

Katherine E. Reed, Ph.D.  
Staff Vice President

3M Environmental, Health and  
Safety Operations

900 Bush Avenue, Building 42-2E-26  
PO Box 33331  
St. Paul, MN 55133-3331  
651 778 4331

AR226 - 3547

RECEIVED

05 SEP -2 PM 10:38

8EHQ-0905-00394

MRH

~~2484895~~

2484895

NO TSCA CBI

8EHQ-81-394

Contain NO CBI

**Certified Mail**

August 26, 2005

Document Processing Center (Mail Code 7407M)

Room 6428

Attention: TSCA 8(e) Coordinator  
Office of Pollution Prevention and Toxics  
U.S. Environmental Protection Agency  
1201 Constitution Avenue, N. W.  
Washington, D. C. 20460

Re: TSCA Section 8(e) Supplemental Notice: Sulfonate-based and Carboxylic-based  
Fluorochemicals, Docket Nos. 8EHQ-1180-374; 8EHQ-0381-0394; 8EHQ-0598-373

Dear Sir:

3M is submitting this notice to supplement its previous submissions on sulfonyl and carboxylic-based fluorochemicals.

3M has recently received the enclosed analytical report which presents data from 36 lots of commercially available normal pooled human serum that were purchased by the 3M Environmental Laboratory and subsequently screened for endogenous fluorochemicals. Each sample was analyzed using a protein precipitation technique and screened for sixteen different fluorochemicals, including perfluorooctane sulfonate (PFOS) ( $C_8F_{17}SO_3^-$ ) and perfluorooctanoic acid (PFOA) ( $C_7F_{15}COOH$ ), using liquid chromatography/tandem mass spectrometry (LC/MS/MS). Much of the data presented in the report was derived from extrapolated curves and should be considered as screening quality data only and not necessarily representative of the general population levels. Not all of the detected chemicals are or were produced by 3M. In addition, the levels of fluorochemicals reported are generally corroborative of previously reported values and/or do not represent "substantial risk" information.

If you have any questions, please do not hesitate to contact Dale L. Bacon at (651) 778-4736.

Sincerely,

*Katherine E. Reed* TRF

Katherine D. Reed, PhD  
Staff Vice President  
Environmental Technology and Safety Services

2005 SEP 14 PM 12:40

RECEIVED  
OPPT NCIC

BEST COPY AVAILABLE



8 9 0 5 0 0 0 0 5 9 8



NO TSCA CBI

## **Analytical Report**

Revised August 12, 2005

Initial Quantitative Screening of Commercial Lots of Human Serum  
Obtained in 2004 for Endogenous Fluorochemicals Using Protein  
Precipitation and Liquid Chromatography/Tandem Mass Spectrometry

Laboratory Request Number: E05-0120

---

### **Testing Laboratory**

3M Environmental Health and Safety Operations  
3M Environmental Laboratory

---

### **3M Environmental Laboratory**

#### **Project Lead**

Mark E. Ellefson

Building 2-3E-09

PO Box 3331

Saint Paul, MN 55133-3331

Phone: 651-778-5405

#### **Testing Facility Management**

William K. Reagen, Ph.D.

Building 2-3E-09

PO Box 3331

Saint Paul, MN 55133-3331

Phone: 651-778-6565

---

### **Requestor**

Dale Bacon

Building 2-3E-09

PO Box 3331

Saint Paul, MN 55133-3331

Phone: 651-778-4736

3M Environmental Laboratory

## *Analytical Report*

*Project Number: E05-0120*

The following analytical report presents data from commercially available normal pooled human serum that were purchased by the 3M Environmental Laboratory and subsequently screened for endogenous fluorochemicals.

Thirty-six separate lots of pooled normal human serum were extracted using a protein precipitation technique and screened for sixteen different fluorochemicals using liquid chromatography/tandem mass spectrometry (LC/MS/MS). Serum samples were received from various suppliers between 9/9/04 and 12/2/04. Samples were analyzed between 02/03/05 and 02/09/05.

This range-finding study was designed to provide lower limits of quantitation (LLOQs) of approximately 100 - 300 ppt. Upon review of the data, it was discovered that many of the target analytes were present at concentrations much lower than the 100 - 300 ppt target level. As an interim solution and until a more definitive study can be completed, signal to noise ratios were evaluated and a limit of detection (LOD) was defined for each analyte. As a result, calibration curves were extrapolated between the LLOQ and the newly defined LOD, allowing screening quality data to be determined for some analytes as low as 19.9 ppt. Therefore, it must be emphasized that much of the data presented in this report was derived from extrapolated curves and should be considered as screening quality data only.

Sample results are summarized in Table 1. The average values reported represent samples in which there was detectable analyte. Sample values that were less than the limit of detection were not included in the average value calculations. Please refer to Section 5 for individual sample results.

Table 1. Summary of Perfluorinated Acid Results

| Analyte                                   | PFOS (ng/mL) | PFOA (ng/mL) | PFNA (ng/mL)    | PFDA (ng/mL) | PFUnA (ng/mL) | PFOS (ng/mL) | PFOA (ng/mL)    | PFNA (ng/mL) | PFDA (ng/mL)    |
|-------------------------------------------|--------------|--------------|-----------------|--------------|---------------|--------------|-----------------|--------------|-----------------|
| Mean                                      | 3.33         | 2.84         | NR <sup>2</sup> | 0.954        | 4.61          | 1.25         | NR <sup>2</sup> | 0.623        | <LOD            |
| Median                                    | 3.33         | 2.5          | NR <sup>2</sup> | 0.954        | 4.59          | 1.09         | NR <sup>2</sup> | 0.660        | <LOD            |
| High                                      | 7.08         | 3.56         | NR <sup>2</sup> | 1.02         | 23.0          | 2.63         | NR <sup>2</sup> | 0.842        | <LOD            |
| Low                                       | 0.507        | 2.45         | NR <sup>2</sup> | 0.888        | 0.477         | 0.337        | NR <sup>2</sup> | 0.312        | <LOD            |
| Standard Deviation                        | 2.17         | 0.627        | NR <sup>2</sup> | 0.0933       | 3.64          | 0.571        | NR <sup>2</sup> | 0.208        | ND <sup>1</sup> |
| Number of Samples with Detectable Analyte | 20/36        | 3/36         | 0/36            | 2/36         | 35/36         | 34/36        | 0/36            | 6/36         | 0/36            |

ND<sup>1</sup> = Not Detected. This analyte was not detected in any of the samples.

