



United States Environmental Protection Agency
Region III
Office of Analytical Services & Quality Assurance



**Sample Submission Procedures
for the
Office of Analytical Services
& Quality Assurance
Laboratory Branch**

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Fort Meade, Maryland 20755-5350

Sample Submission Procedures for OASQA Laboratory Branch

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1.0 Introduction

This document specifies the procedures that are to be followed when submitting samples to the Office of Analytical Services and Quality Assurance (OASQA) Laboratory Branch located at Ft. Meade, Maryland. *These procedures will help ensure that the field and laboratory aspects of the sampling event are linked in a way to produce reliable data of known quality.*

2.0 Project Planning and Analytical Request Preparation and Scheduling

An approved Quality Assurance Project Plan (QAPP) should be in place before project scheduling. For information on QAPPs, please contact a member of the OASQA Technical Services Branch, Quality Assistance Project Plan specialists listed below.

Quality Assistance Project Plan contacts:

Robin Danesi: 410-305-2607 (ph), 410-305-3095 (fax) danesi.robin@epa.gov
Mary Ellen Schultz: 410-305-2746(ph), 410-305-3095(fax) schultz.maryellen@epa.gov
Mike Mahoney: 410-305-2631 (ph), 410-305-3095 (fax) mahoney.mike@epa.gov
Ed Messer: 410-305-2744 (ph), 410-305-3095 (fax) messer.ed@epa.gov

All analytical requests are scheduled through the OASQA Technical Services Branch. See the *EPA Region III, Users' Guide for Acquiring Analytical Services* for instructions on preparing and submitting Analytical Requests, and other documentation requirements. (Current version found at <http://www.epa.gov/region03/esc/labservices.htm>)

Analytical Request contacts:

John Kwedar: 410-305-3021 (ph), 410-305-3095 (fax) kwedar.john@epa.gov
Dan Slizys: 410-305-2734 (ph), 410-305-3095 (fax) slizys.dan@epa.gov
Carroll Harris: 410-305-2625 (ph), 410-305-3095 (fax) harris.carroll@epa.gov

3.0 Sampling

Proper collection and identification of samples, documentation of the collection event (in permanent field records), and submittal of required paperwork (i.e., Chain of Custody, Hazardous Exposure sheets) are all essential parts of a successful sampling event. When samples are not properly collected, preserved, or shipped, the quality of the data may be compromised. If this occurs, the requestor will be notified and given the opportunity to resample or to receive qualified data. Refer to the *OASQA Sample Acceptance Policy* (Attachment 4). In some cases it may be possible for the laboratory to complete the analysis with some alteration. For example, if there is insufficient sample volume, the data may have to be reported with increased quantitation limits or the requestor may be asked to prioritize analyses.

3.1 Collection Requirements

Tables I, II, III and IV show preservatives, minimum volumes/weights, container types and holding times for aqueous and non-aqueous samples submitted to OASQA.

IMPORTANT: All differences due to the requirements of the drinking water program (SDWA) are listed in Table III. In addition, the TABLE NOTES for Tables I, II and III summarize the critical information needed for sample collection. It is essential to

comply with these requirements so that reliable data that meet the needs of the project are produced.

3.2 Holding Times

Samplers must be aware of the holding times for all analyses requested and ship samples to OASQA as quickly as possible. Holding times are calculated *from the time and date of sample collection* and not the date of receipt at the lab. To ensure that OASQA can meet the required holding time, it may be necessary to ship samples at the end of each collection day.

PLEASE NOTE: OASQA does not accept samples on weekends or holidays at this time. Please plan your sample collection accordingly so that samples are collected, shipped and analyzed within holding times.

Data from samples analyzed past the stated holding time must be carefully examined by the data user. These values may be biased low due to possible loss of the parameter(s) of interest, and they will be flagged by the laboratory as such.

3.3 Sample Types

Sample collection types normally used are defined as follows:

Grab sample - An individual sample collected over a period of time generally not exceeding 15 minutes. A grab sample is normally associated with water or wastewater sampling. However, soil, solid, oil, sediment, and liquid hazardous waste samples, for example, may also be considered grab samples.

Composite sample - A sample containing discrete aliquots (1) collected over a defined time period at equal time intervals (time composite), (2) collected in volumes proportional to the flow rate (flow proportional composite), or (3) composited from individual grab samples collected on an area or cross-sectional basis (area composite).

3.4 Blanks

Blank samples are used to identify potential sources of contamination during sampling, shipping, storage and analysis. Blanks should be specified as part of every QAPP or sampling plan. It is highly recommended that field blanks accompany all sample sets, *with the exception of VOAs in Air*.

All water used for blanks must be lab pure water, demonstrated to be free of the parameter(s) of interest. The water may have to be tested prior to use. Commercially available HPLC water is not acceptable for most blank uses. HPLC water is not certified as "organic-free" and/or "metal-free", and therefore, may contain compounds of interest. Blanks that are preserved must be prepared with the *same stock and same volume* of the preservative that was used with the samples.

The blank types normally used are defined as follows:

Sample Matrix ("Field") Blank: The field blank is used to determine whether contamination has been introduced during sample collection, storage and shipment, as well sample handling in the analytical laboratory. Field blanks are prepared by transferring demonstrated analyte-free water to the appropriate sample containers during the time when site-specific samples are collected. These blanks are transported to the field and exposed to the same conditions as site-specific samples

including removal of the container caps and addition of any appropriate preservatives. Field blanks should be collected whenever aqueous samples are collected and at a frequency of one per 20 samples. This sample should be analyzed for the same parameters as those associated with site-specific samples collected from potentially contaminated media.

Trip blank: The trip blank is used to determine whether contamination has been introduced to aqueous samples through cross-contamination during shipment and storage of sample containers, for volatile organic compounds (VOCs) only. Trip blanks should be prepared prior to the sampling event, including preservatives, and are not exposed to field conditions. They may be furnished by the analytical laboratory and will consist of demonstrated analyte-free water provided in the appropriate container (i.e., 40 ml teflon-lined glass vial). Trip blanks should be collected at a frequency of one per each cooler used to store/transport site-specific samples designated for aqueous VOC analyses (or one for each day that VOCs are collected).

