

DRAFT PHASE II QUALITY ASSURANCE
PROJECT PLAN
DUPONT WASHINGTON WORKS SITE
WASHINGTON, WEST VIRGINIA

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CORPORATE REMEDIATION GROUP
An Alliance between
DuPont and URS Diamond

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1.0 PROJECT MANAGEMENT

This Phase II Quality Assurance Project Plan (QAPP) is one of the required deliverables for the Memorandum of Understanding (MOU) between the United States Environmental Protection Agency (USEPA) and E. I. DuPont de Nemours and Company (DuPont) for a Perfluorooctanoic Acid (PFOA) Site-Related Environmental Assessment Program (March 2004). The PFOA Site-Related Environmental Assessment Program will be conducted at and near the DuPont Washington Works Site in Washington, West Virginia. PFOA is the acid form of ammonium perfluorooctanoate (APFO, commonly known as C-8 and historically known as FC-143) and other salts.

This QAPP is written to describe policies, project organization, functional activities, and Quality Assurance/Quality Control (QA/QC) measures intended to achieve the project data quality objectives for sampling activities associated with the Phase II investigation. This QAPP is intended to meet the requirements for conducting the work in accordance with QA/QC field protocols for collecting environmental measurement data.

This QAPP was prepared in general accordance with USEPA documents:

- ❑ EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 (EPA/240/B-1/003, March 2001)
- ❑ EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5 (EPA/240/R-02/009, December 2002)

1.1 Project Background

This QAPP is referenced as part of Appendix 5 in an MOU between USEPA and DuPont for conducting a PFOA Site-Related Environmental Assessment Program. The MOU summarizes DuPont's commitment to provide USEPA with information on PFOA through a PFOA Site-Related Environmental Assessment Program at and near the DuPont Washington Works Site in West Virginia.

The PFOA Site-Related Environmental Assessment Program employs a phased approach. Phase I includes commitments previously made by DuPont that were memorialized in a Letter of Intent (LOI) and that includes environmental monitoring and modeling activities at and near the DuPont Washington Works Site. Phase I also includes other relevant environmental work not within the scope of the LOIs. Many of the Phase I environmental monitoring/modeling commitments have already been conducted. However, some Phase I commitments, such as surface water and groundwater monitoring at the Local, Letart and Dry Run Landfills per West Virginia National Pollutant Discharge Elimination System (WV/NPDES) permits WV0001279, WV0076538, WV0076066 and WV0076244, are ongoing. Details of the LOI commitments and other relevant environmental commitments can be found in Appendices 3 and 4 of the MOU.

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1.2 Project Description

Phase II of the PFOA Site-Related Environmental Assessment Program is made up of commitments that augment and build upon LOI and other relevant environmental commitments. Phase II involves sampling on-site and sampling off-site in both West Virginia and Ohio of various media including:

- ☐ Air
- ☐ Drinking water sources (private and public water sources)
- ☐ Groundwater
- ☐ Surface waters
- ☐ Surface soil/biota
- ☐ Subsurface soil

PFOA is the entity detected by the analytical instrument for all media. Aqueous and soil samples will be analyzed and reported for C-8.

In aqueous solutions, C-8, or ammonium perfluorooctanoate (APFO) forms perfluorooctanoic acid (PFOA). PFOA is extracted from water using C₁₈ solid phase extraction (SPE) cartridges. PFOA is eluted from the SPE cartridge using methanol, resulting in an 8:1 concentration of the sample. Analysis is performed by liquid chromatography-tandem mass spectrometry (LC/MS/MS) using selected reaction monitoring (SRM). Quantitation is accomplished via external standard calibration. Levels of PFOA found are mathematically converted and reported as APFO. Historic water results were recorded in the Corporate Environmental Database (former CED) and were reported as FC-143. Phase II C-8 analytical results for water samples will be recorded in Envista and will be reported as FC-143 for consistency with historical results.

For soil samples, the samples are mixed with methanol, sonicated, centrifuged and filtered. The methanol extracts are analyzed by LC/MS/MS. A known quantity of the labeled compound PFOA-di-13C is added to every sample and to the batch quality control samples prior to extraction. Because the isotopically labeled compound is chemically identical to the compound of concern, it is affected by any interfering substances in the sample to the same extent as the compound of concern. The recovery of the PFOA-di-13C is then used to mathematically correct the final result for the APFO. Historic soil results were recorded in the former CED and were reported as FC-143. Phase II C-8 analytical results for soil samples will be recorded in Envista and will be reported as FC-143 for consistency with historical results.

Air samples will be analyzed and reported for PFOA. PFOA is extracted from each section of an OVS air tube with methanol. Contents of sample tubes will be analyzed separately for vapor and particles greater than 0.3 microns, then composited for a total concentration at each sampling location. Analysis is performed by liquid chromatography/mass spectrometry (LC/MS) using single ion monitoring (SIM). Quantitation is accomplished via external standard calibration. Phase II PFOA analytical results for air samples will be recorded in Envista and will be reported as PFOA, consistent with analytical results obtained during Phase I.

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Method development for PFOA analysis of surface soil/biota samples has been initiated. It is anticipated that samples will be homogenized in the laboratory using a Hobart chopper or the equivalent, so that a uniform subsample may be obtained. Analysis of a prepared extract will be performed using LC/MS/MS. Phase II PFOA analytical results for biota samples will be recorded in Envista and will be reported as PFOA.

All Phase II work will be conducted by DuPont or its authorized representatives. A description of the Phase II work for each media is provided in the Phase II Work Plan. For each media, a brief discussion of the sampling locations, number, and frequency of sampling is presented. The Phase II Work Plan, to which this QAPP is attached as an Appendix, also provides a schedule and reporting requirements.

1.3 Project Organization

DuPont's Corporate Remediation Group (CRG) in Wilmington, Delaware, will be the lead organization for managing the Phase II investigation. Oversight will be provided by the USEPA, West Virginia Department of Environmental Protection (WVDEP), and Ohio EPA (OEPA). DuPont CRG is supported by a contractor, URS Diamond (URSD), who will provide technical professionals that will conduct the Phase II monitoring/sampling activities. Laboratory analytical testing of water and air samples will be conducted by Exygen Research (Exygen), located at State College, Pennsylvania. Severn Trent Laboratories, Inc., Arvada, Colorado (STL - Denver) will perform analysis of subsurface soil samples. Method development for surface soil/biota (organic matter-rich portions and mineral-matter rich portions) analysis has been initiated at Morse Laboratories (Morse), Sacramento, California. Environmental Standards, Inc. (ESI), Valley Forge, Pennsylvania will provide third party data validation services for the project. The contract laboratories will be accredited as required by the state agencies and others, as appropriate.

A description of the program organization and the responsibilities associated with the positions are described in the paragraphs below. The persons described will be charged with ensuring the collection of usable data and assessing measurement systems for precision and accuracy.

An organizational chart for the project is shown in Figure 1. Responsibilities for project team members are summarized below.

1.3.1 Project Director

Mr. Andrew Hartten, DuPont CRG, is the project director for the project. His responsibilities will be as follows:

- ☐ Providing strategic-level review of technical activities
- ☐ Providing direction involving drinking water, groundwater, surface water, and hydrogeologic investigations
- ☐ Approving project-specific procedures and internally prepared plans, drawings, and reports
- ☐ Providing guidance to the project team

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- ☐ Acting as the DuPont representative for the Phase II activities to USEPA, WVDEP, and OEPA

1.3.2 Project Manager

Mr. Mark Houlday, URSD, is the project manager for the site. He is the primary point of contact with DuPont and will be responsible for all technical, financial, and scheduling matters. His other responsibilities will be as follows:

- ☐ Assigning duties to the project team and orienting the team to project needs and requirements
- ☐ Disseminating project-related information from DuPont
- ☐ Acting as liaison with subcontractor organizations (unless specifically delegated to others)
- ☐ Interacting with the QA officer and health and safety officer to ensure that these programs are functioning effectively and safely
- ☐ Serving as the collection point for project team reporting of nonconformance with QA procedures or changes in project documents and activities

1.3.3 Health and Safety Officer

Ms. Katherine Sova, URSD, is the health and safety officer for the project. She will be responsible for developing, reviewing, and approving of the project health and safety plan (HASP). She will ensure that the project HASP is consistent with applicable state and federal regulations and will also be responsible for implementing the HASP.

1.3.4 Project Geologist

The project geologist is to be determined and will be responsible for the following:

- ☐ Coordinating or leading the site investigation and sampling teams
- ☐ Interacting with the project chemist regarding sampling events
- ☐ Evaluating site groundwater and surface water data
- ☐ Leading the preparation of reports and documentation

1.3.5 Technical Consultants

Senior staff members with expertise in the disciplines associated with the site investigation are available to the project as needed. These individuals will participate in the project as directed by the project manager.

1.3.6 Technical and Support Staff

Individuals in this category will participate in the technical activities associated with the project and will be coordinated by the technical lead or project manager.

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1.3.7 Quality Assurance Officer

Mr. Michael Aucoin, URSD, is the QA officer for the project. His responsibilities will be as follows:

- ☐ Developing and reviewing the QAPP
- ☐ Administering the QAPP
- ☐ Supervising day-to-day QA activities
- ☐ Notifying personnel of nonconformance or changes in procedures
- ☐ Determining the system and performance audit schedules, if required

1.3.8 Project Chemist

Mr. Michael Aucoin will also be the project chemist for the project. He will schedule all sample container orders and analytical requests with the laboratory. He will also be the point of contact between the laboratory and project team. He will coordinate review of data generated by the laboratory.

1.3.9 Laboratory Personnel

Oxygen (air and aqueous samples) and STL - Denver (subsurface soil samples) will provide analytical services for this project. Morse is anticipated to provide analysis of surface soil/biota samples. If Morse is unable to provide analysis for surface soil/biota sample extracts, then STL -Denver will provide analysis of these extracts. The regulatory agencies will be notified of any change in the designated laboratories.

The key laboratory personnel for this project will be the laboratory project managers. The analytical laboratory project manager will be responsible for execution of the analytical testing program for the project. The laboratory project manager will be responsible for laboratory analyses and data processing. The laboratory project manager will be the point of contact for the project chemist and QA officer and will be assisted by the laboratory QA director, who is responsible for ensuring that laboratory internal QA procedures are followed and for processing QA data. The laboratory project manager is also responsible for submitting the final data package, including hardcopy deliverable and electronic data deliverable (EDD, see Section 1.6.6 and Appendix A), within the requested turnaround time.

The laboratory has signed a contract with DuPont detailing the terms and conditions for services. This contract includes a guarantee to dispose of samples following analysis in accordance with all pertinent federal, state, and local laws and ordinances.

1.3.10 Data Review Responsibilities

ESI will be the third-party data reviewer for the project. ESI will provide an independent review and validation of approximately 10 percent of all the data points collected. Data generated for select portions of the sampling program, such as residential sampling, will undergo third party validation at a higher rate.

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1.4 Quality Objectives

To ensure that the data collected during the Phase II investigation is sufficient and of consistent quality, data quality objectives (DQOs) for precision, accuracy, and completeness have been established. The sampling and analysis and associated QA efforts are aimed at achieving these DQOs in a timely, cost-effective, and safe manner. Data collected during previous investigations provided baseline information on contamination around the site.

1.4.1 Data Quality Characteristics

DQOs are statements of the level of uncertainty that a decision-maker is willing to accept in results derived from environmental measurements. The uncertainty for sample parameter results may arise from a combination of factors, including sampling procedures, sample matrix characteristics, non-homogeneity of samples, and the inherent accuracy and precision limitations of analysis methods. DQOs are quantitatively and qualitatively described in terms of data quality characteristics (DQCs), which include precision, accuracy, representativeness, completeness, and comparability. These characteristics are defined as follows:

☐ *Comparability*

Comparability expresses consistency in sampling and analytical procedures so that one data set can be compared to another.

☐ *Representativeness*

Representativeness expresses the degree to which sample data accurately and precisely represent a selected characteristic of a population, parameter variations at the sampling point, a process condition, or an environmental condition.

☐ *Precision*

Precision is defined as the agreement between numeric values for two or more measurements that have been made in an identical fashion.

☐ *Accuracy*

Accuracy is the degree of agreement of a measurement with an accepted true value.

☐ *Completeness*

Completeness is a measure of the amount of the usable data obtained from the measurement system compared to the amount that was expected under normal conditions.

1.4.2 Data Quality Objectives

The purpose of the Phase II investigation is to characterize multi-media samples in and near the Washington Works Main Plant, including across the Ohio River in Ohio, and around the associated landfills (Local, Letart, Dry Run) in West Virginia that may have been impacted by plant operations. To accomplish this, the precision, accuracy, and

completeness acceptance criteria required for each media during the Phase II investigation were developed and are listed in Table 1. Aqueous and soil samples will be analyzed for PFOA and reported for C-8. Air and biota samples will be analyzed and reported for PFOA. All measurement data will be calculated and reported in units consistent with specified methodologies (see Section 2.4) to ensure comparability with historical data.

1.4.3 Measurement System Characteristics

Measurement system characteristics are considered to be key performance measures used to indicate that the laboratory is performing the referenced analytical method correctly. These characteristics are range, sensitivity, selectivity, accuracy, and precision.

It should be recognized during the following discussion that PFOA is the entity detected by the analytical instrument for all media. Aqueous and subsurface soil samples will be analyzed for PFOA and reported for C-8. Air and surface soil/biota samples will be analyzed and reported for PFOA.

Range

The reporting range can be represented by the difference between the limit of quantitation (LOQ) and the highest calibration standards analyzed, at which comparable precision and accuracy have been demonstrated. The reporting range of the analytical method can be extended on a sample specific basis by dilution.

The reporting range for PFOA in air, without dilution, is 50 to 1500 nanograms/fraction (ng/fraction), equivalent to 0.1 to 3 micrograms/OVS tube fraction (ug/fraction) when sampling at 1 L/min for 24 hours.