NR<sup>2</sup> = Not Reported. This analyte was detected in one of the 36 samples but appears to be a contaminant or an anomaly; the sample will be reanalyzed and the results will be reported in the definitive study.

Table 2. Summary of Other Perfluorinated and Highly Fluorinated Analyte Results

| Analyte                                     | PFOS (ng/mL)    | PFOA (ng/mL) | PFNA (ng/mL) | PFDA (ng/mL) | PFUnA (ng/mL) | PFOS (ng/mL)    | PFOA (ng/mL) |
|---------------------------------------------|-----------------|--------------|--------------|--------------|---------------|-----------------|--------------|
| Mean                                        | <LOD            | 0.0776       | 0.0348       | 3.27         | 16.3          | <LOD            | 0.0839       |
| Median                                      | <LOD            | 0.0401       | 0.0339       | 3.27         | 16.9          | <LOD            | 0.0819       |
| High                                        | <LOD            | 0.199        | 0.0706       | 5.80         | 32.8          | <LOD            | 0.155        |
| Low                                         | <LOD            | 0.0311       | 0.0210       | 0.780        | 2.71          | <LOD            | 0.0480       |
| Standard Deviation                          | ND <sup>1</sup> | 0.0812       | 0.0122       | 1.52         | 6.71          | ND <sup>1</sup> | 0.0257       |
| Fraction of Samples with Detectable Analyte | 0/36            | 4/36         | 23/36        | 36/36        | 36/36         | 0/36            | 36/36        |

ND<sup>1</sup> = Not Detected. This analyte was not detected in any of the samples.

Peaks were observed in the chromatograms corresponding to linear and branched isomers for PFOS and PFOA. Relative branched and linear isomer ratios were determined for both analytes and are presented below (the human serum value represents an average of % branched isomers for all of the human serum samples that contained detectable levels of each analyte). The % branched isomer results for individual samples are presented in Table 12. In addition, well-characterized lots of PFOS (Lot 171) and PFOA (Lot 332) were analyzed together as a matrix spike (QC050203-004) in rabbit serum. Data from that analysis was evaluated for branched and linear isomer distribution and agrees well with previously obtained NMR data.

Table 3. Summary of PFOA and PFOS Isomer Ratios

| Sample                                                        | PFOA Isomer | PFOS Isomer |
|---------------------------------------------------------------|-------------|-------------|
| Human Serum, average                                          | 6%          | 39%         |
| Matrix Spike (QC050203-004)                                   | 23%         | 32%         |
| TCR-123 (99030-030) PFOA<br>Lot 332 (NMR characterized value) | 22%         |             |
| TCR-696 PFOS<br>Lot 171 (NMR characterized value)             |             | 31%         |

## 2. Reference Substances

Table 4. Reference Substances

| Chemical Name                                   | Chemical Name        | Supplier         | Lot Number          | Purity  | Additional Information (K salt or H) |
|-------------------------------------------------|----------------------|------------------|---------------------|---------|--------------------------------------|
| Heptafluorobutyric Acid                         | PFBA (C4 Acid)       | Aldrich          | TCR-757             | 99.2%   | 0.995                                |
| Nonafluoropentanoic acid                        | NFPA (C5 Acid)       | Aldrich          | TCR-333 (00017-045) | 96.5%   | 0.996                                |
| Perfluorohexanoic acid                          | PFHA (C6 Acid)       | Oakwood Products | TCR-673             | 97.7%   | 0.997                                |
| Tridecafluoroheptanoic acid                     | TDHA (C7 Acid)       | Aldrich          | TCR-267             | 98.2%   | 0.997                                |
| Perfluorooctanoic acid                          | PFOA (C8 Acid)       | 3M               | TCR-123 (99030-030) | 95.0%   | 0.958                                |
| Heptadecafluorononanoic acid                    | C9 Acid              | Oakwood Products | TCR-618             | 98.0%   | 0.998                                |
| Nonadecafluorodecanoic acid                     | C10 Acid             | Oakwood Products | TCR-36              | 98.01%  | 0.998                                |
| Perfluoroundecanoic acid                        | C11 Acid             | Oakwood Products | TCR-619             | 96.4%   | 0.998                                |
| Perfluorododecanoic acid                        | C12 Acid             | Oakwood Products | TCR-37 (SD037)      | 99.65%  | 0.998                                |
| Perfluorobutanesulfonamide                      | FBSA                 | 3M               | TCR-314 (00017-026) | unknown | 0.997                                |
| Perfluorooctanesulfonamide                      | FOSA                 | 3M               | TCR-280 (99131-038) | 98.94%  | 0.997                                |
| Perfluorobutane sulfonate                       | PFBS                 | 3M               | TCR-282 (99131-040) | 97.3%   | 0.885                                |
| Perfluorohexane sulfonate                       | PFHS                 | 3M               | TCR-83 (SE036)      | 98.6%   | 0.911                                |
| Perfluorooctane sulfonate                       | PFOS                 | 3M               | TCR-696             | 86.4%   | 0.927                                |
| 1H, 1H, 2H, 2H,<br>perfluorooctanesulfonic acid | THPFOS               | SynQuest Labs    | TCR-343 (00017-055) | 93.5%   | 0.998                                |
| 1H, 1H, 2H, 2H,<br>perfluorodecane sulfonate    | THPFDS               | Pace/3M          | TCR-627             | 94.7%   | 0.998                                |
| Perfluorooctanoic acid,<br>isotopically labeled | <sup>13</sup> C PFOA | Perkin Elmer     | TCR-744             | >97%    | 0.998                                |

## Sample Preparation

The samples for this study were prepared as follows:

1. The serum samples were allowed to thaw and warm to room temperature.
2. One milliliter of serum (or water in the case of method blanks) was added to a 15 mL screw-capped polypropylene centrifuge tube.
3. Matrix spikes and method blanks were prepared by spiking the serum (or water) with the analytes of interest. In addition,  $^{13}\text{C}$  PFOA was added as a surrogate to all serum samples, calibration standards, matrix blanks, and method blanks at a concentration of 3.01 ng/mL to evaluate extraction method performance.
4. Two milliliters of acetonitrile were added to the serum and the centrifuge tubes were capped and vortex mixed, allowing the serum proteins to precipitate.
5. The tubes were centrifuged at approximately 3000 rcf for 20 minutes to pellet the precipitate.
6. After removal from the centrifuge, the supernatant was transferred to an autovial for analysis.

