



October 22, 2004

RRD OPERATIONAL MEMORANDUM NO. 2

**SUBJECT: SAMPLING AND ANALYSIS - ATTACHMENT 7
LOW LEVEL MERCURY SAMPLING SPECIFICATIONS**

Key definitions for terms used in this document:

NREPA:	The Natural Resources and Environmental Protection Act, 1994 PA 451, as amended
Part 201:	Part 201, Environmental Remediation, of NREPA
Part 211:	Part 211, Underground Storage Tank Regulations, of NREPA
Part 213:	Part 213, Leaking Underground Storage Tanks, of NREPA
MDEQ:	Michigan Department of Environmental Quality
RRD:	Remediation and Redevelopment Division
U.S. EPA:	United States Environmental Protection Agency
Criteria or criterion:	Includes the cleanup criteria for Part 201 and the Risk-based Screening Levels as defined in Part 213 and R 299.5706a(4)
Facility:	Includes "facility" as defined by Part 201 and "site" as defined by Part 213

PURPOSE

This attachment to RRD Operational Memorandum No. 2 provides guidance for the collection of groundwater samples from monitoring wells for analysis using U.S. EPA Method 1631, Revision B; Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, U.S. EPA, Office of Water, EPA-821-R-99-005, May 1999, to evaluate mercury concentrations in groundwater venting to surface water and determine compliance with the groundwater to surface water interface (GSI) criterion. The GSI criterion is based on "total" mercury, i.e., all forms of mercury existing in the groundwater. This includes both inorganic and organic types, dissolved or attached to particulate present in the groundwater.

This attachment is applicable to site investigation and response activities under Part 201, and Part 213 of NREPA.

SUMMARY

The U.S. EPA Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Criteria Levels, July 1996, U.S. EPA, Office of Water, Engineering and Analysis Division, Washington D.C., was used as a reference to develop this attachment. The two-person team approach, as described in Method 1669, "Dirty Hands," and "Clean Hands" sampling was adopted, and quality assurance and control requirements of that method have been incorporated.

Modifications of this method, and other methods, may be proposed and used if found adequate by the MDEQ to produce reliable results for sampling groundwaters for low level mercury. The presentations of information that validate the use of other methods or modifications of this method are the responsibility of the parties proposing their use. This attachment is not intended to be used in place of Method 1669 when the use of that method is required.



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The following documents are rescinded with the issuance of this attachment:

- Environmental Response Division Procedure, "Groundwater Sampling from Monitoring Wells for Low Level Analysis of Mercury" dated April 13, 2001.

APPENDAGE:

Low Level Mercury Sampling and Analysis Specification

This memorandum and its attachment are intended to provide direction and guidance to foster consistent application of Part 201, Part 211, and Part 213 and the associated administrative rules. This document is not intended to convey any rights to any parties or create any duties or responsibilities under the law. This document and matters addressed herein are subject to revision.

LOW LEVEL MERCURY SAMPLING AND ANALYSIS SPECIFICATIONS

Summary

Sampling equipment, materials, and containers are cleaned using high purity chemicals and double bagged for protection from contamination during storage and transportation. Highly purified reagent water is provided to the field personnel for the decontamination of the equipment and collection of field blanks. High purity, diluted, hydrochloric acid (HCl) is also provided to field staff for preservation of the sample.

A two-person team, as described in Method 1669, is used for sample collection. One member of the two-person sampling team is designated as "Dirty Hands," and the second member is designated as "Clean Hands." The individual designated as "Clean Hands" will handle all operations involving contact with the sample bottle and transfer of the samples from the sample collection device to the sample bottle. "Dirty Hands" is responsible for the preparation of the sampler (except the sample container itself), operation of any machinery, and for all other activities that do not involve direct contact with the sample. Sampling teams wear clean non-talc gloves as well as clean, lint-free, outer clothing to protect samples from contamination by lint and dust.

Special precautions are incorporated to minimize contamination. When possible the facility history and results showing previous results of mercury levels at specific locations are used to design the collection process, in order to minimize the chances of cross contamination. Where decontamination of the equipment is required, equipment blanks are taken before each sample. Sample collection is performed by a strict protocol designed to minimize contamination.

Because of the likelihood of positive blanks and the affect they have upon the results, staff should carefully evaluate blank levels before making regulatory decisions. For application to regulatory requirements, it is recommended that blank mercury levels be less than one-fifth of the mercury in the associated sample. This is the guideline recommended in Method 1631.

