

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

STANDARD REFERENCES FOR MONITORING WELLS
SECTION 6.1 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

SECTION 6.1
QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

TABLE OF CONTENTS

Section	Title	Page No.
6.1-1	DEFINITIONS	1
6.1-2	QA/QC Plan	2
6.1-3	FIELD QC SAMPLES	2
6.1-3.1	Trip Blanks	3
6.1-3.2	Equipment Rinsates	3
6.1-3.3	Field Blanks and Duplicates	3
6.1-4	SAMPLE CONTAINER REQUIREMENTS.....	3
6.1-4.1	Preparation of Sample Containers	4
6.1-4.1.1	Preparation of Containers for Semi-Volatile Organic Analytes	4
6.1-4.1.2	Preparation of Containers for Metals and Cyanide Analytes	4
6.1-4.1.3	Preparation of Containers for Volatile Organic Analytes	5
6.1-5	PREPARATION OF PUMPING TUBING.....	5
6.1-6	FORMS AND RECORDS.....	6

APPENDIX

Appendix	Title	Page No.
A	Quality Assurance/Quality Control Checklist and Form for Sampling Monitoring Wells.....	7

6.1 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

6.1-1 DEFINITIONS

The following definitions are used in Sections 6.1-6.5, inclusive.

Accuracy - the degree of agreement of a measurement with an accepted reference value. Accuracy is generally reported as percent recovery, and calculated as:

$$\frac{\text{Measured Value}}{\text{Accepted Value}} \times 100$$

Analyte - the chemical or property for which a sample is analyzed.

Comparability - the expression of information in units and terms consistent with reporting conventions; the collection of data by equivalent means; or the generation of data by the same analytical method. Aqueous samples shall be reported as g/l solid samples shall be reported in units of mg/kg, dry weight.

Completeness - the percentage of valid data obtained relative to that which would be expected to be obtained under normal conditions. Data are judged valid if they meet the stated precision and accuracy goals.

Duplicate - two separate samples taken from the same source by the same person at essentially the same time and under the same conditions that are placed into separate containers for independent analysis.

Episode - a continuous period of time during which sampling activities are undertaken. Cessation of activities for more than 48 hours terminates the episode.

Precision - a measure of the agreement among individual measurements of the same property under prescribed similar conditions. Precision is generally reported as Relative Standard Deviation (RSD) or Relative Percent Difference (RPD). Relative standard deviation is used when three or more measurements are available and is calculated as:

$$\text{RSD} = \frac{\text{Standard Deviation}}{\text{Arithmetic Mean}} \times 100$$

Relative percent difference is used for duplicate measurements and is calculated as:

$$\text{RPD} = \frac{\text{Value 1} - \text{Value 2}}{\text{Arithmetic Mean of Values 1 and 2}} \times 100$$

Quality Assessment - the overall system of activities that provides assurance that quality control activities are done effectively.

Quality Assurance (QA) - all the means taken inside and outside the laboratory to make certain that all laboratories use the same calibration and standardization procedures for reporting results; also, a program which integrates the quality planning, quality assessment, and quality improvement activities within an organization.

Quality Control (QC) - all the means taken by an analyst to ensure that the total measurement systems are calibrated correctly. It is achieved by using EPA reference standards, duplicates, replicates, and sample spikes. Also, the routine application of procedures designed to ensure that the data produced achieve known limits of precision and accuracy.

Replicate - two aliquots taken from the same sample container and analyzed separately. Where replicates are impossible, as with volatile organics, duplicates must be taken.

Rinse - fill container with approximately one-quarter of its total volume, cap and make certain that the rinsate makes contact with all interior surfaces.

6.1-2 QA/QC PLAN

Prior to the initiation of monitoring well sampling activities, a QA/QC Plan will be prepared. The purpose of this document is to provide to on-site personnel an immediate source of information relevant to the work to be undertaken, as well as to initiate the planning and logistics required for a successful sampling episode.

The plan will include, as a minimum:

- Site identity
- Project organization and responsibilities
- Laboratory/shipping information
- Project and data quality objectives
- Chain of custody and sample identification procedures
- Parameters for analysis
- Analytical program with identification of analytical methods
- Field QC samples required
- Sampling and preservation procedures
- List of required equipment
- Equipment decontamination sequence and location
- Calibration of field measurement equipment
- Data quality requirements and assessments
- Signature Block

A sample format, for such a checklist and form, is provided in Appendix A.

6.1-3 FIELD QC SAMPLES

A variety of QC samples are collected in the field and submitted for laboratory analysis. These samples are intended to assess the effectiveness of equipment decontamination, the precision of sampling efforts, the effects of ambient environmental conditions on sensitive analytes (e.g., volatile organics analysis or VOA), and the potential for contaminants attributable to reagents or decontamination fluids. Identifying such potential sources of error is essential to the success of the sampling program and the validity of the environmental data. Each QC sample is described below. As a minimum, each set of ten or fewer field samples should include a trip blank, a duplicate and one sample collected in a sufficient volume to allow the laboratory to perform a matrix spike.

6.1-3.1 Trip Blanks

Trip blanks are samples that originate from analyte-free water taken from the laboratory to the sampling site and returned to the laboratory with the volatile organic samples. One trip blank should accompany each cooler containing volatile organics (VOAs); it should be stored at the laboratory with the samples, and analyzed with the sample set. Trip blanks are only analyzed for VOAs.

6.1-3.2 Equipment Rinsates

Equipment rinsates (sometimes referred to as "equipment blanks" or "sampler blanks") are the final analyte-free water rinse from equipment decontamination in the field and are collected at least once during a sampling episode. If analytes pertinent to the project are found in the rinsate, the results from the blanks will be used to qualify the levels of analytes in the samples. This qualification is made during data validation. The rinsates are analyzed for the same analytes as the samples that have been collected with that equipment. If dedicated sampling equipment is used, this protocol becomes redundant.

6.1-3.3 Field Blanks

Field blanks, also known as source water samples, are samples of the water used in decontamination and steam cleaning in the field. At a minimum, one sample from each episode and each source of water will be collected.

6.1-3.4 Field Replicates and Duplicates

Field Replicates for water samples, except VOA samples, are collected, homogenized, and then split. VOA samples are not mixed, but taken as grab samples. The replicates for water samples should be collected sequentially. Field replicates should be collected at a frequency of 10% per sample matrix (i.e. water or soil) or one replicate for every 10 samples.

To maximize data utility when sampling for analysis by USEPA's Contract Laboratory Program (CLP), which is generally used for litigative quality investigations, the same samples used for field replicates should be taken in sufficient volume to be split by the laboratory and be used as the laboratory replicate or matrix spike. This means that for designated samples, there will need to be a volume sufficient for the normal sample analysis, the field duplicate analysis, and the laboratory matrix spike/ matrix spike duplicate analysis (i.e., up to three times the single sample volume).

Field duplicates are a second aliquot of a sample taken in the field that is treated the same as the original sample in order to determine the precision of the method. They shall be analyzed with every analytical batch or every 20 samples, whichever is greater. This procedure is applicable to all organic and inorganic chemical analytes.

6.1-4 SAMPLE CONTAINER REQUIREMENTS

Sample integrity is assured by use of containers appropriate to both the medium/matrix to be sampled and the analytes of interest. For example, samples intended for semi-volatile organic analyte (SVOA) analyses are collected in glass bottles with teflon-lined caps; samples for volatile organic analyte (VOA) analyses are collected in teflon-septum-capped glass vials with "zero" headspace to minimize diffusive and evaporative losses; and most samples for inorganic analyses are collected in linear polyethylene bottles. Sample containers must be prepared in the laboratory in a manner consistent with USEPA protocols. DEP-approved preparation methods are described below. Bottles may also be purchased precleaned and QC-checked from commercial suppliers. It is generally less expensive and more efficient to purchase the precleaned containers. If precleaned containers are used, the lot number should be reported on the chain-of-custody.

6.1-4.1 Preparation of Sample Containers

Containers should be cleaned based on the analyte of interest. Bottles used to collect hazardous wastes are generally only used once and then discarded.

6.1-4.1.1 Preparation of Containers for Semi-volatile Organic Analyte

Included in this section as semi-volatile organics are: base-neutral extractables, PCBs, pesticides and herbicides. Requires 2-liter amber glass bottles for water samples and 8 oz. clear glass jars for soil, sediment, and sludge samples.

1. Wash containers, closures, and teflon-lined caps in hot tap water with laboratory grade non-phosphate detergent (e.g. Alconox or equivalent).
2. Rinse three times with tap water.
3. Rinse three times with ASTM Type I deionized water.
4. Rinse with technical-grade acetone.
5. Rinse with pesticide grade hexane.
6. Air dry in a contaminant-free environment to get rid of any vapors.

7. Oven dry the glass containers only (1 hour at 105° C).
8. Remove glass containers from oven.
9. Loosely screw teflon-lined caps on containers. Attendant to wear gloves to prevent recontamination; containers not to be removed from preparation room until sealed.

6.1-4.1.2 Preparation of Containers for Metals and Cyanide Analytes

For metals: requires 1-liter clear glass or polyethylene bottles for water samples and 16 oz. clear glass or polyethylene jars for soil, sediment, and sludge samples. For cyanides: requires 1-liter amber glass or polyethylene bottles for water samples and 16 oz. amber glass or polyethylene jars for soil, sediment or sludge samples.

1. Wash bottles and closures in hot tap water with laboratory grade non-phosphate detergent (e.g. Alconox).
2. Rinse three times with tap water.
3. Rinse with 1:1 nitric acid.
4. Rinse three times with tap water.
5. Rinse with 1:1 hydrochloric acid.
6. Rinse three times with ASTM Type II deionized water.
7. Air dry in contaminant-free environment.
8. Place closures on bottles. Attendant to wear gloves to prevent recontamination; bottles not to be removed from preparation room until sealed.

6.1-4.1.3 Preparation of Containers for Volatile Organic Analytes

Requires (2) 40-ml glass vials per water sample and 8-oz wide mouth glass jars with teflon liner for concentrated waste samples.

1. Wash vials, septa, teflon liners, and closures in hot tap water with laboratory grade non-phosphate detergent (e.g. Alconox).
2. Rinse three times with tap water.
3. Rinse three times with ASTM Type I deionized water.
4. Oven dry (in a muffle furnace) the glassware only (1 hour at 105° C).
5. Rinse septa, teflon liners, and closures in methanol.
6. Air dry septa, teflon liners, and closures in a contaminant-free environment.

7. Remove vials and jars from oven.
8. Place septa in closures, teflon side down to face the sample, and place on vials. Put teflon-lined caps on jars. Attendant to wear gloves; vials and jars not to be removed from preparation room until sealed.

6.1-5 PREPARATION OF PUMP TUBING

Adequate lengths of 3/8-inch ID teflon tubing and/or 3/8-inch ID silicone tubing will be prepared by the sampling crew for each sampling episode which requires tubing. Teflon tubing is preferred and can be reused. If tubing shows wear, then discard. A specific procedure for preparing teflon tubing for VOA analysis follows.

The VOA Teflon tubing preparation procedure is as follows:

1. Pump non-phosphate detergent solution (e.g. Alconox) through system for two minutes.
2. Pump clean hot tap water through system for two minutes or until clear, whichever is longer.
3. Pump technical grade acetone through system for two minutes.
4. Pump pesticide grade hexane through system for two minutes.
5. Pump ASTM Type II deionized water through system for five minutes.
 6. Seal tubing ends, close with teflon caps (no wrapping with plastic wraps or "baggies"), and label with date of cleaning.