Quantification was accomplished by high performance liquid chromatography tandem mass spectrometry. The following parameters were used:

**Liquid Chromatograph:** Hewlett-Packard® Series 1100 Liquid Chromatograph system

Analytical column: Keystone® Betasil™ C<sub>18</sub> 2x100mm, 5µm particle size

Column temperature: 30 °C

Stop Time: 20.0 minutes

Flow rate: 300 µL/min

Injection volume: 5 µL

Mobile phase components:

Solvent A: 2.0 mM ammonium acetate in ASTM Type I water

Solvent B: HPLC Grade Methanol

Solvent Gradient:

| <u>Time</u> | <u>%B</u> |
|-------------|-----------|
| 0.00        | 20%       |
| 1.00        | 20%       |
| 14.50       | 90%       |
| 15.50       | 90%       |
| 16.50       | 20%       |
| 20.00       | 20%       |

Mass Spectrometer: MDX Sciex® API 4000 Q-Trap Mass Spectrometer Detector  
Software: Analyst™ Version 1.4  
Ionization Mode: Electrospray Negative  
Temperature: 450 °C  
GS1: 35  
GS2: 45  
Entrance Potential: 10  
Analysis Type: Multiple Reaction Monitoring (MRM)

Table 5. Acquisition Parameters

| Compound                    | Q1 Mass (amu) | Q3 Mass (amu)       | Collision Potential (V) |
|-----------------------------|---------------|---------------------|-------------------------|
| PFBA (C <sub>4</sub> acid)  | 213.0         | 168.9               | -30                     |
| NFPAC <sub>5</sub> Acid     | 262.9         | 219.0               | -30                     |
| PFHA (C <sub>6</sub> acid)  | 313.0         | 268.7, 118.9        | -45                     |
| TDHA (C <sub>7</sub> Acid)  | 362.9         | 318.7, 168.8, 118.9 | -35                     |
| PFOA (C <sub>8</sub> Acid)  | 413.0         | 368.9, 219.0, 169.0 | -40                     |
| C <sub>9</sub> Acid         | 463.0         | 418.7, 268.9, 218.9 | -50                     |
| C <sub>10</sub> Acid        | 512.9         | 468.8, 218.9, 269.1 | -100                    |
| C <sub>11</sub> Acid        | 563.0         | 518.7, 268.9, 218.8 | -45                     |
| C <sub>12</sub> Acid        | 613.0         | 568.7, 168.9, 318.7 | -50                     |
| FBSA                        | 297.9         | 78.0                | -55                     |
| FOSA                        | 497.9         | 77.9                | -90                     |
| PFBS                        | 298.9         | 98.9, 79.9          | -65                     |
| PFHS                        | 398.9         | 98.9, 80.0          | -95                     |
| PFOS                        | 498.9         | 80.0, 99.2, 130.0   | -80                     |
| THPFOS                      | 426.9         | 406.8, 80.9         | -70                     |
| THPFDS                      | 526.9         | 506.7, 81.0         | -70                     |
| PFOA [1,2- <sup>13</sup> C] | 414.9         | 369.8               | -40                     |

When multiple transitions were monitored, the total ion current (TIC) was used to quantify each analyte, thereby providing greater sensitivity than individual Q3 ion measurements.

## 4. Analysis

### 4.1 Calibration

#### 4.1.1 Calibration Curve

An extracted calibration curve was formed by spiking rabbit serum at prescribed levels and extracting the curve in the same manner as the samples. Stock solutions were individually prepared from neat fluorochemicals and diluted in methanol (except PFBA, which was prepared in Milli-Q water to prevent esterification from occurring). Samples were quantified versus an 8-point extracted calibration curve. The regression analysis was performed using a quadratic curve fit weighted 1/x.

Calibration standards were within  $\pm 20\%$  ( $\pm 25\%$  for the Lower Limit of Quantitation (LLOQ)) with a coefficient of determination  $>0.995$ . Low-level calibration standards, which were not within  $\pm 25\%$ , were not included in the calibration curve. Calibration standards that were not greater than twice the method blanks were not included in the calibration curve.

#### 4.1.2 Continuing Calibration Verification (CCV)

A Continuing Calibration Verification (CCV) was run at least once every 10 samples, bracketing the samples with the calibration curve. The CCV recovery was within  $\pm 25\%$  for all analytes.

#### 4.1.3 System Suitability

Five system suitability standards were analyzed before the initial calibration curve and after the last CCV. These samples had area counts with a %RSD of  $\leq 5\%$  and a retention time %RSD of  $\leq 2\%$  when evaluated independently except for the following instances:

Several individual transitions monitored for the C<sub>10</sub> Acid failed the retention time system suitability criteria. However, the data reported was derived from the quantitation of the TIC, which passed system suitability requirements.

All of the system suitability sets for FBSA area counts failed. The %RSDs were 6.2%, 7.5% and 8.4%. However, no samples had area counts above the LLOQ for this analyte therefore the data was accepted.

#### 4.1.4 Lower Limit of Quantitation (LLOQ)

The LLOQ for each analyte was equal to the lowest standard in the calibration curve that met accuracy requirements of 75% - 125%.

#### 4.1.5 Limit of Detection (LOD)

The LOD for each analyte was equal to the area counts (converted to a concentration) that were 2-3 times above the method blank.

#### 4.1.6 Blanks

##### Method Blanks

All of the method blanks in this study had area counts  $< 50\%$  of the LLOQ.

##### Matrix Blanks

Two matrix blanks were prepared in rabbit serum and analyzed with the samples. One matrix blank contained endogenous levels of PFOS that were above the LLOQ. An average endogenous value for PFOS was determined by substituting a number equal to  $\frac{1}{2}$  the LLOQ for BLK050203-003.

Table 6. Average Endogenous PFOS Value Determination

| Sample ID     | PFOS   |
|---------------|--------|
| LLOQ (ng/mL)  | 0.306  |
| Blk050203-003 | < LLOQ |
| Blk050203-004 | 0.477  |
| Average:      | 0.315  |

The average endogenous PFOS value was used to correct the matrix spike response for endogenous levels of PFOS in rabbit serum.

#### 4.1.7 Solvent Blanks

Acetonitrile blanks were analyzed throughout the analytical run. Analyte response was less than ½ the LLOQ in all cases.

#### 4.1.8 Surrogate

<sup>13</sup>C PFOA was added as a surrogate to all serum samples, calibration standards, matrix blanks, and method blanks at a concentration of 3.01 ng/mL to evaluate the extraction method performance. All surrogate recoveries were within ±20%.

