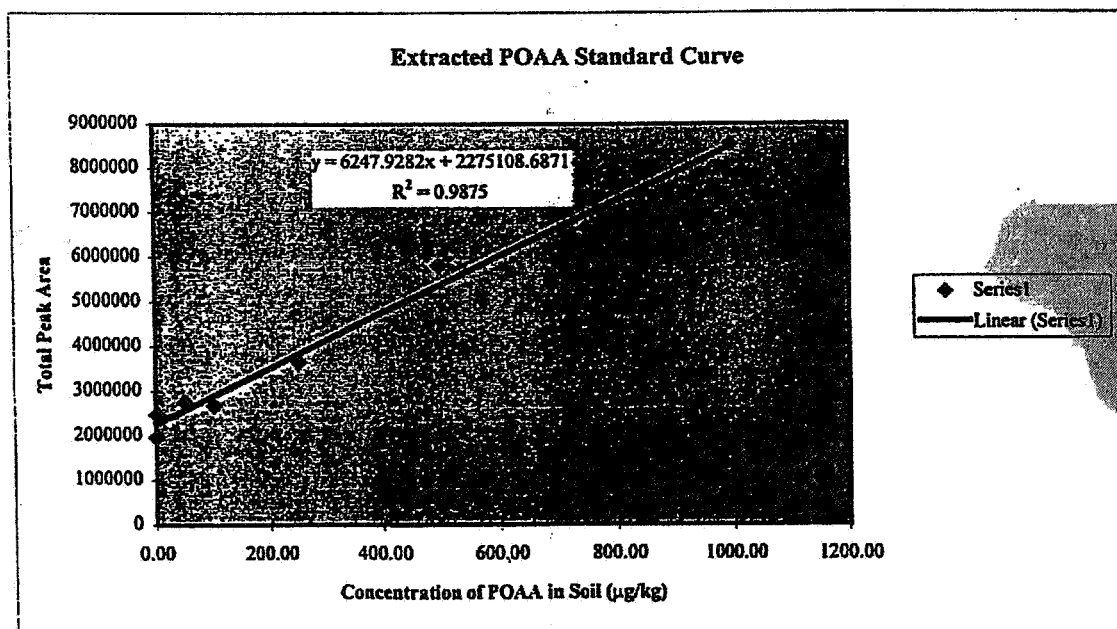
**R-2008 DuPont Soil Data**  
**POAA Standard Curve and Data Table**

**DATA TABLE 1.0**

| Spiked Standard<br>Conc. of POAA in<br>Extract (µg/mL) | Volume of<br>Extract<br>(mL) | Spiked Amount of<br>of POAA in<br>Extract (µg) | Mass of<br>Soil<br>(g) | Conc. of<br>POAA in<br>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<br>Soil (µg/kg) | Peak Area of<br>Extract-a | Peak Area of<br>Extract-b | Total Peak<br>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:

$$\begin{array}{c}
 \text{Conc. of POAA, from} \\
 \text{spiked standards,} \\
 \text{in the extract}
 \end{array}
 \times
 \begin{array}{c}
 \text{Volume} \\
 \text{of} \\
 \text{Extract}
 \end{array}
 \times
 \begin{array}{c}
 \text{Mass} \\
 \text{of} \\
 \text{Soil}
 \end{array}
 \times
 \begin{array}{c}
 \text{Convert} \\
 \text{g to} \\
 \text{kg}
 \end{array}
 =
 \begin{array}{c}
 \text{Conc. of} \\
 \text{POAA in} \\
 \text{Soil}
 \end{array}$$

$$\frac{\mu\text{g}}{\text{mL}} \times \frac{\text{mL}}{1} \times \frac{1}{\text{g}} \times \frac{1000 \text{ g}}{\text{kg}} = \frac{\mu\text{g}}{\text{kg}}$$

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

Using the Method of Standard Addition determine indigent analyte concentration by solving for for the x-intercept:

where  $y = 0$ , and equation of the slope of the line of Peak Area vs. Conc. of POAA in Soil is:

$$y = 6248x + 2275109$$

$$x = -2275109 / 6248$$

$$x = -364$$

concentration of indigent analyte POAA in soil determined to be 364  $\mu\text{g/kg}$

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

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# 3M Environmental Laboratory

## Data Transmittal Summary

Final

| Project Information                                                                       |             |                       |
|-------------------------------------------------------------------------------------------|-------------|-----------------------|
| Lab Request #:                                                                            | 3M Study #: | Contract Laboratory # |
| R2148                                                                                     |             |                       |
| Date Received:                                                                            |             |                       |
| Sponsor or Client: Representative Name<br>Company Name DuPont<br>Company Address<br>Phone |             |                       |
| Project Lead: Kris Hansen (8-6018)<br>Group Leader: Jim Johnson (8-5294)                  |             |                       |
| Analysis Information                                                                      |             |                       |
| 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<br>James D. Johnson (or designee):                        |             |                       |
| Copy Distribution                                                                         |             |                       |
| Internal                                                                                  |             | Sent by: / Date       |
| JDI:<br>QAU (Archives):<br>LIRN System:<br>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       |
| Comments                                                                                  |             |                       |
|                                                                                           |             |                       |

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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ASH010489

EID129588

# 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                                 |         |           |
| R2148-9-2 (MW-5)       | 23264          | 2               | <PQL                                 | <PQL    | <PQL      |
| R2148-10-1 (MW-5)      | 21873          | 2               | <PQL                                 |         |           |
| R2148-10-2 (MW-5)      | 26025          | 2               | <PQL                                 | <PQL    | <PQL      |
| R2148-11-1 (MW-6)      | 11696          | 2               | <MDL                                 |         |           |
| R2148-11-2 (MW-6)      | 11932          | 2               | <MDL                                 | <MDL    | <MDL      |
| R2148-12-1 (MW-6)      | 9132           | 2               | <MDL                                 |         |           |
| R2148-12-2 (MW-6)      | 10595          | 2               | <MDL                                 | <MDL    | <MDL      |

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## 2.0 TEST MATERIALS

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

## 3.0 EXPERIMENTAL-OVERVIEW AND METHODS

### 3.1 Investigative Samples

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

### 3.2 Matrix Spike Samples

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

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 ( $\mu$ g/mL)

$C_e$  = Concentration of ammonium perfluorooctanoate in extract ( $\mu$ g/mL)

$D$  = Dilution Factor

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

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

where,

$\%R$  = Percent recovery of ammonium perfluorooctanoate

$C_m$  = Detected concentration of ammonium perfluorooctanoate in MS sample ( $\mu$ g/mL)

$C_b$  = Average background concentration of ammonium perfluorooctanoate in sample ( $\mu$ g/mL)

$C_{me}$  = Expected concentration of ammonium perfluorooctanoate in MS sample ( $\mu$ g/mL).

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

$C_m = 0.404$   $\mu$ g/mL;

$C_b = 0.00$   $\mu$ g/mL;

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

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.

| <b>Table 2</b><br><b>Matrix Spike Results</b>                                                                                                                                              |                  |                                        |                                                   |                   |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------|----------------------------------------|---------------------------------------------------|-------------------|
| <b>Sample Type</b>                                                                                                                                                                         | <b>Sample ID</b> | <b>Recovered Concentration (µg/mL)</b> | <b>Expected Concentration<sup>1</sup> (µg/mL)</b> | <b>% Recovery</b> |
| DuPont Water                                                                                                                                                                               | R2148-12-1, MS   | 0.404                                  | 0.503                                             | 80                |
|                                                                                                                                                                                            | R2148-12-1, MSD  | 0.401                                  | 0.503                                             | 80                |
|                                                                                                                                                                                            |                  |                                        | <b>Average</b>                                    | <b>80</b>         |
| <b>Notes:</b><br><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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**POAA Analyses in Soil (6/97 Samples)**

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# 3M Environmental Laboratory

## Data Transmittal Summary Final

| Project Information                                                                       |             |                          |
|-------------------------------------------------------------------------------------------|-------------|--------------------------|
| Lab Request #:                                                                            | 3M Study #: | Contract Laboratory #    |
| R2382                                                                                     |             |                          |
| Date Received:                                                                            |             |                          |
| Sponsor or Client: Representative Name<br>Company Name DuPont<br>Company Address<br>Phone |             |                          |
| Project Lead: Kris Hansen (8-6018)<br>Group Leader: Jim Johnson (8-5294)                  |             |                          |
| Analyte Information                                                                       |             |                          |
| 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<br>James D. Johnson (or designee):                        |             |                          |
| Copy List (List all)                                                                      |             |                          |
| Internal                                                                                  |             | Sent by: / Date          |
| JDJ:<br>QAU (Archives):<br>LIRN System:<br>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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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  
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## **Report - Analytical Study Determination of POAA in Soil and Water Lab request - R2382**

### **1.0 Summary**

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

**Table 1. Concentration of POAA in R2382 samples.**

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

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

### **2.0 TEST MATERIALS**

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

### **3.0 EXPERIMENTAL-OVERVIEW AND METHODS**

#### **3.1.1 Sample, soil**

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

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

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

### 3.1 Extraction specifics

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

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

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

### 3.4 ES/MS Analysis specifics

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

Mobile phase: ACN/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 the extract and increases system sensitivity to the target analyte.

### 4.0 DATA ANALYSIS

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

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

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## 5.0 CONCLUSION

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

## 6.0 MAINTENANCE OF RAW DATA AND RECORDS

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

Sample preparation : GML/JJ

Analysis: GML/JJ/kjh

Report preparation: kjh/JJ

ES/MS  
was used

regression  
analysis

ASH010499

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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:**  $(31000 - 15000)/126000 = 0.127 \text{ ug/ml}$

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

**Example:**  $(0.127 \text{ ug/ml} * 50) + .119 \text{ ug/ml} = 6.47 \text{ ug/ml}$

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

**Example:**  $6.47 \text{ ug/ml} * 2 \text{ ml extract/} 1.9999 \text{ g} * 1000 \text{ g/kg} * 1 \text{ mg/} 1000 \text{ ug} = 6.47 \text{ mg/kg POAA}$

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

**Example:**  $6.47 \text{ mg/kg} + 0.43 \text{ mg/kg} = 6.90 \text{ mg/kg}$

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Calculations

R2382\_103097

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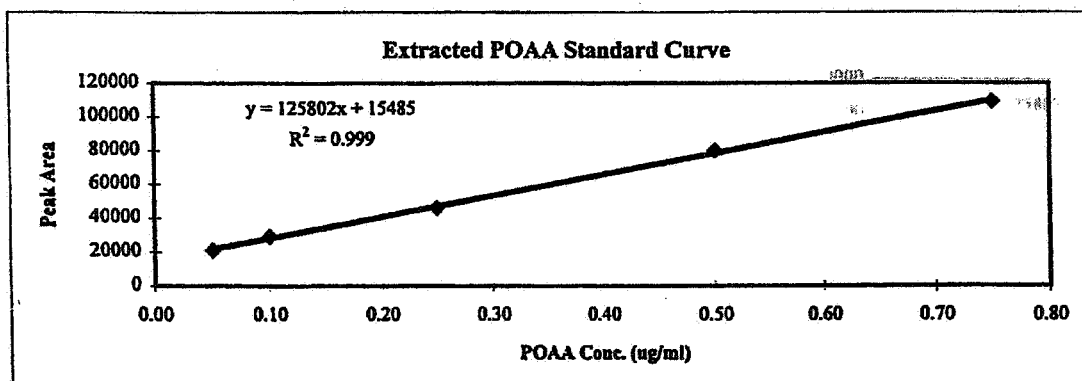
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Figure 1

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

| Conc. of POAA<br>Spiked into Soil<br>(ug/ml) | File D102897B<br>Peak Area of<br>Extracts-a | File D102897B<br>Peak Area of<br>Extracts-b | File D102897B<br>Total Peak Area<br>(a+b) | File D102897D<br>Total Peak Area<br>(a+b) | Average of<br>Initial & Final<br>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<br>Peak-a                     | File D102897D<br>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                                    |                                        |



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

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

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Soil 1 Curve ugml

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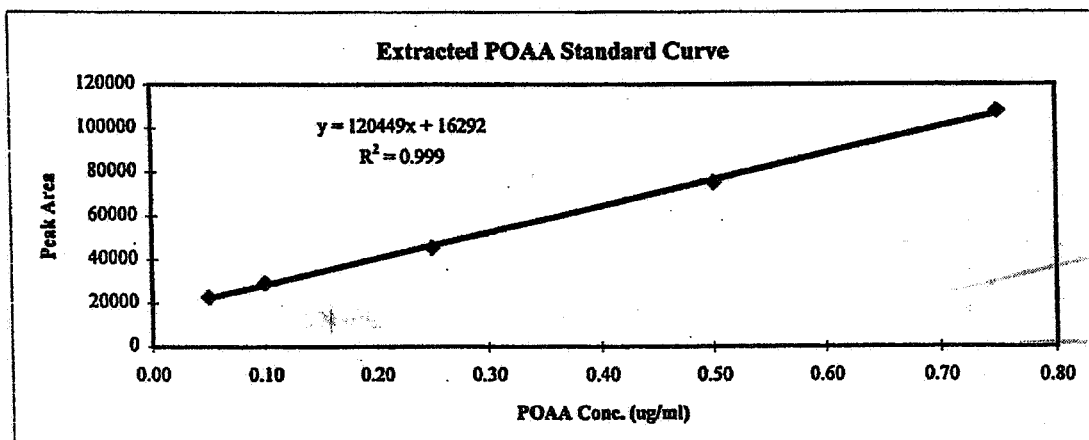
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Figure 2

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

| Conc. of POAA<br>Spiked into Soil<br>(ug/ml) | File D110497B<br>Peak Area of<br>Extracts-a | File D110497B<br>Peak Area of<br>Extracts-b | File D110497B<br>Total Peak Area<br>(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:  $\frac{\text{Y-intercept}}{16000} \quad \frac{\text{Slope (m)}}{120000} \quad \frac{\text{X-intercept}}{-0.133}$

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

ASH010502

11.04 Curve ugml

R2382\_103097

8:58 PM 11/4/97

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

ASH010503

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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.



