Particle Size Measurement at Washington Works

Draft Test Plan

March 2004

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I. INTRODUCTION

This field study is being conducted to measure the particle size distribution of airborne APFO/PFOA particles in emissions from the Washington Works site. This information will be used to better understand transport of this material in ambient air.

II. TEST PLAN

1. Site Description

Monitoring will be conducted in or near the E. I. DuPont manufacturing facility located in Parkersburg, West Virginia.

2. Target Compound

The target compound for this monitoring program is the anion perfluoroctanoate (PFO-) originating from either perfluorooctoanoic acid (PFOA) or ammonium perfluorooctanoate (APFO).

3. Sampling Strategy

Air Sampling Events, Duration and Locations

The particle size program will consist of 3 sampling events conducted over a period of approximately 4 weeks. During each sampling event air emissions will be collected over a 72-hour period at a designated location.

Event 1: The first event will consist of fenceline sampling, located at the predicted maximum according to air dispersion modeling (see Figure 1). Three High Volume Cascade Impactors will be located at the sample location to provide triplicate sampling information. Samplers will be separated with enough distance to avoid influencing each respective airflow. One background sampler will be located to collect predicted upstream concentrations, assuming wind patterns follow predominant North/Northeast directions as expected. (See Figure 1 for background location.)

Event 2: It is expected that samplers for Event 2 will be located within the fenceline, closest to stacks with the highest predicted mass flow rate and particle size. Proposed locations are shown in Figure 2. Exact locations will be finalized after Event 1 is complete.

Event 3: It is expected that samplers for Event 3 will be placed in 2 locations; duplicate samplers will be located at the fenceline, at the point of predicted maximum. Duplicate samplers will also be located at distance from the fenceline to evaluate differences in particle size distribution with distance from the site. Proposed locations are shown in Figure 3. Exact locations will be finalized after Event 1 is complete.