#### 4.2 Laboratory Control Spikes (LCS)

Six Laboratory Control Spikes (LCS) were prepared in rabbit serum and analyzed with the samples. Results were corrected for endogenous levels of analyte in the matrix (where appropriate), and are presented below.

Table 7. Matrix Spike Recoveries in Rabbit Serum

| Sample ID                                                | Conc. (ng/mL) | Conc. (ng/mL) | Recovery (%) | PFOS |
|----------------------------------------------------------|---------------|---------------|--------------|------|
| PFBA (C4 acid)<br>LOD = 0.426 ng/mL<br>LLOQ = 3.00 ng/mL | QC050203-001  | 3.00          | < LLOQ       | NA   |
|                                                          | QC050203-002  | 3.00          | < LLOQ       | NA   |
|                                                          | QC050203-003  | 3.00          | < LLOQ       | NA   |
|                                                          | QC050203-004  | 75.6          | 70           | 93%  |
|                                                          | QC050203-005  | 75.6          | 71.3         | 94%  |
|                                                          | QC050203-006  | 75.6          | 70.2         | 93%  |
| NFPA (C5 acid)<br>LOD = 4.53 ng/mL<br>LLOQ = 15.3 ng/mL  | QC050203-001  | 3.06          | < LLOQ       | NA   |
|                                                          | QC050203-002  | 3.06          | < LLOQ       | NA   |
|                                                          | QC050203-003  | 3.06          | < LLOQ       | NA   |
|                                                          | QC050203-004  | 77.7          | 68.00        | 88%  |
|                                                          | QC050203-005  | 77.7          | 80.50        | 104% |
|                                                          | QC050203-006  | 77.7          | 97.20        | 125% |

NA = Not Applicable

Table 7. Matrix Spike Recoveries in Rabbit Serum (continued)

|                                     |              |      |        |      |       |
|-------------------------------------|--------------|------|--------|------|-------|
| PFHA (C6 acid)<br>LLOQ = 15.1 ng/mL | QC050203-001 | 3.03 | < LLOQ | NA   | NA    |
|                                     | QC050203-002 | 3.03 | < LLOQ | NA   |       |
|                                     | QC050203-003 | 3.03 | < LLOQ | NA   |       |
|                                     | QC050203-004 | 76.8 | 62.4   | 81%  | 20.4% |
|                                     | QC050203-005 | 76.8 | 77.2   | 101% |       |
|                                     | QC050203-006 | 76.8 | 94.2   | 123% |       |
| TDHA (C7 acid)<br>LLOQ = 3.03 ng/mL | QC050203-001 | 3.03 | < LLOQ | NA   | NA    |
|                                     | QC050203-002 | 3.03 | < LLOQ | NA   |       |
|                                     | QC050203-003 | 3.03 | < LLOQ | NA   |       |
|                                     | QC050203-004 | 76.5 | 66.9   | 87%  | 17.7% |
|                                     | QC050203-005 | 76.5 | 80.1   | 105% |       |
|                                     | QC050203-006 | 76.5 | 95.4   | 125% |       |
| C10 ACID<br>LLOQ = 3.12 ng/mL       | QC050203-001 | 3.12 | < LLOQ | NA   | NA    |
|                                     | QC050203-002 | 3.12 | < LLOQ | NA   |       |
|                                     | QC050203-003 | 3.12 | < LLOQ | NA   |       |
|                                     | QC050203-004 | 78.6 | 65.4   | 83%  | 12%   |
|                                     | QC050203-005 | 78.6 | 71.9   | 91%  |       |
|                                     | QC050203-006 | 78.6 | 83.1   | 106% |       |
| C11 ACID<br>LLOQ = 0.297 ng/mL      | QC050203-001 | 2.97 | 2.98   | 100% | 5.1%  |
|                                     | QC050203-002 | 2.97 | 2.71   | 91%  |       |
|                                     | QC050203-003 | 2.97 | 2.95   | 99%  |       |
|                                     | QC050203-004 | 75.3 | 75.8   | 101% | 1.8%  |
|                                     | QC050203-005 | 75.3 | 78.3   | 104% |       |
|                                     | QC050203-006 | 75.3 | 78.2   | 104% |       |
| C12 ACID<br>LLOQ = 0.747 ng/mL      | QC050203-001 | 3.00 | 3.04   | 101% | 3.8%  |
|                                     | QC050203-002 | 3.00 | 3.13   | 104% |       |
|                                     | QC050203-003 | 3.00 | 3.28   | 109% |       |
|                                     | QC050203-004 | 75.6 | 76.2   | 101% | 3.9%  |
|                                     | QC050203-005 | 75.6 | 82.2   | 109% |       |
|                                     | QC050203-006 | 75.6 | 78.3   | 104% |       |

NA = Not Applicable

Table 7. Matrix Spike Recoveries in Rabbit Serum (continued)

|                               |              |      |      |      | RSD  |
|-------------------------------|--------------|------|------|------|------|
| <sup>13</sup> C PFOA          | QC050203-001 | 3.01 | 2.81 | 93%  | 4.8% |
|                               | QC050203-002 | 3.01 | 3.06 | 102% |      |
|                               | QC050203-003 | 3.01 | 2.94 | 98%  |      |
|                               | QC050203-004 | 3.01 | 2.68 | 89%  |      |
|                               | QC050203-005 | 3.01 | 2.91 | 97%  |      |
|                               | QC050203-006 | 3.01 | 2.75 | 91%  |      |
| C9 Acid<br>LLOQ = 0.303 ng/mL | QC050202-001 | 3.03 | 3.23 | 107% | 4.7% |
|                               | QC050202-002 | 3.03 | 3.04 | 100% |      |
|                               | QC050202-003 | 3.03 | 2.95 | 97%  |      |
|                               | QC050202-004 | 75.4 | 77.4 | 103% | 5.3% |
|                               | QC050202-005 | 75.4 | 75.8 | 101% |      |
|                               | QC050202-006 | 75.4 | 69.9 | 93%  |      |
| PFOA<br>LLOQ = 0.298 ng/mL    | QC050202-001 | 2.98 | 2.93 | 98%  | 6.8% |
|                               | QC050202-002 | 2.98 | 2.58 | 87%  |      |
|                               | QC050202-003 | 2.98 | 2.65 | 89%  |      |
|                               | QC050202-004 | 75.6 | 75.2 | 99%  | 1.6% |
|                               | QC050202-005 | 75.6 | 76.4 | 101% |      |
|                               | QC050202-006 | 75.6 | 77.6 | 103% |      |
| FBSA<br>LLOQ = 0.303 ng/mL    | QC050203-001 | 3.03 | 2.96 | 98%  | 0.7% |
|                               | QC050203-002 | 3.03 | 2.92 | 96%  |      |
|                               | QC050203-003 | 3.03 | 2.95 | 97%  |      |
|                               | QC050203-004 | 76.5 | 75.9 | 99%  | 1.4% |
|                               | QC050203-005 | 76.5 | 77.9 | 102% |      |
|                               | QC050203-006 | 76.5 | 77.7 | 102% |      |
| FOSA<br>LLOQ = 0.300 ng/mL    | QC050203-001 | 3.00 | 2.78 | 93%  | 0.8% |
|                               | QC050203-002 | 3.00 | 2.74 | 91%  |      |
|                               | QC050203-003 | 3.00 | 2.74 | 91%  |      |
|                               | QC050203-004 | 75.9 | 76.6 | 101% | 0.7% |
|                               | QC050203-005 | 75.9 | 77.1 | 102% |      |
|                               | QC050203-006 | 75.9 | 76.0 | 100% |      |