**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

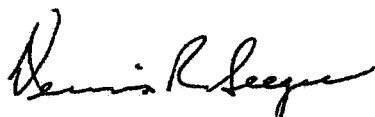
**Instrument Calibration**

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**

**Closing**

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



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

ASH010505

**Appendix A: Report of Laboratory Analysis**

ASH010506

# Method 8260 Results for R2148

| Compound                  | PRL<br>(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            | -                            | -       | 16      | 18      | 26      | 26      |
| Ethyl Ether               | 5             | -                            | -       | -       | -       | -       | -       |
| Trichlorotrifluoroethane  | 5             | 820                          | 730     | 1600    | 1500    | 2300    | 2400    |
| Acrolein                  | 40            | -                            | -       | -       | -       | -       | -       |
| 1,1-Dichloroethene        | 5             | -                            | -       | -       | 3.7 J   | -       | -       |
| Acetone                   | 10            | -                            | 10      | -       | -       | 7.8 J   | 8.0 J   |
| Isopropyl Alcohol         | 60            | -                            | -       | -       | -       | -       | -       |
| Carbon Disulfide          | 5             | -                            | -       | -       | -       | -       | -       |
| Allyl Chloride            | 5             | -                            | -       | -       | -       | -       | -       |
| Methylene Chloride        | 5             | 2.7 J                        | 2.9 J   | -       | -       | 2.3 J   | 2.7 J   |
| tert-Butyl Alcohol        | 20            | 59                           | 49      | -       | -       | -       | -       |
| tert-Methyl Butyl Ether   | 5             | -                            | -       | -       | -       | -       | -       |
| trans-1,2-Dichloroethene  | 5             | -                            | -       | -       | -       | 2.9 J   | 3.0 J   |
| Acrylonitrile             | 40            | -                            | -       | -       | -       | -       | -       |
| Isopropyl Ether           | 5             | -                            | -       | -       | -       | -       | -       |
| 1,1-Dichloroethane        | 5             | -                            | -       | -       | -       | -       | -       |
| 2,2-Dichloropropane       | 5             | -                            | -       | -       | -       | -       | -       |
| Ethyl Acetate             | 10            | -                            | -       | -       | -       | -       | -       |
| cis-1,2-Dichloroethene    | 5             | -                            | -       | -       | -       | 3.3 J   | 3.5 J   |
| 2-Butanone                | 10            | -                            | -       | -       | -       | -       | -       |
| 2-Butanol                 | 60            | -                            | -       | -       | -       | -       | -       |
| Bromochloromethane        | 5             | -                            | -       | -       | -       | -       | -       |
| Tetrahydrofuran           | 10            | -                            | -       | -       | -       | -       | -       |
| Chloroform                | 5             | -                            | -       | -       | -       | 5.5     | 5.9     |
| 1,1,1-Trichloroethane     | 5             | -                            | -       | -       | -       | 14      | 14      |
| Carbon Tetrachloride      | 5             | -                            | -       | -       | -       | -       | -       |
| 1,1-Dichloropropene       | 5             | -                            | -       | -       | -       | -       | -       |
| Isobutanol                | 100           | -                            | -       | -       | -       | -       | -       |
| Benzene                   | 5             | -                            | -       | -       | -       | -       | -       |
| 1,2-Dichloroethane        | 5             | -                            | -       | -       | -       | -       | -       |
| n-Butanol                 | 100           | -                            | -       | -       | -       | -       | -       |
| Trichloroethene           | 5             | 1.8 J                        | 1.5 J   | 140     | 150     | 520     | 570     |
| 1,2-Dichloropropane       | 5             | -                            | -       | -       | -       | -       | -       |
| Dibromomethane            | 5             | -                            | -       | -       | -       | -       | -       |
| Bromodichloromethane      | 5             | -                            | -       | -       | -       | -       | -       |
| 2-Chloroethyl Vinyl Ether | 10            | -                            | -       | -       | -       | -       | -       |
| 2-Nitropropane            | 10            | -                            | -       | -       | -       | -       | -       |
| cis-1,3-Dichloropropene   | 5             | -                            | -       | -       | -       | -       | -       |
| 4-Methyl-2-pentanone      | 10            | -                            | -       | -       | -       | -       | -       |
| Toluene                   | 5             | -                            | -       | -       | -       | -       | -       |
| 4-Methyl-2-Pentanol       | 60            | -                            | -       | -       | -       | -       | -       |

PRL – Practical Quantitation Limit

J – The concentration is below the practical quantitation limit.

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EID129606

# Method 8260 Results for R2148

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

PRL -- Practical Quantitation Limit

J -- The concentration is below the practical quantitation limit.

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# Method 8260 Results for R2148

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

PRL -- Practical Quantitation Limit

J -- The concentration is below the practical quantitation limit.

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ASH010510

EID129609

**3M Environmental Laboratory**

**Volatiles by AED (5/97 Samples)**

11/2/99

ASH010511

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EID129610

# 3M Environmental Laboratory

## Data Transmittal Summary

Preliminary Final (circle one)

|                                                                                                                                                                  |                                     |                       |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------|-----------------------|
| Lab Request #:<br>R2008                                                                                                                                          | 3M Study #:<br>3048/3058 Bigenenvir | Contract Laboratory # |
| Date Received:                                                                                                                                                   |                                     |                       |
| Sponsor or Client: Representative Name<br>Company Name DuPont<br>Company Address<br>Phone                                                                        |                                     |                       |
| Project Lead: Name / Phone KHansen / 80018<br>Group Leader: Name / Phone JDJohnson / 85294                                                                       |                                     |                       |
| Analyte(s) or Test Method #: POAA<br>Sample Matrix: Water, Soil<br>Analysis Dates: 060997 - 062697 Analyst(s): SEMiller<br>Author: SEMiller<br>Data Reviewed by: |                                     |                       |
| Project Lead (or designee):<br>James D. Johnson (or designee):                                                                                                   |                                     |                       |
| Internal                                                                                                                                                         |                                     | Sent by: / Date       |
| JDJ:<br>QAU (Archives):<br>LIRN System:<br>Project Manager: Sue Beach<br>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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EID129611

ASH010512



## 3M Environmental Laboratory - Advanced Method Development Team

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

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

#### 1.0 SUMMARY

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

#### 2.0 INTRODUCTION

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

#### 3.0 TEST MATERIALS

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

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

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 25 $\mu$ L, 50 $\mu$ L, and 100 $\mu$ L. Acetone and methanol spikes (100 $\mu$ L 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 |

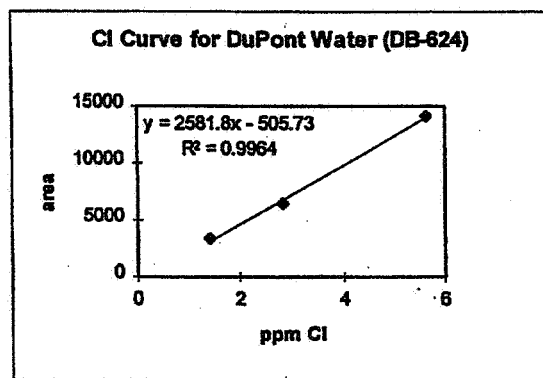
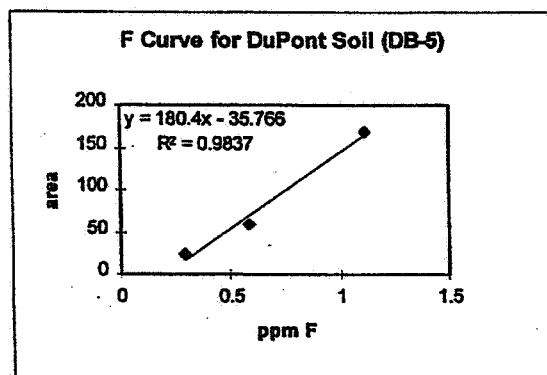
\*minimum quantitation limit: 0.280 ppm F in water, 0.282 ppm F in soil

| 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:



ASH010516

## 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

ASH010517

**3M Environmental Laboratory**

**Volatiles by AED (6/97 Samples)**

ASH010518

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EID129617

### 3M Environmental Laboratory

#### Data Transmittal Summary

Preliminary Final (circle one)

|                                                                                                                                                                                                                        |                                |                       |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------|-----------------------|
| Lab Request #:<br>R2148                                                                                                                                                                                                | 3M Study #:<br>3048 Bigenenvir | Contract Laboratory # |
| Date Received: 070297                                                                                                                                                                                                  |                                |                       |
| Sponsor or Client: Representative Name<br>Company Name DuPont<br>Company Address<br>Phone                                                                                                                              |                                |                       |
| Project Lead: Name / Phone KHansen / 86018<br>Group Leader: Name / Phone JDJohnson / 85294                                                                                                                             |                                |                       |
| Analyte(s) or Test Method #: Fluorine, Carbon, Hydrogen, Chlorine, Bromine<br>Sample Matrix: Water<br>Analysis Dates: 080597 - 082097 Analyst(s): SEMiller<br>Author: SEMiller<br>Data Reviewed by: PA Rethwill 100797 |                                |                       |
| Project Lead (or designee):<br>James D. Johnson (or designee):                                                                                                                                                         |                                |                       |
| Internal                                                                                                                                                                                                               |                                | Sent by: / Date       |
| JDJ:<br>QAU (Archives):<br>LIRN System:<br>Project Manager: Sue Beach                                                                                                                                                  |                                |                       |
| Others (List Recipients / Address / Phone / FAX)                                                                                                                                                                       |                                | Sent by: / Date       |
|                                                                                                                                                                                                                        |                                |                       |
|                                                                                                                                                                                                                        |                                |                       |
|                                                                                                                                                                                                                        |                                |                       |

ASH010519

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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EID129618

### 3M Environmental Laboratory-Advanced Method Development Team

Contact: Kris Hansen  
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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*                                                                  | Results |
|--------|---------|------------------------------------|--------------------------------------------------------------------------|---------|
| DB-5   | 1-8     | halogenated organics               | 0.23-1.3 ppm F, 0.29-0.86 ppm C,<br>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<br>than low standard response       |         |
| DB-5   | 11-12   | C only detected                    | C area less than low standard<br>response                                |         |
| DB-624 | 1-8     | halogenated organics               | 0.23-1.1 ppm F, 0.24-0.50 ppm C,<br>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<br>less than low standard response |         |
| DB-624 | 11-12   | C and F only detected              | C and F areas less than low standard<br>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.



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

\*same low standard concentrations as above

concentrations as above

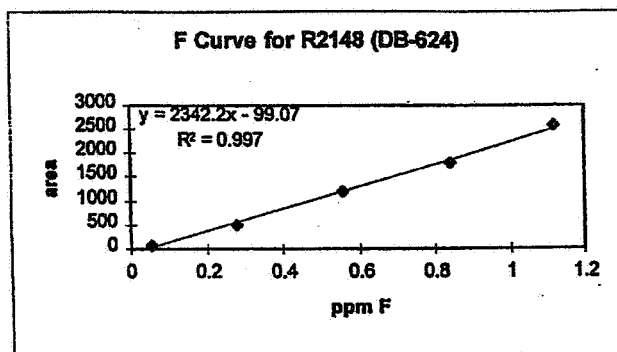
All results were normalized, assuming the density of water = 1g/mL.

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### STANDARD CURVES:

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



### 6.0 CONCLUSIONS

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

### 7.0 MAINTENANCE OF RAW DATA

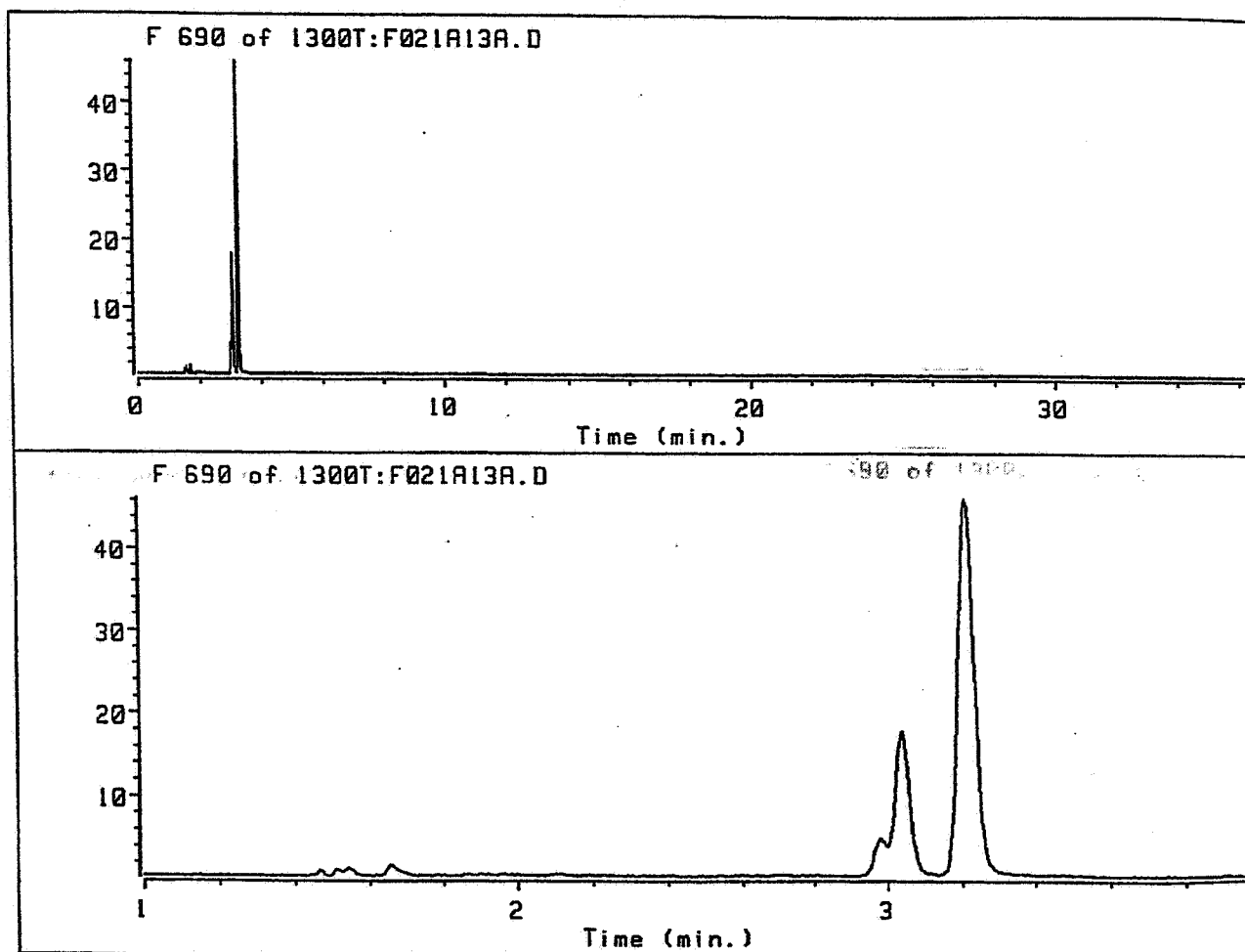
Copies of all data will be filed in the AMDT archive.

### 8.0 APPENDICES

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

SEMiller  
110797

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



3M Environmental Laboratory

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

ASH010528

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EID129627



### 3M Environmental Laboratory

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

|                                                                                                                                                                                                    |             |                                   |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------|-----------------------------------|
| Lab Request #:<br>R2382                                                                                                                                                                            | 3M Study #: | Contract Laboratory #             |
| Date Received: 06/06/97                                                                                                                                                                            |             |                                   |
| Sponsor or Client: Roger Zipfel<br>Dupont Washington Works Plant                                                                                                                                   |             |                                   |
| Project Lead: Name / Phone Kris Hansen / (612) 778-6018                                                                                                                                            |             |                                   |
| Analyte(s) or Test Method #: Total Fluoride<br>Sample Matrix: Soil<br>Analysis Dates: 9/29/97 through 10/01/97 Analyst(s): Daniel Howman<br>Author: Daniel Howman<br>Data Reviewed by: Kris Hansen |             |                                   |
| Internal                                                                                                                                                                                           |             | Sent by: / Date<br>DRH / 11-07-97 |
| Reviewer: James D. Johnson<br>QAU (Archives): Rich Youngblom<br>LIRN System: Denise Appleton<br>Project Manager: Sue Beach                                                                         |             |                                   |
| Others (List Recipients / Address / Phone / FAX)<br>Project Lead: Kris Hansen                                                                                                                      |             | Sent by: / Date<br>DRH / 11-07-97 |

ASH010529

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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EID129628

### **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.

| <b>Summary Table:</b>                            |                     |       |       |        |       |        |       |
|--------------------------------------------------|---------------------|-------|-------|--------|-------|--------|-------|
| <b>Soil Samples from Dupont Washington R2382</b> |                     |       |       |        |       |        |       |
| Fluoride:                                        | Average (mg/Kg):    | 21300 | 20100 | 61,200 | 78300 | 106300 | 82700 |
|                                                  | Standard Deviation: | 2800  | 2400  | 4200   | 6000  | 2100   | 1400  |
| Fluoride:                                        | Average (mg/Kg):    | 59100 | 37600 | 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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### 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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ASH010531

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EID129630

#### 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.

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## 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.