Definitions

1. Trace Metal Grade Reagents – Reagents that make no significant contribution of mercury to the sample.
2. Dirty and Clean Hands: All operations involving contact with the sample bottle, and transfer of the samples from the sample collection device to the sample bottle, are handled by the individual designated as "Clean Hands." An individual designated as "Dirty Hands" is responsible for the preparation of the sampler (except the sample container itself), operation of any machinery, and for all other activities that do not involve direct contact with the sample.

Contamination and Interferences

The need to avoid contamination when collecting samples for extremely low level measurements cannot be over emphasized. Field collection personnel should be familiar with the potential sources of mercury contamination, and implement those steps necessary for adequate control. Field and equipment blanks are used to discover contamination problems during the collection steps.

1. Potential Sources of Mercury Contamination: These include metallic and metal-containing equipment, containers, lab ware, reagents, and de-ionized water, improperly cleaned, and stored equipment, as well as atmospheric sources such as dirt and dust, automobile

- exhaust, laboratory workers, and cigarette smoke. Well construction materials, e.g., the gravel pack and well screen, may also be a source of contamination.
2. **Potential Contamination from Well Construction Materials:** Levels of mercury in groundwater samples can be a result of natural background, well construction material, or environmental contamination. To reliably distinguish the mercury contribution of both natural background and well construction materials from environmental contamination, measurements from up-gradient background wells, constructed in the same manner as down-gradient wells, are necessary. RRD Operational Memorandum No. 4 provides guidance on establishing background.
 3. **Use of Peristaltic Pumps:** Peristaltic pumps have distinct advantages in controlling contamination, and should be used when possible. Most other pumps have metal parts that may come in contact with the sample; hence, pumps must be decontaminated. For peristaltic pumps, only the tubing is in contact with the sample: consequently, clean tubing is all that is necessary to minimize contamination.
 4. **Control:** The best way to control contamination is to minimize exposure of the sample and sampling equipment to possible sources of contamination. When possible, prior knowledge of mercury levels at sampling locations is used for planning collection activities to minimize chances of contamination from high sources, cross contamination resulting from sequentially sampling locations of high and low levels, and cross contamination during storage and transportation. Appropriate equipment and field blanks are used to discover contamination.
 5. **Filtering:** If filtering is determined necessary (see RRD Operational Memorandum No. 2, Attachment 5 for direction on filtering) it must be performed at the laboratory to prevent contamination.
 6. **Preservation:** Preservation at the laboratory is optional for samples not requiring filtering. Unpreserved samples should be sent to the laboratory overnight.

Apparatus and Materials

1. **Disposable Materials:** Disposable materials such as gloves, storage bags, and plastic wrap, may be used new without additional cleaning unless the equipment blank results identify any of these materials as a source of contamination. If new disposable materials are found to be a source of contamination, then a different supplier must be obtained or the materials must be cleaned.
2. **Sample Bottles:** Fluoropolymer (FEP, PTFE) or borosilicate glass, 125 ml to 1 L, depending upon laboratory specifications with fluoropolymer or fluoropolymer lined caps, cleaned according to Method 1669/1631 procedures, with air tight cap. Containers are filled with 0.1 percent HCl (v/v), tightly capped, double bagged in new polyethylene zip-type bags until needed, and stored in cardboard boxes until use. Sample bottles are transferred to the facility with 0.1 percent HCl, or emptied and filled with reagent water for transportation.
3. **Tubing for use with low-flow sampling pump:** Use fluoropolymer tubing in lengths as required to reach the sampling point. Tubing must be cleaned by soaking in a 5-10 percent HCl solution for 8-24 hours, rinsing with reagent water in a clean bench in a clean room, and drying in the clean bench by purging with mercury-free air or nitrogen. Tubing must be double-bagged in clear polyethylene bags, serialized with a unique number to identify it in case of contamination problems, and stored until use.
4. **Peristaltic Pump:** 115V A.C., 12V D.C. internal battery, variable-speed, single head, Cole-Palmer or equivalent, portable, "Masterflex L/S," Catalog No. H-07570-10 drive with Quick Load Pump head, Catalog No. H-07021-24, or equivalent.
 - a. **Tubing for use with peristaltic pump.** Styrene/ethylene/butylene/silicone (SEBS) resin, approximately 3/8 in. internal diameter (i.d.) by approximately 3 ft., Cole-Palmer size 18,