Table 7. Matrix Spike Recoveries in Rabbit Serum (continued)

| Non-Hazardous      |              |      |      |      |      |
|--------------------|--------------|------|------|------|------|
| PFBS               |              |      |      |      |      |
| LLOQ = 0.303 ng/mL | QC050203-001 | 3.03 | 3.06 | 101% | 1.1% |
|                    | QC050203-002 | 3.03 | 3.00 | 99%  |      |
|                    | QC050203-003 | 3.03 | 3.01 | 99%  |      |
|                    | QC050203-004 | 76.8 | 75.8 | 99%  | 1.6% |
|                    | QC050203-005 | 76.8 | 78.3 | 102% |      |
|                    | QC050203-006 | 76.8 | 77.2 | 101% |      |
|                    |              |      |      |      |      |
| PFHS               |              |      |      |      |      |
| LLOQ = 0.315 ng/mL | QC050203-001 | 3.15 | 3.04 | 97%  | 0.8% |
|                    | QC050203-002 | 3.15 | 3.08 | 98%  |      |
|                    | QC050203-003 | 3.15 | 3.04 | 97%  |      |
|                    | QC050203-004 | 79.8 | 78.2 | 98%  | 1.1% |
|                    | QC050203-005 | 79.8 | 79.9 | 100% |      |
|                    | QC050203-006 | 79.8 | 78.6 | 98%  |      |
|                    |              |      |      |      |      |
| THPFOS             |              |      |      |      |      |
| LLOQ = 0.780 ng/mL | QC050203-001 | 3.12 | 3.02 | 97%  | 3.8% |
|                    | QC050203-002 | 3.12 | 2.83 | 91%  |      |
|                    | QC050203-003 | 3.12 | 2.83 | 91%  |      |
|                    | QC050203-004 | 78.9 | 66.0 | 84%  | 19%  |
|                    | QC050203-005 | 78.9 | 80.0 | 101% |      |
|                    | QC050203-006 | 78.9 | 96.5 | 122% |      |
|                    |              |      |      |      |      |
| THPFDS             |              |      |      |      |      |
| LLOQ = 0.303 ng/mL | QC050203-001 | 3.03 | 2.96 | 98%  | 2.8% |
|                    | QC050203-002 | 3.03 | 3.00 | 99%  |      |
|                    | QC050203-003 | 3.03 | 3.12 | 103% |      |
|                    | QC050203-004 | 76.5 | 77.9 | 102% | 1.3% |
|                    | QC050203-005 | 76.5 | 79.8 | 104% |      |
|                    | QC050203-006 | 76.5 | 78.2 | 102% |      |

**Table 8. PFOS Matrix Spike Recovery in Rabbit Serum, Corrected for Endogenous Levels**

| Analyte                  | QC#          | Conc (ng/mL) | Found (ng/mL) | Recovery (%) | Conc (ng/mL) | Found (ng/mL) | Recovery (%) | Conc (ng/mL) | Found (ng/mL) | Recovery (%) |
|--------------------------|--------------|--------------|---------------|--------------|--------------|---------------|--------------|--------------|---------------|--------------|
| PFOS<br>LLOQ=0.303 ng/mL | QC050202-001 | 3.06         | 3.17          | 0.315        | 2.86         | 93%           | 2.5%         |              |               |              |
|                          | QC050202-002 | 3.06         | 3.03          | 0.315        | 2.72         | 89%           |              |              |               |              |
|                          | QC050202-003 | 3.06         | 3.1           | 0.315        | 2.79         | 91%           |              |              |               |              |
|                          | QC050202-004 | 77.4         | 77.4          | 0.315        | 77.1         | 100%          | 1.1%         |              |               |              |
|                          | QC050202-005 | 77.4         | 77.6          | 0.315        | 77.3         | 100%          |              |              |               |              |
|                          | QC050202-006 | 77.4         | 76.1          | 0.315        | 75.8         | 98%           |              |              |               |              |

### 4.3 Accuracy

The accuracy for each analyte reported was calculated by taking the average of the matrix spike recoveries for that analyte. The results are presented below.

**Table 9. Method Accuracy for Individual Analytes**

| Analyte   | % Accuracy | Analyte  | % Accuracy |
|-----------|------------|----------|------------|
| PFBA*     | ± 7%       | C12 Acid | ± 5%       |
| NFPA*     | ± 6%       | FBSA     | ± 1%       |
| PFHA*     | ± 2%       | FOSA     | ± 4%       |
| C7 Acid*  | ± 6%       | PFBS     | ± 1%       |
| PFOA      | ± 4%       | PFHS     | ± 2%       |
| C9 Acid   | ± 1%       | PFOS     | ± 5%       |
| C10 Acid* | ± 7%       | THPFOS   | ± 2%       |
| C11 Acid  | ± 1%       | THPFDS   | ± 1%       |

\*The % Accuracy reported for this analyte is based on the high concentration spike recovery data only because the low concentration spike recoveries were less than the LLOQ.

### 4.4 Sample Related Comments

The extraction and analysis of the samples was performed utilizing a non-validated method. The matrix spike results, <sup>13</sup>C PFOA surrogate recoveries, and the extrapolated calibration curve (between the LLOQ and LOD) should be considered when evaluating the accuracy and precision of the sample results.