The reporting range for C-8 in water, without dilution, is 50 to 1000 nanograms/liter (ng/L).

The reporting range for C-8 in soil, without dilution, is 2 to 100 micrograms/kilogram (ug/kg).

The reporting range for PFOA in biota (vegetation and litter layer, associated root zone and surface soil) is to be determined; method development and validation are ongoing.

Sensitivity

Sensitivity relates to the ability of a measurement system to accurately measure the analyte or property of interest over a range. Sensitivity is defined as the capability of a method to discriminate between measurement responses representing different levels of a variable of interest.

Sensitivity is demonstrated for PFOA, and thus C-8, by injection of a calibration standard at the LOQ and five times the LOQ. In addition, a method detection limit (MDL) study per 40 CFR 136 is performed for each instrument used to analyze samples.

Selectivity

Selectivity is defined as a measurement system's ability to accurately discriminate the analyte of concern from other analytes in the matrix that may interfere.

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Selectivity is demonstrated during analysis of aqueous samples by the presence in the chromatogram of a peak of a daughter ion at 369 amu from a parent of 413 amu. The 413-amu parent corresponds to the PFOA anion, while the daughter ion (369 amu) represents the loss of carbon dioxide. This transition does not discriminate between linear and branched forms of PFOA; therefore, both forms, if present, would be included in the calculation. In addition, the retention time drift for PFOA must not exceed 2% during the run sequence, per the laboratory standard operating procedure (SOP).

Selectivity is demonstrated during analysis of soil samples by the presence in the chromatogram of a peak with daughter ions at 369 and 269 amu from a parent of 413 amu. The ratio of the peak areas of the two PFOA ions must be $\pm 20\%$ of the ion ratios in mid-point ICAL. In addition, the retention time must fall within ± 30 seconds of the retention time observed in the ICAL standards.

Selectivity is demonstrated during air sample analysis by the presence of a peak at 369 amu. The retention time drift for the surrogate standard PFNA must not exceed 2% during the run sequence.

Selectivity is further demonstrated by adequate instrument tunes, and by control of the liquid chromatography-tandem mass spectrometry (LC/MS/MS) cone and quadrupole voltages.

Accuracy

Accuracy is the difference between the mean of the test results for the analyte of interest and the known value of the analyte concentration or value. Accuracy measurements are obtained for PFOA and reflect the accuracy of reported C-8 results.

Accuracy of the C-8 determination for aqueous and soil samples is demonstrated through the use of matrix spikes (MS) with every field sample, or a minimum of one per batch. MS recoveries must fall in the range of 70 to 130%, unless the sample concentration is significantly higher than the spike concentration. In addition, results for laboratory method blanks must be less than the LOQ.

Accuracy of the PFOA determination for air samples is assessed through the use of lab control spikes and surrogate spikes. Recoveries for lab control spikes must be within 75 to 125%. Recoveries for surrogate spikes should fall in the range 75 to 125% as well, however these are advisory limits only.

Precision

Precision is a measure of the variability of test results obtained from applying a measurement system to samples that are ostensibly the same. Precision measurements are obtained for PFOA and reflect the precision of reported C-8 results.

Every field sample may be analyzed as a laboratory replicate in order to generate sample-specific precision data for analysis of PFOA; a minimum of one laboratory replicate per analytical batch must be analyzed. Precision is assessed during the analytical run sequence through analysis of a check standard. The check standard response must be within 15% of the average response of the equivalent concentration calibration standards for aqueous samples, and 20% for soil samples.

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Laboratory replicates and/or check standards may be used to evaluate PFOA precision during air and surface soil/biota samples.

1.5 Training/Certification

The project files shall contain the documentation of training specified in 29 CFR 1910.120(e) for all persons working on-site. The level of training required is dependent upon the person's on-site activities and potential for exposure to hazardous substances and/or other hazards encountered during the operations.

Laboratory quality assurance plans and SOPs address required experience and/or training for the analyst in the laboratory.

Contract samplers have received training in proper sample procedures, including precautions specific to C-8 and PFOA sampling (see Section 2.2.2). In addition, field personnel will be trained by professional industrial hygienists familiar with air sampling equipment (see Section 2.2.9).

Specialized training required by DuPont will also be documented and retained in the project files.

1.6 Documentation and Records

This section describes the process for maintaining document control of the QAPP, as well as field records and laboratory deliverables. It should be noted that the Phase II work plan, containing this QAPP as an Appendix, will be submitted to the EPA public docket (OPPT-2003-0012).

1.6.1 Document Control

Revisions to the QAPP shall be implemented pursuant to procedures set forth in the MOU. Whenever revisions are made or addenda added to the QAPP, the revised document or addenda will be placed on the public docket to ensure that all parties having access to this QAPP will also have access to the revision or addenda.

1.6.2 Field Logbook

Each sampling team will maintain a detailed logbook. The signature of the author and the date of entry, the project name and number and the location will accompany all entries in this log. At the beginning of each sampling day, the designated team member will start the daily log by entering the date and time, the locations to be sampled, weather conditions, field team present, and any potential issues of concern. Other information to be entered into the field logbook includes, but is not limited to, observations of field activities taking place, progress, summary of equipment preparation procedures and a description of any equipment problems (including corrective action), reference to SOPs, and explanations of any deviations from the work plan or SOPs. Detailed records describing groundwater sample collection will be logged on the forms included in Appendix B or on equivalent forms. At the end of the field investigation, the field

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sampling team will deliver copies of all logbook pages and sample collection forms completed during the investigation to the project manager or his designee.

1.6.3 Sample Log

The project geologist, or designated representative, will be responsible for keeping a sample log to record information regarding each sample. The sample log may be maintained in the field logbook. The required information will include, but is not limited to:

- ☐ Project number, facility location
- ☐ Sample location description
- ☐ Sample ID
- ☐ Depth
- ☐ Analyses requested
- ☐ Time, date, sampler name,
- ☐ Equipment used to collect the sample

1.6.4 Laboratory Deliverables

The laboratory project manager will provide the data package described below to the project chemist within the specified turnaround time. Each data package should contain a case narrative, chain-of-custody (COC) forms, and the reportable and supporting data described below.

Completed Documentation

The data package should include copies of the completed field COC forms and documentation, and should also include forms that the laboratory uses to document sample condition upon receipt.

Sample Identification Cross-Reference

Sample identification cross-reference information facilitates the correlation of field and laboratory sample IDs as well as the association of field samples with a particular laboratory batch. The data package should include a listing of COC field sample IDs cross-referenced to the associated laboratory sample IDs. The data package should include an easy and unambiguous means of associating a specific QC sample (for example, the laboratory duplicate, the MS samples, and the laboratory control sample (LCS) with specific field samples.

Test Reports for Samples

Sample test reports provide specific information for each sample regarding analytical results and methods. The data package should include the test reports for all reported data. Analytical results (i.e., detected results and non-detected results) should be adjusted for sample characteristics, laboratory preparations/cleanups, and/or laboratory adjustments. Soil samples should be adjusted for moisture content and reported on a dry

weight basis. All analyte detections above the analyte LOQ should be reported. Detections below the LOQ but above the MDL for soil and water samples will be reported as NQ (not quantifiable). ND indicates the analyte was not detected at the MDL.

Surrogate Recovery Data

The data package should include the surrogate data as applicable to the analytical method performed. The surrogate data can be included on the test report for each sample, or can be included on a summary form, provided that the surrogate results are clearly and unambiguously linked to the sample from which the results were measured. The surrogate data should include the percent recovery (%R) and the laboratory's QC limits.

Laboratory Blank Samples

The data package should include test reports or summary forms for all blank samples (for example, method blanks and preparation blanks) pertinent to sample analyses. Blank sample test reports should contain all of the information (e.g., surrogate data) specified for environmental sample test reports/summary forms. Sample data should not be blank corrected.

Laboratory Control Samples

The data package should include the LCS test reports or LCS result summary forms. A LCS should be included in every preparation batch and taken through the entire preparation, cleanup, and analysis procedures. The LCS samples should contain the target analytes identified for the project applicable to the analytical method performed. The LCS test report, or LCS results summary form, should include the amount of each analyte added, the %R of the amount measured relative to the amount added, and QC limits for each analyte in the LCS.

Matrix Spike Samples

The project MS samples should be spiked with the project-specified analytes. The project MS summary forms should include identification of the compounds in the spike solution, the amount of each compound added to the MS, the parent sample concentration, the concentration measured in the MS, the calculated %R, and the QC limits for %R. The form should also include the laboratory batch number and the laboratory identification number of the sample spiked.

Laboratory Duplicates

If a laboratory duplicate was analyzed, the data package should include the duplicate sample test report summary form. The duplicate sample test report should include the calculated relative percent difference (RPD) between the sample and the sample duplicate results and the QC limits for the RPD. The test report should also include the laboratory batch number and the identification number of the sample.

Narrative

The laboratory should document and report all observed problems and/or anomalies observed by the laboratory that might have an impact on the quality of the data.

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Supporting Data Requirements

Supporting data for each data package should contain instrument printouts, laboratory notebook records, calibration and instrument performance records, and other supporting data deliverables, as described below. Each data sheet and all entries on the data sheet should be legible. Specific reporting formats are not required. However, each record must be clearly labeled with the type of data provided and with either the applicable laboratory quality control batch numbers or applicable dates.

Raw Data

The laboratory should provide the following supporting data for each field sample, laboratory blank, LCS, MS, and duplicate sample, as appropriate for the analytical method:

- ☐ Chromatograms and quantitation reports (chromatographic methods).
- ☐ Real-time instrument printouts (non-chromatographic methods).
- ☐ Laboratory notebook pages (non-chromatographic methods).

If an analyte and concentration are provided on the supporting data, but will not be reported in the test reports, the concentration should be lined out, dated and initialed and a brief explanation for the deletion from the test report should be provided. Suitable explanations include (but are not limited to): "< LOQ" (i.e., analyte concentration is less than laboratory limit of quantitation) or "FP" (i.e., false positive – analyte did not meet method identification requirements).

Internal Standard Summary

If the laboratory utilizes internal standard quantitation, a comparison of the internal standard areas to acceptance criteria should be provided. The comparison may be provided as an internal standard summary form or a summary with each sample or quality control quantitation report/ instrument printout.

Extraction/Digestion Logs

The laboratory should also provide extraction/digestion logs that document initial volumes/weights and final volumes for all field and quality control samples. Logs should identify the preparation method using an EPA method reference number, if appropriate. Quality control samples should be identified on the logs (the laboratory assigned identification that appears on the quality control reports included with the test reports is sufficient).

Instrument Performance Records

The laboratory should provide instrument performance records as appropriate to the analytical method:

- ☐ Bar graph spectrum, chromatogram, instrument performance check summary.
- ☐ Mass calibration and resolution documentation.

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Calibration Records

The laboratory should provide the following supporting data for initial and continuing calibration verifications as appropriate for the analytical method:

- ☐ Initial calibration summary form listing each target analyte, standard identification, the response factor for each target analyte in each standard, average response factor or slope and intercept, and the relative standard deviation (RSD) or correlation coefficient (r).
- ☐ Calibration verification summary form listing each target analyte, standard identification, the response factor for each target analyte in the verification standard, the average response factor or slope and intercept from the initial calibration curve, and the percent difference (%D).
- ☐ Alternatively, the calibration verification summary may list the true concentration, the found concentration, and the percent recovery.
- ☐ Chromatograms and quantitation reports (chromatographic methods).
- ☐ Real-time instrument printouts (non-chromatographic methods).
- ☐ Laboratory notebook pages (non-chromatographic methods).

1.6.5 Report Preparation

The project geologist will be the technical lead for the preparation of reports summarizing the results of the project. Status updates will be provided to USEPA semiannually. A final air report will be submitted after the air verification study is conducted and data reduction and modeling is complete. A Phase II report will be generated, for all media except air, after Phase II is complete and data are reduced and analyzed. All reports will be submitted to USEPA within 30 days for completion.

1.6.6 Electronic Data Deliverables

The project QA officer will manage data for the project in a project database. The laboratory will submit EDDs in a format suitable for input into the project database, as described in Appendix A.

1.6.7 Archival Requirements

Both hardcopy and electronic data must be archived by the laboratory for a minimum of ten years and made available by the laboratory upon request by the regulatory agencies or DuPont. Field records must be archived by CRG for a minimum of ten years from the date of report submittal and made available upon request by the regulatory agencies.

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2.0 DATA GENERATION AND ACQUISITION

The elements in this group address aspects of data generation and acquisition. This section describes the appropriate methods for sampling, measurement and analysis, data collection or generation, data handling, and quality control activities.

2.1 Sampling Process Design

The following elements of the sampling design are included in the Phase II work plan.

- ☐ Types and numbers of samples required;
- ☐ Sampling locations and frequencies;
- ☐ Sample matrices; and
- ☐ Field and laboratory measurement parameters of interest.

Tables 1, 2, and 3 of the Phase II work plan summarize the sampling design. Maps of the sampling locations are included as Figure 1 through 5 of the Phase II work plan.

2.1.1 Sample Naming

Sample labels will clearly identify the particular sample, and include the following:

- ☐ Facility name (location code) and sample ID
- ☐ Time and date sample was taken
- ☐ Sample preservation (if any)
- ☐ Analysis requested.

Sample identification numbers will be assigned, when possible, prior to sample collection in accordance with the DuPont Corporate Environmental Database (CED) sample identifier coding system to facilitate loading of samples and results to the database. All samples will have a three-digit location code as the first three digits, as follows:

- ☐ WWK Washington Works
- ☐ DRL Dry Run Landfill
- ☐ LCL Local Landfill
- ☐ LTL Letart Landfill
- ☐ WWO Washington Works - Ohio

The following digit is the matrix code:

- ☐ A Air
- ☐ D Drinking Water
- ☐ G Groundwater
- ☐ K Blank water

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- ☐ R Biota (vegetation and litter layer)
- ☐ S Soil (subsurface samples)
- ☐ W Surface Water
- ☐ X Surface Soil (root, root zone and soil)
- ☐ Z Other liquid

The following digits define the sampling point, with maximum character length of 16 that includes sample depth in feet in parentheses when applicable (e.g., WWK-S-SB14(0-2)).