Rinsate or sampling equipment blank: The rinsate blank is used to determine whether the sampling equipment decontamination procedure has been adequately performed, thereby assuring that no "carryover" contamination has been introduced before (or during) sample collection. Rinsate blanks are prepared in the field by pouring demonstrated analyte-free water through/over the sampling equipment (including filters) and collecting rinsate in the appropriate sample containers and adding appropriate preservatives. Rinsate blanks should be collected at a frequency of one per 20 samples per matrix per sampling equipment type (or one per day per matrix per equipment type). This sample should be analyzed for the same parameters as those associated with site-specific samples collected from potentially contaminated media.

Temperature blank: The temperature blank is used only to determine whether site-specific samples have been adequately cooled during shipment and storage. Temperature blanks can be prepared any time before or during field sampling activities by adding water to an appropriate sample container such as a VOA vial. Temperature blanks should be collected at a frequency of one per each cooler used to store/transport site-specific samples. The temperature of this sample should be measured upon receipt by the analytical laboratory but not analyzed.

3.5 Laboratory Quality Control Requirements

In addition to the minimum volumes or weights needed to perform a single analysis for each parameter listed in Tables I, II and III, it is essential that the sampler collect enough sample to allow the laboratory to analyze samples for quality control (QC) purposes. **For each parameter, the laboratory will need 3 times the minimum volume for one sample per batch of:**

10 samples for inorganic parameters
20 samples for organic parameters

This volume will be used for the quality control analysis for each batch of samples. If there are more than 10 (inorganic) or 20 (organic) samples per batch, another sample with extra volume should be collected for each group. For example, in a set of 11 to 20 samples for inorganic parameters, there should be extra volume collected for **two** samples.

It is extremely important that sufficient volume be collected for quality control analysis. This is especially true for organic analysis, because of the large volume requirements. A liter of sample will be needed for EACH parameter or analytical fraction, and a sufficient volume for quality control analysis (3 times sample volume) must be collected for each parameter or fraction. For example, for each sample collected for organic analysis for

SVOAs and Pest/PCB with QC, you will need to collect 3 liters for SVOAs and 3 liters for Pest/PCB, for a total of 6 liters. *ON THE OTHER HAND*, please do not collect any more volume than necessary; it is expensive to properly dispose of excess volume and is inefficient for both the sampler and laboratory. (See Section 7.0 Pollution Prevention and EMS.)

4.0 Paperwork Requirements

Region III requires the use of the FORMS II LITE for preparation of the sampling documentation, with certain exceptions (see below). FORMS II LITE is an electronic windows-based application which automates sampling event documentation. It generates a Chain of Custody Form (COC), sample tags, and container labels. FORMS II LITE software is available for downloading at: <http://dyncsdao1.fedcsc.com/itg/forms2lite/>.

When access to FORMS II LITE is not practical, hard copy versions of the paperwork may be used. Instructions for accessing the paper versions of the COC, sample tags, and custody seals are available by contacting the Technical Services Branch Analytical Request Specialists. See Attachments 1 and 2 for examples of completed forms. The following documents must accompany the sample shipment for accurate identification and safety information.

Sample Tag - Each sample must have a sample tag tied to the container and some type of adhesive label with identifying information. Each sample tag and label must be legibly written with indelible (i.e., waterproof) ink. (See Attachment 2 for an example.) The information that is written on the sample label must match the information on the Chain of Custody form (COC). The sample tag must be **tied on each** container so that it will not fall or slip off. Please do not use tape to secure labels or tags since it will loosen and fall off if the containers get cold or wet. No erasures or white outs are allowed. All errors must be corrected with a single line through the error, initialed and dated. For the safety of lab staff, indicate on each sample label and sample tag any preservative used for the samples.

Exposure Data Sheet - Each time samples are collected, the sampler must complete a *Hazard and Risk Exposure Data Sheet* (Attachment 3). This information helps ensure the safety of the lab staff receiving the samples so that proper precautions are taken whenever potentially hazardous samples are encountered. This sheet is a vital part of the OASQA safety program and must be attached to the **OUTSIDE** of at least one shipping container so that it is available for review by the sample managers before opening any coolers or chests.

Chain of Custody (COC) - A COC must accompany each sample shipment. (See Attachment 1 for an example.) A sample shipment without a COC may be rejected by the laboratory. **The COC must be sealed in a water proof zip-locked bag and taped on the inside of the ice chest lid with the samples.** Always use indelible ink (never pencil) for all markings on the COC. The original record must accompany the shipment and a copy retained by the sampler. Each distinct sample must appear on a separate line. It is NOT necessary to have a separate line for each container (or each sample tag). NOTE: *Samples collected for dissolved constituents are considered distinct from the unfiltered aliquot and should be placed on a separate line.* Any writing errors made on the COC must be crossed out with a single line, initialed, dated and rewritten.

Chain of Custody documentation must include:

- site name (project name)
- sampler's name/signature
- sample ID (station number)
- date and time of collection (recorded in 24 hour clock time)
- type of sample (grab or composite)
- sample description (station location) (indicate if sample has been filtered for dissolved components or if it is a field duplicate)
- number of containers
- parameters requested (i.e., tests, methods)
- sample tag numbers (in remarks)
- date, time and signatures for sample receipt and transfer

If problems are found with the documentation or the physical condition of the samples upon arrival at the laboratory, the Sample Scheduling Coordinator may require a Letter to File from the sampler to document problems and corrective actions taken.

5.0 Shipping Requirements

The OASQA laboratory **will not accept any samples shipped with any particulate (dusty) type packing material, especially vermiculite.** We recommend using plastic bags to double-bag glass containers to prevent leakage, then wrapping each container in bubble wrap to prevent breakage. To further assure that any leakage will be contained, the cooler should be lined with a large plastic bag. Bags of ice (sealed to contain the melted water) should be used for cooling the samples. To ensure uniform cooling, the bags must be packed on top and around the samples themselves and not merely placed on the sides of the packed chest. **The loaded cooler must not be heavier than 50 pounds to allow for safe handling.**

SAMPLES PRESERVED BY CHILLING MUST BE SHIPPED WITH SUFFICIENT ICE TO REMAIN AT $\leq 6^{\circ}\text{C}$ WHILE IN TRANSIT. A temperature blank (VOA vial filled with water) must be included in the shipment to allow the laboratory to verify the temperature upon receipt.