A general (suitable also for silicone) tubing preparation procedure is as follows:

1. Pump non-phosphate detergent solution through system for two minutes.
2. Pump clean hot tap water through system for two minutes or until clear, whichever is longer.
3. Pump analyte-free water through system for two minutes.
4. Pump decontamination fluid specified in the site-specific Quality Assurance Plan through system for two minutes.
5. Pump analyte-free water through system for two minutes.
6. Seal tubing ends; wrap (no "saran wrap" or "baggies") and label with date of cleaning.

6.1-6 FORMS AND RECORDS

Documentation of the activities surrounding well purging sample collection, sample preservation, chain of custody, and equipment calibration is critical to subsequent data evaluation and utility. Field data must be recorded in ink in a bound field notebook. Data may be transferred to forms such as those shown in Section 6.2 for ease of calculation and filing.

APPENDIX A

QUALITY ASSURANCE/QUALITY CONTROL CHECKLIST
AND FORM FOR SAMPLING MONITORING WELLS

Quality Assurance/Quality Control Checklist
And Form For Sampling Monitoring Wells

A. Project Identity, Organization and Responsibilities

1. Project Identity

Project Name: _____ Project ID Number: _____
Date of
Address: _____ Sampling: _____

Map Coordinates: _____ UTM or Lat/Long
(circle one)

Site Description: _____

2. Personnel (contact name, firm and telephone number)

Project/site manager: _____

Health and Safety Officer: _____

Quality Assurance Officer: _____

Field Leader: _____

Sampling Personnel: _____

Well Driller: _____

Consultant Firm: _____

Laboratory Performing Analysis: _____

Laboratory Shipping Address: _____

B. Project and Data Quality Objectives

1. Project Description

Objective and Scope Statement: _____

Monitoring Network Design and Rational: _____

Health and Safety Plan to be used at Site: _____

Level A, B, C, or D: _____ Level

2. Data Quality Objectives

Objective of Field Sampling/Data Usage: _____

C. Chain of Custody and Sample Identification Procedures

Chain of Custody Required: _____ (yes or no)

Sample Custody Procedures: _____

Sample Identification Procedures: _____

D. Parameters for Analysis

Monitoring Parameters and Their Frequency of Collection

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

E. Parameter Table

<u>Parameter</u>	<u>Number of Samples</u>	<u>Sample Matrix</u>	<u>Analytical Method Ref</u>	<u>Sample Preservation</u>	<u>Holding Time</u>
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

F. Number of Samples, Blanks, Duplicates, and Matrix Spikes

<u>Matrix: Parameter</u>	<u>Number of Samples</u>	<u>Trip Blanks¹</u>	<u>Equip Rinsates²</u>	<u>Field Blank³</u>	<u>Field Replicates⁴</u>	<u>MS/MSD⁵</u>	<u>Total</u>
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____

- 1 Trip Blanks: One trip blank per cooler containing VOAs
- 2 Equipment Rinsates: At least one per sampling episode
- 3 Field Blanks: At least one per sampling episode and per source of water
- 4 Field Replicates: One for every ten samples
- 5 MS/MSD: Matrix spike and matrix spike duplicates samples are collected at a rate of one each per 20 samples

G. Sampling, Preservation, and Decontamination Procedures

1. Sampling Procedures: _____

2. Preservation Procedures: _____

3. Decontamination Sequence/Procedures: _____

Decontamination Location(s): _____

4. Calibration Procedures and Preventive Maintenance: _____

H. Data Quality Requirements and Assessments

<u>Parameter</u>	<u>Sample Matrix</u>	<u>Detection Limit</u>	<u>Quantitation Limit</u>	<u>Estimated Accuracy</u>	<u>Accuracy Protocol</u>	<u>Estimated Precision</u>	<u>Precision Protocol</u>
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____	_____

Data Representativeness: _____

H. Data Quality Requirements and Assessments (cont.)

Data Comparability: _____

Data Documentation: _____

Data Reduction and Reporting: _____

Data Validation: _____

I. Signature Block

Project Manager:

(print) (signature) (date)

Quality Assurance Manager:

(print) (signature) (date)

Field Leader:

(print) (signature) (date)

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

STANDARD REFERENCES FOR MONITORING WELLS

SECTION 6.2 SAMPLING TECHNIQUES

SECTION 6.2
SAMPLING TECHNIQUES

TABLE OF CONTENTS

Section	Title	Page No.
6.2-1	INITIAL SETUP/EQUIPMENT CALIBRATION	1
6.2-1.1	Specific Conductance-Temperature Meter (based on Y.S.I. S-C-T Meter, Model No. 33).....	2
6.2-1.1.1	Temperature Probe.....	2
6.2-1.1.2	Specific Conductance Meter	2
6.2-1.2	Calibration of Specific Ion Meter (pH/Eh Measurement)	3
6.2-1.2.1	pH Probe	3
6.2-1.2.2	Eh/Platinum Probe	3
6.2-1.3	Calibration of Photoionization Meters	4
6.2-1.3.1	HNU	4
6.2-1.3.2	Photovac T.I.P	5
6.2-1.4	Calibration of Flame Ionization Detectors.....	5
6.2-1.4.1	OVA	5
6.2-2	WELL PURGING	6
6.2-3	SELECTION OF SAMPLING EQUIPMENT	7
6.2-3.1	Bailers.....	8
6.2-3.2	Submersible Pumps.....	9
6.2-3.3	Peristaltic Pumps	10
6.2-3.4	Bladder Pumps	10
6.2-4	GROUNDWATER SAMPLING	10
6.2-4.1	Procedures Applicable Only to Collection of Ground Water Samples for Volatile Organic Analysis	10
6.2-4.2	Procedures Applicable Only to Collection of Ground Water Samples for Metals Determination.....	11
6.2-4.3	Procedures for Collection of Ground Water Samples.....	11
	REFERENCES	13

LIST OF FIGURES

<u>Figures</u>	<u>Title</u>	<u>Page No.</u>
6.2-1	Field Instrumentation Quality Assurance Record	15
6.2-2	Modified Kemmerer Sampler	16
6.2-3	Teflon or Stainless Steel Bailer	17
6.2-4	Volatile Organic Collection Bottle	18
6.2-5	Sample Data Record	19

6.2 SAMPLING TECHNIQUES

6.2-1 INITIAL SETUP/EQUIPMENT CALIBRATION

Monitoring of ground water wells should proceed from the upgradient or background wells to the downgradient or contaminated wells as best as can be determined.

The area immediately surrounding the well should be cleared of debris and the ground surface covered with plastic sheeting to minimize contact of instruments with surface soils. The monitoring procedure is as follows:

1. Check the well for proper identification and location.
2. Measure and record the height of the protective casing above the ground.
3. After unlocking the well and removing any well caps, measure and record the ambient and well-mouth organic vapor levels using a Photoionization detector (PID). If a check for methane gas is required, then the use of a Flame Ionization Detector (FID) is necessary. If the ambient air quality at breathing level reaches an action level as described in the Health and Safety Plan, the sampler shall utilize the appropriate safety equipment.
4. Measure and record the distance between the top of the uncapped well and the top of the protective casing.
5. Measure from the top of the well casing to the static water level with accuracy to the nearest 0.01 foot. Then measure and record the depth to the bottom of the well. Upon removing the water level measuring device, rinse it with the decontamination fluid specified for the sampling episode and then either potable or deionized (DI) water, as specified for the episode.
6. Calculate the volume of standing water in the well by first determining the area:

$$(1) \quad \text{Area} = \pi r^2$$

where,

$\pi = 3.142$, and
 r = radius, measured as the diameter of the well
divided by 2

Calculate the volume of water in the well using the formula:

$$(2) \quad \text{Volume} = \text{Area} \times h$$

where,

Area = surface calculated according to equation (1),
and,

h = the distance between the water surface in the well and the bottom of the well.

Calibrate each piece of instrumentation prior to each day's use or as specified by its manufacturer. If possible, gross calibration may best be accomplished prior to the instrument leaving the office and then rechecked and fine-tuned on each day of sampling in the field. Each instrument should be recalibrated in the field on an as-needed basis. The recalibration frequency will depend on the number of measurements made, the time between measurements, and the type of samples. The operator's experience is also critical. Recalibration may be necessary before each measurement until the operator is confident that a particular instrument is stable.

Data are recorded, in ink in a field notebook and may be transferred to a form similar to that shown in Figure 6.2-1. The procedures described below apply to the specific instrument noted. If other instruments are used, similar procedures must be developed or the manufacturer's calibration procedures followed.

6.2-1.1 Specific Conductance Temperature Meter (based on Y.S.I. S-C-T Meter, Model No. 33)

6.2-1.1.1 Temperature Probe

1. Using a National Bureau of Standards-approved thermometer, immerse both temperature probes into a beaker of water and note any differences for the field probe.
2. Recalibrate as necessary.
3. Document calibration in a field notebook.

6.2-1.1.2 Specific Conductance Meter

1. Calibrate meter and probe using the calibration control and the red-line on the meter dial (Y.S.I. S-C-T Meter, Model No. 33).
2. Turn the function switch to read conductivity x10 and then depress the cell test button, noting the deflection. If the needle falls more than 2 percent of the reading, clean the probe and retest.

3. Using at least two standard solutions, which will most likely bracket the expected values for conductivity, note accuracy of the water and probe and clean probe if necessary. If stock standards are purchased or prepared by field personnel, they must be verified periodically against an EPA or NBS traceable standard.
4. Document calibration in a field notebook. Field notes should include: instrument serial number, the batch number of the calibration solutions and who did the calibration.

6.2.1.2 Calibration of Specific Ion Meter (pH/Eh Measurement)

6.2-1.2.1 pH Probe

1. If using refillable probes, remove electrode cap and check that filling solution is above the filling mark.
2. Immerse the probe in the pH 7 buffer solution and adjust the calibration control to read the appropriate pH. Check the pH buffer solution for correct pH value at the equilibrated temperature.
3. Remove the probe, rinse with distilled water and then immerse in either or both the pH 4 or pH 10 buffer solution, depending on the expected pH of the sample. Document calibration in a field notebook.
4. If the meter does not register the correct pH for that buffer solution, carefully adjust the calibration knob (i.e., slope control or sometimes called efficiency control) of the instrument to obtain the pH of the buffer.
5. After rinsing, insert the pH probe into the flow cell and allow the probe to come to equilibrium with the sample water.
6. Storage of the pH electrode should be in accordance with the manufacturer's instructions. These procedures vary from ambient air to specific storage solutions.

6.2-1.2.2 Eh/Platinum Probe

1. Check that the Eh or platinum probe is clean and the platinum band or tip is unoxidized. If dirty, polish with emery paper.
2. Immerse the probe and the reference probe, if required, into the calibration solution. Record the mV reading and the temperature and compare with the expected value ($\pm 10-20$ mV).
3. Rinse the probe with distilled water and insert into the flow cell. Allow for temperature equilibration and record the sample Eh.

4. At the end of the day, the platinum probe should be stored in water.

6.2-1.3 Calibration of Photoionization Meters

6.2-1.3.1 HNU

On a daily basis, calibrate this instrument (manufactured by HNU Systems of Newton, Massachusetts) according to the following general instructions. See manufacturer's manual for more specific instructions. Note that the probe must be attached to the instrument in order to operate.

1. Turn the function switch to the "battery check" position. The needle on the meter should read within or above the green battery area on the scale plate. If the needle is in the lower position of the battery arc, the instrument should be recharged prior to any calibration. If red LED comes "on", the battery should be recharged.
2. Turn the function switch to the "on" position. In this position the UV light source should be on.
3. To zero the instrument, turn the function switch to the "standby" position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counterclock-wise rotation yields a downscale deflection. If the span adjustment setting is changed after zero is set, the zero should be rechecked and adjusted if necessary. Wait 15-20 seconds to ensure that the zero reading is stable. If necessary, readjust the zero.

The instrument is now ready for calibration by switching the function switch to the proper range.