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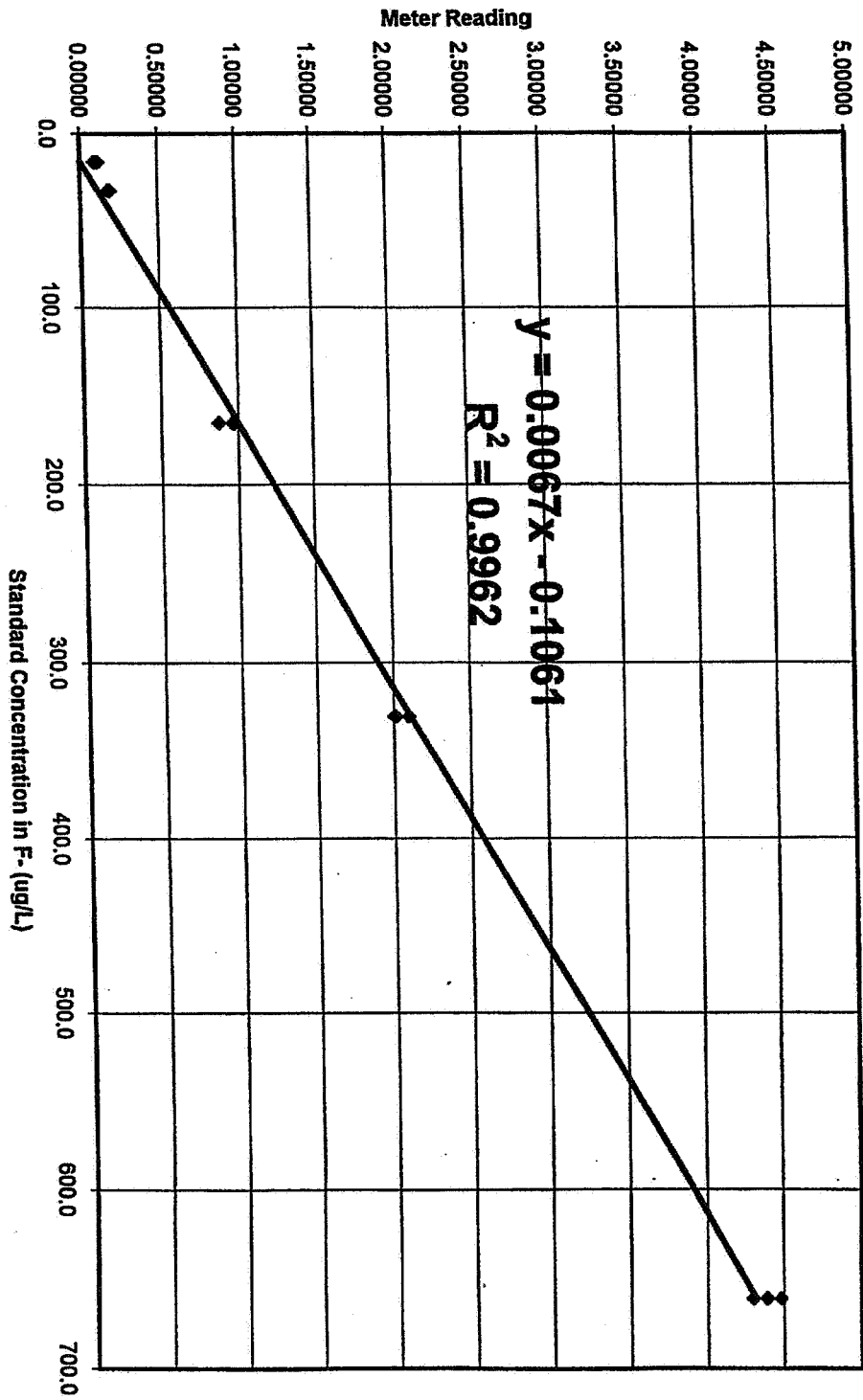
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Standard Curve POAA



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| Project: R2382                                                           |          |                    |                        |                      |                                  |               |        |                                                                                             |
|--------------------------------------------------------------------------|----------|--------------------|------------------------|----------------------|----------------------------------|---------------|--------|---------------------------------------------------------------------------------------------|
| Dupont Washington Soil Samples                                           |          |                    |                        |                      |                                  |               |        |                                                                                             |
| Calculations for Standard Curve                                          |          |                    |                        |                      |                                  |               |        |                                                                                             |
| Calculated F- (mg/L) = Meter Reading * Collection Volume / Sample Volume |          |                    |                        |                      |                                  |               |        |                                                                                             |
| Sample ID                                                                | Dilution | Sample Volume (mL) | Collection Volume (mL) | Calculated F- (mg/L) | Standard F- Concentration (mg/L) | Meter Reading | % Rec. | Comments                                                                                    |
| Analysis: DRH 9-29-97                                                    |          |                    |                        |                      |                                  |               |        |                                                                                             |
| QC 4.33PPM ERA CHECK                                                     | 2        |                    |                        |                      |                                  | 2.062         | 95.25  |                                                                                             |
| BLANK-1                                                                  | 1        | 0.01               | 3.0                    |                      |                                  | 0.05853       |        |                                                                                             |
| BLANK-2                                                                  | 1        | 0.01               | 3.0                    |                      |                                  | 0.03891       |        |                                                                                             |
| BLANK-3                                                                  | 1        | 0.01               | 3.0                    |                      |                                  | 0.02618       |        |                                                                                             |
| 25PPM W397-919                                                           | 1        | 0.01               | 3.0                    | 18.3                 | 16.5                             | 0.06097       | 110.69 |                                                                                             |
| 25PPM W397-919                                                           | 1        | 0.01               | 3.0                    | 16.5                 | 16.5                             | 0.05506       | 99.96  |                                                                                             |
| 25PPM W397-919                                                           | 1        | 0.01               | 3.0                    | 12.8                 | 16.5                             | 0.04256       | 77.26  |                                                                                             |
| 25PPM W397-919                                                           | 1        | 0.01               | 3.0                    | 13.9                 | 16.5                             | 0.04638       | 84.20  |                                                                                             |
| QC 1.0PPM CHECK                                                          | 1        | 0.01               | 3.0                    |                      |                                  | 0.9685        | 96.85  |                                                                                             |
| 50PPM W397-920                                                           | 1        | 0.01               | 3.0                    | 30.5                 | 33.1                             | 0.1016        | 92.22  | Due to low meter readings and low recovery, decided to rerun using 20uL as sample volume!!! |
| 50PPM W397-920                                                           | 1        | 0.01               | 3.0                    | 26.2                 | 33.1                             | 0.08735       | 79.29  |                                                                                             |
| 50PPM W397-920                                                           | 1        | 0.01               | 3.0                    | 21.7                 | 33.1                             | 0.07239       | 65.71  |                                                                                             |
| 250PPM W397-921                                                          | 1        | 0.01               | 3.0                    | 111.7                | 165.3                            | 0.3724        | 67.61  |                                                                                             |
| 250PPM W397-921                                                          | 1        | 0.01               | 3.0                    | 128.4                | 165.3                            | 0.4279        | 77.68  |                                                                                             |
| QC 1.0PPM CHECK                                                          | 1        |                    |                        |                      |                                  | 0.9845        | 98.45  |                                                                                             |
| BLANK-1                                                                  | 1        | 0.02               | 3.0                    |                      |                                  | 0.07984       |        |                                                                                             |
| BLANK-2                                                                  | 1        | 0.02               | 3.0                    |                      |                                  | 0.02783       |        |                                                                                             |
| BLANK-3                                                                  | 1        | 0.02               | 3.0                    |                      |                                  | 0.02694       |        |                                                                                             |
| 25PPM W397-919                                                           | 1        | 0.02               | 3.0                    | 13.7                 | 16.5                             | 0.09136       | 82.93  |                                                                                             |
| 25PPM W397-919                                                           | 1        | 0.02               | 3.0                    | 17.2                 | 16.5                             | 0.1149        | 104.25 |                                                                                             |
| 25PPM W397-919                                                           | 1        | 0.02               | 3.0                    | 16.0                 | 16.5                             | 0.1067        | 96.86  |                                                                                             |
| 50PPM W397-920                                                           | 1        | 0.02               | 3.0                    | 26.5                 | 33.1                             | 0.1764        | 80.04  |                                                                                             |
| 50PPM W397-920                                                           | 1        | 0.02               | 3.0                    | 28.6                 | 33.1                             | 0.1906        | 86.50  |                                                                                             |
| 50PPM W397-920                                                           | 1        | 0.02               | 3.0                    | 26.5                 | 33.1                             | 0.1764        | 80.04  |                                                                                             |
| QC 1.0PPM CHECK                                                          | 1        |                    |                        |                      |                                  | 0.9725        | 97.25  |                                                                                             |
| 250PPM W397-921                                                          | 1        | 0.02               | 3.0                    | 130.6                | 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         | 90.95  |                                                                                             |
| 500PPM W397-922                                                          | 1        | 0.02               | 3.0                    | 314.4                | 330.5                            | 2.096         | 95.13  |                                                                                             |
| 1000PPM W397-923                                                         | 1        | 0.02               | 3.0                    | 645.2                | 661.0                            | 4.301         | 97.51  |                                                                                             |
| 1000PPM W397-923                                                         | 1        | 0.02               | 3.0                    | 672.1                | 661.0                            | 4.481         | 101.68 |                                                                                             |
| 1000PPM W397-923                                                         | 1        | 0.02               | 3.0                    | 658.5                | 661.0                            | 4.390         | 99.63  |                                                                                             |
| QC 1.0PPM CHECK                                                          | 1        |                    |                        |                      |                                  | 0.9646        | 96.46  |                                                                                             |
| Notebook Reference: Carlton-SA-2, p.20-21                                |          |                    |                        |                      |                                  |               |        |                                                                                             |

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| Project: R2382                                                                   |          |                   |                        |               |                      |                       |                    |            |          |
|----------------------------------------------------------------------------------|----------|-------------------|------------------------|---------------|----------------------|-----------------------|--------------------|------------|----------|
| Soil Samples from Dupont Washington                                              |          |                   |                        |               |                      |                       |                    |            |          |
| Calculations for Total Fluoride in Soil                                          |          |                   |                        |               |                      |                       |                    |            |          |
| Calculated F- (mg/L) = (Meter Reading + Intercept) / Slope                       |          |                   |                        |               |                      |                       |                    |            |          |
| Calculated F- (mg/Kg) = Calculated F- (mg/L) * Collection Volume / Sample Weight |          |                   |                        |               |                      |                       |                    |            |          |
| Sample ID                                                                        | Dilution | Sample Weight (g) | Collection Volume (mL) | Meter Reading | Calculated F- (mg/L) | Calculated F- (mg/Kg) | Statistics (mg/Kg) | % Recovery | Comments |
| Analysis: DRH 9-30-97                                                            |          |                   |                        |               |                      |                       |                    |            |          |
| QC 4.33PPM ERA CHECK                                                             | 2        |                   |                        | 2.079         |                      |                       |                    | 96.0       |          |
| BLANK-1                                                                          | 1        |                   | 3.0                    | 0.07719       |                      |                       |                    |            |          |
| BLANK-2                                                                          | 1        |                   | 3.0                    | 0.05811       |                      |                       |                    |            |          |
| BLANK-3                                                                          | 1        |                   | 3.0                    | 0.05168       |                      |                       |                    |            |          |
| R2382-1-1                                                                        |          | 0.0203            | 3.0                    | 0.8047        | 136                  | 20,100                | AVE 21300          |            |          |
| R2382-1-2                                                                        |          | 0.0231            | 3.0                    | 0.8870        | 148                  | 19,200                | STD 2800           |            |          |
| R2382-1-3                                                                        |          | 0.023             | 3.0                    | 1.155         | 188                  | 24,500                | CV 13              |            |          |
| R2382-2-1                                                                        |          | 0.0236            | 3.0                    | 0.8080        | 136                  | 17,300                | AVE 20100          |            |          |
| R2382-2-2                                                                        |          | 0.0215            | 3.0                    | 0.9163        | 153                  | 21,300                | STD 2400           |            |          |
| R2382-2-3                                                                        |          | 0.0219            | 3.0                    | 0.9542        | 158                  | 21,700                | CV 12              |            |          |
| QC 1.0PPM CHECK                                                                  | 1        |                   |                        | 0.9698        |                      |                       |                    | 97.0       |          |
| R2382-3-1                                                                        |          | 0.0204            | 3.0                    | 2.485         | 387                  | 56,900                | AVE 61200          |            |          |
| R2382-3-2                                                                        |          | 0.0225            | 3.0                    | 2.983         | 461                  | 61,500                | STD 4200           |            |          |
| R2382-3-3                                                                        |          | 0.0202            | 3.0                    | 2.841         | 440                  | 65,300                | CV 7               |            |          |
| R2382-4-1                                                                        |          | 0.0235            | 3.0                    | 3.668         | 563                  | 71,900                | AVE 78300          |            |          |
| R2382-4-2                                                                        |          | 0.0227            | 3.0                    | 3.898         | 598                  | 79,000                | STD 6000           |            |          |
| R2382-4-3                                                                        |          | 0.0225            | 3.0                    | 4.109         | 629                  | 83,900                | CV 8               |            |          |
| R2382-4-4 w/10uL spike of 10,000ppm POAA                                         |          | 0.023             | 3.0                    | 8.189         | 940                  | 122,600               |                    | 51.4       |          |
| R2382-5-1                                                                        |          | 0.0228            | 3.0                    | 5.349         | 814                  | 107,100               | AVE 106300         |            |          |
| R2382-5-2                                                                        |          | 0.023             | 3.0                    | 5.436         | 827                  | 107,900               | STD 2100           |            |          |
| R2382-5-3                                                                        |          | 0.0226            | 3.0                    | 5.136         | 782                  | 103,900               | CV 2               |            |          |
| QC 1.0PPM CHECK                                                                  | 1        |                   |                        | 0.9856        |                      |                       |                    | 98.6       |          |
| R2382-6-1                                                                        |          | 0.021             | 3.0                    | 3.835         | 588                  | 84,000                | AVE 82700          |            |          |
| R2382-6-2                                                                        |          | 0.021             | 3.0                    | 3.789         | 581                  | 83,000                | STD 1400           |            |          |
| R2382-6-3                                                                        |          | 0.0214            | 3.0                    | 3.774         | 579                  | 81,200                | CV 2               |            |          |
| R2382-7-1                                                                        |          | 0.0218            | 3.0                    | 2.983         | 461                  | 63,400                | AVE 59100          |            |          |
| R2382-7-2                                                                        |          | 0.0201            | 3.0                    | 2.588         | 402                  | 60,000                | STD 4900           |            |          |
| R2382-7-3                                                                        |          | 0.021             | 3.0                    | 2.415         | 376                  | 53,800                | CV 8               |            |          |
| R2382-8-1                                                                        |          | 0.022             | 3.0                    | 1.811         | 286                  | 39,000                | AVE 37600          |            |          |
| R2382-8-2                                                                        |          | 0.0216            | 3.0                    | 1.841         | 291                  | 40,400                | STD 3600           |            |          |
| R2382-8-3                                                                        |          | 0.0215            | 3.0                    | 1.503         | 240                  | 33,500                | CV 10              |            |          |
| QC 1.0PPM CHECK                                                                  | 1        |                   |                        | 0.9777        |                      |                       |                    | 97.8       |          |
| R2382-9-1                                                                        |          | 0.0209            | 3.0                    | 1.485         | 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         | 216                  | 31,900                | CV 4               |            |          |
| QC 1.0PPM CHECK                                                                  | 1        |                   |                        | 0.9737        |                      |                       |                    | 97.4       |          |
| Analysis: DRH 10-1-97                                                            |          |                   |                        |               |                      |                       |                    |            |          |
| QC 4.33PPM ERA CHECK                                                             | 2        |                   |                        | 2.019         |                      |                       |                    | 93.3       |          |
| BLANK-1                                                                          | 1        |                   | 3.0                    | 0.1319        |                      |                       |                    |            |          |
| BLANK-2                                                                          | 1        |                   | 3.0                    | 0.06430       |                      |                       |                    |            |          |
| BLANK-3                                                                          | 1        |                   | 3.0                    | 0.05910       |                      |                       |                    |            |          |
| R2382-10-1                                                                       |          | 0.0203            | 3.0                    | 1.665         | 264                  | 39,100                | AVE 41200          |            |          |
| R2382-10-2                                                                       |          | 0.0201            | 3.0                    | 1.741         | 276                  | 41,100                | STD 2200           |            |          |
| R2382-10-3                                                                       |          | 0.0208            | 3.0                    | 1.909         | 301                  | 43,400                | CV 5               |            |          |
| R2382-11-1                                                                       |          | 0.0218            | 3.0                    | 1.288         | 208                  | 28,600                | AVE 30300          |            |          |
| R2382-11-2                                                                       |          | 0.0223            | 3.0                    | 1.471         | 235                  | 31,700                | STD 1600           |            |          |
| R2382-11-3                                                                       |          | 0.022             | 3.0                    | 1.402         | 225                  | 30,700                | CV 5               |            |          |
| QC 1.0PPM CHECK                                                                  | 1        |                   |                        | 0.9420        |                      |                       |                    | 94.2       |          |
| Notebook Reference: Carlton-SA-2, p.21-23                                        |          |                   |                        |               |                      |                       |                    |            |          |