The chest must be sealed with strapping tape and custody seals on the outside. The custody seal must be placed so that it will be broken when the chest is opened. Department of Transportation (DOT) and/or Federal Express approved shipping containers must be used. *If the samples have been identified as 'environmental laboratory samples' such as those defined in Appendix D of the Sample Shipping Procedures reference (see Section 8.1), then the shipment process is not regulated. In most cases, preserved water, wastewater, and sediment samples accepted by OASQA for analysis are considered environmental samples. If the samples have been classified as 'hazardous materials', **only certified personnel are allowed to ship the containers**, according to the Dangerous Goods Regulations promulgated by the International Air Transport Authority (IATA) (see Section 8.2). At least one member of the sampling team should be aware of the DOT and IATA legal requirements for shipping these types of materials.*

6.0 Shipment Notification

Always notify the *Sample Scheduling Coordinator (SSC)* when samples have been shipped. In addition, notify the SSC as soon as possible if a scheduled shipment has been canceled or there are any changes in the number or types of samples. These changes may affect the

laboratory's scheduled workload. **All notifications or changes should be phoned, faxed, or e-mailed on or BEFORE the expected shipping date.** Sample shipments cannot be accepted before or after normal business hours (7:30am - 4:30pm), on weekends or Federal Holidays.

Sample Scheduling Coordinator (SSC) Contacts:

Patricia Sosinski: 410-305-2667 (ph), 410-305-3093 (fax), sosinski.pat@epa.gov

Kevin Martin: 410-305-3032 (ph), 410-305-3040 (fax), martin.kevin@epa.gov

Mailing Address:

P. Sosinski/Kevin Martin

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NOTE: *If any part of the Sample Submission Guidelines is unclear or if you want to verify the requirements for collection and shipping, please call one of the contacts listed above.* Many times problems can be avoided if issues are clarified before the samples are collected and sent. In addition, if there is a need for parameters not listed in this document, the laboratory may be able to accommodate special requests. Please call the Technical Services Branch contacts listed in Section 2.0 for information.

7.0 Pollution Prevention and EMS

It is the Environmental Science Center's policy to integrate environmental stewardship into our operations and we have therefore instituted an Environmental Management System (EMS). This means that we manage our organizations and our programs in a manner that protects the environment, the safety of our employees, and the public health. In support of this policy, the OASQA lab is committed to the promotion of pollution prevention (P2) awareness and the Agency's waste reduction strategies. To support our P2 goals, the lab requests that samplers take a common sense approach to the collection of samples with respect to how much volume is collected. Of course, the most important consideration must be the need for enough volume to constitute a representative sample, and to accommodate the analysis requested. But remember - - --**many parameters can be combined together to avoid excess volumes.** Since the laboratory must pay to dispose of the material after analyses, if at all possible, please combine samples for all analytes requiring the same container and preservative in a minimum number of containers.

Examples of parameters for aqueous samples which are commonly combined are: (1) metals + mercury + hardness; or (2) ammonia + TOC + TP + TKN. Parameters for solid samples have few preservative requirements; therefore, most inorganic or organic parameters can be combined in one container. It is especially important to consolidate parameters when collecting solid samples because of the difficulty in disposing of the excess sample. Solids should be collected in a single 8 oz. container for either the organic or inorganic parameters. For additional guidance on combining samples, please contact the *Sample Scheduling Coordinator (SSC)*.

8.0 References

8.1 Letter from U.S. Department of Transportation (DOT) stating DOT's position on shipping preserved environmental samples dated February 13, 2003.

(<http://www.epa.gov/epawaste/hazard/testmethods/pdfs/pres-samp.pdf>).

8.2 Dangerous Goods Regulations, International Air Transport Authority (IATA). Current Edition (document changes annually).

8.3 EPA Order 1000.18, February 16, 1979,

(<http://epawww.epa.gov/rmpolicy/ads/transorders.htm>

8.4 Contract Laboratory Program (CLP) Guidance for Field Samplers, OSWER 9240.0-35, EPA540-R-07-06, (<http://www.epa.gov/superfund/programs/clp/guidance.htm#sample>).

8.5 EPA Region III, Users' Guide for Acquiring Analytical Services, current version

http://www.epa.gov/region03/esc/5users_guide_rev.pdf

8.6 Sample Submission Procedures for the Office of Analytical Services & Quality Assurance Laboratory Branch, current version

<http://www.epa.gov/region03/esc/labservices.htm>

8.7 National Environmental Laboratory Accreditation Conference (NELAC) Standards, current revision. <http://www.nelac-institute.org/standards.php>

8.8 "40 CFR Parts 122, 136, 141, 143, 430, 455, and 465; Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures, Final Rule" Federal Register Vol. 72, No. 47 (Monday, March 12, 2007) 11200 – 11249.

<http://www.epa.gov/EPA-WATER/2007/March/Day-12/w1073.pdf>

8.9 "40 CFR Parts 136 and 503; Guidelines Establishing Test Procedures for the Analysis of Pollutants; Analytical methods for Biological Pollutants in Wastewater and Sewage Sludge; Final Rule" Federal Register Vol. 72, No. 57 (Monday, March 26, 2007) 14220 – 14233.

<http://www.epa.gov/fedrgstr/EPA-WATER/2007/March/Day-26/w1455.pdf>

8.10 Electronic Code of Federal Regulations e-CFR; Approved analytical methods for NPDES 40 CFR part 136.

<http://ecfr.gpoaccess.gov/cgi/t/text/text->

[idx?c=ecfr&tpl=/ecfrbrowse/Title40/40cfr136_main_02.tpl](http://ecfr.gpoaccess.gov/cgi/t/text/text-idx?c=ecfr&tpl=/ecfrbrowse/Title40/40cfr136_main_02.tpl)

TABLE NOTES

The following information applies to all parameters listed in the Sample Requirements Tables I, II, and III. Preservations, holding times, and container types are taken from the specific methods used for analysis or from Federal Register promulgated requirements.

