New York State Department of Environmental Conservation

Division of Water

Standard Operating Procedure:

Sample Handling, Transport, and Chain of Custody

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SOP # 101- Update Log[1]

Prepared/Revised By:	**Date**	**Approved By**	**Revision No:**	**Summary of Changes**
DOW staff	August 2003	Larry Bailey	101-02	
DOW staff	March 2011	RoseAnn Garry	101-11	
DOW staff	March 2014	Jason Fagel	101-14	
DOW staff	March 2015	RoseAnn Garry	101-15	
Liz Nystrom (USGS) Jeff Lojpersberger	May 2018 June 2018	Rose Ann Garry	3.0	Deleted Non-point source section. Deleted PISCES section Non substantive changes Amended Section 10.3 groundwater sampling equipment and procedure.
Jeff Lojpersberger	April 2019			Clarified Field blank definition. Added Distilled water and preservation method summary to Appendix Table 1
Brian Duffy, Meredith Streeter, Gavin Lemley	April 2020	Rose Ann Garry	101-20.COV-1.0	Updates for COVID-19 procedure modifications. Update to mercury preservation in Table 1.

[1] The detailed 'Update Log' for DOW SOPs was adopted in 2016. The log may not be complete for updates conducted prior to 2016.

[2] 'No substantive changes' include updating references, correcting typographical errors, and clarifying certain language to make the document more useful and effective.

1. Table of Contents

2. Scope and Applicability

This standard operating procedure (SOP) covers field processing, sample handling, shipping, and chain of custody of samples. The SOP describes the steps required to maintain and document the integrity of the sample after collection through analysis. This guide does not cover sample collection procedures or analysis of the sample. For source and matrix specific sample collection details please refer to the appropriate collection SOP. The main objective of proper sample handling is to ensure there are no significant changes in the composition of the sample before it is analyzed. This SOP is to be followed unless project objectives or physical conditions make it inappropriate. In such a case, the exact procedures followed, or deviations from the SOP must be documented and a copy submitted to the Division of Water Quality Assurance Officer for possible incorporation into future updates to this SOP.

COVID-19 update: In order to minimize exposure and to protect staff while continuing to execute the core mission of Division of Water and its water quality monitoring programs, modifications were developed that incorporate social distancing recommendations driven by COVID-19. Modifications to protocols for collection of water quality data as part of DOW monitoring programs are contained within this SOP. All sampling scenarios described within require that samplers wear gloves throughout the day to minimize direct contact with equipment surfaces. This is in addition to described protocols requiring samplers to wear gloves to minimize sample contamination. Where applicable, all protocols described below should be conducted in accordance with guidance provided in SOP#603-20 Guidance for Field Work During COVID-19 Pandemic.

3. Summary of Method

The nature of various parameters makes it necessary to perform some form of processing in the field to maintain sample integrity during storage. Analysis of some constituents require that the sample be partitioned into smaller fractions and/or be preserved to minimize qualitative and quantitative changes to the sample.

Procedures for handling and preserving a sample are contingent upon both the collection process and the specific parameter to be analyzed. Delivery of the sample to the analytical laboratory should be as soon as possible and without contravening the integrity of the sample.

4. Definitions

40 CFR Part 136 – Title 40 (Protection of Environment) is a part of the United States Code of Federal Regulations. Title 40 covers environmental regulations that are promulgated by the US Environmental Protection Agency (EPA), based on the provisions of United States laws (statutes of the U.S. Federal Code).

Chain of Custody – The process of documenting that a sample has been in the physical possession of or under a form of control that has prevented the tampering or alteration of its characteristics from the time it was collected until it was analyzed.

Equipment Blank – assess the potential of cross contamination of samples due to insufficient decontamination of sampling equipment. At a predetermined sampling site the field crew uses an aliquot of de-ionized/distilled water and processes it sequentially through each component of the sampling system in the same manner and using the same equipment as the environmental sample(s).

Field Blank – assess the potential for contamination from field conditions during sampling. A container of deionized/distilled water is included with the supplies for a sampling event. At a predetermined sampling site, the field crew opens the designated container of field blank water brought into the field. The deionized/distilled water is exposed to the air for approximately the same amount of time that it takes to collect a sample then pours the water directly into parameter specific sample bottles. The field blank is processed along with the environmental samples.