4. Using non-toxic gas mixtures of known concentration available from the manufacturer in pressurized containers, connect the cylinder with the analyzed gas mixture to the end of the probe with a piece of tubing. Open the valve of the pressurized container until a slight flow is indicated and the instrument draws in the volume of sample required for detection. Now adjust the span potentiometer so that the instrument is reading the stated value of the calibration gas.
5. If the instrument span setting is changed, the instrument should be turned back to the "standby" position and the electronic zero should be readjusted, if necessary. If the instrument does not calibrate, it may be necessary to clean the probe or the lamp connection. Record calibration information in a field notebook. Along with the instrument serial number, calibrating gas batch number, and the calibrator's name.

6.2-1.3.2 Photovac T.I.P.

On a daily basis, calibrate this instrument (manufactured by Photovac International of Huntington, New York) according to the following general instructions. See manufacturer's manual for more specific instructions.

1. Turn power switch on by first pulling the knob out and then up. Allow T.I.P. to warm up for five minutes prior to use. Turn span knob to max (9) and turn the zero knob to zero.
2. Attach "zero air" cylinder to T.I.P. inlet using PVC tubing. Zero instrument using zero knob only. (T.I.P. is very sensitive, so a stable reading of absolute zero is difficult and not necessary to achieve.)
3. Attach isobutylene cylinder to T.I.P. inlet. Use the span knob to adjust T.I.P. reading to the concentration number on the isobutylene cylinder (usually 60 ppm). Remove cylinder. T.I.P. is now calibrated and ready for use. Check randomly the calibration as T.I.P. has tendency to drift.
5. When finished, turn power off by pulling switch out and down. Recharge instrument overnight. Note that the Battery charger must be pushed into place and then screwed into bottom of T.I.P.

6.2-1.4 Calibration of Flame Ionization Detectors

6.2-1.4.1 OVA.

This instrument, manufactured by the Foxboro Co. of Foxborough, Massachusetts, is used for the rapid analysis of volatile organics in soil, water or air. Results obtained from soil/sediment samples are considered semi-quantitative.

1. Connect the appropriate probe to the Probe/Readout Assembly. Turn the pump switch on and leak check the flow system by plugging the end of the probe momentarily; the sample flow rate indicator should drop to zero. Turn the pump switch off. Turn INSTR switch on and allow 5 minutes for warmup. Turn the pump switch on and verify that the battery is charged.
2. Open the H₂ tank valve and supply valve. Depress the igniter button. If the unit is in proper working order, the flame ionization detector will ignite in 1 to 6 seconds. Do not depress the igniter for more than 6 seconds. If the instrument does not light, allow it to run for several minutes and repeat the ignition procedure.
3. Check the calibration using a calibration check gas before and after use. Most units are factory calibrated to methane.

6.2-2 WELL PURGING

The following statement, taken from Handbook for Sampling and Sample Preservation of Water and Wastewater (EPA, 1982) summarizes the importance of well purging in order to obtain representative groundwater samples.

"The importance of proper sampling of wells cannot be over emphasized. Even though the well being sampled may be correctly located and constructed, special precautions must be taken to ensure that the sample taken from that well is representative of the groundwater at that location and that the sample is neither altered nor contaminated by the sampling and handling procedure."

To obtain a representative sample of the groundwater it must be understood that the composition of the water within the well casing and in close proximity to the well is probably not representative of the overall groundwater quality at that sampling site. This is due to the potential presence of drilling contaminants near the well and because environmental conditions, such as the oxidation-reduction potential near the well, may differ from the conditions in the surrounding water-bearing materials. For these reasons it is frequently suggested that a well be pumped or bailed until it is thoroughly flushed of standing water and contains fresh water from the aquifer. The recommended length of time required to pump or bail a well before sampling is dependent on many factors including the characteristics of the well, the hydrogeologic nature of the aquifer, the type of sampling equipment being used, and the parameters being sampled. The time required may range from the time needed to pump or bail one bore volume to the time needed to pump several bore volumes. A common procedure is to pump or bail the well until a minimum of three to five bore volumes have been removed or the well has been bailed to dryness whichever comes first.

In order to calculate the one bore volume in the well the following formula should be used:

$$\text{Volume} = \pi r^2 h$$

where,

$$\pi = 3.142,$$

r = radius, measured as the inside diameter of the well divided by 2

h = the distance between the water surface in the well and the bottom of the well.

Following the measurements and calculations described above, sampling will commence in the sequence below, utilizing the appropriate purging technique [1(a) through 1(d)]:

1. Lower the pump intake into the well. For shallow groundwater situations, the intake of the suction tubing or the submersible pump will be lowered to the top of the well screen and the well purged of the required volumes. Available alternatives to this procedure may be utilized in certain situations:

(a) If the well screen is 20 feet or longer (i.e., making pumping from the top impractical), the intake line should be lowered to the approximate mid-point of the screened portion of the well or in highly permeable formations. The intake portion can be moved up and down the entire water column.

(b) If the well is situated in tight formations such as tills, clays or rock, purging of the well should be performed near the top of the well screen. Pumping or purging at this level until one to three volumes have been purged will facilitate removal of standing well water without creating a large artificial gradient toward the well.

(c) Under certain circumstances, the pump intake may be placed just below the water surface and purging initiated. As the water surface lowers, the pump intake is lowered to remain below the water surface.

(d) When using a submersible pump in conjunction with an inflatable packer system, the packer should be placed just above the top of the well screen and inflated according to the packer manufacturer's instructions. The volume of stagnant water to be purged should be calculated based on the depth below the packer. The packer is not deflated until sampling is complete.

2. Connect the instrumentation header to the pump discharge and begin flushing the well. Monitor the in-situ parameters (pH, Eh, temperature, and specific conductivity) and measure the volume of groundwater being pumped. Alternatively, in-situ parameters may be monitored in a beaker filled from the pump discharge. Purging of the standing well water is considered complete when one of the following is achieved:
 - o a minimum of three well volumes has been purged, and in-situ parameters have stabilized; or
 - o five well volumes have been purged; or
 - o the well has been pumped dry.
3. Record the in-situ parameters and the purging methodology utilized. All future sampling of this well must be preceded by the same purging method.

6.2-3 SELECTION OF SAMPLING EQUIPMENT

The sampling device to be utilized must be selected based upon both the physical characteristics of the well and the analytes to be determined. Aspects to be considered include:

- analytes of concern
 - volatile organics
 - semivolatile organics
 - metals
- depth to well screen relative to static water level
- floating versus dissolved versus suspended contaminants versus non-aqueous liquids that are heavier than water
- well diameter
- safety

Typical sampling devices include and are further described below in Sections 6.2-3.1 through 6.2-3.4.

- bailers
- submersible pumps
- peristaltic pumps
- bladder pumps

Other pumps may be utilized for well purging but are not recommended for sample collection.

The following devices are generally not acceptable for collecting samples for analysis:

- gas-driven piston pump
- suction lift pumps
- submersible diaphragm pump
- gas-lift sampler
- impeller pump

6.2-3.1 Bailers

Bailers (available in Teflon, PVC and stainless steel) are the only sampling devices currently recognized for sampling of volatile organics. However, their use is labor-intensive and may be discontinued after collecting samples for volatile organic analysis. Bailers may also enhance positive bias when multiple phases exist in a well. This condition should be evaluated prior to sampling.

A modified Kemmerer sampler, Figure 6.2-2, is often used for sampling surface water as well as ground water. Figure 6.2-3 shows a standard bailer with bottom check valve.

- Advantages of Bailers:
 - Can be constructed from a wide variety of materials
 - Economical and convenient enough so that a separate bailer may be dedicated to each well; some bailers are disposable
 - No external power source required

- Reduces outgassing of volatile organics
- o Disadvantages of Bailers:
 - Impractical for purging large volumes of stagnant water from a well
 - Transfer of water sample from bailer to sample bottle can result in aeration
- Cross-contamination may occur if equipment is not adequately cleaned after each use

6.2-3.2 Submersible Pumps

Submersible pumps are generally acceptable for all sampling activities except VOA sample collection. The reason for this is that submersible pumps may introduce air into the sample causing the volatilization and loss of the constituents being tested for. They are most often used for well purging and, therefore, may be utilized for sampling immediately after purging.

Other than bailing for VOA, submersible pumps are considered one of the most efficient means of sampling moderately deep (50-150') wells. They are difficult to transport, but accomplish sampling more quickly than bailers or peristaltic pumps and have variable flow rates (high for purging; low for sampling). Submersible pumps are not recommended for highly turbid waters due to rotor-binding problems.

- o Advantages:
 - Can be used to sample or purge several monitoring wells in a brief period of time
 - Dependent upon size of pump and pumping depths, relatively large pumping rates are possible
 - Can be dedicated to a single well if desired
- o Disadvantages:
 - Submersible pumps currently available require a minimum well casing inside diameter of two inches
 - Require relatively large amount of support equipment service
 - Not suitable for sampling organics
 - Must be decontaminated if used for more than one well

6.2-3.3 Peristaltic Pumps

Peristaltic pumps may be used for sampling of all analytes except VOA sample analysis. Their use is limited to vertical lifts of about 20 feet. These pumps never contact the sample, so decontamination is not an issue as new tubing is utilized instead. This aspect is especially appealing for shallow, highly contaminated ground water sampling. Transportation is relatively easy and flow may be regulated. Turbid waters will not affect pump operation.

6.2-3.4 Bladder Pumps

Bladder pumps for environmental sampling are available with silicone or teflon bladders. These pumps require a source of compressed air and, therefore, are more difficult to transport than other pumps. However, they provide lift for sampling up to 400' deep. Flow may be varied for purging or sampling by changing the cycle rate. They provide marginally better results than submersible pumps in highly turbid waters. Due to the large internal area, bladder pumps are considered more difficult to decontaminate than other pumps.

6.2-4 GROUNDWATER SAMPLING

Water sample containers are generally filled directly from the source, the sampler or the pump discharge without special considerations. A major exception is the collection of Volatile Organic Analyte (VOA) samples.

6.2-4.1 Procedures Applicable Only to Collection of Ground Water Samples for Volatile Organic Analysis

VOA samples must be collected as specified below. Each sample is taken in duplicate.

1. Uncap the sample bottle (Figure 6.2-4), taking care not to touch the teflon-faced septum (shiny side). If the septum is contaminated in any way, it should be replaced.
2. If a chlorine residual is potentially present, check for chlorine content with Potassium Iodine (KI) paper, a chlorine residual comparator, or a DPD kit. Note that KI paper will not detect low levels of residual chlorine and comparators are subject to interferences from turbidity and sample color. Thus EPA recommends using DPD kits (DPD colorimetric method).

If a residual chlorine content is detected, add four drops of concentrated Hydrochloric Acid to the sample container prior to filling the bottle.

3. Fill the sample vial slowly from bailer or equivalent discharge point, minimizing air entrainment, until the vial overflows.
4. Place the teflon-faced silicone rubber septum on the convex meniscus, teflon side (shiny side) down, and screw cap on (see Figure 6.2-4).
5. Invert the bottle; tap lightly to check for air bubbles.
6. If air bubbles are present, open the bottle, and add additional sample to eliminate air bubbles, then reseal and repeat STEP 5.

6.2-4.2 Procedures Applicable Only to Collection of Ground Water Samples for Metals Determination

Generally if the well is pumped ahead of time the sample should not have to be filtered. In some cases (e.g. water is very silty), field filtration of samples may be required. This

should be determined in advance and communicated to the laboratory. This is an important distinction since data obtained from the analysis of filtered samples are termed "dissolved" constituents while those from non-filtered samples are termed "total".

Filtering of any sample collected for organic analysis should be avoided. Allowing the samples to settle prior to analysis followed by decanting of the liquid is the preferred technique to avoid loss of organic constituents.