1 BLANKS

All parameters except VOAs in Air require the collection of at least one blank sample as described in Section 3.4

* *Field Blank* -- collected for every parameter to ensure that containers and preservatives (if required) are contamination free.

* *Trip Blank* -- collected for aqueous volatile organics (VOCs) only. It must be prepared prior to the sampling trip. NOTE: A Trip Blank is not required for VOCs in Air.

* *Rinsate or Equipment Blank* -- collected if samples were filtered in the field or otherwise treated by mechanical or physical means to check for carry-over contamination.

* *Temperature Blank* -- placed in cooler whenever samples are required to be kept on ice. This is used for temperature verification only; it is not analyzed.

2 LABORATORY QUALITY CONTROL REQUIREMENTS

The sampler must collect 3 times the minimum volume for at least one sample per batch of 10 inorganic samples or 20 organic samples for each parameter or analytical fraction--to use for quality control (QC) analysis. *It is extremely important that sufficient volume be collected.* (See Section 3.5 for further explanation.)

3 POLLUTION PREVENTION AND HAZARDOUS WASTE REDUCTION

If the sampling requirements (for preservation, sample containers, etc.) are exactly the same, **then one sample can be taken for several parameters in a single container if the volume will accommodate all analyses** (see Section 7.0 for further explanation). It is especially important to consolidate parameters when collecting solid samples because of the difficulty in disposing of the excess sample. Solids should be collected in a single 8 oz. container for either the organic or inorganic parameters. *If a parameter **cannot be combined** with other parameters, it is noted in the table.*

4 COMPOSITING SAMPLES

Normally, samples will not be composited in the laboratory. If it is appropriate or advantageous, compositing in the lab will be considered on a case-by-case basis. Samples for the determination of volatiles are not to be composited in the field because of possible loss of analyte.

5 DISSOLVED ANALYSIS

Samples collected for analysis of dissolved components must be filtered in the field. A filtered Field Blank must also be collected for each new lot of filters. These are considered separate samples from the unfiltered aliquot, so a separate sample number will be needed on the chain-of-custody form.

6 DECHLORINATION

Dechlorinate only those samples which actually contain chlorine. Chlorine presence may be determined using a color wheel or Hach kit.

If any of part of this document is unclear or if you want to verify the requirements, please call the Sample Scheduling Coordinator (SSC) for clarification. It is highly recommended that samples be transported to the laboratory as soon as possible after collection.

TABLE I
Aqueous Sample Requirements ¹

Parameter	Sample Type ^{4,5}	Container Type	Minimum Volume ^{2,3}	Preservation	Holding Time (from time of collection)
Acidity	Grab or Composite	Glass or Plastic	500 ml	cool, ≤ 6 °C	14 days
Alcohols	Grab or Composite	Glass VOA 40-ml vial with Teflon lined cap	10 ml	cool, ≤ 6 °C	7 days
Alkalinity	Grab or Composite	Glass or Plastic	500 ml	cool, ≤ 6 °C	14 days
Anions by IC: Bromide Chloride Fluoride Nitrite Nitrate Ortho Phosphate Sulfate	Grab or Composite	Glass or Plastic	200 ml	cool, ≤ 6 °C	48 hrs for Nitrate, Nitrite and Ortho Phosphate 28 days - all others
Ammonia	Grab or Composite	Glass or Plastic	200 ml	cool, ≤ 6 °C, sulfuric acid to pH<2	28 days
Biochemical Oxygen Demand (BOD)	Grab or Composite	Glass or Plastic	500 ml	cool, ≤ 6 °C	48 hours
Bromide	Grab or Composite	Glass or Plastic	200 ml	cool, ≤ 6 °C	28 days
Carbonaceous Biochemical Oxygen Demand (CBOD)	Grab or Composite	Glass or Plastic	500 ml	cool, ≤ 6 °C	48 hours
Chemical Oxygen Demand (COD)	Grab or Composite	Glass or Plastic	200 ml	cool, ≤ 6 °C, sulfuric acid to pH<2	28 days
Chloride	Grab or Composite	Glass or Plastic	200 ml	none	28 days
Chlorophyll & Pheophytin	Grab	Wrap in aluminum foil and plastic bag. <i>Protect from sunlight!</i>	See method	See method for field filtration/extraction procedure.	Filters: store at -20°C for up to 21 days
Coliform (total & fecal)	Grab	Glass or Plastic, <i>Sterile container</i>	120 ml with 1-inch air space in bottle	cool, ≤ 6 °C, <i>0.2ml/120ml of sterile 10 % sodium thiosulfate</i> ⁶	6 hours

TABLE I
Aqueous Sample Requirements ¹

Parameter	Sample Type ^{4,5}	Container Type	Minimum Volume ^{2,3}	Preservation	Holding Time (from time of collection)
Color	Grab or Composite	Glass or Plastic	200 ml	cool, $\leq 6^{\circ}\text{C}$	48 hours
Corrosivity (pH by 9040)	Grab or Composite	Plastic	200 ml	none	analyze as soon as possible
Total Cyanide	Grab or Composite	Glass or Plastic	200 ml	<p>Superfund: Test for sulfide & oxidizing agents & treat as per ILM05.4, 8.1.1. Add sodium hydroxide to pH>12, cool 4°C</p> <p>NPDES: In the absence of interferences, add sodium hydroxide to pH>12, cool $\leq 6^{\circ}\text{C}$</p> <p>If interferences are present, follow footnotes from Table II, 3/12/07 FR</p>	<p>Superfund: 14 days</p> <p>NPDES: 48 hours if sulfides are present, otherwise 14 days.</p>
1,4 - Dioxane	Grab	Glass VOA 40-ml vial w/Teflon lined cap	120 ml (3 vials filled with no headspace)	cool, $\leq 6^{\circ}\text{C}$	14 days
Diesel Range Organics (DROs)	Grab or Composite	Amber glass bottle w/Teflon lined cap	1000 ml (Cannot combine parameters)	acidify with 1:1 HCl to pH<2; , cool to $\leq 6^{\circ}\text{C}$	14 days
Dissolved Organic Carbon (DOC)	Grab or Composite	Glass or Plastic	100 ml	filter in field, sulfuric acid to pH<2, cool $\leq 6^{\circ}\text{C}$	28 days
Fluoride	Grab or Composite	Glass or Plastic	200 ml	none	28 days
Hardness	Grab or Composite	Glass or Plastic	200 ml	nitric acid to pH<2	6 months
Heterotrophic Plate Count (HPC)	Grab	Glass or Plastic, <i>Sterile container</i>	120 ml with 1-inch air space in bottle	cool, $<10^{\circ}\text{C}$, 0.2ml/120ml of sterile 10 % sodium thiosulfate ⁶	8 hours