- Cat. No. G-06464-18, or approximately ¼ in. i.d., Cole-Palmer size 17, Catalog No. G06464-17, or equivalent. Tubing is cleaned and stored as provided above.
- b. Tubing for connection to peristaltic pump as provided above. Fluoropolymer, 3/8 or ¼ in. outside diameter (o.d.), in lengths required to reach the point of sampling. Tubing is cleaned and stored as provided above. If necessary, more aggressive cleaning (e.g., concentrated nitric acid) may be used.
5. Bladder Pump: QED¹ model MP-SP-4P.
 - a. Water Level Meter – Provided as part of the QED bladder pump equipment, QED part number MP30-150.
 - b. Controller – Provided as part of the QED bladder pump equipment, QED part number MP-15.
 - c. Bladders – QED Bladder Kit, part number 38360. Unless it is known, the bladders do not contribute to contamination, the bladders must be cleaned and stored as provided above.
 - d. Spare CO2 Tank – QED part number 38304.
 6. Water Quality Instruments: Use instruments capable of measuring temperature, hydrogen ion activity (pH), specific conductance, redox, dissolved oxygen, and turbidity to determine when formation water is entering the pump. With the equipment provided to staff, a separate meter is necessary for turbidity measurements.
 7. Gloves: Clean, non-talc polyethylene, latex, vinyl, or polyvinylchloride (PVC): various lengths.
 8. Gloves: PVC—Fisher Scientific Part No. 11-394-100B, or equivalent.
 9. Wind Suit: Suitable to protect samples from contamination from lint and dust. Unlined, long sleeve wind suit consisting of pants and jacket constructed of nylon or synthetic fiber are suitable. Tyvek® suits are used in this procedure.
 10. Storage bags: Clean, zip-type, non-vented, colorless polyethylene (various sizes). Large size bags are needed for storage of the pump during transportation between sampling locations.
 11. Plastic Wrap: Clean, colorless polyethylene.
 12. Cooler: Clean, nonmetallic, with white interior for shipping samples.
 13. Ice: Use ice to keep samples chilled during shipment. Chemical packs are less effective.
 14. Carboys: Dedicate one specific carboy for “Reagent Water.”
 15. Plastic Decontamination Tubs: Containers of various sizes to immerse the submersible pump, sampling tubing, and the wetted parts of the water level meter and multi-parameter monitor. Four tubs are needed, one for a soap solution, one for tap water rinse solution, one for reagent water rinse, and one to hold the reagent water for obtaining field blanks.
 16. Pipette: Automatic pipette, capable of dispensing 10.0 ml and automatic tip ejector.
 17. Pipette Tips: Colorless, 10 ml, for use with automatic pipette. Pipette tips must be cleaned and stored as described under tubing above.

Reagents

1. Reagent Water: Ultra pure deionized water, starting from a pre-purified (distilled, reverse osmosis, etc.) source, 18 Megaohms minimum, provided in a carboy suitable to prevent mercury contamination. The water should be tested at the laboratory for suitability for sampling. The quantity needed depends on the amount of water needed for each decontamination cycle and the number of wells sampled. The laboratory should provide this water.
2. Preservative: Hydrochloric acid (HCl), 6 N (normal) made from Trace Metal Grade acid and reagent water, and tested to contain less than 0.5 ng/L of mercury. The laboratory should provide this reagent.



3. Soap: Alconox² CITRANOX®, suitable for cleaning instruments for low level mercury sampling. Prepare a 2 percent solution as per the manufacturer's instructions.

Site Sampling Plans and Sample Delivery Strategies to Minimize Contamination

1. Sample Collection Strategy: Sample collection activities should be designed that will minimize the potential for cross contamination.
 - a. If possible, use previous facility data showing mercury levels at the locations to be sampled. If mercury data is not available, use other information to make a judgement whether the mercury level is suspected to be high or low. For example, if data is available for other metal levels, the relative levels of these metals may be a good indicator of whether high or low mercury levels are suspected.
 - b. Arrange the sampling sequence in order of their known or expected levels of mercury. Collect samples starting from locations known to have the lowest and approximate same levels of mercury, and proceed to those of higher levels. In this manner, if decontamination procedures fail to remove all residual mercury, the effect on samples will be minimized.
 - c. Group samples so that samples of high and low levels are separately grouped in storage and transportation. For purposes of separating samples based on expected concentration levels, samples believed to have concentrations more than 200 ng/L of mercury should be identified as high level samples, and low level samples less than or equal to 200 ng/L.
2. Sample Information Provided to the Laboratory: Laboratory areas and instrumentation used for low level analysis of mercury are extremely clean and designed to prevent mercury contamination from outside sources. Processing a sample with an extremely high level of mercury in these areas can result in contamination of the area and instrumentation, resulting in delays and additional expense. Using the evaluation described above, provide information to the laboratory regarding the known or expected levels of mercury for each location sampled. Information useful to the laboratory and recommended to be provided is as follows:

Mercury (Hg) Level	Provide to Laboratory
Hg levels not known and high levels expected.	Expected > 200 ng/L
Hg levels not known and low levels expected.	Expected < 200 ng/L
Hg levels previously found	Provide Data
Hg levels and expectations not known	Not Known

Sample Collection, and Handling Considerations

Sampling precautions should be taken as follows:

1. Use low-flow rates (0.5 L/min.) during both purging and sampling to maintain minimal draw-down in the well.³
2. Place the sampling pump intake at the proper sampling point.
3. Minimize disturbance of the stagnant water column above the screened interval during water level measurement and sampling device insertion.
4. Make proper adjustments to stabilize the flow rate as soon as possible.
5. Monitor water quality indicators during purging.
6. Collect unfiltered samples to represent contaminant loading and transport potential in the subsurface system.