## 5 Individual Sample Data

Tables 10 and 11 summarize individual sample data.

Table 10. Perfluorinated Acid Results

| Sample ID | PFCA   | PFBA | PFHxA | PFHpA  | PFDA  | PFUnA | PFNSA | PFTrA | PFOS |
|-----------|--------|------|-------|--------|-------|-------|-------|-------|------|
| TNA 6840  | 5.19   | 2.50 | <LOD  | <LOD   | 5.48  | 1.52  | <LOD  | <LOD  | <LOD |
| TNA 6843  | 0.990* | <LOD | <LOD  | <LOD   | 0.581 | 0.574 | <LOD  | <LOD  | <LOD |
| TNA 6847  | 0.507* | <LOD | <LOD  | <LOD   | 4.61  | 0.976 | <LOD  | <LOD  | <LOD |
| TNA 6934  | <LOD   | <LOD | <LOD  | <LOD   | 7.69  | 2.26  | <LOD  | <LOD  | <LOD |
| TNA 6935  | 1.17*  | <LOD | <LOD  | <LOD   | 5.19  | 1.63  | <LOD  | 0.451 | <LOD |
| TNA 6944  | 7.08   | <LOD | <LOD  | <LOD   | 7.38  | 1.45  | <LOD  | <LOD  | <LOD |
| TNA 6959  | 4.91   | <LOD | <LOD  | <LOD   | 3.13  | 2.45  | <LOD  | 0.700 | <LOD |
| TNA 6960  | 3.32   | <LOD | <LOD  | <LOD   | 4.59  | 1.11  | <LOD  | <LOD  | <LOD |
| TNA 6967  | 4.09   | <LOD | <LOD  | <LOD   | 5.30  | 1.62  | <LOD  | <LOD  | <LOD |
| TNA 6973  | 6.73   | 3.56 | <LOD  | 0.888* | 5.51  | 1.64  | <LOD  | <LOD  | <LOD |
| TNA 6975  | 2.95*  | <LOD | <LOD  | <LOD   | 5.52  | 1.31  | <LOD  | <LOD  | <LOD |

Table 10. Perfluorinated Acid Results (continued)

| Sample ID          | PFNA<br>(ng/mL) | PFNA<br>(ng/mL) | PFNA<br>(ng/mL) | PFNA<br>(ng/mL) | PFNA<br>(ng/mL) | PFNA<br>(ng/mL) | PFNA<br>(ng/mL) | PFNA<br>(ng/mL) | PFNA<br>(ng/mL) |
|--------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 1100<br>(ng/mL)    | 2.05            | 15.2            | 2.05            | 2.05            | 0.238           | 0.30            | 2.05            | 0.238           | 0.30            |
| 1501<br>(ng/mL)    | 1.25            | 2.37            | 1.25            | 1.25            | 0.298           | 0.303           | 1.25            | 0.298           | 0.303           |
| TNA 6977**         | 5.04            | <LOD            | <LOD            | <LOD            | 0.477           | <LOD            | <LOD            | <LOD            | <LOD            |
| TNA 6981           | 6.76            | 2.45            | <LOD            | <LOD            | 4.12            | 1.28            | <LOD            | <LOD            | <LOD            |
| TNA 6992           | 0.690*          | <LOD            | <LOD            | <LOD            | 4.76            | 0.912           | <LOD            | <LOD            | <LOD            |
| TNA 6994           | <LOD            | <LOD            | <LOD            | <LOD            | 1.15            | 0.337           | <LOD            | <LOD            | <LOD            |
| TNA 6995           | 1.98*           | <LOD            | 18.4***         | 1.02*           | 3.58            | 2.63            | <LOD            | 0.815           | <LOD            |
| TNA 6997           | 3.90            | <LOD            | <LOD            | <LOD            | 3.46            | 2.52            | <LOD            | 0.842           | <LOD            |
| TNA 7000           | 3.34            | <LOD            | <LOD            | <LOD            | 23.0            | 1.08            | <LOD            | <LOD            | <LOD            |
| TNA 7006           | 3.19            | <LOD            | <LOD            | <LOD            | 4.18            | 1.09            | <LOD            | <LOD            | <LOD            |
| TNA 6938           | <LOD            | <LOD            | <LOD            | <LOD            | 5.00            | 0.979           | <LOD            | <LOD            | <LOD            |
| TNA 6939           | <LOD            | <LOD            | <LOD            | <LOD            | 2.32            | 0.941           | <LOD            | 0.312           | <LOD            |
| TNA 6941           | 3.56            | <LOD            | <LOD            | <LOD            | 5.82            | 1.69            | <LOD            | 0.620           | <LOD            |
| TNA 6950           | <LOD            | <LOD            | <LOD            | <LOD            | 3.87            | 0.723           | 14.0***         | <LOD            | <LOD            |
| TNA 6953           | 0.621*          | <LOD            | <LOD            | <LOD            | 5.49            | 0.725           | <LOD            | <LOD            | <LOD            |
| TNA 6955           | <LOD            | <LOD            | <LOD            | <LOD            | 2.54            | 0.562           | <LOD            | <LOD            | <LOD            |
| TNA 6962           | <LOD            | <LOD            | <LOD            | <LOD            | 4.68            | 0.941           | <LOD            | <LOD            | <LOD            |
| TNA 6964           | <LOD            | <LOD            | <LOD            | <LOD            | 5.61            | 1.58            | <LOD            | <LOD            | <LOD            |
| TNA 6966           | <LOD            | <LOD            | <LOD            | <LOD            | 4.73            | 1.35            | <LOD            | <LOD            | <LOD            |
| TNA 6970           | <LOD            | <LOD            | <LOD            | <LOD            | 6.00            | 1.42            | <LOD            | <LOD            | <LOD            |
| TNA 6972           | <LOD            | <LOD            | <LOD            | <LOD            | 3.40            | 1.09            | <LOD            | <LOD            | <LOD            |
| TNA 6978**         | <LOD            | <LOD            | <LOD            | <LOD            | <LOD            | <LOD            | <LOD            | <LOD            | <LOD            |
| TNA 6979           | <LOD            | <LOD            | <LOD            | <LOD            | 3.32            | 0.981           | <LOD            | <LOD            | <LOD            |
| TNA 6982           | <LOD            | <LOD            | <LOD            | <LOD            | 1.48            | 0.664           | <LOD            | <LOD            | <LOD            |
| TNA 6984           | <LOD            | <LOD            | <LOD            | <LOD            | 2.84            | 0.955           | <LOD            | <LOD            | <LOD            |
| TNA 6986           | 0.530*          | <LOD            | <LOD            | <LOD            | 2.08            | 0.645           | <LOD            | <LOD            | <LOD            |
| TNA 6988           | <LOD            | <LOD            | <LOD            | <LOD            | 2.57            | 0.881           | <LOD            | <LOD            | <LOD            |
| Mean               | 3.33            | 2.84            | NA              | 0.954*          | 4.61            | 1.25            | NA              | 0.623           | NA              |
| Median             | 3.33            | 2.50            | NA              | 0.954*          | 4.59            | 1.09            | NA              | 0.660           | NA              |
| High               | 7.08            | 3.56            | NA              | 1.02*           | 23.0            | 2.63            | NA              | 0.842           | NA              |
| Low                | 0.507*          | 2.45            | NA              | 0.888*          | 0.477           | 0.337           | NA              | 0.312           | NA              |
| Standard Deviation | 2.17            | 0.627           | NA              | 0.0933          | 3.64            | 0.571           | NA              | 0.208           | NA              |
| # OF HITS          | 20              | 3               | 0               | 2               | 35              | 34              | 0               | 6               | 0               |