TABLE I
Aqueous Sample Requirements ¹

Parameter	Sample Type ^{4,5}	Container Type	Minimum Volume ^{2,3}	Preservation	Holding Time (from time of collection)
Hexavalent Chromium	Grab	Glass or Plastic	200 ml	Field filter, add ammonium sulfate buffer to pH= 9.3 – 9.7, cool $\leq 6^{\circ}\text{C}$ See Footnote 20, Table II, 3/12/07 FR	28 days
HPLC/MS screen	Grab or Composite	Amber glass bottle w/Teflon lined cap	1000 ml (Cannot combine parameters)	cool, $\leq 6^{\circ}\text{C}$	7 days
Ignitability	Grab or Composite	Glass VOA 40-ml vial	240 ml (six 40 mL vials)	none	analyze as soon as possible
Infrared (IR) screen	Grab or composite	Glass or Plastic	100 ml	cool, $\leq 6^{\circ}\text{C}$ – no preservative	analyze as soon as possible
Mercury	Grab or Composite	Glass preferred Plastic ok	200 ml	nitric acid to pH<2 (acid may be added at the lab if safety precautions warrant)	28 days
Metals	Grab or Composite	Glass or Plastic	200 ml	nitric acid to pH<2 (acid may be added at the lab if safety precautions warrant)	6 months
Nitroaromatics + Nitramines, Explosives	Grab or Composite	Amber glass bottle w/Teflon lined cap	1000 ml (Cannot combine parameters)	cool, $\leq 6^{\circ}\text{C}$	7 days
Nitrogen, Nitrate	Grab or Composite	Glass or Plastic	200 ml	cool, $\leq 6^{\circ}\text{C}$	48 hours
Nitrogen, Nitrite	Grab or Composite	Glass or Plastic	200 ml	cool, $\leq 6^{\circ}\text{C}$	48 hours
Nitrogen, Nitrate & Nitrite combined	Grab or Composite	Glass or Plastic	200 ml	cool, $\leq 6^{\circ}\text{C}$ sulfuric acid to pH<2	28 days
Nitrogen, Total Kjeldahl (TKN)	Grab or Composite	Glass or Plastic	200 ml	cool, $\leq 6^{\circ}\text{C}$, sulfuric acid to pH<2	28 days

TABLE I
Aqueous Sample Requirements ¹

Parameter	Sample Type ^{4,5}	Container Type	Minimum Volume ^{2,3}	Preservation	Holding Time (from time of collection)
Total Nitrogen (TN)	Grab or Composite	Glass or Plastic	250 mL	cool, ≤ 6 °C, sulfuric acid to pH<2	28 days
Nitroglycerin	Grab or Composite	Amber glass bottle with Teflon lined cap	1000 ml (Cannot combine parameters)	cool, ≤ 6 °C	7 days
Oil & Grease	Grab	Glass wide-mouth jar (<i>contact lab for jar details</i>)	two - 1000 ml containers (Cannot combine parameters)	cool, ≤ 6 °C, sulfuric or hydrochloric acid to pH<2	28 days
Perchlorate	Grab or Composite	Glass or Plastic	200 mL	none, but must avoid temperature extremes	28 days
Polyaromatic Hydrocarbons (PAHs) by GC/MS	Grab or Composite	Amber glass bottle w/Teflon lined cap	one - 1000 ml containers (Cannot combine parameters)	cool, ≤ 6 °C	7 days
PCB Congeners	Grab or Composite	Amber glass bottle w/Teflon lined cap	two - 1000 mL containers (cannot combine parameters)	cool, ≤ 6 °C	1 year
PCB/Pesticides	Grab or Composite	Amber glass bottle w/Teflon lined cap	one - 1000 ml containers (Cannot combine parameters)	cool, ≤ 6 °C	7 days NPDES Pesticides – if not pH 5-9, Extract within 72 hours
Phenol, Total	Grab or Composite	Glass	1000 ml	Dechlorinate ⁶ with excess ferrous ammonium sulfate, sulfuric acid to ≤ 6 °C, cool, 4°C	28 days

TABLE I
Aqueous Sample Requirements ¹

Parameter	Sample Type ^{4,5}	Container Type	Minimum Volume ^{2,3}	Preservation	Holding Time (from time of collection)
Phosphorus, Ortho	Grab or Composite	Glass or Plastic	200 ml	cool, ≤ 6 °C, must be field filtered for NPDES compliance	48 hours
Phosphorus, Total	Grab or Composite	Glass or Plastic	200 ml	cool, ≤ 6 °C, sulfuric acid to pH<2	28 days
Semivolatiles (SVOAs)	Grab or Composite	Amber glass bottle w/Teflon lined cap	one - 1000 ml containers (Cannot combine parameters)	cool, ≤ 6 °C	7 days
Solids, Total (TS)	Grab or Composite	Glass or Plastic	500 ml	cool, ≤ 6 °C	7 days
Solids, Total Dissolved (TDS)	Grab or Composite	Glass or Plastic	500 ml	cool, ≤ 6 °C	7 days
Solids, Total Suspended (TSS)	Grab or Composite	Glass or Plastic	500 ml	cool, ≤ 6 °C	7 days
Sulfate	Grab or Composite	Glass or Plastic	200 ml	cool, ≤ 6 °C	28 days
Sulfide	Grab	Glass or Plastic	600 ml (2 - 300 ml BOD bottles)	cool, ≤ 6 °C, zinc acetate & sodium hydroxide to pH>9	7 days
TCLP - Semivolatiles (SVOAs)	Grab or Composite	Amber glass bottle w/Teflon lined cap	At least 1000 ml	cool, ≤ 6 °C (unless cooling causes precipitation of the waste)	14 days
TCLP - Herbicides	Grab or Composite	Amber glass bottle w/Teflon lined cap	At least 1000 ml	cool, ≤ 6 °C (unless cooling causes precipitation of the waste)	14 days
TCLP - Metals	Grab or Composite	Glass or Plastic	At least 1000 ml	cool, ≤ 6 °C (unless cooling causes precipitation of the waste)	180 days
TCLP - Mercury	Grab or Composite	Glass or Plastic	At least 1000 ml	cool, ≤ 6 °C (unless cooling causes precipitation of the waste)	28 days

