Appendix A

Standard Operating Procedures

STANDARD OPERATING PROCEDURE

Decontamination of Field Sampling Equipment

Revision 1

November 24, 2006

Approved By:	Andrea Nord			11/17/06
	Print	QA Manager(s)	Signature	Date
	KEVIN McGILP			11/17/06
	Print	Field Technician(s)	Signature	Date

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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	CF	Date:	03-03-09
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for the Decontamination of Field Sampling Equipment

Purpose

The purpose of this procedure is to define the process used for decontaminating all sampling-related equipment including pumps, meters, and materials coming into contact with actual sampling equipment or with sampling personnel. Bailers, protective gear, and filtration devices will be discarded after one use. Stainless steel bailers are used once and returned to an independent laboratory for decontamination.

Applicability

This procedure is applicable to all personnel who are collecting samples and/or decontaminating sampling and field equipment

Equipment

Alconox®
Scrub brush made of inert materials
Distilled or Deionized rinse water
Bucket
Field Log Data Sheets
Field Log Cover Sheets
Field Log Data Reports

Responsibilities

The Equipment Technician is responsible for ensuring all field equipment has been thoroughly decontaminated and prepared for use out in the field. The field technician(s) are responsible for decontamination in the field at each individual sampling point.

Procedure

Decontamination of monitoring well equipment will be performed by the field technician(s) before sampling and after working at each sampling point. All equipment will be handled in a manner that minimizes cross-contamination between points. After cleaning, the equipment will be visibly inspected to detect any residues or other substances that may exist after normal cleaning. If inspection reveals that decontamination was insufficient, the decontamination procedures will be repeated.

Equipment will be decontaminated in the following manner:

1. Equipment that does **not** contact sample water or the inside of the well:

    a. Rinse with clean control water.

    b. Inspect for remaining particles or surface film and repeat cleaning and rinse procedures if necessary.

2. Equipment that contacts sample water or the inside of the well:

    a. Clean (inside and outside where possible) with an Alconox®/clean-water solution applied with a scrub brush made of inert materials.

    b. Rinse with clean control water.

    c. Inspect for remaining particles or surface film and repeat cleaning and rinse procedures if necessary.

    d. Shake off remaining water and allow to air dry.

The internal surfaces of pumps and tubing that cannot be adequately cleaned by the above methods alone will also be cleaned by circulating decontamination fluids through them. The fluids will be circulated through this equipment in the order shown above. Special care will be exercised to ensure that the "rinse" fluids will be circulated in sufficient quantities to completely flush out contaminants and detergents.

When transporting or storing equipment after cleaning, the equipment will be protected in a manner that minimizes the potential for contamination.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

Documentation

The technician(s) will document the field equipment decontamination procedures on Field Log Data Sheets, Field Log Cover Sheets, and Field Log Data Reports or a project dedicated Field Log book.

Attachments

Attachment 1: Field Sampling Report
Attachment 2: Field Log Cover Sheet
Attachment 3: Field Log Data Sheet

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

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Laboratory Analysis Status

<Name inserts here>
Environmental Technician

Document1

Attachment 2
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Attachment 3
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample Time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:								
Casing diameter:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						
Others present:								
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)								
CASING & CAP: COLLAR: LOCK: OTHER:								
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:								
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-								
oil,grease- bacteria- total metal- filtered metal- methane- filter-								
Others:								

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\FieldLogDataSheet.doc

STANDARD OPERATING PROCEDURE

Soil Sample Collection Tools Decontamination – Level I

Revision 2

March 3, 2009

Approved By:	Andrea Nord			03/03/09
	Print	QA Manager(s)	Signature	Date
	Chris J. Frerich			03/03/09
	Print	Field Technician(s)	Signature	Date

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Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for the Soil Sample Collection Tools Decontamination – Level I

Purpose

The purpose of this SOP is to describe the proper techniques for equipment decontamination to meet level I protocol.

Applicability

This SOP applies to any field technician who is collecting environmental samples or is otherwise tasked with decontaminating field equipment for level I decontamination protocol.

Equipment

Tap water
Alconox®

Brush
Deionized water or distilled water
Bucket
Gloves

Responsibilities

The environmental technician(s) and/or Equipment technician is responsible for the proper equipment decontamination; quality control procedures and documentation.

Discussion

A variety of samplers (split-barrel, split-barrel with brass liners, piston sampler, backhoe, hand-auger, or shovel) may be used to retrieve soil from sampling locations. The soil sample will either be sealed within the sampler (e.g., collecting volatile samples) or the soil sample will be transferred to laboratory-supplied containers depending on the analysis to be conducted on the soil sample. The equipment required to transfer the soil from the sampler to the laboratory-supplied sample containers includes: stainless-steel spoons or scoops and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan.

Decontamination Procedures

All soil sampling equipment will be carefully cleaned before and during soil or sediment sampling. All sampling tools including split-barrels, stainless-steel spoons and scoops will be cleaned before use and between samples in the following manner:

1. Clean in a tap water and Alconox® solution, using a brush if necessary to remove particulate matter and films.

2. Rinse three times with tap water.

3. Rinse three times with deionized or distilled water.

4. Inspect equipment and repeat procedure if any residual soil or visible contaminants are present.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

Documentation

The environmental and/or equipment technician is responsible for the proper decontamination of the equipment and the proper documentation in the Field Sampling Report and /or Field Log book.

Attachments

Attachment 1: Field Sampling Report

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

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Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

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STANDARD OPERATING PROCEDURE

Soil Sample Collection Tools Decontamination – Level II

Revision 2

March 3, 2009

Approved By:	Andrea Nord			03/03/09
	Print	QA Manager(s)	Signature	Date
	Chris J. Frerich			03/03/09
	Print	Field Technician(s)	Signature	Date

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Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for the Soil Sample Collection Tools Decontamination – Level II

Purpose

The purpose of this SOP is to describe the proper techniques for equipment decontamination to meet level II protocol.

Applicability

This SOP applies to any field technician who is collecting environmental samples or is otherwise tasked with decontaminating field equipment for level II decontamination protocol.

Equipment

Tap water
Alconox®
Brush
Deionized water or distilled water
Bucket
Methanol
Aluminum Foil
Chem-wipe™
Gloves

Responsibilities

The environmental technician(s) and/or equipment technician is responsible for the proper equipment decontamination; quality control procedures and documentation.

Discussion

A variety of samplers (split-barrel, split-barrel with brass liners, piston sampler, backhoe, hand-auger, or shovel) may be used to retrieve soil from sampling locations. The soil sample will either be sealed within the sampler (e.g., collecting volatile samples) or the soil sample will be transferred to laboratory-supplied containers depending on the analysis to be conducted on the soil sample. The equipment required to transfer the soil from the sampler to the laboratory-supplied sample containers includes: stainless-steel spoons or scoops and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan.

Decontamination Procedures

All soil sampling equipment will be carefully cleaned before and during soil sampling. All sampling tools including split-barrels, stainless-steel spoons and scoops will be cleaned before use and between samples in the following manner:

1. Clean in a tap water and Alconox® solution, using a brush if necessary to remove particulate matter and films.

2. Rinse three times with tap water. Discharge water to the ground.

3. Rinse three times with deionized or distilled water. Discharge water to ground.

4. Rinse once with methanol. Collect and containerize the methanol rinse.

5. Inspect equipment and repeat procedure if any residual soil or visible contaminants are present.

6. Dry sampler with Chem-wipe™ or appropriate disposable replacement.

At the completion of the work day, the samplers should be decontaminated following the procedure outlined above and wrapped in aluminum foil for storage.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

Documentation

The environmental and/or equipment technician is responsible for the proper decontamination of the equipment and the proper documentation in the Field Sampling Report and /or Field Log book.

Attachments

Attachment 1: Field Sampling Report

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

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Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

STANDARD OPERATING PROCEDURE

Field Screening Soil Samples

Revision 2

August 27, 2007

Approved By:	Andrea Nord	Andrea Nord		8/27/07
	Print	QA Manager(s)	Signature	Date
	KEVIN McGILP			8/27/07
	Print	Field Technician(s)	Signature	Date

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and the SOP still reflects current practice.

Initials:	CF	Date:	03-03-09
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedure for Field Screening Soil Samples

Purpose

To describe the procedure for properly screening soil or sediment samples in the field.

Applicability

This procedure applies to all field technicians responsible for field screening soil or sediment samples.

Definitions

PPE Personal protective equipment
PID Photoionization Detector
FID Flame Ionization Detector

Equipment

PPE (gloves, safety glasses)
Project Health and Safety Plan
Quart-sized-self-sealing Polyethylene bag
Photoionization detector (PID)
Flame ionization detector (FID)
Thermometer
Indelible ink pen or pencil
Stainless-steel spoon
Squirt bottle with tap water
Logbook
Alconox®
Brush

Responsibilities

The environmental technician(s) is responsible for the proper sample identification; field screening procedures; field equipment and calibration; quality control procedures and documentation.

Procedure

The field screening techniques for soils are as follows: (1) visual examination; (2) odor; (3) headspace organic vapor screening; and (4) oil sheen. The results of these four screening procedures may be used to screen soil samples for possible contamination.

- **Visual Examination.** A visual examination of the soil sample will include noting any discoloration of the soil or visible oiliness or tar.

- **Odor.** The sampler will note odor only if noticed incidentally while handling the soil sample. Samplers will not unduly expose themselves to sample odors. Odor will be described as light, moderate, or strong, and appropriate description of the type and odor, if evident.

- **Headspace Organic Vapor Screening.** The polyethylene bag headspace method recommended by the Minnesota Pollution Control Agency will be used in the field to screen soils suspected to contain volatile organic compounds. The screening method is intended to be used in conjunction with other "real time" observations.

  The following equipment is required to conduct headspace organic vapor screening: photoionization or flame ionization detector (PID or FID), self-sealing quart-sized polyethylene bag, a log book or record sheet, and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan (PHASP). The meter shall be calibrated daily or more frequently if suspect data is obtained.

  The following procedure will be used for checking the calibration of the flame ionization detector:

  FID calibration check is conducted using a two point calibration process with methane gas. Calibrate the instrument by analyzing the calibration gas at 100 ppm and 1,000 ppm. If instrument values exceed ± 5% from true value, then the FID needs to be recalibrated.

  Reference the Standard Operating procedure for the TVA1000B (FID) for further information.

  The following procedure will be used for checking the calibration of the PID:

  PID calibration check is conducted using isobutylene calibration gas. Analyze a sample of the calibration gas, evaluate result, if result exceeds ± 5% from true value, then the PID needs to be recalibrated.

  Reference the Standard Operating procedure for the HNU PI-101 for further information.

The following procedure will be used for conducting headspace organic vapor screening:

1. Soil samples collected from a split-barrel sampler or a direct-push (i.e., Geoprobe®) sample liner will be collected immediately after opening the barrel or liner. If the sample is collected from an excavation wall, soil pile, or backhoe bucket, it will be collected from a freshly exposed surface.

2. Half-fill the bag with the sample to be analyzed using a stainless-steel spoon or a gloved hand and immediately seal it.

3. Agitate the bag for 15 seconds. Manually break up any soil clumps within the bag.

4. Allow headspace development for approximately 10 minutes. The sample should be kept in a shaded area out of direct sunlight. Ambient temperatures during headspace development should be recorded. When ambient temperatures are below 50°F, headspace development should be conducted inside a heated vehicle or building.

5. Agitate the bag for an additional 15 seconds.

6. Quickly puncture the bag with the sampling probe to a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particles.

7. Record the highest meter response as the headspace concentration. The maximum response will likely occur between 0 to 5 seconds.

8. When using a FID, it may be necessary to correct for methane. In this case, take a reading first with carbon filter, then without. This will require two duplicate bag samples. The second reading less the first is the headspace adjusted for methane. Adjusted readings less than zero are considered zero. Methane correction is not necessary if a PID is used.

- **Oil Sheen Test.** The oil sheen or hydrocarbon is a method used to immediately determine the approximate magnitude of coal tar contamination in soil by observation of the sample in the field. The test is useful in soils which do not have a high binding capacity with polyaromatic hydrocarbons (PAHs) (i.e., the PAHs are free on the surface of the soil particles and can be released by a stream of water).

  The equipment required to conduct the oil sheen test includes: a stainless-steel spoon, a squirt bottle filled with tap water, a log book or recording sheet, and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan. Decontamination of the spoon between test events will consist of scrubbing the surface of the spoon with a solution of Alconox® in water using a brush and then rinsing the spoon with water.

  The procedure for conducting the oil sheen test consists of obtaining approximately 50 grams (about 30 cc) of representative soil with the spoon and then directing a stream of water onto the soil in the spoon with the squirt bottle until the soil is saturated and water begins to collect around the soil. The amount of oil sheen present on the water is determined by observation and the results of the test are reported as a magnitude of oil sheen observed: none, trace, light, moderate, heavy or rainbow. The test results, sample location, and observations of the sample's appearance and odor are recorded in the log book.

  The specific soil types at the area of investigation should be accounted for when performing the oil sheen test. The best results are obtained in silts, sands, and/or gravels with low organic content. The results obtained from clayey soils may appear deceptively low. Typical descriptions of each test result are given below.

Oil Sheen Test Result	Description
None	No sheen detected.
Trace	Possible or faint oil sheen observed (may not continue to generate sheen as additional water is added).
Light	Obvious sheen that may not cover entire water surface
Moderate	Definite oil sheen that covers entire surface, but "rainbow colors" not distinguishable.
Heavy	Definite oil film or product that does not display rainbow colors.
Rainbow	Definite oil sheen, film or product that displays rainbow colors.

Interferences

Interferences on the test can be caused by any contaminant which will cause an oil sheen on water. The samples will be carefully observed for characteristic appearance or odors which may indicate a possible contaminant other than coal tar. Sunlight and low temperatures may interfere with headspace development. Water and soil particles may interfere with PID and FID readings.

Documentation

The technician(s) will document the soil sampling events in a project dedicated field logbook or on field log data sheets.

Attachments

Attachment 1: Field Sampling Report
Attachment 2: Field Log Data Sheet

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

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Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

Attachment 2
Field Log Data Sheet

Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:									Number of Containers/ Analysis																	
Location:																										
Project #:																										
Project Name:																										
Sample Identification	Collection		Matrix			Type			2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:		
	Date	Time	Soil	Sludge		Grab	Comp.	QC																		
1.																										
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3.																										
4.																										
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STANDARD OPERATING PROCEDURE

Direct-Push Soil and Groundwater Sample Collection (Geoprobe®)

PCDOCS No.: 246127

Revision 2

March 13, 2009

Approved By:

Andrea Nord		03-13-09
Print QA Manager(s)	Signature	Date

John W. Juntilla		03-13-09
Print Field Technician(s)	Signature	Date

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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials: _____ _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Standard Operating Procedures for the Direct-Push Soil and Groundwater Sample Collection (Geoprobe®)

Purpose

The purpose of this standard operating procedures (SOP) is to describe the procedures for the collection of soil and/or groundwater samples when Geoprobe® field methods are used.

Applicability

This SOP will be utilized wherever direct-push (i.e., Geoprobe®) methods are employed for the retrieval of soil or groundwater from designated sampling locations.

Equipment

Direct-push soil sampling rig
Direct-push sampler liner
Direct-push probe
Extension rods
Screen (four-foot lengths)
Polyethylene tubing
Pump (peristaltic or vacuum)
Pre-cleaned-certified Sampling Containers
Alconox®
Deionized or tap water
Stainless steel spoons, scoops or trowels
Clean pair of surgical gloves
Appropriate personal protective equipment
Field notebook and/or Field Log Data Sheets
Chain of Custody Form
Sample Labels
Coolers
Bagged ice
Tape
Field balance (for soils)
Water-proof ink pen

References

Procedures for Ground Water Monitoring, Minnesota Pollution Control Agency Guidelines, December 1986
EPA: Title 40 of the Code of Federal Regulations

Responsibilities

The environmental technician(s) or geologist is responsible for the proper collection of soil and water samples, sample identification, quality control procedures, and documentation.

Procedure

1. Approximately one week before the sampling event, the appropriate sample containers should be ordered from the laboratory.

2. Before leaving for the site, account for all the containers.

3. When the sample is ready to be collected label the containers with the following information:
   - Project number
   - Location sampled
   - Individual collecting the samples
   - Date and time of sample collection
   - Sample analysis (if required by the laboratory)

   Note: Use an indelible permanent pen to avoid ink bleeding.

4. Put on a new pair of disposable sampling gloves at each sampling location.

Soil Sampling with a Direct-push Soil Boring Rig:

A. Preparation of Soil Sampling Equipment

All soil sampling equipment will be carefully cleaned before use. All sampling tools including stainless steel spoons/scoops/trowels will be cleaned before use and in between sampling locations by cleaning with deionized or tap water and Alconox®, using a brush if necessary to remove particulate matter or films and rinsed thoroughly with deionized water. To prevent sample cross-contamination, the sampler will put on a new pair at each sampling location. Direct-push sampler liners (soils) are one-time use and disposable.

B. Soil Sample Collection

Soils are generally continuously sampled using the direct push method. This method utilizes steel drive rods and a 2-inch outside diameter (O.D.) soil core sampler with a dedicated 1.75-inch inside diameter (I.D) removable acetate liner. The probe rods and sampling unit are driven to the desired sampling depth by the static weight of the carrier vehicle and hydraulic hammer percussion. Two or four-foot sample cores are typically collected. The assembly is brought to the surface and the soil sample is exposed by cutting open the acetate plastic liner. In most investigations, the entire cores are field screened for moisture, odor, oil sheen, discoloration and the presence of organic soil vapors and classified in accordance with ASTM D-2488, Standard Practice for Description and Identification of Soils

(Visual/Manual Method.) Soil sample field screening procedures are described in a separate standard operating procedure description.

1. Collecting Volatile Organic Samples

   Note: Samples for volatile analysis should be collected prior to any other analysis.

   A. Before beginning the collection of VOC soil samples, verify field balance using a 50 gram weight. If the balance is off by ± 5 grams, recalibrate the instrument following the manufacturer's recommendations.

   B. Cut open the liner using a knife or similar utensil.

   C. Using a stainless-steel spoon/trowel and a field balance, to collect 25 grams of soil in a laboratory-provided tarred sample container. Because samples for VOCs cannot be weighed and then collected in the pre-tarred jar, it is recommended that a 25 gram aliquot be measured and discarded first, to gauge the approximate volume of the 25 gram aliquot (based on soil type). Then, collect another equal aliquot for preservation and analysis. Depending on the laboratory that supplied the container, methanol may be provided in a snap-cap vial that will be opened and poured over the soil in the pre-tarred container or container will been received with the appropriate volume of methanol already added. In this case, avoid splashing the methanol when adding the soil volume.

   D. Wipe the jar lip and screw threads to remove soil and ensuring a tight seal with the lid of the container.

   E. Cool the sample to approximately 4°C immediately after collection.

2. Collecting Semivolatile Organic or Metals Samples (or any other soil sample)

   A. Cut open the liner using a knife or similar utensil.

   B. Retrieve sample using a clean stainless steel spoon/trowel. Fill sample jar, wipe the jar lip and screw threads to remove soil and ensuring a tight seal with the lid of the container. No preservatives are required for soil samples except VOCs.

   C. Cool the sample to approximately 4°C immediately after collection.

Groundwater Sampling with a Direct-push Soil Boring Rig:

Groundwater samples will be collected by advancing the direct-push probe to the desired sampling depth. When the sampling depth is reached, small diameter extension rods will be run through the steel probe rods to push out the expendable drive point. Next, a one-inch screen (four-foot length) is extended into the formation. Following screen placement, polyethylene tubing is placed into the temporary well, and a peristaltic pump (or equivalent) is used to draw water samples to the surface to be placed in appropriate vials or bottles for laboratory analysis.

After each well is constructed, the probe rods are washed in an Alconox®/water mixture and rinsed with water. The polyethylene tubing is discharged after each sample was collected and new tubing used for the

collection of the next sample. The temporary well locations will be abandoned following all State regulations.

Container volume, type, and preservative are important considerations in groundwater sample collection. Container volume must be adequate to meet laboratory requirements for quality control, split samples, or repeat examinations. The container type or construction varies with the analysis required: (1) septum-sealed 40-ml glass vial is used for volatile organic compounds; (2) semivolatile analyses usually require a glass container (note—amber-tinted glass prevents sunlight from affecting the sample); and (3) polyethylene containers are used for general parameters, metals, and inorganics. The analytical laboratory will preserve the container before shipment. Preservation and shelf life vary; contact the laboratory to determine if an on-hand container is still useful.

A. Groundwater Sample Collection

1. Volatiles—Use caution because concentrated acid may be present. Do not rinse or overfill glass vials. Hold bottle in one hand, the cap right side up in the other. Pour slowly, avoiding air bubbles and overfilling the vial. Cap tightly, invert the bottle, and tap gently. If any air bubbles appear in the vial, discard and collect sample in a new vial. After collecting the required number of vials (usually sets of 2 or 3, depending on the laboratory), insert them in a Ziplock® plastic bag and place in a cooler with ice.

2. Semivolatiles—Fill container slowly with a minimum headspace and cap tightly. Do not rinse glass containers. Place container directly in a cooler with ice.

3. Filtered Metals—Typically field filtering of groundwater samples collected from a Geoprobe® boring is not advised. Undeveloped temporary borings of this type will likely contain significant solids that would require several attempts to filter adequately. In these cases, the laboratory('s) can perform this filtering, if necessary. However this would require an **unpreserved** aliquot of sample for filtration and preservation (of nitric acid) at the laboratory. Should field filtering be required, see the Barr Engineering Co. Standard Operating Procedure for Filtering Groundwater Samples). Pour sample into metals sample container, minimizing headspace and avoiding spillage. Use caution handling metals containers because of nitric acid. Place directly in a cooler with ice.

4. Other Organics or Inorganics—Containers may contain acid(s), use caution when handling. Fill containers appropriately, rinsing any general unpreserved containers three times, minimizing splashing and spillage. Place container directly in a cooler with ice.

Quality Control Samples

The effectiveness of the sample handling techniques is monitored by collecting both preserved and unpreserved field blank samples. For additional information, consult the Barr Engineering Co. SOP for the Collection of Quality Control Samples.

Field (or Masked) duplicate samples will be collected to measure relative sampling (and laboratory) precision. The ratio of quality control samples are generally 1 field blank/field duplicate per twenty samples, however, specific project requirements may be determined by the QAPP/SAP for the project. These samples are collected at the same time using the same procedures, equipment, and types of containers as the required samples. They are also preserved in the same manner and are either co-located or split and submitted for the same analyses as the native sample(s).

Trip blanks are only applicable when sampling/analyzing for volatile organics. Their purpose is to determine if contamination has occurred as a result of improper sample container cleaning, contaminated blank source water, sample contamination during storage and transport due to exposure to volatile organics, or other environmental conditions during sampling and analysis. The water will be free of contaminants. The trip blanks are prepared, sealed and labeled appropriately at the lab, and transported to the field in the same containers as the sample vials. These blanks are not opened in the field. They are transferred to the coolers designated for volatile sample storage and transport and accompany the samples to the analytical laboratory.

Field blanks (or Rinsate Blanks) are used to evaluate the effects of sampling cross-contamination caused by inadequately decontaminated equipment. Their purpose is to determine if contamination has occurred as a result of improper equipment cleaning. Field blanks are prepared onsite by pouring analyte-free water through decontaminated sample collection equipment (bailer or pump) and collecting the rinsate in the appropriate sample container. The field blanks will be handled in the same manner as the sample group for which they are intended (i.e., blanks will be stored and transported with the sample group).

The volume of the sample obtained should be sufficient to perform all required analyses with an additional amount collected to satisfy the needs for quality control, split samples, or repeat examinations. The QA Staff should be consulted for any specific volume requirements.

The elapsed time between sample collection and initiation of each laboratory analysis will fall within a prescribed time frame. Holding times for samples required by this project are prescribed by EPA: Title 40 of the Code of Federal Regulations.

Water and Soil Sample Storage

The samples will be bubble wrapped or bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain of custody documentation. Samples will be kept cold (approximately 4°C) until receipt at the laboratory, where they are to be stored in a refrigerated area. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of fiberglass tape. All samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Note: Samples may have to be stored indoors in winter to prevent freezing.

Interferences/Discussion

Volatile and low-level mercury samples must be collected prior to any other analyses and metals must be collected prior to cyanide samples to avoid possible cross-contamination or other potential data quality issues. After collection, all samples should be handled as few times as possible. Samplers should use extreme care to ensure that samples are not contaminated. If samples are placed in a cooler, samplers should ensure that melted ice cannot cause sample containers to become submerged, as this may result in cross-contamination. Plastic bags, such as Ziplock® bags, should be used when small sample containers (e.g., VOC vials) are placed in coolers to prevent cross-contamination.

Some compounds can be detected in the parts per billion and/or parts per trillion range. Extreme care will be taken to prevent cross-contamination of these samples. A clean pair of new, disposable gloves will be

worn for each sample location. Sample containers for source samples or samples suspected of containing high concentrations of contaminants are placed in separate plastic bags and coolers immediately after collecting, preserving and tagging. Sample collection activities will proceed progressively from the least contaminated area to the most contaminated area (when known).

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

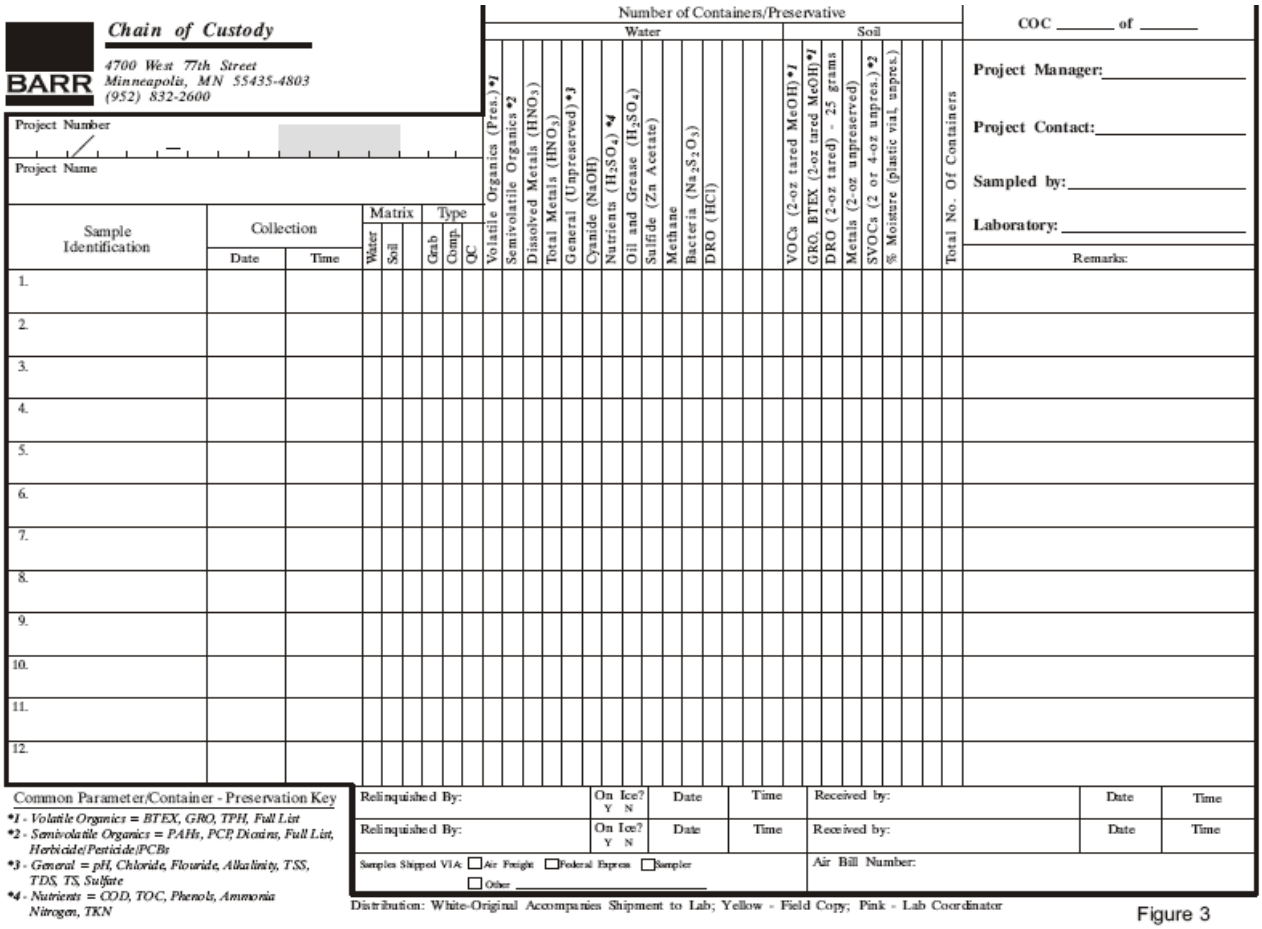
Documentation

The technician(s) will document the type and number of samples collected during each field event. All sample information will be documented in the field notebook, field log data sheet and chain-of-custody record.

Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Sampling Report
Attachment 5: Field Log Data Sheet

Attachment 1
Chain of Custody Form



Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number
Project Name

Number of Containers/Preservative

Water: Volatile Organics (Pres.) *1; Semivolatile Organics *2; Dissolved Metals (HNO_3); Total Metals (HNO_3); General (Unpreserved) *3; Cyanide (NaOH); Nutrients (H_2SO_4) *4; Oil and Grease (H_2SO_4); Sulfide (Zn Acetate); Methane; Bacteria ($Na_2S_2O_3$); DRO (HCl)

Soil: VOCs (2-oz tared MeOH) *1; GRO, BTEX (2-oz tared MeOH) *1; DRO (2-oz tared) - 25 grams; Metals (2-oz unpreserved); SVOCs (2 or 4-oz unpres.) *2; % Moisture (plastic vial, unpres.)

Total No. Of Containers

COC _____ of _____

Project Manager: _____

Project Contact: _____

Sampled by: _____

Laboratory: _____

Sample Identification	Collection Date	Collection Time	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Remarks
1.								
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.								
11.								
12.								

Common Parameter/Container - Preservation Key

*1 - Volatile Organics = BTEX, GRO, TPH, Full List
*2 - Semivolatile Organics = PAHs, PCP, Dioxins, Full List, Herbicide/Penticide/PCBs
*3 - General = pH, Chloride, Flouride, Alkalinity, TSS, TDS, TS, Sulfate
*4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other _____				Air Bill Number:		

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

Figure 3

CHAIN OF CUSTODY

Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

Preservative: _____

Sampled By: _____

Sample Location: _____

_____

Attachment 3
Custody Seal – if applicable

Custody Seal

Date_____ Project_____

Signature _____Container#_____ of_____

Attachment 4
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Attachment 5
Field Log Data Sheet

Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:
Location:
Project #:
Project Name:

Sample Identification	Collection		Matrix			Type			Number of Containers/ Analysis																	
	Date	Time	Soil	Sludge		Grab	Comp.	QC	2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:
1.																								
2.																								
3.																								
4.																								
5.																								
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20.																								