Figure 1: Proposed Air Sampling Locations - Event 1

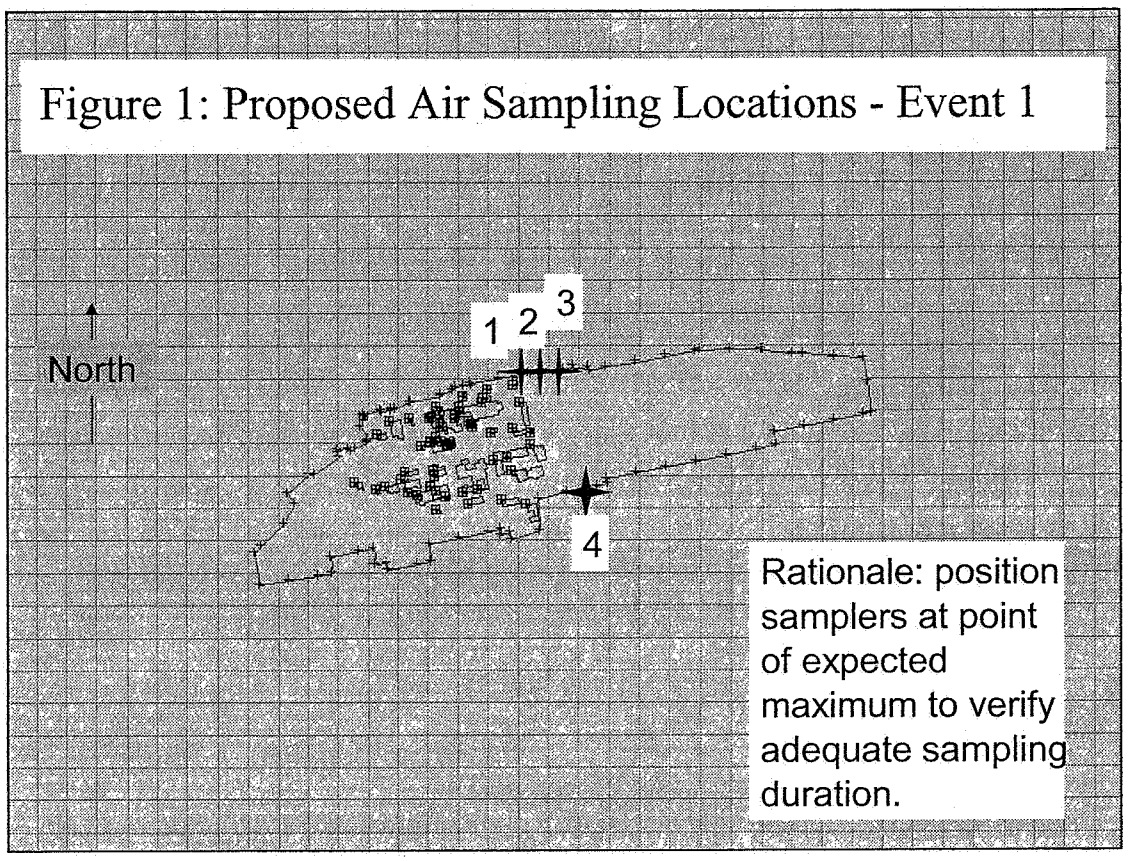


Figure 2: Proposed Air Sampling Locations - Event 2

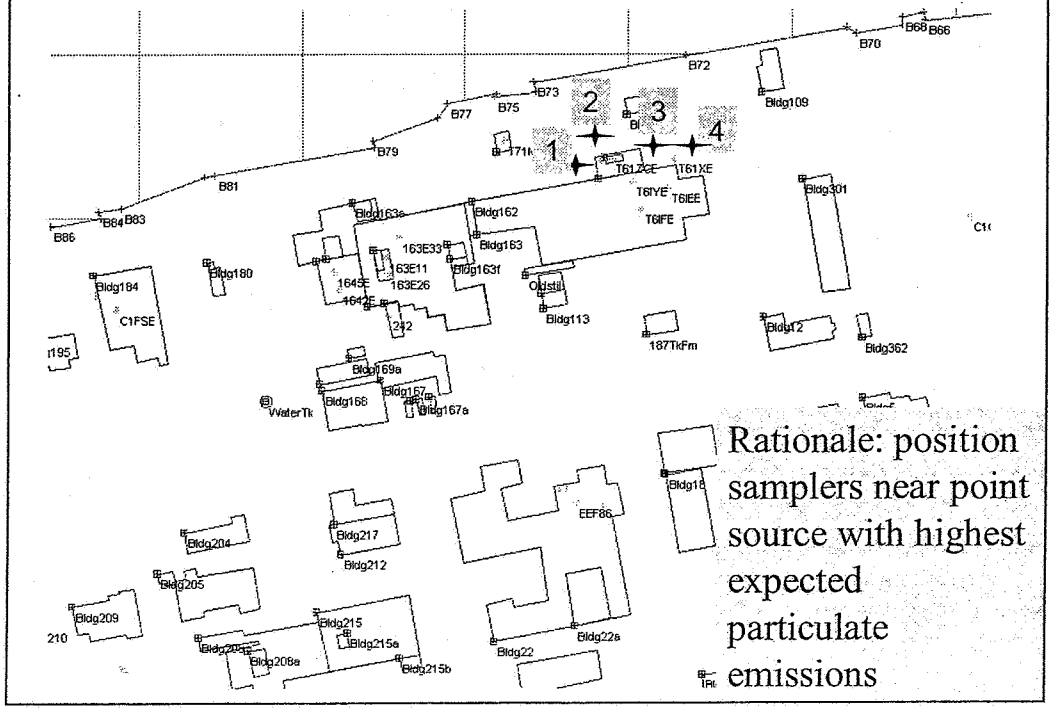
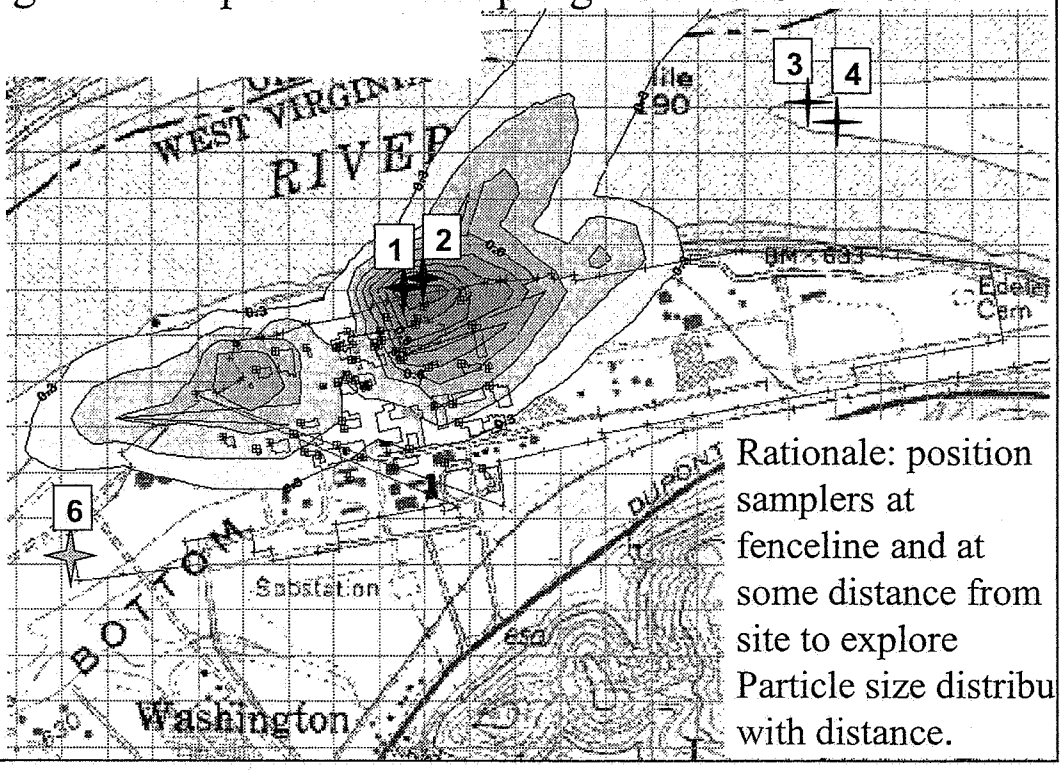