TABLE I
Aqueous Sample Requirements ¹

Parameter	Sample Type ^{4,5}	Container Type	Minimum Volume ^{2,3}	Preservation	Holding Time (from time of collection)
TCLP - Pesticides	Grab or Composite	Amber glass bottle w/Teflon lined cap	At least 1000 ml	cool, ≤ 6 °C (unless cooling causes precipitation of the waste)	14 days
TCLP/ZHE - Volatiles (VOAs)	Grab	Glass VOA 40 - ml vial w/Teflon lined cap	160 ml (4 vials filled with no headspace)	cool, ≤ 6 °C	14 days
Total Toxic Organics (TTO) Customer to supply compound lists	Follow requirements for PCB/Pests, SVOAs, and VOAs.	Follow requirements for PCB/Pests, SVOAs, and VOAs.	Follow requirements for PCB/Pests, SVOAs, and VOAs.	Follow requirements for PCB/Pests, SVOAs, and VOAs.	Same as for PCB/Pests, SVOAs, and VOAs.
Total Organic Carbon (TOC)	Grab or Composite	Glass or Plastic	100 ml	cool, ≤ 6 °C, sulfuric acid to pH<2	28 days
Volatiles (VOAs)	Grab	Glass VOA 40-ml vial w/Teflon lined cap	120 ml (3 vials filled with no headspace)	cool, ≤ 6 °C Dechlorinate ⁶ with sodium thiosulfate (10mg/40ml) BEFORE acidifying with 1:1 hydrochloric acid to pH<2. For RCRA and NPDES, samples must be submitted both preserved and unpreserved <i>if 2-chloroethyl vinyl ether is an analyte of interest</i> . This is due to losses of 2-chloroethyl vinyl ether in acidified samples.	7 days unpreserved 14 days preserved

TABLE II
Non-Aqueous Sample Requirements ¹

Parameter	Sampling Type ⁴	Container Type	Appropriate Weight ^{2,3}	Preservation	Holding Time (from time of collection)
Anions by IC: Bromide Chloride Fluoride Nitrite Nitrate Ortho Phosphate Sulfate	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, ≤ 6 °C	none
Benthic Invertebrate Taxonomy	Grab	Glass	N/A	70% ethanol	none
Chemical Oxygen Demand (COD)	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, ≤ 6 °C	28 days
Cyanide, Total	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, ≤ 6 °C	28 days
Diesel Range Organics (DROs)	Grab or Composite	Amber Glass (4 oz. wide- mouth w/Teflon lined lid)	50 g	cool, ≤ 6 °C	14 days
Dry Weight, Percent	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	none	none
Grain Size	Grab	Heavy plastic bag or glass jar	500 g	cool, ≤ 6 °C	none
Hexavalent Chromium	Grab	Glass (4 oz. wide- mouth)	50 g	cool, ≤ 6 °C	30 days
HPLC/MS screen	Grab or Composite	Amber Glass (4 oz. wide- mouth w/Teflon lined lid)	50 g	cool, ≤ 6 °C	14 days
Infrared (IR) Screen	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, ≤ 6 °C	see parameter of interest
Mercury	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, ≤ 6 °C	28 days
Metals	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, ≤ 6 °C	6 months

TABLE II
Non-Aqueous Sample Requirements ¹

Parameter	Sampling Type ⁴	Container Type	Appropriate Weight ^{2,3}	Preservation	Holding Time (from time of collection)
Metals in Wipes	Grab	Plastic digestion tube provided by OASQA	NA	None Include 5 unused wipes for lab QC	6 months
Metals in Air Filters	Grab	Sealed plastic bag	NA	None Include 5 unused filters for lab QC	6 months
Metals in Dust	Grab	Vacuum bag	NA	None	6 months
Nitroaromatics + Nitramines, Explosives	Grab or Composite	Amber Glass (4 oz. wide- mouth w/Teflon lined lid)	50 g	cool, $\leq 6^{\circ}\text{C}$	14 days
Nitrogen, Total Kjeldahl (TKN)	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, $\leq 6^{\circ}\text{C}$	28 days
Nitroglycerin	Grab or Composite	Amber Glass (4 oz. wide- mouth w/Teflon lined lid)	50 g	cool, $\leq 6^{\circ}\text{C}$	14 days
Paint Filter Test	Grab	Glass or plastic	500 g	cool, $\leq 6^{\circ}\text{C}$	none
Perchlorate	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, $\leq 6^{\circ}\text{C}$	none
Polyaromatic Hydrocarbons (PAHs) by GC/MS	Grab or Composite	Amber Glass (4 oz. wide- mouth w/Teflon lined lid)	50 g	cool, $\leq 6^{\circ}\text{C}$	14 days
PCB Congeners	Grab or Composite	Amber Glass (4 oz. wide- mouth w/Teflon lined lid)	50 g	cool, $\leq 6^{\circ}\text{C}$	1 year
PCB/Pesticides	Grab or Composite	Amber Glass (4 oz. wide- mouth w/Teflon lined lid)	50 g	cool, $\leq 6^{\circ}\text{C}$	14 days
PCBs in oil	Grab	Glass VOA 40-ml vial w/Teflon lined cap	10 g	cool, $\leq 6^{\circ}\text{C}$	none