STANDARD OPERATING PROCEDURE

SOIL SAMPLE COLLECTION

Revision 2.0

March 3, 2009

Approved By:	Andrea Nord			03-06-09
	Print	QA Manager(s)	Signature	Date
	Chris J. Frerich			03-06-09
	Print	Field Technician(s)	Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedure for Soil Sample Collection

Purpose

To describe the collection of soil samples for volatiles, semivolatiles, metals, inorganics, bacteria, and dioxin analysis in soil.

Applicability

This procedure applies to the collection of soil samples by the sampling technician(s). It identifies each container type (volume, construction, preservative) required for each category of analyses, their corresponding holding times and collection procedures from a variety of sources.

Definitions

Holding Time. Period of time between sample collection and when the sample is analyzed.

Sample Preservation. The stability of analytes depends upon how well the samples are preserved.

Equipment

Sampler media
Pre-cleaned-certified Sampling Containers
Stainless Steel Spoons
Balance
Coolers
Ziploc® Baggy
Ice
Water-proof ink pen or pencil
Gloves
Alconox®
Chain of Custody Form
Sample Label
Custody Seal – if applicable
Field Sampling Report
Field Log Cover Sheet
Field Log Data Sheet

Responsibilities

The Field Operations/QA Officer or the environmental technician(s) will order the sample containers prior to the sampling event. The environmental technician(s) is responsible for the proper collection of soil samples, sample identification, quality control procedures, and documentation.

Procedure

Examples of samplers include split-barrel, split-barrel with brass liners, Geoprobe® sleeves, piston samplers, backhoe, or shovels may be used to retrieve soil from sampling locations. Depending upon the analyses to be conducted on the soil sample, the soil sample will either be sealed within the liner or sleeve or the soil sample will be transferred to a certified-laboratory-supplied container. The equipment required to transfer soil from the sampler to the sample container includes: stainless steel spoons, or scoops and the appropriate personal protective equipment necessary for collection and handling of soil. Volatile samples will be collected from representative areas of soil that were least disturbed first, then the remaining soil will be mixed and collected for the remaining analyses.

All soil sampling equipment will be carefully cleaned before and during soil sampling. All sampling tools including split-barrels, stainless steel spoons and scoops will be cleaned before use and between samples in the following manner: (1) clean with tap water and a phosphate–free detergent such as

Alconox®, using a brush if necessary to remove particulate matter and films; (2) rinse three times with tap water; and (3) rinse three times with deionized water. To prevent sample cross-contamination, the sampler will discard the outer pair of sample gloves and put on a new pair between each sample event.

Collecting Volatile Organic Samples

Soil samples will be collected for analysis by either a drilling apparatus equipped with a split-barrel, core barrel sampler or by hand excavation. Volatile samples should be collected first. The soil selected for collection should be the most undisturbed sample possible.

It is important to note that there are different jar sizes and sampling media available for collecting a soil sample for volatile organic compounds (VOCs). The table below describes the sample volumes and preservation techniques for the most common sampling media.

Summary of Typical Sampling Media and Soil Volumes Used for Volatile Organic Compound Determination

VOC Sample Media	Preservative	Volume of Preservative (mL)	Volume of Sample (g)
2 oz. glass jar with PTFE-lined lid	MeOH, cool 4 °	10	10
	MeOH, cool 4 °	25	25
4 oz. glass jar with PTFE-lined lid	MeOH, cool 4 °	10	10
	MeOH, cool 4 °	25	25
Encore® Sampler			
5 gram device	Freeze or extrude into chemical preservative	Maintain a 1:1 ratio of soil to preservative if chemical preservation is used.	5
25 gram device	Freeze or extrude into chemical preservative	Maintain a 1:1 ratio of soil to preservative if chemical preservation is used.	25
Terracore® Kit			
1 MeOH and 2 water preserved glass vial	MeOH, cool 4 °	5	5
	Water Submersion, cool 4 °	5	5
1 MeOH and 2 sodium bisulfite preserved glass vials	MeOH, cool 4 °	5	5
	Sodium Bisulfite, cool 4 °	5	5

The following procedure applies to soil samples retrieved with a drilling apparatus equipped with a split-barrel sampler or core barrel with liners (Skip to the next section if Encore® sampler or other coring device is used):

1. Open the split-barrel sampler.

2. Open a representative liner containing soil.

3. Using a stainless-steel spoon, weigh the desired aliquot (25 g. or 10 g.) of a representative soil sample on a field balance. Once a weight/volume estimate has been established, discard the soil and collect untouched soil, from the same source for step 4.

4. Using a stainless-steel spoon, place soil in a laboratory-provided-pre-weighed sample container containing methanol (avoid splashing the methanol).

5. Wipe the container lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

6. Cool the sample to approximately 4±2°C immediately after collection.

The following procedure applies to the collection of hand-excavated soil samples:

1. Dig to the desired sampling interval, exposing fresh soil surface to sample.

2. Collect a large sample on a shovel or in a bucket auger and bring it to the surface or collect the sample directly from the fresh soil surface.

3. Using a stainless-steel spoon, weigh the desired aliquot (25 g. or 10 g.) of a representative soil sample on a field balance. Once a weight/volume estimate has been established, discard the soil and collect untouched soil, from the same sample source for step 4.

4. Using a stainless-steel spoon, place the desired aliquot (25 g. or 10 g.) of soil in a pre-weighed-laboratory-provided sample container containing methanol (avoid splashing the methanol).

5. Wipe the jar lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

6. Cool the sample to 4±2°C immediately after collection

Collecting Volatile Organic Samples with the Encore® Sampler or other soil coring device

The following procedure applies collecting VOC samples of soil with the Encore® sampler device:

1. Hold the Encore® coring body and push plunger down until small o-ring rests against tabs to ensure the plunger moves freely.

2. Depress locking lever on T-Handle. Place coring body plunger end first into the open end of the T- Handle, aligning the slots on the coring body with the locking pins in the T-Handle. Twist coring body clockwise to lock pins in slots. Check to insure sampler is locked in place.

3. Turn T-handle with T-up and coring body down. This positions the plunger bottom flush with bottom of coring body. Using T-Handle, push sampler into soil until coring body is completely

full. When full the small o-ring will be centered in the T-Handle viewing hole. Remove excess soil from the coring body exterior.

4. Cap the coring body while it is still on the T-Handle. Push and twist cap over bottom until grooves on locking arms seat over ridge on coring body. Remove from T-Handle, lock plunger by rotating extended plunger rod fully counterclockwise until wings rest firmly against tabs, and attach label.

5. Cool the sample to 4±2°C immediately after collection.

Collecting Semivolatile Organic, Wet Chemistry and Metals Samples– except WI DRO

Soil samples will be collected for analysis by either a drilling rig equipped with a Geoprobe® sleeve, split-barrel, core barrel sampler or by hand excavation.

Please review the SOP for Direct Push Soil and Groundwater Sample Collection when Geoprobe® sleeves are used.

The following procedure applies to soil samples retrieved with a drilling rig equipped with a split-barrel sampler or core barrel with brass liners:

1. Open the split-barrel sampler.

2. Select a representative brass liner filled completely with soil.

3. Wrap the ends of the brass liners with heavy-duty aluminum foil, taking care to not piece the foil. Tape the foil to the brass liner with duct tape to ensure a seal. Cover the ends of the liner with plastic caps or duct tape to fully protect the foil.

4. Cool the sample to 4±2°C immediately after collection.

The following procedure applies to the collection of hand-excavated soil samples or core barrel samples:

1. Dig to the desired sampling interval, exposing fresh soil surface to sample.

2. Collect a large sample on a shovel or in a bucket auger and bring it to the surface or collect the sample directly from the fresh soil surface.

3. Using a stainless-steel spoon, composite the soil, pack the soil into the sample jars, leaving no headspace.

4. Wipe the container lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

5. Cool the sample to 4±2°C immediately after collection.

WI Diesel Range Organic (WIDRO) Samples

Soil samples will be collected for analysis by either a drilling apparatus equipped with a split-barrel, core barrel sampler or by hand excavation. Volatile samples should be collected first. The soil selected for collection should be the most undisturbed sample possible.

The following procedure applies to soil samples retrieved with a drilling apparatus equipped with a split-barrel sampler or core barrel with liners:

1. Open the split-barrel sampler.

2. Open a representative liner containing soil.

3. Using a stainless-steel spoon, weigh 25 ± 5 grams of a representative soil sample on a field balance. Once a weight/volume estimate has been established, discard the soil and collect untouched soil, from the same source for step 4.

4. Using a stainless-steel spoon, place 25 ± 5 grams of soil in a laboratory-provided-pre-weighed sample container.

5. Wipe the container lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

6. Cool the sample to 4±2°C immediately after collection.

The following procedure applies to the collection of hand-excavated soil samples:

1. Dig to the desired sampling interval, exposing fresh soil surface to sample.

2. Collect a large sample on a shovel or in a bucket auger and bring it to the surface or collect the sample directly from the fresh soil surface.

3. Using a stainless-steel spoon, weigh 25 ± 5 grams of a representative soil sample on a field balance. Once a weight/volume estimate has been established, discard the soil and collect untouched soil, from the same sample source for step 4.

4. Using a stainless-steel spoon, place 25 ± 5 grams of soil in a pre-weighed-laboratory-provided sample container containing methanol (avoid splashing the methanol).

5. Wipe the container lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

6. Cool the sample to 4±2°C immediately after collection

Collecting Soil Quality Control Samples

Trip blanks are only used when sampling for volatile organics. Their purpose is to determine if contamination has occurred as a result of improper sample container cleaning, sample contamination during storage and transport due to exposure to volatile organics, or other environmental conditions during sampling or analysis. Trip blank samples are prepared prior to the sampling events by the laboratory providing the sample containers. The certified-pre-weighed methanol (MeOH) containers will be free of contaminants. The trip blank samples are prepared by the lab, sealed, labeled appropriately by the lab, and transported to the field in the same containers as the sample containers.

These blanks are not opened in the field. They are transferred to the cooler designated for volatile sample storage and transport and accompany the samples to the analytical laboratory.

Field (or Masked) duplicate samples will be collected to measure relative sampling precision. Five percent of all samples collected are collected in duplicate. These samples are collected at the same time using the same procedures, equipment, and types of containers as the required samples. They are also preserved in the same manner and are either co-located or split and submitted for the same analyses as the required samples.

Some general considerations will be taken into account when planning and conducting sampling operations. The sampler will take into consideration the required sample volume, sample holding times, sample handling, and special precautions for trace contaminant sampling.

The volume of the sample obtained should be sufficient to perform all required analyses with an additional amount collected to satisfy the needs for quality control, split samples, or repeat examinations. The Laboratory Coordinator should be consulted for any specific volume requirements.

The elapsed time between sample collection and initiation of each laboratory analysis will fall within a prescribed time frame. Holding times for samples required by this project are prescribed by EPA: Title 40 of the Code of Federal Regulations.

After collection, all samples should be handled as few times as possible. Samplers should use extreme care to ensure that samples are not contaminated. If samples are placed in a cooler, samplers should ensure that melted ice cannot cause sample containers to become submerged, as this may result in cross-contamination. Plastic bags, such as Ziplock® bags, should be used when small sample containers are placed in coolers to prevent cross-contamination.

Some compounds can be detected in the parts per billion and/or parts per trillion range. Extreme care will be taken to prevent cross-contamination of these samples. A clean pair of new, disposable gloves will be worn for each sample location. Sample containers for source samples or samples suspected of containing high concentrations of contaminants are placed in separate plastic bags and coolers immediately after collecting, preserving and tagging. Sample collection activities will proceed progressively from the least contaminated area to the most contaminated area (when known).

Sample Storage

Immediately after samples are collected, they will be placed in a cooler containing bagged ice. Samples will be kept cold (approximately 4±2°C) until receipt at the laboratory, where they are to be stored in a refrigerated area. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of tape. All samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Note: Samples may have to be stored indoors in winter to prevent freezing.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

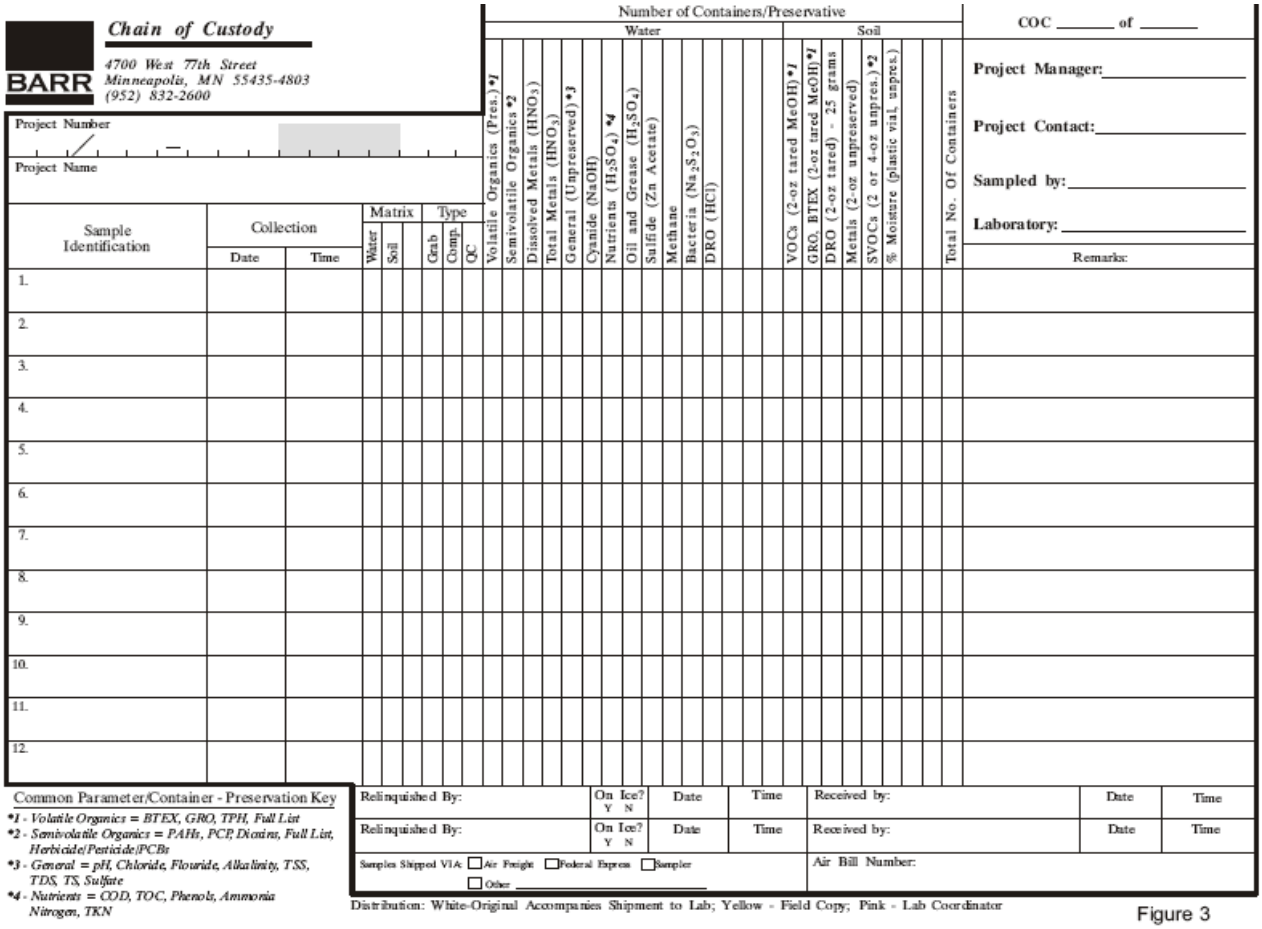
Documentation

The technician(s) will document the soil sampling events in a project dedicated field logbook or on field log data sheets. They will also document the type and number of bottles the field log data sheets and chain-of-custody record. The analysis for each bottle and the laboratory used will be documented on the chain-of-custody record. The sampling request form will document which sampling containers are used for which soil samples.

Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Log Data Sheet

Attachment 1
Chain of Custody Form



Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number
Project Name

Number of Containers/Preservative

Water: Volatile Organics (Pres.)*1; Semivolatile Organics *2; Dissolved Metals (HNO_3); Total Metals (HNO_3); General (Unpreserved)*3; Cyanide (NaOH); Nutrients (H_2SO_4)*4; Oil and Grease (H_2SO_4); Sulfide (Zn Acetate); Methane; Bacteria ($Na_2S_2O_3$); DRO (HCl)

Soil: VOCs (2-oz tared MeOH)*1; GRO, BTEX (2-oz tared MeOH)*1; DRO (2-oz tared) - 25 grams; Metals (2-oz unpreserved); SVOCs (2 or 4-oz unpres.)*2; % Moisture (plastic vial, unpres.)

Total No. Of Containers

COC _____ of _____

Project Manager: _____
Project Contact: _____
Sampled by: _____
Laboratory: _____

Sample Identification	Collection Date	Collection Time	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Remarks
1.								
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.								
11.								
12.								

Common Parameter/Container - Preservation Key
*1 - Volatile Organics = BTEX, GRO, TPH, Full List
*2 - Semivolatile Organics = PAHs, PCP, Dioxins, Full List, Herbicide/Pesticide/PCBs
*3 - General = pH, Chloride, Flouride, Alkalinity, TSS, TDS, TS, Sulfate
*4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other _____				Air Bill Number:		

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

Figure 3

CHAIN OF CUSTODY

Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

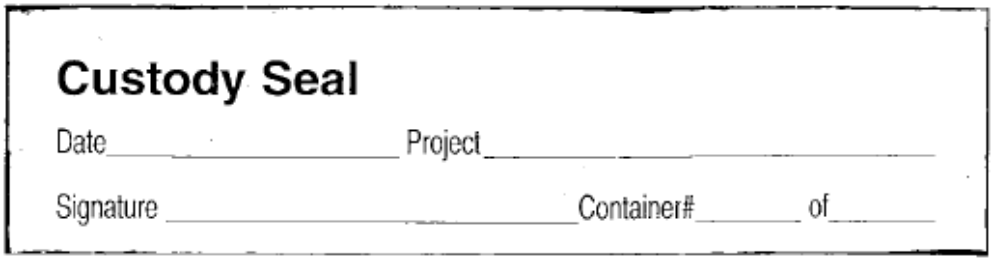
Preservative: _____

Sampled By: _____

Sample Location: _____

_____

Attachment 3
Custody Seal



Attachment 4
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:
Location:
Project #:
Project Name:

Sample Identification	Collection		Matrix			Type			Number of Containers/ Analysis																		
	Date	Time	Soil	Sludge		Grab	Comp.	QC	2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:
1.																								
2.																								
3.																								
4.																								
5.																								
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STANDARD OPERATING PROCEDURE

Soil Sample Compositing

Revision 1

November 3, 2006

Approved By:	Andrea Nord			11/17/06
	Print	QA Manager(s)	Signature	Date
	KEVIN McGILP			11/17/06
	Print	Field Technician(s)	Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	CF	Date:	03-03-09
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for Soil Sample Compositing

Purpose

The purpose of this SOP is not to define the representative number of sub-samples, but to describe the procedures of compositing several discrete samples into one representative sample for analysis.

Applicability

This SOP applies to samples collected from any site where it is determined that samples be composited prior to analysis at the laboratory.

Definitions

Sub-sample A representative, homogeneous portion or aliquot of a sample that is removed from an individual sample or the aggregate sample for preparation and measurement of the sample submitted for analysis.
Composite Sample A collection of more than one sample of the same medium from the same type of surface, such that multiple samples can be combined and analyzed as a single sample. ***Discrete Sample*** A sample that originated from a specific area at a specific time.

Equipment

Stainless steel spoons or scoops
Large stainless steel mixing bowl
Sampler media
Pre-cleaned-certified Sampling Containers
Coolers
Ziploc® Baggy
Ice
Water-proof ink pen or pencil
Gloves
Tap water
Deionized water
Drill rig

References

Barr Engineering Co. SOP Soil Sample Collection

Responsibilities

The environmental technician(s) is responsible for the proper sample identification; field screening procedures; quality control procedures and documentation. The role of the Health and Safety Officer is to oversee all aspects of job safety. The Barr Project Manager in conjunction with the client develops the site specific work plan or sample and analysis plan to define the scope of work.

Discussion

Both composite and discrete samples can be used for environmental investigations. Composite samples are valuable in characterizing a large area or volume of soil. Detailed guidance for sub-sample collection is given from specific programs (e.g., Minnesota Department of Agriculture Agricultural Chemical Incidents; MCPA's Leaking Petroleum Storage Tanks). In general, sampling the total investigation area and final numbers of sub-samples should be appropriate to meet the data quality objectives for the project. The work plan or SAP should contain the detailed information regarding the ratio of the total investigation soil area to final composited sample numbers.

Discrete soil samples identified for compositing can be collected in several ways. These include a drilling rig equipped with a split-barrel, core-barrel sampler, or by hand excavation. Additional information on soil sample collection can be found in the SOP for soil sample collection.

Procedure

The samples should be labeled discretely, and stored at 4ºC until each individual sample is obtained. A minimum volume of soil obtained during discrete sampling will be dependent on the final analytical requirements for the composite sample. A minimum volume of soil sufficient to fill two 4- or 8-ounce glass or Teflon containers should be obtained for compositing. This volume would be ample for analysis of semivolatiles, PCBs, pesticides, metals.

Note: Analytical samples should not be collected from polyethylene bags sometimes used for field screening purposes. Volatile organic samples should not be composited, due to aeration of the sample during mixing.

A. Sampling Equipment Preparation
   All soil compositing equipment will be carefully cleaned between uses in following manner: (1) clean with tap water and TSP using a brush, if necessary, to remove particulate matter and films; (2) rinse three times with tap water; and (3) rinse three times with deionized water. To prevent sample cross-contamination, the sampler will discard the outer pair of sample gloves and put on a new pair between each compositing event.

B. Compositing Discrete Samples

   1. After individual samples have been obtained, compositing begins by documenting the discrete sample locations to be included in a final composited sample. Appropriate laboratory containers should be labeled with this final sample identifier and the date of collection.

   2. Retrieve from storage the samples selected for compositing. One container from each discrete sample location should remain in storage in case individual sample confirmations are necessary.

   3. Empty the entire contents of each container into the stainless steel mixing bowl, removing any large debris or rocks. Mix thoroughly.

   4. Fill appropriate laboratory sample containers.

5. Complete chain-of-custody documentation.

6. Immediately after samples are composited, they should be placed in a cooler containing ice or ice packs and cooled at 4°C for shipment to the laboratory.

Sample Storage

The samples will be bubble wrapped or bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain of custody documentation. Samples will be kept cold (approximately 4°C) until receipt at the laboratory, where they are to be stored in a refrigerated area. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of fiberglass tape. All samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Note: Samples may have to be stored indoors in winter to prevent freezing.

Investigative Derived Waste Sample Collection

Ensure enough representative sample is collected to provide adequate sample volume for all analyses used to characterize the IDW for disposal purposes. When collecting solid IDW sample volume, take moisture content into consideration when determining how much sample to provide to the laboratory for TCLP analysis. If the solid sample contains <5 percent by volume solid material, then enough liquid/solid sample must be obtained to provide the laboratory with an adequate supply to meet TCLP sample volume requirement guidelines. See the IDW Sample Collection SOP for further information.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

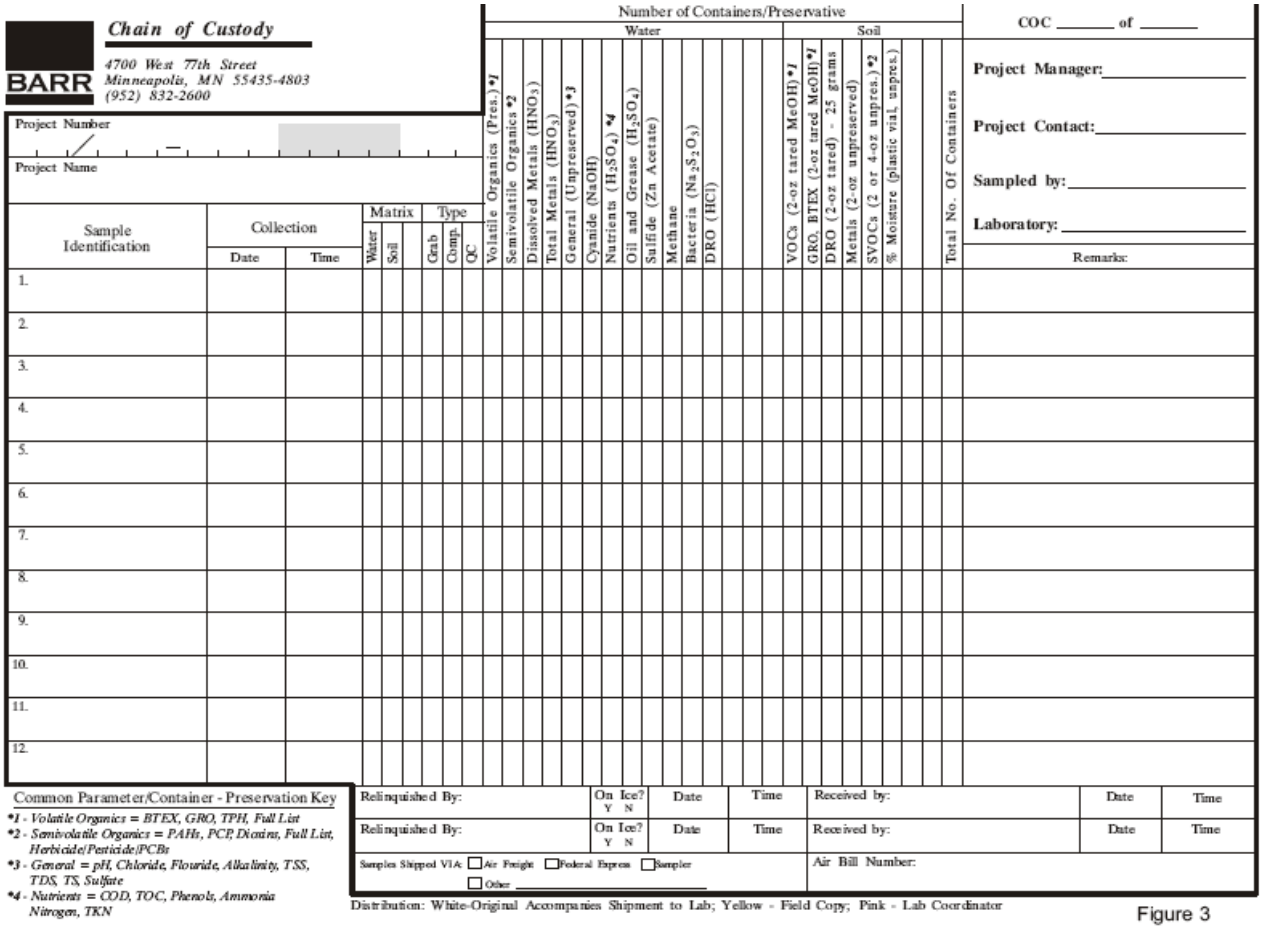
Documentation

The technician(s) will document the soil sampling events on field log data sheets or project dedicated Field Log book. They will also document the type and number of bottles on both the field log data sheet and chain-of-custody record. The analysis for each bottle and the laboratory used will be documented on the chain-of-custody record. The sampling request form will document which sampling containers are used for which soil samples.

Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Log Data Sheet - Soil

Attachment 1
Chain of Custody Form



Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number
Project Name

Number of Containers/Preservative

Water: Volatile Organics (Pres.)*1; Semivolatile Organics *2; Dissolved Metals (HNO_3); Total Metals (HNO_3); General (Unpreserved)*3; Cyanide (NaOH); Nutrients (H_2SO_4)*4; Oil and Grease (H_2SO_4); Sulfide (Zn Acetate); Methane; Bacteria ($Na_2S_2O_3$); DRO (HCl)

Soil: VOCs (2-oz tared MeOH)*1; GRO, BTEX (2-oz tared MeOH)*1; DRO (2-oz tared) - 25 grams; Metals (2-oz unpreserved); SVOCs (2 or 4-oz unpres.)*2; % Moisture (plastic vial, unpres.)

Total No. Of Containers

COC _____ of _____

Project Manager: _____
Project Contact: _____
Sampled by: _____
Laboratory: _____

Sample Identification	Collection Date	Collection Time	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Remarks
1.								
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.								
11.								
12.								