Figure 3: Proposed Air Sampling Locations - Event 3



Rationale: position samplers at fenceline and at some distance from site to explore Particle size distribu with distance.

Number of Samples

A total of 75 samples will be analyzed over the duration of the program. The samples will include:

- One field blank per sampling event
- Six samples from each of the four sampling locations per event (five stages + one back-up sample for each sampler)
- A total of 25 samples per event x 3 events = 75 samples

4. Sampling Equipment and Methods

Sampling will be conducted using Model 235 Five-Stage High Volume Cascade Impactors, manufactured by Tisch Environmental, as shown in Figure 4. (See Appendix A for equipment specifications.) This method involves drawing a measured volume of ambient air through a series of filters in the cascading impactor, representing various particle "cut-sizes". The species collected in each stage will be preserved and shipped to a laboratory for separate analysis of PFO-. The sampling protocol will follow EPA methods established in 40 CFR 50.11, Appendix B, July 1, 1975) that describes a high volume method for determination of suspended particulate matter in the atmosphere. (See Appendix B for the regulatory standards text.) This test program will adapt the EPA protocol to include a cascade impactor with the following cut sizes:

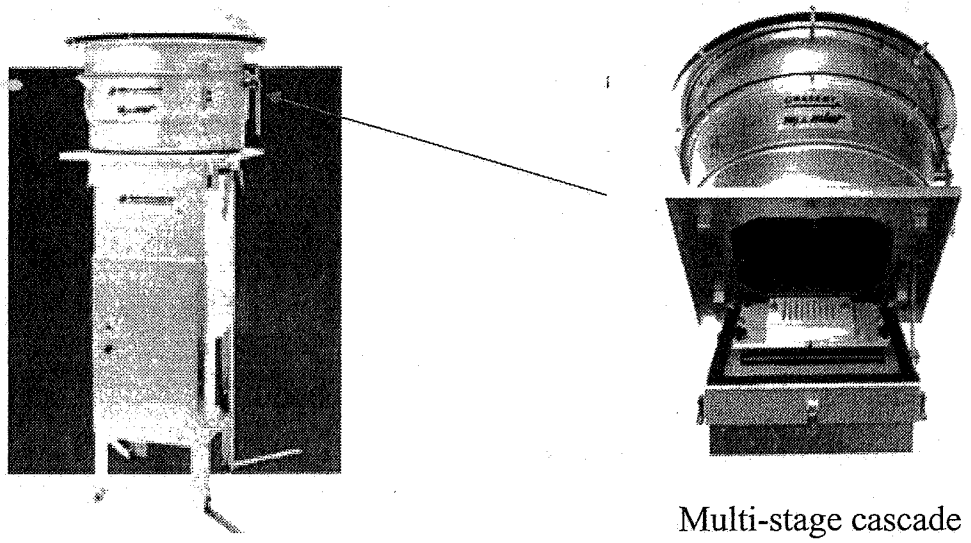
Stage 1 – greater than 7.2 microns
Stage 2 – 3.0 to 7.2 microns
Stage 3 – 1.5 to 3.0 microns