TABLE II
Non-Aqueous Sample Requirements ¹

Parameter	Sampling Type ⁴	Container Type	Appropriate Weight ^{2,3}	Preservation	Holding Time (from time of collection)
pH by 9045C	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	none	Analyze as soon as possible
Phenol, Total	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, ≤ 6 °C	28 days
Phosphorus, Total	Grab or Composite	Glass (4 oz. wide-mouth)	50 g	cool, ≤ 6 °C	28 days
Semivolatiles (SVOAs)	Grab or Composite	Amber Glass (4 oz. wide-mouth w/Teflon lined lid)	50 g	cool, ≤ 6 °C	14 days
TCLP - Semivolatiles (SVOAs)	Grab or Composite	Amber Glass (8 oz. wide-mouth w/Teflon lined lid)	200 g	cool, ≤ 6 °C	14 days
TCLP - Herbicides	Grab or Composite	Amber Glass (8 oz. wide-mouth w/Teflon lined lid)	200 g	cool, ≤ 6 °C	14 days
TCLP - Mercury	Grab or Composite	Glass (8 oz. wide-mouth)	200 g	cool, ≤ 6 °C	28 days
TCLP - Metals	Grab or Composite	Glass (8 oz. wide-mouth)	200 g	cool, ≤ 6 °C	180 days
TCLP - Pesticides	Grab or Composite	Amber Glass (8 oz. wide-mouth w/Teflon lined lid)	200 g	cool, ≤ 6 °C	14 days
TCLP/ZHE - Volatiles (VOAs)	Grab	Glass VOA 40-ml vial w/Teflon lined cap	160 g (4 vials filled with no headspace)	cool, ≤ 6 °C	14 days
Total Organic Carbon (TOC)	Grab	Glass (8 oz. wide-mouth)	50 g	cool, ≤ 6 °C	none
Volatiles (VOAs)	Grab	See attachment 5	See Attachment 5	See Attachment 5	48 hrs to preserve or analyze

TABLE II**Non-Aqueous Sample Requirements ¹**

Parameter	Sampling Type ⁴	Container Type	Appropriate Weight ^{2,3}	Preservation	Holding Time (from time of collection)
Volatile Organic Compounds (VOCs) in air (TO14a/15)	Grab or Composite	Summa canister supplied by lab	6 L	none	30 days

TABLE III
SDWA Program Special Requirements ^{1, 6}

Parameter	Sampling Type ^{4,5}	Container Type	Minimum Volume ^{2,3}	Preservation	Holding Time (from time of collection)
Coliform (total & fecal)	Grab	Glass or Plastic, <i>Sterile</i>	120 ml with 1-inch air space in bottle	cool, <10°C, 0.2ml/120ml of sterile 10 % sodium thiosulfate ⁶	30 hours
Heterotrophic Plate Count (HPC)	Grab	Glass or Plastic, <i>Sterile container</i>	120 ml with 1-inch air space in bottle	cool, <10°C, 0.2 ml/120ml of sterile 10% sodium thiosulfate ⁶	8 hours
Nitrate (NO ₃ -N)	Grab or Composite	Glass or Plastic	200 ml	From a chlorinated source and cooled to ≤6 °C	14 days
Pesticides	Grab or Composite	Amber glass bottle w/Teflon lined cap	two - 1000 ml containers (Cannot combine parameters)	cool, ≤6 °C, 6 N HCl to pH ≤ 2, dechlorinate ⁶ w/50 mg sodium sulfite	7 days
Total Trihalomethanes (TTHMS)	Grab	Glass VOA 40-ml vial w/Teflon lined cap	120 ml (3 vials filled with no headspace)	cool, ≤6 °C, dechlorinate ⁶ with 3 mg/40ml sodium thiosulfate	14 days
Volatiles (VOAs)	Grab	Glass VOA 40-ml vial w/Teflon lined cap	120 ml (3 vials filled with no headspace)	cool, ≤6 °C, dechlorinate ⁶ with either 25mg/40ml ascorbic acid OR 3mg/40ml sodium thiosulfate BEFORE acidifying with 1:1 hydrochloric acid to pH<2. Note: If residual chlorine is measured in the field at >5mg/L, to each vial add an additional 25 mg of ascorbic acid OR 3 mg of sodium thiosulfate per each increment of 5 mg/L residual chlorine. Avoid adding excess dechlorination agent.	14 days preserved

Attachment 1 - Example Chain-of-Custody

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

CHAIN OF CUSTODY RECORD

REGION 3
Curtis Bldg., 6th & Walnut Sts.
Philadelphia, Pennsylvania 19106

PROJ. NO. DAS or NSF-E		PROJECT NAME SITE NAME				NO. OF CON- TAINERS	REMARKS									
SAMPLERS: (Signature) JANE DOE / Jane Doe																
STA. NO.	DATE 2005	TIME	COMP.	GRAB	STATION LOCATION		TAG #s									
1A	3/28	11:00		X	Upstream A	3	X	X	X						5951-5953	5951-HNO ₃
1A-D	3/28	11:00		X	upstream A-dup	3	X	X	X						5954-5956	5954-HNO ₃
2A	3/28	8:00	X		downstream A	1		X							5957	8-hr comp 8:00 thru 15:00
EQB	3/28	18:00		X	EQUIPMENT BLANK	1		X							5958	
Relinquished by: (Signature) John Doe			Date / Time 3/29/05 7:00		Received by: (Signature)			Relinquished by: (Signature)			Date / Time		Received by: (Signature)			
Relinquished by: (Signature)			Date / Time		Received by: (Signature)			Relinquished by: (Signature)			Date / Time		Received by: (Signature)			
Relinquished by: (Signature)			Date / Time		Received for Laboratory by: (Signature)			Date / Time		Remarks						

Distribution: Original Accompanies Shipment; Copy to Coordinating Field Office

Attachment 2 – Example Sample Tag

Project Code DAS or NSF #	Station No. 1A	Month/Day/Year 3/28/05	Time 11:00	Designate		Preservative: Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>
				Comp.	Grab <input checked="" type="checkbox"/>	
Station Location Upstream A				Samplers (Signatures) <i>Jane Doe</i>		ANALYSES
				BOD		
				Solids (TSS) (TDS) (SS)		
				COD, TOC, Nutrients		
				Phenolics		
				Mercury		<input checked="" type="checkbox"/>
				Metals		
				Cyanide		
				Oil and Grease		
				Organics GC/MS		
				Priority Pollutants		
				Volatile Organics		
				Pesticides		
				Mutagenicity		
				Bacteriology		
Remarks:						
HNO ₃ preservative						
Tag No.				Lab Sample No.		
3065951						

Attachment 3 – Example Hazard and Risk Exposure Data Sheet

**Region III, Office of Analytical Services and Quality Assurance
Ft Meade, Maryland
HAZARD AND RISK-EXPOSURE DATA SHEET
LEVELS OF PERSONAL PROTECTION DURING SAMPLING**

BACKGROUND

Under the authority Section 104 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) of 1980, Section 311 of the Clean Water Act, and Subtitle I of the Resource Conservation and Recovery Act (RCRA), EPA has been delegated the responsibility to undertake response actions with respect to the release or potential release of oil, petroleum, or hazardous substances that pose a substantial threat to human health or welfare, or the environment.