Common Parameter/Container - Preservation Key
*1 - Volatile Organics = BTEX, GRO, TPH, Full List
*2 - Semivolatile Organics = PAHs, PCP, Dioxins, Full List, Herbicide/Pesticide/PCBs
*3 - General = pH, Chloride, Flouride, Alkalinity, TSS, TDS, TS, Sulfate
*4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other _____				Air Bill Number:		

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

Figure 3

CHAIN OF CUSTODY

Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

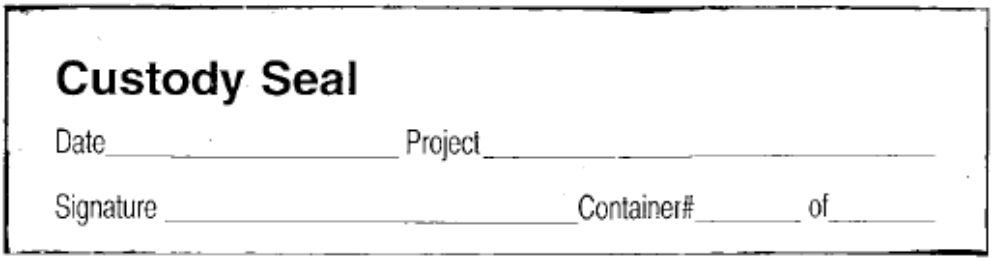
Preservative: _____

Sampled By: _____

Sample Location: _____

_____

Attachment 3
Custody Seal – if applicable



Attachment 4
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

Attachment 5
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

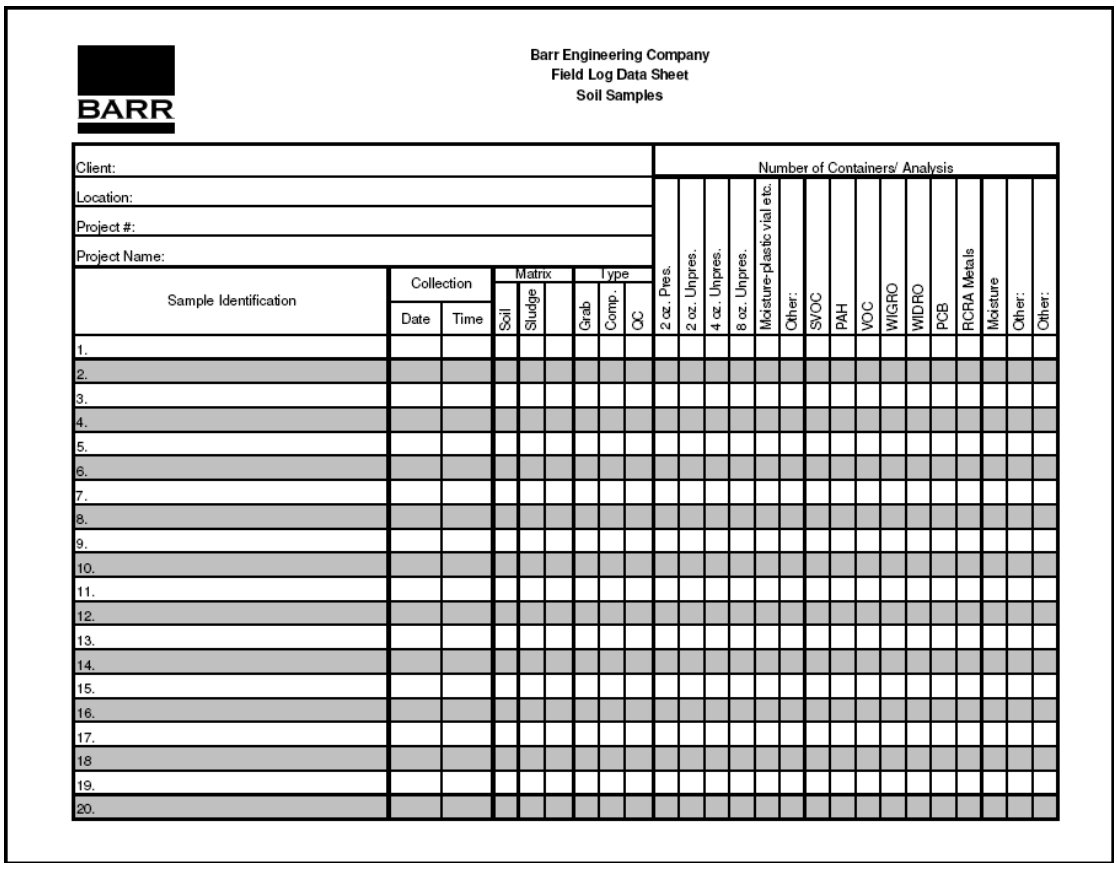
Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Attachment 6
Field Log Data Sheet



Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:
Location:
Project #:
Project Name:

Sample Identification	Collection		Matrix			Type			Number of Containers/ Analysis																
	Date	Time	Soil	Sludge		Grab	Comp.	QC	2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:
1.																									
2.																									
3.																									
4.																									
5.																									
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18																									
19.																									
20.																									

STANDARD OPERATING PROCEDURE

Collection of Quality Control Samples

Revision 2

March 3, 2009

Approved By:	Andrea Nord	Andrea Nord	03-03-09
	Print QA Manager(s)	Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for the Collection of Quality Control Samples

Purpose

To describe the procedures used in the collection of quality control samples; equipment blanks, field blanks, masked duplicate samples (i.e. field duplicate samples), matrix spikes and matrix spike duplicate and trip blank samples.

Applicability

This procedure applies to sample definition, collection and handling techniques used by the technician(s) and the laboratory in regards to quality control samples.

Equipment

Laboratory certified containers appropriate for the required analysis
Nitrile or vinyl gloves
Bailer
Chain of custody
Sample Labels
Sample containers/media
Analyte-free water

Definitions

Equipment Blank. The equipment blank sample is made up from analyte-free water that is rinsed on or through sample collection equipment. The rinse water is collected in the appropriate sample container(s) and submitted for analysis. The equipment blank samples are used to determine the following; the effectiveness of field cleaning procedures and to determine any source of contamination in a trip blank sample. The purpose of the equipment blank sample is to determine if the sampling devices and apparatus for sample collection have been adequately cleaned before they are shipped to the field site.

Field Blank. Field blank samples (or Rinsate Blanks) are prepared on-site. The field technician pours analyte-free water through decontaminated sample collection equipment (bailer or pump, hand-trowel, etc.) and collects the "rinsate" in the appropriate sample container(s). The field blank samples will be handled in the same manner as the sample group for which they are intended (i.e. blanks will be stored and transported with the sample group). The purpose of the field blank sample is to determine whether the field or sample transporting procedures and environments have contaminated the sample.

Field (or Masked) Duplicates. Field duplicate samples are: two identical aliquots of a sample, collected in separate sample bottles at the same time, and placed under identical circumstance using a duel inlet sampler or by splitting a larger aliquot. They are treated exactly the same throughout field and laboratory procedures. Analyses of field duplicate samples give a measure of the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

Matrix Spikes (MS) and Matrix Spike Duplicate (MSD). Matrix spike and matrix spike duplicates are two identical aliquots of an environmental sample to which known quantities of analytes are added (spiked) in the laboratory. The MS and MSD are prepared and analyzed exactly like their project (native) sample aliquot. Generally, it is required that three separate sample aliquots are collected in the field for each analysis. One aliquot is analyzed to determine the background concentrations in the project sample, a second sample aliquot serves as the MS sample and the third sample aliquot serves as the MSD. The purpose of the MS and MSD is to quantify the bias and precision caused by the sample matrix. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS and MSD corrected for background concentrations.

Trip Blank. A trip blank sample is made up of contaminant-free water and is prepared prior to sampling event by the laboratory providing the sampling containers. The purpose of the trip blank sample is to determine if contamination has occurred from any of the following sources; improper sampling container cleaning, contaminated source water, sample contamination during storage and transportation due to exposure to contaminants or any other environmental conditions during sampling. Trip blank samples apply to VOC samples only.

References

Procedures for Ground Water Monitoring, Minnesota Pollution Control Agency Guidelines, December 1986

EPA: Title 40 of the Code of Federal Regulations

Responsibilities

The sampling technician(s) are responsible for the accurate collection of quality control samples. The laboratory is responsible for the accurate set-up and analysis of quality control samples.

Procedure

The ratio of quality control samples are generally 1 field blank/field duplicate per twenty samples, however, specific project requirements may be determined by the QAPP/SAP for the project.

A. Masked duplicate sample:

1. Collect samples by rotating sampling containers from original sample to the field (masked) duplicate sample (using the same exact methods for both).

2. Preserve, store, and transport the field duplicate sample in the same manner as the original sample.

3. Submit the field duplicate sample to the laboratory for the same analyses as the original sample.

B. Trip blank Samples:

1. Trip blank samples are sealed prior to sampling (prepared by the laboratory performing the VOC analysis).

2. Transport trip blank samples to the site in the sample storage cooler containing the VOC vials used for collecting project samples for the sampling event.

3. Trip blank sample containers are not to be opened in the field.

4. Transport trip blank samples back to the laboratory in the sample storage cooler. There must be one set of trip blank samples per sample cooler containing VOC samples from the Site.

5. The trip blanks should be listed on the chain-of-custody along with the other samples and the analysis required. (Trip blanks are only provided for VOCs analyses).

   Note: Labeling of all sample blank containers follow the SOP for the collection of groundwater, soil, or sediment samples.

C. Field, Rinsate, or Equipment blanks:

1. Obtain the appropriate sampling containers and desired amount (analyte-free) water from the laboratory. (Generally, blanks are taken for each parameter of interest.)

2. Pour analyte-free water through decontaminated sample collection equipment (bailer or pump, hand-trowel, etc.) and collecting the "rinsate" in the appropriate sample containers.

3. Seal the field blank sample containers and store with other samples collected (should be handled in the same manner).

Filtered equipment blank:

1. Pour or pump (analyte-free) water into and/or through the groundwater sampling filter.

2. Begin filtering (as described in the standard operating procedure for Filtering Groundwater Samples).

Note: The filtered equipment blank is usually conducted for dissolved metals or dissolved organic carbon samples only.

Sample Storage

The samples will be bubble wrapped or bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain of custody documentation. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of strapping tape. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

Documentation

The technician(s) will document the type and number of quality control samples collected during each field event. All sample information will be documented in the field notebook, field log data sheet and chain-of-custody record.

Attachments

Attachment 1 – Field Log Data Sheet
Attachment 2 – Chain of Custody Form
Attachment 3 – Sample Label – Example
Attachment 4 – Custody Seal – Example

Attachment 1
Field Log Data Sheet – Soil Samples

Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:									Number of Containers/ Analysis																				
Location:																													
Project #:																													
Project Name:																													
Sample Identification	Collection		Matrix			Type			2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:					
	Date	Time	Soil	Sludge		Grab	Comp.	QC																					
1.																													
2.																													
3.																													
4.																													
5.																													
6.																													
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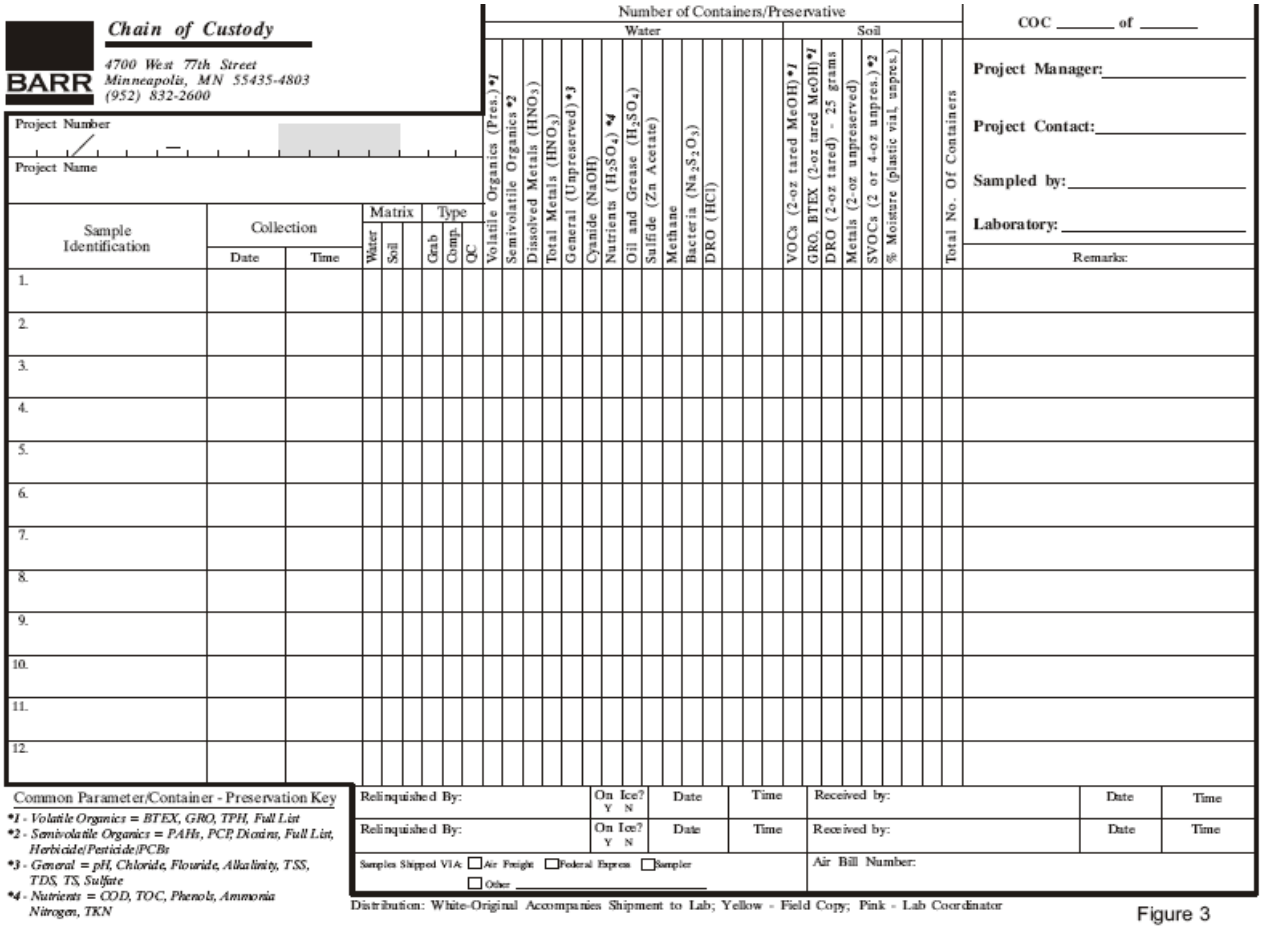
19.																													
20.																													

Attachment 2
Chain of Custody Form



Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number
Project Name

Number of Containers/Preservative

Sample Identification	Collection Date	Collection Time	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Water: Volatile Organics (Pres.) *1	Water: Semivolatile Organics *2	Water: Dissolved Metals (HNO_3)	Water: Total Metals (HNO_3)	Water: General (Unpreserved) *3	Water: Cyanide (NaOH)	Water: Nutrients (H_2SO_4) *4	Water: Oil and Grease (H_2SO_4)	Water: Sulfide (Zn Acetate)	Water: Methane	Water: Bacteria ($Na_2S_2O_3$)	Water: DRO (HCl)	Soil: VOCs (2-oz tared MeOH) *1	Soil: GRO, BTEX (2-oz tared MeOH) *1	Soil: DRO (2-oz tared) - 25 grams	Soil: Metals (2-oz unpreserved)	Soil: SVOCs (2 or 4-oz unpres.) *2	Soil: % Moisture (plastic vial, unpres.)	Total No. Of Containers	Remarks
1.																										
2.																										
3.																										
4.																										
5.																										
6.																										
7.																										
8.																										
9.																										
10.																										
11.																										
12.																										

COC _____ of _____

Project Manager: _____

Project Contact: _____

Sampled by: _____

Laboratory: _____

Common Parameter/Container - Preservation Key

*1 - Volatile Organics = BTEX, GRO, TPH, Full List
*2 - Semivolatile Organics = PAHs, PCP, Dioxins, Full List, Herbicide/Pesticide/PCBs
*3 - General = pH, Chloride, Flouride, Alkalinity, TSS, TDS, TS, Sulfate
*4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other _____ | Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

Figure 3

CHAIN OF CUSTODY

Attachment 3
Example - Sample label

Client _____

Project Number: _____

Date: _____ Time _____

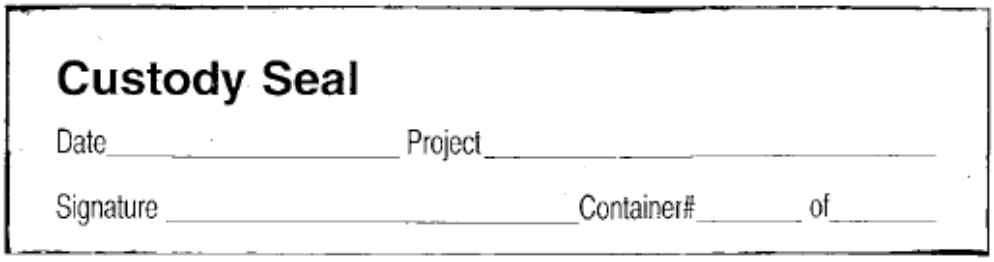
Preservative: _____

Sampled By: _____

Sample Location: _____

_____

Attachment 4
Custody Seal



STANDARD OPERATING PROCEDURE

Documentation of a Chain of Custody

Revision 2

March 3, 2009

Approved By:	Andrea Nord			03/03/09
	Print	QA Manager(s)	Signature	Date
	Chris J. Frerich			03/03/09
	Print	Field Technician(s)	Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for Documentation of a Chain-of-Custody

Purpose

The purpose of this procedure is to describe how to properly document information on a Chain-of-Custody (COC) form.

Applicability

These procedures apply to anyone, any time a COC is required.

Definitions

Chain-of-Custody A legally binding document that identifies sample identification, analyses required, and shows traceable possession of samples from the time they are obtained until they are introduced as evidence in legal proceedings.

Equipment

Chain of Custody form
Indelible ink pen

References

Groundwater sampling guidelines and groundwater and surface water sampling procedures by Barr Engineering Company.

Responsibilities

The environmental technician(s)/field technician(s) are responsible for accurate and complete documentation on the COC.

Procedure

The COC is the most important sampling document, it must be filled out accurately and completely every time.

Completing a Chain-of-Custody

1. The COC should be completed prior to leaving the sampling location.

2. Complete one COC or more as needed for each cooler of samples.

3. The COC must contain the following information:

    a. Project number
    b. Sample identification
    c. Date and time of sample collection
    d. Container type and number
    e. Sample matrix
    f. Whether the sample is a grab, composite, or blank sample
    g. Project manager
    h. Project contact
    i. Project name
    j. Project number
    k. Laboratory name
    l. Analyses required
    m. Signature of sampler(s)
    n. Signature of transferee
    o. Date and time of transfer
    p. Method of transport and any shipping numbers
    q. If sample preservation check conducted in the field indicates:
    1) additional preservation is required for inorganic samples. Note this on the COC or perform a pH adjustment and note the volume, concentration and preservative type on the COC. Or,
    2) that a VOC sample is not properly preserved, note this on the COC, request a 7 day TAT due to the analytical method holding time is 7 days from collection.

4. The COC should always accompany the cooler of samples associated with the COC.

    a. Distribution of the COC pages:

    Pages one and two go to the laboratory, page three goes to the lab coordinator, and the fourth page is the field copy.

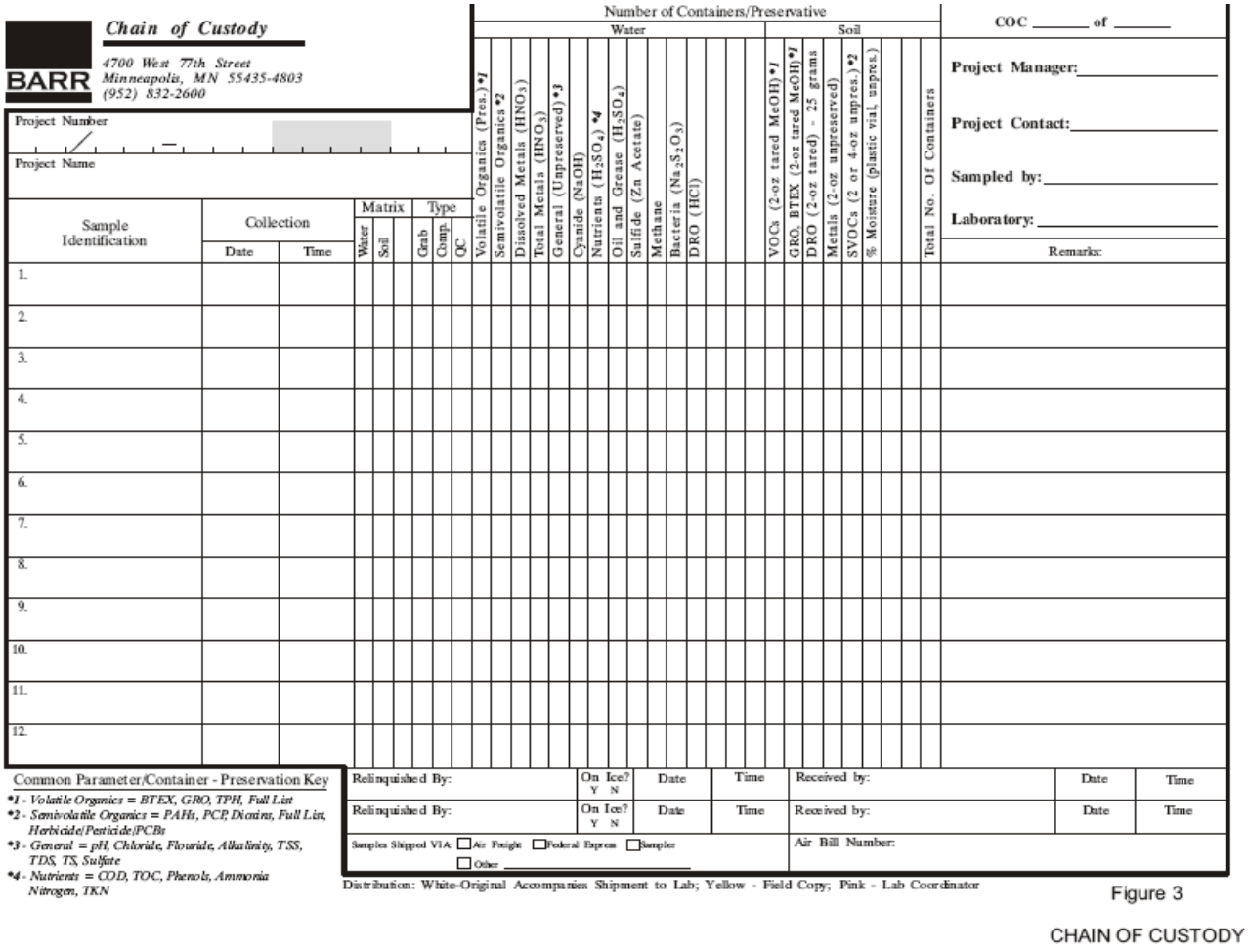
Documentation

The Chain-of-Custody form is the documented proof of possession of samples collected. This is documented by field personnel collecting the samples and the laboratory receiving the samples.

Attachments

Attachment 1: Chain of Custody Form

Attachment 1
Chain of Custody Form



Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number
Project Name

Number of Containers/Preservative

Sample Identification	Collection Date	Collection Time	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Water: Volatile Organics (Pres.) *1	Water: Semivolatile Organics *2	Water: Dissolved Metals (HNO_3)	Water: Total Metals (HNO_3)	Water: General (Unpreserved) *3	Water: Cyanide (NaOH)	Water: Nutrients (H_2SO_4) *4	Water: Oil and Grease (H_2SO_4)	Water: Sulfide (Zn Acetate)	Water: Methane	Water: Bacteria ($Na_2S_2O_3$)	Water: DRO (HCl)	Soil: VOCs (2-oz tared MeOH) *1	Soil: GRO, BTEX (2-oz tared MeOH) *1	Soil: DRO (2-oz tared) - 25 grams	Soil: Metals (2-oz unpreserved)	Soil: SVOCs (2 or 4-oz unpres.) *2	Soil: % Moisture (plastic vial, unpres.)	Total No. Of Containers	Remarks
1.																										
2.																										
3.																										
4.																										
5.																										
6.																										
7.																										
8.																										
9.																										
10.																										
11.																										
12.																										

COC _____ of _____

Project Manager: _____

Project Contact: _____

Sampled by: _____

Laboratory: _____

Common Parameter/Container - Preservation Key

*1 - Volatile Organics = BTEX, GRO, TPH, Full List
*2 - Semivolatile Organics = PAHs, PCP, Dicainz, Full List, Herbicide/Pesticide/PCBs
*3 - General = pH, Chloride, Flouride, Alkalinity, TSS, TDS, TS, Sulfate
*4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other _____				Air Bill Number:		

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

Figure 3

CHAIN OF CUSTODY

STANDARD OPERATING PROCEDURE

Calculation of Purge Volumes for Groundwater Sampling Wells

Revision 2

February 27, 2009

Approved By:

Print	QA Manager(s)	Signature	Date
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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

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Standard Operating Procedures for the Calculation of Purge Volumes for Groundwater Sampling Wells

Purpose

The purpose of this procedure is to describe the methods used in calculating and measuring purge volumes.

Applicability

The procedure applies to the amount of water that is purged out of a well before sampling can occur.

Definition

Purge volume is a specific amount of water removed from a well before sampling.

Equipment

Calculator
Field Logbook

Reference

Groundwater and Surface Water Sampling Procedures by Barr Engineering Co.

Discussion

The procedure will show that a variety of calculations must be carried out before purge volumes are known.

Responsibilities

The sampling technician(s) conducting the purging of the well are responsible for the purge volumes.

Procedure

Calculating and Measuring Purge Volumes

1. Calculate the volume of standing water in the well (using the following equation):

   Note: There is a precalculated chart to determine the volume of standing water (Figure 1).

a. $V = (\pi)(r^2)(h)$

V = Volume in cubic feet of standing water
π = 3.14
r = Radius of the well casing or hole (in feet)
h = Height of the column of water in the well (in feet)

(h = water level - total well depth)

2. Convert the volume of standing water in the well from cubic feet to gallons using the following equation:

a. WV = (V)(7.48)

WV = Well volume in gallons

3. Determine the amount of water to be purged (using this equation):

a. VP = (WV)(NWV)

VP = Volume of water pumped
WV = Well volume in gallons
NWV = Number of well volumes that monitoring plan requires to be purged

4. Estimate the time it will take for the well to be purged (time pumped).

a. Determine the flow rate of the well.

Flow meter— If installed on well, it can be simply read to obtain the flow rate.

No-flow meter— The rate can be obtained by using a container marked in volumes and calculating the amount of time it takes for the container to fill with purge water.

Note: See Standard Operating Procedures for Measuring Well Pumping Rates.

b. Divide the volume of water pumped in the well by the flow rate.

Stabilization Test Measurements

Collection of stabilization test measurements shall begin at the same time as groundwater purging prior to sample collection is initiated. Well stabilization measurements will be collected and recorded at the start of the purging process and once every well volume during the purging process, with a minimum of one measurement collected per well volume removed. A well volume will be measured as the volume of water that occurs in each well from the base of the well to the water level measurement collected prior to initiation of purging. Once three well volumes have been removed, the well may be sampled after three consecutive measurements, collected at the intervals described above, are within the ranges presented below:

Specific Conductance:	±5% of the most recent reading (temperature corrected)
pH	±5% of the most recent reading (in pH units)
Temperature	±5% of the most recent reading (in degrees Celsius)
Oxidation Reduction Potential (Eh)	±20 mV of the most recent reading

Collect samples only after a minimum of three water-column volumes have been purged and stabilization of field water-quality parameters has been demonstrated by meeting the target criteria defined in the preceding paragraph. Field staff shall check operator procedures, equipment functioning, and well construction information for potential problems. In particular, field staff shall re-evaluate whether or not water is being withdrawn from the appropriate depth to effectively evacuate the well.

If all the checks produce no new insight, a decision might be made to collect samples after five or more water-column volumes have been purged even if field measurements have not stabilized. If the well was purged dry, it shall be allowed to recharge and the samples will be collected.

However, if either circumstance applies, the following procedure is required: Before authorizing the laboratory to analyze the samples, the meaningfulness and value of completing laboratory analysis of the sampling suite will be evaluated by reviewing the results of field measurements, well construction data, site hydrology, etc. Where such data is presented, it will be clearly documented that stabilization was not achieved; at a minimum, this fact will be reported on the Field Log Data Sheets and in the Field Sampling Report.

Documentation

The technicians will document flow rate, well volume, time pumped, volume pumped, water level, total well depth and stabilization test measurements on the field log data sheet.

Figures

Figure 1 – Volume of Water in Casing or Hole

Attachments

Attachment 1 – Field Log Data Sheet

Figure 1

Volume of Water in Casing or Hole

Diameter of Casing or Hole (In)	Gallons per Foot of Depth	Cubic Feet per Foot of Depth	Liters per Meter of Depth	Cubic Meters per Meter of Depth
1	0.041	0.0055	0.509	0.509×10^{-3}
1½	0.092	0.0123	1.142	1.142×10^{-3}
2	0.163	0.0218	2.024	2.024×10^{-3}
2½	0.255	0.0341	3.167	3.167×10^{-3}
3	0.367	0.0491	4.558	4.558×10^{-3}
3½	0.500	0.0668	6.209	6.209×10^{-3}
4	0.653	0.0873	8.110	8.110×10^{-3}
4½	0.826	0.1104	10.26	10.26×10^{-3}
5	1.020	0.1364	12.67	12.67×10^{-3}
5½	1.234	0.1650	15.33	15.33×10^{-3}
6	1.469	0.1963	18.24	18.24×10^{-3}
7	2.000	0.2673	24.84	24.84×10^{-3}
8	2.611	0.3491	32.43	32.43×10^{-3}
9	3.305	0.4418	41.04	42.04×10^{-3}
10	4.080	0.5454	50.67	50.67×10^{-3}
11	4.937	0.6600	61.31	61.31×10^{-3}
12	5.875	0.7854	72.96	72.96×10^{-3}
14	8.000	1.069	99.35	99.35×10^{-3}
16	10.44	1.396	129.65	129.65×10^{-3}
18	13.22	1.767	164.18	164.18×10^{-3}
20	16.32	2.182	202.68	202.68×10^{-3}
22	19.75	2.640	245.28	245.28×10^{-3}
24	23.50	3.142	291.85	291.85×10^{-3}
26	27.58	3.687	342.52	342.52×10^{-3}
28	32.00	4.276	397.41	397.41×10^{-3}
30	36.72	4.909	456.02	456.02×10^{-3}
32	41.78	5.585	518.87	518.87×10^{-3}
34	47.16	6.305	585.68	585.68×10^{-3}
36	52.88	7.069	656.72	656.72×10^{-3}

1 gallon = 3.785 liters
1 meter = 3.281 feet
1 gallon water weighs 8.33 lbs. = 3.785 kilograms
1 liter water weighs 1 kilogram = 2.205 lbs.
1 gallon per foot of depth = 12.419 liters per foot of depth
1 gallon per meter of depth = 12.419×10^{-3} cubic meters per meter of depth

Attachment 1
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample Time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:								
Casing diameter:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						
Others present:								
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form) CASING & CAP: COLLAR: LOCK: OTHER:								
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:								
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-								
oil,grease- bacteria- total metal- filtered metal- methane- filter-								
Others:								

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

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STANDARD OPERATING PROCEDURE

Purging Groundwater Wells

PDCOCS No.: 213629

Revision 3

April 27, 2009

Approved By:

Andrea Nord		04/27/09
Print	QA Manager(s) Signature	Date
Kim Johannessen		04/27/09
Print	Field Technician(s) Signature	Date

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Standard Operating Procedures
Purging Groundwater Wells

Purpose

The purpose of this SOP is to describe the procedures for purging a well using a variety of techniques described in this SOP.

Applicability

This SOP applies to environmental technicians who are responsible for purging wells using one of the following techniques described in this SOP.

Definitions

Well purging is the removal of a known volume of water from a well so sampling can occur. This removal can be achieved by using two techniques: (1) without in-place plumbing; or (2) with in-place plumbing.

Drawdown: the lowering of the static water level due to the removal of the groundwater.
Note: See SOP for definition of static water level.

Equipment

Trisodium phosphate (TSP) solution
Tap water
Brush
Deionized water
Container marked in volume increments
Sterile gloves
Bailer (Stainless steel or new disposable polyethylene)
Peristaltic pump
Submersible pump
Nylon line
Discharge hose

References

Groundwater Sampling Guidelines by MPCA

Responsibilities

The environmental technician(s) is responsible for the proper well purging procedures; and documentation.