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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 #:<br>R-2148                                                                                                                                                                                                                                                                                                            | 3M Study #:<br>Bigenenvir | Contract Laboratory # |
| Date Received:                                                                                                                                                                                                                                                                                                                      |                           |                       |
| Sponsor or Client: Representative Name Dale Bacon / Robert Howell<br>Company Name 3M<br>Company Address 935 Bush Ave. , St. Paul, MN<br>Phone 778-4736 / 778-7540                                                                                                                                                                   |                           |                       |
| Project Lead: Name / Phone Kris Hansen<br>Group Leader: Name / Phone 612-778-6018                                                                                                                                                                                                                                                   |                           |                       |
| Analyte(s) or Test Method #: Total Fluorine, Fluoride Ion, Adsorbable Organic Fluoride<br>Sample Matrix: Dupont Waters<br>Analysis Dates: 8/15/97-8/25/97 Analyst(s): Jan Schutz , Nancy Bergman<br>Author: Jan Schutz<br>Data Reviewed by:<br>Project Lead (or designee): Pat Rethwill 10/03/97<br>James D. Johnson (or designee): |                           |                       |
| Internal                                                                                                                                                                                                                                                                                                                            |                           | Sent by: / Date       |
| JDJ: Kris Hansen                                                                                                                                                                                                                                                                                                                    |                           | JGS /11/07/97         |
| QAU (Archives): Rich Youngblom                                                                                                                                                                                                                                                                                                      |                           | JGS /11/07/97         |
| LIRN System: Denise Appleton                                                                                                                                                                                                                                                                                                        |                           | JGS /11/07/97         |
| Project Manager: Sue Beach                                                                                                                                                                                                                                                                                                          |                           | JGS /11/07/97         |
| Others (List Recipients / Address / Phone / FAX)                                                                                                                                                                                                                                                                                    |                           | Sent by: / Date       |
|                                                                                                                                                                                                                                                                                                                                     |                           |                       |
|                                                                                                                                                                                                                                                                                                                                     |                           |                       |
|                                                                                                                                                                                                                                                                                                                                     |                           |                       |

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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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EID129637

## 3M Environmental Laboratory - Advanced Method Development Team

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

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

#### 1.0 SUMMARY

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

The following table contains a summary of the results.

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

#### 2.0 INTRODUCTION

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

#### 3.0 TEST MATERIALS

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

3M Environmental Laboratory, Lab Request R2148

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## INSTRUMENTATION

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

## OPERATING CONDITIONS

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

## REAGENTS

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

## 5.0 DATA ANALYSIS

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

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

A standard curve for total fluorine was generated by combusting 0.1 mL aliquots of 2.0, 5.0, 20, 50, and 100 µg/mL POAA standard (POAA is 66.1% fluoride) in the Dohrmann DX2000 Modified Organic Halide Analyzer. The off-gasses were collected in 3 mL of 1:1 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 µg/mL fluorine as POAA through two carbon columns. The carbon columns were combusted in the Modified Dohrmann DX2000 Organic Halide Analyzer. The effluent was collected in 3 mL of 1:1 TISAB II / Milli-Q water and analyzed with the Orion meter. Using least squares linear regression, plotting the fluoride concentration of the standard on the x axis, and the Orion meter response on the y axis, the following curve was generated:  $Y = 0.2894x + 0.0196$  and  $R^2 = 0.9949$ .

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**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<br>Sample<br>(mL) | Orion Meter<br>Reading<br>( $\mu\text{g/mL F-}$ ) <sup>1</sup> | Spiked $\mu\text{g/mL}$<br>F- in Sample<br>* |
|----------------------------|----------------------------|----------------------------------------------------------------|----------------------------------------------|
| 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.  $Y = 0.0341x - 0.0157$   $R^2 = 0.9988$

\*ammonium perfluorooctanoate standard is 66.1 % Fluoride

**Total Fluorine Sample Analysis**

| Sample ID       | Meter * | Quantity of<br>Sample (mL) | Total $\mu\text{g/mL}$ *<br>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 Schutz  
09-30-97

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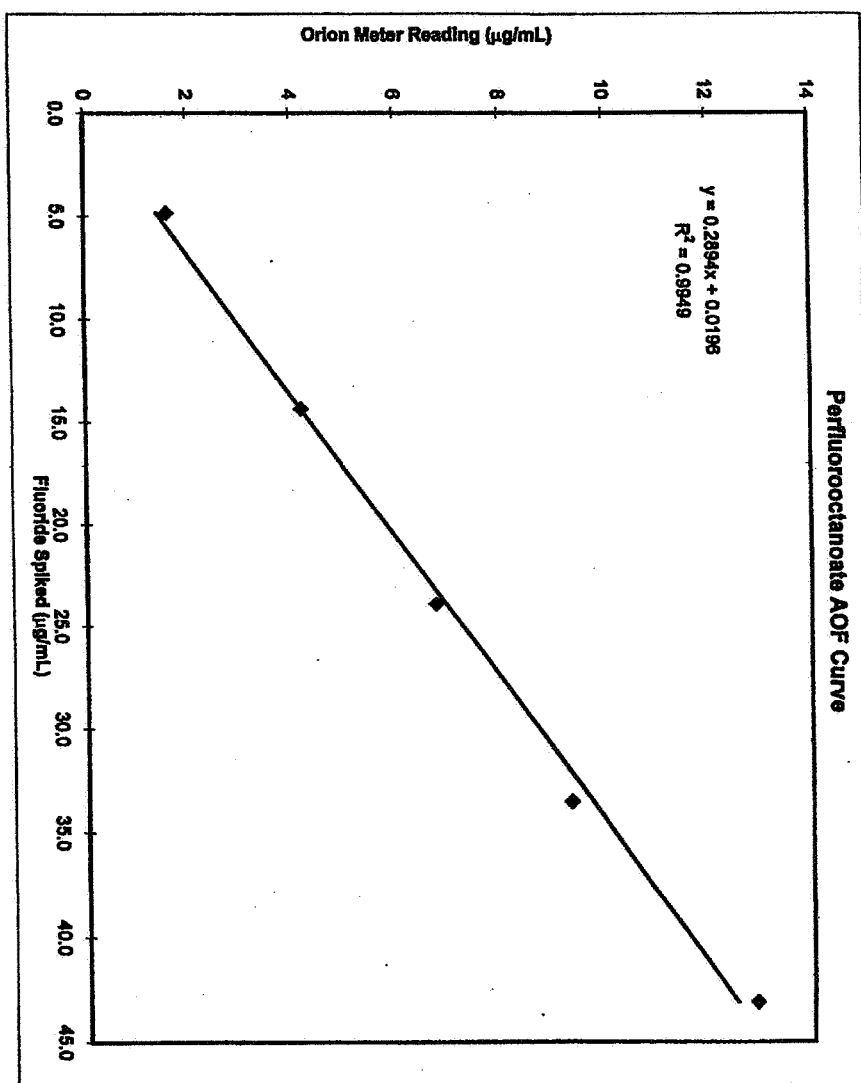
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Curve

ASH010544

| F-Conc.<br>in Standard | Meter<br>Reading |
|------------------------|------------------|
| 4.9                    | 1.6166           |
| 14.4                   | 4.1827           |
| 23.9                   | 6.7477           |
| 33.5                   | 9.3122           |
| 43.1                   | 12.8672          |



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Orion Data

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## 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   | 0.1622                        | 3.0              | 1               | 1                  | 0.4565             |                                |                                |                             |            |
| Sid # 1 bottom | 0.4536                        | 3.0              | 1               | 100                | 0.01361            |                                |                                |                             |            |
| Sid # 2 bottom | 0.3701                        | 3.0              | 1               | 100                | 0.01110            |                                |                                |                             |            |
| Sid # 3 bottom | 0.3643                        | 3.0              | 1               | 100                | 0.0109296          |                                |                                |                             |            |
| Sid # 4 bottom | 0.1815                        | 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                | 0.00163            |                                |                                |                             |            |
| Sid # 1 top    | 1.1820                        | 3.0              | 1               | 100                | 0.03486            | 1.616                          | 0.048                          | 0.049                       | 100%       |
| Sid # 2 top    | 3.8126                        | 3.0              | 1               | 100                | 0.1144             | 4.183                          | 0.125                          | 0.144                       | 87%        |
| Sid # 3 top    | 6.3634                        | 3.0              | 1               | 100                | 0.1915             | 6.748                          | 0.202                          | 0.239                       | 85%        |
| Sid # 4 top    | 8.1307                        | 3.0              | 1               | 100                | 0.2739             | 9.312                          | 0.279                          | 0.335                       | 83%        |
| Sid # 5 top    | 12.764                        | 3.0              | 1               | 100                | 0.3829             | 12.887                         | 0.387                          | 0.431                       | 90%        |
| QC check 5.0   | 5.098                         | 1.0              | 1               | 1                  | 5.098              |                                |                                |                             |            |

## FLUORIDE ION ANALYSIS

Total Fluoride

| Sample ID        | Meter Read | TISAB Vol | Dilution | Sample Vol | (µg/mL) |  |  |  |      |
|------------------|------------|-----------|----------|------------|---------|--|--|--|------|
| QC check 0.50 pp | 0.5018     | 1.0       | 1        | 1.0        | 0.502   |  |  |  | 100% |
| R2148-1          | 0.1005     | 1.0       | 2        | 1.0        | 0.201   |  |  |  |      |
| R2148-2          | 0.1017     | 1.0       | 2        | 1.0        | 0.203   |  |  |  |      |
| R2148-3          | 0.0813     | 1.0       | 2        | 1.0        | 0.163   |  |  |  |      |
| R2148-4          | 0.0801     | 1.0       | 2        | 1.0        | 0.160   |  |  |  |      |
| R2148-5          | 0.0701     | 1.0       | 2        | 1.0        | 0.140   |  |  |  |      |
| R2148-6          | 0.0686     | 1.0       | 2        | 1.0        | 0.139   |  |  |  |      |
| R2148-7          | 0.0567     | 1.0       | 2        | 1.0        | 0.113   |  |  |  |      |
| R2148-8          | 0.0556     | 1.0       | 2        | 1.0        | 0.111   |  |  |  |      |
| R2148-9          | 0.0465     | 1.0       | 2        | 1.0        | 0.093   |  |  |  |      |
| R2148-10         | 0.0463     | 1.0       | 2        | 1.0        | 0.093   |  |  |  |      |
| R2148-11         | 0.0491     | 1.0       | 2        | 1.0        | 0.098   |  |  |  |      |
| R2148-12         | 0.0497     | 1.0       | 2        | 1.0        | 0.099   |  |  |  |      |
| QC check 0.50 pp | 0.5039     | 1.0       | 1        | 1.0        | 0.504   |  |  |  | 101% |