GENERAL

This form is to be used when collecting Environmental Samples (i.e. streams, farm ponds, wells, soils etc.) and for Hazardous Sample (i.e. drums, storage tanks, lagoons, leachates, hazardous waste sites). This information is intended for use as a guide for the safe handling of these laboratory samples in accordance with EPA and OSHA regulations. The sample classification(s) and levels of personal protection used by the sampler in all situations will enable the analyst to be better aware of potential exposure to substances in air, splashes of liquids, or other direct contact with material due to work being done.

DEGREE OF PROTECTION

- _____ Level A: Highest level of respiratory, skin, and eye protection needed. Fully encapsulated suit, respirator self-contained (Tank type).
- _____ Level B: Highest level of respiratory protection but lesser level of skin protection needed. Chemical suit, respirator self-contained (Tank type).
- _____ Level C: Lesser level of respiratory protection than Level B. Skin protection criteria are similar to Level B. Chemical suit, canister respirator/cartridge
- _____ Level D: Work uniform without any respirator or skin hazards. Lab coat, gloves etc.

CLASSIFIED FIELD SAMPLES

_____ Environmental _____ Hazardous _____ Comb. (Env. & Haz.) _____ Radioactive

Site Name: _____ Sampling Date: _____

Sta No. _____/_____/_____/_____/_____/_____

Field pH: _____/_____/_____/_____/_____/_____
(must be taken prior to submission of aqueous samples)

Sampler: _____ Work Phone Number: _____

Personal observations at time of sampling (surroundings):
Sample collection observations (physical sample, odors etc.):

Attachment 4 - OASQA Sample Acceptance Policy

OASQA Sample Acceptance Policy

The following are those conditions which indicate that the integrity of the sample may have been jeopardized, either during the actual sampling event or during its shipment to the lab. If one or more of these conditions exist, the laboratory will consult with the requester to determine whether to reject the sample(s) for analysis or to perform a qualified analysis. If the sample(s) is (are) rejected, the sampler will be given the opportunity to resample. If the analysis is performed, the data will be reported with qualifiers explaining why the data may have been compromised, and the potential impact on the data. In some cases it may be possible for the laboratory to complete the analysis with some alteration. For example, if there is insufficient sample volume, the data may have to be reported with increased quantitation limits or the requestor may be asked to prioritize analytical requests.

The condition of sample(s) and shipment will be documented on the OASQA Shipment Documentation Form. The Sample Scheduling Coordinator may request a Letter-To-File from the sampler to document additional critical details. Any actions taken because of the compromised condition of a sample will be noted in the laboratory's information management database and in the report narrative sent the requester.

Conditions which may jeopardize the integrity of the sample:

- Not collected in appropriate containers.
- If cooling is required for the requested analytes, samples are received at greater than 6 degrees C or missing the temperature blank.
- Not properly preserved as outlined in Tables I, II, and/or III of the OASQA Sample Submission Procedures.
- Received past the analytical holding time.
- Samples tampered with during shipment. (Example: custody seal has been broken)
- Insufficient sample to perform sample analysis or the quality control analysis.
- Sample identification incorrect, incomplete, or missing.
- Chain-of-custody documentation not available, inaccurate or incomplete.
- Samples inappropriate for requested analysis. (Example: decomposed condition)
- Leaking or broken container.
- Lack of a trip blank with samples collected for volatile analysis.

Note: One other condition which would cause samples to be rejected by OASQA is if the samples are suspected to contain dioxin. At this time, this facility is not prepared to handle the potential hazard of dioxin contamination.

Attachment 5 - Region III 5035A Fact Sheet**Region III 5035A Fact Sheet**

May 15, 2003, Revision No: 2

Field Samplers Guide to the Collection and Handling of Soil Samples for Volatile Organic Analysis using SW 846 Method 5035A**Summary:**

The purpose of this fact sheet is to specify procedures for the collection and handling by **field samplers** of soil samples for volatile organic analysis (VOA) in Region III. SW-846 Method 5035A is the collection method required for analysis of soil samples for VOA. This method incorporates chemical preservatives and sample storage techniques to limit volatilization and biodegradation of organic compounds. Method 5035A is applicable to both low/medium and high level soil samples.

Collection Procedures:

- Soil samples being analyzed for volatile organic compounds collected via Method 5035A should not be chemically preserved in the field.
- Samples should be collected using the following collection options:

Option 1: For most Soil types

Number of samples: 4 EnCore (or similar closed-sampling vessel)¹ samplers
4 QC EnCore samplers
1 40 mL vial for moisture analysis

¹EnCore samplers (or similar sample collection device, refer to Section 4.5 of Method 5035)

Samples must be cooled to 4°C upon collection and during shipment and bagged individually upon collection.

Samples must arrive at the laboratory within 24 hours.

Samples must be analyzed or preserved by the lab within 48 hours of collection.

Option 2: For Non-Cohesive Granular Material (wet, rocky, sediments, etc.)

Number of samples: 4 40mL vials (sampler may use wide mouth jars if sample not amenable to smaller vials)
2 QC 40 mL vials
1 40 mL vial for moisture analysis

Samples must be cooled to 4°C upon collection and during shipment.

Samples must arrive at the laboratory within 24 hours.

Samples must be analyzed or preserved by the lab within 48 hours of collection.