When field filtering is required, an appropriate filter medium must be selected to avoid potential sample contamination during the filtering process. If the water is thought to be contaminated by organic solvents, use of filter media such as cellulose or polycarbonate must be avoided; glass fiber or teflon filters should be used. Glass fiber filters should be rinsed in acid followed by a deionized water rinse prior to use for filtering trace metal or nutrient samples. Filtration procedures are described in Table 6.3-1. In all cases, the laboratory should be advised if samples have been filtered in the field. A general outline of the sampling procedure is as follows, refer to Section 6.3 for additional information.

1. Fill the sample container from sampling device or equivalent discharge point.
2. Add sufficient 1:1 Nitric Acid to the sample to bring the pH down to 2.0 for sample preservation.

6.2-4.3 Procedures for Collection of Ground Water Samples

General procedures for collecting groundwater samples are described below. See Section 6.3 for more specific instructions.

1. After purging, lower the sampling device to the middle of the screened interval or mid-point of the static water level. If the analysis to be performed is for lighter-than-water chemical species, then the sampling device should be lowered to the top of the water column for sample collection.
2. Collect the sample(s). Volatile and semivolatile samples are filled directly from a bailer with as little agitation as possible.

Other samples should be placed directly into the appropriate container from the bailer or pump discharge.

3. Remove the pump or bailer from the well and, if necessary, decontaminate the pump, tubing or bailer by flushing with decontamination fluid specified in the site-specific quality assurance specifications. Preferably, field decontamination should be avoided. Otherwise, extreme caution must be exercised to avoid introducing contamination. Up to one pint of solvent is used as needed. Rinse the bailer with one gallon of potable or deionized water. Rinse again with deionized water.

4. Do not use deionized water stored in plastic (polyethylene) containers for field use as the deionized water will leach phthalates from polyethylene storage containers and may actually introduce contamination if used for on-site equipment clean up. Nalgene, glass, or Teflon bottles should be used.
5. Complete sample Data Record (see Figure 6.2-5) after each well is sampled.
6. Secure the well cap and lock.
7. Place samples in a cool place (4° C) immediately for transport to laboratory.

REFERENCES

EPA, 1982, Handbook for sampling and sample preservation of water and wastewater:
Cincinnati, OH, EPA-600/4-82-029, 402 p.

SECTION 6.2
SAMPLING TECHNIQUES

LIST OF FIGURES

<u>Figure</u>	<u>Title</u>	<u>Page No.</u>
6.2-1	Field Instrumentation Quality Assurance Record	15
6.2-2	Modified Kemmerer Sampler	16
6.2-3	Teflon or Stainless Steel Bailer	17
6.2-4	Volatile Organic Collection Bottle	18
6.2-5	Sample Data Record	19

FIELD INSTRUMENTATION & MATERIAL QUALITY ASSURANCE RECORD		
PROJECT <input style="width: 90%;" type="text"/>	JOB NUMBER <input style="width: 90%;" type="text"/>	DATE <input style="width: 90%;" type="text"/>
FIELD INSTRUMENTATION CALIBRATION DATA		
EQUIP. TYPE/I.D.	BATTERY CONDITION	CALIBRATION INFORMATION
_____	_____	pH 4 _____ pH 7 _____ pH 10 _____
_____	_____	pH 4 _____ pH 7 _____ pH 10 _____
_____	_____	pH 4 _____ pH 7 _____ pH 10 _____
_____	_____	COND STD. _____/_____ COND STD. _____/_____
_____	_____	COND STD. _____/_____ COND STD. _____/_____
_____	_____	COND STD. _____/_____ COND STD. _____/_____
DISSOLVED OXYGEN	_____	AVG. WINKLER VALUE _____ PPM METER VALUE _____ PPM
REDOX	_____	ZOBELL SOL. VALUE _____ METER VALUE _____
PHOTOIONIZATION METER	_____	ZERO/ZERO AIR? <input type="checkbox"/> YES <input type="checkbox"/> NO SPAN GAS VALUE _____ PPM EQUIV. METER VALUE _____ PPM EQUIV.
_____	_____	ZERO/ZERO AIR? <input type="checkbox"/> YES <input type="checkbox"/> NO SPAN GAS VALUE _____ PPM EQUIV. METER VALUE _____ PPM EQUIV.
OTHER	_____	_____
FLUIDS/MATERIALS RECORD		
DEIONIZED WATER SOURCE: <input type="checkbox"/> LAB <input type="checkbox"/> PORTABLE SYSTEM <input type="checkbox"/> OTHER _____		
TRIP BLANK WATER SOURCE: <input type="checkbox"/> LAB, LOT NO. _____ <input type="checkbox"/> OTHER, TYPE _____ ID _____		
DECONTAMINATION FLUIDS: <input type="checkbox"/> METHYL HYDRATE; LOT NO. _____ <input type="checkbox"/> OTHER, TYPE _____ ID _____		
HNO ₃ /DI RINSE SOLUTION: <input type="checkbox"/> LAB, LOT NO. _____		
FILTRATION PAPER ID: (IN LINE) MANUF/TYPE _____ LOT NO. _____/_____ (VACUUM) MANUF/TYPE _____ LOT NO. _____		
CHEMICALS USED: <input type="checkbox"/> HNO ₃ LOT NO. _____ ZnAOC LOT NO. _____ <input type="checkbox"/> H ₂ SO ₄ LOT NO. _____ OTHER LOT NO. _____ <input type="checkbox"/> HCL LOT NO. _____ OTHER LOT NO. _____ <input type="checkbox"/> NaOH LOT NO. _____		
SAMPLER SIGNATURE _____		

Source: ABB-ES

Figure 6.2-1

Field Instrumentation Quality Assurance Record.

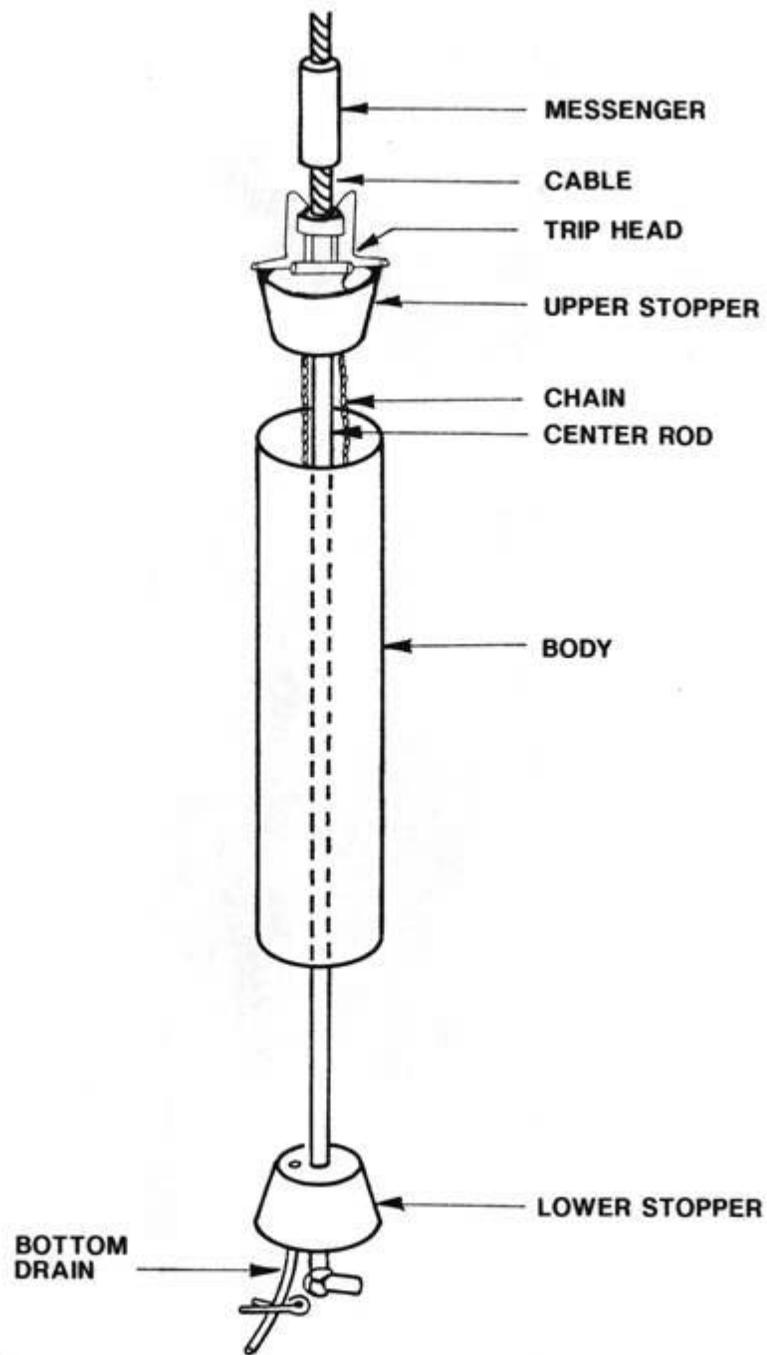
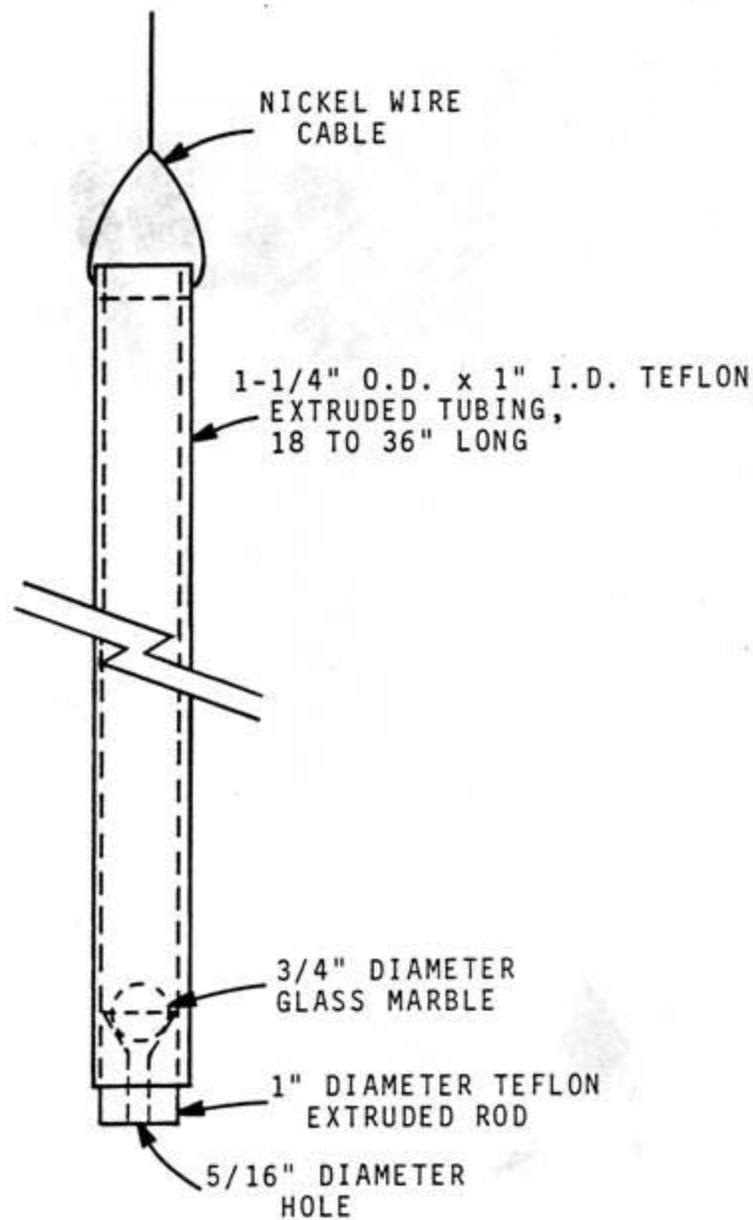


Figure 6.2-2

Source: EPA (1982)

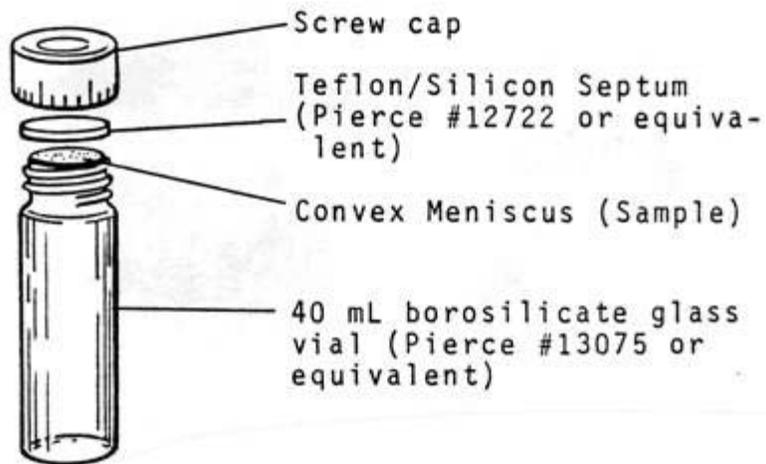
Modified Kemmerer Sampler.