7. Filtering (if necessary): If it is not feasible to collect samples representative of the water flowing in the aquifer, and filtering is determined necessary, (see RRD Operational Memorandum No 2 – Attachment 5 for direction on filtering), collect duplicate samples and identify one of these to be filtered and preserved upon receipt at the laboratory. Appropriate arrangements must be made with the laboratory to ensure the filtering and subsequent preservation is accomplished for identified samples immediately upon receipt. Arrangements with the laboratory to utilize appropriate filters should be made well in advance of sample collection, so that immediate filtering and preservation at the laboratory can be accomplished upon receipt of samples.
8. Water samples should not be taken immediately following well development. Sufficient time should be allowed for the groundwater flow regime in the vicinity of the monitoring well to stabilize and to approach chemical equilibrium with the well construction materials. This lag time will depend on facility conditions and methods of installation but often exceeds one week.
9. Well purging is nearly always necessary to obtain samples of water flowing through the formation associated with the screened interval. The required purging procedure relies on the stabilization of several water quality parameters to determine when formation water is being pumped. The pH, specific conductance, redox, dissolved oxygen, and turbidity are monitored for this purpose. Temperature is also measured and recorded during this process but is not used as an indicator for formation water. Data on pumping rate draw-down, not to exceed 0.1 meter, and volume required for parameter stabilization can be used as a guide for conducting subsequent sampling activities.
10. Water Level Measurements and Monitoring: Well depth should be obtained from the well logs. Since measuring to the bottom of the well casing will cause re-suspension of the settled solids and require longer purging times for turbidity equilibration, measure well depth after sampling is completed. The water level measurement should be taken from a permanent reference point, which is surveyed relative to ground elevation.

Sample Collection using Bladder Pumps

1. Upon arrival at the sample location, one member of the two-person sampling team is designated as “Dirty Hands,” and the other as “Clean Hands.”
2. An area, expected or known to be free of high levels of mercury, is selected.
3. The team removes the bags containing the pump, monitoring instruments, tubing, carbon dioxide (CO₂) cartridges, gloves, plastic wrap, and wind suits, from the coolers or storage containers in which they are packed.
4. The team puts on Wind Suits and PVC gloves.
5. The team generates the Initial Equipment Blank, following the steps listed under Decontamination and Initial Equipment Blank.
6. The team proceeds to the sampling location.
7. The team opens the well.
8. The team changes gloves.
9. Keeping both bags together, Dirty Hands opens the outer bag containing the pump.
10. Clean Hands opens the inner bag and removes the pump.
11. Clean Hands lowers the submersible sampling pump into the monitoring well. Lower the pump slowly and carefully to the middle of the screened interval or slightly above the middle. This should minimize excessive mixing of the stagnant water above the screen with water in the screened interval and minimize suspension of solids from the bottom of the well.
12. Dirty Hands opens bag containing static water level meter. Clean Hands removes water level meter. Clean Hands sets up the water level meter.