Field duplicate samples will receive a sample identification, which is in the same format as that of the original samples, but will have a "DUP" suffix (e.g., WWK-S-SB14(0-2)-DUP).

Matrix spike and matrix spike duplicate samples will receive sample identifications that are similar to that of the original sample, with the exception that the matrix spike and matrix spike duplicate sample identification will have "MS" or "MSD", respectively, as a suffix (e.g., WWK-S-SB14(0-2)-MSD).

Trip (if any) and equipment blanks will receive sample identification which will include the location and matrix code, and a sequential number (e.g., WWK-K-EQBLK-1).

Samples collected for dissolved metals analysis, if any, will have a "DIS" suffix (e.g., LCL-G-MW-4-DIS).

2.2 Sampling Methods

Phase II involves sampling on-site and sampling off-site in both West Virginia and Ohio of various media including:

- ☐ air
- ☐ drinking water sources (private and public water sources)
- ☐ groundwater
- ☐ surface water
- ☐ soil
- ☐ biota (vegetation)

In addition, the groundwater-sampling program includes installing shallow monitoring wells at the LHWA well field in Ohio. DuPont will install four new shallow monitoring wells as part of this investigation, contingent upon receiving permission of the property owner.

2.2.1 Preliminary Activities

The following preliminary activities will be completed before sampling personnel enter the field to ensure proper preparation for each sampling event:

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- ☐ Coordination between sampling and laboratory personnel will be established so that sample integrity is retained at all times during the sampling event.
- ☐ The laboratory will be notified of each upcoming sampling event so it can prepare the list of parameters to be analyzed for each site, the replicate requirements, and the number of extra bottles needed, if any, for quality control testing will be specified to the laboratory manager.
- ☐ All proper field forms (i.e., field logbooks, custody seals, and COC forms) will be prepared for use to enable proper documentation of the sampling event.
- ☐ A preliminary inspection and calibration of all field equipment will be performed to ensure accurate measurements of field parameters (i.e., pH, specific conductance, dissolved oxygen, turbidity, and temperature).
- ☐ All field personnel will be trained in the sampling protocols contained herein.

The following steps will be performed before beginning each sampling event to ensure sampling is implemented correctly and safely:

- ☐ The sample location will be identified.
- ☐ All equipment to be used during the sampling event will be inspected, pre-cleaned, and decontaminated.
- ☐ Field meters to be used during sampling (i.e., pH and specific conductance meters) will be checked to ensure proper calibration and precision response. Buffer and standard solutions will be laboratory checked to ensure their accuracy.
- ☐ Pumps used for air sampling will be calibrated as described in Section 2.2.9.
- ☐ All forms to be used in the field (e.g., field logbook, and COC forms) will be assembled.
- ☐ Sampling personnel will review sampling protocols. In addition, health and safety protocols will be reviewed to help ensure that no injuries occur during the sampling event.
- ☐ The locations of underground utilities will be determined and marked, as appropriate. Washington Works excavation or other permits, if necessary, will be obtained.

2.2.2 General Instructions for All Sampling

Water sample bottles will not be pre-rinsed with site water prior to sample collection. Latex or nitrile gloves will be worn during sampling activities and replaced between samples. All samples, except for air sample collection (OVS) tubes, will be held chilled (temperature ranging from not frozen to 6°C) with wet ice from collection to shipping.

In order to minimize the possibility of introducing C-8 contamination into samples, the following protocol will be followed:

- ☐ Avoid fluoropolymers
- ☐ Avoid aluminum foil

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- ☐ Do not use Post-a-Notes
- ☐ Avoid blue ice
- ☐ Avoid pre-wrapped foods or snacks
- ☐ Wear clothing that has been washed at least six times.
- ☐ Use only containers supplied by contract laboratory.

The project geologist or a senior member of the field team will be responsible for sampling and laboratory coordination. The laboratory will provide necessary sample containers with the shipping containers (i.e., shuttles). Containers and any preservative added to the containers will be in accordance with EPA document SW-846 protocol. All samples requiring refrigeration will be shipped at approximately 4°C (temperature ranging from not frozen to 6°C).

Field equipment will consist of some or all of the following:

- ☐ Polyethylene (nalgene) collection bottles (laboratory provided)
- ☐ Field sampling record
- ☐ Sufficient ice to maintain the samples at approximately 4°C (temperature ranging from not frozen to 6°C)
- ☐ Methanol (VWR Scientific laboratory grade or equivalent) and deionized/distilled water
- ☐ Conductivity meter, pH meter, temperature probe, Redox probe, dissolved oxygen probe, turbidity meter, or multi-probe Sonde with a Flow-Thru cell
- ☐ Glass beakers
- ☐ PID and/or FID for organic vapor analysis
- ☐ Pumps and/or bailers for purging
- ☐ Rope
- ☐ Stainless-steel or polypropylene leader to attach rope to sampling device
- ☐ Non-fluoropolymer bailers or other sampling devices (preferably dedicated, pre-cleaned, or disposable)
- ☐ OSHA Versatile Sampling (OVS) tube with a 0.3 micron filter will be used (cat # 226-58 or equivalent, SKC, Inc., Eighty Four, Pennsylvania).
- ☐ Sample pumps such as the Universal PCXR4
- ☐ DC-LITE Primary Flowmeter, or equivalent
- ☐ Stainless-steel knife, trowels, spoons, scoops, and bowls
- ☐ Soil punch, or equivalent

Preparing for sampling includes acquiring all of the necessary monitoring equipment listed above and site-specific information to perform the required monitoring.

2.2.3 Soil Boring and Soil Sampling

The construction of four shallow wells at the LHWA well field will be directed by the DuPont contracted oversight geologist. Well construction details will be dictated by subsurface conditions encountered at each location. A roto sonic-drilling rig will be used to drill the wellbore and install the four shallow wells. The proposed locations for soil borings at the LHWA and shallow water monitoring wells are presented in Figure 5 of the Phase II Work Plan.

The sonic drilling method uses high frequency mechanical oscillations combined with rotation to advance a sampling core vertically downward into unconsolidated materials, such as those encountered in the LHWA well field. The technique is quick and generates minimal drill cuttings. The roto sonic-drilling rig will be used to collect a continuous soil core at each of the four well installation locations using a 4-inch diameter core sampler. Descriptive logs of the all soil cores will be logged by the DuPont contracted oversight geologist in the field using the Unified Soil Classification System (USCS). In addition, qualitative moisture content within the core samples will also be recorded. During drilling, vegetable oil and other non-petroleum-based products will be used as lubricating oils to minimize potential contamination of the borehole and/or well.

In all LHWA borings, soils from the following depths will be sampled:

- ☐ at the surface to a depth of six inches (a surface soil/biota sample)
- ☐ at 5-foot intervals below ground surface (bgs) to the top of the sand and gravel aquifer (approximately 30 feet below grade)

Surface soil sampling will be conducted in association with shallow well installation at LHWA and also during the synoptic surface soil/biota sampling events. Surface soil sample collection methods are discussed in Section 2.2.10 of this QAPP.

Subsurface soil samples will be transferred from the liner to a stainless steel mixing bowl for homogenization. Soil samples will be transferred to polyethylene collection bottles (supplied by the laboratory) using stainless steel spoons or trowels. A Chem-wipe™, or equivalent, moistened with methanol (VWR Scientific laboratory grade or equivalent) should be used to wipe the exterior of the sample collection bottle prior to sampling, and again after the filled bottle is capped.

Field equipment will consist of some or all of the following:

- ☐ Polyethylene (nalgene) collection bottles (laboratory provided)
- ☐ Plastic sheet liners
- ☐ Field sampling record and soil boring logs
- ☐ Stainless-steel knife, trowels, spoons, scoops, and bowls

Soil samples will be analyzed for C-8 using the analytical methodology discussed in Section 2.4 of this QAPP. Sampling devices used during the investigation (i. e. sample tubes, soil spoons, etc.) will be decontaminated as described in Section 2.2.12 of this QAPP after each use to avoid cross contamination within and between sample locations.

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2.2.4 Well Construction in the LHWA Well Field

Shallow monitoring wells will be constructed using Schedule 40, flush jointed four-inch polyvinyl chloride (PVC) plastic screen and riser materials. The screened unit of the monitoring well will be a five-foot length of factory-slotted, 10-slot (.010) PVC well screen. The riser material will also be constructed of Schedule 40, flush jointed four inch PVC. The suggested sandpack will be placed in the annulus of the borehole to two feet above the screened interval. Two feet of bentonite pellets will be placed above the sandpack and hydrated with water. The remainder of the borehole annulus will be tremie piped to the surface with a bentonite/grout mixture. All material placement will be checked with a measurement tape during installation. The monitoring wells will be completed with six-inch outer steel casing with a locking cap, a concrete pad, and traffic protection bollards. The setting of the well, lengths of pipe and placement of construction materials will be based on subsurface conditions encountered during the drilling of the boreholes and at the discretion of the DuPont contracted oversight geologist.

Proper decontamination (see Section 2.2.12 of this QAPP for these procedures) will be performed during this investigation to ensure against cross-contamination within and between sample locations. All well riser pipe and screen used for well construction will be certified cleaned and wrapped by the distributor. Prior to moving onto a new well installation location, the drill rig and associated equipment will be thoroughly cleaned at a decontamination pad using pressurized, supplied, water.

2.2.5 Well Development

Following installation, each new well will be thoroughly developed by pumping and/or bailing to remove fines from the screened interval. Once constructed, the new wells will be allowed to cure a minimum of twelve hours before being developed. All wells will be developed for a minimum of one hour and until at least ten well volumes have been purged and turbidity levels are noticeably decreased as determined by the DuPont oversight geologist. Well development procedures will be dictated by specific subsurface conditions at each of the four locations; well purging for development will not exceed 2 hours per well.

2.2.6 Groundwater Sampling (Wells)

Groundwater will be sampled from residential and monitoring wells, including the newly installed wells, and specified production wells. Procedures for sampling production wells are included in Section 2.2.7.

Prior to initiating sampling activities at a given location (e.g., landfill), a complete round of depth to water levels will be measured to the nearest one hundredth of a foot.

To the extent possible, monitoring wells will be purged and sampled beginning from the least suspected to most suspected contaminated well to minimize the potential for cross-contamination.

Prior to sampling, monitoring wells will be purged using low-flow methodology. All wells will be purged with low-flow type submersible and/or bladder pumps. The depth of

the purge pump intake will depend on well yields. The ideal intake for the pump is at the mid-section of the screened interval in the well and may be adjusted as the water level responds to pumping. Total well depths necessary to calculate the required pump placement will be tabulated and will accompany the sampling team in the field. If the screened interval is unknown, the pump will be placed approximately one foot from the bottom of the well.

During well purging, pH, specific conductance, turbidity, dissolved oxygen, salinity, oxidation/reduction potential, color, odor, and temperature will be measured. Purging will be considered complete when readings of pH, specific conductance, and temperature have stabilized to within 10 percent of the previous set of readings or when one hour of purging has been reached. Once purging is complete, the well will be sampled through the discharge tubing of the pump by directly filling the laboratory-supplied sample containers.

Sample containers will be filled to at least the container shoulder. After the sample containers are filled, they will be labeled appropriately and placed in a sample shuttle containing ice or ice packs. Samples requiring refrigeration for preservation will be stored at approximately 4°C (temperature ranging from not frozen to 6°C) during storage and shipment.

The following procedure will be followed during groundwater sampling:

1. Wipe the exterior of the sampling bottle using a Chem-wipe™, or equivalent, moistened with methanol (VWR Scientific laboratory grade or equivalent).
2. Remove the bottle cap, wipe the bottle lip using a Chem-wipe™, or equivalent, moistened with methanol (VWR Scientific laboratory grade or equivalent), and fill from the bailer or discharge tubing. Do not use a secondary container to fill the bottle.
3. Recap the sample bottle.
4. Wipe the bottle using a Chem-wipe™, or equivalent, moistened with methanol (VWR Scientific laboratory grade or equivalent) and affix a sample label, unless the label was affixed by the laboratory.
5. Place the sample in a cooler of ice.
6. Complete the COC form.

To ensure against cross-contamination between groundwater sampling locations, the sampler collecting the groundwater samples will wear clean, disposable latex or nitrile gloves and will limit his/her contact with the samples. When possible, laboratory-cleaned or disposable sampling equipment should be used (e.g., discharge tubing). Sample bottles and containers will be prepared by the contracted laboratory and will be sealed to ensure cleanliness. Sample bottles will not be cleaned or reused in the field. Monitoring instruments and sample pumps will be cleaned in accordance with Section 2.2.12 of this QAPP.

Groundwater samples will be analyzed for C-8 using the analytical methodology discussed in Section 2.4 of this QAPP.

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2.2.7 Groundwater Sampling (Taps)

Groundwater sampled from taps includes residential locations, finished water, and production wells. All taps will be flushed for two minutes prior to collecting the water sample. Purging before sampling is not required for production wells when the pumps are "in-service" prior to sampling. If production wells are not "in-service", the samplers will request that the production well be turned for a period of one hour prior to sampling the production well. After one hour has passed, the taps will be flushed for two minutes and then the water will be sampled. Samples from production wells will be collected from in-line sample ports within each well while the pumps are operating.