Holding Time – the length of time a sample can be stored after collection and prior to analysis with no significant changes in the composition of the pertinent constituents and therefore can be considered valid for the analysis.

Trip Blank – is a check on in-transit sample contamination. It originates with the analytical laboratory using deionized water and accompanies the sample bottle from initial distribution by the laboratory through final analysis. The trip blank is ***not*** opened until analysis. The trip blank(s) follows the same storage and transport procedures as the sample bottles used for a collection event.

5. Health and Safety Warnings

This standard does not address all safety concerns associated with the field processing of samples and the handling of chemical reagents. The reader is referred to the Division of Water's (DOW) 2019 Health and Safety Program (HASP) and is recommended to follow the appropriate health and safety practices consistent with handling chemical preservatives.

COVID-19 update: According to SOP 603-20 DOW Guidance for Field Work During COVID-19 Pandemic, samplers should wear gloves throughout the sampling day and thus should minimize direct contact with equipment surfaces. However, during daily activities indirect contact with equipment surfaces may occur and therefore external surfaces may need to be disinfected. If equipment is not to be used for more than 5 days, normal cleaning procedures and storage are sufficient to ensure disinfection. In

instances where equipment is to be used by another individual within 5 days, disinfection of external surfaces such as handles may be cleaned with supplied disinfectant. Care should be taken to not use disinfectant in ways that will interfere with sample integrity such as cleaning churn nozzles, the interior of the churn, or other surfaces that the sample matrix may contact.

6. Cautions

6.1 Know how to use, handle, and store chemicals safely and appropriately.

6.2 Wear and maintain assigned personal protective equipment.

6.3 Know how to safely and responsibly dispose of preservation chemicals (i.e., acids).

7. Interference

7.1 Introduction of contaminants from equipment when field processing the sample.

7.2 Introduction of contaminants from the sample collector.

7.3 Incorrect chemical treatment used in preserving a sample.

7.4 Mislabeling of sampling bottles.

7.5 Samples held past established holding times.

8. Personnel Qualifications

All staff responsible for handling samples shall be familiar with the procedures outlined in this standard, the DOW's HASP and the Quality Assurance Project Plan (QAPP) for the sampling project prior to conducting water quality sampling.

COVID-19 update: All samplers should also be familiar with Guidance for Field Work During COVID-19 Pandemic (SOP#603-20).

9. General Equipment and Supplies

9.1 Chain of Custody (COC)

9.2 Chemicals and materials for cleaning and decontaminating equipment

9.3 Collection Chamber and chamber bags for groundwater sampling

9.4 Field sheets or logbooks

9.5 Filtering apparatus and filters that are compatible with parameters being filtered.

9.6 Flow meters and portable meters for measuring dissolved oxygen, pH, et. al.

9.7 Global Positioning System

9.8 Sample labels and tags

9.9 Sample containers

9.10 Tubing and cables

9.11 Permanent markers and pens

9.12 Pre-chilled coolers and ice packs

9.13 Packaging and shipping supplies – sample seals, Parafilm, tape, and poly bags

9.14 Personal protective equipment – gloves, safety glasses, and boots

9.15 Deionized Water/distilled water

9.16 Preservatives – Refer to EPA 40 CFR 136.3 Table 2 for water samples. All other media verify with specific SOPs and or QAPPs.

10. Sample Handling Procedure

10.1 The following steps should be followed for all types of samples prior to sample collection: (1) verify what, if any, field processing requirements are needed for the constituents to be analyzed, (2) assemble and collect equipment necessary for sample collection, handling and transport, (3) prepare documentation (COC, field sheets and logbooks) pertaining to sample collection, handling, and transport, (4) pre-label collection bottles and sample bottles, and (5) establish and maintain a clean working area.

10.2 Samples are to be collected and handled to prevent contamination at detectable levels. When trace level monitoring is being performed, samples must be collected and handled using the “clean hands/dirty hands” procedures described in US EPA Method 1669 (1996), with one person (clean hands) taking the samples, wearing double-layers of latex or vinyl, powderless gloves and the other person (dirty hands), also wearing double gloves, performing procedures related to equipment setup.