Stage 4 – 0.95 to 1.5 microns
Stage 5 – 0.49 to 0.95

Collection substrates for this test program are specified as cellulose filters. This medium was selected to maximize the capture of the species of interest, PFO-. Preliminary laboratory testing was performed to evaluate the effectiveness of cellulose filters. Results are summarized in Appendix C.

Impactors will operate at a nominal flow rate of 40 standard cubic feet per minute (scfm) over the 72-hour sampling period. Sampling equipment will be mounted on wooden pallets for stability. Power will be supplied to each location via AC power. Field logs will be provided to record pertinent information during sampling events. (See Appendix D for sample log sheet.)

Figure 4: High Volume Sampler With Cascade Impactor



Size selective particulate high volume sampler

Multi-stage cascade impactor, ranging from ~ 7 to 0.49 micron cut sizes

5. Analytical Methods

Cellulose filters will be extracted and PFO- determined at Exygen Research, State College, PA. Exygen will use analytical methodology that has been submitted to the public docket under OPPT-2003-0012-0040. Results will be reported as PFOA to align with previous air studies. The methodology uses a methanol extraction with liquid chromatograph/mass spectrometer/mass spectrometer (LC/MS/MS) to detect PFO-. Method detection limits are approximated at 50 ppt in water.

Quality Assurance and Quality Control

Quality Assurance (QA) includes a description of actions taken to ensure data measurements are of sufficient quality to meet the overall goals of the program. QA consists of things such as personnel training, standardization of procedures, documentation, data validation and data quality evaluations. Quality control (QC) describes the operational techniques and activities that are used to fulfill requirements for quality. QC consists of activities such as routine instrument checks, flow rate checks, duplicate samples, blanks, and calibration. Various QA/QC procedures are described in this section.

Shipping Protocol

Samples will be shipped to Exygen Research in State College, PA for analysis. A standardized protocol for sample preservation, transport and storage and chain-of-custody information are included in Appendix E.

Calibration Procedures

The calibration of samplers will be performed prior to and after each of the 3 sampling events. Each instrument will be calibrated in accordance with procedures outlined in 40 CFR Part 50 Appendix B, section 9.

QC Samples

QC field blanks will be collected during each sampling event to measure possible contamination introduced by sampling procedures, media, equipment, or shipment. Field blanks will be handled in the same manner as actual samples. A total of three field blanks will be collected for the monitoring program. One sample spike will also be provided by Exygen and inserted into the final stage of one sampler during the final sampling event. This sample will provide information about laboratory recovery.

IV. RESULTS

Following the completion of laboratory analysis of filters at each stage, concentrations of PFOA will be available to calculate the mass distribution from greater than 7.2 microns, to less than 0.49 microns.

Appendix A

Equipment Specifications

http://www.pacwill.ca/230seri.htm

230 Series Hi-Volume Impactor

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This is a multi-stage cascade impactor which attaches to any standard high volume air sampler and fractionated suspended particulates into as many as six size fractions (model 235), nominally: 7.2 microns and greater, 3.0 microns, 1.5 microns, 0.95 microns, 0.49 microns, and less than 0.49 microns (back-up filter in hi-vol. sampler). Andersen Instruments' High Volume Cascade Impactors have been fully calibrated and field-tested by EPA and other research laboratories. The Series 230 provides a simple, accurate, yet low-cost means of obtaining the particle size distribution and respirable mass fraction of suspended particulates in outdoor and occupational environments. The unique slotted impactor design features the ultimate precision, predictability, and sharpness of cut-off, combined with compactness, ease-of-use, and the lowest internal losses.