The following procedure will be followed during sampling from taps:

1. Locate an appropriate tap water source (prior to any treatment systems).
2. Wipe the tap water faucet or port and the exterior of the sampling bottle using a Chem-wipe™, or equivalent, moistened with methanol (VWR Scientific laboratory grade or equivalent).
3. Open the valve and allow water to run for at least two minutes to flush the valve system and supply lines.
4. Remove the bottle cap, wipe the bottle lip using a Chem-wipe™, or equivalent, moistened with methanol (VWR Scientific laboratory grade or equivalent), place the bottle under the tap, and fill. If the bottle will not fit under the tap faucet, then look for another appropriate source. Do not use a secondary container to fill the bottle.
5. Recap the sample bottle.
6. Wipe the bottle using a Chem-wipe™, or equivalent, moistened with methanol (VWR Scientific laboratory grade or equivalent) and affix a sample label, unless the label was affixed by the laboratory.
7. Place the sample in a cooler of ice.
8. Complete the COC form.

To ensure against cross-contamination between groundwater sampling locations, the sampler collecting the groundwater samples will wear clean, disposable latex and/or nitrile gloves and limit his/her contact with the samples. When possible, laboratory-cleaned or disposable sampling equipment should be used (e.g., discharge tubing). Sample bottles and containers will be prepared by the contracted laboratory and will be sealed to ensure cleanliness. Sample bottles will not be cleaned or reused in the field.

Groundwater samples will be analyzed for C-8 using the analytical methodology discussed in Section 2.4 of this QAPP.

2.2.8 Surface Water Sampling of Springs and Cisterns

Surface water sampling includes sampling of springs and cisterns. The following procedure will be followed during surface water sampling:

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1. Wipe the exterior of the sampling bottle using a Chem-wipe™, or equivalent, or equivalent, moistened with methanol (VWR Scientific laboratory grade or equivalent).
2. Submerge the sample bottle below the water surface and unscrew the bottle cap.
3. Fill the water bottle by turning the bottle parallel to the water surface and slowly rotating so that the mouth of the bottle is up right. This procedure will ensure that water from the surface microlayer is not pulled into the sample bottle.
4. Recap the sample bottle under water.
5. Wipe the bottle using a Chem-wipe™, or equivalent, or equivalent, moistened with methanol (VWR Scientific laboratory grade or equivalent) and affix a sample label, unless the label was affixed by the laboratory.
6. Place the sample in a cooler of ice.
7. Complete the COC form.

Alternatively, a decontaminated or disposable bailer may be used to collect samples from cisterns.

To ensure against cross-contamination between surface water sampling locations, the sampler collecting the surface water samples will wear clean, disposable latex and/or nitrile gloves and limit his/her contact with the samples. When possible, laboratory-cleaned or disposable sampling equipment should be used (e.g., bailers). Sample bottles and containers will be prepared by the contracted laboratory and will be sealed to ensure cleanliness. Sample bottles will not be cleaned or reused in the field.

Surface water samples will be analyzed for C-8 using the analytical methodology discussed in Section 2.4 of this QAPP.

2.2.9 Air Modeling Verification Study Sampling

Phase II ambient air sampling will be conducted at nine locations, seven in Ohio and two in West Virginia. These air sampling locations are shown in Figure 1 and listed in Table 1 of the Phase II work plan. Sampling locations were selected to represent a complete range (i.e. radius) of wind directions from the Washington Works facility. In addition, some sampling locations were selected at different distances from the Washington Works Facility, but in the same wind direction, to help evaluate gradients. Seven sampling events are scheduled over a six-week period. Sampling duration will be 24 hours per event. A blank air sampling tube will be submitted with each sampling event.

The target compound for this monitoring program is PFOA, the free acid form of C-8. For collection and analysis of PFOA, the OSHA Versatile Sampling (OVS) tube with a 0.3 micron filter will be used (cat # 226-58 or equivalent, SKC, Inc., Eighty Four, Pennsylvania). This tube is uniquely designed to simultaneously capture aerosols and vapors. The OVS tube contains a plastic retaining ring and glass fiber frits as fraction A. The first section of XAD resin beads and the first PUF filter are fraction B. The second section of XAD resin beads and the second PUF filter are fraction C.

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Pumps will be required at each sampling location to provide airflow through sample tubes. Sample pumps such as the Universal PCXR4, capable of 500 mL to 1500 mL/minute flowrate will be specified. The samplers will be set to run at approximately 1 liter per minute for a total volume of 1.44 cubic meters over the 24-hour sampling period. The actual pump flow rate will be verified using a DC-Lite primary flowmeter (available through SKC, Inc.). This "dry" calibration utilizes a flow cell volume and crystal timing mechanism which is certified NIST-traceable. Three readings are taken at the start of sampling and averaged for an initial pump rate. Following the sampling period a final calibration is performed to verify the pump rate. The final pump rate must be within 10% of the initial pump rate. Sampling equipment will be mounted on poles and covered as needed to provide protection from weather and other potential damage. Power will be supplied to each location via AC power or battery supplied power for remote locations. Field personnel will be trained by professional industrial hygienists familiar with OVS equipment. Field logs will be provided to record pertinent information during sampling events. (See Appendix C for sample log sheet.) Sample tubes (OVS) will be provided by the contracted laboratory and should be stored in a clean, dry location. Sample tubes and collected samples are not required to be chilled, however sample tubes and collected samples should not be subjected to temperature extremes.

The following procedure will be followed during sample collection:

1. 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.
2. Following sample collection, the OVS tubes are capped and placed in a Zip-loc™, or equivalent, bag. Seal the Zip-loc™, or equivalent, bag.
3. Complete the sample label and COC form, including the volume of air sampled through the tube. The completed sample label is attached to the outside of the Zip-loc™ bag, or equivalent.
4. Pack the samples in a small box or cooler with bubble wrap. Include the completed COC form.
5. Seal the box or cooler with at least one (1) custody seal.
6. Complete a mailing label and airbill and ship by overnight carrier.
7. Samples should be shipped to the laboratory same day as collection, and will be received at the laboratory within 72 hours of collection.

Ambient air samples will be analyzed as described in Section 2.4 of this QAPP.

2.2.10 Meteorological Measurements

Meteorological measurements will be taken to support air modeling activities during each sampling event using an on-site meteorological station. The use of an on-site meteorological station ensures the data are representative of conditions at the perimeter of the site.

Data collected during each 24-hour sampling event will include:

- ☐ Hourly average wind speed

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- ☐ Hourly average wind direction
- ☐ Hourly average ambient air temperature
- ☐ Calculation of stability class

2.2.11 Surface Soil/Biota (Vegetation) Sampling

For the surface soil/biota sampling in Ohio and West Virginia, DuPont proposes conducting two synoptic sampling events, one following a "wet" period and one following a "dry" period. The objective of sampling following a "wet" and "dry" period is to evaluate if precipitation has an effect on the concentration of PFOA measured. Meteorological data will be monitored prior to and during the "wet" and "dry" sampling events.

The "wet" period will be defined using the USEPA stormwater sampling requirements (40CFR122.21(g)(7)). A "wet" period or acceptable storm to collect surface water samples is defined by the USEPA as a 72 hour period with less than 0.1 inches of rainfall followed by a rainfall event that is forecasted not to vary more than 50 percent of the average or median event for the sampling area. Sampling locations in Ohio and West Virginia are located in the Northeast Rain Zone (USEPA July 1992, exhibit 2.8); the representative storm or "wet" event will range from 5.6 to 16.8 hours in duration and will include between 0.25 and 0.75 inches of rainfall. Soil and biota samples will be collected as soon as possible, but not to exceed 24 hours after the acceptable storm event. The "dry" sampling event will be defined in this investigation as five consecutive days with less than 0.1 inches of rainfall. Soil and biota samples will be collected within 24 hours of the acceptable "dry" period.

Meteorological data is available from a weather station located at the Washington Works Facility and from the Parkersburg Airport, in Parkersburg, WV. Meteorological data from these two sources will be obtained covering the time period between the "wet" period prior to the sampling event and the "wet" sampling event and covering the time period between the "wet" period prior to the sampling event and the "dry" event.

At each sampling location, two samples will be collected immediately adjacent to each other during each sampling event. Each sample will consist of a six-inch core of vegetation and soil. The first sample will be submitted for laboratory analysis and the second sample, which will be a larger diameter sample, will be used to determine the soil classification at the location.

The first sample will be divided in the field into two portions, the organic-rich portion (vegetation and litter layer) and the mineral matter-rich portion (roots, root zone and the material below the root zone). The objective of dividing the sample into a biota (organic-rich) portion and soil (mineral matter-rich) portion is to evaluate if the PFOA is detectable in both portions and to compare the PFOA concentration between the organic-rich portion and the mineral matter-rich portion.

The following procedure will be followed during surface soil/biota sample collection:

1. 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.

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2. The soil punch, or equivalent, will be pushed or driven into the ground to a depth of six inches and then will be pulled back out of the ground.
3. The surface soil/biota sample will be removed from the soil punch, or equivalent.
4. The surface soil/biota sample will be divided in the field into the two portions, the organic-rich portion and the mineral matter-rich portion.
5. Each soil/biota sample portion will be placed separately in a Zip-loc™ bag, or equivalent.
6. The sample label and COC form will be completed.
7. The completed sample label is attached to the outside of the Zip-loc™ bag, or equivalent, and placed in a sample shuttle containing ice or ice packs.
8. Samples will be kept at approximately 4°C (temperature ranging from not frozen to 6°C) during storage and shipment to the laboratory.

Sampling devices used during the investigation (i. e. soil punches, etc.) will be decontaminated as described in Section 2.2.12 of this QAPP after each use to avoid cross contamination within and between sample locations.

For the samples collected during the "dry" event, the organic-rich portion will be washed with deionized water, prior to processing the soil samples for analysis, to differentiate between adsorbed (sticking) or "external" PFOA removed by the deionized wash water and PFOA incorporated within the organic material itself. The washed organic-rich portion and the wash water will be analyzed for PFOA. The mineral-rich portion of select samples collected during the "dry" event will also be washed for comparison. For these samples the washed mineral-matter rich portion and the wash water will be analyzed for PFOA.

The deionized water wash procedure is as follows:

1. Place a 10 mg sample portion (weighed to 0.1 mg) into a 250 mL polypropylene centrifuge tube.
2. Add 100 mL of deionized or distilled or liquid chromatographic grade water.
3. Place on wrist action shaker for 30 minutes.
4. Centrifuge, and decant.

The deionized water wash procedure described above will be performed by the laboratory. The soil/biota sample fractions and wash water will be analyzed as described in Section 2.4 for PFOA.

The second, larger diameter sample will be used for soil classification according to ASTM D2487. Soil classification will be based on the results of grain size determination and Atterburg Limits, and may include results of specific gravity, organic content and moisture content.

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2.2.12 Surveying

All newly installed monitoring wells, each soil/biota sampling location, and each air sampling location will be surveyed by a State of Ohio or State of West Virginia licensed surveyor, as appropriate. Elevations will be surveyed to the nearest 0.01-foot using the nearest convenient permanent surveyed benchmark for each location. During surveying, the following data (as appropriate) will be measured:

- ☐ horizontal locations (i.e., Northings and Eastings)
- ☐ the elevation of the ground surface
- ☐ the elevation of the top of the PVC casing (for wells)

2.2.13 Decontamination

All equipment in direct contact with the material to be sampled will be decontaminated prior to sampling to prevent cross-contamination of samples collected. In addition, care will be taken so as not to allow anything to come into contact with a sample or sample area, which may affect its composition.

Sampling equipment may include, but is not limited to, spoons, bowls, bailers, tubing, and pumps. All of these items will come in direct contact with the sample and have potential to impact analytical results. Therefore, care will be taken to ensure the cleanliness of all sampling equipment. When possible, pre-cleaned or disposable sampling equipment will be used (e.g., dedicated bailers or polyethylene tubing for sampling wells). Field decontamination will be permitted for, provided the following method is applied:

- ☐ Wipe off any residual sludge or water with a Chem-wipe™, or equivalent,, or equivalent.,
- ☐ Rinse the equipment with deionized water.
- ☐ Rinse the equipment with methanol (VWR Scientific laboratory grade or equivalent).
- ☐ Place in zip-sealed bag until next use

Where pumps are used and are not dedicated, the pumps will be cleaned and flushed prior to and between each use. The pumps will get an external, tap water rinse, followed by a 2-gallon flush of potable water through the pump. This must be followed by a deionized water rinse of the outside of the pump.

In addition to the decontamination procedures outlined above, the person collecting the sample will wear clean latex or nitrile gloves and will limit his/her contact with the samples.

Sample bottles and containers will be prepared by the contracted laboratory and will be sealed to ensure cleanliness. Sample bottles will not be cleaned in the field.

A personnel decontamination area will be set up at each sample location prior to starting sampling activities. Procedures for the decontamination of protective equipment and the

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removal of respiratory and personal protection clothing to avoid transfer of constituents from clothing to the body are discussed in the HASP.

To the extent that it is economically feasible and technically acceptable, disposable personal protective equipment (PPE) will be used. Where the work scope restricts use of disposable PPE, decontamination facilities will be provided.

2.2.14 Waste Management

All disposable equipment and other materials that are not decontaminated for reuse will be disposed of in an acceptable manner. All waste generated during the investigation will be disposed of off-site in accordance with the Project-specific Waste Management Procedure for Letart Landfill, Local Landfill, Dry Run Landfill Washington Works Plant and Designated Off-site Areas (November 2001) and Project-specific Waste Management Plan for Washington Works Consent Order Sampling, Little Hocking Water Association Investigation, Porterfield, Ohio (May 2002).

2.3 Sampling Handling and Custody

Sample chain-of-custody will be initiated in most cases by the laboratory with the selection and preparation of sample containers. To reduce the chance for error, the number of personnel assuming custody of the samples and sample containers will be held to a minimum.