Source: EPA (1982)

Figure 6.2-3

Teflon or Stainless Steel Bailer.



Source: EPA (1982)

Figure 6.2-4

Volatile Organic Collection Bottle.

PAGE _____ OF _____

PROJECT JOB NUMBER DATE

SAMPLE LOCATION ID LOCATION ACTIVITY START: END:

WATER LEVEL / WELL DATA

WELL DEPTH FT MEASURED TOP OF WELL CASING STICK-UP FT CASING/WELL DIFF. FT
 HISTORICAL TOP OF PROT. CASING (FROM GROUND)

WATER DEPTH FT WELL MATERIAL: PVC _____ WELL LOCKED?: YES NO WELL DIA. 2 INCH 4 INCH 6 INCH WATER LEVEL EQUIP. USED:
 SS _____ NO 6 INCH ELECT. COND. PROBE
 _____ _____ _____ FLOAT ACTIVATED
 _____ _____ _____ PRESS. TRANSDUCER

HEIGHT OF WATER COLUMN FT X .16 GAL/FT (2 IN.) GAL/VOL _____
 .65 GAL/FT (4 IN.) = TOTAL GAL PURGED _____
 1.5 GAL/FT (6 IN.) _____
 _____ GAL/FT (_____ IN.) _____

WELL INTEGRITY: YES NO
 PROT. CASING SECURE
 CONCRETE COLLAR INTACT
 OTHER _____

EQUIPMENT DOCUMENTATION

PURGING/SAMPLING EQUIP. USED: PURGING SAMPLING IF USED FOR:

PERISTALTIC PUMP EQUIPMENT ID _____ DECONTAMINATION FLUIDS USED:
 SUBMERSIBLE PUMP _____ ALL THAT APPLY AT LOCATION
 BAILER _____ ETHYL ALCOHOL
 PVC/SILICON TUBING _____ DEIONIZED WATER
 TEFLON/SILICON TUBING _____ TSP SOLUTION
 AIR LIFT _____ HEXANE
 HAND PUMP _____ HNO3/D.I. WATER SOLUTION
 IN-LINE FILTER _____ POTABLE WATER
 PRESS/VAC FILTER _____ NONE
 _____ _____
 _____ _____

FIELD ANALYSIS DATA

AMBIENT AIR VOA PPM WELL MOUTH PPM FIELD DATA COLLECTED IN-LINE IN CONTAINER

PURGE DATA	@ _____ GAL	SAMPLE OBSERVATIONS:				
TEMPERATURE, DEG C	_____	_____	_____	_____	_____	<input type="checkbox"/> TURBID
pH, units	_____	_____	_____	_____	_____	<input type="checkbox"/> COLORED
SPECIFIC CONDUCTIVITY (umhos/cm. @ 25 deg.c)	_____	_____	_____	_____	_____	<input type="checkbox"/> CLOUDY
OXIDATION-REDUCTION, +/- mv	_____	_____	_____	_____	_____	<input type="checkbox"/> CLEAR
DISSOLVED OXYGEN, ppm	_____	_____	_____	_____	_____	<input type="checkbox"/> ODOR

SAMPLE COLLECTION REQUIREMENTS
 IF REQUIRED AT THIS LOCATION

ANALYTICAL PARAMETER	<input type="checkbox"/> IF FIELD FILTERED	PRESERVATION METHOD	VOLUME REQUIRED	<input type="checkbox"/> IF SAMPLE COLLECTED	SAMPLE BOTTLE ID'S
<input type="checkbox"/>	<input type="checkbox"/>	_____	_____	<input type="checkbox"/>	_____/_____/_____/_____/_____
<input type="checkbox"/>	<input type="checkbox"/>	_____	_____	<input type="checkbox"/>	_____/_____/_____/_____/_____
<input type="checkbox"/>	<input type="checkbox"/>	_____	_____	<input type="checkbox"/>	_____/_____/_____/_____/_____
<input type="checkbox"/>	<input type="checkbox"/>	_____	_____	<input type="checkbox"/>	_____/_____/_____/_____/_____
<input type="checkbox"/>	<input type="checkbox"/>	_____	_____	<input type="checkbox"/>	_____/_____/_____/_____/_____
<input type="checkbox"/>	<input type="checkbox"/>	_____	_____	<input type="checkbox"/>	_____/_____/_____/_____/_____
<input type="checkbox"/>	<input type="checkbox"/>	_____	_____	<input type="checkbox"/>	_____/_____/_____/_____/_____
<input type="checkbox"/>	<input type="checkbox"/>	_____	_____	<input type="checkbox"/>	_____/_____/_____/_____/_____

NOTES:

SIGNATURE OF SAMPLER _____
 Source: ABB-ES

Figure 6.2-5
 Sample Data Record.

SECTION 6.3
SAMPLE HANDLING

TABLE OF CONTENTS

<u>Section</u>	<u>Title</u>	<u>Page No.</u>
6.3-1	FILTRATION	1
6.3-2	HOLDING TIMES AND PRESERVATION	1
6.3-3	SHIPPING	2
6.3-4	TRACKING	3
	REFERENCES.....	4

LIST OF FIGURES

<u>Figure</u>	<u>Title</u>	<u>Page No.</u>
6.3-1	Sample Splitting Flow Chart.....	6
6.3-2	Sample Analytical Request Form (Example)	7
6.3-3	Sample Tracking Form (Example)	8

LIST OF TABLES

<u>Table</u>	<u>Title</u>	<u>Page No.</u>
6.3-1	Standard Field Filtration Procedures	9
	(a) In-line Filtration.....	9
	(b) Vacuum Filtration	9
	(c) Pressure Filtration	10
6.3-2	Recommended Sample Containers, Preservation Techniques and Holding Times for Volatiles and Semi Volatiles Organics (SW-846, 3rd Edition)	12
6.3-3	Required Containers Preservation Techniques, and Holding Times-Liquid Matrix Only (RCRA TGED and SW-846)	14
6.3-4	Sample Containers, Preservation and Holding Requirements (EPA's Contract Laboratory Protocol Samples)	15
6.3-5	Sample Container, Preservation and Holding Requirements (Clean Water Act Samples).....	16

6.3 SAMPLE HANDLING

6.3-1 FILTRATION

In some cases, field filtration of samples may be required. This should be determined in advance and communicated to the laboratory. This is an important distinction since data obtained from the analysis of filtered samples are termed "dissolved" constituents while those from non-filtered samples are termed "total". In the case of metals, another option is the collection of an unfiltered sample followed by a milder digestion than that used in the total metals determination; results from this analysis are termed "total recoverable metals".

Filtering of any sample collected for organic analysis should be avoided. Allowing the samples to settle prior to analysis followed by decanting of the liquid is the preferred technique to avoid loss of organic constituents.

When field filtering is required, an appropriate filter medium must be selected to avoid potential sample contamination during the filtering process. If the water is thought to be contaminated by organic solvents, use of filter media such as cellulose or polycarbonate must be avoided; glass fiber or teflon filters should be used. Glass fiber filters should be rinsed in acid followed by a deionized water rinse prior to use for filtering trace metal or nutrient samples. Filtration procedures are described in Table 6.3-1.

A typical sample splitting flow chart is shown in Figure 6.3-1.

6.3-2 HOLDING TIMES AND PRESERVATION

Sample holding times are specified for the initiation of chemical analyses, usually beginning at the time of sample collection but occasionally (e.g., EPA's Contract Laboratory Program) beginning at the time of sample receipt at the laboratory. This determination must be made prior to sampling to allow proper logistical planning for sample shipments. Holding times also vary with the regulatory basis under which analyses are conducted. It is essential that the laboratory be consulted before sampling takes place in order to properly schedule the work. This will ensure that the laboratory will have a staff member available to receive the samples and that the laboratory can analyze the sample within the appropriate holding time.

Unless the proper sample bottle preparation and sample preservation measures are taken in the field, sample composition can be altered by contamination, degradation, biological transformation, chemical interactions, and other factors during the time between sample collection and analysis. Steps taken to maintain the in-situ characteristics required for analysis may include refrigeration of samples at 4°C, freezing, pH adjustment, and chemical fixation. Samples are preserved according to the protocol established for the specific analytical method and for specific regulatory requirements selected to obtain the desired data. These requirements are established on a case-by-case basis.

Holding times and preservation requirements for several analytes under different regulatory bases are presented in Tables 6.3-2, 6.3-3, 6.3-4 and 6.3-5.

6.3-3 SHIPPING

Sample containers are generally packed in picnic coolers for shipment. Bottles are to be packed tightly so that no motion is possible. Styrofoam, vermiculite, and "bubble pack" are suitable for most instances. Some materials that are considered to be highly hazardous require special preparation, containerization and labeling. Department of Transportation (DOT) guidelines and regulations should be consulted prior to shipment of any materials considered to be hazardous. Ice is placed in double "Ziploc" bags and added to the cooler along with all paperwork in a separate "Ziploc" bag. Sealed containers of heat transfer fluids (e.g., "Blue Ice") may also be used. Solid carbon dioxide (dry ice) is not an acceptable alternative. The cooler top is then taped shut. Custody seals and taping of coolers are generally required.

The standard procedure followed for shipping environmental samples to the analytical laboratory is, as follows:

1. All shipping of environmental samples collected must be done through overnight delivery service.

Note: Samples must not be shipped unless:
 - (a) next-day arrival is quarantined by the delivery service, and
 - (b) the receiving laboratory has agreed to be open to receive them.
2. Prior to leaving for the field, the task leader responsible for sample collection must notify the laboratory manager of the number, type and approximate collection and shipment dates for the samples. If the number, type or date of shipment changes due to site constraints or program changes, the task leader must notify the laboratory of the changes. This notification from the field also needs to occur when sample shipments will arrive on Saturdays.
3. If prompt shipping and laboratory receipt of the samples cannot be guaranteed (i.e., Sunday arrival), the task leader will be responsible for proper storage of the samples until adequate transportation arrangements can be made. Proper storage requires that the samples be refrigerated, and in some cases, locked in a secure location. Storing samples in one's car or leaving them in the office are not acceptable procedures.
4. The laboratory should be notified if advance if parameters such as BOD or holding times <48 hrs are included in the shipment.

These communications are necessary to allow the laboratory enough time to prepare for the samples' arrival.