Discussion

Purging of a groundwater well is an important factor in the sampling process. It prepares the well by removing required volumes of water (according to the sampling plan) prior to sampling. The purging is needed to stabilize the well to allow for representative sample collection.

One method of purging is to pump the well until three to five times the volume of standing water in the well is removed. A second method is to pump the well until the groundwater's specific conductance, temperature, pH, dissolved oxygen and oxidation reduction potential (ORP) stabilize. Normally, a combination of the two methods is used; i.e., specific conductance, temperature, pH, ORP and dissolved oxygen are measured at intervals and the volume purged is monitored. If a well is pumped dry, this constitutes an adequate purge and the well can be sampled following recovery. All well purging equipment will be cleaned between wells with tap water and Trisodium phosphate (TSP) solution and rinsed with tap water as described in the SOP for Tool Decontamination – Level I.

Purging can be done using a bailer, a peristaltic or a submersible pump.

Procedure

Well Purging

A. Technique for purging a well without in-place pumping:

1. Bailer

a. Put on gloves for skin protection and to prevent sample contamination.

b. Remove foil from bailer top (stainless steel), bailer body (stainless steel), and check valve (Teflon). A disposable polyethylene bailer can be used in place of the stainless steel bailer.

c. Connect all three parts together. It may be possible to connect additional bailer body pieces together, increasing the volume removed with each lowering of the bailer.

d. Using a cord reel or similar device, secure the bailer to the cord reel rope.

e. Empty the water collected from the bailer into a measuring bucket.

f. Continue the process, until the correct volume of water has been purged and the well has stabilized.
Note: See SOP for well stabilization testing.

g. Cut the rope from the bailer after purging is finished.

h. Place used top, bailer, and check valve in a dirty bailer cooler to be cleaned.
Note: If a disposable bailer was used, it cannot be reused and must be disposed of.

2. Bailer (H) – bailer hose

A bailer is used for slow-recovering wells with an inside diameter less than 2 inches and a depth to groundwater greater than 25 feet. A laboratory-cleaned stainless steel bailer with a Teflon check valve or new disposable polyethylene bailer with a check valve is attached to a downrigger and support assembly. Teflon-coated wire and stainless steel wire are both acceptable for hauling stainless steel bailers. Polyethylene bailers can be hauled using stainless steel wire or new nylon line.

1. Put on gloves to protect skin.

2. Remove foil from bailer (stainless steel) and check valve (Teflon).

3. Connect these two parts together and connect them to a 40-foot suction hose.
   Note: Bailer (H) can only be used on wells with total well depths of approximately 40 feet or less.

4. Lower the hose and bailer into the well until the bailer is partially submerged below the static water level.
   Note: If well goes dry, the bailer needs to be on the bottom of the well (due to drawdown).

5. Begin to surge the hose up and down; the result will be water pumping out of the well from the suction hose.

6. Collect purged water in a measuring bucket.

7. Continue to purge until the desired amount is purged or the well goes dry (see monitoring plan for volumes required to be purged).

8. Remove hose from well, put bailer and check valve in dirty bailer cooler, rinse hose with distilled water.

3. Centrifugal Pump

a. Put on gloves to protect skin.

b. Remove foil from bailer (stainless steel) and check valve (Teflon); connect together.

c. Connect bailer assembly to a 40-foot suction hose.

Note: Centrifugal pumps will not pump at depths greater than 30 feet without surging (bailer [C]).

d. Submerge the bailer assembly with attached hose about 2 feet into the static groundwater.

e. Screw the other end of the hose onto the intake of centrifugal pump (make sure the connection is tight to ensure suction).

f. Prime the pump by pouring water into the priming water filler cap.

g. Start centrifugal pump:
Step 1: turn pump on by the switch on the side of the pump
Step 2: pull recoil rope to start pump

h. Surge hose to get the water up.

i. Continue priming until the water pumps by itself.

j. Adjust flow (with check valve located on discharge of the pump) to desired flow rate.

k. Check flow rate with the measurement bucket (gpm).

Note: If flow rates are under 1 gpm, the centrifugal pump should not be used.

l. Continue pumping until desired purge volumes are achieved.

m. Remove bailer and hose from well, turn off pump and disconnect the hose from the intake.

n. Disconnect bailer from the hose, put the bailer and check valve into the dirty bailer cooler; rinse the hose with distilled water.

o. Discharge purged water from pump by unscrewing the drain plug (bottom of the pumps); rinse pump.

4. Peristaltic Pump

This pump is used when the water level is within suction lift, i.e., within about 22 feet of the ground surface. It usually is a low-volume suction pump with low pumping rates suitable for sampling shallow, small-diameter wells.

a. Cut tubing to desired length.

b. Connect tubing to pump head, leaving 1 to 2 feet for discharge line.

c. Lower tubing into the well water (1 to 2 feet below surface).

d. Turn on pump and set speed at the desired rate of flow.

5. 4-inch Submersible Pump

This pump may be used to purge water samples from any depth. Variable rate submersible pumps are available to fit inside 2-inch or larger wells.

a. Put on gloves to protect skin.

b. Attach purging hose to the pipe connected on the top submersible pump.
Note: Either a 40- or 60-foot hose can be used, or both, whichever is appropriate.

c. Lower the submersible pump slowly into the well.

d. Lower pump until it is completely submersed into the water hang in casing.
Note: It can usually be lowered 5 to 6 feet under the water, unless draw-down in the well occurs.

e. Connect the pump to the generator with an extension cord.

f. Start the generator:
Step 1: turn switch to start
Step 2: put choke on
Step 3: pull recoil rope
Step 4: let generator idle until it is running smooth

g. Turn on power (which is located on the front of the generator).
Note: Submersible should be running; if not, turn off the generator and check connections.

h. Adjust flow rate to desired rate with the valve.

i. Measure the flow rate with the measuring bucket (gpm).

j. Turn off the generator after desired purge volume has been achieved.

k. Pull up the pump. The technician must be especially careful not to let the hose and wire get under or on the side of the pump.

l. Disconnect and disassemble all of the submersible pump apparatus; rinse accordingly.

6. 1.5-inch Submersible Pump

This is a type of submersible pump that can be used in 2-inch or larger diameter wells. It can purge water from depths down to 200 feet depending on pump model and manufacturer. This pump may be used as a submersible pump alternate for lower-volume wells.

a. Attach ⅜-inch tubing to pump intake and lower to desired depth.

b. Cut off tubing, allowing additional tubing length for discharge.

c. Plug the pump into the controller. Pump will begin pumping using the variable speed controller. There are a variety of speed controllers available, typically designed for a specific pump.

d. Attach the controller battery clips to the 12v DC power supply.

e. Turn on the controller and dial the speed control to the desired flow rate. This is especially useful if the well has low recharge rates. The controller can slow the purge rate down to the optimum rate.

7. 6-Inch Submersible Pump

a. Put on gloves to protect skin.

b. Attach hose reel onto well casing.

c. Loosen retainer pins from pump holder and place in well.

d. Loosen retainer pins from hose reel and lower pump with reel handle to desired depth (about 2 feet below static water level).

e. Connecting hoses and power cords:
Step 1: connect discharge hose to hose reel
Step 2: connect (110, 220 volt) controller power patch cord to hose reel
Step 3: connect controller power cord to appropriate 110, 220 receptacle on generator

f. Start the generator:
Step 1: turn switch to start
Step 2: put choke on
Step 3: pull recoil rope
Step 4: let generator idle until it is running smoothly

g. Turn on AC switch if applicable

h. Turn controller switch on (make sure LCD display reads zero before setting flow rate), adjust the flow rate with the speed control knob to desired rate.

i. Measure the flow rate with the measuring bucket (gpm).
Note: Submersible pump should be running; if not, turn off the generator and check connections.

j. Shut down system after desired purge volume has been achieved:
Step 1: turn controller switch off and turn speed control to zero
Step 2: turn off AC switch
Step 3: turn off generator
Step 4: disconnect controller power patch cord from generator
Step 5: disconnect controller power patch cord from hose reel
Step 6: disconnect discharge hose from hose reel

k. Unlock retainer pin and reel up the hose and submersible pump and lock into pump holder.

l. Rinse the hose and pump with distilled water.

B. With In-place Plumbing

1. **Dedicated pumps** are submersible pumps that are permanently installed in a well.

    a. Put on gloves to protect skin.

    b. Start the generator:
    Step 1: turn switch to "on"
    Step 2: turn on choke
    Step 3: pull recoil rope
    Step 4: let generator idle until it is running smooth

    c. Connect the pump to the generator with an extension cord.

    d. Connect the pipe, elbow, and valve to the discharge pipe of the submersible pump (located at the top of the well).

    e. Turn on power from the generator to the pump.
    Note: If the pump does not start, check the connection from the generator to the pump.

    f. When water flows from discharge of the pump, adjust the flow according to desired flow rate (using the discharge check valve).

    g. Use measuring bucket to determine the appropriate flow rate (gpm).

    h. After the appropriate purge volume is achieved. Sample collection can occur (before shutting off the generator and pump).

    i. Turn off the generator.

    j. Disconnect all of the appropriate connections and take the pipe, elbow, and valve off.

    Note: Each dedicated pump has its own pipe, elbow, and valve. These pieces are left at each well.

Discussion

In general, peristaltic pumps are used for wells with water levels less than 22 feet in depth. Submersible pump may be used for wells with lower water levels. Bailers are used for wells with water levels below 25 feet and diameters less than 2 inches.

When peristaltic pumps are used, only the intake line is placed into the well. When submersible pump are used, the pump and discharge hose are lowered into the water column.

The pump/hose assembly used in purging should be lowered into the top of the standing water column and not deep into the water. This is done so that the purging will "pull" water from the formation into the screened area of the well and up through the casing so that the entire static volume can be removed. If the pump/hose is placed deep into the water column,

the water above the pump may not be removed, and the subsequent samples collected by bailer may not be representative of the groundwater. The exception to placing the intake at the top of the water level is during low-flow purging and sampling. For low-flow purging and sampling the intake should be placed at or just above midscreen to capture water within the formation (see Barr's Low-flow Purging and Sampling SOP's).

If well recovery (groundwater reentering the well from the surrounding formation) is at least as rapid as the pumping rate, the pump/hose may be left hanging at the initial level until an adequate volume of water is removed. If the pumping rate exceeds the well's recovery rate, the pumping rate will be adjusted.

A laboratory-cleaned bailer with a Teflon check valve or new disposable polyethylene bailer with a check valve is attached to a support base and downrigger by stainless steel or Teflon-coated wire. The bailer assembly is lowered into the top of the water column. When the bailer has filled, it is removed from the well and the water is poured into a bucket marked in quarts/liters for volume measurement.

Purge Rate. The purge rate for a given well depends on several factors including the well volume and the depth to water. The well volume and depth to water will determine the type of pump used in purging the well. Different types of pumps give different types of flow rates. The flow rate will be determined in the field according to individual well performance. The purge rate with submersible pumps will be up to approximately 25 gpm. The purge rate with a peristaltic pump will be up to approximately 1 gpm. The purge rate should be held constant during stabilization testing.

Measuring Well Pumping Rate. If a flow meter is installed on the well, simply read the meter. If no meter is available, the pumping rate can be determined by using a container marked in volume increments such as quarts or liters and a stopwatch to time how long it takes for the container to fill with purge water. Be aware that changes in the flow rate will affect the amount of time required to purge the necessary amount of water from the well.

Measuring Purge Volume. The volume of standing water in the well is calculated first to determine the amount of purge water that needs to be removed from the well. The water level must be measured in order to determine the volume of standing water. The volume of standing water in the well is calculated using the following equation:

$$V = (\pi)(r^2)(h)$$

where: V = volume, in cubic feet
π = 3.14
r = radius of the well casing or hole (in feet)
h = height of the column of water in the well (in feet)

Then convert the volume of water standing in the well from cubic feet to gallons by multiplying the volume by 7.48.

Then determine the amount of water that must be purged by multiplying the gallons of standing water in the well by the number of well volumes that are required to be purged.

Documentation

The environmental technician(s) will document the procedures used in purging wells on the Field Log Cover Sheet, Field Log Data Sheet and or Field Log book.

Attachments

Attachment 1: Field Log Data Sheet

Attachment 1
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:	**Monitoring Point:**
Location:	**Date:**
Project #:	**Sample Time:**

GENERAL DATA		STABILIZATION TEST						
Barr lock:								
Casing diameter:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						

Others present:
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)
CASING & CAP: COLLAR: LOCK: OTHER:
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-
oil,grease- bacteria- total metal- filtered metal- methane- filter-
Others:

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

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STANDARD OPERATING PROCEDURE

For Well Stabilization and Well Stabilization Testing

Revision 1

March 5, 2009

Approved By:

Andrea Nord	Andrea Nord	03/05/09
Print	QA Manager(s) Signature	Date

Steve Ivers	Steve Ivers	03/05/09
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Initials: _____	Date: _____
Initials: _____	Date: _____
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Initials: _____	Date: _____
Initials: _____	Date: _____

Standard Operating Procedures for
Well Stabilization and Well Stabilization Testing

Purpose

The purpose of procedure is to describe the methods used for well stabilization and stabilization testing of a well.

Applicability

Well stabilization is important to ensure that the water sampled will be representative of aquifer conditions.

Definitions

Stabilized When the required amount of water has been purged and the specific conductance, temperature, pH and potentially ORP of the groundwater are within acceptable limits for three consecutive readings.

ORP Reduction/oxidation potential. ORP is the potentiometric measurement in which the potential (or tendency) of the medium for electron transfer is sensed by an inert metal electrode and read relative to a reference electrode that is immersed in the same medium.

References

Quality Assurance Manual: Ground Water and Surface Water Sampling Procedures; Barr Engineering Co. Procedures for Groundwater Monitoring: MPCA Guidelines.

YSI Environmental. YSI Model 556 MPS Water Quality Monitoring System Operations Manual

YSI Environmental. *Measuring ORP on YSI 6-series Sondes: Tips, Cautions, and Limitations Tech Note*. YSI 2001/2005.

Responsibilities

The environmental technician(s) will be responsible for testing and recording stabilization test information.

Procedures

A. Well Stabilization:

1) Field Water Quality Measurements

Specific conductance, pH, temperature, dissolved oxygen and potentially ORP (oxidation reduction potential) will be measured in the field immediately before sample collection. These measurements, as well as measurement conditions and the steady-state value for each

field water-quality parameter, will be recorded on the Field Log Data Sheet. Instrument calibration information will be recorded as part of the field sampling report.

All measurements will be taken within a closed flow-through cell designed to allow measurement of these parameters while minimizing changes in temperature, pressure, and dissolved gases from the in-situ aquifer environment. The flow-through cell has:

- Airtight fittings for installation of all probes.
- An intake that is connected directly to the pump discharge line.
- A discharge line that is connected to the flow-through cell with an airtight connection.

The following rules should be followed when using the flow-through cell:

- The flow-through cell will be shielded from strong winds and, on hot days, it will be shielded from direct sunlight.
- The flow of groundwater through the cell will be maintained as continuous and steady as practical throughout the measurement period.
- Discharge velocities through the cell should be kept below 1.5 gallons per minute.

The operation of the probes will be as follows:

- All probes will be fully immersed without touching the sides of the airtight, non-metallic flow-through cell.
- All probes will be allowed to equilibrate with well water before recording measurements.

Specific procedural details for measurement of individual field water-quality parameters are specified below. General care, maintenance, calibration procedures, and operation of each measurement device will follow manufacturer's specifications as detailed in the instruction/owner's manual for each device. Specific procedures for measurement of individual field water-quality parameters are described below.

Specific Conductance, Temperature, pH, and ORP (reduction/oxidation potential)

These measurements will be taken using the YSI Model 556 MPS Water Quality Monitoring System or equivalent. This device will be operated (including calibration) following the manufacturer's instructions.

Dissolved Oxygen

Dissolved oxygen measurements will be taken using the YSI Model 556 MPS dissolved oxygen meter or equivalent. Personnel using dissolved oxygen measuring equipment will have read the manufacturer's instruction manual once carefully before making dissolved oxygen measurements. Special care will be taken to store the probe in a humid environment and to otherwise protect the delicate membrane on the end of the probe. The membrane will be replaced every two to four weeks.

The dissolved oxygen meter will be calibrated according to manufacturer's specifications before taking measurements. When dissolved oxygen readings less than or equal to approximately 1.0 mg/L are expected, the meter will be calibrated in a mode that enhances accuracy at low concentrations. The calibration details will be recorded in the field log.

Measurements will be taken as follows:

a. The membrane at the tip of the probe will be checked visually to verify that it is in good condition.

b. After allowing the dissolved oxygen probe to equilibrate with a continuously replenished supply of aquifer water, the first measurement will be recorded.

To be considered valid, readings should appear stable on the display. If unstable readings are recorded, they will be footnoted and the unstable measurement conditions will be clearly stated in the final field sampling report. Readings will be reported to the nearest 0.01 mg/L dissolved oxygen.

2. Criteria for Stabilization

Field water-quality parameters will be measured for stabilization after each water-column volume is purged. One water-column volume is defined as the volume of a cylinder with a height (h) equal to that of the static water-column inside the well and a diameter (d) equal to the diameter of the well casing.

Volume = $\pi (d/2)^2 h$

Three consecutive measurements which meet the criteria listed below will be used to demonstrate stabilization:

- Temperature ±5% of the most recent reading (in degrees Celsius)
- Specific conductance (temperature corrected EC) ±5% of the most recent reading
- Dissolved oxygen ±5% of the most recent reading (in mg/L)
- pH ±5% of the most recent reading (in pH units)
- ORP Reading must be within ±0.01 units depending on the accuracy of the meter used.

Samples for laboratory analysis will be collected only after purging a maximum of five water-column volumes and achieving stabilization of field water-quality parameters. If field parameters do not stabilize after five water-column volumes, then field staff will verify that the probes and related equipment are functioning properly and that operator error is not an issue. Samples will be collected after five (or more) water-column volumes have been purged, even if field measurements have not stabilized. In such a case, the field log sampling and analysis report will clearly state that stabilization was not achieved.

B. Well Stabilization Testing:

Stabilization test samples are collected either from the flowing well discharge water or a bailer (depending on the purging method used). The sample is collected in a plastic bottle that has been rinsed three times with the sample.

Probes from both meters are placed in the collection bottle; readings are allowed to stabilize. Record the readings.

Note: Operation of the meters is explained in the SOPs for pH and conductivity meters.

The acceptable limits for specific conductance, temperature, pH and ORP are as shown:

- Specific Conductance—Reading from 0 to 500 must be within ±5 µmhos/cm @ 25°C. Reading from 500 to 5,000 must be within ±50 µmhos/cm @ 25°C.
- Temperature—Readings must be within ±0.5°C.
- pH—Reading must be within ±0.1 units.
- ORP—Reading must be within ±0.01 units depending on the accuracy of the meter used.

Documentation

The technician(s) shall document readings on the Field Log Data Sheet in the stabilization columns.

Attachments

Attachment 1: Field Sampling Report
Attachment 2: Field Log Cover Sheet
Attachment 3: Field Log Data Sheet

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

<Name inserts here>
Environmental Technician

Attachment 2
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Attachment 3
Field Log Data Sheet

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:		Time/ Volume	Temp. ºC	Cond. @ 25	PH	ORP mV	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*		NA						
Static well level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume purged:								
Duplicate collected:								
Sample collection by:								
Others present:		Well condition:						
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment Other: sump								
VOC Semi-volatile General Nutrient Cyanide DRO Sulfide								
Oil, grease Bacteria Total Metal Filtered Metal Methane Filter								
Others:								

STANDARD OPERATING PROCEDURE

Collection of Each Type of Groundwater Sample from Monitoring Wells, Residential Wells and Residential Systems

Revision 2

February 27, 2009

Approved By:

Print	QA Manager(s)	Signature	Date
Andrea Nord	Andrea Nord		2/27/09

Print	Field Technician(s)	Signature	Date
Kim Johannessen	Kim Johannessen		2/27/09

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for the Collection of Each Type of Groundwater Sample from Monitoring Wells, Residential Wells and Residential Systems

Purpose

The purpose of this procedure is to describe the collection of water samples for volatiles, semivolatiles, metals, inorganics, bacteria, and dioxin from monitoring wells, residential wells and residential systems.

Applicability

This procedure applies to the stabilization of monitoring wells and subsequent collection of groundwater samples by the sampling technician(s). It identifies each container type (volume, construction, preservative) required for each category of analyses, their corresponding holding times and collection procedures from monitoring wells, residential wells and residential systems.

Definitions

Headspace. The air space between the container top and the water sample level.

Holding Time. Period of time between sample collection and when the sample is analyzed.

Sample Preservation. The stability of analytes depends upon the proper preservation technique and preservation acceptance criteria as defined by EPA Title 40 of the Code of Federal Regulations and corresponding method criteria.

Equipment

Sampler media
Pre-cleaned-certified Sampling Containers
Coolers
Ziploc® Baggy
Ice
Water-proof ink pen or pencil
Bailer (Stainless Steel or Polyetheylene)
Gloves
Water Quality Meter
Sample label
Chain of Custody Form
Alconox

References

Quality Assurance Manual: Groundwater and Surface Water Sampling Procedures, Barr Engineering Co.; American Water Works Association: Pocket Guide to Water Sampling; Environmental Sampling, A Summary, the Radian Corporation.
Ground Water Sampling Guidelines by MPCA
EPA: Title 40 of the Code of Federal Regulations

Responsibilities

The Field Operations/QA Officer or the environmental technician(s) will order the sample containers prior to the sampling event. The environmental technician(s) is responsible for the proper collection of monitoring wells, residential wells, and residential system groundwater samples; sample identification; quality control procedures; sample filtering and documentation.

Procedure

I. Obtain Sampling Media

Approximately one week before the sampling event, the sample containers should be ordered from the laboratory.

Note: Container volume, type, and preservative are important considerations in sample collection. Container volume must be adequate to meet laboratory requirements for quality control, split samples, or repeat examinations. The container type or construction varies with the analysis required. The analytical laboratory will preserve the container before shipment. Preservation and shelf life vary; contact the laboratory to determine if an on-hand container is still useful.

II. Measure Water Level, Well Depth and Purge

Once the water level and well depth measurements have been taken and the well has been purged in accordance to Barr's Calculation of Purge Volumes for Groundwater Sampling Wells SOP and allowed to stabilize, the technician can begin groundwater sampling.

Stabilization Test Measurements

Collection of stabilization test measurements shall begin at the same time as groundwater purging prior to sample collection is initiated. Well stabilization measurements will be collected and recorded at the start of the purging process and once every ten minutes during the purging process, with a minimum of one measurement collected per well volume removed. A well volume will be measured as the volume of water that occurs in each well from the base of the well to the water level measurement collected prior to initiation of purging. Once three well volumes have been removed, the well may be sampled after three consecutive measurements, collected at the intervals described above, are within the ranges presented below:

Specific Conductance:	$\pm$5% of the most recent reading (temperature corrected)
pH	$\pm$5% of the most recent reading (in pH units)
Temperature	$\pm$5% of the most recent reading (in degrees Celsius)
Oxidation Reduction Potential (Eh)	$\pm$20 mV of the most recent reading

Collect samples only after a minimum of three water-column volumes have been purged and stabilization of field water-quality parameters has been demonstrated by meeting the target criteria defined in the preceding paragraph. Field technician will check operator procedures, equipment functioning, and well construction information for potential problems. In particular, field staff will re-evaluate whether or not water is being withdrawn from the appropriate depth to effectively evacuate the well.

If all the checks produce no new insight, a decision might be made to collect samples after five or more water-column volumes have been purged even if field measurements have not stabilized. If the well was purged dry, it shall be allowed to recharge and the samples will be collected.

However, if either circumstance applies, the following procedure is required: Before authorizing the laboratory to analyze the samples, the meaningfulness and value of completing laboratory analysis of the sampling suite will be evaluated by reviewing the results of field measurements, well construction data, site hydrology, etc. Where such data is presented, it will be clearly documented that stabilization was not achieved; at a minimum, this fact will be reported on the field data sheets and in the Field Sampling Report.

III. Groundwater Sampling

1. Monitoring Wells (Permanent or Temporary)

1.a Monitoring wells may either be installed permanently or temporarily. They are constructed for the collection of groundwater samples. These monitoring wells have a wide variety of diameters. Groundwater samples might also be collected out of a pit or a drilled hole.

1. Put on sampling gloves to protect the sample and skin.

   Note: New sampling gloves are needed for each well. Never reuse old gloves.

2. Prepare sampling containers by filling out the label with the following information:

   – Project number
   – Location sampled
   – Individual collecting the samples
   – Date and time of sample collection
   – Sample analysis (if required by the lab)

   Note: Use an inedible permanent pen to avoid ink bleeding.

3. Sampling

   a. Sampling Technique Using a Polyethelene Bailer (1) or Stainless Steel Bailer (2):

      1. Polyetheylene bailer and Cord reel and rope— Tie the rope to the bailer and lower the bailer into the well with the cord reel.

      2. Remove foil from the bailer top (stainless steel).

3. Connect the rope to the bailer top.

4. Remove foil from the bailer body (stainless steel) and the check valve (Teflon).

5. Connect these two parts together, screw these pieces into the bailer top.

6. Slowly rotate the cord reel to lower the bailer into the top of the water column.

   Note: Make sure not to stir up the water with the bailer, thus volatizing the samples.

7. Keep the bailer in the top portion of the water column when collecting the sample.

8. When the bailer is filled, slowly rotate the cord reel to retrieve the bailer out of the well.

9. Collect samples by utilizing steps outlined in this SOP.

10. After all of the samples are collected, place the samples in a sampling cooler with ice.

11. Disassemble the sampling apparatus.

    Step 1: Cut rope several feet above bailer
    Step 2: Dismantle bailer assembly
    Step 3: Place bailer parts into a dirty bailer cooler (cooler is then sent to lab for decontamination of bailers)

12. After sampling is completed, clean sampling apparatus with alconox or equivalent and distilled water.

b. Sampling technique utilizing a peristaltic pump:

   Used in cases where water depth is less than approximately 25 feet.

   This pump is used when the water level is within suction lift, i.e., within about 25 feet of the ground surface. It usually is a low-volume suction pump with low pumping rates suitable for sampling shallow, small-diameter wells.

   1. Cut tubing to desired length.

   2. Connect tubing to pump head, leaving 1 to 2 feet for discharge line.

   3. Lower tubing into the well water (1 to 2 feet below surface).

   4. Turn on pump and set speed at the desired rate of flow.

2. Residential Sampling—potable water supply

2.a Residential sampling is sampling conducted on a potable water supply. It is very important that these samples are representative of that water supply. The sampling point must be located ahead of any filtering devices or water conditioners. The highest standard of sampling technique is required for residential well sampling.

1. Put on sampling gloves to prevent contamination of the samples..

2. Purge private wells before sampling (including taking pH, conductivity, and temperature).

   Note: Rule of thumb—at least one well and storage tank volume should be removed. A 15-minute purging period is usually sufficient for residential wells.

3. Prepare sampling containers by filling out the label with the following information:
   – Project number
   – Location sampled
   – Individual collecting the samples
   – Date and time of sample collection
   – Sample analysis (if required by the lab)

4. Unscrew sampling container top (do not let the container or container top touch anything).

   Note: If applicable, collect the volatile samples first, proceeding to the least volatile.

5. Collect sample from the purge tap.

6. After completing the collection of the samples, place samples in a cooler with ice.

7. Turn off the tap; clean up any mess made by sampling.

3. Residential Systems (water supply system)

3.a Residential systems is sampling done on a water supply system. It must be representative of the water quality of that system. Preferably, a sampling tap will be ahead of the storage tank and close to the well head. Sample collection from this tap in the system must be from a steady stream of water.

1. Select a tap that is free from exterior contamination (remove anything attached to the faucet).

- If bacterial samples are to be collected, flame the end of the tap with a lighter or match to sterilize the tap.

2. Put on sampling gloves to prevent contamination of the samples

3. Turn on water tap; make sure the water is a steady stream out of the tap.

   Note: If water is not a steady stream, find a new tap. Also, make sure the tap is not leaking by the valve handle.

4. The water tap should be run steadily for two to three minutes or a sufficient time to permit clearing of the service line. Take pH, conductivity, and temperature.

5. Prepare sampling containers by filling out the label with the following information:

   - Project number
   - Location sampled
   - Individual collecting the samples
   - Date and time of sample collection
   - Sample analysis (if required by the lab)

6. Without changing water flow, the sample(s) can be collected.

   Note: Make sure there is no water splash up into the sampling container or cap. If applicable, collect the volatile samples first, proceeding to the least volatile.

7. Place sampling containers in the appropriate cooler with bagged ice.

8. Clean up any mess made by the sampling event.

3.b Collecting Field Samples

To ensure sample integrity, collect volatile samples first, then proceed to the least volatile method required for the site.

1. Volatiles and WI Gasoline range organics (WIGRO)– Samples to be analyzed for volatile organics will be collected in two or three 40-ml vials with Teflon®-lined septum caps. Use caution because concentrated acid may be present. Do not rinse glass vials. Hold bottle in one hand, the cap right side up in the other. Allow a slow stream of water to run into the 40-ml vial. The vial should be held at an angle while filling to prevent water from falling directly to the bottom of the container and becoming overly disturbed. While holding the vial vertically, add the water sample until a small meniscus forms on the top of the sample container. Avoid air bubbles and overfilling the vial. Cap tightly, invert the bottle, and tap gently. If any air bubbles appear in the vial, discard and collect sample in a new vial. These samples will be cooled to approximately 4°C. After collecting the required number of vials, insert them in a zip-lock plastic bag and place in a cooler with ice.

If prescribed by site-specific situations a duplicate volatile sample may be collected and field checked with a pH indicator strip to assess the pH of the sample. If the pH is greater than 2, the laboratory will be instructed to reduce the holding time of that day's samples to the 7-day holding period used for unpreserved samples.

2. Semivolatiles (includes: Pesticides, PCB, Herbicides, BNAs, Dioxin and Furans)– Samples to be analyzed for semivolatile organics will be collected in a 1-liter amber glass jar with a Teflon-lined septum cap for each fraction. Fill container slowly with a minimum headspace and cap tightly. Do not rinse glass containers. Place container directly in a cooler with ice. These samples will be cooled to approximately 4°C.

   Note: For Dioxin and furan analysis, the bottles must be preserved with 80 mg. sodium thiosulate if they are being collected from a chlorinated source.

3. WI Diesel Range Organics (WIDRO) – Samples to be analyzed for WIDRO are to be collected in a 1-liter amber glass jar with a Teflon-lined septum cap and preserved with 1:1 HCl to a pH or less than 2. Fill container slowly with a minimum of headspace and cap tightly. Do not rinse glass containers. Place container directly into a cooler with ice. These samples will be cooled to approximately 4°C.