10.3 Groundwater

10.3.1 This applies to private and public wells, monitoring/observation wells as well as springs.

10.3.2 Equipment

10.3.2.1 Automatic composite sampler with energy source

10.3.2.2 Pre-cleaned 5-gallon container and tubing appropriate for sample type. Or if required for special water chemistry constituents; a sample collection chamber and chamber bags to collect the groundwater sample to prevent atmospheric contamination of the samples. (PVC frame with clear bag).

10.3.2.3 YSI multimeter, Hydrolab, or an in-line flow cell unit

10.3.3 Reagents

10.3.3.1 Refer to EPA 40 CFR 136.3 Table 2 for chemical preservation requirements.

10.3.4 Handling Procedure

10.3.4.1 The collection point for the sample can be either before or after the pressure tank if the tank has a bladder that prevents contact of the water with air within the tank. For monitoring/observation wells no pressure tank will exist. If a site includes a treatment system, the sample is taken before the system or after the treatment system if it is turned off and the lines are well flushed.

10.3.4.2 Each well site to be sampled will have a clean set of tubing for the stabilization process as well as the actual sampling.

10.3.4.3 *After pressure tank*: Water is extracted from the well at the sample collection point at a slow rate and circulated through clean five-gallon containers using PFTE tubing with the multi-meter probe in the container.

10.3.4.3.1 Five-gallon containers are connected in series to handle the necessary volume of water to reach stabilization.

10.3.4.3.2 The water should be run to waste until the pump turns on.

10.3.4.4 *Before pressure tank*: The valve should be opened only enough for a steady, laminar, non-turbulent flow of water.

10.3.4.5 If the sample is to be taken from a kitchen faucet the aerator, if any, should be removed and the sample is taken as described in Section 10.3.4.3.

10.3.5 Storage and Shipping

10.3.5.1 As soon as sampling is finished at a site, all samples should be securely packed for delivery or shipment to the designated laboratory. Delivery and/or shipment methods must be selected to meet required holding times. Sample storage, if necessary or practical, must adhere to the preservation requirements for the samples. Refer to EPA 40 CFR 136.3, Table 2 for parameter specific preservation and holding time requirements. See Sections 11-14 for additional guidance on storage and shipping requirements.

10.4 Ambient surface waters

10.4.1 This applies to water column samples collected from water bodies such as reservoirs, streams and rivers.

10.4.2 Equipment

10.4.2.1 Compositing container –churn splitter or integrating carboy

10.4.2.2 Filtration apparatus and filters (0.45 µm) that are compatible with the parameters being filtered.

10.4.2.3 Sample containers – certified cleaned and free of contamination. Container material to be compatible with parameter being analyzed. Refer to 40 Code of Federal Regulations Part 136 for guidance and verify bottle requirements, size, and type with analytical laboratory.

10.4.2.4 Gloves - latex or nitrile, powder free

10.4.2.5 Pre-cleaned filter apparatus/cartridges – verify manufacture's specifications of filters with analysis needs

10.4.2.6 Power source suited for water contact – if required for filtering equipment

10.4.2.7 Pre-cleaned sample bottles – verify bottle requirements, size, and type with analytical laboratory.

10.4.3 Reagents: Verify type and quantity of preservative with analytical laboratory.

10.4.3.1 H_2SO_4 for preserving nutrient samples to pH <2

10.4.3.2 HNO_3 for preserving metals samples to pH <2

10.4.3.3 Carbonated water, Formalin-Rose Bengal solution, and acid Lugols for preserving phytoplankton tows

10.4.3.4 5 mg/l $MgCO_3$ solution for preserving chlorophyll-*a* samples

10.4.4 Handling Procedures

10.4.4.1 Use a churn splitter to facilitate the withdrawal of a representative subsample from a large composite sample of ambient water. Verify with analytical laboratory and/or 40 CFR Part 136 what parameters may be composited and what must be collected as a direct grab sample.