13. Clean Hands connects the multi-meter flow through cell to the pump outlet.
14. Dirty Hands turns on the submersible pump, sets the pump for the allowable water level draw-down (not to exceed 0.1 meters), and slowly pumps the water while monitoring the water level to assure that that the pumping rate does not result in draw-down of the water level. With the QED bladder pump in this standard operating procedure (SOP), the pump will turn off automatically if this level is exceeded. As the well is pumped, water quality parameters are monitored to determine when formation water is flowing through the pump. Formation water is considered to be flowing, if three consecutive measurements of the water quality parameters, conducted at 3-5 minute intervals, meet the following requirements:
 - a. Turbidity, within ± 10 percent.
 - b. pH, within ± 0.1 pH units.
 - c. Specific conductance, within 3 percent.
 - d. Redox, within ± 10 millivolts
 - e. Dissolved oxygen, within ± 10 percent. If dissolved oxygen is used for comparison to criteria or a mixing zone calculation, the dissolved oxygen calibration must be corrected for local barometric pressure and elevation. The equipment in this procedure (YSI multi-parameter meter) automatically corrects the dissolved oxygen for these conditions.
15. After stabilization, Clean Hands disconnects the meter.
16. The team changes gloves.
17. Dirty Hands retrieves the sample containers required, and unzips their outer bags. Retrieve two sample containers if filtering is required, for duplicate samples, or for field blanks. If split samples are to be generated a larger size container is required, at least twice the size of normal samples.
18. Dirty Hands prepares the label(s).
19. Clean Hands opens the inner bag, removes the sample container, and reseals the inner bag.
20. Clean Hands removes the cap for the sample being collected, and while holding the cap upside down, discards the diluted acid into a waste carboy, or empties the reagent water onto the ground.
21. If a field blank is being generated, proceed as follows:
 - a. Clean Hands opens the inner bag and places the emptied sample bottle and its cap in its inner bag. This bottle is to be identified as the field blank.
 - b. Clean Hands obtains another sample bottle from its inner bag, removes and, discards its cap.
 - c. Clean Hands retrieves the field blank bottle, and pours the contents of the sample bottle into the field blank bottle.
 - d. Skip to step 27 below.
22. Clean Hands rinses the sample bottle and cap three times with the formation water flowing from the pump, and collects the sample from the flowing tube.
23. Clean Hands caps the sample, opens the inner bag, and places the sample in its inner bag.
24. If filtering is required or a duplicate sample is to be taken, Steps 18 through 23 are repeated to immediately take another sample.
25. For samples required to be filtered or preserved at the laboratory, skip to step 27 below.
26. Preserve each sample taken as follows:
 - a. Dirty Hands opens the outer bag containing the preservative, pipette, and tips.
 - b. Clean Hands opens the inner bag, opens the preservative, retrieves the pipette, and prepares it for dispensing.

- c. Use the information included in Sample Preservation and Holding Time for the correct amount of preservative. Clean Hands pipettes the required amount of preservative into the sample container(s), ejects the pipette tip into the waste container, places the pipette back into its inner bag, recaps the preservative, and seals the inner bag.
 - d. Dirty Hands seals the outer bag for the preservative.
27. Clean Hands caps the sample(s), opens the inner bag(s) for the sample(s), places the sample bottle(s) into the inner bag(s), and seals the inner bag(s).
28. Dirty Hands seals the outer bag(s), writes sample identification information in permanent ink on the outside of the plastic bag, places the sample(s) in the cooler (on ice), and closes the cooler.
29. Dirty Hands measures and records the depth to the bottom of the well.
30. Dirty Hands records the sample number(s) in the sampling log, water quality parameters, and notes any unusual observations.
31. Clean Hands removes the equipment from the well, removes the water level meter, and places them into bags for transportation.
32. Both Dirty and Clean Hands move to the decontamination area with the equipment.
33. Decontamination Between Sampling Locations steps are used to decontaminate the equipment.
34. Generating the Equipment Blank steps are used to collect an equipment blank.
35. If other samples are to be taken at the facility, the team proceeds to the next sampling location, and collects another sample beginning with step 6 above.
36. If samples are to split, proceed as follows:
 - a. The team selects a suitable place for splitting samples.
 - b. The team changes gloves.
 - c. Dirty Hands opens the cooler, removes the bag containing the sample to be split. The volume of this sample must be at least twice the volume of normal samples.
 - d. Dirty Hands removes two bags with sample containers, and unzips their outer bags. These containers will hold the split samples.
 - e. Dirty Hands prepares the label(s).
 - f. Clean Hands opens the inner bags holding all containers, removes the containers, removes the caps of all containers and places them in their respective inner bags.
 - g. Clean Hands discards the diluted acid from the two sample containers, into a waste carboy, or empties the reagent water onto the ground.
 - h. Clean Hands pours from the container holding the sample to be split, into each of the sample containers.
 - i. Clean Hands discards the container that held the sample to be split.
 - j. Clean Hands retrieves the caps, seals the samples with their respective caps, places the samples into their inner bags, and seals the inner bags.
 - k. Dirty Hands seals the outer bag(s), writes sample identification information in permanent ink on the outside of the plastic bag, places the sample(s) in the cooler (on ice), and closes the cooler.
 - l. Equipment blanks associated with the respective samples must be provided to both parties receiving split samples.
 - m. Repeat steps for each additional split sample.
 - n. Information specific for splitting samples must be documented. If others request split samples, use the MDEQ Laboratory's chain of custody sheet. If the MDEQ is requesting the split sample, and a chain of custody is not forthcoming from the sampler, use the MDEQ chain of custody, fill out information, sign it, and request this be signed by the provider of the samples.