4. Other Organics – Containers may contain acid, use caution when handling. Fill containers appropriately, minimizing headspace and avoiding spillage. Place container directly in a cooler with ice.

5. Metals

   - Total Metals – Samples to be analyzed for metals will be collected in a 500-mL or 1-liter polyethylene jar with a polyethylene-lined closure. These samples will be preserved in by the lab with a 1:1 (50%) solution of Nitric Acid to reduce the pH of the sample to less than 2.

   - Filtered Metals – Select the appropriate Corning filter size, either 250-ml or 500-ml volume (see Standard Operating Procedures for filtering groundwater samples). Pour filtered sample into metals sample container, minimizing headspace and avoiding spillage. Use caution handling metals containers because of nitric acid. Place directly in a cooler with ice.

6. Phenolics – Samples to be analyzed for phenol will be collected in a 1-liter glass jar. These samples will be preserved in the field with sulfuric acid to reduce the pH of the sample to less than 2 and cooled to approximately 4°C.

7. Oil and Grease by hexane extraction – Samples to be analyzed for Oil and Grease will be collected in a 1-liter glass jar with a Teflon-lined septum cap preserved to a pH or less than 2 with either 1:1 hydrochloric acid or 1:1 sulfuric acid . These samples will be cooled to approximately 4°C.

8. Cyanide – Groundwater samples to be analyzed for cyanide will be collected in a 1-liter polyethylene container with a polyethylene cap and preserved with sodium hydroxide to pH greater than 12 and cooled to approximately 4°C.

9. Collecting General Chemistry Samples – Samples to be analyzed for sulfate, chloride, carbonate, and bicarbonate will be collected in 1-liter plastic jars. These samples will be cooled to approximately 4°C.

10. Bacteria – Plastic bottles or glass containers preserved with 10 mg of sodium thiosulfate are used for bacterial sample collection. Care should be taken not to contaminate the container before collecting the sample. Fill the container within 1 inch of the top. This allows the laboratory to shake and mix the contents before analysis. Close and seal the Whirl Pak; grasp the wire ends and flip the pack in a circular motion several times and twist the wires together. Pack the containers carefully in a cooler with ice.

IV. Collecting Quality Control Samples

The effectiveness of the sample handling techniques is monitored by collecting both preserved and unpreserved field blank samples.

Field (or Masked) duplicate samples will be collected to measure relative sampling precision. Five percent of all samples collected are collected in duplicate. These samples are collected at the same time using the same procedures, equipment, and types of containers as the required samples. They are also preserved in the same manner and are either co-located or split and submitted for the same analyses as the required samples.

Trip blanks are only used when sampling for volatile organics. Their purpose is to determine if contamination has occurred as a result of improper sample container cleaning, contaminated blank source water, sample contamination during storage and transport due to exposure to volatile organics, or other environmental conditions during sampling and analysis. Trip blanks are prepared prior to the sampling events by the laboratory providing the sample containers. The water will be free of contaminants. The trip blanks are prepared by the lab, sealed and labeled appropriately at the lab, and transported to the field in the same containers as the sample vials. These blanks are not opened in the field. They are transferred to the coolers designated for volatile sample storage and transport and accompany the samples to the analytical laboratory.

Field blanks (or Rinsate Blanks) are used to evaluate the effects of onsite equipment contaminants. Their purpose is to determine if contamination has occurred as a result of improper equipment cleaning. Field blanks are prepared onsite by pouring analyte-free water through decontaminated sample collection equipment (bailer or pump) and collecting the rinsate in a sample container. The field blanks will be handled in the same manner as the sample group for which they are intended (i.e., blanks will be stored and transported with the sample group).

Some general considerations will be taken into account when planning and conducting sampling operations. The sampler will take into consideration the required sample volumes, sample holding times, sample handling, and special precautions for trace contaminant sampling.

The volume of the sample obtained should be sufficient to perform all required analyses with an additional amount collected to satisfy the needs for quality control, split samples, or repeat examinations. The Laboratory Coordinator should be consulted for any specific volume requirements. Multiple sample containers are always required for volatile organic compound (VOC) analyses.

The elapsed time between sample collection and initiation of each laboratory analysis will fall within a prescribed time frame. Holding times for samples required by this project are prescribed by EPA: Title 40 of the Code of Federal Regulations.

After collection, all samples should be handled as few times as possible. Technicians should use extreme care to ensure that samples are not contaminated. If samples are placed in a cooler, technicians should ensure that melted ice cannot cause sample containers to become submerged, as this may result in cross-contamination. Plastic bags, such as Ziplock® bags, should be used when small sample containers (e.g., VOC vials) are placed in coolers to prevent cross-contamination.

Some compounds can be detected in the parts per billion and/or parts per trillion range. Extreme care will be taken to prevent cross-contamination of these samples. A clean pair of new, disposable gloves will be worn for each sample location. Sample containers for source samples or samples suspected of containing high concentrations of contaminants are placed in separate plastic bags and coolers immediately after collecting, preserving and tagging. Sample collection activities will proceed progressively from the least contaminated area to the most contaminated area (when known).

Sample Storage

Place samples as soon as possible in a cooler containing bagged ice. Samples must be kept cold (4 ±2°C) at all times until delivery to the laboratory. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of fiberglass tape. Samples must be secure to prevent tampering with or loss of samples. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Note: Samples may have to be stored indoors in winter to prevent freezing.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

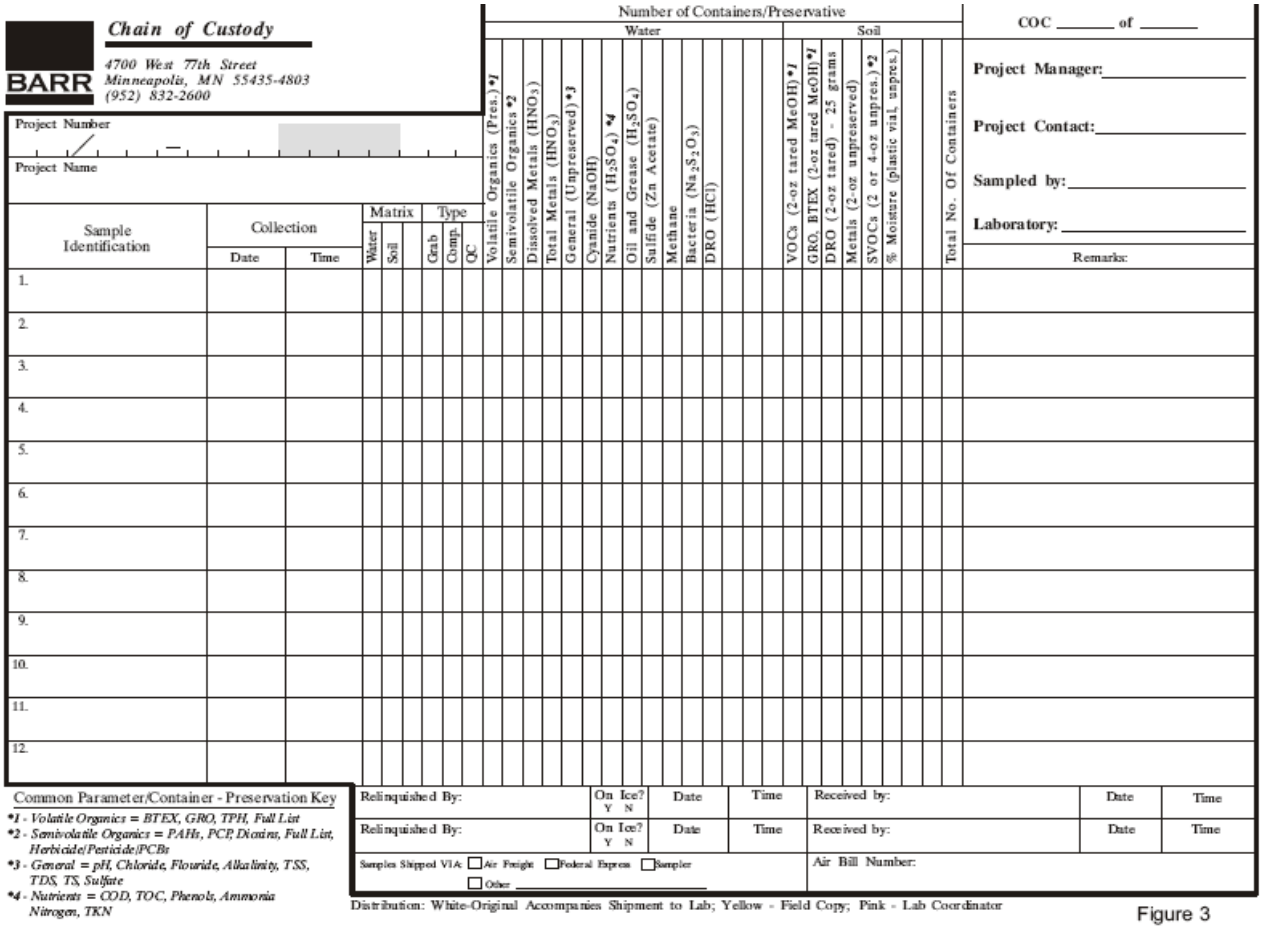
Documentation

The technician(s) will document the groundwater sampling events on field log data sheets, field log cover sheets, and field log data reports. They will also document the type and number of bottles on both the field log data sheet and chain-of-custody record. The analysis for each container and the laboratory used will be documented on the chain-of-custody record.

Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Sampling Report
Attachment 5: Field Log Cover Sheet
Attachment 6: Field Log Data Sheet

Attachment 1
Chain of Custody Form



Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number
Project Name

Number of Containers/Preservative

Sample Identification	Collection Date	Collection Time	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Water: Volatile Organics (Pres.) *1	Water: Semivolatile Organics *2	Water: Dissolved Metals (HNO_3)	Water: Total Metals (HNO_3)	Water: General (Unpreserved) *3	Water: Cyanide (NaOH)	Water: Nutrients (H_2SO_4) *4	Water: Oil and Grease (H_2SO_4)	Water: Sulfide (Zn Acetate)	Water: Methane	Water: Bacteria ($Na_2S_2O_3$)	Water: DRO (HCl)	Soil: VOCs (2-oz tared MeOH) *1	Soil: GRO, BTEX (2-oz tared MeOH) *1	Soil: DRO (2-oz tared) - 25 grams	Soil: Metals (2-oz unpreserved)	Soil: SVOCs (2 or 4-oz unpres.) *2	Soil: % Moisture (plastic vial, unpres.)	Total No. Of Containers
1.																										
2.																										
3.																										
4.																										
5.																										
6.																										
7.																										
8.																										
9.																										
10.																										
11.																										
12.																										

COC _____ of _____

Project Manager: _____

Project Contact: _____

Sampled by: _____

Laboratory: _____

Remarks:

Common Parameter/Container - Preservation Key
*1 - Volatile Organics = BTEX, GRO, TPH, Full List
*2 - Semivolatile Organics = PAHs, PCP, Dioxins, Full List, Herbicide/Pesticide/PCBs
*3 - General = pH, Chloride, Flouride, Alkalinity, TSS, TDS, TS, Sulfate
*4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other _____				Air Bill Number:		

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

Figure 3

CHAIN OF CUSTODY

Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

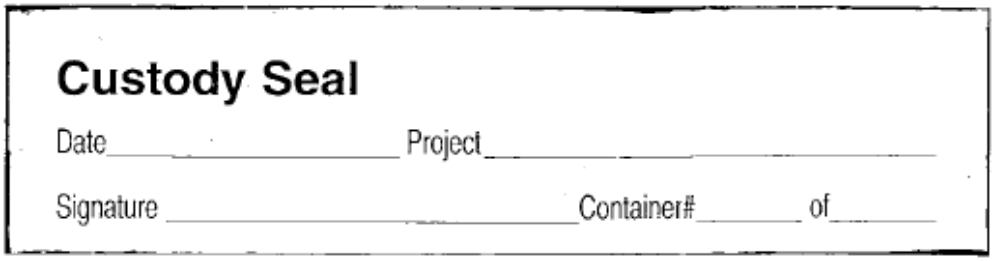
Preservative: _____

Sampled By: _____

Sample Location: _____

_____

Attachment 3
Custody Seal – if applicable



Attachment 4
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

Attachment 5
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Attachment 6
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample Time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:								
Casing diameter:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						
Others present:								
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)								
CASING & CAP: COLLAR: LOCK: OTHER:								
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:								
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-								
oil,grease- bacteria- total metal- filtered metal- methane- filter-								
Others:								

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\FieldLogDataSheet.doc

STANDARD OPERATING PROCEDURE

Filtering of Groundwater and Surface Water Samples

Revision 1

November 3, 2006

Approved By:	Andrea Nord	Andrea Nord		11/17/06
	Print	QA Manager(s)	Signature	Date
	Steve Iverson	Steve Iverson		11/16/06
	Print	Field Technician(s)	Signature	Date

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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	SDI	Date:	03-05-09
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for the Filtering of Groundwater and Surface Water Samples

Purpose

To describe the filtering process for groundwater and surface water samples to remove silt, clay, and particles.

Applicability

These procedures apply to the filtering of groundwater and surface water for laboratory analysis.

Equipment

Ziploc Baggies
Cooler
Bagged Ice
Chain of Custody Form
Sample Label
Talc-free latex or vinyl gloves
0.45 micron pore size filter
0.60 micron pore size filter – required if prefiltering the sample
Peristaltic or vacuum Pump
Tubing
Bubble Wrap

References

Corning Disposable Sterile Filter Information Booklet.

Responsibilities

The environmental technicians are responsible for the filtering of groundwater and surface water samples. The Field Operations/QA Officer or the environmental technician(s) will order the sample containers prior to the sampling event. The environmental technician(s) is responsible for the proper collection of groundwater and surface water samples, sample identification, quality control procedures, and documentation.

Procedure

Vacuum Pump - Filtering Process

1. Collect groundwater or surface water sample in an unpreserved sample container (filtering must be done within 15 minutes of collection).

2. Pour groundwater or surface water sample into 200-ml or 500-ml Corning Disposable Sterile Filter, depending on volume needed.

3. The filters must be 0.45 micron pore size.

Note: Prefiltering may be needed if sample is too turbid. The prefilter will filter particles up to 0.60 micron pore size.

a. Add prefilter to filter by placing it over the filter membrane (extends the life of the filter).
b. Filter membrane must be covered completely by prefilter to work properly.
c. Prefilter must be placed rough side up to be effective.

4. Attach vacuum pump to filter; turn on power.

5. Filter groundwater or surface water sample until amount of sample needed is filtered.

   Note: Additional filters may be needed to get enough sample volume.

6. After filtering is complete, pour contents into the appropriate sample container, dispose of filter. Depending upon groundwater conditions, additional filters may be required.

In-line Perastaltic Pump - Filtering Process

1. Attach 0.45 micron pore size filter to the end of purge tubing, ensuring direction of flow is correct. (filtering must be done within 15 minutes of collection).

2. Place appropriate sample container at the filter outlet

3. Turn on peristaltic pump until desired volume is achieved, then dispose of in-line filter. A new filter must be used for each sampling location. Depending upon groundwater conditions, additional filters may be required.

4. After filtering is complete, pour contents into the appropriate sample container, dispose of filter. Depending upon groundwater conditions, additional filters may be required.

Quality Control Samples

Equipment Blank An aliquot of reagent water that is subjected in the laboratory to all aspects of sample collection and analysis, including contact with all sampling devices and apparatus. The purpose of the equipment blank is to determine if the sampling devices and apparatus for sample collection have been adequately cleaned before they are shipped to the field site. An acceptable equipment blank must be achieved before the sampling devices and Apparatus are used for sample collection. One equipment blank must be collected per site or every 10 samples, whichever is more frequent.

Field Blank An aliquot of water that is placed in a sample container in the laboratory, shipped to the field, and treated as a sample in all respects, including contact with the sampling devices and exposure to sampling site conditions, filtration, storage and preservation, and all analytical procedures. The purpose of the field blank is to determine whether the field or sample transporting procedures and environments have contaminated the sample. One field blank must be collected per site or every 10 samples, whichever is more frequent.

Field Duplicates(FD1 and FD2) Two identical aliquots of a sample collected in separate sample bottles at the same time and placed under identical circumstance using a duel inlet sampler or by splitting a larger aliquot and treated exactly the same throughout filed and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection,

preservation, and storage, as well as with laboratory procedures. One set of field duplicates must be collected per site or every 10 samples, whichever is more frequent.

Sample Storage

The samples will be bubble wrapped or bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain of custody documentation. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of fiberglass tape. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

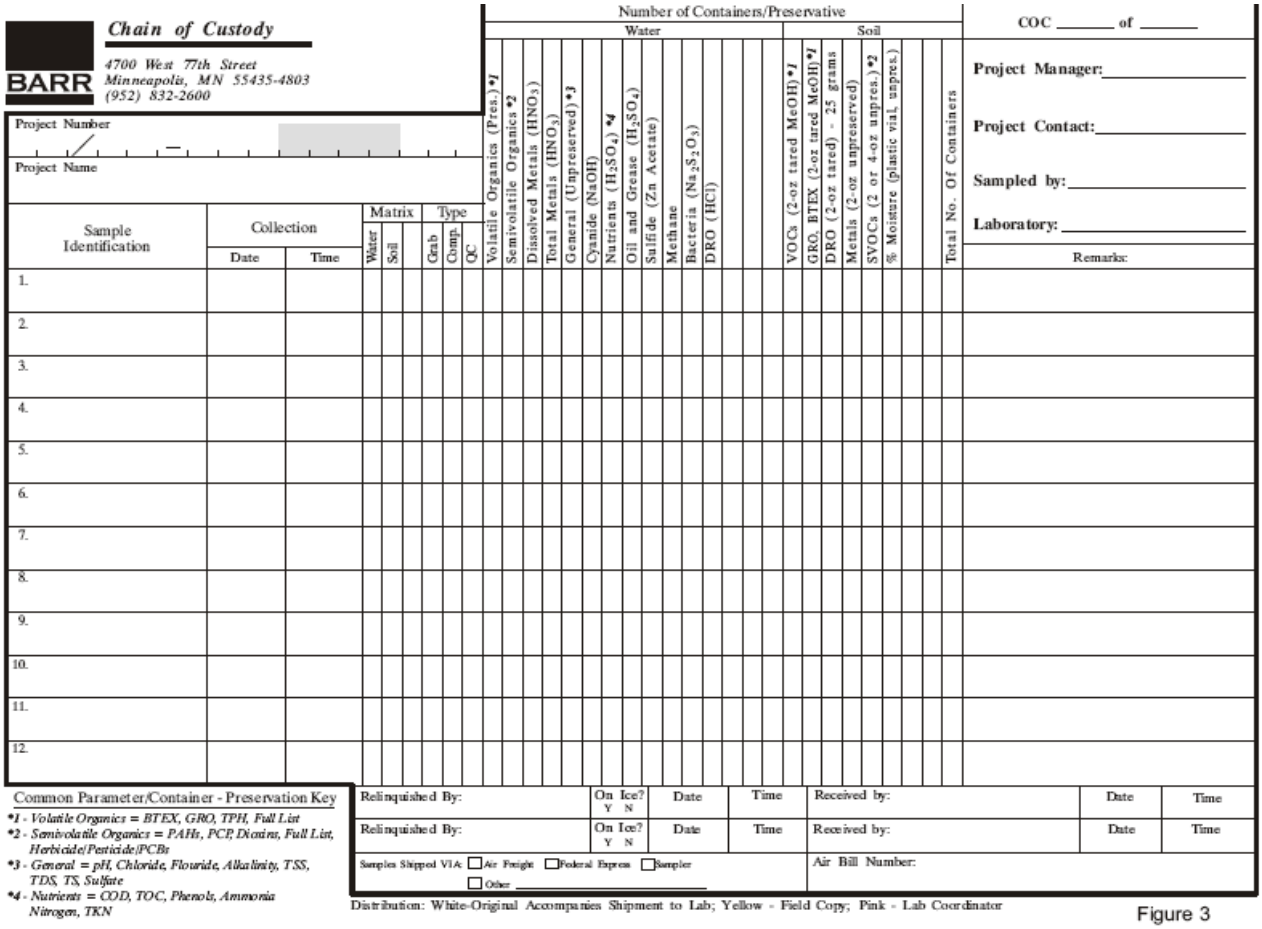
Documentation

The technician(s) will document the water sampling events on field log data sheets, field log cover sheets, and field log data reports. The technicians will document the number of filters and prefilters used for each sample filtered on the field log data sheet. They will also document the type and number of bottles on both the field log data sheet and chain-of-custody record. The analysis for each bottle and the laboratory used will be documented on the chain-of-custody record. The sampling request form will document which sampling containers are used for which water samples.

Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Sampling Report
Attachment 5: Field Log Cover Sheet
Attachment 6: Field Log Data Sheet

Attachment 1
Chain of Custody Form



Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number
Project Name

Number of Containers/Preservative

Water: Volatile Organics (Pres.)*1; Semivolatile Organics *2; Dissolved Metals (HNO_3); Total Metals (HNO_3); General (Unpreserved)*3; Cyanide (NaOH); Nutrients (H_2SO_4)*4; Oil and Grease (H_2SO_4); Sulfide (Zn Acetate); Methane; Bacteria ($Na_2S_2O_3$); DRO (HCl)

Soil: VOCs (2-oz tared MeOH)*1; GRO, BTEX (2-oz tared MeOH)*1; DRO (2-oz tared) - 25 grams; Metals (2-oz unpreserved); SVOCs (2 or 4-oz unpres.)*2; % Moisture (plastic vial, unpres.)

Total No. Of Containers

COC _____ of _____

Project Manager: _____
Project Contact: _____
Sampled by: _____
Laboratory: _____

Sample Identification	Collection Date	Collection Time	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Remarks
1.								
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.								
11.								
12.								

Common Parameter/Container - Preservation Key
*1 - Volatile Organics = BTEX, GRO, TPH, Full List
*2 - Semivolatile Organics = PAHs, PCP, Dioxins, Full List, Herbicide/Pesticide/PCBs
*3 - General = pH, Chloride, Flouride, Alkalinity, TSS, TDS, TS, Sulfate
*4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other _____				Air Bill Number:		

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

Figure 3

CHAIN OF CUSTODY

Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

Preservative: _____

Sampled By: _____

Sample Location: _____

_____

Attachment 3
Custody Seal – if applicable

Custody Seal

Date_____ Project_____

Signature _____Container#_____ of_____

Attachment 4
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

<Name inserts here>
Environmental Technician

Document1

Attachment 5
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Attachment 6
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample Time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:								
Casing diameter:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						
Others present:								
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)								
CASING & CAP: COLLAR: LOCK: OTHER:								
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:								
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-								
oil,grease- bacteria- total metal- filtered metal- methane- filter-								
Others:								

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\FieldLogDataSheet.doc

STANDARD OPERATING PROCEDURE

for Field Measurement of Water Temperature and pH Using the YSI Model 60

PCDOCS No.: 248885

Revision 0

March 12, 2009

Approved By:

Andrea Nord		03-12-09
Print	QA Manager(s) Signature	Date
STEVE JUERS		03-12-09
Print	Field Technician(s) Signature	Date

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Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____
Initials: _____	Date: _____

Standard Operating Procedures for Field Measurement of Water Temperature and pH Using the YSI Model 60

Purpose

The purpose is to describe the method by which temperature and pH measurements on water samples will be made and documented in the field.

Applicability

This procedure defines the proper technique for measuring water temperature and pH with the YSI Model 60 in depths up to 100 feet.

Equipment

YSI Model 60 pH Meter
Distilled or Deionized Water
Kim Wipes
4 pH Buffer Solution/KCL solution– necessary only when performing a thee point calibration
7 pH Buffer Solution
10 pH Buffer Solution
Nitrile Gloves
Field Log Data Sheet
Meter Calibration Summary Form

References

YSI 1999. *YSI Model 60 Handheld pH and Temperature System Operations Manual.* YSI Incorporated, Sept. 1999.

Responsibilities

The environmental technicians are responsible for the proper operation, maintenance, and checking calibration of the YSI Model 60.

Discussion

The YSI Model 60 instrument requires a two point calibration, at pH 7 and 10, at the beginning of each day of sampling. A three point calibration is necessary (pH 4, 7 and 10) if it is suspected that the water you will be testing has a pH of 6.5 or lower. The instrument has a self-diagnostic routine it runs each time the instrument is turned on. For the best results, calibrate the instrument as close to sample temperature as possible.

Specifications

Temperature	Range	-5 to +75°C
	Resolution	0.1°C
	Accuracy	±0.15°C ±1lsd
pH	Range	0 to 14 units
	Resolution	0.01 unit
	Accuracy	±0.1 unit within 10°C of calibration, +0.2 unit within 20°C

Field Calibration

Calibrate the YSI Model 60 at the beginning of each day of water sample analysis.

1. Press the **ON/OFF** key.
2. Wait several seconds while the instrument completes its self-diagnostic test.
3. Verify the instrument is reading in °C.
3. Rinse the pH probe with distilled or deionized water and blot dry with a Kim Wipe.
4. Immerse the pH and temperature probes in the pH 7 solution making sure that both the pH and temperature sensors are immersed in solution.
5. Wait approximately 5 minutes until the pH and temperature readings to stabilize (i.e. pH doesn't vary more than 0.01 for 2 minutes).
6. Enter the calibration menu by pressing the **UP ARROW** and **DOWN ARROW** keys simultaneously, the display should now read **CAL** at the bottom of the screen and **STAND** should be flashing and the main display will show 7.00.

   Note: The YSI Model 60 automatically adjusts the pH reading based upon the actual sample temperature at the time of the pH reading, therefore the pH values displayed with during calibration will vary with temperature.
7. Press the **ENTER** key: the display will still show **CAL** at the bottom, **STAND** will stop flashing and the pH value is shown with the middle decimal flashing. When the reading stabilizes, the decimal will stop flashing, press and hold he **ENTER** key to save the calibration point. The YSI Model 60 will flash **SAVE** on the display along with the **OFS** to indicate that the offset value has been saved.
8. **SLOPE** will now appear on the display and will be flashing. This indicates that the slope is ready to be set using a second pH buffer solution (pH 10). Rinse the probe with distilled or deionized water and blot dry with a Kim Wipe.
9. Immerse the pH and temperature probes in the pH 10 buffer solution and wait approximately 5 minutes until the pH reading stabilize.
10. Press the **ENTER** key. The display should now show **CAL** at the bottom, **SLOPE** will stop flashing and the pH calibration value (automatically sensed by the instrument) is shown with the right decimal point flashing. When the reading has stabilized, the decimal point will stop flashing. Press and hold the **ENTER** key when the reading stabilizes to save the first slope. The display will flash **SAVE** along with **SLP** to indicate that the first slope value has been saved. **SLOPE** will start flashing again indicating that the slope is ready to be set using a third pH buffer solution (pH 4). The system is now calibrated at two points. If you are only performing a two point calibration, press the **MODE** key to return to normal operation. If a three point calibration is required, follow steps 11 through 13.

    STOP HERE IF PERFORMING A TWO POINT CALIBRATION.
11. If a three point calibration is required, fill a container with the pH 4 buffer solution and immerse the probe into the solution, making sure the temperature sensor is immersed.

12. Press the **ENTER** key: the display will still show **CAL** at the bottom, **SLOPE** will stop flashing and the pH calibration value is shown with the left decimal flashing. When the reading stabilizes, the decimal will stop flashing, press and hold he **ENTER** key to save the second SLOPE.
13. The Model 60 will flash **SAVE** on the display along with **SLP** to indicate that the second slope value has been saved. The system is now calibrated at three points and will return to normal operation.

Testing the Calibration

1. Rinse the probe with distilled or deionized water and blot dry with a Kim Wipe.
2. Re-immerse the probe into the pH 7 buffer solution.
3. Wait for the pH reading to stabilize (not varying by more than 0.01 for 2 minutes)
4. Record the reading. If the difference between the expected and actual pH readings are ≤ 0.1 pH unit, you may begin analyzing water samples. If the difference exceeds 0.01 pH unit, recalibrate the instrument. If the instrument does not properly calibrate after 3 or 4 tries, contact the Field Equipment Technician and replace the instrument or send it in for servicing.
5. Rinse the probe with distilled or deionized water and blot dry with a Kim Wipe. You can now begin measuring the pH of water samples in the field.

Field Measurements

1. Place the probe in water, submerging it until the temperature sensor is under water. **Please ensure that the probe is NOT touching the bed of the river, or is in any sediment.** Also, please ensure that the meter itself is not submerged, and is above water level.
2. Shake gently to remove any trapped air bubbles and wait for the readings to stabilize (approximately 60 seconds or so).
3. The Model 60 has three modes: pH, which displays pH and temperature; Recall, which allows previously stored pH readings to be displayed; and Erase all, which erases all previously stored data. As we will be taking pH readings directly onto the field data sheet, there is no need to store data in the meter.
4. Take the pH readings, recording them directly onto the field log data sheet.
5. Rinse the probe with distilled or deionized water and blot dry with a Kim Wipe in between each water sample analysis.
6. Place the probe back in the upper right socket.
7. Press the **ON/OFF** key to turn the meter off.

Quality Control Samples

Replicate sample measurements should be taken a minimum of one per twenty project samples or a minimum of one per day. Method blanks must be one for every batch of samples analyzed.

Interferences

To ensure accurate pH measurements, make sure the that both the pH and temperature sensors are submersed in the buffer solution or water sample and make sure that there are no air bubbles on the pH probe when taking pH readings.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

Documentation

The field technician will document the YSI Model 60 calibration on the Meter Calibration Summary Form and record the water sample temperature and pH data on the Field Log Data Sheet.

Attachments

Attachment 1: Field Sampling Report
Attachment 2: Field Log Cover Sheet
Attachment 3: Field Log Data Sheet
Attachment 4: Meter Calibration Summary Form

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Attachment 2
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Attachment 3
Field Log Data Sheet

BARR

Client:		**Monitoring Point:**						
Location:		**Date:**						
Project #:		**Sample time:**						
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:		Time/ Volume	Temp. ºC	Cond. @ 25	PH	ORP/ unit	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*		NA						
Static well level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume purged:								
Duplicate collected:								
Sample collection by:								
Others present:		Well condition:						
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment Other: sump								
VOC Semi-volatile General Nutrient Cyanide DRO Sulfide								
Oil, grease Bacteria Total Metal Filtered Metal Methane Filter								
Others:								

Attachment 4
Meter Calibration Summary Form

MCS-1

BARR ENGINEERING COMPANY
METER CALIBRATION SUMMARY

PROJECT _____

TECHNICIAN _____

Meter type and number	Date	Time	Temperature C	Standard Used	Meter Reading	Slope	Conductivity Redline
Conductivity	Date	Solution Used	Cell Result				
Cell Check							
ORP Probe	Date	Temp.	ORP Reading	Calculation Result			
Check							
231+,- 10mV @ 25C							

231mV = Display Value + [(Display Temp. - 25 C) x (1.3 mV)]

WEATHER CONDITIONS

Date	Wind Direction	Wind Speed	Temperature F	Cloud Cover	Comments

Comments: _____

STANDARD OPERATING PROCEDURE

Maintenance and Operation of the YSI Model 556 MPS Water Quality Monitoring System

PCDOCS No.: 232050

Revision 1.0

April 27, 2009

Approved By:

Andrea Nord		04-27-09
Print QA Manager(s)	Signature	Date
Kim Johannessen		04-27-09
Print Field Technician(s)	Signature	Date

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Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:		Date:	03-05-09
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for the Maintenance and Operation of the YSI Model 556 MPS Water Quality Monitoring System

Purpose

The purpose of this SOP is to clearly define the procedures required to accurately measure dissolved oxygen, conductivity, temperature, pH and oxidation reduction potential (ORP) in the field using the YSI Model 556 MPS water quality system.