10.4.4.2 For all parameters that can be composited, collect (using an appropriate collection method) a volume of water sufficient to fill all sample containers plus an additional 1-2 liters. Additional water is needed to allow for proper sample mixing in churn splitter or integrating carboy.

10.4.4.3 Withdrawal of subsamples from the churn splitter

10.4.4.3.1 Maintain a churning rate of about 9 inches per second. Establish and maintain the churning rate before beginning to withdraw subsamples.

10.4.4.3.1.1 COVID-19 update: churning may be conducting by a single person operating the churn shaft, opening the spigot, and filling bottles.

10.4.4.3.2 Do not allow the churn disc to break the water surface during mixing of the sample. In doing so chemical changes may occur and the sample will no longer be representative.

10.4.4.3.3 Do not draw down water below a point about 2 inches above the spigot for non-dissolved fractions.

10.4.4.3.4 Subsamples are filled in the order that will minimize possible contamination of the subsequent sample containers. Samples that require no preservative are filled first. Samples for nitrogen series parameters are filled before bottles that are preserved with nitric acid. Samples with sulfur as a target analyte are filled prior to bottles with sulfuric acid preservation.

Filtered samples are drawn last unless their preservation method conflicts with the above fill order guidance.

10.4.4.4 Unfiltered samples/Non-preserved bottles

10.4.4.4.1 When contract laboratories provide sample bottles that are certified cleaned do not rinse with the collected water.

10.4.4.4.2 Transfer the sample from the compositing container into a labeled sample container.

10.4.4.4.3 When collecting for parameters that require acid preservation, fill the bottle with the sample add the appropriate volume of acid to preserve the sample.

10.4.4.4.4 Secure the cap to the bottle.

10.4.4.4.5 Rotate bottle side to side to ensure proper mixing of the preservative.

10.4.4.5 Unfiltered samples/Pre-preserved bottles

10.4.4.5.1 Pre-preserved sample bottles are not to be rinsed with the water contained in the sampling container or churn.

10.4.4.5.2 Use caution when opening pre-preserved bottles. Bottles are to be tilted away from you or other people to avoid exposure to acidic fumes.

10.4.4.5.3 Open churn spout and fill the sample bottles to the appropriate level (volume). When adding water to bottles pre-preserved with acid use extra care. Keep bottle tilted away from you. Do not to overfill the bottle.

10.4.4.5.4 Secure the cap to the bottle

10.4.4.5.5 Rotate bottle side to side to ensure proper mixing of the preservative.

10.4.4.6 Filtered samples:

10.4.4.6.1 Filter samples only after all whole water samples have been drawn, unless preservation method contravenes the fill order guidance in **10.4.4.3.4.**

10.4.4.6.2 It is not necessary to continue churning during filtration as all whole water samples have already been drawn.

10.4.4.6.3 All Filtering equipment, filters and tubing are to be free from contaminants. Set up filtering equipment in a clean area.

10.4.4.6.4 Attach filtration tubing to the filtering equipment.

10.4.4.6.5 Maintain sufficient vacuum pressure to draw entire sample through the tubing and filter, or if using peristaltic pump maintain approximately 300 rpm.

10.4.4.6.6 Submerge the intake end of the filtration tubing in the churn. Position the outlet end of tubing either over a collection bucket or ground where sample water may flow onto.

10.4.4.6.7 Allow the sample to run through the system for a few seconds to allow an ample volume of water to process through the system before filling sample bottles.

10.4.4.6.8 Preserve the sample with the appropriate acid if the pre-cleaned sample bottles are not pre-acidified.

10.4.5 Storage and Shipping

10.4.5.1 Samples are to be stored at ≤6°C (unless noted otherwise) and shipped immediately.

10.5 Lakes

10.5.1 Equipment

10.5.1.1 Churn splitter or integrating carboy

10.5.1.2 Filtration apparatus and filters (0.45 µm mixed ester and glass fiber)

10.5.1.3 Sample containers – polyfulfonate bottles (organics and inorganics), 50 mL polypropylene centrifuge vials (zooplankton), and 20 mL borosilicate glass vials (chlorophyll)

10.5.2 Reagents

10.5.2.1 H_2SO_4 for preserving nutrient samples to pH <2

10.5.2.2 HNO_3 for preserving metals samples to pH <2

10.5.2.3 Carbonated water, Formalin-Rose Bengal solution, and acid Lugols for preserving phytoplankton tows

10.5.2.4 5 mg/l $MgCO_3$ solution for preserving chlorophyll-*a* samples

10.5.3 Handling Procedures

10.5.3.1 Regardless of the sample collection method used, collect a volume of water sufficient to fill all sample containers plus an additional 1-2 liters to allow for proper mixing in churn or mixing carboy.