On-site monitoring and sampling data will be controlled and entered onto appropriate records. Personnel involved in the chain-of-custody and transfer of samples will be trained on the procedures and their importance and purpose prior to sampling initiation.

2.3.1 Chain-of-Custody

Sample custody procedures are summarized below. COC procedures are intended to maintain and permanently document sample possession from the time of collection to disposal, in accordance with EPA guidelines. A sample is considered to be under a person's custody if:

- ☐ it is in that person's possession;
- ☐ it is in that person's view, after being in that person's possession;
- ☐ it was in that person's possession and was locked up by them to prevent tampering; or
- ☐ it has been placed in a designated secure area by that person.

2.3.2 Field Chain-of-Custody

A COC form will accompany the sample container from the initial sample container selection and preparation at the laboratory to sample collection and preservation in the field to the return of the samples to the laboratory. The COC form will trace the path of each individual sample container by means of a unique identification number. When

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requested, sample designation/location numbers will be pre-printed by the laboratory on the COC form and bottle labels.

The project manager or field team leader will notify the laboratory of upcoming field sampling activities and the subsequent transfer of samples to the laboratory. This notification will include information concerning the number and type of samples to be shipped as well as the anticipated date of arrival. Sample shipping containers (i.e., shuttles or coolers) will be provided by the laboratory. The shipping containers will be insulated. A sample container partially filled with water will be included in each shuttle to serve as a temperature blank. All sample bottles within each shipping container will be individually controlled and labeled. Sample identification labels will be provided by the laboratory. All sample bottle labels will include the following information:

- ☐ Site name
- ☐ Sample number
- ☐ Analysis required
- ☐ Preservatives

Personnel receiving the sample containers will verify the integrity of the seals on each cooler. Shuttles with broken seals will be returned to the laboratory with the contents unused, assuming the cooler is intact. The receiving personnel will break the seal, inspect the contents for breakage, and sign the COC form to certify receipt of the sample containers. A temporary seal then will be affixed to each cooler.

Once sample containers are filled, they will be placed immediately in the cooler on ice to maintain the samples at approximately 4°C (temperature ranging from not frozen to 6°C). The field sampler will indicate the sample designation/location number in the space provided on the COC form for each sample, unless COC forms are preprinted. Date and time of sample collection will be entered by the field sampler. The COC forms will be signed and placed in the cooler. The samples should be shipped to the laboratory on the same day as they were collected and will be delivered to the laboratory no later than 72 hours after sample collection. The cooler with samples will be shipped to the laboratory using an overnight express service.

The "remarks" column of the COC form will be used to record specific considerations associated with sample acquisition such as sample type, container type, sample preservation methods, analysis to be performed, and the possible need to dilute the sample due to indication of high levels of contamination. The source of reagents, field blank water, and supplies will be documented on the COC form or the field notebook. The laboratory will maintain a file of the completed original forms. Copies will be submitted as part of the final analytical report. If samples are split and sent to different laboratories, each sample will receive a unique COC form.

Examples of sample labels and COC forms are included in Figures 3 and 4.

2.3.3 Laboratory Chain-of-Custody

Receiving, storing, and tracking samples submitted to the laboratory will be conducted according to laboratory SOPs intended to ensure that invalid laboratory data resulting

from sample contamination, loss, deterioration, or tampering has not occurred. Internal laboratory COC procedures will follow laboratory SOPs.

2.4 Analytical Methods

Phase II investigation multi-media samples collected at and near the Washington Works Site will be analyzed by a DuPont-approved laboratory. PFOA is the entity detected by the analytical instrument for all media. Aqueous and soil samples will be analyzed for PFOA and reported for C-8. This is a mathematical conversion accomplished by multiplying the PFOA result times the ratio of the molecular weight of C-8 over the molecular weight of PFOA. Air and biota samples will be analyzed and reported as PFOA.

Chemical parameters, sample containers, holding times, and preservatives are presented in Table 2. The analytical procedure used by the laboratory will be consistent with applicable laboratory SOPs as specified in Table 3.

Quantitative reporting threshold limits are sample dependent and may vary as the sample matrix varies. Factors influencing the threshold limits include sample matrix, interferences, and high concentrations of analytes. The LOQ to be reported for each media was determined based on an evaluation of the concentration of interest and laboratory background concentrations and is intended to enable the data user to make technically correct decisions for the purposes of the Phase II investigation. The actual LOQs may vary from sample to sample in accordance with standard laboratory practices (e.g., dilution resulting from high analyte concentration).

2.4.1 Aqueous Samples

C-8, or ammonium perfluorooctanoate (APFO) forms perfluorooctanoic acid (PFOA) in aqueous solution. PFOA is extracted from water using C₁₈ solid phase extraction (SPE) cartridges. PFOA is eluted from the SPE cartridge using methanol, resulting in an 8:1 concentration of the sample. Analysis is performed by LC/MS/MS using selected reaction monitoring (SRM). Quantitation is accomplished via external standard calibration. Levels of PFOA found are mathematically converted and reported as APFO.

Additional details concerning the analytical method for aqueous samples are found in EPA public docket OPPT-2003-0012-0040.

2.4.2 Soil Samples

Soil samples are mixed with methanol and sonicated. The methanol extracts are centrifuged and filtered. The methanol extracts are analyzed by LC/MS/MS. PFOA is separated from other components on an HPLC C₁₈ column with a mobile phase mixture of water containing 0.1% formic acid and methanol. The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode. The instrument is calibrated at 6 concentration levels. The parent ion for PFOA is 413 amu. The daughter ions used for analysis by LC/MS/MS are at 369 and 219 amu. Sample results are quantitated using isotope dilution. The isotope dilution technique allows correction for analytical bias encountered when analyzing more chemically complex environmental samples. A

known quantity of the labeled compound PFOA-di-13C is added to every sample and to the batch quality control samples prior to extraction. Because the isotopically labeled compound is chemically identical to the compound of concern, it is affected by any interfering substances in the sample to the same extent as the compound of concern. The recovery of the PFOA-di-13C is then used to mathematically correct the final result for the APFO.

Additional details concerning the analytical method for soil samples are found in EPA public docket OPPT-2003-0012-xxxx.

2.4.3 Biota Samples

Method development has been initiated for the biota samples. It is anticipated that samples will be homogenized in the laboratory using a Hobart chopper or the equivalent, so that a uniform subsample may be obtained. Analysis of a prepared extract will be performed using LC/MS/MS. Biota samples will be analyzed and reported for PFOA.

Additional details concerning the analytical method for biota samples will be submitted to EPA public docket OPPT-2003-0012-xxxx.

2.4.4 Air Samples

PFOA is extracted from each section of an OVS air tube with methanol. The plastic retaining ring and glass fiber frits are fraction A. The first section of XAD resin beads and the first PUF filter are fraction B. The second section of XAD resin beads and the second PUF filter are fraction C. Contents of sample tubes will be analyzed separately for vapor and particles greater than 0.3 microns, then composited for a total concentration at each sampling location. Analysis is performed by liquid chromatography/mass spectrometry (LC/MS) using single ion monitoring (SIM). Quantitation is accomplished via external standard calibration. Levels of PFOA found are reported for air samples.

Additional details concerning the analytical method for air samples are found in EPA public docket OPPT-2003-0012-317 and OPPT-2003-0012-318.

2.5 Quality Control

As part of the QA program, samples will be collected and prepared in the field and laboratory to provide control over the collection of environmental measurements and subsequent review, interpretation, and validation of generated analytical data. Two types of field QC samples will be prepared or collected: field (e.g., equipment rinsate) blanks and field duplicate samples. The two types of QA samples are discussed in more detail below. Split samples may also be collected by third parties, as discussed below. Laboratory QC samples included are also discussed below.

2.5.1 Field Blanks

Field blanks will be collected during sampling of solid and aqueous media. These blanks will provide a check on possible sources of contamination such as sample bottle preparation, blank water quality, and sample handling. Field blanks also will be used to

indicate potential contamination from ambient air or sampling equipment used to collect and transfer samples.

Field blanks will be collected using two identical sets of pre-cleaned sample containers. One set of containers will be empty and will serve as the sample containers to analyze. The second set of containers will be filled at the laboratory with laboratory-demonstrated analyte-free water. The water will originate from one common source and physical location within the laboratory and will be the same water as the method blank water used by the laboratory. Field blanks will be handled, transported, and analyzed in the same manner as the samples acquired that day. After sampling at the suspected most impacted field location, analyte-free water will be rinsed over decontaminated sampling equipment and placed in the empty sample container for analysis (it may be necessary for the laboratory to provide extra water to ensure sufficient volume of blank water to eliminate headspace). The rationale for collecting equipment rinsate samples at the suspected most impacted area is to simulate a worst-case scenario regarding sampling equipment cross-contamination and ambient air contributions to sample contamination. When there is no sampling equipment to be rinsed, as for tap water sampling, a bottle to bottle transfer of the blank water will take place in order to collect a field blank sample. Field blanks should return to the laboratory with the same set of sample bottles they accompanied to the field.

Field blanks for air samples will consist of an OVS tube shipped from the laboratory to the field, and returned unopened to the laboratory from the field. Field blank OVS tubes should not be held on-site for more than sixty days after the sampling event.

Other guidelines for the use and integrity of field blanks include:

- ☐ Field blank water samples should not be held on-site for more than four days after the sampling event.
- ☐ The clock governing holding times for the field blank will begin at the time of sample collection when analyzed using EPA document SW-846 protocol.
- ☐ Field blanks will be collected and analyzed at a rate of at least one per day and at a rate of one in 20 (minimum of one per day) samples. Field blanks will be analyzed for the same parameters for which the environmental samples collected that day are analyzed.

2.5.2 Field Duplicate Samples

Collecting duplicate samples allows the evaluation of the laboratory's performance by comparing the analytical results of two samples from the same location. Duplicates will be collected for air and aqueous samples. Although moisture content, particle size, and adsorption properties of various soils inhibit the ability to achieve replicability, soil duplicate samples will also be collected. Duplicates will be collected for all media at a rate of 5 percent, or one per sampling event.

Duplicates of solid and aqueous samples will be obtained by alternately filling sample containers from the sample source. Sample locations for duplicate soil samples will be selected to ensure collection of representative samples and sufficient sample volume to fulfill all QA/QC protocols.

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Duplicates of air samples will be obtained as co-located air tube samples. Procedures for collecting air, water, and solid samples are provided in Section 2.2 of this QAPP.

2.5.3 Split Samples

To maintain the integrity of any samples split with other parties, primary contractor personnel and sampling equipment will be used to collect all samples. Other parties, if any, will provide their own equipment (e.g., sample containers, blank containers, preservatives, COC forms) and should adhere to the protocols outlined in this QAPP.

2.5.4 Laboratory Blank Samples

Analytical results for laboratory blanks provide a means to evaluate laboratory precision and bias, and other potential contamination and carry-over problems. Laboratory blanks are carried through applicable sample preparation and analysis procedures. Laboratory blanks are analyzed for PFOA and must be less than the LOQ.

2.5.5 Laboratory Control Samples

LCS are analyte-free water or solid matrix that is spiked with PFOA at the level or levels specified in the laboratory SOP. The LCS is carried through the entire sample preparation and analysis procedures. LCS results are used to assess method/laboratory accuracy. The LCS is not the continuing calibration verification or check standard analysis.

2.5.6 Laboratory Replicate

Laboratory duplicates, or replicates, are generated in the laboratory and analyzed to assess method/laboratory precision in the matrix of concern.

2.5.7 Matrix Spikes

Matrix spikes provide information about the effect of the sample matrix on the preparation and measurement methodology. MS samples are spiked and analyzed by the laboratory to facilitate identification of effects of the particular matrix of interest on analytical results, particularly biasing of results. Sufficient sample volume will be collected for at least one sample in each batch of 20 or fewer field samples so that MS samples can be prepared for analysis. MS samples are spiked with PFOA at levels comparable to the level found in the unspiked sample, up to a maximum as specified in the laboratory SOP.

2.5.8 Surrogates

Surrogate recovery data are used to evaluate the precision of the analytical method on a sample specific basis. Surrogates are compounds similar to the target analytes in chemical composition and behavior but are not normally found in environmental samples. A surrogate standard is not employed for aqueous sample analysis. Perfluorononanoic acid is the surrogate standard for solid sample analysis. Nonadecafluorodecanoic acid is

the surrogate standard specified for air sample analysis. Surrogates are spiked into all field samples and laboratory quality control samples.

2.5.9 Internal Standards

Internal standard areas are used to correct sample results affected by losses in the analytical system. Internal standards are measured amounts of compounds not normally found in environmental samples. The labeled compound PFOA-di-13C is added to all soil field samples and laboratory quality control samples for the analysis of PFOA.

2.6 Instrument/Equipment Testing, Inspection, and Maintenance

The purpose of this element is to specify procedures used to verify that instruments and equipment are maintained in sound operating condition and are capable of acceptable performance. A program will be implemented to ensure that routine calibration and maintenance is performed on all field instruments.

2.6.1 Instrument/Equipment Testing and Inspection

A calibration program will be implemented to ensure that routine calibration and maintenance is performed on all field instruments. Field team members familiar with field calibrations and equipment operations will maintain instrument proficiency by performing the prescribed calibration procedures outlined in the operation and field manuals accompanying the field monitoring instruments. Air monitoring instruments (e.g., PID) used in the field to collect data for sample screening and health and safety purposes will be calibrated each day prior to the initiation of fieldwork using the manufacturer's instructions. The instruments will be calibrated using appropriate zero and indicator gases.

The pH, conductivity, and temperature meters will be calibrated prior to each day's use according to the manufacturer's instructions. More frequent calibrations will be performed as necessary to maintain analytical integrity. The pH meter will be calibrated at a minimum of two values that bracket the anticipated pH values of the samples to be analyzed and that are three pH units or more apart. The conductivity meter will be calibrated using a standard solution of known conductivity.