Orion Data

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| AOF ANALYSIS           |               | AOF Calc. ( $\mu\text{g}/\text{mL}$ ) = ((total meter reading - intercept) / slope) / sample volume |                 |                 |                                  |  | Calc. Adsorbable Organic Fluorine ( $\mu\text{g}/\text{mL}$ ) |  | Comments                   |  | Recovery % |  |
|------------------------|---------------|-----------------------------------------------------------------------------------------------------|-----------------|-----------------|----------------------------------|--|---------------------------------------------------------------|--|----------------------------|--|------------|--|
| Sample ID              | Meter Reading | Collect Vol (mL)                                                                                    | Dilution Factor | Sample Vol (mL) | Meter Reading Total (top&bottom) |  |                                                               |  |                            |  |            |  |
| QC check 5.0 ppm       | 5.039         | 1.0                                                                                                 | 1               | 1               | 5.110                            |  |                                                               |  |                            |  | 101%       |  |
| AOF Instr Blank Bottom | 0.0280        | 3.0                                                                                                 | 1               | 1               | 6.044                            |  |                                                               |  |                            |  |            |  |
| R2148-1 bottom         | 0.3019        | 3.0                                                                                                 | 1               | 85              | 6.080                            |  |                                                               |  |                            |  |            |  |
| R2148-2 bottom         | 0.2972        | 3.0                                                                                                 | 1               | 70              | 6.236                            |  |                                                               |  |                            |  |            |  |
| R2148-3 bottom         | 0.1222        | 3.0                                                                                                 | 1               | 85              | 7.79                             |  |                                                               |  |                            |  |            |  |
| R2148-4 bottom         | 0.1368        | 3.0                                                                                                 | 1               | 90              |                                  |  |                                                               |  |                            |  |            |  |
| R2148-5 bottom         | 0.2861        | 3.0                                                                                                 | 1               | 85              |                                  |  |                                                               |  |                            |  |            |  |
| R2148-6 bottom         | 0.2803        | 3.0                                                                                                 | 1               | 85              |                                  |  |                                                               |  |                            |  | 98%        |  |
| QC check 5.0 ppm       | 4.903         | 1.0                                                                                                 | 1               | 1               | 125.2                            |  | 5.089                                                         |  | Read above top std, re-run |  |            |  |
| R2148-1 Top            | 124.9         | 3.0                                                                                                 | 1               | 85              | 103.0                            |  | 5.083                                                         |  | Read above top std, re-run |  |            |  |
| R2148-2 Top            | 102.7         | 3.0                                                                                                 | 1               | 70              | 6.776                            |  | 0.275                                                         |  |                            |  | 99%        |  |
| R2148-3 Top            | 6.654         | 3.0                                                                                                 | 1               | 85              | 11.29                            |  | 0.458                                                         |  |                            |  |            |  |
| R2148-4 Top            | 11.16         | 3.0                                                                                                 | 1               | 85              |                                  |  | 1.168                                                         |  | Read above top std, re-run |  |            |  |
| R2148-5 Top            | 30.15         | 3.0                                                                                                 | 1               | 90              | 30.44                            |  | 1.030                                                         |  | Read above top std, re-run |  |            |  |
| R2148-6 Top            | 25.09         | 3.0                                                                                                 | 1               | 85              | 25.37                            |  |                                                               |  |                            |  | 99%        |  |
| QC check 5.0 ppm       | 4.965         | 1.0                                                                                                 | 1               | 1               |                                  |  |                                                               |  |                            |  |            |  |
| MIII Q Blk Bottom      | 0.0565        | 3.0                                                                                                 | 1               | 100             |                                  |  |                                                               |  |                            |  |            |  |
| R2148-1 bottom         | 0.1633        | 3.0                                                                                                 | 1               | 5               |                                  |  |                                                               |  |                            |  |            |  |
| R2148-2 bottom         | 0.1910        | 3.0                                                                                                 | 1               | 5               |                                  |  |                                                               |  |                            |  |            |  |
| R2148-3 bottom         | 0.1423        | 3.0                                                                                                 | 1               | 10              |                                  |  |                                                               |  |                            |  |            |  |
| R2148-4 bottom         | 0.1128        | 3.0                                                                                                 | 1               | 10              |                                  |  |                                                               |  |                            |  |            |  |
| R2148-5 bottom         | 0.2298        | 3.0                                                                                                 | 1               | 50              |                                  |  |                                                               |  |                            |  |            |  |
| R2148-6 bottom         | 0.1881        | 3.0                                                                                                 | 1               | 50              |                                  |  |                                                               |  |                            |  |            |  |
| R2148-7 bottom         | 0.0531        | 3.0                                                                                                 | 1               | 100             | 0.110                            |  | 0.0031                                                        |  |                            |  |            |  |
| MIII Q blk Top         | 6.361         | 3.0                                                                                                 | 1               | 5               | 6.544                            |  | 4.5091                                                        |  |                            |  |            |  |
| R2148-1 top            | 6.899         | 3.0                                                                                                 | 1               | 5               | 6.090                            |  | 4.1951                                                        |  |                            |  |            |  |
| R2148-2 top            | 3.093         | 3.0                                                                                                 | 1               | 10              | 3.236                            |  | 1.1113                                                        |  |                            |  |            |  |
| R2148-3 top            | 2.116         | 3.0                                                                                                 | 1               | 10              | 2.228                            |  | 0.7632                                                        |  |                            |  |            |  |
| R2148-4 top            | 2.466         | 3.0                                                                                                 | 1               | 50              | 2.696                            |  | 0.1849                                                        |  |                            |  |            |  |
| R2148-5 top            | 1.836         | 3.0                                                                                                 | 1               | 50              | 2.024                            |  | 0.1386                                                        |  |                            |  |            |  |
| R2148-6 top            | 2.04          | 1.0                                                                                                 | 2               | 1               |                                  |  | 4.072                                                         |  |                            |  | 94%        |  |
| QC cal check 4.33 ppm  |               |                                                                                                     |                 |                 |                                  |  |                                                               |  |                            |  |            |  |

Orion Data

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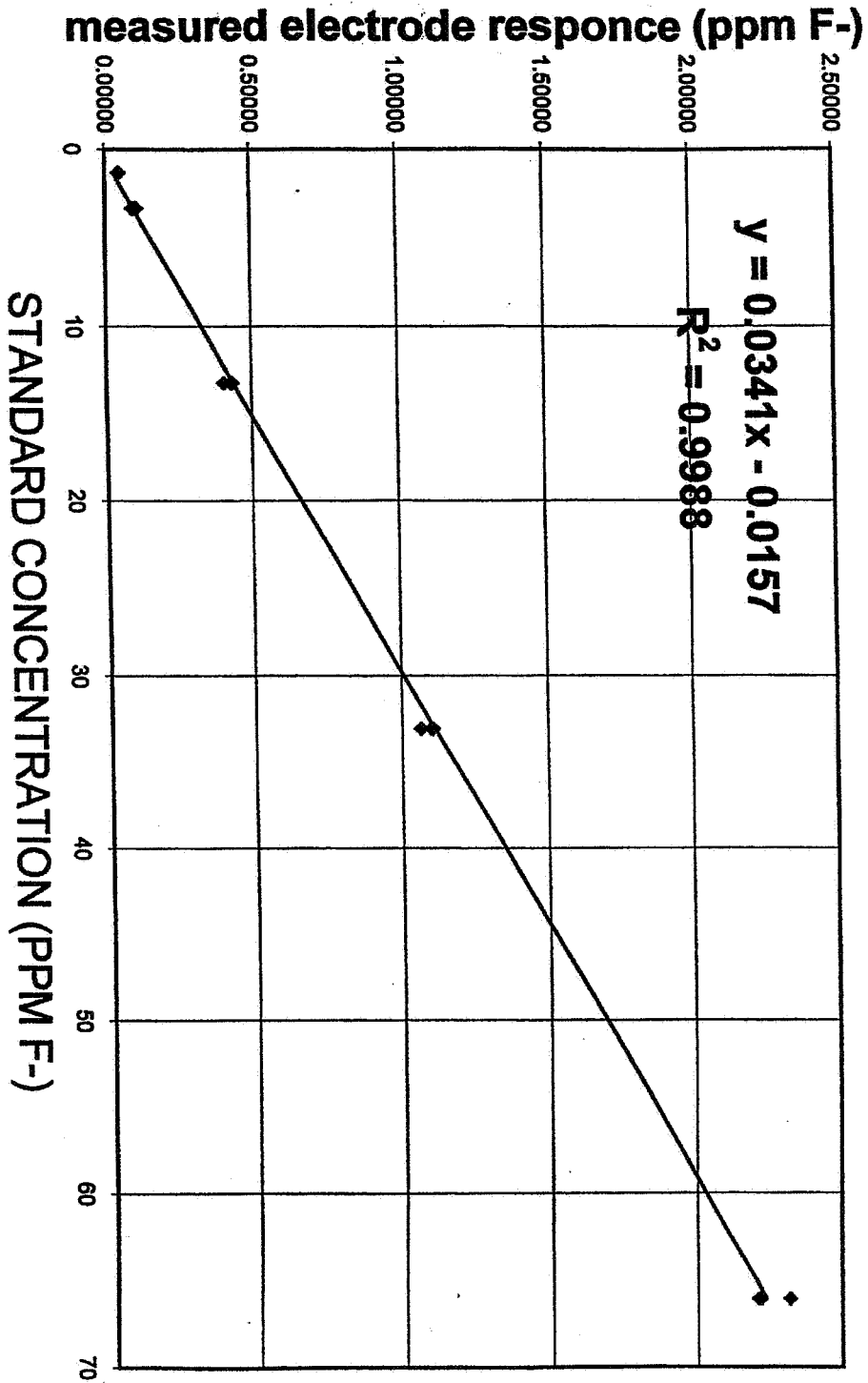
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| AOF ANALYSIS          |               | AOF Calc. (µg/mL) = ((total meter reading - intercept) / slope) / sample volume |                 |                 |                                  |  | Calc. Adsorbable Organic Fluorine (top&bottom) |  | Comments                   |  | Recovery % |  |
|-----------------------|---------------|---------------------------------------------------------------------------------|-----------------|-----------------|----------------------------------|--|------------------------------------------------|--|----------------------------|--|------------|--|
| Sample ID             | Meter Reading | Collect Vol (mL)                                                                | Dilution Factor | Sample Vol (mL) | Meter Reading Total (top&bottom) |  |                                                |  |                            |  |            |  |
| R2148-9 bottom        | 0.2705        | 3.0                                                                             | 1               | 50              |                                  |  |                                                |  |                            |  |            |  |
| R2148-10 bottom       | 0.2784        | 3.0                                                                             | 1               | 50              |                                  |  |                                                |  |                            |  |            |  |
| R2148-11 bottom       | 0.1188        | 3.0                                                                             | 1               | 50              |                                  |  |                                                |  |                            |  |            |  |
| R2148-12 bottom       | 0.0713        | 3.0                                                                             | 1               | 50              |                                  |  |                                                |  |                            |  |            |  |
| R2148-9 top           | 1.2856        | 3.0                                                                             | 1               | 50              | 1.5561                           |  | 0.1062                                         |  |                            |  |            |  |
| R2148-10 top          | 1.6588        | 3.0                                                                             | 1               | 50              | 1.9372                           |  | 0.1325                                         |  |                            |  |            |  |
| R2148-11 top          | 0.3520        | 3.0                                                                             | 1               | 50              | 0.4707                           |  | 0.0312                                         |  | read below low std, re-run |  |            |  |
| R2148-12 top          | 0.2683        | 3.0                                                                             | 1               | 50              | 0.3396                           |  | 0.0221                                         |  | read below low std, re-run |  |            |  |
| R2148-7 MS Bottom     | 0.1134        | 3.0                                                                             | 1               | 100             |                                  |  | 0.1079                                         |  |                            |  |            |  |
| R2148-7 MS Top        | 3.0297        | 3.0                                                                             | 1               | 100             | 3.143                            |  | 0.1079                                         |  |                            |  | 103%       |  |
| QC cal check 4.33 ppm | 2.228         | 1.0                                                                             | 2               | 1               |                                  |  | 4.456                                          |  |                            |  |            |  |
| R 2148-11 bottom      | 0.0760        | 3.0                                                                             | 1               | 100             |                                  |  |                                                |  |                            |  |            |  |
| R 2148-12 bottom      | 0.0839        | 3.0                                                                             | 1               | 100             |                                  |  |                                                |  |                            |  |            |  |
| R 2148-11 Top         | 0.4608        | 3.0                                                                             | 1               | 100             | 0.5368                           |  | 0.0176                                         |  |                            |  |            |  |
| R 2148-12 Top         | 0.4518        | 3.0                                                                             | 1               | 100             | 0.5357                           |  | 0.0175                                         |  |                            |  |            |  |
| QC check 0.060 ppm    | 0.4988        | 1.0                                                                             | 1               | 1               |                                  |  |                                                |  |                            |  | 98%        |  |

POAA CURVE

ASH010548

# Thermally Extracted Perfluorooctanoate Curve



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Perfluorooctanoate Standard Curve

| Sample ID             | FACTOR | SAMPLE | COLLECT | CALC. | POAA  | Actual  | % REC | COMMENTS              |
|-----------------------|--------|--------|---------|-------|-------|---------|-------|-----------------------|
|                       |        | (ML)   | (ML)    | (PPM) | (PPM) | (PPM)   |       |                       |
| ERA 4.33PPM STD       |        |        |         |       |       |         |       |                       |
| QC 1.0PPM STD CHECK   |        |        |         |       |       |         |       |                       |
| BLK-1                 | 1      | 0.1    | 3       | 1.47  |       | 0.0491  | 101.6 |                       |
| BLK-2                 | 1      | 0.1    | 3       | 1.02  |       | 0.0340  |       |                       |
| BLK-3                 | 1      | 0.1    | 3       | 0.48  |       | 0.0161  |       |                       |
| POAA 1.0 PPM W397-795 | 1      | 0.1    | 3       | 1.16  | 0.661 | 0.0386  | 176   | BELOW DETECTION LEVEL |
| POAA 1.0 PPM W397-795 | 1      | 0.1    | 3       | 0.97  | 0.661 | 0.0323  | 147   | USE 2.0PPM STD DATA   |
| POAA 1.0 PPM W397-795 | 1      | 0.1    | 3       | 1.40  | 0.661 | 0.0467  | 212   | NOT INCLUDED IN CURVE |
| ERA 4.33PPM STD       |        |        |         |       |       |         |       |                       |
| QC 1.0PPM STD CHECK   |        |        |         |       |       |         |       |                       |
| BLK-1                 | 1      | 0.1    | 3       |       |       | 0.01599 |       |                       |
| BLK-2                 | 1      | 0.1    | 3       |       |       | 0.01455 |       |                       |
| BLK-3                 | 1      | 0.1    | 3       |       |       | 0.01491 |       |                       |
| POAA 2.0PPM W397-801  | 1      | 0.1    | 3       | 1.473 | 1.322 | 0.04911 | 111   | 111                   |
| POAA 2.0PPM W397-801  | 1      | 0.1    | 3       | 1.473 | 1.322 | 0.04911 | 111   | 111                   |
| POAA 2.0PPM W397-801  | 1      | 0.1    | 3       | 1.397 | 1.322 | 0.04655 | 106   | 106                   |
| POAA 5.0 PPM W397-796 | 1      | 0.1    | 3       | 2.82  | 3.31  | 0.0939  | 85.2  |                       |
| POAA 5.0 PPM W397-796 | 1      | 0.1    | 3       | 3.22  | 3.31  | 0.1074  | 97.5  |                       |
| POAA 5.0 PPM W397-796 | 1      | 0.1    | 3       | 2.99  | 3.31  | 0.0998  | 90.6  |                       |
| QC 1.0 PPM CHECK      |        |        |         |       |       |         |       |                       |
| POAA 20PPM W397-797   | 1      | 0.1    | 3       | 12.10 | 13.22 | 0.4033  | 91.5  |                       |
| POAA 20PPM W397-797   | 1      | 0.1    | 3       | 12.80 | 13.22 | 0.4268  | 96.9  |                       |
| POAA 20PPM W397-797   | 1      | 0.1    | 3       | 12.96 | 13.22 | 0.4321  | 98.1  |                       |
| POAA 50PPM W397-798   | 1      | 0.1    | 3       | 31.92 | 33.05 | 1.064   | 96.6  |                       |
| POAA 50PPM W397-798   | 1      | 0.1    | 3       | 33.12 | 33.05 | 1.104   | 100   |                       |
| POAA 50PPM W397-798   | 1      | 0.1    | 3       | 32.97 | 33.05 | 1.099   | 99.8  |                       |
| POAA 100PPM W397-799  | 1      | 0.1    | 3       | 66.54 | 66.10 | 2.218   | 101   |                       |
| POAA 100PPM W397-799  | 1      | 0.1    | 3       | 69.60 | 66.10 | 2.320   | 105   |                       |
| POAA 100PPM W397-799  | 1      | 0.1    | 3       | 66.27 | 66.10 | 2.209   | 100   |                       |
| QC 1.0 PPM CHECK      |        |        |         |       |       |         |       |                       |
| 0.9930 99.3           |        |        |         |       |       |         |       |                       |