10.5.3.2 Unfiltered samples

10.5.3.2.1 Non-preserved sample bottles: When contract laboratories provide sample bottles that are certified cleaned do not rinse with the collected water. For non-certified clean bottles, transfer a small amount of water from the compositing container or collection device into the sample bottle and cap. Invert to completely line the inner walls of the bottle. Rinse bottles to help ensure the removal of any residues from storage procedures.

10.5.3.2.2 Pre-preserved sample bottles are not to be rinsed with the water contained in the sampling container or churn. Open mixing churn spout and fill the sample bottles to the appropriate level (volume).

10.5.3.2.3 Transfer the sample from the compositing container into suitable labeled sample container.

10.5.3.2.4 If using non-preserved bottles to collect parameters that require acid preservation, before capping the bottle, add the appropriate volume of acid to preserve the sample.

10.5.3.3 Filtered samples Chlorophyll a and DOC

10.5.3.3.1 Transfer the sample(s) from the compositing container into a clean graduate cylinder and measure and record the sample volume.

10.5.3.3.2 Filter the sample through a 0.45 µm glass micro-fiber filter.

10.5.3.3.3 Certified pre-cleaned sample bottles are not to be acclimated with the sampling medium.

10.5.3.3.4 Maintain sufficient vacuum pressure to draw entire sample through a clean filter.

10.5.3.3.5 Once filtration is completed, release pressure and transfer the sample from a port on the receiver flask into a suitable, labeled sample container for DOC.

10.5.3.3.6 If more volume of water is needed for the chlorophyll a continue with filtering from step 10.5.3.3.1 record the volume of water needed on the chlorophyll a bottle (Sample volume required for chlorophyll a analyses is specified by the laboratory and identified in the QAPP for each program.).

10.5.3.3.7 Fold filter into quarters and place in tin foil square, fold tin foil square to completely cover filter and place into the sampling container.

10.5.3.3.8 Preserve the sample with the appropriate acid if the pre-cleaned sample bottles are not pre-acidified.

10.5.3.4 Filtered samples Orthophosphorus

10.5.3.4.1 Filter the sample through a 0.45 µm phosphorus free filter.

10.5.3.4.1 Maintain sufficient vacuum pressure to draw entire sample through a clean filter.

10.5.3.4.2 Once filtration is completed, release pressure and transfer the sample from a port on the receiver flask into a suitable, labeled sample container.

10.5.4 Storage and Shipping

10.5.4.1 Samples not shipped immediately should be frozen and shipped as soon as possible to avoid sample receipt at a time when the receiving laboratory is closed.

10.5.5 Once preserved, plankton samples can be stored at room temperature until analysis

10.6 Sediments

10.6.1 Equipment

10.6.1.1 Electric, gravity box, or push corer or extruder

10.6.1.2 Standard and Petite Ponar® Dredge Samplers

10.6.1.3 Trowel or Scoop

10.6.2 Reagents

10.6.2.1 Alconox® powdered or Liqui-nox liquid detergents for cleaning sampling equipment

10.6.2.2 Deionized/distilled water for rinsing equipment

10.6.3 Handling Procedure

10.6.3.1 Trowel or Scoop

10.6.3.1.1 Stand facing the direction of flow and approach the sampling location from the downstream direction. Do not disturb the sediment prior to collecting the sample.

10.6.3.1.2 Scoop the sample in the upstream direction of flow.

10.6.3.1.3 Transfer the sample directly into an appropriate sample container or, if compositing, to a clean stainless steel or Nalgene mixing pan or bucket.