NA = Not Applicable

\*Analyte concentration was below the LLOQ and above the LOD.

\*\*TN-A-6977 and TN-A-6978 are from the same lot of pooled serum.

\*\*\*Analyte was detected in one of the 36 samples but appears to be a contaminant or an anomaly; the sample will be reanalyzed and the results will be reported in the definitive study.

Based on <sup>13</sup>C PFOA surrogate recovery, the overall method uncertainty is +/- 20%.

15

Table 11. Sample Results for Additional Perfluorinated and Highly Fluorinated Analytes

| Sample ID  | PFOS | PFOA    | PFNA    | PFDA  | PFUnA | PFHxA | PFNA    |
|------------|------|---------|---------|-------|-------|-------|---------|
| TNA 6840   | <LOD | <LOD    | <LOD    | 4.39  | 23.0  | <LOD  | 0.124*  |
| TNA 6843   | <LOD | <LOD    | 0.0228* | 0.780 | 3.42  | <LOD  | 0.155*  |
| TNA 6847   | <LOD | 0.0458* | <LOD    | 2.05  | 23.7  | <LOD  | 0.145*  |
| TNA 6934   | <LOD | <LOD    | 0.0304* | 2.41  | 32.8  | <LOD  | 0.0531* |
| TNA 6935   | <LOD | <LOD    | 0.0433* | 2.14  | 16.2  | <LOD  | 0.0820* |
| TNA 6944   | <LOD | <LOD    | 0.0706* | 4.73  | 20.4  | <LOD  | 0.0860* |
| TNA 6959   | <LOD | <LOD    | <LOD    | 3.75  | 14.8  | <LOD  | 0.0649* |
| TNA 6960   | <LOD | <LOD    | 0.0282* | 4.52  | 19.6  | <LOD  | 0.0819* |
| TNA 6967   | <LOD | 0.199*  | 0.0358* | 4.27  | 21.3  | <LOD  | 0.0857* |
| TNA 6973   | <LOD | <LOD    | <LOD    | 5.18  | 20.5  | <LOD  | 0.0818* |
| TNA 6975   | <LOD | <LOD    | 0.0393* | 4.98  | 19.4  | <LOD  | 0.0622* |
| TNA 6977** | <LOD | <LOD    | 0.0339* | 0.844 | 3.01  | <LOD  | 0.0916* |
| TNA 6981   | <LOD | 0.0311* | 0.0313* | 1.77  | 17.4  | <LOD  | 0.103*  |
| TNA 6992   | <LOD | <LOD    | 0.0429* | 4.45  | 19.8  | <LOD  | 0.0826* |
| TNA 6994   | <LOD | <LOD    | <LOD    | 1.71  | 3.50  | <LOD  | 0.0616* |
| TNA 6995   | <LOD | <LOD    | 0.0366* | 3.79  | 15.1  | <LOD  | 0.0853* |
| TNA 6997   | <LOD | <LOD    | 0.0479* | 3.81  | 15.2  | <LOD  | 0.0707* |
| TNA 7000   | <LOD | <LOD    | 0.0568* | 5.68  | 22.9  | <LOD  | 0.0638* |
| TNA 7006   | <LOD | <LOD    | 0.0412* | 4.90  | 20.2  | <LOD  | 0.0667* |
| TNA 6938   | <LOD | <LOD    | 0.0226* | 2.78  | 15.6  | <LOD  | 0.0601* |
| TNA 6939   | <LOD | <LOD    | <LOD    | 1.79  | 6.23  | <LOD  | 0.0625* |
| TNA 6941   | <LOD | <LOD    | 0.0224* | 3.08  | 23.4  | <LOD  | 0.0577* |
| TNA 6950   | <LOD | <LOD    | <LOD    | 3.24  | 14.3  | <LOD  | 0.0909* |
| TNA 6953   | <LOD | <LOD    | 0.0226* | 5.24  | 22.1  | <LOD  | 0.0480* |
| TNA 6955   | <LOD | <LOD    | <LOD    | 3.29  | 10.1  | <LOD  | 0.0493* |
| TNA 6962   | <LOD | <LOD    | 0.0312* | 5.22  | 19.8  | <LOD  | 0.0705* |
| TNA 6964   | <LOD | <LOD    | 0.0374* | 5.80  | 21.7  | <LOD  | 0.101*  |
| TNA 6966   | <LOD | <LOD    | 0.0229* | 4.62  | 17.5  | <LOD  | 0.0620* |
| TNA 6970   | <LOD | <LOD    | <LOD    | 4.06  | 17.0  | <LOD  | 0.137*  |
| TNA 6972   | <LOD | <LOD    | 0.0345* | 3.09  | 12.7  | <LOD  | 0.0653* |
| TNA 6978** | <LOD | <LOD    | 0.0210* | 0.945 | 2.71  | <LOD  | 0.0998* |
| TNA 6979   | <LOD | 0.0344* | 0.0228* | 2.31  | 16.7  | <LOD  | 0.102*  |
| TNA 6982   | <LOD | <LOD    | <LOD    | 1.26  | 9.37  | <LOD  | 0.0790* |
| TNA 6984   | <LOD | <LOD    | <LOD    | 1.73  | 15.8  | <LOD  | 0.0911* |
| TNA 6986   | <LOD | <LOD    | <LOD    | 1.41  | 11.3  | <LOD  | 0.0683* |