Applicability

This procedure is applicable to field personnel who will be using the YSI Model 556 MPS to measure dissolved oxygen, conductivity, temperature, pH and ORP in the field.

Definitions

ORP Oxidation Reduction Potential
MPS Multi-Probe System

Equipment

YSI Model 556 MPS
O-ring lubricant
Four alkaline "C" batteries
Mild soap
Water
ChemWipes
Screwdrivers
Conductivity standard
pH buffer solution (pH 7.00 and 10.00)
ORP solution (Zobel)
Zobel solution value chart
Moist sponge
Calibration cup
Field Log Data Sheet

References

YSI Model 556 MPS water quality system Operations Manual

Responsibilities

The environmental technician(s) is responsible for the proper sample identification; field screening procedures; field equipment and calibration; quality control procedures and documentation.

Instrument

The rugged and reliable YSI 556 MPS (Multi-Probe System) combines the versatility of an easy-to-use, easy-to-read hand-held unit. It features a waterproof, impact-resistant case and it simultaneously measures dissolved oxygen, conductivity, temperature, pH and ORP.

Maintenance/Installation

1. Instrument

The 556 requires occasional battery replacement and cleaning. Four alkaline "C" cells in the 556 provide 180 hours of operation. Battery life is displayed on the keypad. When the fuel gauge is low, it is time to change batteries.

a. Loosen the four screws in the battery lid on the back of the instrument.
b. Insert four "C" batteries in the clips following the polarity labels on the bottom of the battery compartment.
c. Check the gasket for proper placement and place the lid.
d. Do not over tighten the screws.
e. Clean the display pad with a mild soap and water solution.
f. Wipe the solution on and off.
g. Follow with a clean water wipe.

2. The Probe Module

To prepare the probe module for calibration and operation, the sensors need to be installed into the connectors on the probe module bulkhead. Whenever you install, remove or replace a sensor, it is important that the probe module and all the sensors be dry. This will prevent water from entering the port.

a. Unscrew and remove the probe sensor guard.
b. Using the sensor installation tool, unscrew and remove the sensor port plugs.
c. Locate the port with the connector that corresponds to the sensor that is to be installed.
d. Apply a thin coat of o-ring lubricant to the o-rings on the connector-side of the sensor.
e. Be sure that the probe module sensor port is free of moisture and insert the sensor into the correct port.
f. Gently rotate the sensor until the two connectors align.
g. With connectors aligned, screw down the sensor nut using the installation tool.
h. Repeat these steps for all sensors.

3. Instrument/Cable Connection

a. Line up the pins and guides on the cable with the holes and indentations on the cable connector at the bottom of the 556 instrument.
b. Holding the cable firmly against the cable connector, turn the locking mechanism clockwise until it snaps into place.

Calibration

All of the sensors, except temperature, require daily calibration to assure high performance. This will show specific calibration procedures for all sensors that require calibration. Make sure that the sensors are completely submersed when calibration values are entered. For maximum accuracy, use a small amount of calibration solution to pre-rinse the probe module. Have room temperature water on hand to rinse the probes between calibration solutions. Make sure to dry the probe module between rinses and calibration solutions. Be sure that port plugs are installed in all ports where sensors are not installed.

To access the calibration screen:

a. Press the on/off key to display the run screen.
b. Press the escape key to display the main menu screen.
c. Use the arrow keys to highlight the calibrate selection.
d. Press the enter key and the calibration screen is displayed.

1. Conductivity Calibration

a. Go to the calibrate screen as described above.
b. Use the arrow key to highlight the conductivity selection.
c. Press enter. The conductivity calibration screen is displayed.
d. Select the specific conductance selection. Press enter.
e. Place the correct volume of conductivity standard into a clean calibration cup.
f. Carefully immerse the sensor end of the probe module into the solution. The sensor must be completely immersed past its vent hole.
g. Gently move the probe up and down to remove any bubbles from the cell.
h. Use the keypad to enter the calibration value of the standard you are using. Be sure to enter the value in ms/cm@25ºC.
i. Press enter; the conductivity calibration screen is displayed. Allow at least one minute for temperature equilibration before proceeding. The current values for all enabled sensors will appear on the screen.
j. Observe the reading under specific conductance. When the reading shows no significant change for 30 seconds, press enter. The screen will indicate that the calibration has been accepted and prompt you to press enter. This returns you to the conductivity calibrate selection screen.
k. Press escape to return to the calibrate menu.
l. Rinse the probe module and dry.

2. Dissolved Oxygen Calibration

[Note: The instrument must be on for at least 20 minutes to polarize the DO sensor before calibrating. Calibrating any one option (% or µg/L) automatically calibrates the other.]

a. Go to the calibrate screen.

b. Use the arrow keys to highlight the dissolved oxygen selection. Press enter. The dissolved oxygen calibration screen is displayed.
c. Use the arrow keys to highlight the DO% selection. Press enter. The DO barometric pressure entry screen is displayed.
d. Place ⅛ inch of water in the bottom of the calibration cup and screw it on the probe module (only engage one or two threads to ensure the DO sensor is vented to the atmosphere).
e. Use the keypad to enter the current local barometric pressure. (If the unit has the optional barometer, no entry is required.)
f. Press enter and the DO% saturation calibrating screen is displayed. Allow 10 minutes for the air in the calibration cup to become water-saturated and for the temperature to equilibrate before proceeding.
g. Observe the reading under DO%. When the reading shows no significant change for 30 seconds, press enter. The screen will indicate that the calibration has been accepted and prompt you to press enter again. This will return you to the DO calibration screen.
h. Press escape to return to the calibrate menu.
i. Rinse the probe and dry.

Note: A moist sponge kept with the probe sensor guard to prevent the dissolved oxygen membrane from drying out.

3. pH Calibration

a. Go to the calibrate screen and select the pH selection.
b. Press enter, and the pH calibration screen is displayed.
c. Select the two-point option. Press enter. The pH entry screen is displayed.
d. Place the correct amount of pH buffer into a clean calibration cup. (Note: for maximum accuracy, the pH buffers you choose should be within the same pH range as the water you are sampling.)
e. Carefully immerse the sensor end of the probe module into the solution.
f. Gently rotate the probe up and down to remove any air bubbles.
g. Use the keypad to enter the calibration value of the buffer you are using. Press enter. The pH calibration screen is displayed.
h. Allow one minute for temperature equilibrium before proceeding. The current values of all enabled sensors will appear on the screen.
i. Observe the reading under pH. When the reading shows no significant change for 30 seconds, press enter. The screen will indicate the calibration has been accepted and prompt you to press enter again to continue.
j. Press enter. This returns you to the specified pH calibration screen.
k. Rinse the probe modules, calibration cup and sensors, and dry.
l. Repeat the above steps using the second pH buffer.
m. Press enter. This returns you to the pH calibration screen.
n. Press escape to return to the calibrate screen.
o. Rinse the probe and dry.

4. ORP Calibration

a. Go to the calibrate screen and use the arrows to highlight the ORP selection.
b. Press enter. The calibration screen is displayed.
c. Place the correct amount of a known ORP solution (Zobel) into a clean calibration cup. (Note: before proceeding, make sure the sensor is dry and, ideally, rinse it with ORP solution.)
d. Carefully immerse the sensor end of the probe up and down to remove any air bubbles.

e. Use the keypad to enter the correct value of the calibration solution you are using at the current temperature. Refer to the Zobel solution value chart.
f. Press enter. The ORP calibration screen is displayed.
g. Allow at least one minute for temperature equilibration before proceeding.
h. Observe the reading under ORP.
i. When the reading shows no significant change for 30 seconds, press enter. The screen will indicate that the calibration has been accepted and prompt you to press enter again to continue.
j. Rinse the probe and dry. The meter is now calibrated and ready for use.

If any calibrations fail, contact the Equipment Technician or manufacturer immediately or obtain a replacement instrument.

Quality Control Samples

Replicate sample measurements should be taken a minimum of one of twenty project samples per type of measurement. Method Blanks must be one for every batch of samples analyzed.

Safety

Please refer to the proper MSDS sheets or the Project Health and Safety Plan to determine the proper PPE required for use with the calibration solutions and reagents listed in this SOP prior to working with these chemicals.

Interferences

Rinse the probe sensor between instrument readings with water and dab dry to ensure accurate results.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for environmental pollution.

Documentation

The field technician will document the YSI Model 556 MPS dissolved oxygen, conductivity, temperature, pH and ORP data on the Field Log Data Sheet.

Attachments

Attachment 1: Field Sampling Report
Attachment 2: Field Log Cover Sheet
Attachment 3: Field Log Data Sheet
Attachment 4: Meter Calibration Summary Form

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

<Name inserts here>
Environmental Technician

Attachment 2
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

::ODMA\PCDOCS\DOCS\232050\1

Attachment 3
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:	**Monitoring Point:**
Location:	**Date:**
Project #:	**Sample Time:**

GENERAL DATA		STABILIZATION TEST						
Barr lock:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						

Others present:

WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)

CASING & CAP: COLLAR: LOCK: OTHER:

MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:

VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-

oil,grease- bacteria- total metal- filtered metal- methane- filter-

Others:

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\FieldLogDataSheet.doc

Attachment 4
Meter Calibration Summary Form

MCS-1

BARR ENGINEERING COMPANY
METER CALIBRATION SUMMARY

PROJECT _____

TECHNICIAN _____

Meter type and number	Date	Time	Temperature C	Standard Used	Meter Reading	Slope	Conductivity Redline
Conductivity	Date	Solution Used	Cell Result				
Cell Check							
ORP Probe	Date	Temp.	ORP Reading	Calculation Result			
Check							
231+,- 10mV @ 25C							
231mV = Display Value + [(Display Temp. - 25 C) x (1.3 mV)]							

WEATHER CONDITIONS

Date	Wind Direction	Wind Speed	Temperature F	Cloud Cover	Comments

Comments: _____

STANDARD OPERATING PROCEDURE

for Routine Level General Chemistry Data Validation

PCDOCS No.: 248821

Revision 2.1

March 16, 2009

Approved By: Michael Dupay _____

Print	QA Manager(s)	Signature	Date
Michael Dupay			03-16-09

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for Routine Level General Chemistry Data Validation

Purpose

This SOP is intended as a guidance document for the routine level validation of general chemistry data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine general chemistry data validation including a variety of approved methods not limited to the following analyses:

Chromium VI (Hexavalent Chromium)	Nitrate (or Nitrite) only
Alkalinity as $CaCO_3$	Nitrate + Nitrite
Ammonia	pH – *in lab*
BOD (Biological Oxygen Demand)	Phosphorus, total
COD (Chemical Oxygen Demand)	Sulfate
Chloride	Sulfide
Conductance, Specific – *in lab*	Total Dissolved Solids (TDS)
Cyanide (CN^- as HCN)	Total Kjeldahl Nitrogen (TKN)
Fluoride	Total Organic Carbon (TOC)
Hardness	Total Suspended Solids (TSS)
HEM (Oil and Grease)	

In the case of specific analyses not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

Definitions

Blank. A sample designed to assess specific sources of contamination.

BOD. Biological Oxygen Demand. The BOD test is an empirical bioassay-type test which measures the dissolved oxygen consumed by microbial life while assimilating and oxidizing organic matter in a sample.

COD. Chemical Oxygen Demand. The COD test determines the quantity of oxygen required to oxidize organic matter in a waste sample.

Contamination. A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

Duplicate. A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Field Blank. Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the Laboratory. A field blank includes trip blanks, equipment blanks, etc.

Field Duplicate. A duplicate sample generated in the field, not in the Laboratory.

HCl. Hydrochloric acid. Used as a sample preservative in some analyses.

HNO_3. Nitric acid. Used as a sample preservative in some analyses.

H_2SO_4. Sulfuric acid. Used as a sample preservative for some analyses.

Initial Calibration. Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument.

Instrument Blank. A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD). A control sample of known composition. LCSs and LCSDs are processed using the same sample preparation, reagents, and analytical methods employed for samples received. Sometimes referred to as a LFB (Laboratory Fortified Blank).

LFB. Laboratory Fortified Blank. See *Laboratory Control Sample*.

Matrix. The predominant material of which the sample to be analyzed is composed (e.g. water, soil, sediment, etc.)

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS). Aliquot of the sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate (MSD). A second aliquot of the same as the MS sample that is fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine precision of the method.

Method Detection Limit (MDL). The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99% probability that it is different from the blank. MDL studies performed by the laboratory should be consistent with SW-846, Ch. 1.

Method Blank. An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.

NaOH. Sodium hydroxide. Used as a preservative in some analyses.

Narrative. Portion of the data package which includes laboratory, contact, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Retention Time (RT). The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT Window established for that compound. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or

- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

TDS. Total Dissolved Solids. The amount of filterable residue in a given water sample.

TKN. Total Kjeldahl Nitrogen. The combination of organically bound nitrogen and ammonia (NH_3 and NH_4^+) in biological wastewater.

TOC. Total Organic Carbon. The carbon bound in an organic compound in waters and used as an indicator of water quality. Source of nutrients for undesirable biological growth.

TSS. Total Suspended Solids. The amount of non-filterable residue in a given water sample.

ZnAc + NaOH. Zinc acetate and sodium hydroxide. Used as a preservative of samples in the analysis for sulfide.

Equations

For % Recovery (%R or %Rec):

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where,

%R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample
In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of the associated approved analytical methods (EPA, ASTM, NPDS, etc.) and *Standard Methods for the Examination of Water and Wastewater*, 20th Ed. (Parts 1020A and 1020B).

Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

All samples should meet acceptance criteria for their respective analyses (and matrices) in the charts attached to the end of this SOP

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Attachments 1* and *2*, consider qualification with an "**h**".

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before (receipt). While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

It is understood that the method recommends that pH is a parameter that should be measured in the field. However, for conformational measurements in the laboratory, a recommended maximum holding time of 7 days from sample collection will be used for as a guideline for qualification. QAPP and SAP requirements may differ from this recommendation and professional judgment should be applied before qualifying any data.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

While not required for all methods, method blanks are recommended for all but pH analyses. Refer to *Attachments 1 and 2* at the end of this SOP for individual method requirements for method blank evaluation.

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the method blank. In such cases, it may be appropriate to qualify the affected data with "******" (usable value, QA/QC criteria not met).

Table 1 – Guidelines for the Evaluation of Blank Contamination

Sample Result	Recommended Action
Non-detect	No action required
<5x blank concentration	Qualify with "**b**"
>5x blank concentration	Use professional judgment
Gross contamination	Qualify associated samples with "******"

Note: "******" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. Per method (laboratories may have developed other acceptable QC measures), LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix

Not all methods require an LCS (or equivalent, such as a LFB). *Attachments 1 and 2* should be consulted to determine those analyses that require an LCS.

Laboratory control samples contain a known amount of each target compound and the percent recoveries are evaluated based either on the laboratory's internally-generated acceptance windows or default acceptance criteria when laboratory limits are not assigned generally fall between 75-125% recovery. *Table 2* presents the recommended guidelines for evaluating LCS/LCSD recoveries and qualification of samples from the associated batch.

Table 2 – LCS/LCSD Recovery Guidelines		
Spike Recovery	**Sample Concentration**	**Recommended Action**
< Lower Limit	Non-detect	Qualify with "*****" If LCS recovery is < 10%, consider "******"
	Detected	Qualify with "*****"
Between Lower and Upper Limits	Non-detect or Detected	Acceptable, no qualification.
> Upper Limit	Non-detect	No qualification required.
	Detected	Qualify with "*****"; If LCS recovery is >> upper limit, use professional judgment

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met.

IV. Laboratory Duplicates

Laboratory duplicates are analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and performance evaluation (PE) standards may not be used for duplicate analysis and are only evaluated for samples with concentrations greater than five times (>5x) the MDL. When methods require duplicates, they should be analyzed for each matrix.

In general, laboratory duplicates should be analyzed 1 duplicate in every 20 sample (where required). In some cases, a matrix spike duplicate may be considered an acceptable laboratory duplicate for methods requiring a matrix spike.

Duplicate results are compared to the associated native result for evaluation, using the equation for RPD defined in the *Equations* section in the beginning of this SOP.

RPD values are calculated only for results above the reporting limit and only if the following qualifiers do not apply: b, U, < and **.

Use laboratory acceptance criteria to evaluate RPDs, when available. The guidelines in *Table 3* may be used when laboratory acceptance criteria is not available.

Table 3 – Duplicate RPD Guidelines	
Matrix	**Recommended Action**
aqueous	if RPD is <20%, no action is required
	if RPD is >20%, but both results are <5x RL, no action is required
	if RPD is >20% and both results are >5x RL, qualify with *****
soil/sediment	if RPD is <35%, no action is required
	if RPD is >35%, but both results are <5x RL, no action is required
	if RPD is >35% and both results are >5x RL, qualify with *****

If both samples are non-detect, the RPD is not calculated.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are also used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the same equation as found in the *Equations* section in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs of <20-30% for aqueous samples and <30-40% for soil or sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample is detected and greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results based on field duplicate RPDs.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix may have on the sample preparation procedures and analytical results. While not required by every method, matrix spikes are typically analyzed 1 in 20 samples where required.

However, the frequency may also be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.).

If a matrix spike does not meet acceptance criteria and is not associated with the specific project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (greater than 4 times the native concentration (>4x)), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery defined in the *Equations* section in the beginning of this SOP.

If laboratory or QAPP acceptance criteria are provided for the percent recoveries of matrix spikes, those criteria should be used for the evaluation of the data. If acceptance criteria are not provided, percent recoveries between 75-125% are generally acceptable for matrix spikes.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity.

In general, matrix spikes and matrix spike duplicates may be evaluated as follows:

Table 4 – MS/MSD Recovery Guidelines

% Recovery of MS/MSD	Native Concentration	Recommended Action
<< Lower Limit (e.g. < 20%)	Non-detect	Consider qualifying with "**"
	Detected	Qualify with "*"
< Lower Limit	Non-detect	Qualify with "*"
	Detected	Qualify with "*"
Between Lower and Upper Limits	Non-detect or Detected	No qualification required
> Upper Limit	Non-detect	No qualification required
	Detected	Qualify with "*"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS/MSD pairs using the equation for RPD evaluation in the *Equations* section in the beginning of this document. Generally, acceptance criteria for MS/MSD are <20-30% RPD for aqueous samples and <30-40% for soils/sediments, but professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

IX. Attachments

Attachment 1: QC/QA Recommendations and Requirements Chart for Water Samples
Attachment 2: QC/QA Recommendations and Requirements Chart for Soil Samples
Attachment 3: Routine Level Quality Control Report
Attachment 4: Barr Qualifiers/Footnotes
Attachment 5: Revisions to SOP

Attachment 1
QC/QA Recommendations and Requirements Chart for Water Samples

	Recommended Hold Time						Required Preservation						QC Requirements					
Parameter (*Alternate Name*)	24 Hours (or ASAP)	48 Hours	7 Day	14 Day	28 Day	180 Day	Ice Only (or ≤ 6°C)	HCl	HNO_3	H_2SO_4	NaOH	ZnAc + NaOH	Method Blank	LCS	LSCD	Duplicate	MS	MSD
Chromium VI (*Hexavalent Chromium*)	X						X						X	X				
Alkalinity, as $CaCO_3$				X			X						R	R		R	R	
Ammonia					X					X			X	X		R	X	
BOD (*Biological Oxygen Demand*)		X					X						R			R		
COD (*Chemical Oxygen Demand*)					X					X			X			R		
Chloride					X		B						X	X	O	O	X	O
Conductance, specific – *in lab*					X		X						R	R		R		
Cyanide (*CN^- as HCN*)				X							X		X	X			X	
Fluoride					X		B						X	X	O	O	X	O
Hardness						X			X				R	R		R		
Nitrate (or Nitrite) only		X					X						X	X		O	X	O
Nitrate + Nitrite					X					X			X	X			X	
Oil and Grease (HEM)					X			X[b]		X[b]			X	X			X	R
pH[a] – *in lab*			X				X							R		R		
Phosphorus, total					X					X			R	R		R	R	
Sulfate					X		X						X	X	O	O	X	O
Sulfide			X									X	R	R		R	X	
Total Dissolved Solids (*TDS*)			X				X						R	R	R	R		
Total Kjeldahl Nitrogen (*TKN*)					X					X			R	R		R	R	
Total Organic Carbon (*TOC*)					X			X[b]		X[b]			X	R		R	X	
Total Suspended Solids (*TSS*)			X				X						R	R	R	R		

a Preferably in the field, otherwise 7 days
b Either preservative may be used (to pH <2)
R Recommended QA/QC test, not method requirement
X Method requirement
O Optional requirement (one must be used)
B No preservation is required, but ice is recommended for all samples

Attachment 2
QC/QA Recommendations and Requirements Chart for Soil Samples

	Recommended Hold Time					Required Preservation					QC Requirements					
Parameter (*Alternate Name*)	24 Hours (or ASAP)	48 Hours	7 Day	14 Day	28 Day	Ice Only (or ≤ 6°C)	HCl	H_2SO_4	NaOH	ZnAc + NaOH	Method Blank	LCS	LSCD	Duplicate	MS	MSD
Chromium VI (*Hexavalent Chromium*)					X	X					X	X		O	O	
Ammonia					X			X			X	X		R	X	
Chloride					X	X					X	X	O	O	X	O
Cyanide (*CN^- as HCN*)				X					X		X	X			X	
Fluoride					X	X					X	X	O	O	X	O
Nitrate (or Nitrite) only		X				X					X	X		O	X	O
Nitrate + Nitrite					X			X			X	X			X	
pH[a] – *in lab*			X			X						R		R		
Phosphorus, total					X			X			R	R		R	R	
Sulfate					X	X					X	X	O	O	X	O
Sulfide			X							X	R	R		R	X	
Total Kjeldahl Nitrogen (*TKN*)					X			X			R	R		R	R	
Total Organic Carbon (*TOC*)					X		X[b]	X[b]			X	R		R	X	

a Preferably in the field, otherwise 7 days
b Either preservative may be used (to pH <2)
R Recommended QA/QC test, not method requirement
X Method requirement
O Optional requirement (one must be used)

Attachment 3
Routine Level Quality Control Report

Barr Project # _____		***Project Name:*** _____			
Laboratory: _____		***Sample ID Event or COC#*** _____			
Lab Report # _____		***Matrix:***	***Soil*** _____	***Required Analysis:***	***VOC*** _____
Report Date: _____			***Water*** _____		***SVOC*** _____
Review By: _____	***Date:*** _____		***Air*** _____		***Metal*** _____
			Other _____		***GenChem*** _____
		Holding Times Met: ☐ ***Yes*** ☐ ***No***			
		Comments:			

Accuracy Data:	MS/MSD % Recovery Yes / No Sample ID_____	LCS/LCSD % Recovery
VOC		
SVOC		
Metals		
Other		

Precision Data:	MS/MSD RPDs, % Yes / No Sample ID_____	LCS/LCSD RPDs, %
VOC		
SVOC		
Metals		
Other		

Surrogate Standards Data		
Organics:		Inorganic Sample Dups:
VOC		Frequency: ____
SVOC		Results:

Blank Data:	Field Blank	Trip Blank (VOC Only)	Laboratory/Method Blank
VOC			
SVOC			
Metals			
Other			

Completeness Check: 100% Yes / No Comments:	**Historical Comparison:** N/A ____ Comments:

Masked/Blind Duplicate Results: **N/A**____ **Sample**_____

	Native Result	Duplicate Result	Native Result	Duplicate Result
VOC				
SVOC				
Metals				
Other				

Qualifiers/Qualifier Summary: **Yes / No** **(Note any TB, FB and MB affected)**

Sample Parameter	Add Qualifier	Remove Qualifier	Retain Qualifier

Other Actions Taken: Revised Report Requested _____	Lab Exception Report Completed: _____
Summary:	

Attachment 4
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes

--	Not analyzed/not available.
DLND	Not detected, detection limit not determined.
ND	Not detected.
AT	Sample chromatogram is noted to be atypical of a petroleum product.
a	Estimated value, calculated using some or all values that are estimates.
B	The reported value is less that the Contract Required Detection Limit (CRDL) but greater than or equal to the Instrument Detection Limit (IDL).
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
J	Associated value is an estimate.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
U	Not detected.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.

Attachment 5
Revisions to PCDOCS No.: 248821

Revision Number	Date of Revision	Section	Revision Made
2.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		Attachments	Added Attachment 5
		Attachment 1	Corrections to hold times and preservation requirements
		Attachment 2	Corrections to hold time requirements

STANDARD OPERATING PROCEDURE

for Routine Level Metals Data Validation

PCDOCS No.: 248176

Revision 2.1

March 16, 2009

Approved By:	Michael Dupay		03-16-09
	Print QA Manager(s)	Signature	Date

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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	_____	Date:	_____
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Standard Operating Procedures for Routine Level Metals Data Validation

Purpose

This SOP is intended as a guidance document for the routine level validation of metals data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine metals data validation for analysis by:

- ICP/AES (Methods EPA 200.7 or EPA 6010C)
- ICP/MS (Methods EPA 200.8 or EPA 6020A)
- Mercury (Methods EPA 245.1/245.5, EPA 7470A/7471B and EPA 1631E (including appendix))
- Any of the above in conjunction with TCLP procedure (EPA 1311)

Validation of Contract Laboratory Program (CLP) data should be performed in accordance with to the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004)*.

Definitions

AFS. Atomic Fluorescence Spectroscopy. A flame is used to solvate and atomize the sample, and a lamp emits light at a specific wavelength into the flame to excite the analyte atoms in the flame. The atoms of certain elements fluoresce and emit light in a different direction. The intensity of this fluorescing light is used for quantifying the amount of analyte element in the sample.

Blank. A sample designed to assess specific sources of contamination.

Duplicate. A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Field Blank. Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the Laboratory. A field blank includes trip blanks, equipment blanks, etc.

Holding Time. The maximum recommended amount of time samples may be held before they are processed.

HNO_3. Nitric acid. Used as a preservative.

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD). A control sample of known composition. LCSs and LCSDs are processed using the same sample preparation, reagents, and analytical methods employed for samples received.

Matrix. The predominant material of which the sample to be analyzed is composed (e.g. water, soil, sediment, etc.)

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike* (MS) *and Matrix Spike Duplicate (MSD). Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology.

Method Detection Limit (MDL). The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99% probability that it is different from the blank.

Method (Preparation) Blank. An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

Narrative. The portion of the data package which includes laboratory, contact, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Reporting Limit (RL). The RL is the lowest reported concentration, provided on the sample-analysis data report, after corrections have been made for sample dilution, sample weight, and (for soils and sediments) amount of moisture in the sample.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

TCLP. Toxicity Characteristic Leaching Procedure. A test designed to determine whether a waste is hazardous or requires treatment to become less hazardous; also can be used to monitor treatment techniques for effectiveness.

Equations

For % Recovery (%R or %Rec):

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where: %R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample

In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where: RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004)* and quality control recommendations outlined in:

- **EPA Methods 200.7/6010C** – *"Determination of Metals in Waters and Wastes by ICP-AES"*, 1994/February 2007
- **EPA Methods 200.8/6020A –** *"Determination of Trace Elements in Waters and Wastes by ICP-MS"*, 1994/February 2007
- **EPA Methods 245.1/245.5** – *"Determination of Mercury in Water by CVAAS/ Automated Cold Vapor Technique"*, 1994/1974
- **EPA Method 1631E (including Appendix)** – *"Mercury in Water by Oxidation, Purge and Trap, and CVAAS"*, August 2002
- **EPA Methods 7470A/7471B** – *"Mercury in Liquid/Solid Waste (Manual Cold Vapor Technique)"*, September 1994/February 2007

Responsibilities

The laboratory is responsible for generating metals data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the metals data in accordance with this document, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

The recommended hold time and preservation acceptance criteria are in *Table 1*.

Table 1 – Recommended Holding Times and Preservation

Compound	Matrix	Temperature	Preservative	Maximum Hold Time
Mercury	aqueous	< 6° C	HNO_3 < 2 pH	28 days
	aqueous (low level)	< 6° C	Pre-tested hydrochloric acid or bromium chloride	28 days
	sediment/soil	< 6° C	ice	28 days
	sediment/soil (low level)	< 6° C	Pre-tested hydrochloric acid or bromium chloride	28 days
All other metals	aqueous	< 6° C	HNO_3 < 2 pH	180 days
	sediment/soil	< 6° C	ice	180 days

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil) may not be necessarily be subjected to the same hold time recommendations.

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before receipt by the laboratory. While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

Low-level mercury considerations

Low-level mercury (Method 1631E) must be collected directly into a specially cleaned, pretested, fluoropolymer bottle using sample handling techniques specially designed for collection of mercury at trace levels and preserved with pre-tested hydrochloric acid (required for methyl mercury) or bromium chloride. Borosilicate glass bottles may be used if mercury is the only target analyte. Samples not collected in the correct type of container may be qualified with an "**h**". These samples may be shipped unpreserved provided:

- the sample is collected in a fluoropolymer bottle
- the bottle contains no headspace and is capped tightly
- sample temperature was maintained between 0-4°C, and
- the samples are acid-preserved within 48 hours of sampling.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group, or each batch digested (whichever is more frequent).
- Field blank collection and analysis frequency is project-specific.
- Low-level mercury method requires *at least* three method blanks per run per analytical batch.

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the method blank. In such cases, it may be appropriate to qualify the affected data with "******" (usable value, QA/QC criteria not met).

Table 2 – Guidelines for the Evaluation of Blank Contamination	
Sample Result	**Recommended Action**
Non-detect	No action required
<5x blank concentration	Qualify with "b"
>5x blank concentration	Use professional judgment
Gross contamination	Qualify associated samples with "**"

Note: "**" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix
- For low-level mercury, ongoing precision and recovery (OPR) samples are run before and after each analytical batch. Quality control samples (QCS) should be from a different source and analyzed once per analytical batch.