Following calibration, each instrument will be tagged identifying the person who calibrated the instrument and the calibration date. Calibration records for each field instrument used during the investigation will be maintained, and copies of the records will be stored in the project QA files. An example form record (for the PID) is presented as Figure 4.

Calibration of the liquid chromatograph-tandem mass spectrometer (LC/MS/MS) and other analytical equipment will be performed according to laboratory SOPs. Mass calibration checks meeting criteria must be performed. The correlation coefficient (R) for calibration curves generated for C-8 analysis must be ≥ 0.992 ($R^2 \geq 0.985$). If calibration results do not meet the criteria listed in the SOP, then appropriate steps must be taken to adjust instrument operation, and the standards or the relevant set of samples should be reanalyzed.

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2.6.2 Instrument/Equipment Maintenance

All field equipment will be subjected to a routine maintenance program, before and after each use. The routine maintenance program for each piece of equipment will be in accordance with the manufacturer's operations and maintenance manual. All equipment will be cleaned and checked for integrity after each use. Repairs will be performed immediately after any defects are observed and before the item of equipment is used again. Equipment parts with a limited life (e.g., such as batteries, membranes, and some electronic components) will be periodically checked and replaced or recharged as necessary according to the manufacturer's specifications.

Each piece of field equipment will have its own log sheet that contains the equipment identification number, information on maintenance procedures, and the date and nature of the last maintenance. Since most equipment will be used on an irregular, as-needed basis, all equipment will be properly stored when not in use.

Laboratory equipment maintenance will be regularly performed by the subcontracted laboratory. It will be the laboratory's responsibility to maintain and document the maintenance of properly functioning equipment so that the data is usable and reproducible. Upon request, a description of the laboratory's equipment, maintenance procedures will be provided by the subcontracted laboratory.

2.7 Instrument Calibration and Frequency

The purpose of this element is to define calibration procedures that will be used to generate environmental measurements. Specific requirements for calibration of analytical instruments are described in the field and laboratory SOPs.

2.8 Inspection/Acceptance Requirements for Supplies and Consumables

The purpose of this element is to establish and document a system for inspecting and accepting supplies and consumables that may affect the quality of the project data. Laboratory or field consumables or supplies that will come into contact with samples must be documented to be free of contamination prior to use. Examples of consumables and supplies include latex or nitrile gloves, glassware, soap or detergent, sample containers, reagents, and reagent water. Field consumables and supplies are also demonstrated to be free of contamination through the collection of equipment blanks. Laboratory consumables and supplies are demonstrated to be free of contamination through the preparation and analysis of laboratory blanks. The laboratory Quality Assurance Manual should identify critical supplies, such as calibration gases or standards, solvents or reagents, and the acceptance criteria for these supplies.

2.9 Data Acquisition Requirements

This element describes the types of non-measurement data needed for project implementation or decision-making. Non-measurement data may include computer

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databases, programs, literature files, and historical databases. This type of non-measurement data is not required for this phase of the project.

2.10 Data Management

This element describes the project data management process, including standard record-keeping procedures, and data storage and retrieval from electronic media.

2.10.1 Logbooks and Forms

Laboratory and field records shall be maintained by appropriate personnel and shall be sufficiently detailed to allow for reconstruction of the collection, handling, preparation, and analysis procedures performed on the samples. These procedures shall be documented in logbooks or on forms. It is sufficient to identify the SOPs and record any deviation from the SOPs in the logbooks or on the forms. Logbook pages and forms shall be initialed and dated by the person making the entry. Entries shall be legible. If errors are made, the error is crossed out with a single line, initialed, and dated by the person making the correction. Maintenance and calibration records must be traceable to the person using the instrument and to the specific instrument.

2.10.2 Data Storage and Retrieval

Field records and laboratory records shall be archived for a minimum of ten years from the date the record was generated. Software and hardware used to generate, store, and retrieve the records shall be kept on file with the records.

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3.0 ASSESSMENT AND OVERSIGHT

Internal audits and assessments will be performed by the organization primarily responsible for conducting the task being audited. For example, DuPont CRG may conduct an assessment of field sample collection activities and the contract laboratory will perform internal audits.

A technical systems audit of field activities is an on-site, qualitative review of the sampling system to ensure that the activity is being performed in compliance with this QAPP. A technical systems audit of field sampling activities is not planned for this phase of the project.

The contract laboratories will be accredited by the state agencies and others, as appropriate. A technical systems audit of the laboratory is not planned for this phase of the project.

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4.0 DATA VALIDATION AND USABILITY

The elements of this group address the QA activities that occur after the data collection phase of the project is completed. Implementation of these elements determines if the data conform to the specified criteria and satisfy the project objectives.

4.1 Data Review, Verification and Validation

The review performed on the data at each level shall be documented, beginning with the laboratory's review of the analytical results through the independent data review performed by the data user, and finally review by the appropriate state or other agency. The intent is to capture the review effort of each party to minimize duplicative efforts, to ensure that critical elements of the review process are not overlooked, and to set in place a system that can be audited or inspected.

In addition, air data modeling activities are discussed below.

4.1.1 Laboratory Review

The laboratory utilized for this project shall have implemented a quality assurance program that meets the requirements of a recognized organization such as an appropriate state agency, National Environmental Laboratory Accreditation Program (NELAP), or International Organization for Standardization (ISO). The laboratory shall review the data for technical acceptance.

The laboratory analyst is responsible for the reduction of raw data and shall clearly identify any problems or anomalies that might affect the quality of the data. The analyst shall review 100 percent of the data and shall verify that data reduction protocols are correct. At least 10% of the data shall be reviewed independently by a senior analyst or by the supervisor of the laboratory analyst. Both the analyst and independent review shall include:

- ☐ Calibrations and calibration verifications,
- ☐ Instrument and system performance checks,
- ☐ Blanks,
- ☐ LCS recoveries and precision,
- ☐ MS/MSD recoveries and precision,
- ☐ Duplicate sample precision,
- ☐ Compound identification and quantification,
- ☐ Surrogate recoveries, if applicable,
- ☐ Internal standard areas, if applicable,
- ☐ Serial dilutions, if applicable,
- ☐ Interference check sample results, if applicable, and

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- ☐ Post-digestion spike recoveries, if applicable.

The laboratory QA personnel shall review the completed data packages and perform a reasonableness check review on the completed data packages. The QA personnel shall ensure that all deliverables are present, that qualifiers have been applied to the data and that nonconformance and other issues have been address in the case narrative. Either the independent reviewer or the laboratory QA personnel shall perform a QA check on 100% of hand-entered data and 5% of electronically transferred data.

4.1.2 Data Reporting

The laboratory will report PFOA and/or C-8 results to three significant figures. Results will be reported to the LOQ. Reported concentrations will not be corrected for contaminants found in associated method and field blanks. Deliverables will include a narrative and appropriate laboratory raw data and QC summary forms.

The laboratory will report results for a laboratory replicate of each soil or aqueous field sample, or a minimum of one per batch. These results will be evaluated for precision by comparing the field sample result to the corresponding laboratory replicate result:

- ☐ If both results are less than the LOQ, the replicate sample for that analyte is considered to have passed the precision criteria.
- ☐ If one or both results is between one and five times the LOQ, the replicate is considered to have met the criteria if the two results differ by less than the LOQ.
- ☐ If one result is less than the LOQ and the other is not, and if the two results differed by a value less than the LOQ, the replicate is said to have met the acceptance criteria.
- ☐ Finally, if both results are at least five times the LOQ, the replicate is considered to have met the criteria if the relative percent difference (RPD) between the two results is less than or equal to 20%. RPD is the absolute value of the difference of two measurements divided by their average.

When the precision criteria outlined above is met, DuPont will report the average of the field sample and lab replicate results reported by the laboratory. If criteria for precision is exceeded, the higher of the sample and lab replicate results will be reported by DuPont. Finally, when one result (from the sample/lab replicate pair) is above the LOQ and one below, the result which is above the LOQ will be reported. C-8 results for soil and aqueous samples will be recorded in the Corporate Environmental Database (formerly CED, now Envista) and reported as FC-143 for consistency with historical results. PFOA results for air and biota samples will be recorded in Envista and reported as PFOA.

4.1.3 Phase II Air Modeling Activity

The Emission Inventory includes Source Characterization and Methods for Estimating Emissions.

Source Characterization

The following information will be collected and used as input into air dispersion modeling:

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- Stack locations
- Stack heights
- Stack diameters
- Stack gas exit temperatures
- Stack gas flow rates or exit velocities
- Emission rates

Methods for Estimating Emissions

Site-specific operational data will be used to relate production data to emissions levels using a factor. Through a combination of actual stack monitoring (for vents with highest emissions) and mass balance estimates (for vents with very small emissions), a factor has been established for each PFOA emission point to estimate the quantity of emissions released at a given production rate. This factor will be combined with production data to calculate PFOA emissions during each 24-hour sampling event.

Meteorological Data

On-site meteorological data will be collected during each sampling event, as described in a previous section. Concurrent twice-daily upper air observations will be collected from the upper air station located in Wilmington, OH in order to compute hourly mixing depth. Both sets of data will be combined and preprocessed by Trinity consultants of Dallas, Texas, to be used as input to the air dispersion model. Missing data and measured wind speeds of less than 1.0 m/s will be treated consistent with the recommendations in EPA's "Meteorological Monitoring Guidance for Regulatory Model Applications". An anemometer height of approximately 9.14 meters will be used for modeling analysis.

Model Selection and Methodology

The Industrial Source Complex Short Term Model (ISCST3), version 02035 will be used to conduct air dispersion modeling. All modeling will be performed in accordance with the procedures in EPA's Guideline on Air Quality Models (40 CFR Part 51, Appendix W). All model options will be set to the U.S. EPA default version of ISCST3.

PFOA emission sources will be evaluated for downwash effects from surrounding buildings. EPA's Building Profile and Input Program (BPIP) will be used to provide wind direction specific building parameters. All buildings on site will be evaluated to determine if they could potentially impact the stack by causing building downwash effects.

The area surrounding Washington Works is primarily non-urban. The U.S. EPA procedures classify land use within 3 kilometers of the site by the Auer method. Previous review of the U.S. Geological Survey (USGS) maps, aerial photographs, and site visits indicate the area is well over 50% non-urban. The Washington Works facility is located within the Ohio River valley, and is surrounded by significant terrain features on both sides of the river valley. As a result, terrain elevations will be considered in the modeling analysis.

An averaging time of 24-hours will be used to simulate the duration of field sampling.

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Receptor Selection

Receptors will be placed at exact coordinates of the perimeter sampling locations shown in Figure 1 of the Work Plan. A Global Positioning System (GPS) will be used to identify UTM coordinates of each sample location. The aboveground elevation of intake tubing for each sample location will also be measured and input into the ISCST3 model.

4.2 Verification and Validation Methods

Data verification is the process of verifying that qualitative and quantitative information generated relative to a given sample is complete and accurate.

All data will be reviewed by an internal DuPont team for compliance with the laboratory SOP and usability according to prepared checklists (see Appendix D).

Ten percent of the data points will be validated by a third party reviewer, such as ESI, for compliance with the laboratory SOP and data usability. A higher percentage of data points will [MAY??] undergo third party validation for specified sampling efforts (e.g. residential sampling). *Region III Modifications to the National Functional Guidelines* will be used as a guide for report formatting and application of qualifiers. Validation will take place concurrent with data reporting in order to expedite reporting of results. A formal report will be generated by the validator, which will include judgments on data usability and data qualifiers applied by the validator. This report will be forwarded, if requested, to the regulatory agencies.

A data usability summary (DUS) will be prepared by the project quality assurance officer.

4.2.1 Data Usability Summary

The DUS should include an examination of

- ☐ the reportable data;
- ☐ the supporting data;
- ☐ the field notes and data associated with the sampling event(s); and
- ☐ the project objectives.

The Project Quality Assurance Officer will generate a DUS that contains a review of the deficiencies identified in the data, qualifiers identifying biases and unreliable data, assessments of field and laboratory performance, overall precision and accuracy, representativeness and completeness of the data set. The DUS should provide the following information:

- ☐ Samples and analytical parameters reviewed
- ☐ Field data reviewed
- ☐ QC parameters reviewed
- ☐ Review criteria for each QC parameter
- ☐ Specific samples and constituents that did not meet criteria and applied qualifiers

□ Usability of the data

The DUS, or the conclusions reached in the DUS, will be included in project periodic reports and the final report submitted for the Phase II investigation.

4.3 Reconciliation with Data Quality Objectives

The DUS shall describe the effect of the uncertainty associated with results qualified as estimated that may affect the usability of the data in meeting data quality objectives. The DUS should include an evaluation of how representative the analytical results are of the medium being evaluated, based on measures such as sampling design, replicate analyses, and quality control results. It shall also include a discussion on the sufficiency of the valid data set for meeting project objectives. The DUS shall also contain a listing of data that have been rejected during validation or that have been considered unusable for meeting specific project or data quality objectives. The DUS shall include a discussion of the consequences of having rejected or unusable data.

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TABLES

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TABLES

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Table 1

**DUPONT WASHINGTON WORKS PHASE II INVESTIGATION
PRECISION, ACCURACY, AND COMPLETENESS
OBJECTIVES**

Parameter	Media	Precision (Relative Difference)	Accuracy (Relative Recovery)	Completeness
C-8*	Water	20	70-130	90**
C-8*	Soil	20	70-130	90**
PFOA***	Air	20	70-130	90**
C-8*	Biota****	40	50-150	90**

*also known as FC-143 or ammonium perfluorooctanoate (APFO).