**Table 11. Sample Results for Additional Perfluorinated and Highly Fluorinated Analytes (continued)**

| Sample ID          | PFSA<br>ng/L | PFSA<br>ng/L | PFSA<br>ng/L | PFOS<br>ng/L | PFOS<br>ng/L | PFOS<br>ng/L | PFOS<br>ng/L |
|--------------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|
| LLOQ (ng/L)        | 0.003        | 0.003        | 0.003        | 0.003        | 0.003        | 0.003        | 0.003        |
| LOD (ng/L)         | 0.0046       | 0.0058       | 0.0046       | 0.0046       | 0.0046       | 0.0046       | 0.0046       |
| TNA 6988           | <LOD         | <LOD         | <LOD         | 1.79         | 16.5         | <LOD         | 0.0979*      |
| Mean               | NA           | 0.0776*      | 0.0348*      | 3.27         | 16.3         | NA           | 0.0839*      |
| Median             | NA           | 0.0401*      | 0.0339*      | 3.27         | 16.9         | NA           | 0.0819*      |
| High               | NA           | 0.199*       | 0.0706*      | 5.80         | 32.8         | NA           | 0.155*       |
| Low                | NA           | 0.0311*      | 0.0210*      | 0.780        | 2.71         | NA           | 0.048*       |
| Standard Deviation | NA           | 0.0812       | 0.0122       | 1.52         | 6.71         | NA           | 0.0257       |
| # OF HITS          | 0            | 4            | 23           | 36           | 36           | 0            | 36           |

NA = Not Applicable

\*Analyte concentration was below the LLOQ and above the LOD.

\*\*TN-A-6977 and -6978 are from the same lot of pooled serum.

Based on <sup>13</sup>C PFOA surrogate recovery, the overall method uncertainty is +/- 20%.

**Table 12. Isomer Ratios for Individual Samples**

| Sample ID  | Sample Origin    | Sum of Isomers | Sum of Isomers |
|------------|------------------|----------------|----------------|
| TNA 6840   | California       | 11%            | 40%            |
| TNA 6843   | Missouri         | 13%            | 40%            |
| TNA 6847   | Pennsylvania     | 4.1%           | 43%            |
| TNA 6934   | Pennsylvania     | 0.8%           | 45%            |
| TNA 6935   | New York         | 12%            | 37%            |
| TNA 6944   | Michigan         | 3.4%           | 35%            |
| TNA 6959   | Virginia         | 4.3%           | 40%            |
| TNA 6960   | Virginia         | 4.5%           | 38%            |
| TNA 6967   | Maryland         | 9.5%           | 43%            |
| TNA 6973   | Montreal, Quebec | 4.3%           | 42%            |
| TNA 6975   | Montreal, Quebec | 4.0%           | 42%            |
| TNA 6977** | Massachusetts    | < LLOQ*        | 39%            |
| TNA 6981   | Texas            | 3.7%           | 43%            |
| TNA 6992   | California       | 4.2%           | 40%            |
| TNA 6994   | California       | 4.0%           | 39%            |
| TNA 6995   | California       | 6.0%           | 41%            |
| TNA 6997   | California       | 4.0%           | 41%            |
| TNA 7000   | California       | 17%            | 41%            |
| TNA 7006   | California       | 6.3%           | 41%            |
| TNA 6938   | New York         | 2.7%           | 44%            |
| TNA 6939   | New York         | 4.0%           | 35%            |
| TNA 6941   | New York         | 3.0%           | 36%            |

Table 12. Isomer Ratios for Individual Samples (continued)

|            |                    |         |     |
|------------|--------------------|---------|-----|
| TNA 6950   | Michigan           | 0.3%    | 39% |
| TNA 6953   | Michigan           | 10%     | 39% |
| TNA 6955   | Michigan           | 3.0%    | 35% |
| TNA 6962   | Virginia           | 6.8%    | 41% |
| TNA 6964   | Virginia           | 5.4%    | 42% |
| TNA 6966   | Virginia           | 7.9%    | 40% |
| TNA 6970   | Maryland           | 6.7%    | 39% |
| TNA 6972   | Maryland           | 5.1%    | 38% |
| TNA 6978** | Massachusetts      | < LLOQ* | 41% |
| TNA 6979   | Massachusetts      | 7.6%    | 30% |
| TNA 6982   | Texas              | 6.0%    | 41% |
| TNA 6984   | Texas              | 1.3%    | 31% |
| TNA 6986   | Texas              | 0.1%    | 43% |
| TNA 6988   | Texas              | 4.5%    | 33% |
|            |                    |         |     |
|            | Average:           | 6%      | 39% |
|            | Standard Deviation | 3%      | 3%  |

\*Sample value was < LLOQ

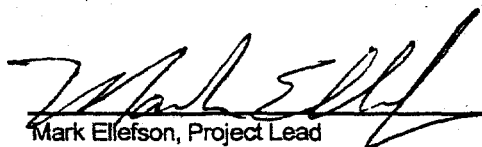
\*\*TN-A-6977 and TN-A-6978 are from the same lot of pooled serum.

All original raw data and the analytical report have been archived at the 3M Environmental Laboratory. The test materials and analytical reference standard reserve samples, as well as the samples pertaining to this project are archived at the 3M Environmental Laboratory.

Reserve samples, digital copies of original data and all original paper data will be retained in the archives of 3M Environmental Laboratory for a period of at least 10 years following report signing.

Under the conditions of the study, endogenous fluorochemicals were found to be present in all samples. PFHS, PFOS, and THPFDS were detected in every lot of serum while PFOA was present in detectable levels in all but one sample. Conversely, FBASA, THPFOS, and the C<sub>12</sub> acid were not detected in any of the samples using the present methodology. <sup>13</sup>C PFOA surrogate recoveries were all within ±20%.

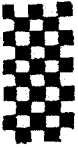
8. Signatures

 08/12/05  
Mark Ellefson, Project Lead Date

 08/12/05  
William Reagen, Laboratory Manager Date

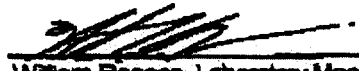
The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.

\_\_\_\_\_  
Quality Assurance Representative Date



8. Signatures

 08/12/05  
Mark Elfrson, Project Lead Date

 08/12/05  
William Reagan, Laboratory Manager Date

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

 8/12/05  
Quality Assurance Representative Date