Laboratory control samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 9) and the percent recoveries are evaluated based either on the laboratory's internally generated acceptance windows or default method criteria (as given in *Table 3*).

Table 3 – Guidelines for Laboratory Control Sample Recoveries		
Matrix	**Acceptance Criteria**	**Action**
aqueous	80% to 120% recovery	if LCS > upper limit and samples are non-detect, no action; if detections, qualify with "*"
		if LCS is between < lower limit, use professional judgment when considering qualifying with "*"
		if LCS is << lower limit and samples are non-detect, qualify with "**"; if detections, qualify with "*"
sediment/soil	70% to 130% recovery	if LCS > 130%, and samples are non-detect, no action; if detections, qualify with "**"
		if LCS < 70% qualify detections with "*"; use professional judgment when considering non-detections with "**"

Note: "*"indicates the reported value is estimated and QA/QA criteria were not met.
"**" indicates the reported value is unusable and QA/QC criteria were not met.

IV. Laboratory Duplicates

Laboratory duplicates are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and performance evaluation (PE) standards may not be used for duplicate analysis and duplicate RPDs are only evaluated for samples with concentrations greater than five times (>5x) the MDL. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water), or
- One from each SDG

MS/MSD duplicate pairs may be substituted for laboratory duplicates.

Duplicate results are compared to the associated native result for evaluation, using the equation for RPD defined above.

Use laboratory acceptance criteria to evaluate RPDs, where available. When acceptance criteria is not available, use the following:

Table 4 – Guidelines for Laboratory Duplicate RPDs	
% RPD	**Action**
RPD is < upper limit	no action is required
RPD is > upper limit	if both results are <5x RL, no action is required
RPD is > upper limit	if both results are >5x RL, consider qualifying with "*****".

Note: "*"indicates the reported value is estimated and QA/QA criteria were not met.

If both samples are non-detect, the RPD is not calculated.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are also used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs between 20-30% for aqueous samples and 30-40% for soil and sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample is detected and greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix may have on the sample preparation procedures and analytical results. Matrix spike and matrix spike duplicate samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 9).

Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix, or
- 1 per sample delivery group

However, the frequency may be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.). In some cases, MS/MSD analysis is not required.

If a matrix spike does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (>4x), spike recovery can not be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery provided in the *Equations* section in the beginning of this SOP.

If laboratory acceptance criteria are provided for the percent recoveries of matrix spikes, those criteria should be used for the evaluation of the data. If acceptance criteria are not provided, percent recoveries between 75-125% are generally considered acceptable for matrix spikes.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity.

Table 5 – Guidance for Matrix Spike Evaluation	
Spike Result	**Recommended Action**
% recovery > upper acceptance limit	Non-detects, no qualification
	Detects, qualify with "*****"
% recovery meets acceptance limits	No qualification
% recovery is between 20% and lower acceptance limit	Non-detects, qualify with "*****"
	Detects, qualify with "*****"
% recovery is below 20%	Non-detects, use professional judgment; consider qualifying with "******"
	Detects, qualify with "*****"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation for RPD evaluation in the *Equations* section in the beginning of this document. Generally, acceptance criteria for MS/MSD are <20-30% RPD for aqueous samples and <30-40% for soils/sediments, but professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

IX. Additional Tables

Table 9 – Number of Target Compounds Required by NELAP and MN Rules for LCS/LCSD and MS/MSD samples	
Number of Target Parameters	**Required Number of Spiked Compounds**
1-10 analytes	Spike *all* compounds
11-20 analytes	*At least* 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike *at least* 16 compounds

X. Attachments

Attachment 1: Routine Level Quality Control Report
Attachment 2: Barr Qualifiers/Footnotes
Attachment 3: Revisions to SOP

Attachment 1
Routine Level Quality Control Report

Barr Project # _____		***Project Name:*** _____			
Laboratory: _____		***Sample ID Event or COC#*** _____			
Lab Report # _____		***Matrix:***	***Soil*** _____	***Required Analysis:***	***VOC*** _____
Report Date: _____			***Water*** _____		***SVOC*** _____
Review By: _____	***Date:*** _____		***Air*** _____		***Metal*** _____
			Other _____		***GenChem*** _____
		Holding Times Met: ☐ ***Yes*** ☐ ***No***			
		Comments:			

Accuracy Data:	MS/MSD % Recovery Yes / No Sample ID_____	LCS/LCSD % Recovery
VOC		
SVOC		
Metals		
Other		

Precision Data:	MS/MSD RPDs, % Yes / No Sample ID_____	LCS/LCSD RPDs, %
VOC		
SVOC		
Metals		
Other		

Surrogate Standards Data		
Organics:		Inorganic Sample Dups:
VOC		Frequency: ____
SVOC		Results:

Blank Data:	Field Blank	Trip Blank (VOC Only)	Laboratory/Method Blank
VOC			
SVOC			
Metals			
Other			

Completeness Check: 100% Yes / No Comments:	**Historical Comparison:** N/A ____ Comments:

Masked/Blind Duplicate Results: **N/A**____ **Sample**_____

	Native Result	Duplicate Result	Native Result	Duplicate Result
VOC				
SVOC				
Metals				
Other				

Qualifiers/Qualifier Summary: **Yes / No** **(Note any TB, FB and MB affected)**

Sample Parameter	Add Qualifier	Remove Qualifier	Retain Qualifier

Other Actions Taken: Revised Report Requested _____	Lab Exception Report Completed: _____
Summary:	

Attachment 2
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes

--	Not analyzed/not available.
DLND	Not detected, detection limit not determined.
ND	Not detected.
AT	Sample chromatogram is noted to be atypical of a petroleum product.
a	Estimated value, calculated using some or all values that are estimates.
B	The reported value is less that the Contract Required Detection Limit (CRDL) but greater than or equal to the Instrument Detection Limit (IDL).
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
J	Associated value is an estimate.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
U	Not detected.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.

Attachment 3:
Revisions to PCDOCS No.: 248176

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections;
		IX	Changed to Section X
		Attachments	Added Attachment 3
		IX (new)	Added Table 9.

STANDARD OPERATING PROCEDURE

for Routine Level Polychlorinated Biphenyls (PCB), Aroclor™, Pesticide and Herbicide Data Validation

PCDOCS No.: 248817

Revision 1.1

March 16, 2009

Approved By: Michael Dupay _____ _____ 03-16-09

Print QA Manager(s) Signature Date

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Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____

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Initials: _____ Date: _____

Initials: _____ Date: _____

Standard Operating Procedures for Routine Level Polychlorinated Biphenyls (PCB), Aroclor™, Pesticide and Herbicide Data Validation

Purpose

This SOP is intended as a guidance document for the routine level validation of polychlorinated biphenyls (PCBs), Aroclor™, herbicide and pesticide data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine level PCB, Aroclor™, herbicide and pesticide data validation by the analytical methods including, but not limited to:

- GC/ECD for Pesticides (EPA Methods 608/8081B)
- GC/ECD or GC/ELCD for PCBs/Aroclor™ (EPA Method 8082A)
- GC/FPD or GC/NPD for Organophosphorous Compounds (EPA Method 8141B)
- GC/ECD for Herbicides (EPA Method 8151A)

Validation of Contract Laboratory Program (CLP) data should be performed in accordance with to the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008)*.

Definitions

Aroclor™. A trademarked name for a mixture of polychlorinated biphenyls (PCBs) used in a variety of applications including additives in lubricants, heat transfer dielectric fluids, adhesives, etc.

Blank. An analytical sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

Contamination. A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

Field Blank. A blank used to provide information about contaminants that may be introduced during sample collection.

Herbicide. Any substance, or mixture of substances, intended to prevent the growth of or to destroy terrestrial or aquatic weeds. Weeds are any woody or non-woody undesirable vegetation.

GC/ECD. Gas Chromatography/Electron Capture Detector. Measures electron capturing compounds (usually halogenated) by creating an electrical field in which molecules exiting a GC column can be detected by the drop in current in the field. Identifies potential compounds based on retention time in a GC column, but the identification of the compound is not selective with one detector. Additional conformational analyses must be performed (either GC/MS or a separate GC/ECD with a different stationary phase on the column).

GC/FPD. Gas Chromatography/Flame Photometric Detector. The flame photometric detector (FPD) measures sulfur and phosphorus containing compounds, measuring chemiluminescent reactions from these compounds in a hydrogen / air flame.

GC/MS. Gas Chromatography/Mass Spectrometry (sometimes Mass Selective Detector (MSD)). An analytical technique used to measure the mass-to-charge ratio of ions. A MS detector can selectively identify a compound based on the compound's mass-to-charge ratio and generally does not require secondary confirmation.

GC/NPD. Gas Chromatography/Nitrogen-Phosphorus Detector. The nitrogen phosphorus detector (NPD) is a highly sensitive but specific detector similar to an FID. It gives a strong response to organic compounds containing nitrogen and/or phosphorus.

GC/PID. Gas Chromatography/Photoionization Detector. Uses ultraviolet light to ionize analyte exiting from a GC column. The resultant electrical charge produces a measurable current on a detector.

Initial Calibration. Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument.

Instrument Blank. A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Laboratory Control Sample (LCS). The LCS is an internal laboratory Quality Control (QC) sample designed to assess the capability of the laboratory to perform the analytical method.

Matrix. The predominant material of which the sample to be analyzed is composed.

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS). Aliquot of the sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate (MSD). A second aliquot of the same sample that is fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine precision of the method.

Method Blank. A reagent aqueous sample spiked with internal standards, and surrogate standards (or DMCs), that is carried throughout the entire analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.

Narrative. The portion of the data package which includes laboratory, contact person, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

Pesticide. Any substance or mixture of substances intended for preventing, destroying, repelling, or lessening the damage of any pest.

Polychlorinated Biphenyls (PCBs). A group of toxic, persistent chemicals used in electrical transformers and capacitors for insulating purposes, and in gas pipeline systems as a lubricant. The sale and new use of PCBs were banned by law in 1979.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Retention Time (RT). The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT Window established for that compound. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

Semi-Volatile Organic Compounds (SVOC). An organic compound which has a boiling point higher than water and which may vaporize when exposed to temperatures above room temperature. Semi-volatile organic compounds include phenols and polynuclear aromatic hydrocarbons (PAH).

Surrogates (Surrogate Standard). Compounds added to every blank, sample, including Laboratory Control Sample (LCS/LCSD), Matrix Spike/Matrix Spike Duplicate (MS/MSD), and standards; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

TCLP. Toxicity Characteristic Leaching Procedure. A test designed to determine whether a waste is hazardous or requires treatment to become less hazardous; also can be used to monitor treatment techniques for effectiveness.

Equations

For % Recovery (%R or %Rec):

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where,

%R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample
In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008)*, and quality control recommendations outlined in:

- **EPA Methods 608** – *"Organochlorine Pesticides and PCBs"*
- **EPA Method 8081B** – "Organochlorine Pesticides by Gas Chromatography", February 2007.
- **EPA Method 8082A –** *"Polychlorinated Biphenyls (PCBs) by Gas Chromatography"*, February 2007.
- **EPA Method 8141B –** *"Organophosphorus Compounds by Gas Chromatography: Capillary Column Technique"*, February 2007.
- **EPA Method 8151A** – *"Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization"*, December 1996.
- **EPA Method 1311** – *"Toxicity Characteristic Leaching Procedure"* July 1992.

Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the data in accordance with this SOP, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

The recommended hold time and preservation acceptance criteria are in *Table 1*.

Table 1 – Recommended Hold Times and Preservation

Compound	Matrix	Temp.	Preservative	Maximum Hold Time
PCBs/Aroclor™/ Pesticides (EPA 8081/8082)	aqueous	< 6° C	ice	7 days extraction/ addl. 40 days analysis
	sediment/ soil	< 6° C	ice	14 days extraction/ addl. 40 days analysis
PCBs/Pesticides (EPA 608)	aqueous	< 6° C	ice (if >72 hrs to extraction, preserve to pH 5-9 with NaOH and/or H_2SO_4)	72 hours extraction unpreserved/ 7 day extraction preserved/ addl. 40 days analysis
Herbicides (EPA 8151)	all matrices	< 6° C	ice	7 day extraction/ addl. 40 days analysis

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil) may not be necessarily be subjected to the same hold time recommendations.

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before receipt by the laboratory. While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group, or each batch digested (whichever is more frequent).
- The laboratory should analyze a method blanks at least once every 20 samples.
- Field blank collection and analysis frequency is project-specific.

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the method blank. In such cases, it may be appropriate to qualify the affected data with "******" (usable value, QA/QC criteria not met).

Table 2 – Guidelines for the Evaluation of Blank Contamination

Sample Result	Recommended Action
Non-detect	No action required
<5x blank concentration	Qualify with "**b**"
>5x blank concentration	Use professional judgment
Gross contamination	Qualify associated samples with "******"

Note: "******" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Surrogates Standards

Recovery limit guidelines are presented in the table below. Keep in mind that the laboratory may have different limits and compounds than those recommended. Recommended surrogate compounds are in *Tables 6 and 7* in ***Section IX*****.** Laboratory or QAPP assigned limits should be used where provided.

All samples (blanks, spiked samples, project samples, QC samples) should contain surrogates. If a sample does not contain surrogates, professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of surrogate spikes may not be applicable if dilution of the sample was required.

Table 3 – Guidelines for Surrogate Standard Recoveries			
Analysis	**Sample Concentration**	**Surrogate recovery**	**Recommended Action**
PCB/ Aroclor™/ Pesticides/ Herbicides	Non-detect	< 10% recovery	Qualify associated compounds with "**"
		< lower recovery limit	Qualify associated compounds with "*"
		Within or > acceptance criteria	No action
	Detections above reporting limits	< lower recovery limit	Qualify associated compounds with "*"
		Within acceptance criteria	No action
		> upper recovery limit	Qualify associated compounds with "*"

Note: "*"indicates that the reported value is estimated and QA/QA criteria were not met;
"**" indicates that the reported value is unusable and QA/QC criteria were not met.

In instances where surrogate recoveries are marginally outside acceptance criteria (within 10% of recovery limits), additional consideration should be given to historical results (where available) and general recovery trends in related samples of the same report before qualifying the sample.

IV. Laboratory Control Samples (LCS)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. Per method (laboratories may have developed other acceptable QC measures), LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix

Laboratory control samples contain a known amount of each target compound and the percent recoveries are evaluated based either on the laboratory's internally generated acceptance windows or default method criteria (as presented in *Table 4*). Herbicides do not currently have EPA-recommended recovery acceptance criteria. For the purposes of this SOP, use the recommended guidelines for LCS spike recoveries of PCBs/Aroclor™ to evaluate data (50-150% recoveries are acceptable).

Table 4 – Guidelines for Laboratory Control Sample Recoveries		
Analysis	**Acceptance Criteria**	**Recommended Action**
PCBs/Aroclor™	50-150% recovery (Aroclor™ 1016 and Aroclor™ 1260 are the recommended spike compounds)	if LCS > 150% & samples are non-detect, no action; if detections, qualify with "*"
		if LCS < 50%, qualify samples with "**"
		if LCS < 10%, qualify detects with "**" qualify non-detects with "**"
Pesticides	See Table 6 in **Section IX** for EPA-recommended compounds and recoveries	if LCS > upper limit & samples are non-detect, no action; if detections, qualify with "*"
		if LCS < lower limit, qualify samples with "**"
		if LCS < 10%, qualify detects with "**" qualify non-detects with "**"

Note: "**"indicates that the reported value is estimated and QA/QA criteria were not met;
"**" indicates that the reported value is unusable and QA/QC criteria were not met.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs of <20-30% for aqueous samples and <30-40% for soil or sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results are dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix may have on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix, or
- 1 per sample delivery group

However, the frequency may be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.). In some cases, MS/MSD analysis is not required.

If a matrix spike does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (greater than 4 times (>4x)), spike recovery criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery provided in the *Equations* section in the beginning of this SOP.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity.

In general, matrix spikes and matrix spike duplicates may be evaluated as follows:

Table 5 – Guidelines for Matrix Spike Evaluation	
Spike Result	**Recommended Action**
% recovery > upper acceptance limit	Non-detects, no qualification
	Detects, qualify with "*****"
between upper and lower limits	No qualification
% recovery is between 20% and lower acceptance limit	Non-detects, qualify with "*****"
	Detects, qualify with "*****"
% recovery is below 20%	Non-detects, use professional judgment; consider qualifying with "******"
	Detects, qualify with "*****"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS/MSD pairs using the equation for RPD evaluation provided in the *Equations* section in the beginning of this SOP. Generally, acceptance criteria for MS/MSD are <20-30% RPD for aqueous samples and <30-40% for soils or sediments, but professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

Note: Pesticides, herbicides, PCBs and Aroclors™ **require** additional ECD or GC/MS confirmation of tentatively identified compounds (TIC), using a separate column. This may occur at the same time as the initial analysis using a dual-column GC with an additional detector; or a second, separate analysis via EPA 8270 (See Barr SOP for SVOC Data Validation if positive detections occur). Herbicides are sufficiently identified by a single column if a GC/MS is used for analysis. If there is indication that confirmational analysis was not performed for the remaining parameters, professional judgment should be used to critically evaluate the usability of the data as reported.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

IX. Additional Tables

The following tables should be used for guidance purposes *only*. Priority should be given to laboratory limits and associated target compounds when provided by the laboratory. Use professional judgment before applying any of the data in the following tables to the validation process.

Table 6 – Recommended Guidance for LCS Compounds and Recovery for Pesticides

Compound	Recovery limits (%)
4,4'-DDE	50-150
Dieldrin	30-130
Endosulfan sulfate	50-120
Endrin	50-120
gamma-BHC	50-120
gamma-Chlordane	30-130
Heptachlor epoxide	50-150

Table 7 – Recommended Surrogates

Analysis	Recommend Surrogate
PCBs/Aroclor™/Pesticides	Tetrachloro-m-xylene (TCX)
	Decachlorobiphenyl (DCB)
Herbicides	2,4-Dichlorophenylacetic acid (DCAA)

X. Attachments

Attachment 1: Routine Level Quality Control Report
Attachment 2: Barr Qualifiers/Footnotes
Attachment 3: Revisions to SOP

Attachment 1
Routine Level Quality Control Report

Barr Project # _____		***Project Name:*** _____			
Laboratory: _____		***Sample ID Event or COC#*** _____			
Lab Report # _____		***Matrix:***	***Soil*** _____	***Required Analysis:***	***VOC*** _____
Report Date: _____			***Water*** _____		***SVOC*** _____
Review By: _____	***Date:*** _____		***Air*** _____		***Metal*** _____
			Other _____		***GenChem*** _____
		Holding Times Met: ☐ ***Yes*** ☐ ***No***			
		Comments:			

Accuracy Data:	MS/MSD % Recovery Yes / No Sample ID_____	LCS/LCSD % Recovery
VOC		
SVOC		
Metals		
Other		

Precision Data:	MS/MSD RPDs, % Yes / No Sample ID_____	LCS/LCSD RPDs, %
VOC		
SVOC		
Metals		
Other		

Surrogate Standards Data		
Organics:		Inorganic Sample Dups:
VOC		Frequency: ____
SVOC		Results:

Blank Data:	Field Blank	Trip Blank (VOC Only)	Laboratory/Method Blank
VOC			
SVOC			
Metals			
Other			

Completeness Check: 100% Yes / No Comments:	**Historical Comparison:** N/A ____ Comments:

Masked/Blind Duplicate Results: **N/A**____ **Sample**_____

	Native Result	Duplicate Result	Native Result	Duplicate Result
VOC				
SVOC				
Metals				
Other				

Qualifiers/Qualifier Summary: **Yes / No** **(Note any TB, FB and MB affected)**

Sample Parameter	Add Qualifier	Remove Qualifier	Retain Qualifier

Other Actions Taken: Revised Report Requested _____	Lab Exception Report Completed: _____
Summary:	

Attachment 2
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes

--	Not analyzed/not available.
DLND	Not detected, detection limit not determined.
ND	Not detected.
AT	Sample chromatogram is noted to be atypical of a petroleum product.
a	Estimated value, calculated using some or all values that are estimates.
B	The reported value is less that the Contract Required Detection Limit (CRDL) but greater than or equal to the Instrument Detection Limit (IDL).
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
J	Associated value is an estimate.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
U	Not detected.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.

Attachment 3:
Revisions to PCDOCS No.: 248817

Revision Number	Date of Revision	Section	Revision Made
1.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		Attachments	Added Attachment 3

STANDARD OPERATING PROCEDURE

for Routine Level Semivolatile Organic Compounds (SVOC) and Polycyclic Aromatic Hydrocarbons (PAH) Data Validation

PCDOCS No.: 248818

Revision 3.1

March 16, 2009

Approved By: Michael Dupay _____

Print	QA Manager(s)	Signature	Date
			03-16-09

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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for Routine Level Semivolatile Organic Compounds (SVOC) and Polycyclic Aromatic Hydrocarbons (PAH) Data Validation

Purpose

This SOP is intended as a guidance document for the routine level validation of semivolatile organic compounds (SVOC) and Polycyclic Aromatic Hydrocarbons (PAH) data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine SVOC (including PAHs, PCPs) and diesel range organics (DRO) data validation by the analytical methods including, but not limited to:

- GC/MS for SVOCs (EPA Method 8270D and 8270D SIM)
- GC/FID for PAHs (EPA Method 8100)
- HPLC for PAHs (EPA Method 8310)
- Wisconsin (WI) DRO (SW-141)
- GC/FID for DRO (EPA Method 8015C)
- TCLP/SVOC (EPA Methods 1311/8270D)

Validation of Contract Laboratory Program (CLP) data should be performed in accordance with to the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008)*.

Definitions

Blank. An analytical sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

Contamination. A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

DRO. Diesel Range Organics. Organic range corresponding to a hydrocarbon range of C_{10} - C_{28} and a boiling point range between approximately 170°C and 430°C. Other organic compounds, including chlorinated hydrocarbons, phenols, phthalate esters, polynuclear aromatic hydrocarbons, kerosene, fuel oils and heavier oils, are measurable.

Deuterated Monitoring Compounds (DMCs). Compounds added to every semivolatile calibration standard, blank, and sample used to evaluate the efficiency of the extraction/purge and trap procedures, and the performance of the Gas Chromatograph/Mass Spectrometer (GC/MS) systems. DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are not expected to be naturally detected in the environmental media.

Field Blank. A blank used to provide information about contaminants that may be introduced during sample collection.

GC/FID. Gas Chromatography/Flame Ionization Detector. As components elute from the GC's column they pass through the flame and are burned, producing ions. The ions propagate an electric current, which is the signal output of the detector. The greater the concentration of the component, the more ions are produced, and the greater the current.

GC/MS. Gas Chromatography/Mass Spectrometry (sometimes Mass Selective Detector (MSD)). An analytical technique used to measure the mass-to-charge ratio of ions. A MS detector can selectively identify a compound based on the compound's mass-to-charge ratio and generally does not require secondary confirmation.

HCl. Hydrochloric acid.

HPLC. High Performance Liquid Chromatography. A chromatographic technique for separating and analyzing mixtures of substances, using a packed column with small particles coated with the stationary phase and where the mobile phase is pumped through the column with a high pressure pump. For the purposes of these analyses, a fluorescence or UV (ultraviolet) detector is used to identify the chromatographic separations.

Initial Calibration. Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument.

Internal Standards. Compounds added to every volatile calibration standard, blank, sample, or sample extract, including the Laboratory Control Sample (LCS), at a known concentration, prior to analysis. Internal standards are used to monitor instrument performance and quantitation of target compounds.

Instrument Blank. A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Laboratory Control Sample (LCS). The LCS is an internal laboratory Quality Control (QC) sample designed to assess the capability of the laboratory to perform the analytical method.

Matrix. The predominant material of which the sample to be analyzed is composed.

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS). Aliquot of the sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MeOH. Methanol.

Method Blank. A reagent aqueous sample spiked with internal standards, and surrogate standards (or DMCs), that is carried throughout the entire analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.

Narrative. Portion of the data package which includes laboratory, contact, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Retention Time (RT). The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT Window established for that compound. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

SIM. Selected Ion Monitoring. Method of GC/MS scanning that focuses on only a few ions for detection. Using this technique increases the sensitivity of a mass spectrometry detector as much as 500-fold.

Semivolatile Organic Compounds (SVOC). An organic compound which has a boiling point higher than water and which may vaporize when exposed to temperatures above room temperature. Semivolatile organic compounds include phenols and polynuclear aromatic hydrocarbons (PAH)

Surrogates (Surrogate Standard). Compounds added to every blank, sample, including Laboratory Control Sample (LCS/LCSD), Matrix Spike/Matrix Spike Duplicate (MS/MSD), and standards; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

TCLP. Toxicity Characteristic Leaching Procedure. A test designed to determine whether a waste is hazardous or requires treatment to become less hazardous; also can be used to monitor treatment techniques for effectiveness.

Equations

For % Recovery (%R or %Rec):

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where,

%R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample
In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (January 2005)*, and quality control recommendations outlined in:

- **SW-141** – *"Wisconsin DRO"*, September 1995;
- **EPA Method 1311** – *"Toxicity Characteristic Leaching Procedure"*, July 1992;
- **EPA Method 8015B** – *"Nonhalongenated Organics Using GC/FID"*, February 2007;
- **EPA Method 8100** – *"Polynuclear Aromatic Hydrocarbons"*, September 1986;
- **EPA Method 8270** – *"Semivolatile Organic Compounds by GC/MS"*, February 2007; and
- **EPA Method 8310** – *"Polynuclear Aromatic Hydrocarbons*, September 1986.

Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the data in accordance with this SOP, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

The recommended hold time and preservation acceptance criteria are in *Table 1*.

Table 1 – Recommended Hold Times and Preservation

Compound	Matrix	Temperature	Preservative	Maximum Hold Time
SVOCs /PAHs	aqueous	< 6° C	ice	7 days extraction/ addl. 40 days analysis
	sediment/soil	< 6° C	ice	14 days extraction/ addl. 40 days analysis
WI DRO	aqueous	< 6° C	HCl <2 pH	7 days extraction/ addl. 40 days analysis
	sediment/soil	< 6° C	ice	14 days extraction/ addl. 40 days analysis
TCLP	all matrices	< 6° C	ice	14 days TCLP extraction / 7 days prep. extraction / addl. 40 days analysis

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil) may not be necessarily be subjected to the same hold time recommendations.

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before receipt by the laboratory. While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

A separate sample (without preservative) should be collected for each soil/sediment sample to be analyzed for DRO for the determination of moisture content. Samples without moisture content determination should be reported as wet weight.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group, or each batch digested (whichever is more frequent).
- The laboratory should analyze a method blanks at least once every 20 samples.
- At least one method blank should be analyzed with each concentration level (e.g. low or medium).
- Field blank collection and analysis frequency is project-specific.

Table 2 – Guidance for the Evaluation of Blank Contamination

Analyses	Positive Detection in Blank	Sample Result	Recommended Action
SVOCs/ DRO/ PAHs	Common laboratory contaminants (e.g. common phthalate esters)	Non-detect	No action required
		<10x blank concentration	Qualify with "**b**"
		>10x blank concentration	Use professional judgment
	All other target parameters	Non-detect	No action required
		<5x blank concentration	Qualify with "**b**"
		>5x blank concentration	Use professional judgment
Any analysis	Gross contamination (e.g. saturated peaks of target analytes in blanks)	All detections	Qualify with "******"
SVOC 8270 SIM	All target parameters	Non-detect	No action required
		< 20x blank concentration	Qualify with "**b**"
		> 20x blank concentration	Use professional judgment

Note: "******" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Deuterated Monitoring Compounds (DMC), (Surrogates)

Deuterated Monitoring Compounds (DMC) are also frequently known as System Monitoring Compounds (SMC) or Surrogates. Keep in mind that the laboratory may have different limits and compounds than those recommended. *Table 7* in **Section IX** presents the *recommended* compounds and recovery limits (per Guidelines) for DMCs used by laboratories (SVOCs only). Associated methods may provide additional guidance. Laboratory or QAPP assigned limits should be used where provided.

All samples (blanks, spiked samples, project samples, QC samples) should contain DMCs or surrogates. If a sample does not contain DMCs or surrogates, professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of DMCs or surrogates may not be applicable if dilution of the sample was required.

Table 3 – Guidance for the Recovery of Deuterated Monitoring Compounds

Analysis	Sample Concentration	DMC/surrogate recovery	Recommended Action
SVOC/ SVOC SIM	Sample is non-detect or has concentrations of associated target compounds less than reporting limit (RL)	< 10% recovery	Qualify associated target compounds with "**"
		< lower recovery limit	Qualify with associated target compounds with "*"
		within or > acceptance limits	No action
	Sample has detectable concentrations of associated target compounds above reporting limit (RL)	< lower recovery limit	Qualify with associated target compounds with "*"
		within acceptance limits	No action
		> upper recovery limits	Qualify with associated target compounds with "*"

Note: "*"indicates that the reported value is estimated and QA/QA criteria were not met;
"**" indicates that the reported value is unusable and QA/QC criteria were not met.

In instances where the DMC or surrogate recoveries are marginally outside acceptance criteria (within 10% of recovery limits), additional consideration should be given to historical results (where available) and general recovery trends in related samples of the same report before qualifying the sample.

In all cases, only the target compound(s) associated with the DMC that fails to meet acceptance criteria may require qualification.

Table 8 in **Section IX** presents the recommended DMCs with their associated target compounds for SVOCs *only*. Bear in mind that laboratory methods may deviate from the recommended guidance and surrogate failures should be evaluated based on laboratory SOPs, especially in regard to which DMCs (surrogates) are associated with which target compounds.

Not all DMC/surrogates are utilized in all SVOC analyses. If alternate or fewer surrogates are used, the following guidelines are recommended:

Table 4 – Guidance for the Recovery of Deuterated Monitoring Compounds (If Fewer DMCs than National Function Guidelines Recommend Are Used)

DMC/Surrogate recoveries	Recommended Action
One DMC < 10% recovery	All detects of same fraction (Acid or Base/Neutral), qualify with "*"
	All non-detects, qualify with "**"
One DMC (or two DMC of different fractions), between 10% recovery and lower recovery limit	No action required
Two or more DMC of the same acid or base/neutral fraction between 10% recovery and lower recovery limit	All detects of same fraction (Acid or Base/Neutral), qualify with "*"
	All non-detects, qualify with "**"
Two or more DMC of the same acid or base/neutral fraction above the upper recovery limit	All detects of same fraction (Acid or Base/Neutral), qualify with "*"
	All non-detects, qualify with "**"
One DMC above the upper recovery limit	No action

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met.

PAH analysis by Method 8100 (GC/FID) requires only that one surrogate be used and does not specify which surrogate is to be used. 2-fluorobiphenyl and 1-fluoronaphthalene are the recommended surrogate compounds, but the choice is open to the laboratory performing the analysis, provided adequate chromatographic separations can be demonstrated. PAH analysis by Method 8310 (HPLC) has similar recommendations and requirements. The recommended (but not required) surrogate is decafluorobiphenyl for this method.