**Completeness objective may be 100% for designated sampling events.

***Perfluorooctanoic acid

****Vegetation including litter layer, root zone with soil.

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Table 2

**DUPONT WASHINGTON WORKS PHASE II INVESTIGATION
SUMMARY OF CONTAINERS, HOLDING TIMES, AND
PRESERVATION**

Test	Media	Container	Holding time	Preservation
C-8***	Water	P	14 days	Cool 4°C
C-8***	Soil	P	14 days	Cool 4°C
PFOA****	Air	OVS tubes	14 days	Ambient
C-8***	Biota*****	Zip-loc bags	14 days	Cool 4°C

*Hold times are advisory.

**All samples except air (OVS tubes) are to be stored at a nominal 4°C (temperature range is not frozen to 6°C).

***also known as FC-143 or ammonium perfluorooctanoate (APFO).

****Perfluorooctanoic acid

*****Vegetation including litter layer, root zone with soil.

P = Polyethylene

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Table 3

DUPONT WASHINGTON WORKS PHASE II INVESTIGATION
ANALYTICAL METHODOLOGY

Parameter	Media	Laboratory	Analytical Method	EXP-000480 01M-008-046 Revision 1
C-8**	Water	Exygen	01M-008-046 Revision 1	0040
C-8**	Soil	STL - Denver	DEN-LC-0012 Revision 2.1	----
PFOA***	Air	Exygen	Study P419	----
C-8**	Biota****	Morse	tbd	----

*Methods are from laboratory standard operating procedure (SOP) or validation of SOP.

**also known as FC-143 or ammonium perfluorooctanoate (APFO).

***Perfluorooctanoic acid

****Vegetation including litter layer, root zone with soil.

tbd = to be determined

Exygen = Exygen Research, State College, Pennsylvania

STL - Denver = Severn Trent Laboratories, Arvada, Colorado

Morse = Morse Laboratories, Sacramento, California

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FIGURES

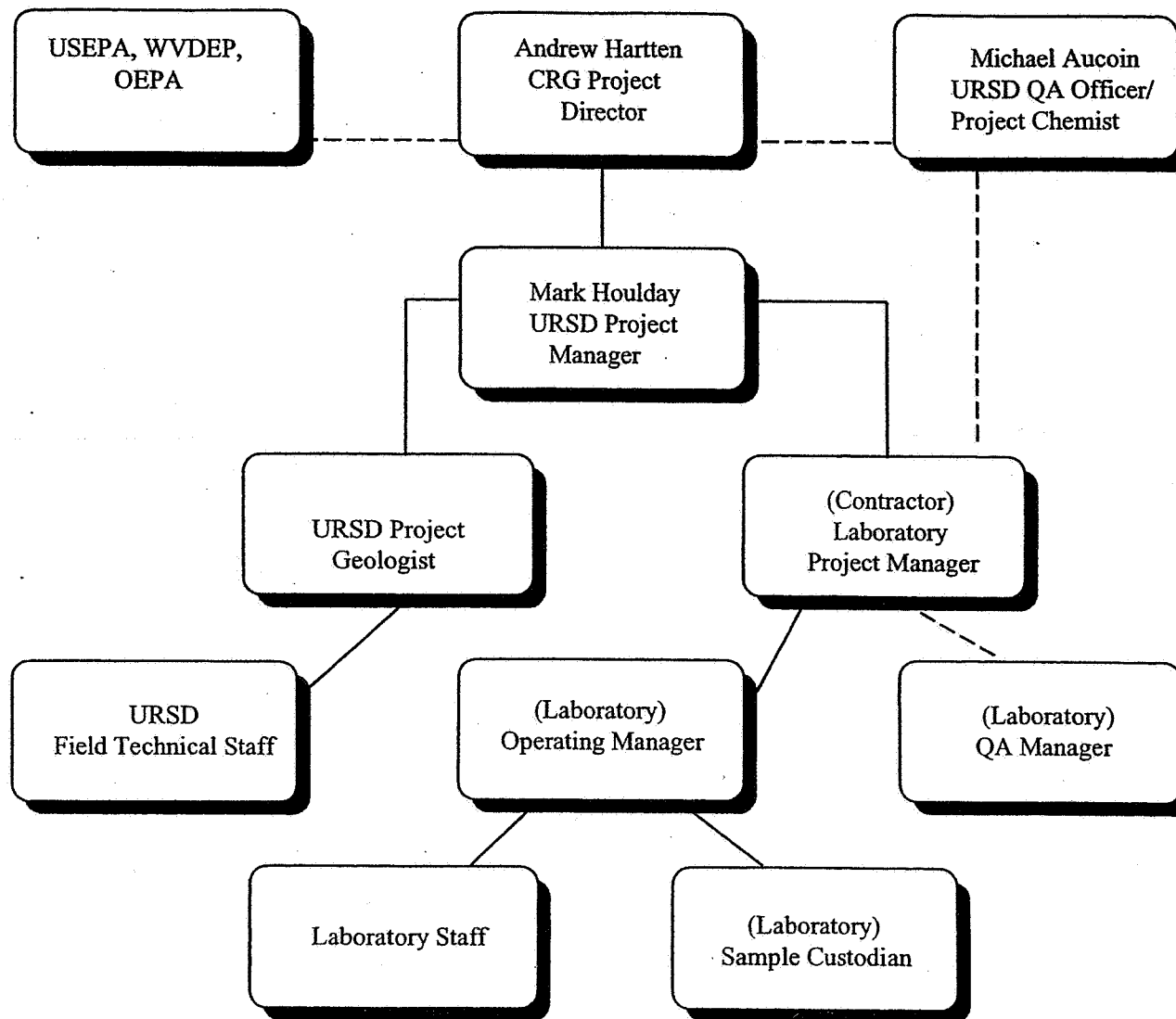
EXP000481

FIGURES

EXP000482

Figure 1

PROJECT ORGANIZATION DIAGRAM



— Line of authority
- - - Line of communication

EXP000483

Figure 2
Sample Label
Quality Assurance Project Plan
DuPont Washington Works Phase II Investigation

Client	_____
Sample ID	_____
Location	_____
Analysis	_____
Preservative	_____
Collection Date/Time	_____
Collected By	_____

6112155 B-1100

EXP000484

Figure 3

[illegible]

DAILY INSTRUMENT CALIBRATION CHECK SHEET
Quality Assurance Project Plan
DuPont Washington Works Phase II Investigation

INSTRUMENT: _____

SERIAL NO.: _____

[illegible]

EXP000486

Figure 5

AUDIT CHECKLIST
Quality Assurance Project Plan
DuPont Washington Works Phase II Investigation

PROJECT: _____ **PROJECT MANAGER:** _____

SITE LOCATION: _____

AUDITOR: _____ **DATE:** _____

Question	Yes	No	Comment/Documentation
Field:			
1. Was an on-site safety officer appointed?			
2. Did site personnel receive a copy of the site-specific sampling and analytical plan in a timely manner to allow for sufficient review?			
3. Are copies available in the field during sampling?			
4. Was a briefing held off site, before any site work was begun, to acquaint personnel with sampling equipment, assign field responsibilities, and review safety procedures?			
5. Do field personnel have a field notebook?			
6. Are the site survey grid stakes present?			
7. Do the number and location of samples collected follow the procedures as specified in the site-specific sampling and analysis plan?			
8. Are samples labeled?			
9. Are samples being collected following the procedures?			
10. Was a chain-of-custody form filled out for all samples collected?			
11. Are samples preserved as specified?			
12. Are the number, frequency, and type of samples (including blanks and duplicates) collected as described in the sampling analysis plan?			
13. Are the number, frequency, and type of measurements and observations taken as specified in the site-specific sampling and analysis plan?			

Figure 5

AUDIT CHECKLIST
(Continued)

Question	Yes	No	Comment/Documentation
Field:			
14. Are operating procedures for field equipment available?			
15. Is a record maintained of the calibration of field equipment?			
16. Is field equipment being calibrated as required?			
17. Are geophysical cross sections correlated to geologic data?			
18. Is safety equipment being used by field personnel?			
19. Is emergency safety equipment available as required in the health and safety plan?			
20. Are well designations clearly labeled (i.e., well numbers)?			
21. Are caps on wells locked if not being used?			

Figure 6

CORRECTIVE ACTION REQUEST
Quality Assurance Project Plan
DuPont Washington Works Phase II Investigation

Number: _____ Date: _____

To: _____

You are hereby requested to take corrective actions indicated below and as otherwise determined by you (A) to resolve the noted condition and (B) prevent it from reoccurring. Your written response is to be returned to the project quality assurance officer by _____

Condition: _____

Reference Documents: _____

Recommended Corrective Actions: _____

Originator	Date	Approval	Date	Approval	Date
------------	------	----------	------	----------	------

Corrective Action

(A) Resolution: _____

(B1) Prevention: _____

(B2) Affected Documents: _____

Signature: _____ Date: _____

O.A. Followup

Corrective Action Verified By : _____

Date: _____

EXP000489

APPENDICES

EXP000490

APPENDIX A

EXP000491

APPENDIX A

ELECTRONIC DATA DELIVERABLE (EDD) FORMAT

EXP000492

**DuPont Lab Network
Envista EDD Specification**

Version 1.0

April 25, 2003

EXP000493

Introduction

The DuPont Corporate Remediation Group (CRG) maintains a corporate environmental database (Envista) that stores field data, analytical results, QA/QC results, water levels, and other information resulting from the activities of the DuPont environmental projects. Much of this data is provided by analytical labs or sampling contractors performing analytical and sampling services for DuPont. To optimize loading data generated by these contractors, a format of an ASCII text file has been developed for conveying data to DuPont for loading into the database. These text files will be referred to as Envista EDD files. Following is a description of the Envista EDD specification.

General Information

Envista EDD files are electronically submitted to CRG in an agreed upon manner. The EDD must match the hardcopy report in terms of samples, tests, analytes, and results. Also, DuPont requires the lab composite results such that only one result is reported for each analyte (i.e., the lab submits only the result judged best when a sample is re-analyzed for particular analytes due to exceeding calibration range, etc.)

Normally, all data for a particular sample delivery group will be contained in one file. This group is normally referred to as a lot (or group), which makes up a normal reporting/invoicing group and usually consists of samples for a given project and site that the lab has received in one day, including all associated QC samples and results. Note that QC results may be contained in more than one EDD if field samples from different lots were analyzed in the same QC batch.

All data must be written in text format (ASCII). Each data field within a record must be delimited by the caret (^) symbol. Therefore, each record must begin with a ^. If another format is required, arrangements must be made with the DuPont Envista database administrator and another format will be provided. This format must be agreed upon between the lab and DuPont prior to the delivery of the data to DuPont.

Some data fields are required to be populated, while others are populated depending on the project circumstances or the particular data being reported. These requirements are described in the "When Required" column of the EDD specification in Table 1. The length of each field must not exceed the width specified in the "Width" column of the EDD specification in Table 1, or the data will be truncated. Fields designated as a number (N) in the "Width" column of Table 1 must contain a text string that will convert to a number. The record format of the deliverable is positional and therefore, each field must be listed in the order specified in Table 1. Also, null or blank fields must be delimited. For example, if MDL was not applicable to a sample, the field would be designated with a ^^ indicating the MDL was null. Each field begins with a ^ and ends with a ^, but the trailing ^ also becomes the beginning ^ for the next field. Missing data, truncated data, or improperly ordered fields within a record may result in the deliverable being judged to be incomplete.

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There is only one type of record, and all records must be the same format (i.e., flat file format). Note that this is changed from the previous DuPont CED EDD specification.

Note that different laboratory samples (samples with different Lab_IDs) may come from the same field sample. This is preferable for results that are from different lab samples that may have the same parent field sample, such as composited results from different dilutions.

Samples taken for matrix spike/matrix spike duplicates (MS/MSD) and laboratory replicates (REP) are QC samples that have field samples. If the field sample is from DuPont and is in the current lot for the current project, the parent or unspiked sample and result information should be included in the EDD and the Field_Sample_ID for the MS, MSD, and REP should be included for those records. If the parent field sample is not from a DuPont site or is from a DuPont site but not the current site and project, the field sample and result should not be included, and the field sample name will be null for the MS, MSD, and REP. Lab originated (QA/QC) samples such as lab control spikes or method blanks will not have a field sample information (field sample ID, date and time sampled, etc.).

The Batch Number is a unique number that identifies the laboratory QC batch. Note that this is changed from the previous DuPont CED EDD specification.

QA/QC results involving relative percent recoveries and relative percent differences, e.g. MS/MSDs, REPs, lab control spikes and lab control spike duplicates (LCS/LCSD), and surrogates must also include these recoveries and differences plus the maximum and minimum recoveries and differences that are acceptable, as applicable. For example, an MS sample requires a result, the relative percent recovery, and the maximum and minimum permissible relative percent recovery. An MSD sample requires a result, the relative percent recovery, the relative percent difference, the maximum and minimum permissible relative percent recovery, and the maximum permissible relative percent difference (see Table 2).

DuPont will no longer require the laboratories to comment on items already flagged in the hardcopy report. For example, laboratories will not be required to comment on poor matrix spike/matrix spike duplicate recoveries, poor laboratory control sample/control sample duplicate recoveries, poor surrogate recoveries, and method blank contamination (if flagged in the hardcopy report).

Laboratories will be required to comment on issues not flagged in the hardcopy report. For example, if samples had to be reextracted/redigested/reanalyzed for any reason, hold times missed, sample was reextracted due to poor surrogate recovery and outcome of reanalysis, sample received without correct preservation, not enough volume to perform matrix spike/matrix spike duplicate samples, etc., laboratories will provide a comment on the analysis report or in the case narrative. Note that comments and/or case narratives should also be included electronically in the comment field, or may be submitted as a separate electronic file in an agreed upon format.