The samples are shipped to the laboratory together with the Chain of Custody (COC) documents described in Section 6.4 and an applicable Analytical Request Form (see example Figure 6.3-2).

6.3-4 TRACKING

Figure 6.3-3 is an example of a sample tracking form. Tracking of samples should commence at the time of sample container label generation. A site-specific database of anticipated sample collection should be created, then updated as analytical request forms and chain-of-custody forms are received from the field. This database can be hand-scribed, but tracking of data is better done in a computerized format, that is able to sort through and organize the data according to different parameters.

A letter of receipt from the laboratory provides the information to verify the following:

- analytical program
- turn-around time
- laboratory internal identification numbers
- chain-of-custody for shipped samples

REFERENCES

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act (Federal), 40 CFR Part 136.

RCRA Groundwater Monitoring Technical Enforcement Guidance Document, US EPA, Office of Waste Programs Enforcement (OSWER), Sept 1986.

Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, 3rd Edition, Proposed Update Package, "SW-846", Dec 1987.

User Guide to the Contract Laboratory Program, EPA, Office of Environmental Response and Remediation (OERR)/Contract Laboratory Program (CLP), Dec 1988.

SECTION 6.3
SAMPLE HANDLING

LIST OF FIGURES

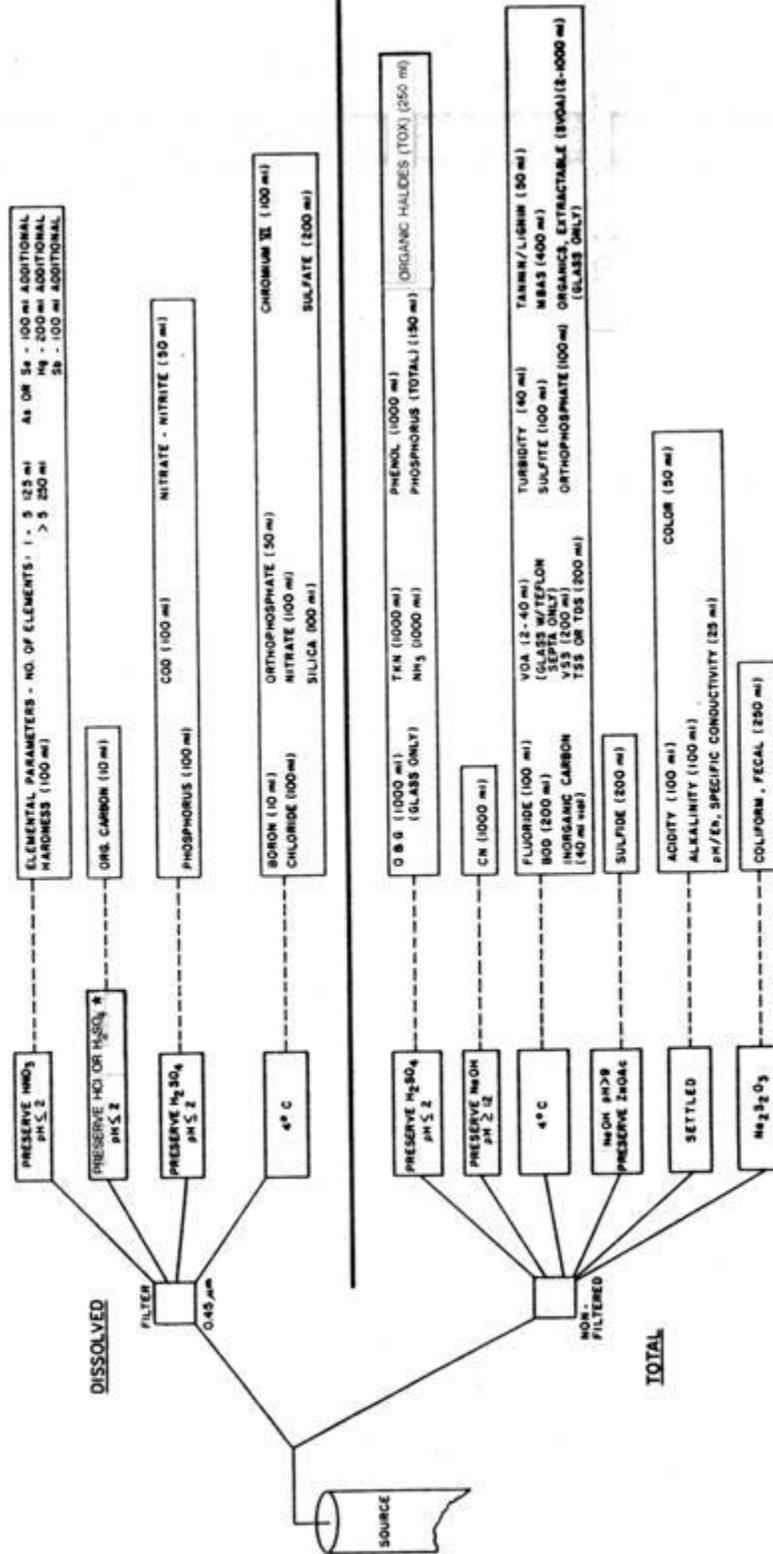
<u>Section</u>	<u>Title</u>	<u>Page No.</u>
6.3-1	Sample Splitting Flow Chart6
6.3-2	Sample Analytical Request Form (Example)	7
6.3-3	Sample Tracking Form (Example)8

LIST OF TABLES

<u>Section</u>	<u>Title</u>	<u>Page No.</u>
6.3-1	Standard Field Filtration Procedures	9
	(a) In-line Filtration	9
	(b) Vacuum Filtration	9
	(c) Pressure Filtration	10
6.3-2	Recommended Sample Containers, Preservation Techniques and Holding Times for Volatiles and Semi Volatiles Organics (SW-846, 3rd Edition)	12
6.3-3	Required Containers Preservation Techniques, and Holding Times- Liquid Matrix Only (RCRA, TGED, and SW-846)	14
6.3-4	Sample Containers, Preservation and Holding Requirements (EPA's Contract Laboratory Protocol Samples)	15
6.3-5	Sample Container, Preservation and Holding Requirements (Clean Water Act Samples)	16

SAMPLE SPLITTING FLOW CHART-GROUNDWATER/SURFACE WATER MONITORING

PARAMETER & MINIMUM VOLUMES REQUIRED



* Preservative (HCl or H₂SO₄) depends upon instrument manufacturer's directions - consult with lab.
NOTE ALL SAMPLES SHOULD BE KEPT AT 4°C. REGARDLESS OF PRESERVATIVE

Figure 6.3-1
Sample Splitting Flow Chart.

Source: ABB-ES

CLIENT INFORMATION: NAME _____
COMPANY _____
MAILING ADDRESS _____
JOB NUMBER _____

ANALYSES REQUESTED BY: TECHNICAL PROJECT PROFESSIONAL _____
APPROVED BY: PROJECT MANAGER _____

SAMPLE IDENTIFICATION	LAB NUMBERS	DATE SAMPL'D	SAMPL'D BY	ANALYSES REQUIRED
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

DATE RECEIVED _____ TYPE OF SAMPLE _____
LAB LOCATION _____ LIST ANY HAZARDS _____
RESULTS DUE _____
CLIENT ID. NO. _____

SOLID WASTE DATA FILE
 DATA DOCUMENTATION REQ'D
 ENTERED IN COMPUTER

SPECIAL PROCEDURE

ADDITIONAL INFORMATION OR SPECIAL PROCEDURES _____

Source: ABB-ES

Figure 6.3-2
Sample Analytical Request Form.

Date: _____

Page: _____

Project: _____

Job Number: _____

Sample ID	Lab Number	Matrix	Analysis	Date Sampled	Hardcopy Received	Box Number	File Name	A	V	S	Final Table	Comments
0150118xx01xx	8253-026	Sediment	CLP-COP	9/09/88	11/16/88	4607-805-A	BR18	X				
0150118xx01xx	8256-021	Sediment	CLP-CIP	9/09/88	11/04/88	4607-803-B	BR08	X				MS/MSD
0150118xx01xx	8256-021	Sediment	CLP-COP	9/09/88	11/16/88	4607-805-A	BR18	X				MS/MSD
0150119xx01xx	8256-022	Sediment	CLP-CIP	9/09/88	11/04/88	4607-803-B	BR08	X				
0150119xx01xx	8256-022	Sediment	CLP-COP	9/09/88	11/16/88	4607-805-A	BR18	X				
0150103xx01xx	8252-030	Sediment	CLP-CIP	9/07/88	11/04/88	4607-803-B	BR08	X				
0150103xx01xx	8252-030	Sediment	CLP-COP	9/07/88	10/31/88	4607-803-A	BR05	X				
0150304xx01xx	8252-031	Sediment	CLP-CIP	9/07/88	11/04/88	4607-803-B	BR08	X				
0150304xx01xx	8252-031	Sediment	CLP-COP	9/07/88	10/31/88	4607-803-A	BR05	X				
0150305xx01xx	8252-032	Sediment	CLP-CIP	9/07/88	11/04/88	4607-803-B	BR08	X				
0150305xx01xx	8252-032	Sediment	CLP-COP	9/07/88	10/31/88	4607-803-A	BR05	X				
0150918xx01xx	8267-003	Sediment	CLP-CIP	9/22/88	11/08/88	4607-804-B	BR16	X				
0150918xx01xx	8267-003	Sediment	CLP-COP	9/22/88	11/17/88	4607-805-C	BR21	X				No VOA
0155118xx01xx	8253-029	Soil	CLP-CIP	9/08/88	11/04/88	4607-803-B	BR08	X				
0155118xx01xx	8253-029	Soil	CLP-COP	9/08/88	11/16/88	4607-805-A	BR18	X				
015006xx01xx	8253-012	Water	CLP-CIP	9/08/88	11/04/88	4607-803-B	BR08	X				
015006xx01xx	8253-012	Water	CLP-COP	9/08/88	11/09/88	4607-804-C	BR11	X				
0150118xx01xx	8256-014	Water	CLP-CIP	9/09/88	11/04/88	4607-803-B	BR08	X				
0150118xx01xx	8256-014	Water	CLP-COP	9/09/88	11/01/88	4607-804-D	BR13	X				
0150118xx01xx	8256-013	Water	CLP-CIP	9/09/88	11/04/88	4607-803-B	BR08	X				
0150118xx01xx	8256-013	Water	CLP-COP	9/09/88	11/01/88	4607-804-D	BR13	X				
0150118xx01xx	8256-015	Water	CLP-CIP	9/09/88	11/04/88	4607-803-B	BR08	X				
0150118xx01xx	8256-015	Water	CLP-COP	9/09/88	11/01/88	4607-804-D	BR13	X				
015015xx01xx	8253-017	Water	CLP-CIP	9/08/88	11/04/88	4607-803-B	BR08	X				
015015xx01xx	8253-017	Water	CLP-COP	9/08/88	11/09/88	4607-804-C	BR11	X				
015018xx01xx	8256-016	Water	CLP-CIP	9/09/88	11/04/88	4607-803-B	BR08	X				
015018xx01xx	8256-016	Water	CLP-COP	9/09/88	11/01/88	4607-804-D	BR13	X				
015019xx01xx	8256-017	Water	CLP-CIP	9/09/88	11/04/88	4607-803-B	BR08	X				
015019xx01xx	8256-017	Water	CLP-COP	9/09/88	11/01/88	4607-804-D	BR13	X				
0150918xx01xx	8267-002	Water	CLP-CIP	9/22/88	11/08/88	4607-804-B	BR15	X				
0150918xx01xx	8267-002	Water	CLP-COP	9/22/88	11/17/88	4607-805-C	BR21	X				
018001xx01xx	8252-001	Water	CLP-COP/VOA	9/06/88	10/31/88	4607-803-A	BR04	X				
018002xx01xx	8253-003	Water	CLP-COP/VOA	9/08/88	11/09/88	4607-804-C	BR11	X				

Figure 6.3-3
Sample Tracking Form.