Sample Collection using Peristaltic Pumps

1. Upon arrival at the sample location, one member of the two-person sampling team is designated as “Dirty Hands,” and the other as “Clean Hands.”
2. The team opens the well to be sampled.
3. An area, expected or known to be free of high levels of mercury, is selected. Sampling should proceed from lowest to highest expected level of contamination.
4. The team removes the bags containing the pump, batteries, monitoring instruments, SEBS resin tubing, gloves, plastic wrap, and wind suits, from the coolers or storage containers in which they are packed.
5. The team puts on Wind Suits and PVC gloves.
6. Dirty Hands removes the pump from its storage bag and opens the bag containing SEBS resin tubing.
7. Clean Hands installs the tubing in the well. Lower the tubing slowly and carefully to the middle of the screened interval or slightly above the middle, to minimize excessive mixing of the stagnant water above the screen with water in the screened interval, and to minimize resuspension of solids from the bottom of the well.
8. Clean Hands installs tubing on the pump.
9. Dirty Hands opens bag with water level meter.
10. Clean Hands removes water level meter and lowers it into the well.
11. Clean Hands connects the multi-parameter meter flow through the cell to the pump outlet.
12. Dirty Hands turns on the peristaltic pump and slowly pumps the water while monitoring the water level to assure that that the pumping rate does not result in excessive draw-down of the water level (not to exceed 0.1 meters). As the well is pumped, water quality parameters are monitored to determine when formation water is flowing through the pump. Formation water is considered to be flowing if three consecutive measurements of the water quality parameters, conducted at 3-5 minute intervals, meet the following requirements:
 - a. Turbidity, within ± 10 percent.
 - b. pH, within ± 0.1 pH units.
 - c. Specific conductance, within 3 percent.
 - d. Redox, within ± 10 mv.
 - e. Dissolved oxygen, within ± 10 percent. If dissolved oxygen is used for comparison to criteria or a mixing zone calculation, the dissolved oxygen calibration must be corrected for local barometric pressure reading and elevation. The equipment in this procedure (YSI multi-parameter meter) automatically corrects the dissolved oxygen for these conditions.
13. After stabilization, Clean Hands disconnects the meter.
14. The team changes gloves.
15. Dirty Hands opens the cooler containing the sample bottle, and unzips the outer bag containing the sample container. If the sample is to be split, a larger size container is required at least twice the size of normal samples. If filtering is necessary, a field blank is being generated, or a duplicate sample is to be taken, Dirty Hands unzips the outer bag of another sample container.
16. Dirty Hands prepares the sample label(s).
17. Clean Hands opens the inner bag, removes the sample container, and reseals the inner bag.
18. Clean Hands unscrews the cap, and while holding the cap upside down, discards the diluted acid into a waste carboy, or empties the reagent water onto the ground.
19. If a field blank is being generated, proceed as follows:

- a. Clean Hands places the sample bottle and its cap in its bag. This is to be identified as the field blank.
 - b. Clean Hands obtains another sample bottle from its bag, unscrews and discards the cap.
 - c. Clean Hands retrieves the field blank bottle, and pours the contents of the other bottle into the field blank bottle, discards this other bottle, retrieves the cap of the field blank and caps the field blank.
 - d. Skip to step 22 below.
20. Clean Hands rinses the sample bottle and cap three times with the formation water, and collects the sample from the flowing tube.
 21. Clean Hands caps the sample.
 22. Clean Hands places a label on the sample container, and places it in its inner bag.
 23. If filtering is required, or a duplicate sample is to be taken, steps 17 through 22 are repeated to immediately take another sample.
 24. For samples required to be filtered, and samples requiring preservation at the laboratory, skip to step 26 below.
 25. Preserve sample as follows:
 - a. Dirty Hands opens the outer bag containing the preservative, pipette, and tips.
 - b. Clean Hands opens the inner bag, opens the preservative, retrieves the pipette, prepares it for dispensing, and pipettes the required amount of preservative into the sample container(s). Use the information included in Sample Preservation and Holding Time for the correct amount of preservative.
 - c. Clean Hands ejects the pipette tip into the waste container, places the pipette back into its inner bag, and seals the inner bag.
 - d. Clean Hand caps the preservative, places it in its inner bag, and seals the inner bag.
 - e. Dirty Hands seals the outer bags for the pipette and preservative.
 26. Clean Hands caps the sample(s), opens the inner bag(s) for the sample(s), places the sample bottle(s) into the inner bag(s), and seals the inner bag(s).
 27. Dirty Hands seals the outer bag(s), writes sample identification information on the outer bag, places the sample(s) in the cooler (on ice), and closes the cooler.
 28. Dirty Hands measures and records the depth to the bottom of the well.
 29. Dirty Hands records the sample number(s) in the sampling log, water quality parameters, and notes any unusual observations.
 30. Clean Hands removes the equipment from the well, removes the water level meter, and places them into bags for transportation.
 31. Both Dirty and Clean Hands move to the decontamination area with the equipment.
 32. Decontamination Between Sampling Locations steps are used to decontaminate the water level meter and multi-parameter meter. The SEBS resin tubing is replaced prior to sampling each new monitoring well.
 33. If other samples are to be collected, the team proceeds to the next sampling location, and collects another sample beginning with the step 1.
 34. If samples are to be split, follow the steps in Sample Collection Using Bladder Pumps, starting with step 36.