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11/7/97 3:17 PM

ASH010550

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

CALCULATED (PPM F-) =

(meter reading + intercept) / slope)

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

|                     | SAMPLE   | COLLET   | Actual           | CALC.      | STATS    | % REC |
|---------------------|----------|----------|------------------|------------|----------|-------|
|                     | VOL (ML) | VOL (ML) | Reading (PPM F-) | F (PPM F-) | (PPM F-) |       |
| Date: 11/6/97       |          |          |                  |            |          |       |
| ERA 4.33PPM STD     |          |          |                  |            |          |       |
| QC 1.0PPM STD CHECK |          |          | 2.162            |            |          | 98.86 |
|                     |          |          | 0.9864           |            |          | 98.64 |
| BLK-1               | 0.1      | 3        | 0.01599          |            |          |       |
| BLK-2               | 0.1      | 3        | 0.01455          |            |          |       |
| BLK-3               | 0.1      | 3        | 0.01491          |            |          |       |
| QC 1.0PPM CHECK     |          |          | 0.9743           |            |          | 97.43 |
| R2148-11-1          | 0.1      | 3        | 0.05555          | 2.09       | AVE 2.33 |       |
| R2148-11-2          | 0.1      | 3        | 0.06031          | 2.23       | STD 0.30 |       |
| R2148-11-3          | 0.1      | 3        | 0.07499          | 2.66       | CV 12.8  |       |
| R2148-12-1          | 0.1      | 3        | 0.1699           | 5.44       | AVE 4.23 |       |
| R2148-12-2          | 0.1      | 3        | 0.1233           | 4.08       | STD 1.14 |       |
| R2148-12-3          | 0.1      | 3        | 0.09247          | 3.17       | CV 27.0  |       |
| R2148-10-1          | 0.1      | 3        | 0.08379          | 2.92       | AVE 2.81 |       |
| R2148-10-2          | 0.1      | 3        | 0.06284          | 2.30       | STD 0.47 |       |
| R2148-10-3          | 0.1      | 3        | 0.09400          | 3.22       | CV 16.6  |       |
| QC 1.0 PPM CHECK    |          |          | 0.9663           |            |          | 96.63 |
| R2148-9-1           | 0.1      | 3        | 0.1042           | 3.52       | AVE 2.96 |       |
| R2148-9-2           | 0.1      | 3        | 0.07910          | 2.78       | STD 0.49 |       |
| R2148-9-3           | 0.1      | 3        | 0.07286          | 2.60       | CV 16.4  |       |
| R2148-8-1           | 0.1      | 3        | 0.1361           | 4.45       | AVE 4.15 |       |
| R2148-8-2           | 0.1      | 3        | 0.1279           | 4.21       | STD 0.34 |       |
| R2148-8-3           | 0.1      | 3        | 0.1131           | 3.78       | CV 8.24  |       |
| QC 1.0PPM STD CHECK |          |          | 0.9624           |            |          | 96.24 |

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11/7/97 3:17 PM

TABLE continued : R2148 F- DETERMINATION: SAMPLES DUPONT INC.

| Sample ID            | VOL (ML) | COLLET (ML) | Actual (PPM F) | Calc. (PPM F) | STATS     | % REC |
|----------------------|----------|-------------|----------------|---------------|-----------|-------|
| ERA 4.33PPM STD      |          |             |                |               |           |       |
| QC 1.0 PPM STD CHECK |          |             | 0.9781         |               |           | 97.81 |
| BLK-1                | 0.1      | 3           | 0.05542        | 2.09          | AVE 1.71  |       |
| BLK-2                | 0.1      | 3           | 0.04169        | 1.68          | STD 0.36  |       |
| BLK-3                | 0.1      | 3           | 0.03074        | 1.36          | CV 21.2   |       |
| R2148-7-1            | 0.1      | 3           | 0.1088         | 3.65          | AVE 3.98  |       |
| R2148-7-2            | 0.1      | 3           | 0.1075         | 3.61          | STD 0.60  |       |
| R2148-7-3            | 0.1      | 3           | 0.1435         | 4.67          | CV 15.1   |       |
| R2148-4-1            | 0.1      | 3           | 0.1323         | 4.34          | AVE 3.54  |       |
| R2148-4-2            | 0.1      | 3           | 0.1028         | 3.48          | STD 0.77  |       |
| R2148-4-3            | 0.1      | 3           | 0.08024        | 2.81          | CV 21.6   |       |
| QC 1.0PPM STD CHECK  |          |             | 0.9507         |               |           | 95.07 |
| QC 1.0PPM STD CHECK  |          |             | 0.9941         |               |           | 99.41 |
| R2148-3-1            | 0.1      | 3           | 0.1003         | 3.40          | AVE 3.29  |       |
| R2148-3-2            | 0.1      | 3           | 0.09065        | 3.12          | STD 0.15  |       |
| R2148-3-3            | 0.1      | 3           | 0.09833        | 3.34          | CV 4.5    |       |
| R2148-6-1            | 0.1      | 3           | 0.1024         | 3.46          | AVE 3.29  |       |
| R2148-6-2            | 0.1      | 3           | 0.09567        | 3.26          | STD 0.17  |       |
| R2148-6-3            | 0.1      | 3           | 0.09102        | 3.13          | CV 5.11   |       |
| R2148-5-1            | 0.1      | 3           | 0.1225         | 4.05          | AVE 4.21  |       |
| R2148-5-2            | 0.1      | 3           | 0.1538         | 4.97          | STD 0.69  |       |
| R2148-5-3            | 0.1      | 3           | 0.1075         | 3.61          | CV 16.4   |       |
| QC 1.0PPM STD CHECK  |          |             | 0.9623         | 28.68         |           | 96.23 |
| R2148-1-1            | 0.1      | 3           | 0.1809         | 5.77          | AVE 7.99  |       |
| R2148-1-2            | 0.1      | 3           | 0.2924         | 9.04          | STD 1.9   |       |
| R2148-1-3            | 0.1      | 3           | 0.2972         | 9.18          | CV 24.1   |       |
| R2148-2-1            | 0.1      | 3           | 0.4648         | 14.09         | AVE 15.76 |       |
| R2148-2-2            | 0.1      | 3           | 0.4517         | 13.71         | STD 3.2   |       |
| R2148-2-3            | 0.1      | 3           | 0.6487         | 19.48         | CV 20.5   |       |
| QC 1.0PPM STD CHECK  |          |             | 0.9623         |               |           | 96.23 |

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ASH010551

3M Environmental Laboratory

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

ASH010552

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EID129651



**FINAL REPORT COMPLETED: October 31, 1997**

ASH010553

**Susan A. Beach**  
Senior Environmental Biologist  
3M Environmental Laboratory  
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## 1.0 Introduction

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

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

## 2.0 Results

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

|          | DuPont Sample No. | Sulfate, mg/kg | Sulfite, mg/L | Nitrite Nitrogen, mg/kg | pH in CaCl2 | pH in water | CEC, meq/100 g | % Moisture, (as-rec'd) |
|----------|-------------------|----------------|---------------|-------------------------|-------------|-------------|----------------|------------------------|
| R2382-1  | SS-1 0-2'         | 98             | <2            | 0.41                    | 7.2         | 7.7         | 15.8           | 12.3                   |
| R2382-2  | SS-1 4-6'         | 99             | <2            | 0.41                    | 7.3         | 7.7         | 18.4           | 12.7                   |
| R2382-3  | SS-1 8-10'        | 73             | <2            | 0.36                    | 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                   |

ASH010554

### 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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## 3M Ecotoxicology and Environmental Fate Laboratory

### 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

### 3M Ecotoxicology and Environmental Fate Laboratory

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  $\text{NH}_4\text{OAc}$  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

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

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

#### 6.4 Nutrient Analyses

Aliquots of the 0.500 mm-sieved soil were submitted to Minnesota Valley Testing Laboratories, Inc. (MVTTL) 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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**3M Ecotoxicology and Environmental Fate Laboratory**

**Copies of Raw Data and Contract Laboratory Reports**

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ENVIRONMENTAL LABORATORY

WORKSHEET

LR R2382  
PAGE 1 OF 1  
DATE 8/5/97  
ANALYST 273

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 thru the center was removed for analysis. The rest of the intact cores were refrigerated @ 20°C in the dark until needed. The core sample was mixed then split for inorganic and organic analysis.

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EID129658

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

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

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12382

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DATE

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## ANALYSIS FORM

|    |        | A   | B | C | D | E | F | G | H | I | J | K | L | M |
|----|--------|-----|---|---|---|---|---|---|---|---|---|---|---|---|
| 1  | 123821 | 180 |   |   |   |   |   |   |   |   |   |   |   |   |
| 2  |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 3  |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 4  | 2      | 210 |   |   |   |   |   |   |   |   |   |   |   |   |
| 5  |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 6  | 3      | 200 |   |   |   |   |   |   |   |   |   |   |   |   |
| 7  |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 8  | 4      | 200 |   |   |   |   |   |   |   |   |   |   |   |   |
| 9  |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 10 | 5      | 210 |   |   |   |   |   |   |   |   |   |   |   |   |
| 11 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 12 | 6      | 220 |   |   |   |   |   |   |   |   |   |   |   |   |
| 13 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 14 | 7      | 200 |   |   |   |   |   |   |   |   |   |   |   |   |
| 15 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 16 | 8      | 130 |   |   |   |   |   |   |   |   |   |   |   |   |
| 17 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 18 | 9      | 150 |   |   |   |   |   |   |   |   |   |   |   |   |
| 19 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 20 | 10     | 110 |   |   |   |   |   |   |   |   |   |   |   |   |
| 21 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 22 | 11     | 12  |   |   |   |   |   |   |   |   |   |   |   |   |
| 23 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 24 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 25 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 26 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 27 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 28 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 29 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 30 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 31 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 32 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 33 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 34 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 35 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 36 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 37 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 38 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 39 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 40 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 41 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 42 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 43 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 44 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 45 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 46 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |
| 47 |        |     |   |   |   |   |   |   |   |   |   |   |   |   |

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

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

ANALYST: [Signature]  
DATE: 8/27/94

ASH010563





# LABORATORIES, Inc.

P.O. BOX 249, 1126 N. FRONT STREET  
NEW ULM, MN 56073-0249  
PHONE (507) 354-8517 WATS (800) 782-3557 FAX (507) 359-2890

**WE ARE AN EQUAL OPPORTUNITY EMPLOYER**



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

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

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 Koebele*  
Anthony R. Koebele  
By and for Minnesota Valley Testing Laboratories, Inc.

ASH010565

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

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EID129664



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

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

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

*a Kuehl*

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## Analysis of 3M Samples

Page 1 of 1

| <u>Analyte</u>   | <u>Detection Level</u>   | <u>Method Reference</u>                     |
|------------------|--------------------------|---------------------------------------------|
| 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]

ASH010567

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EID129666

DATE: 08/21/1997

3M ENVIRONMENTAL LABORATORY  
CONTRACT LABORATORY WORK ORDER BY PARAMETER

LAB REQUEST NO. R2382

CONTRACT LAB : MVTL

PROJECT LEAD: RD HOWELL

PROJECT NUMBER : B10ENENVIR

TELEPHONE :

SHIP DATE :

8/24/97

FAX : 612-778-6176

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

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EID129667



**Copies of Soil Methods**

ASH010569

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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.

#### SOIL REACTION TESTS

pH  
Lime Requirement  
Gypsum Requirement

#### SOLUBLE SALTS ANALYSIS

Electrical Conductivity  
Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>  
Alkalinity

#### PHYSICAL CHARACTERISTICS

Soil texture and classification

#### SOLUBLE SALTS ANALYSIS

Electrical Conductivity

### 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 RRR)

ASH010570

97 # 125

### 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).

ASH010571

## 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).

ASH010572

## 3M ENVIRONMENTAL LABORATORY

### SOIL pH METHOD

#### PRINCIPLE OF THE SOIL pH METHOD

Soil pH is one of the most indicative measurements of the chemical properties of a soil. Whether a soil is acidic, neutral, or basic has much to do with the solubility of various compounds, the relative bonding of ions to exchange sites, and the activity of various microorganisms. Three soil pH ranges are particularly informative: a pH <4 indicates the presence of free acids generally from oxidation of sulfides; a pH <5.5 suggests the likely occurrence of exchangeable Al; and a pH from 7.8 to 8.2 indicates the presence of  $\text{CaCO}_3$ .

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

#### RANGE AND SENSITIVITY

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

#### INTERFERENCES

Factors that may influence the measured pH include:

- The nature and type of inorganic and organic constituents that contribute to soil acidity. (Hydrogen ions may dissociate from the exchange sites or may be displaced by hydrolysis.)
- The soil/solution ratio (1:1 is the most commonly used).
- The salt or electrolyte content ( $\text{H}^+$  are displaced by the cations of salts contained in the soils, in addition, the salts also displace exchangeable Al, which upon hydrolysis increases the  $\text{H}^+$  in solution).
- The  $\text{CO}_2$  content ( $\text{CO}_2$  from the atmosphere or soil air) dissolves in water forming carbonic acid ( $\text{H}_2\text{CO}_3$ ) which can lower the pH. In the actual measurement of soil pH, the soil and water are shaken so they come to equilibrium with the  $\text{CO}_2$  in the air, there is no effect on the pH measurement. Only in soils of very low  $[\text{H}^+]$  where the pH is considerably above 7.0 and particularly in soils containing free  $\text{CaCO}_3$  does the  $\text{CO}_2$  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  $\text{CaCl}_2$  is recommended to minimize differences caused by some of the above factors. This dilute salt solution masks small differences in salt contents without displacing a large fraction of the  $\text{H}^+$  or  $\text{Al}^{3+}$ . In addition, errors due to the liquid junction potential are decreased.

#### PRECISION AND ACCURACY

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

ASH010573

## EQUIPMENT AND REAGENTS

- 1). pH meter equipped with a combination electrode (or soil pH electrode, Cole-Parmer Model #5992-60) and automatic temperature compensation (ATC) probe.
- 2). Standard buffers, pH 7.0 and pH 4.0.
- 3). 50 mL conical, polypropylene centrifuge tubes.
- 4). Automatic pipets.
- 5). Gyratory shaker.
- 6). Millipore Milli-Q™ water.
- 7). Calcium chloride ( $\text{CaCl}_2$ ) solution, 1 M or 0.01 M.

## $\text{pH}_w$ AND $\text{pH}_s$ PROCEDURES

- 1). Calibrate pH meter with commercially prepared buffer solutions of pH 7.0 and 4.0 according to the instrument instruction manual.
- 2). Weigh 10.000 g of 2.00 mm air-dried soil into a 50 mL conical centrifuge tube.
- 3). With automatic pipet, add 10 mL of Milli-Q™ water to each tube.
- 4). Mix thoroughly for 5 minutes, preferably on a gyratory shaker.  
(Option: mix for one hour.)
- 5). Let stand for 10 minutes.  
(Option: let stand for one hour.)
- 6). Insert the electrodes into the container. Note: the test mixture after settling will have an upper, relatively clear layer (supernatant layer) and a lower layer of opaque soil suspension. Immerse the electrode into this mixture until the pH sensitive bulb is covered by the opaque soil suspension while leaving the reference contact in the supernatant layer.
- 7). Allow time for the electrode to reach equilibrium (~1 to 3 min.) and record as soil pH in water,  $\text{pH}_w$ .
- 8). To determine the soil pH in 0.01 M  $\text{CaCl}_2$ , add 0.10 mL of 1M  $\text{CaCl}_2$  solution to the soil water suspension.
- 9). Mix intermittently for 30 minutes.  
(Option: mix 30 minutes, let stand 30 minutes.)
- 10). Insert electrodes, and record as soil pH in 0.01 M  $\text{CaCl}_2$ ,  $\text{pH}_s$ . Alternatively, the soil pH in 0.01M  $\text{CaCl}_2$  may be determined directly by substituting 0.01 M  $\text{CaCl}_2$  for the water in step 2.
- 11). If the lime requirement is to be determined on the samples ( $\text{pH} < 6.9$ ), save them for this purpose after reading the  $\text{pH}_w$  or  $\text{pH}_s$ .