Source: ABB-ES

STANDARD FIELD FILTRATION PROCEDURE

(a) IN-LINE FILTRATION

Equipment

1. A portable 102-mm acrylic backflushing filter unit
2. 102-mm diameter filter papers, 0.45- μ m membrane filters
3. Deionized (DI) rinse water
4. 1:1 reagent grade nitric acid rinse solution

Procedures

1. After assembling filter paper into filter holder, attach in-line filter assembly to discharge line of sampling pump. Open by-pass valve completely.
2. Turn sampling pump on, and slowly turn by-pass valve closed, allowing flow into the filter. Remove trapped air through the filter bleed valve, if necessary.
3. Rinse barrel and filter holder assembly between samples with three rinses of reagent water. The rinse sequence when elemental parameters will be analyzed is: DI water - 1:1 reagent grade nitric acid - DI water.

(b) VACUUM FILTRATION

1. Two sets of either glass funnel type or self-contained polysulfone filters with sintered glass discs or polysulfone filter plates.
2. 47-mm diameter filter papers, 0.45- μ m membrane filters.
3. Vacuum pump or ISCO peristaltic pump with silicone tubing
4. DI rinse water
5. 1:1 reagent grade nitric acid rinse solution.

Procedures

1. Thoroughly rinse sintered glass disc, filter funnel, and stem polysulfone filter units with DI water.

Table 6.3-1

Standard Field Filtration Procedures.

(b) VACUUM FILTRATION (cont.)

2. On the basis of visual clarity of sample, prefiltering with larger pore filters may be required. If sample has a heavy clay content, organics, or suspended matter, prefiltration through a 3.0- or 5.0- μm membrane filter may be necessary.
3. Place membrane filter on filter holder with minimum handling.
4. Attach filter holder with filter to filter funnel and receiver.
5. Swirl and slowly pour contents of sample bottle into filter funnel.
6. Attach suction tubing to filter flask and vacuum pump (or ISCO pump). Pump is turned on in the vacuum mode.
7. Filter a small portion of the sample and discard filtrate after rinsing flask with sample filtrate.
8. If prefiltering was required, pass the sample through a 0.45- μm membrane filter using another filtering apparatus.
9. Transfer the filter sample to appropriate bottles.
10. Rinse filtration equipment between samples with at least three rinses of DI water. The rinse sequence, when elemental parameters are to be analyzed, is: DI water - 1:1 reagent grade nitric acid - DI water.

(c) PRESSURE FILTRATION

Equipment

1. Pressure filter apparatus consisting of 1-liter barrel filter, filter holder and pressure hose connectors.
2. Source of pressurized inert gas, (e.g., tank of nitrogen, argon, etc.).
3. 147-mm filter papers, 0.45- μm membrane filter.
4. DI rinse water.
5. 1:1 reagent grade nitric acid rinse solution.

Procedures

1. If filter barrel has sample valve, assemble with 0.45- μm membrane filter and attach pressure hose.

**Table 6.3-1
(continued)
Standard Field Filtration Procedures.**

(c) PRESSURE FILTRATION (cont.)

2. If filter barrel does not have a sample valve, assemble filter paper on filter holder.
3. Turn barrel upside down and pour sample into barrel.
4. Place filter holder and filter onto barrel assembly, making sure to align O-ring for a positive seal.
5. Attach swing-away bolts and tighten hand-tight.
6. Turn over filter assembly and attach pressure hose assembly.
7. Slowly turn on pressurized gas and increase pressure regulator to a maximum of 20 psi.
8. Collect filtrate from bottom of barrel assembly.
9. Rinse barrel and filter holder assembly between samples with three rinses of DI water. The rinse sequence when elemental parameters will be determined is: DI water - 1:1 reagent grade nitric acid - DI water.

**Table 6.3-1
(continued)
Standard Field Filtration Procedures.**

Parameter	Container	Preservative	Holding Time
<u>Volatile Organics</u>			
Concentrated Waste Samples	8-oz. widemouth glass with Teflon liner	None	14 days
<u>Liquid Samples</u>			
No Residual Chlorine Present	2 40-ml vials with Teflon lined septum caps	4 drops conc. HCl, Cool, 4° C	14 days
Residual Chlorine Present	20 40-ml vials with Teflon lined septum caps	Collect sample in a 4 oz. soil VOA container which has been pre-preserved with 4 drops of 10% sodium thio-sulfate. Gently mix sample and transfer to a 40-ml VOA vial that has been pre-preserved with 4 drops of conc. HCl, Cool to 4° C	14 days
Acrolein and Acrylonitrile	2 40-ml vials with Teflon lined septum	Adjust to pH 4-5, Cool, 4° C	14 days
Soil/sediments and sludges	4-oz. (120-ml) wide-mouth glass with Teflon liner	Cool, 4° C	14 days

Table 6.3-2
(page 1 of 2)
Recommended Sample Containers,
Preservation Techniques, and Holding
Times for Volatiles and Semi-Volatile Organics.
(SW-846, 3rd Edition)

Source: Proposed Update 3rd. Ed. SW-846

Parameter	Container	Preservative	Holding Time
<u>Semi-Volatile Organics/Organochlorine Pesticides/PCBs</u>			
Concentrated Waste Samples	8-oz. widemouth glass with Teflon liner	None	Samples must be extracted within 14 days and extract analyzed within 40 days following extraction.
Water Samples			
No Residual Chlorine Present	1-gal. or 2 1/2-gal. amber glass with Teflon liner	Cool, 4° C	Samples must be extracted within 7 days and extracts analyzed within 40 days following extraction.
Residual Chlorine Present	1-gal. or 2 1/2-gal. amber glass with Teflon liner	Add 3 ml 10% sodium thiosulfate per gallon, Cool, 4° C	Samples must be extracted within 7 days and extracts analyzed within 40 days following extraction.
Soil/Sediments and Sludges	8-oz. widemouth glass with Teflon liner	Cool, 4° C	Samples must be extracted within 14 days and extract analyzed within 40 days following extraction.

Source: Proposed Update 3rd. Ed. SW-846

Table 6.3-2
(page 2 of 2)
Recommended Sample Containers,
Preservation Techniques, and Holding
Times for Volatiles and Semi-Volatile Organics.
(SW-846, 3rd Edition)

Name	Container ¹	Preservation	Maximum holding time
Bacterial Tests:			
Coliform, total	P, G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃	6 hours
Inorganic Tests:			
Chloride	P, G	None required	28 days
Cyanide, total and amenable to chlorination	P, G	Cool, 4°C; if oxidizing agents present add 5 mL 0.1N NaAsO ₂ per L or 0.06 g of ascorbic acid per L; adjust pH>12 with 50% NaOH.	14 days
Hydrogen ion (pH)	P, G	None required	Analyze immediately
Nitrate	P, G	Cool, 4°C	48 hours
Sulfate	P, G	Cool, 4°C	28 days
Sulfide	P, G	Cool, 4°C, add zinc acetate	7 days
Metals:			
Chromium VI	P, G	Cool, 4°C	24 hours
Mercury	P, G	HNO ₃ to pH<2	28 days
Metals, except chromium VI and mercury	P, G	HNO ₃ to pH<2	6 months
Organic Tests:			
Oil and grease	G	Cool, 4°C ²	28 days
Organic carbon, total (TOC)	P, G	Cool, 4°C ²	28 days
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C ³	14 days
Purgeable aromatic hydrocarbons	G, Teflon-lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ^{2,3}	14 days
Acrolein and acrylonitrile	G, Teflon-lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ , Adjust pH to 4-5	14 days
Phenols	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Benzidines	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Phthalate esters	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitrosamines	G, Teflon-lined cap	Cool, 4°C, store in dark, 0.008% Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
PCBs	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitroaromatics and cyclic ketones	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ , store in dark	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ , store in dark	7 days until extraction, 40 days after extraction
Haloothers	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Chlorinated hydrocarbons	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Dioxins and Furans	G, Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Total organic halides (TOX)	G, Teflon-lined cap	Cool, 4°C ²	8 days
Pesticides	G, Teflon-lined cap	Cool, 4°C, pH 5-9	7 days until extraction, 40 days after extraction
Radiological Tests:			
Alpha, beta and radium	P, G	HNO ₃ to pH<2	6 months

¹Polyethylene (P) or Glass (G)

²Adjust to pH<2 with H₂SO₄, HCl or solid NaHSO₄.

³Free chlorine must be removed prior to addition of HCl by exact addition of Na₂S₂O₃.

Table 6.3-3

Source: RCRA TGED and SW-846

Required Containers Preservation Techniques, and Holding Times-Liquid Matrix Only. (RCRA TGED and SW-846)

Parameter	Container	Preservative	Holding Time	
			Soil	Water
Volatiles by gas chromatography/mass spectrometry (GC/MS)	Water - 40 mL glass vial with Teflon-lined septa	Cool, 4°C	10 days	10 days
	Soil-Glass with Teflon-lined septa			
Polychlorinated biphenyl (PCB)/pesticides	G, Teflon-lined lid	Cool, 4°C	Extract within 10 days, analyze 40 days	Extract within 5 days, analyze 40 days
	G, Teflon-lined lid	Cool, 4°C	Extract within 10 days, analyze 40 days	Extract within 5 days, analyze 40 days
Metals	P, G	HNO ₃ to pH<2	6 months	6 months
	P, G	HNO ₃ to pH<2	26 days	26 days
	P, G	NaOH to pH>12 Cool 4°C add 0.6g ascorbic acid if residual chlorine present	14 days	14 days
Chromium VI	P, G	Cool, 4°C	24 h	24 h

Note: P = polyethylene
G = glass

Table 6.3-4

Sample Containers,
Preservation and Holding Requirements.
(EPA's Contract Laboratory Protocol Samples)

Source: EPA CLP

Parameter Name	Type	Container(1)	Size	Preservation(2)	Maximum Holding Time(3)
Bacterial Tests					
Coliform, fecal and total	P, G		250 ml	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃	6 hours
Fecal streptococci	P, G		250 ml	Same as above	6 hours
Inorganic Tests					
Acidity	P, G		100 ml	Cool, 4°C	14 days
Alkalinity	P, G		100 ml	Cool, 4°C	14 days
Ammonia	P, G		1000 ml	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days
Biochemical oxygen demand	P, G		200 ml	Cool, 4°C	48 hours
Bromide	P, G		100 ml	None required	28 days
Biochemical oxygen demand, carbonaceous	P, G		100 ml	Cool, 4°C	48 hours
Chemical oxygen demand	P, G		100 ml	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days
Chloride	P, G		100 ml	None required	28 days
Chlorine, total residual	P, G		in field	None required	Analyze immediately
Color	P, G		50 ml	Cool, 4°C	48 hours
Cyanide, total and amenable to chlorination	P, G		1 l	Cool, 4°C, NaOH to pH >12, 0.6g ascorbic acid	14 days
Fluoride	P		100 ml	None required	28 days
Hardness	P, G		100 ml	HNO ₃ to pH <2, H ₂ SO ₄ to pH <2	6 months
Hydrogen ion (pH)	P, G		25 ml	None required	Analyze immediately
Kjeldahl and organic nitrogen	P, G		1 l	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days
Metals					
Chromium VI	P, G		100 ml	Cool, 4°C	24 hours
Mercury	P, G		150 ml	HNO ₃ to pH <2	28 days
Metals, except chromium VI and mercury	P, G		1-5 parameters-100ml 6-10 parameters-125ml >10 parameters-150ml	Same as above	6 months
Nonconventional Pollutants					
Nitrate	P, G		100 ml	Cool, 4°C	48 hours
Nitrate-nitrite	P, G		50 ml	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days
Nitrite	P, G		100 ml	Cool, 4°C	48 hours
Oil and grease	G		1 l	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days
Organic carbon	P, G		10 ml	Cool, 4°C, HCl or H ₂ SO ₄ to pH <2*	28 days
Orthophosphate	P, G		50 ml	Filter immediately, cool, 4°C	48 hours
Oxygen, dissolved probe	G	bottle and top	in field	None required	Analyze immediately
Winkler	G	Same as above	200 ml	Fix on site and store in dark	8 hours
Phenols	G		1 l	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days
Phosphorus (elemental)	G		100 ml	Cool, 4°C	48 hours
Phosphorus, total	P, G		150 ml	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days
Residue, total	P, G		200 ml	Cool, 4°C	7 days
Residue, filterable	P, G		200 ml	Cool, 4°C	48 hours
Residue, nonfilterable (TSS)	P, G		200 ml	Cool, 4°C	7 days
Residue, settleable	P, G		1 l	Cool, 4°C	48 hours
Residue, volatile	P, G		200 ml	Cool, 4°C	7 days
Silica	P	see metals		Cool, 4°C	28 days

* Consult with laboratory; choice of acid depends upon instrument manufacturer.