Decontamination and Initial Equipment Blank

1. Dirty Hands prepares the decontamination solutions.
2. Dirty Hands opens outer bag containing tubing and pump bladder.
3. Dirty Hands opens bags containing pump and water level meter.
4. Dirty Hands removes the pump.

5. Dirty Hands holds the pump while Clean Hands removes the bladder from the inner bag and places the bladder on the pump. Clean Hands removes tubing from the inner bag and installs tubing on pump and controller.
6. Dirty Hands lowers pump into tub 1 containing the soap solution.
7. Dirty Hands turns on controller and pumps three volumes of soap solution through the pump and tubing.
8. Clean Hands moves the pump to tub 2 containing tap water.
9. Dirty Hands turns on controller to pump three volumes of tap water through the pump.
10. Clean Hands moves the pump to tub 3 and pumps three volumes of reagent water.
11. Clean Hands places the pump in tub 4 containing reagent water.
12. An equipment blank is taken following steps in Generating the Equipment Blank.
13. Clean Hands removes the water level meter from its storage bag, decontaminates the water level meter by successively cleaning with solutions from tub 1, 2, and 3, and places the meter into a clean storage bag.

Decontamination Between Sampling Locations

1. The team changes gloves.
2. Dirty Hands prepares the decontamination solutions.
3. Dirty Hands lowers pump into tub 1 containing the 2 percent Alconox/tap water solution.
4. Dirty Hands turns on controller and pumps three volumes of Alconox solution through the pump.
5. Clean Hands moves the pump to tub 2 containing tap water (fresh tap water should be used between each sampling location.)
6. Dirty Hands turns on controller to pump three volumes of tap water through the pump.
7. Clean Hands moves the pump to tub 3 and pumps three volumes of reagent water (fresh reagent water should be used between each sampling location.)
8. Clean Hands changes gloves.
9. Dirty Hands opens outer bag containing tubing and pump bladder.
10. Dirty Hands changes gloves.
11. Dirty Hands removes the pump from tub 3.
12. With Dirty Hands holding the pump, Clean Hands removes the bladder from the inner bag and places the bladder on the pump. Clean Hands removes tubing from the inner bag and installs tubing on pump and controller.
13. Clean hands places pump in reagent water in tub 4.
14. The team changes gloves.
15. An equipment blank is taken following steps in Generating the Equipment Blank.
16. Clean Hands places the pump in the storage bag or proceeds to place pump in monitoring well.
17. Clean Hands removes the water level meter from its storage bag, decontaminates the water level meter by successively cleaning with solutions from tub 1, 2, and 3, and places the meter back into a clean storage bag or into the monitoring well.
18. Clean Hands changes gloves.

Generating the Equipment Blank

1. One equipment blank is generated for each location sampled.
2. With the submersible pump in tub 4 holding the fresh reagent water, Dirty Hands turns on the pump and allows several volumes of reagent water to be pumped.
3. The team changes gloves.

4. Dirty Hands opens the box or cooler containing the sample bottles, and unzips the bag containing a sample container. If a split sample is scheduled to be taken, Dirty Hands unzips another bag containing a sample container.
5. Clean Hands opens the inner bag, removes the sample container, and reseals the inner bag.
6. Dirty Hands reseals the outer bag.
7. Clean Hands unscrews the cap, and while holding the cap upside down, discards the diluted acid into a waste carboy, or empties the reagent water on the ground.
8. As reagent water is flowing through the pump, Clean Hands collects the sample by emptying the solution from the sample bottle, rinsing the sample bottle and cap three times with the flowing reagent water, and collecting the sample from the flowing tube.
9. If preservation is performed at the laboratory, skip to step 11.
10. Preserve sample(s) as follows:
 - a. Dirty Hands opens the outer bag holding the automatic pipette and preservative.
 - b. Clean Hands opens the inner bag containing the preservative and automatic pipette, opens the preservative bottle, and pipettes 10 ml of the preservative into the sample bottle.
 - c. Clean Hands recaps the preservative bottle, removes the pipette tip, and places the preservative and pipette back into its bag.
 - d. Clean Hands seals the inner bag holding the preservative and pipette.
 - e. Dirty Hands seals the outer bag.
 - f. Clean Hands opens the inner bag for the sample, places the sample bottle into the inner bag, and seals the inner bag.
11. Dirty Hands seals the outer bag, opens the sample cooler, places the equipment or field blank in the cooler (on ice), and closes the cooler.
12. Dirty Hands records the sample in the sampling log as the "Equipment Blank".
13. If the scheduled sample to be taken is a split sample, follow the steps in [Sample Collection Using Bladder Pumps](#), starting with step 36.
14. Clean Hands removes the pump from the tub, places it in a clean protective bag, and seals the bag.