## REFERENCES

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

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

ASH010574

# 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.

ASH010575

## 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  $25^\circ\text{C}$ ) 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.

ASH010576



## CALCULATION

CEC EQUATION:

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

\* % 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).

ASH010577

**3M Environmental Laboratory**

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ASH010578

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EID129677

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1m4m

|         |                                   |  |         |          |            |
|---------|-----------------------------------|--|---------|----------|------------|
| Ship to | 4m 3M Environmental Technology &  |  | Date    | 05/08/97 | No. 38-972 |
|         | 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               |  | B/L     |          |            |

| 4m Quantity | Product Code / Description | Weight |
|-------------|----------------------------|--------|
| 4m 1        | Container of Water Samples | 48 lbs |
| 4m          |                            |        |
| 4m          |                            |        |
| 4m          |                            |        |
| 4m          |                            |        |
| 4m          |                            |        |
| 4m          |                            |        |
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| 4m          |                            |        |
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|Hazardous Material? N

4mValue for Customs

| MATERIAL CLASSIFICATION

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|Corrosive Toxic

4m

|Flammable Other

4m

0m

4m|

\*\* AUTHORIZATION \*\*

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ASH010579

106 & 125

EID129678

107 of 1.

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NFV 11-107 FORM 7-00

1mSHIPPING MEMORANDUMOm  
(FOR NO-CHARGE SHIPMENTS ONLY)

1m4m

Om

Ship to 4m 3M Environmental Technology &  
4m Service Laboratory, B-2-3E-09  
4m 935 Bush Avenue  
4m St Paul, Minesota 55144-1000

|Date 06/02/97  
|Routing  
|F.O.B.  
|Date  
|Shipped  
|B/L

No. 38-9732010m

Om  
Om  
Om  
Om  
Om

4m Attn: Robert Howell

4m Quantity | Product Code / Description

Weight

Om

4m 1 | Container/Soil sample

30 lbs

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Special Inst 4mShip Overnight 6/2--Must be delivered Tues. AM

SHIP OVERNIGHT

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4mCharge (Cost Code) 8309019511001280

|MSDS Sheets Required?

Om

4mPurchase Order No.

|Hazardous Material? N

Om

4mValue for Customs

| MATERIAL CLASSIFICATION

Om

Ship From 4mStores, Bldg. 4

|Corrosive

Toxic

Om

4m

|Flammable

Other

Om

4m

Om

4m

Om

4m

Om

4m

Om

4m

Om

\*\* AUTHORIZATION \*\*

ASH010581

108 of 125

EID129680

TITLE OF PROJ OR STUDY DUPONT WASHINGTON WORKS PROJ OR STUDY NO \_\_\_\_\_  
SUBJECT PARKERSBURG, WV WORKS \_\_\_\_\_

COMPUTER \_\_\_\_\_ DATE \_\_\_\_\_  
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" CHAIN OF CUSTODY "

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

FOR USE BY ROBERT NEWELL, 3M

Dan Weber

304-863-4415

ASH010582

109 of 125

USE STANDARDS FOR MINIMUM ESSENTIAL INSTALLATIONS

EID129681

# 3M Environmental Laboratory Request Form

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

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

Name **DLB / RDT** Location **DuPont Soil and Groundwater** Requester **Phone No.** **Date Received** **6/6/97** Project Lead **505** Expected Completion Date **12-31-97**

Project Description (40 Character Max) **DuPont Soil and Groundwater** In this a TSCA 610 project (see back side for instructions) **Yes** ☒ No ☐ Dept. No. (Mandatory) **60** Sub. Acct. **See comments** Class/Job/Project No. **60** Planning Status **60** Quality Review **60**

MUST BE CHECKED ☐ Yes ☒ No ☐ TSCA ☐ CWA ☐ CMA ☐ SDWA ☐ MSDS ☒ Customer Service

Purpose of Testing ☐ RCRA ☐ TSCA ☐ CWA ☐ CMA ☐ SDWA ☐ MSDS ☒ Customer Service

Safety Concerns/Special Handling

Comments

DuPont Washington Works samples for FC-143 analysis.  
 Split charge 50:50 between 3058 & 3048 BLOEUVENIR

ASH010583

| Sample No. (Range 0-999) | Sample Code (12 Character Max) | Sample Date & Time (MM/DD/YY, HH:MM) | Sample Description (12 Character Max) | 3M I.D. (11 Character Stock No.) | Computer Code | Class    | Mail Date | Disp. Date |
|--------------------------|--------------------------------|--------------------------------------|---------------------------------------|----------------------------------|---------------|----------|-----------|------------|
| 1                        | DUPONT WASH                    | 5/8/97 1:48pm                        | 5/8/97 1:48                           | 5/8/97 1:50                      | 5/8/97 1:50   | DI Blank |           |            |
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Form 10620 - P - PWO

White - Environmental Lab Archives

Pink - Contractor

Canary - Disbursements

Copies To:

EID129682

1mSHIPPING MEMORANDUM0m  
(FOR NO-CHARGE SHIPMENTS ONLY)

|                      |                                                          |                                              |                               |            |
|----------------------|----------------------------------------------------------|----------------------------------------------|-------------------------------|------------|
| 1m4m                 |                                                          |                                              |                               | 0m         |
| Ship to              | 4m 3M Environmental Technology &                         |                                              | Date 06/26/97 No. 38-9737950m |            |
|                      | 4m Service Laboratory, B-2-3E-09                         |                                              | Routing                       | 0m         |
|                      | 4m 935 Bush Avenue                                       |                                              | F.O.B.                        | 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 7                 |                                                          | Coolers containing water samples/water & ice |                               | 40 each 0m |
| 4m                   |                                                          |                                              |                               | 0m         |
| 4m                   |                                                          |                                              |                               | 0m         |
| 4m                   |                                                          |                                              |                               | 0m         |
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| 4m                   |                                                          |                                              |                               | 0m         |
| 4m                   |                                                          |                                              |                               | 0m         |
| Special Inst         | 4mNeed 7 labels, Overnight Air, Deliver 6/27 before noon |                                              |                               | 0m         |
| Freight Class        | 4m                                                       |                                              |                               | 0m         |
|                      | 4m                                                       |                                              |                               | 0m         |
|                      | 4m                                                       |                                              |                               | 0m         |
| 4mOriginator         | VERA L WIGAL                                             | Phone                                        | 304-863-4895                  | 0m         |
| 4mCharge (Cost Code) | 8309066742001280                                         |                                              | MSDS Sheets Required?         | 0m         |
| 4mPurchase Order No. |                                                          |                                              | Hazardous Material? N         | 0m         |
| 4mValue for Customs  |                                                          |                                              | MATERIAL CLASSIFICATION       | 0m         |
| Ship From            | 4mStores, Bldg. 4                                        |                                              | Corrosive Toxic               | 0m         |
|                      | 4m                                                       |                                              | Flammable Other               | 0m         |
|                      | 4m                                                       |                                              |                               | 0m         |
|                      | 4m                                                       |                                              |                               | 0m         |
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|                      | 4m                                                       |                                              |                               | 0m         |
|                      | 4m                                                       |                                              |                               | 0m         |

\*\* AUTHORIZATION \*\*

ASH010584

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EID129684

113 of 12:

EID129685

**DISTRIBUTION:** Original - LAB, Yellow - LAB, Yellow - LAB, Pink - Chem



☐ LKW Canpro Analytical Laboratories, Inc.  
 50 Balthazar, Unit 12, Waterloo, Ontario, Canada N2V 2G5  
 (519) 747-2575 FAX (519) 747-3908  
☐ CVO 2300 NW Walnut Boulevard  
 Corvallis, Oregon 97330-5538  
 (541) 752-4271 FAX (541) 752-0278

114 of 12

[illegible]

**Instructions and Agreement Provisions on Reverse Side**

**DISTRIBUTION:** Original - LAB, Yellow - LAB, Pink - Chem

115 of 12

**DISTRIBUTION:** Original - LAB, Yellow - LAB, Pink - Client



☐ LMG 2567 Fairlane Drive  
 Montgomery, AL 36118-1692  
 (334) 271-1444 FAX (334) 271-3428  
☐ LRD 5080 Caterpillar Road  
 Redding, CA 96003-1412  
 (916) 244-5227 FAX (916) 244-4109

□ LKW Carveo Analytical Laboratories, Inc.,  
 50 Bathurst, Unit 12, Waterloo, Ontario, Canada N2V 2C5  
 (519) 747-2575 FAX (519) 747-3808  
 □ CVO 2300 NW Walnut Boulevard  
 Corvallis, Oregon 97330-3538  
 (541) 752-4271 FAX (541) 752-0276

CCC

116 of 12.

|                                                               |  |                                    |  |                                                                                                                                        |  |                                                    |  |
|---------------------------------------------------------------|--|------------------------------------|--|----------------------------------------------------------------------------------------------------------------------------------------|--|----------------------------------------------------|--|
| Project #                                                     |  | Purchase Order #                   |  | Requested Analytical Method #                                                                                                          |  | THIS AREA FOR LAB USE ONLY                         |  |
| Project Name<br><b>D. FORT WASHINGTON WORKS</b>               |  |                                    |  | Lab #                                                                                                                                  |  | Page of                                            |  |
| Company Name<br><b>D. FORT</b>                                |  |                                    |  | Lab PM                                                                                                                                 |  | Custody Review                                     |  |
| Project Manager or Contact & Phone #<br><b>(302) 492-6028</b> |  | Report Copy to:                    |  | PH                                                                                                                                     |  | Custody Seal Y N                                   |  |
| Requested Completion Date:                                    |  | Site ID                            |  | Sample Disposal:<br>Depose <input checked="" type="checkbox"/> Return <input type="checkbox"/>                                         |  | Ice Y N                                            |  |
| Sampling<br>Date<br><b>6/26/97</b>                            |  | Time<br><b>1335</b>                |  | Type<br>COM<br>GRA<br>T<br>P<br>W<br>A<br>T<br>E<br>R<br>S<br>I<br>T<br>I<br>A<br>L                                                    |  | CLIENT SAMPLE ID<br>(9 CHARACTERS)<br><b>MLW-6</b> |  |
| LAB<br>QC                                                     |  | TOTAL # OF CORRECTIONS<br><b>2</b> |  | Preservative<br><b>C-8</b><br><b>C-7</b><br><b>SURFACTANTS</b><br><b>NITRATE</b><br><b>METHANE</b><br><b>SULFATE</b><br><b>SULFIDE</b> |  | QC Level 1 2 3 Other                               |  |
| Cooler Temperature                                            |  | Alternate Description              |  | Lab ID<br><b>ASH010589</b>                                                                                                             |  |                                                    |  |
| Sampled By & Title<br><b>Rowlesley</b>                        |  | Date/Time<br><b>6/26/97</b>        |  | Relinquished By<br><b>Rowlesley</b>                                                                                                    |  | Date/Time<br><b>6/26/97</b>                        |  |
| Received By<br><b>[Signature]</b>                             |  | Date/Time<br><b>6/26/97</b>        |  | Shipped Via<br><b>UPS</b>                                                                                                              |  | Shipping #                                         |  |
| Special Instructions:                                         |  |                                    |  |                                                                                                                                        |  |                                                    |  |

**Instructions and Agreement Provisions on Reverse Side**

**DISTRIBUTION:** Original - LAB, Yellow - LAB, Pink - Client

# CH2MHILL Analytical Services

☐ LMG 2597 Fairlane Drive  
 Montgomery, AL 36116-1622  
 (334) 271-1444 FAX (334) 271-3428  
☐ LRD 5090 Campbell Road  
 Redding, CA 96003-1412  
 (916) 244-5227 FAX (916) 244-4108

☐ LKW Camtro Analytical Laboratories, Inc.  
 50 Balford, Unit 12, Waterloo, Ontario, Canada N2V 2C5  
 (616) 747-2575 FAX (616) 747-3808  
☐ CVO 2300 NW Walnut Boulevard  
 Corvallis, Oregon 97330-3538  
 (541) 752-4271 FAX (541) 752-0276

COC #

117 of 12

|                                                                    |  |                                         |  |                                                                                                 |  |                             |  |
|--------------------------------------------------------------------|--|-----------------------------------------|--|-------------------------------------------------------------------------------------------------|--|-----------------------------|--|
| Project #                                                          |  | Purchase Order #                        |  | Requested Analytical Method #                                                                   |  | THIS AREA FOR LAB USE ONLY  |  |
| Project Name<br><b>DUPONT WASHINGTON ROCKS</b>                     |  | Company Name<br><b>DUPONT</b>           |  | Project Manager or Contact & Phone #<br><b>(302) 992-6820</b>                                   |  | Report Copy to:             |  |
| Requested Completion Date:                                         |  | Site ID                                 |  | Sample Disposal:<br><input checked="" type="checkbox"/> Dispose <input type="checkbox"/> Return |  | Lab #                       |  |
| Sampling Type<br>C G A W S I A<br>O R A T O I R<br>M A T E R I A L |  | Matrix                                  |  | CLIENT SAMPLE ID<br>(8 CHARACTERS)                                                              |  | Lab PM                      |  |
| Date<br><b>6/24/97</b>                                             |  | Time<br><b>1150</b>                     |  | XX MW-5                                                                                         |  | Custody Review              |  |
| LAB CC                                                             |  | TOTAL # OF CONTAINERS                   |  | C-B                                                                                             |  | Custody Seal Y N            |  |
|                                                                    |  |                                         |  | C-7                                                                                             |  | Ics Y N                     |  |
|                                                                    |  |                                         |  | SURFACTANTS                                                                                     |  |                             |  |
|                                                                    |  |                                         |  | METHANE                                                                                         |  |                             |  |
|                                                                    |  |                                         |  | NITRATE                                                                                         |  |                             |  |
|                                                                    |  |                                         |  | SULFIDE                                                                                         |  |                             |  |
|                                                                    |  |                                         |  | SULFATE                                                                                         |  |                             |  |
|                                                                    |  |                                         |  | Preservative                                                                                    |  |                             |  |
|                                                                    |  |                                         |  | QC Level 1 2 3 Other                                                                            |  |                             |  |
|                                                                    |  |                                         |  | Cooler Temperature                                                                              |  |                             |  |
|                                                                    |  |                                         |  | Alternate Description                                                                           |  | Lab ID                      |  |
|                                                                    |  |                                         |  | ASH010590                                                                                       |  |                             |  |
| Sampled By & Title<br><b>RON WESLEY</b>                            |  | Phone (day and night times)             |  | Date/Time<br><b>6/24/97</b>                                                                     |  | Date/Time<br><b>6/24/97</b> |  |
| Received By<br><b>Mike Brown</b>                                   |  | Phone (day and night times)             |  | Date/Time<br><b>6/24/97</b>                                                                     |  | Date/Time<br><b>6/24/97</b> |  |
| Special Instructions:                                              |  | Relinquished By<br><b>Paul B. (202)</b> |  | Phone (day and night times)                                                                     |  | Date/Time<br><b>6/24/97</b> |  |
| Shipped Via<br>UPS                                                 |  | FedEx                                   |  | Other                                                                                           |  | Shipping #                  |  |

Instructions and Agreement Provisions on Reverse Side

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

# 3M Environmental Laboratory Request Form

Address

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

Lab Request No.