Particles are sized aerodynamically

It is only the particle size applicable to the determination of the "respirable" particle mass fraction, particle transport, and particulate pollution control equipment. The nominal flow rate is 40 CFM, which falls in the standard range specified by EPA. Particles are collected on small, light-weight reflection substrates. Both these features provide large particulate samples and unsurpassed measurement sensitivity. The final stages, Stages 5 and 6, provide important data points in the sub-micron range.

Principle of Operations:

Suspended particulates enter the High Volume Cascade Impactor through the parallel slots in the first impactor stage, and particles larger than the particle cut-off size of the first stage impact on the slotted collection substrate. The air stream then passes through the slots in the collection substrate, accelerates through narrower slots in the second impactor stage, and the remaining particles greater the particle cut-off size of the second stage impact on the second collection substrate and so on. The width of the slots is constant for each impactor stage, but is smaller for each succeeding stage, and most of the smaller particles eventually acquire sufficient momentum to impact on one of the collection substrates. After the last impactor stage, the remaining time particles are collected by the back-up filter in the high volume sampler. The collection substrate and back-up filter are weighed before and after sampling to determine the particle size distribution. Since all particles are collected, the impactor also yields total particles mass concentration.

Features:

- The flow rate is 40 CFM
- The maximum number of impactor stages available is 5 (Model 235) or 6 (Model 236
- There are two impactor cut-points in sub micron range
- The tare weight of collection substrates is 1.2 g
- The collection substrates are ail to the same configurations
- Gaskets are not needed

Appendix B

40 CFR 50.11, Appendix B, July 1, 1975

National Primary and Secondary Ambient Air Quality Standards

Appendix C

Summary of Laboratory Work to Evaluate Cellulose Filters

Appendix D
Sample Field Log

PLACEHOLDER FOR RAMCON'S FIELD LOG

DuPont Washington Works
Air Sampling Program

Sampler(s): _____ **FedEx Airbill #:** _____ **Event Number** _____

Pump #	Station #	Exygen ID	Calibration (pre)	Date	Start Time	Date	Stop Time	Calibration (post)	Total Minutes	Total Volume
669842	AMS-1									
669908	AMS-2									
669878	AMS-3									
669809	AMS-4									
669769	AMS-5									
669848	AMS-6									

Field Blank: yes
yes or no

Weather:

SAFER operational:
yes or no

Comments:

Appendix E

Sample Shipment Protocol

SUGGESTED SAMPLE HANDLING, CHAIN-OF-CUSTODY, PRESERVATION and SHIPPING – AIR SAMPLES

The person collecting the sample should wear clean latex or nitrile disposable lab or exam gloves and should limit his/her contact with the samples.

Cellulose filters will be provided by the contracted laboratory and should be stored in a clean, dry location. Fresh filters and collected samples are not required to be chilled, however neither should be subjected to temperature extremes.

In order to minimize the possibility of introducing APFO contamination into samples, the following protocol should be followed:

- Avoid fluropolymers.
- Avoid aluminum foil.
- Do not use self-stick memo notes.
- Avoid blue ice (samples are shipped at ambient temperature).
- Avoid pre-wrapped foods or snacks.
- Wear clothing that has been washed at least six times.
- Use only containers supplied by contract laboratory.

Following sample collection, filters should be placed in a zip-loc bag. Seal the zip-loc bag.

Complete the sample label and chain-of custody (COC) form, including the volume of air sampled. The completed sample label is attached to the outside of the zip-loc bag. Pack the samples in a small box or cooler with adequate packing. Include the completed COC form.

Seal the box or cooler with at least one (1) custody seal.
Complete a mailing label and airbill and ship by overnight carrier to:

Exygen Research
3058 Research Drive
State College, PA 16801
814-272-1039

Contacts at the laboratory are Shawn Robb and John Flaherty.
Samples should be shipped to the laboratory same day as collection, and will be received at the laboratory within 72 hours of collection.