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Table 1. DuPont Envista EDD flat file field format			
Field Name	When Required	Maximum Length (C=Character, N=number)	Description
Lot_ID	All Records	C30	Identifier for the lot (group of samples that came lab receives in same shipment, and is the typical EDD/hardcopy report/invoice unit)
Lab_ID	All Records	C30	ID used to identify the sample at the lab
Field_Sample_ID	All Field samples (FS, TB, FB, EB) and MS/MSD/REP when parent is DuPont sample for project	C60	Field sample ID or Client ID (normally from chain of custody)
Sample_Type	All Records	C30	Designates the type of sample as a Field Sample (FS), Equipment Blank (EB), Field Blank (FB), or Trip Blank (TB), Matrix Spike (MS), Matrix Spike Duplicate (MSD), Lab Control Spike (LCS), Lab Control Spike Duplicate (LCSD), Lab Duplicate (REP), Method Blank (MB)
Collection_date	Field samples (FS, TB, FB, EB) only	C9	Date sample was collected (Format is DD-MMM-YY, e.g. 15-JAN-97)
Collection_time	Field samples (FS, TB, FB, EB) only	C4	Time sample was collected (Format is HHMM military time, e.g., 1:30 p.m. is 1330, 9:30 a.m. is 0930)
Receipt_date	Field samples (FS, TB, FB, EB) only	C9	Date received at the lab (Format is DD-MMM-YY, e.g. 15-JAN-97)
Sample_Matrix	All Records	C30	Matrix of the sample (Water, soil, sediment, air, oil...) as collected
Lab_Name	All Records	C30	The name or code for the lab (these will be assigned by DuPont for each lab)
Project_Name	All Records	C30	Name assigned to the project
Sample_comments	As applicable	C600	Lab comments regarding handling of samples (e.g. Sample came in above temperature.) Note that comments may be provided as a separate electronic file in an agreed upon format.
Batch_ID	All Records	C30	Unique ID that identifies the group of samples prepared & analyzed together (QC batch)
Analyte_name	All Records	C60	Compound or sample property tested for.
CAS_No	All Records	C30	Chemical Abstract number of the analyte or lab assigned number (without dashes preferred).
Result_Mod	See Table 2	C30	"<" if result is non-detect, otherwise null
Result	See Table 2	C20	Result of an analysis. If non-detect, use reporting limit; either PQL or MDL as specified by the project. For high-resolution isotope dilution methods, use EDL for non-detects.
PQL	See Table 2	C20	Practical Quantitation Limit for an analysis in same units as result, adjusted for sample amount and

EXP000496

			dilution. For high-resolution isotope dilution methods, use EDL.
MDL	See Table 2	C20	Method Detection Limit for an analysis in same units as result, adjusted for sample amount and dilution. For high-resolution isotope dilution methods, this would be null.
Reporting_Units	See Table 2	C30	Units that the analysis is reported in (mg/l, ug/kg, etc.)
Upper_Error	As applicable	C20	Upper error range (e.g., for rad data, + error measurement)
Lower_Error	As applicable	C20	Lower error range (e.g., for rad data, - error measurement)
Qualifiers	As applicable	C30	Flags applied to the analysis to qualify the data.
Dilution_Factor	All Records	N20	Sample dilution factor. If not diluted, enter one (1).
Analysis_Method	All Records	C30	Method used to run an analysis
Analysis_Date	All Records	C9	Date that the analysis was run (Format is DD-MMM-YY, e.g. 15-JAN-97)
Analysis_Time	All Records	C4	Time that the analysis was run (Format is HHMM military time. e.g. 1:30 p.m. is 1330, 9:30 a.m. is 0930)
Prep_Method	When prep performed	C30	Method used to prep an analysis (if prep method = analysis method, then this should be 'METHOD' without the quotes, or can be null).
Prep_Date	When prep performed	C9	Date that the sample was prepared. (Format is DD-MMM-YY, e.g. 15-JAN-97)
Prep_Time	When prep performed	C4	Time that the sample was prepared (Format is HHMM military time. e.g. 1:30 p.m. is 1330, 9:30 a.m. is 0930)
Preprep_Method	When preprep performed	C30	Method used to preprep/leach an analysis (e.g., 1310, 1311, 1312).
Preprep_Date	When preprep performed	C9	Date that the sample was prepreped (Format is DD-MMM-YY, e.g. 15-JAN-97)
Preprep_Time	When preprep performed	C4	Time that the sample was prepreped (Format is HHMM military time. e.g. 1:30 p.m. is 1330, 9:30 a.m. is 0930)
Analyte_Type	All Records	C30	Target analyte (FS), Surrogate (SU), tentatively identified compound (TIC) or Internal Standard (IS)
Filtered	All Records	C1	Total (T) or Dissolved (D)
Dry_wet_basis	All Records	C2	D for dry weight basis, W for wet weight basis, NS for (air, wipe, etc.)
Instrument	All Records	C30	Lab defined identifier for instrument on which analysis was performed.
TIC_number	For TIC results	N2	Tentatively identified compound number beginning with 1 and consecutively numbered to include all TIC's found in the sample. If no TICS are found, can either submit 1 TIC result with ND as result, or not include TIC result record
Spike_added	See Table 2	N20	Amount of the analyte spiked into a quality control sample, in same units as result
RPR	See Table 2	N20	Relative percent recovery. For MS/MSD, if concentration in unspiked parent sample > 4x spike amount, RPR should be null and enter NC as qualifier. For surrogates, if dilution factor > 4, RPR should be null, and enter NC as qualifier.

Min_RPR	See Table 2	N20	Lowest recovery limit acceptable for the method
Max_RPR	See Table 2	N20	Highest recovery limit acceptable for the method
RPD	See Table 2	N20	Relative percent difference between duplicate sample types (sample & REP, MS & MSD, LCS & LCSD). For MSD, if concentration in unspiked parent sample > 4x spike amount, RPD should be null and enter NC as qualifier.
Max_RPD	See Table 2	N20	Highest relative percent difference allowed for the method/analyte
Initial_weight_volume	Optional	N18	Initial sample weight or volume before any preps or prepreps
Initial_weight_volume_units	If initial weight/volume included	C30	Units for initial sample weight/volume
Final_weight_volume	Optional	N18	Final sample weight/volume after any preps or prepreps
Final_weight_volume_units	If final weight/volume included	C30	Units for final sample weight/volume
DuPont_Cost_Code	Optional	C30	The DuPont cost code (specifies method/matrix/deliverable/turnaround time) from Exhibit B of the current contract. This is currently optional, but will be required in the future.
Result_Comments	As applicable	C600	Any result specific comments. Note that comments may be provided as a separate electronic file in an agreed upon format.
Fraction	All Records	C30	This field is used to differentiate laboratory samples and results as required. For example, some labs use the LabID (2 nd field in this list) to identify the client field sample, which does not sufficiently differentiate lab samples and results from different dilutions. This field could also be a more specific internal Lab ID

EXP000498

Table 2. Fields Required for each Sample Type and Surrogates/Internal Standards								
Fields	SAMPLE Types—> FS, EB, TB, FB	MS	MSD	MB	LCS	LCSD	REP	Surrogates
Result_Mod (if non-detect)	X	X	X	X	X	X	X	
Result	X	X	X	X	X	X	X	
MDL (not TICs)	X	X	X	X	X	X	X	
PQL (not TICs)	X	X	X	X	X	X	X	
Spike_Added		X	X		X	X		
RPR		X	X		X	X		X
Min_RPR		X	X		X	X		X
Max_RPR		X	X		X	X		X
RPD			X			X	X	
Max_RPD			X			X	X	
Reporting_Units	X	X	X	X	X	X	X	

EXP000499

APPENDIX B

EXP000500

APPENDIX B

GROUNDWATER SAMPLE COLLECTION FORM

EXP000501

Corporate Remediation Group - Field Book

Site: _____ Event: _____ Date: _____ Time: _____

Personnel: _____ **Project Manager:** _____

Well ID: _____ Permit No: _____ VO Vapors: PIONEER 02: _____

Weather Conditions: ☒ Clear ☐ Cloudy ☐ Other ☐ Wind: 1000 ft Temp: 70 °F

Well Depth: _____ LNAPL: _____ Purge Method: _____

Depth to Water: _____ DNAPL: _____ Purge Start: _____

Water Column: _____ Casing Dia: _____ Purge Stop: _____

Well Vol: _____ Conv. Factor: _____ Parameter Collection Time: _____

Well Vol (3x): _____ Purge Rate: _____ Water Level Stable @: _____

Parameters					Sample	Analysis Time
pH						
Temperature (°C)						
Specific Conductance (µmho)						
Dissolved Oxygen (mg/l)						
Redox (mV)						
Turbidity (ntu)						
Color						
Odor						

Sample Date: _____ **Sample Collection Time:** _____ **Sample Method:** _____

[illegible]

Analyst Name: _____

Analyst Signature: _____ Date: _____

000070

EXP000502

APPENDIX C

EXP000503

APPENDIX C

AIR SAMPLE COLLECTION FORM

EXP000504

**DuPont Washington Works
Air Sampling Program**

Sampler(s):

FedEx Airbill #:

Event Number

Pump #	Station #	Oxygen ID	Calibration (pre)	Date	Start Time	Stop Time	Calibration (post)	Total Minutes	Total Volume

Hours	Minutes	Hours	Minutes	Hours	Minutes	Hours	Minutes	Hours	Minutes
0	0	5	300	10	600	15	900	20	1200
1	60	6	360	11	660	16	960	21	1260
2	120	7	420	12	720	17	1020	22	1320
3	180	8	480	13	780	18	1080	23	1380
4	240	9	540	14	840	19	1140	24	1440

Field Blank: yes or no

Weather:

SAFER operational: yes or no

Comments:

EXP000505

APPENDIX D

EXP000506

APPENDIX D

C-8 AND PFOA DATA REVIEW CHECKLISTS

EXP000507

Location -

Jobname -

Lab - Exygen Research

Lab Report No. -

NARRATIVE - confirm target compound is APFO, review text against SOP for sample prep/analysis, note if QC criteria not being met

RESULTS SUMMARY -

Confirm sample results reported as APFO (QC samples, raw data may show PFOA)

Evaluate precision for each sample and lab rep pair - if both results $> 5X$ LOQ calculate RPD, compare to 20% (40% for solids); if either or both results are $\leq 5X$ LOQ, calculate difference (using LOQ for non detects) and compare to LOQ (2X LOQ for solids)

If precision criteria met, report average of sample/lab rep; if precision criteria not met, or if one result above and one below LOQ, report the higher of the two results. Verify lab reported average or higher of sample and lab rep results, as appropriate.

MATRIX SPIKE RECOVERY - spike recovery must meet criteria (70-130%) unless sample concentration is $> 4X$ spike concentration. Spike value is 500 - 500,000 ng/L.

COC REVIEW/SAMPLE RECEIPT

Samples relinquished by field

Samples received at lab next day

Samples packed in wet ice

Sample temperature upon receipt (not frozen to 6 C)

HOLD TIME - 14 days from date of collection to analysis date

Location -

Jobname -

Lab - Exygen Research

Lab Report No. -

RUN SEQUENCE -

Compare Masslynx sample list to raw data report, including run sequence no

Verify non-detects are less than LOD area value

Verify slope and intercept on raw data report are from correct curve (final, not initial)

Check for:

Initial 6 point calibration

Each standard repeated during sequence

Check standard (250 ppt) within 15% of average response for 250 ppt calibration standards

LCS @ LOQ (recovery should be 70-130%)

LCS @ 10X LOQ (recovery should be 70-130%)

Recalculate one or more results

METHOD BLANKS - less than LOQ

LC/MS/MS OPERATING CONDITIONS

LM/HM Res 1 - should be 13 - 14

LM/HM Res 2 - should be 11-14, not greater than LM/HM Res 1

Cone voltage - should be 10

Scanning method - 413-369 transition

MASS CALIBRATION

Confirm sample analysis within one week of mass calibration meeting criteria

All masses must be found within 0.2 amu of known masses

Location -

Jobname -

Lab - Exygen Research

Lab Report No. -

INITIAL CALIBRATION - initial 6 standards, with no weighting

Correlation coefficient $R \geq 0.992$

Coefficient of determination $R^2 \geq 0.985$

CONTINUING CALIBRATION - all standards, with 1/x weighting, no more than two standards excluded

$R^2 \geq 0.985$

RETENTION TIMES - no more than 2% drift during run sequence

COMMENTS

Reviewer -

Date -

Location -

Jobname -

Lab - Exygen Research

Lab Report No. -

NARRATIVE - confirm target compound is PFOA (C8 Acid), review text against SOP for sample prep/analysis, note if QC criteria not being met

RESULTS SUMMARY -

Confirm sample results reported as PFOA (C8 Acid)

Confirm air volume samples same for all fractions within a sample

Confirm concentration $A > B > C$ reported for each fraction within a sample

For each sample, if B is non detect, then C = NA (not analyzed)

COC REVIEW/SAMPLE RECEIPT

Samples relinquished by field

Samples received at lab next day

Samples shipped at ambient temperature

HOLD TIME - apply 14 days from date of collection to analysis date (this is an advisory limit only)

PFOA Air Data Package Review

page 2 of 3

Location -

Jobname -

Lab - Exygen Research

Lab Report No. -

RUN SEQUENCE -

Check for:

Initial calibration (6 point or more)

Each standard repeated during sequence

Solvent blank following the high standard

LCS (recovery should be 75-125%)

Surrogates (advisory recovery limits are 75-125% - note excursions)

Recalculate one or more results

METHOD BLANKS - less than LOQ (0.1 ug/fraction)

LC/MS OPERATING CONDITIONS

Signal 1 (C8 acid) -369 amu

Signal 2 (C10 surrogate) - 469 amu

TUNE

Confirm sample analysis within one month of tune meeting criteria

All masses must be found within 0.2 amu of known masses

Location -

Jobname -

Lab - Exygen Research

Lab Report No. -

INITIAL CALIBRATION -

Correlation coefficient $R \geq 0.992$

RETENTION TIMES - no more than 2% drift of surrogate peak during run sequence

COMMENTS

Reviewer -

Date -