Sample Preservation and Holding Time

1. Preservation: Samples are transported on ice during shipment to the laboratory. Samples are preserved in the field using 10 ml/L 6N HCl per liter of sample. If filtering and preservation is required at the laboratory, equivalent amounts of HCl per liter of sample can be used.
2. Laboratory Processing of Filtered/Preserved Samples: If filtering and preservation is to be performed at the laboratory, make arrangements with the laboratory for receipt of samples well in advance. If special filters are necessary, these must be provided to the laboratory prior to sample collection activities or arrangements made with the laboratory to ensure they are available upon sample receipt. It is not advisable to plan sampling immediately proceeding non-working days for the laboratory. Upon shipment of samples to a laboratory, it is good practice to immediately contact the laboratory. If the laboratory is not advised of these arrangements, extra effort and expense must be incurred to ensure necessary filtering and preservation.
3. Sample analysis must be performed within 28 days of sample collection.



Quality Assurance/Quality Control

Equipment Blank: The equipment blanks are used to verify the equipment is free from contamination prior to the collection of the sample. (See Decontamination and Initial Equipment Blank and Generating the Equipment Blank)

1. Frequency of Collection: Collect one initial equipment blank, and an equipment blank per monitoring well sampled.
2. Evaluation Criteria: If the mercury concentration in the blank is greater or equal to 0.5 ng/L, or greater than one-fifth of sample concentration, whichever is higher, the associated sample result is an estimate and may be unusable for regulatory application.
3. Corrections: If the initial equipment blank indicates contamination, above 0.5 ng/L, review the process used for cleaning, and have reagents replaced as appropriate.

Field Blanks: The purpose of field blanks is to assess the suitability of the container, preservative, and sample handling. The field blank is generated by simply pouring the solution provided in one of the sample containers into another sample container whose contents have been emptied at the facility. (See Sample Collection Using Bladder Pumps step 21 and Sample Collection using Peristaltic Pumps step 19)

1. Frequency of Collection: One per facility, per day, or one per sampling event, whichever is greater.
2. Evaluation: If the mercury concentration in the blank is greater or equal to 0.5 ng/L, or greater than one-fifth of sample concentration, whichever is higher, the associated sample result is an estimate and may be unusable for regulatory application.

Field Duplicates: The purpose of field duplicates is to assess the precision for the field sampling and analytical process. A field duplicate is collected by filling a second sample container, in rapid succession after the first sample, from the outlet of the sampling stream.

1. Frequency of Collection: Collect duplicates minimally for every 10 samples collected, or at the frequency specified in the project objectives. If possible, select a location with detectable amounts of mercury.

Split Samples: Split samples are used to independently confirm results of the laboratory performing the analysis. Typically a laboratory known to produce valid, unbiased results, is selected as the laboratory to which the split samples are sent.

1. Collection: Split samples are created by dividing one sample collected in the field into two aliquots. This requires the collection of at least twice the volume of sample normally collected, properly preserved if field preservation is performed. Because of the influence that equipment blanks may have upon the use of the data, an equipment blank associated with the sample should be provided along with the split sample. This will require the generation of two equipment blanks prior to the collection of the sample to be split.

Footnotes

1. QED, P.O. Box 3726, Ann Arbor, MI 48160.
2. Alconox, Inc., 30 Glenn Street, Suite 309, White Plains, NY, 10603.
3. Puls, R. W. and Barcelona, M. J. 1996 Low-Flow (Minimal Draw Down) Ground-Water Sampling Procedures, EPA Ground Water Issues, U.S. EPA Office of Research and Development, EPA/540/S-95/504.



Disclaimer

Mention of specific manufacturers and associated instruments does not constitute endorsement by the MDEQ RRD of that manufacturer and equipment.

This SOP is intended to be a performance-based method. Acceptance of results using modifications of this procedure, and using other procedures, will depend upon the demonstration of equivalent performance.