R2148

Date Received

Page 1 of 3

Environmental Lab

Project Lead

JDU

Planning Status

JDU

Quality Review

9/11/97

Expected Completion Date

9/11/97

Class/Priority

9/11/97

Date Shipped

9/11/97

Project Description (40 Character Max)

Purest Water

Is this a TSCA 816 project? (See back side for instructions)

YES

MUST BE CHECKED

YES

Dep't. No. (Main)

3048

Sub. Acct.

3048

Class/Job/Project No.

816 ENVIR

Plant Code

816 ENVIR

Purpose of Testing

RCRA

TSCA

CWA

CAA

SDWA

MSDS

MSDS

Comments

Two samples of each.

Sample No. (Range 0-999)

1

Sample Code (12 Character Max)

MW-1-1

Sample Date & Time (MM/DD/YY, HH:MM)

6/26/97

Sample Description (12 Character Max)

MW-1 1 of 2

3M I.D. (11 Character Stock No.)

71297 81297

Computer Code

71297 81297

Class

71297 81297

Avail. Date

7/12/97

Due Date

8/29/97

Sample No. (Range 0-999)

2

Sample Code (12 Character Max)

MW-1-2

Sample Date & Time (MM/DD/YY, HH:MM)

6/26/97

Sample Description (12 Character Max)

MW-1 2 of 2

3M I.D. (11 Character Stock No.)

71297 81297

Computer Code

71297 81297

Class

71297 81297

Avail. Date

7/12/97

Due Date

8/29/97

Sample No. (Range 0-999)

3

Sample Code (12 Character Max)

MW-2-1

Sample Date & Time (MM/DD/YY, HH:MM)

6/26/97

Sample Description (12 Character Max)

MW-2 1 of 2

3M I.D. (11 Character Stock No.)

71297 81297

Computer Code

71297 81297

Class

71297 81297

Avail. Date

7/12/97

Due Date

8/29/97

Sample No. (Range 0-999)

4

Sample Code (12 Character Max)

MW-2-2

Sample Date & Time (MM/DD/YY, HH:MM)

6/26/97

Sample Description (12 Character Max)

MW-2 2 of 2

3M I.D. (11 Character Stock No.)

71297 81297

Computer Code

71297 81297

Class

71297 81297

Form 10620 - P - PWD

White - Environmental Lab Archives

Pink - Contractor

Canary - Disturbments

Copies To:

EID129690

|                       |             |
|-----------------------|-------------|
| Lab Request No. R2148 |             |
| Date Received         | Page 2 of 3 |

ASH010592

## Comments

EID129691



## Form 10820 - 14-C-PW0

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

R2148

|      |        |
|------|--------|
| Page | 3 of 3 |
|------|--------|

ASH010593

ASH010593

### Safety Concerns/Special Handling

## Comments

ASH010593

[illegible]

### While - Environmental Lab Archives

**Pink - Contractor**

### Canary - Requisition

1mSHIPPING MEMORANDUM0m  
(FOR NO-CHARGE SHIPMENTS ONLY)

1m4m

0m

|         |                                   |         |          |                |    |
|---------|-----------------------------------|---------|----------|----------------|----|
| Ship to | 4m 3M Environmental Technology &  | Date    | 06/24/97 | No. 38-9737408 | 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 |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |
| 4m          |                                   |         | 0m |

Special Inst 4mOvernight-EAGLE USA AIR, Delivie Before Noon 0m

Freight Class 4m 0m

4m 0m

4m 0m

4mOriginator VERA L WIGAL Phone 304-863-4895 0m

4mCharge (Cost Code) 8309066742001280 |MSDS Sheets Required? 0m

4mPurchase Order No. |Hazardous Material? N 0m

4mValue for Customs | MATERIAL CLASSIFICATION 0m

Ship From 4mStores, Bldg. 4 |Corrosive Toxic 0m

4m |Flammable Other 0m

4m | 0m

4m | 0m

4m | 0m

4m | 0m

\*\* AUTHORIZATION \*\*

ASH010594

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EID129693



☐ LMG 2567 Fairlane Drive  
 Montgomery, AL 36116-1822  
 (334) 271-1444 FAX (334) 271-3428  
☐ LRD 5090 Caterpillar Road  
 Redding, CA 96003-1412  
 (916) 244-5227 FAX (916) 244-4108

• LKW Carrypro Analytical Laboratories, Inc.  
 50 Balfour, Unit 12, Waterloo, Ontario, Canada N2V 2C5  
 (519) 747-2575 FAX (519) 747-3908  
 • CVO 2300 NW Walnut Boulevard  
 Corvallis, Oregon 97330-3538  
 (541) 752-4271 FAX (514) 752-0276

**COC #**

[illegible]

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☐ LTKW Caystro Analytical Laboratories, Inc.,  
 50 Ballhurst, Unit 12, Mississauga, Ontario, Canada N2V 2C5  
 (519) 747-2575 FAX (519) 747-3808  
☐ CVO 2300 NW Weiland Boulevard  
 Corvallis, Oregon 97330-3538  
 (541) 752-4271 FAX (541) 752-0276

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EID129695

# 3M Environmental Laboratory Request Form



Lab Request No. **R2382**

Date Received **8/5/94**

Page **1 of 2**

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

**Name** DLBach **Location** Port Washington Works **Requester** CS Study **Phone No.** 3058 **Date Received** 8/5/94 **Environmental Lab** RDH/KS **Expected Completion Date** 8/29/97

**Project Description (10 Characters Max.)** DLBach **Is this a TSCA Site project (see back side for instructions)?** Yes **Dept. No. (Main)** 3058 **Sub. Acct.** Bigenewiro **Classified/Project No.** 10 **Planning Status** 10 **Class/Proj.** 10 **Date Shipped** 8/29/97

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

ASH010597

| Sample No. (Range 0-999) | Sample Code (12 Character Max) | Sample Date & Time (MM/DD/YY, HH:MM) | Sample Description (12 Character Max) | 3M I.D. (11 Character Stock No.) |
|--------------------------|--------------------------------|--------------------------------------|---------------------------------------|----------------------------------|
| 1                        | MW-1 Site Cont                 | 4/23/97 1550                         | 4-6'                                  | 4/23/97 1610                     |
| 2                        |                                |                                      |                                       | 4/23/97 1620                     |
| 3                        |                                |                                      |                                       | 4/23/97 1630                     |
| 4                        |                                |                                      |                                       | 4/23/97 1640                     |
| 5                        |                                |                                      |                                       | 4/23/97 1650                     |
| 6                        |                                |                                      |                                       | 4/23/97 1700                     |
| 7                        |                                |                                      |                                       | 4/23/97 1710                     |
| 8                        |                                |                                      |                                       | 4/23/97 1720                     |
| 9                        |                                |                                      |                                       | 4/23/97 1730                     |
| 10                       |                                |                                      |                                       | 4/23/97 1740                     |
| 11                       |                                |                                      |                                       | 4/23/97 1750                     |
| 12                       |                                |                                      |                                       | 4/23/97 1800                     |
| 13                       |                                |                                      |                                       | 4/23/97 1810                     |
| 14                       |                                |                                      |                                       | 4/23/97 1820                     |
| 15                       |                                |                                      |                                       | 4/23/97 1830                     |
| 16                       |                                |                                      |                                       | 4/23/97 1840                     |
| 17                       |                                |                                      |                                       | 4/23/97 1850                     |
| 18                       |                                |                                      |                                       | 4/23/97 1900                     |
| 19                       |                                |                                      |                                       | 4/23/97 1910                     |
| 20                       |                                |                                      |                                       | 4/23/97 1920                     |
| 21                       |                                |                                      |                                       | 4/23/97 1930                     |
| 22                       |                                |                                      |                                       | 4/23/97 1940                     |
| 23                       |                                |                                      |                                       | 4/23/97 1950                     |
| 24                       |                                |                                      |                                       | 4/23/97 2000                     |
| 25                       |                                |                                      |                                       | 4/23/97 2010                     |
| 26                       |                                |                                      |                                       | 4/23/97 2020                     |
| 27                       |                                |                                      |                                       | 4/23/97 2030                     |
| 28                       |                                |                                      |                                       | 4/23/97 2040                     |
| 29                       |                                |                                      |                                       | 4/23/97 2050                     |
| 30                       |                                |                                      |                                       | 4/23/97 2100                     |
| 31                       |                                |                                      |                                       | 4/23/97 2110                     |
| 32                       |                                |                                      |                                       | 4/23/97 2120                     |
| 33                       |                                |                                      |                                       | 4/23/97 2130                     |
| 34                       |                                |                                      |                                       | 4/23/97 2140                     |
| 35                       |                                |                                      |                                       | 4/23/97 2150                     |
| 36                       |                                |                                      |                                       | 4/23/97 2200                     |
| 37                       |                                |                                      |                                       | 4/23/97 2210                     |
| 38                       |                                |                                      |                                       | 4/23/97 2220                     |
| 39                       |                                |                                      |                                       | 4/23/97 2230                     |
| 40                       |                                |                                      |                                       | 4/23/97 2240                     |
| 41                       |                                |                                      |                                       | 4/23/97 2250                     |
| 42                       |                                |                                      |                                       | 4/23/97 2300                     |
| 43                       |                                |                                      |                                       | 4/23/97 2310                     |
| 44                       |                                |                                      |                                       | 4/23/97 2320                     |
| 45                       |                                |                                      |                                       | 4/23/97 2330                     |
| 46                       |                                |                                      |                                       | 4/23/97 2340                     |
| 47                       |                                |                                      |                                       | 4/23/97 2350                     |
| 48                       |                                |                                      |                                       | 4/23/97 2400                     |
| 49                       |                                |                                      |                                       | 4/23/97 2410                     |
| 50                       |                                |                                      |                                       | 4/23/97 2420                     |
| 51                       |                                |                                      |                                       | 4/23/97 2430                     |
| 52                       |                                |                                      |                                       | 4/23/97 2440                     |
| 53                       |                                |                                      |                                       | 4/23/97 2450                     |
| 54                       |                                |                                      |                                       | 4/23/97 2460                     |
| 55                       |                                |                                      |                                       | 4/23/97 2470                     |
| 56                       |                                |                                      |                                       | 4/23/97 2480                     |
| 57                       |                                |                                      |                                       | 4/23/97 2490                     |
| 58                       |                                |                                      |                                       | 4/23/97 2500                     |
| 59                       |                                |                                      |                                       | 4/23/97 2510                     |
| 60                       |                                |                                      |                                       | 4/23/97 2520                     |
| 61                       |                                |                                      |                                       | 4/23/97 2530                     |
| 62                       |                                |                                      |                                       | 4/23/97 2540                     |
| 63                       |                                |                                      |                                       | 4/23/97 2550                     |
| 64                       |                                |                                      |                                       | 4/23/97 2560                     |
| 65                       |                                |                                      |                                       | 4/23/97 2570                     |
| 66                       |                                |                                      |                                       | 4/23/97 2580                     |
| 67                       |                                |                                      |                                       | 4/23/97 2590                     |
| 68                       |                                |                                      |                                       | 4/23/97 2600                     |
| 69                       |                                |                                      |                                       | 4/23/97 2610                     |
| 70                       |                                |                                      |                                       | 4/23/97 2620                     |
| 71                       |                                |                                      |                                       | 4/23/97 2630                     |
| 72                       |                                |                                      |                                       | 4/23/97 2640                     |
| 73                       |                                |                                      |                                       | 4/23/97 2650                     |
| 74                       |                                |                                      |                                       | 4/23/97 2660                     |
| 75                       |                                |                                      |                                       | 4/23/97 2670                     |
| 76                       |                                |                                      |                                       | 4/23/97 2680                     |
| 77                       |                                |                                      |                                       | 4/23/97 2690                     |
| 78                       |                                |                                      |                                       | 4/23/97 2700                     |
| 79                       |                                |                                      |                                       | 4/23/97 2710                     |
| 80                       |                                |                                      |                                       | 4/23/97 2720                     |
| 81                       |                                |                                      |                                       | 4/23/97 2730                     |
| 82                       |                                |                                      |                                       | 4/23/97 2740                     |
| 83                       |                                |                                      |                                       | 4/23/97 2750                     |
| 84                       |                                |                                      |                                       | 4/23/97 2760                     |
| 85                       |                                |                                      |                                       | 4/23/97 2770                     |
| 86                       |                                |                                      |                                       | 4/23/97 2780                     |
| 87                       |                                |                                      |                                       | 4/23/97 2790                     |
| 88                       |                                |                                      |                                       | 4/23/97 2800                     |
| 89                       |                                |                                      |                                       | 4/23/97 2810                     |
| 90                       |                                |                                      |                                       | 4/23/97 2820                     |
| 91                       |                                |                                      |                                       | 4/23/97 2830                     |
| 92                       |                                |                                      |                                       | 4/23/97 2840                     |
| 93                       |                                |                                      |                                       | 4/23/97 2850                     |
| 94                       |                                |                                      |                                       | 4/23/97 2860                     |
| 95                       |                                |                                      |                                       | 4/23/97 2870                     |
| 96                       |                                |                                      |                                       | 4/23/97 2880                     |
| 97                       |                                |                                      |                                       | 4/23/97 2890                     |
| 98                       |                                |                                      |                                       | 4/23/97 2900                     |
| 99                       |                                |                                      |                                       | 4/23/97 2910                     |
| 100                      |                                |                                      |                                       | 4/23/97 2920                     |

# **3M Environmental Laboratory** **Request Form - Continued**

Form 10820 - 14-C-PWO

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

Safety Concerns/Special Handling

Comments

ASH010598

|                 |               |
|-----------------|---------------|
| Lab Request No. | <b>R2282</b>  |
| Date Received   | <b>2 of 2</b> |

| Sample No.                         | (Range 0-999) →      | 6           | 7       | 8       | 9       | 10      | 11      |
|------------------------------------|----------------------|-------------|---------|---------|---------|---------|---------|
| Sample Code                        | (12 Character Max) → | MW-15HES-11 |         |         |         |         |         |
| Sample Date                        | (MM/DD/YY) →         | 4/23/97     | 4/23/97 | 4/23/97 | 4/23/97 | 4/23/97 | 4/23/97 |
| Sample Description                 | (12 Character Max) → | 10-22'      | 24-26'  | 29-30'  | 32-34'  | 36-38'  | 38-40'  |
| 3M I.D. (11 Character Stock No.) → |                      |             |         |         |         |         |         |
| Test Required                      |                      |             |         |         |         |         |         |
| Computer Code                      |                      |             |         |         |         |         |         |
| Glab                               |                      |             |         |         |         |         |         |
| AV Date                            |                      |             |         |         |         |         |         |
| ID Date                            |                      |             |         |         |         |         |         |

3M 203  
 3M 203  
 3M 203

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