Table 6.3-5
(page 1 of 2)
Sample Container,
Preservation and Holding Requirements.
(Clean Water Act Samples)

Parameter Name	Container(1)		Preservation(2)		Maximum Holding Time(3)
	Type	Size			
Specific conductance	P, G	25 mL	Cool, 4°C		28 days
Sulfate	P, G	250 mL	Cool, 4°C		28 days
Sulfide	P, G	200 mL	Cool, 4°C, add zinc acetate plus sodium hydroxide to pH >9		7 days
Sulfite	P, G	100 mL	None required		Analyze immediately
Surfactants	P, G	400 mL	Cool, 4°C		48 hours
Temperature	P, G	25 mL	None required		Analyze in field
Turbidity	P, G	40 mL	Cool, 4°C		48 hours
Organic Tests					
Purgeable halocarbons	G, Teflon-lined septum	40 mL	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ (4)		14 days (5)
Purgeable aromatic hydrocarbons	Same as above	40 mL	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ (4), HCl to pH 2		14 days (5)
Acrolein and acrylonitrile	Same as above	40 mL	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ (4), Adjust pH to 4-5		14 days (5)
Phenols	G, Teflon-lined cap	1 L	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ (4)		7 days until extraction, 40 days after extraction
Benzidines	Same as above	1 L	Same as above		7 days until extraction
Phthalate esters	Same as above	1 L	Cool, 4°C		7 days until extraction, 7 days after extraction
Nitrosamines	Same as above	1 L	Cool, 4°C, store in dark, 0.008% Na ₂ S ₂ O ₃		Same as above
PCBs, acrylonitrile	Same as above	1 L	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ (4)		Same as above
Nitroaromatics and isophorone	Same as above	1 L	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ (4)		Same as above
Polynuclear aromatic hydrocarbons	Same as above	1 L	Same as above		Same as above
Haloethers	Same as above	1 L	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ (4)		Same as above
Chlorinated hydrocarbons	Same as above	1 L	Cool, 4°C		Same as above
TCDD	Same as above	1 L	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ (4)		Same as above
Volatile Organics	G, Teflon-lined septum	40 mL	Cool, 4°C		14 days (5)
Semi-Volatiles	G, Teflon-lined cap	1 L	Cool, 4°C		7 days until extraction, 40 days after extraction
Pesticides Tests					
Pesticides	Same as above	1 L	Cool, 4°C, pH 5-9		Same as above
Radiological Tests					
Alpha, beta and radium	P	1 L	HNO ₃ to pH <2		6 months

- (1) Appropriate sample containers: P = polyethylene, G = glass.
- (2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- (3) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples (preserved, as required) may be held before analyses and still be considered valid. Some samples may not be stable for the maximum time period given in the table. A permit or monitoring laboratory is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.
- (4) Use Na₂S₂O₃ (sodium thiosulfate) only if chlorine is present.
- (5) 7 days if unpreserved.

Table 6.3-5
(page 2 of 2)
Sample Container,
Preservation and Holding Requirements.
(Clean Water Act Samples)

Source: EPA Guidelines Clean Water Act

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

STANDARD REFERENCES FOR MONITORING WELLS

SECTION 6.4 CHAIN-OF-CUSTODY

SECTION 6.4
CHAIN-OF-CUSTODY

TABLE OF CONTENTS

<u>Section</u>	<u>Title</u>	<u>Page No.</u>
6.4-1	PURPOSE	1
6.4-2	IMPLEMENTATION	1

LIST OF FIGURES

<u>Figure</u>	<u>Title</u>	<u>Page No.</u>
6.4-1	Chain-of-Custody Form	4
6.4-2	Example of a Properly Filled Out Chain-of-Custody Form	5

6.4 CHAIN-OF-CUSTODY

6.4-1 PURPOSE

A chain-of-custody (COC) program must be followed during sample handling activities from the field through laboratory operations. This program is designed to assure that each sample is accounted for at all times. Field data sheets, COC records, and sample labels must also be completed by the appropriate sampling and laboratory personnel for each sample.

The objective of the sample custody identification and control system is to assure, to the extent practicable, that:

- all samples are uniquely identified;
- the correct samples are analyzed for the correct parameters and are traceable through their records;
- important sample characteristics are preserved;
- samples are protected from loss or damage;
- any processing of samples (e.g., filtration, preservation) is documented;
- a defensible forensic record of sample integrity is established; and
- client confidentiality is maintained.
- a sample is considered under a chain-of-custody if it meets all of the following criteria:
 - (1) the sample is in your custody,
 - (2) the sample is in your view, after being in your possession,
 - (3) the sample is in your possession and then you locked it up to prevent tampering, and
 - (4) the sample is in a designated, secured area (locked area with limited access).

6.4-2 IMPLEMENTATION

The chain-of-custody procedure begins in the field and establishes a "paper trail" so that sample possession can be traced.

The standard COC protocol used by DEP is as follows:

- Pre-prepare labels for each sample that includes identification, date and time of collection, sample parameters to be analyzed, any preservatives added, and the name of sample collector.
- Record the procedures and amounts of reagents or supplies necessary for each sample including sample preparation and preservation.
- Record date and time of sampling, sampling locations, sample bottle identification, and specific sample acquisition measures on the chain-of-custody forms.
- Complete standard field data record forms to establish sample custody in the field before sample shipment (see Section 6.3).

The COC description section requires:

- a unique identification of each sample;
- the name(s), address(es), and telephone number(s) of the sampler(s) and the person shipping the samples and all subsequent transfers of custody;
- the type and method of analysis requested (sometimes this can be put on the field data sheet that accompanies the chain-of-custody form);
- the date and time that the samples were taken and delivered for shipping; and
- the names of those responsible for receiving the samples at the laboratory.

The COC record is used to:

- document the identity of a sample and its handling from its first existence as a sample until analysis and data reduction are completed;
- Custody records trace a sample from collection through all transfers of custody until it is transferred to the analytical laboratory. Internal laboratory records document the custody of the sample through its final disposition.

A typical COC record is shown in Figure 6.4-1. At least four copies of the COC must be available, 4-part NCR forms are preferred. The original and one copy must accompany the samples to the laboratory; one copy is retained by the sampling crew chief; the last copy is placed in the project file. The signed original is retained by the laboratory. Return

copies of original signed by receiving laboratory to sampling crew chief and to the project file.

Where litigation is likely, custody seals must be used on sample shipments to avoid any question of tampering. In any event, routine use of custody seals is good practice.

LIST OF FIGURES

<u>Section</u>	<u>Title</u>	<u>Page No.</u>
6.4-1	Chain-of-Custody Record4
6.4-2	Example of a Properly Filled Out Chain-of-Custody Form5

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

STANDARD REFERENCES FOR MONITORING WELLS
SECTION 6.5 DECONTAMINATION OF SAMPLING EQUIPMENT

SECTION 6.5
DECONTAMINATION OF SAMPLING EQUIPMENT

TABLE OF CONTENTS

<u>Section</u>	<u>Title</u>	<u>Page No.</u>
6.5-1	PURPOSE	1
6.5-2	PROCEDURE.....	1

6.5 DECONTAMINATION OF SAMPLING EQUIPMENT

6.5.1 PURPOSE

Ideally, sampling equipment, (e.g., bailers, pumps, tubing, filter holders) will be dedicated to each sampling location and precleaned in the laboratory prior to each sampling episode, thus eliminating the need for field decontamination of sampling equipment. The use of disposable sampling equipment is more expensive but totally contaminant free. When this is not possible, field decontamination of such equipment must occur prior to collection of each set of samples.

Decontamination of reusable equipment used to collect samples is essential in order to maintain chemical data integrity between sampling locations. In general, decontamination should allow for adequate cleaning of the drilling and sampling tools for the contaminants found at any given site. Different chemicals or mixtures of chemicals will require the use of different cleaning methods or compounds.

6.5-2 PROCEDURE

The method of choice for decontamination should be that which most fully removes site contaminants from the sampling equipment with least interference to the ultimate chemical analysis. Site and weather conditions frequently impose constraints upon the preferred method.

The general decontamination methods and compounds that can be used are as follows:

- Do not use distilled water stored in plastic bottles as the plastic contains too many contaminants. Bring deionized water in either nalgene bottles or teflon bottles from the laboratory.
- Equipment to be utilized in the collection of samples for metals analysis should be cleaned by the following steps:
 1. Wash equipment with a non-phosphate detergent-solution (e.g. Alconox) and a brush.
 2. Rinse thoroughly with tap water.
 3. Rinse with 1:1 nitric acid.
 4. Rinse the equipment thoroughly with deionized water (either ASTM type I or II).
 5. For water samples, rinse the equipment two to three times with the media being sampled before collecting a sample.
 6. Repeat this procedure at each location.

- Equipment to be used for collection of samples for TPH, oil identification, and oil and grease analyses should be cleaned by the following steps:
 1. Wash equipment with a non-phosphate detergent-solution (e.g. Alconox) and a brush.
 2. Rinse with tap water.
 3. Rinse with reagent grade methanol.
 4. Rinse thoroughly with deionized water.
 5. For surface water or ground water, rinse the equipment two to three times with the media being sampled prior to collecting a sample.
 6. Repeat this at each location.
- Equipment to be used for collection of semi-volatile organics (which include base-neutral extractables, PCBs, herbicides and pesticides) should be cleaned by the following steps:
 1. Wash equipment with a non-phosphate detergent-solution (e.g. Alconox) and a brush.
 2. Rinse with tap water.
 3. Rinse with technical grade acetone.
 4. Rinse with pesticide grade hexane.
 5. Rinse thoroughly with deionized water.
 6. For water samples, rinse the equipment two to three times with the media being sampled before collecting the sample.
 7. Repeat this procedure at each sampling location.
- Equipment used for collection of samples for volatile organics analysis should be cleaned by the following steps:
 1. Wash equipment with a non-phosphate detergent solution (e.g. Alconox) and a brush.
 2. Rinse with tap water.

3. Rinse with reagent grade methanol.
 4. Rinse thoroughly with deionized water.
 5. For water samples, rinse the equipment two to three times with the media being sampled before collecting a sample.
 6. Repeat this procedure at each location.
- Steam cleaning is another acceptable technique for field decontamination.

The source of rinse water is often from nearby public sources. This should be noted and a sample collected as described in Section 6.1-3.3.

More than one method or compound may be used in series for a particular site. In extreme cases, disposable equipment is recommended over decontamination. This is because the level of effort and costs required to adequately clean the equipment and dispose of the cleaning solutions may not be warranted.