For DRO analysis, surrogates are not required by the method. If used, the method requires that the surrogates must not elute within the diesel range organics (DRO) window. Surrogates recommended by the method are nonane (C_9) and nonacosane (C_{29}). Use professional judgment and the above table as guidance for evaluating surrogates in DRO samples.

IV. Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. Per method (laboratories may have developed other acceptable QC measures), LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix
- One LCS every 20 samples of the same matrix (WI DRO methods require an additional LCSD analysis every 20 samples)

Laboratory control samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 10) and the percent recoveries are evaluated based either on the laboratory's internally generated acceptance windows or default method criteria (as presented in the following table).

Table 5 – Guidelines for Evaluating Laboratory Control Sample Recoveries

Analysis	Matrix	Acceptance Criteria	Recommended Action
SVOC and associated analyses	aqueous/ sediment/ soil	no guidance from EPA, use laboratory acceptance criteria or professional judgment (generally, 75-125% recovery is acceptable)	if LCS > upper limit & samples are non-detect, no action; if detections, qualify with "*"
			if LCS < lower limit, qualify samples with "*****"
			if LCS << lower limit, qualify detects with "*****" qualify non-detects with "******"
DRO	aqueous	75-115% recovery <20% RPD	if LCS > 115% & samples are non-detect, no action; if detections, qualify with "*"
			if LCS < 75%, qualify samples with "*****"
	soil/sediment	70-120% recovery <20%RPD	if LCS > 120% & samples are non-detect, no action; if detections, qualify with "*****"
			if LCS < 70%, qualify samples with "*****"

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met.

RPDs are calculated for LCS/LCSD pairs using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are also used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs <20-30% for aqueous samples and <30-40% for soil and sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample is detected and much greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results based upon field duplicate RPDs.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix may have on the sample preparation procedures and analytical results. Matrix spike and matrix spike duplicate samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 10).

Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples (does not apply to WI DRO)
- 1 per preparation batch per matrix, or
- 1 per sample delivery group

However, the frequency may be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.). In some cases, MS/MSD analysis is not required.

If a matrix spike does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (greater than four times (>4x)), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery provided in the *Equations* section in the beginning of this SOP.

If laboratory acceptance criteria are provided for the percent recoveries of matrix spikes, those criteria should be used for the evaluation of the data. If acceptance criteria are not provided by the laboratory, the percent recoveries recommended by the Guidelines are presented in *Table 9* in **Section IX** can be used for guidance.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be limited by the sampling precision and inherent sample homogeneity.

In general, matrix spikes and matrix spike duplicates may be evaluated as follows:

Table 6 – Guidance for Matrix Spike Evaluation

Spike Result	Recommended Action
% recovery > upper acceptance limit	Non-detects, no qualification
	Detects, qualify with "*****"
% recovery meets acceptance limits	No qualification
% recovery is between 20% and lower acceptance limit	Non-detects, qualify with "*****"
	Detects, qualify with "*****"
% recovery is below 20%	Non-detects, use professional judgment; consider qualifying with "******"
	Detects, qualify with "*****"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS/MSD pairs using the equation for RPD evaluation provided in the *Equations* section in the beginning of this SOP. Generally, acceptance criteria for MS/MSD is <20-30% RPD for aqueous samples and <30-40% for soils/sediments, but professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

IX. Additional Tables

The following tables should be used for guidance purposes *only* for samples being analyzed for SVOCs. Priority should be given to laboratory limits and associated target compounds when provided by the laboratory. Use professional judgment before applying any of the data in the following tables to the validation process.

Table 7 – Recommended Guidance for DMC/Surrogate Recovery		
DMC	**Recovery limits (%) for aqueous samples**	**Recovery limits (%) for soil/sediment samples**
2,4-Dichlorophenol-d_3	37-105	23-104
2-Chlorophenol-d_4	41-106	13-101
2-Nitrophenol-d_4	40-108	16-104
4-6-Dinitro-2-methylphenol-d_2	22-104	1-121
4-Chloroaniline-d_4	1-145	1-145
4-Methylphenol-d_8	25-111	8-100
4-Nitrophenol-d_4	33-116	16-166
Acenaphthylene-d_8	41-107	20-97
Anthracene-d_{10}	44-110	22-98
Benzo(a)pyrene-d_{12}	32-121	43-111
Bis-(2-chloroethyl) ether-d_8	40-105	12-98
Dimethylphthalate-d_6	47-114	43-111
Fluorene-d_{10}	42-111	40-108
Nitrobenzene-d_5	43-108	16-103
Phenol-d_5	39-106	17-103
Pyrene-d_{10}	52-119	51-120
Fluoranthene-d_{10} **(SIM)**	50-150	50-150
2-Methylnaphthalene-d_{10} **(SIM)**	50-150	50-150

Table 8 – DMC and Associated Target Compounds		
DMC (alphabetical)	**Associated Target Compounds**	
2,4-Dichlorophenol-d_3	2,3-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol	2,3,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol 2,3,4,6-Tetrachlorophenol
2-Chlorophenol-d_4	2-Chlorophenol	
2-Nitrophenol-d_4	Isophorone	2-Nitrophenol
4-6-Dinitro-2-methylphenol-d_2	4,6-Ditritro-2-methylphenol	
4-Chloroaniline-d_4	4-Chloroaniline Hexachlorocyclopentadiene	3,3'-Dichlorobenzidine
4-Methylphenol-d_8	2-Methylphenol 4-Methylphenol	2,4-Dimethylphenol
4-Nitrophenol-d_4	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol	4-Nitrophenol 4-Nitroaniline
Acenaphthylene-d_8	Naphthalene 2-Methylnaphthalene 2-Chloronapthalene	Acenaphthylene Acenaphthene
Anthracene-d_{10}	Hexachlorobenzene Atrazine	Phenanthrene Anthracene
Benzo(a)pyrene-d_{12}	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
Bis-(2-chloroethyl) ether-d_8	Bis-(2-chloroethyl) ether 2,2'-oxybis(1-chloropropane)	bis(2-Choloethoxy) methane
Dimethylphthalate-d_6	Caprolactum 1,1'-Biphenyl Dimethylphthalate Diethylphthalate	Di-n-butylphthalate Butylbenzylphthalate bis(2-ethylhexyl)phthalate Di-n-octylphthalate
Fluorene-d_{10}	Dibenzofuran Fluorene 4-Chlorophenyl-phenylether	4-Bromophenyl-phenylether Carbazole

Table 8 – DMC and Associated Target Compounds (Continued)		
DMC	**Associated Target Compounds**	
Nitrobenzene-d_5	Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene	2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosdiphenylamine
Phenol-d_5	Benzaldehyde	Phenol
Pyrene-d_{10}	Fluoranthrene Pyrene	Benzo(a)anthracene Chrysene
SIM DMC and Associated Target Compounds		
Fluoranthene-d_{10}	Fluoranthene Pyrene Benzo(a)antheacene Chrysene Benzo(b)fluoranthene	Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
2-Methylnaphthalene-d_{10}	Naphthalene 2-Methylnaphthalene Ancenaphthylene Acenaphthene	Fluorene Pentachlorophenol Phenanthrene Anthracene

Table 9 – Recommended MS/MSD Recoveries and RPD				
Compound	**%Recovery for Water Samples**	**RPD for Water Samples**	**%Recovery for Soil/Sediment Samples**	**RPD for Soil/Sediment Samples**
2,4-Dinitrotoluene	24 – 96	0 – 38	28 – 89	0 – 47
2-Cholorphenol	27 – 123	0 – 40	25 – 102	0 - 50
4-Chloro-3-methylphenol	23 - 97	0 – 42	26 – 103	0 – 33
4-Nitrophenol	10 – 80	0 – 50	11 – 114	0 – 50
Acenaphthene	46 – 118	0 – 31	31 – 137	0 – 19
N-Nitroso-di-n-propylamine	41 – 116	0 – 38	41 – 126	0 – 38
Pentachlorophenol	9 – 103	0 – 50	17 – 109	0 – 47
Phenol	12 - 110	0 - 42	26 - 90	0 - 35
Pyrene	26 – 127	0 - 31	35 – 142	0 – 36

Table 10 – Number of Target Compounds Required by NELAP and MN Rules for LCS/LCSD and MS/MSD samples	
Number of Target Parameters	**Required Number of Spiked Compounds**
1-10 analytes	Spike *all* compounds
11-20 analytes	*At least* 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike *at least* 16 compounds

X. Attachments

Attachment 1: Routine Level Quality Control Report
Attachment 2: Barr Qualifiers/Footnotes
Attachment 3: Revisions to SOP

Attachment 1
Routine Level Quality Control Report

Barr Project # _____		***Project Name:*** _____			
Laboratory: _____		***Sample ID Event or COC#*** _____			
Lab Report # _____		***Matrix:***	***Soil*** _____	***Required Analysis:***	***VOC*** _____
Report Date: _____			***Water*** _____		***SVOC*** _____
Review By: _____	***Date:*** _____		***Air*** _____		***Metal*** _____
			Other _____		***GenChem*** _____
		Holding Times Met: ☐ ***Yes*** ☐ ***No***			
		Comments:			

Accuracy Data:	MS/MSD % Recovery Yes / No Sample ID_____	LCS/LCSD % Recovery
VOC		
SVOC		
Metals		
Other		

Precision Data:	MS/MSD RPDs, % Yes / No Sample ID_____	LCS/LCSD RPDs, %
VOC		
SVOC		
Metals		
Other		

Surrogate Standards Data		
Organics:		Inorganic Sample Dups:
VOC		Frequency: ____
SVOC		Results:

Attachment 1
Routine Level Quality Control Report

Blank Data:	Field Blank	Trip Blank (VOC Only)	Laboratory/Method Blank
VOC			
SVOC			
Metals			
Other			

Completeness Check: 100% Yes / No Comments:	**Historical Comparison:** N/A ____ Comments:

Masked/Blind Duplicate Results: **N/A**____ **Sample**_____

	Native Result	Duplicate Result	Native Result	Duplicate Result
VOC				
SVOC				
Metals				
Other				

Qualifiers/Qualifier Summary: **Yes / No** **(Note any TB, FB and MB affected)**

Sample Parameter	Add Qualifier	Remove Qualifier	Retain Qualifier

Attachment 1
Routine Level Quality Control Report

Other Actions Taken: Revised Report Requested _____	Lab Exception Report Completed: _____
Summary:	

Attachment 2
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes

--	Not analyzed/not available.
DLND	Not detected, detection limit not determined.
ND	Not detected.
AT	Sample chromatogram is noted to be atypical of a petroleum product.
a	Estimated value, calculated using some or all values that are estimates.
B	The reported value is less that the Contract Required Detection Limit (CRDL) but greater than or equal to the Instrument Detection Limit (IDL).
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
J	Associated value is an estimate.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
U	Not detected.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.

Attachment 3
Revisions to PCDOCS No.: 248818

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		IX	Added Table 10
		Attachments	Added Attachment 3

STANDARD OPERATING PROCEDURE

for Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO) and Total Petroleum Hydrocarbons (TPH) Data Validation

PCDOCS No.: 248819

Revision 3.1

March 16, 2009

Approved By:

Michael Dupay		03-16-09
Print QA Manager(s)	Signature	Date

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Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for Routine Level Volatile Organic Compounds (VOC) Gasoline Range Organics (GRO) and Total Petroleum Hydrocarbons (TPH) Data Validation

Purpose

This SOP is intended as a guidance SOP for the routine level validation of volatile organic compounds (VOC) data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine VOC (including BTEX and TPH) and gasoline range organics (GRO) data validation by the analytical methods including, but not limited to:

- GC/MS and GC/MS SIM (EPA Method 8260B)
- GC/PID or GC/ECD (EPA Method 8021B)
- Wisconsin (WI) GRO (EPA Method 8015C)
- TCLP VOCs (EPA Methods 1311/8260B)

Validation of Contract Laboratory Program (CLP) data should be performed in accordance with to the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008)*.

Definitions

Blank. An analytical sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

BTEX. An acronym that stands for Benzene, Toluene, Ethylbenzene, and Xylenes.

Contamination. A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

Deuterated Monitoring Compounds (DMCs). Compounds added to every volatile calibration standard, blank, and sample used to evaluate the efficiency of the extraction/purge and trap procedures, and the performance of the Gas Chromatograph/Mass Spectrometer (GC/MS) systems. DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are not expected to be naturally detected in the environmental media.

Field Blank. A blank used to provide information about contaminants that may be introduced during sample collection.

GC/ECD. Gas Chromatography/Electron Capture Detector. Measures electron capturing compounds (usually halogenated) by creating an electrical field in which molecules exiting a GC column can be detected by the drop in current in the field. Identifies potential compounds based on retention time in a GC column, but the identification of the compound is not selective with one

detector. Additional conformational analyses must be performed (either GC/MS or a separate GC/ECD with a different stationary phase on the column).

GC/FID. Gas Chromatography/Flame Ionization Detector. As components elute from the GC's column they pass through the flame and are burned, producing ions. The ions propagate an electric current, which is the signal output of the detector. The greater the concentration of the component, the more ions are produced, and the greater the current.

GC/MS. Gas Chromatography/Mass Spectrometry (sometimes Mass Selective Detector (MSD)). An analytical technique used to measure the mass-to-charge ratio of ions. A MS detector can selectively identify a compound based on the compound's mass-to-charge ratio and generally does not require secondary confirmation.

GC/PID. Gas Chromatography/Photoionization Detector. Uses ultraviolet light to ionize analyte exiting from a GC column. The resultant electrical charge produces a measurable current on a detector.

GRO. Gasoline Range Organics. Light-range petroleum products, including gasoline, with petroleum hydrocarbon compounds corresponding to an alkane range from the beginning of n-hexane (C_6) to beginning of n-decane (C_{10}) and with a boiling point range between approximately 60 - 170 degrees Centigrade.

HCl. Hydrochloric acid.

Initial Calibration. Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument.

Internal Standards. Compounds added to every volatile calibration standard, blank, sample, or sample extract, including the Laboratory Control Sample (LCS), at a known concentration, prior to analysis. Internal standards are used to monitor instrument performance and quantitation of target compounds.

Instrument Blank. A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Laboratory Control Sample (LCS). The LCS is an internal laboratory Quality Control (QC) sample designed to assess the capability of the laboratory to perform the analytical method.

Matrix. The predominant material of which the sample to be analyzed is composed.

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS). Aliquot of the sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate (MSD). A second aliquot of the same as the MS sample that is fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine precision of the method.

MeOH. Methanol.

Method Blank. A reagent aqueous sample spiked with internal standards, and surrogate standards (or DMCs), that is carried throughout the entire analytical procedure. The method blank

is used to define the level of contamination associated with the processing and analysis of samples.

MTBE. Methyl-Tertiary-Butyl-Ether. A gasoline additive, intended to reduce air pollution, that has sometimes contaminated groundwater through releases from leaking underground fuel storage tanks.

Narrative. The portion of the data package which includes laboratory, contact person, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

$Na_2S_2O_4$. Sodium Hydrosulfite. A chemical used to preserve aqueous VOC samples if residual chlorine is present.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Retention Time (RT). The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT Window established for that compound. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

SIM. Selected Ion Monitoring. Method of GC/MS scanning that focuses on only a few ions for detection. Using this technique increases the sensitivity of a mass spectrometry detector as much as 500-fold.

Surrogates (Surrogate Standard). Compounds added to every blank, sample, including Laboratory Control Sample (LCS/LCSD), Matrix Spike/Matrix Spike Duplicate (MS/MSD), and standards; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

TCLP. Toxicity Characteristic Leaching Procedure. A test designed to determine whether a waste is hazardous or requires treatment to become less hazardous; also can be used to monitor treatment techniques for effectiveness.

TPH. Total Petroleum Hydrocarbons. A measure of the concentration or mass of petroleum hydrocarbon constituents present in a given amount of soil or water. The term "total" is a misnomer--few, if any, of the procedures for quantifying hydrocarbons are capable of measuring all fractions of petroleum hydrocarbons present in the sample. Volatile hydrocarbons are usually lost in the process and not quantified, and some non-petroleum hydrocarbons are sometimes included in the analysis.

Trip Blank. A blank used to provide information about contaminants that may be introduced during sample transport.

Volatile Organic Compounds (VOC). Organic chemical compounds that have high enough vapor pressures under normal conditions to significantly vaporize and enter the atmosphere.

Equations

For % Recovery (%R or %Rec):

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where,

%R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample
In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (January 2005)*, and quality control recommendations outlined in:

- **Minnesota Rules 4740.2020 – 4740.2120** – *State of Minnesota Rules,* October 2006,
- **SW-140 –** *Wisconsin GRO (WI GRO),* September 1995,
- **EPA Method 8260B** – *"Volatile Organic Compounds by GC/MS",* December 1996,
- **EPA Method 8015C** – *"Nonhalogenated Organics Using GC/FID"*, February 2007,
- **EPA Method 8021B** – *"Aromatic and Halogenated Volatiles by GC using PID and/or ECD",* December 1996, and
- **EPA Method 1311** – *"Toxicity Characteristic Leaching Procedure"* July 1992.

Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the data in accordance with this SOP, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

The recommended hold time and preservation acceptance criteria are in *Table 1*.

Table 1 – Recommended Hold Times and Preservation

Compound	Matrix	Temperature	Preservative	Maximum Hold Time
VOC (including BTEX and MTBE)	aqueous	< 6° C	HCl <2 pH	14 days
	aqueous	< 6° C	unpreserved	7 days
	sediment/soil	< 6° C	25 mL MeOH in jar pre-weighed by laboratory	14 days
WI GRO	aqueous	< 6° C	HCl <2 pH	14 days
	sediment/soil	< 6° C	25 mL MeOH in jar pre-weighed by laboratory	14 days
TPH	aqueous	< 6° C	HCl or H_2SO_4 <2 pH	7 day extraction/ addl. 40 days analysis
	sediment/soil	< 6° C	not required	14 days extraction/ addl.40 days analysis
TCLP	all matrices	< 6° C	no preservative	14 days extraction/ addl. 14 days analysis

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil,) may not be necessarily be subjected to the same hold time recommendations.

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before receipt by the laboratory. While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

Special considerations for Holding Times of VOC samples

Aqueous samples should be received without headspace and soil samples typically require 25 grams of soil to 25 mL methanol (other volumes may be used, but the ratio of grams of soil to mL of methanol should be 1:1). Some headspace may be self-evolving in aqueous samples at sites with characteristically high pH levels and this should be considered before qualification of the results.

Aqueous samples with residual chlorine present should additionally have a 10% $Na_2S_2O_4$ solution added in addition to the HCl preservative to dechlorinate the sample. Samples with residual chlorine might warrant qualification with an "**h**" if not preserved correctly.

A separate sample (without preservative) should be collected for each soil sample to be analyzed for VOC, BTEX or WI GRO for the determination of moisture content. Samples without moisture content determination should be reported as wet weight.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group, or each batch digested (whichever is more frequent). The laboratory should analyze a method blanks at least once every 12 hours.

- Field blank collection and analysis frequency is project-specific.

- Trip blanks should be placed in each transport cooler containing VOC sample containers prior to shipment into the field and remain with the associated VOC samples submitted to the laboratory for VOC analysis; including sample storage through analysis.

Table 2 – Guidance for the Evaluation of Blank Contamination		
Positive Detection in Blank	**Sample Result**	**Recommended Action**
Common laboratory contaminants (e.g. methylene chloride, acetone, toluene, 2-butanone (MEK), carbon disulfide, and cyclohexane)	Non-detect	No action required
	<10x blank concentration	Qualify with "**b**"
	>10x blank concentration	Use professional judgment
All other target parameters	Non-detect	No action required
	<5x blank concentration	Qualify with "**b**"
	>5x blank concentration	Use professional judgment
Gross contamination (e.g. saturated peaks of target analytes in blanks)	All detections	Qualify with "******"

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Deuterated Monitoring Compounds (DMC aka Surrogates)

Deuterated Monitoring Compounds (DMC) are also frequently known as System Monitoring Compounds (SMC) or Surrogates. Keep in mind that the laboratory may have different limits and compounds than those recommended. *Table 6* in **Section IX** presents the *recommended* compounds and recovery limits (per Guidelines) for DMCs used by laboratories (VOCs only). Laboratory-assigned limits should be used where provided.

All samples (blanks, spiked samples, project samples, QC samples) should contain DMCs or surrogates. If a sample does not contain DMCs or surrogates, professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of DMCs or surrogates may not be applicable if dilution of the sample was required.

Table 3 – Guidance for the Recovery of Deuterated Monitoring Compounds		
Sample Concentration	**DMC or surrogate recovery**	**Recommended Action**
Sample is non-detect or has concentrations of associated target compounds less than reporting limit (RL)	< 10% recovery	Qualify associated target compounds with "******"
	< lower recovery limit	Qualify with associated target compounds with "*****"
	within acceptance limits	No action
	> upper recovery limits	No action
Sample has detectable concentrations of associated target compounds above reporting limit (RL)	< 10% recovery	Qualify with associated target compounds with "*****"
	< lower recovery limit	Qualify with associated target compounds with "*****"
	within acceptance limits	No action
	> upper recovery limits	Qualify with associated target compounds with "*****"

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met;

In instances where the DMC or surrogate recoveries are marginally outside acceptance criteria (within 10% of recovery limits), additional consideration should be given to historical results (where available) and general recovery trends in related samples of the same report before qualifying the sample.

In all cases, only the target compound(s) associated with the DMC that fails to meet acceptance criteria may require qualification.

Table 7 in **Section IX** presents the recommended DMCs with their associated target compounds. Bear in mind that laboratory methods may deviate from the recommended guidance and surrogate failures should be evaluated based on laboratory SOPs, especially in regard to which DMCs (surrogates) are associated with which target compounds.

For WI GRO analysis, surrogates are not required by the method. If used, the method requires that the surrogates must not elute within the gasoline range organics (GRO) window. Surrogates recommended by the method are nonane (C_9) and nonacosane (C_{29}). Use professional judgment and the above table as guidance for evaluating surrogates in WI GRO samples.

IV. Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. Per method (laboratories may have developed other acceptable QC measures), LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix
- One LCS/LCSD pair every 20 samples of the same matrix for WI GRO analysis

Laboratory control samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 9) and the percent recoveries are evaluated based either on the laboratory's internally generated acceptance windows or default method criteria (as given below).

Table 4 – Guidelines for Evaluating Laboratory Control Sample Recoveries

Analysis	Matrix	Acceptance Criteria	Recommended Action
VOC and associated analyses	aqueous/ sediment/ soil	no guidance from EPA, use laboratory acceptance criteria or professional judgment (generally, 75-125% recovery is acceptable)	if LCS > upper limit & samples are non-detect, no action; if detections, qualify with "*"
			if LCS < lower limit, qualify samples with "***"**
			if LCS << lower limit, qualify detects with "***"** qualify non-detects with "****"**
GRO	aqueous	75-115% recovery <20% RPD	if LCS > 115% & samples are non-detect, no action; if detections, qualify with "*"
			if LCS < 75%, qualify samples with "***"**
	soil/sediment	70-120% recovery <20%RPD	if LCS > 120% & samples are non-detect, no action; if detections, qualify with "***"**
			if LCS < 70%, qualify samples with "***"**

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met.

RPDs are calculated for LCS/LCSD pairs using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are also used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs between 20-30% for aqueous samples and 30-40% for soil and sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample is detected and greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results based upon field duplicate RPDs.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spike and matrix spike duplicate samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 9).

Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix, or
- 1 per sample delivery group

However, the frequency may be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.). In some cases, MS/MSD analysis is not required.

If a matrix spike does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (greater than four times (>4x)), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery provided in the *Equations* section in the beginning of this SOP.

If laboratory acceptance criteria are provided for the percent recoveries of matrix spikes, those criteria should be used for the evaluation of the data. If acceptance criteria are not provided by the laboratory, the percent recoveries recommended by the Guidelines are presented in *Table 8* in **Section IX** may be used for guidance.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity.

In general, matrix spikes and matrix spike duplicates may be evaluated as follows:

Table 5 – Guidance for Matrix Spike Evaluation

Spike Result	Recommended Action
% recovery > upper acceptance limit	Non-detects, no qualification
	Detects, qualify with "*****"
% recovery meets acceptance limits	No qualification
% recovery is between 20% and lower acceptance limit	Non-detects, qualify with "*****"
	Detects, qualify with "*****"
% recovery is below 20%	Non-detects, use professional judgment; consider qualifying with "******"
	Detects, qualify with "*****"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation for RPD evaluation provided in the *Equations* section in the beginning of this SOP. Generally, acceptance criteria for MS/MSD is <20-30% RPD for aqueous samples and <30-40% for soils/sediments, but professional judgment should be used for difficult matrices and the acceptance criteria should be adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

IX. Additional Tables

The following tables should be used for guidance purposes *only*. Priority should be given to laboratory limits and associated target compounds when provided by the laboratory. Use professional judgment before applying any of the data in the following tables to the validation process.

Table 6 – Recommended Guidance for DMC/Surrogate Recovery (alphabetical)

DMC	Recovery limits (%) for aqueous samples	Recovery limits (%) for soil samples
1,1,2,2-Tetrachloroethane-d_2	73-125	56-161
1,1-Dichloroethane-d_2	55-104	45-132
1,2-Dichlorobenzene-d_4	80-131	70-131
1,2-Dichloroethane-d_4	78-129	79-122
1,2-Dicloropropane-d_6	79-124	74-124
1,4-Dioxane-d_8	50-150	50-150
2-Butanone-d_5	49-155	20-182
2-Hexanon-d_5	28-135	17-184
Benzene-d_6	77-124	80-121
Chloroethane-d_5	71-131	61-130
Chloroform-d	78-121	72-123
Toluene-d_8	77-121	78-121
trans-1,3-Dichloropropene-d_4	73-121	72-130
Vinyl Chloride-d_3	65-131	68-122

Table 7 – Target Compounds Associated with DMCs (alphabetical)		
DMC	**Associated Target Compounds**	
1,1,2,2-Tetrachloroethane-d_2	1,1,2,2-Tetrachloroethane	1,2-Dibromo-3-chloropropane
1,1-Dichloroethane-d_2	trans-1,2-Dichloroethene 1,1-Dichloroethene	cis-1,2-Dichloroethene
1,2-Dichlorobenzene-d_4	Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene	1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene
1,2-Dichloroethane-d_4	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride	Methyl-tert-butyl ether 1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
1,2-Dicloropropane-d_6	Cyclohexane Methylcyclohexane	1,2-Dichloropropane Bromodichloromethane
1,4-Dioxane-d_8	1,4-Dioxane	
2-Butanone-d_5	Acetone	2-Butanone
2-Hexanon-d_5	4-Methyl-2-pentanone	2-Hexanone
Benzene-d_6	Benzene	
Chloroethane-d_5	Dichlorodifluoromethane Chloromethane Bromomethane	Chloroethane Carbon disulfide
Chloroform-d	1,1-Dichloroethane Bromochloromethane Chloroform	Dibromochloromethane Bromoform
Toluene-d_8	Trichloroethene Toluene Tetrachloroethene Ethylbenzene	o-Xylene m,p-Xylene Styrene Isopropylbenzene
trans-1,3-Dichloropropene-d_4	cis-1,3-Dichloropropene trans-1,3-Dichloropropene	1,1,2-Trichloroethane
Vinyl Chloride-d_3	Vinyl chloride	

Table 8 – EPA-recommended MS/MSD limits for VOCs				
Compound	**% Rec., Aqueous**	**% RPD, Aqueous**	**% Rec., Soil/Sediment**	**% RPD, Soil/Sediment**
1,1-Dichloroethane	61-145	< 14	59-172	< 22
Trichloroethene	71-120	< 14	62-137	< 24
Benzene	76-127	< 11	66-142	< 21
Toluene	76-125	< 13	59-139	< 21
Chlorobenzene	75-130	< 13	60-133	< 21

Table 9 – Number of Target Compounds Required by NELAP and MN Rules for LCS/LCSD and MS/MSD samples	
Number of Target Parameters	**Required Number of Spiked Compounds**
1-10 analytes	Spike *all* compounds
11-20 analytes	*At least* 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike *at least* 16 compounds

X. Attachments

Attachment 1: Routine Level Quality Control Report
Attachment 2: Barr Qualifiers/Footnotes
Attachment 3: Revisions to SOP

Attachment 1
Routine Level Quality Control Report

Barr Project # _____		***Project Name:*** _____			
Laboratory: _____		***Sample ID Event or COC#*** _____			
Lab Report # _____		***Matrix:***	***Soil*** _____	***Required Analysis:***	***VOC*** _____
Report Date: _____			***Water*** _____		***SVOC*** _____
Review By: _____	***Date:*** _____		***Air*** _____		***Metal*** _____
			Other _____		***GenChem*** _____
		Holding Times Met: ☐ ***Yes*** ☐ ***No***			
		Comments:			

Accuracy Data:	MS/MSD % Recovery Yes / No Sample ID_____	LCS/LCSD % Recovery
VOC		
SVOC		
Metals		
Other		

Precision Data:	MS/MSD RPDs, % Yes / No Sample ID_____	LCS/LCSD RPDs, %
VOC		
SVOC		
Metals		
Other		

Surrogate Standards Data		
Organics:		Inorganic Sample Dups:
VOC		Frequency: ____
SVOC		Results:

Blank Data:	Field Blank	Trip Blank (VOC Only)	Laboratory/Method Blank
VOC			
SVOC			
Metals			
Other			

Attachment 1 (continued)
Routine Level Quality Control Report

Completeness Check: 100% Yes / No Comments:	**Historical Comparison:** N/A ____ Comments:

Masked/Blind Duplicate Results: **N/A**____ **Sample**_____

	Native Result	Duplicate Result	Native Result	Duplicate Result
VOC				
SVOC				
Metals				
Other				

Qualifiers/Qualifier Summary: **Yes / No** **(Note any TB, FB and MB affected)**

Sample Parameter	Add Qualifier	Remove Qualifier	Retain Qualifier

Other Actions Taken: Revised Report Requested _____	Lab Exception Report Completed: _____

Summary:

Attachment 2
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes

--	Not analyzed/not available.
DLND	Not detected, detection limit not determined.
ND	Not detected.
AT	Sample chromatogram is noted to be atypical of a petroleum product.
a	Estimated value, calculated using some or all values that are estimates.
B	The reported value is less that the Contract Required Detection Limit (CRDL) but greater than or equal to the Instrument Detection Limit (IDL).
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
J	Associated value is an estimate.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
U	Not detected.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.

Attachment 3:
Revisions to PCDOCS No.: 248819

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections;
		IX	Added Table 9
		Attachments	Added Attachment 3

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