10.6.3.1.4 Homogenize sample and then transfer sample directly to an appropriately labeled sample container.

10.6.3.2 Ponar Samplers

10.6.3.2.1 Arrange the Ponar with the jaws in the open position. Push the spring loaded release pin into the release holes on the lever arms.

10.6.3.2.2 Slowly lower the sampler through the water column to the substrate, being careful not to prematurely trip the jaws closed.

10.6.3.2.3 Raise the sampler to the surface and slowly decant any free liquid through the screens on the top of the sampler.

10.6.3.2.4 Place the sampler in a clean stainless steel or Nalgene tub. Try not to use sediment that has come in contact with the sides of the sampler.

10.6.3.2.5 For standard sediment evaluation, open the jaws by pushing down on the hoisting ring and lift the sampler out of the tub exposing the sediment sample.

10.6.3.2.6 Homogenize the sample for 2-3 minutes and transfer sample directly to the appropriate labeled sample container using clean spatulas or scoops.

10.6.3.3 Corers

10.6.3.3.1 Measure the water depth and entire corer length (bottom of tube to top of extension rod) prior to coring.

10.6.3.3.2 Place the sampler in a vertical position in the water and allow the core tube to touch the sediment surface.

10.6.3.3.3 Push the sampler into the sediment.

10.6.3.3.4 Slowly withdraw the sampler from the sediment. Immediately cap and tape bottom of the core tube.

10.6.3.3.5 Drain excess water above sediment interface.

10.6.3.3.6 Remove the excess core tube using a pipe cutter. Cap and tape the top of the core tube.

10.6.4 Storage and Shipping

10.6.4.1 Samples are to be stored at 4°C until and shipped immediately in a cooler with ice packs.

10.7 Stream Biomonitoring

10.7.1 Equipment

10.7.1.1 Pre-cleaned, 32-ounce, wide-mouth polyethylene jars with aluminum foil-lined plastic caps

10.7.1.2 Pre-cleaned, 4-ounce, wide-mouth glass jars

10.7.1.3 U.S. No. 30 Standard Sieve, U.S. with 8-inch diameter

10.7.2 Reagents

10.7.2.1 Ethyl alcohol, 95%

10.7.3 Handling Procedure

10.7.3.1 Kick and dip net samples: The contents of the pan are sieved through a U.S. no. 30 standard sieve and transferred to a wide-mouth quart jar. 95% ethyl alcohol is added to fill the jar. Samples with large amounts of organic matter, such as algae, are drained of the initial preservative after thorough mixing and preserved with fresh ethyl alcohol.

10.7.3.2 Multiplate samples: The slurry containing the sample is poured into a U.S. no. 30 standard sieve. The residue is rinsed with river water, funneled, and placed in a 4-ounce glass jar. 95% ethyl alcohol is added to fill the jar.

10.7.3.3 Ponar samples: Contents of the Ponar sampler are sieved in a plastic sieve bucket with a U.S. Standard No. 30 mesh sieve bottom, and are transferred to a wide mouth quart jar. 95% ethyl alcohol is added to fill the jar. Samples with large amounts of organic matter or dense silt-clay are drained of the initial preservative, after thorough mixing, and preserved with fresh alcohol.

10.7.3.4 Tissue samples: Organisms are placed in pre-cleaned 4-ounce wide-mouth glass jars containing water from the stream being sampled. In the laboratory, specimens are emptied into a pre-cleaned Petri dish and examined under a dissecting stereo-microscope to eliminate large foreign particles.

10.7.3.4.1 Mollusk tissues, except zebra mussels, are removed from the shells for analysis.

10.7.3.4.2 Crayfish are measured for carapace length.

10.7.4 Storage and Shipping

Store and ship the samples to the laboratory following the steps below and Sections 11-14

10.7.4.1 Kick samples, dip net samples, multiplate samples, and Ponar samples: samples are stored shelved or boxed at room temperature.

10.7.4.2 Tissue samples: prior to freeze-drying, samples are stored on ice in a cooler, or in a refrigerator. Following freeze-drying, samples are stored shelved or boxed at room temperature.

10.8 Wastewater

10.8.1 Equipment

10.8.1.1 Automatic composite sampler (ISCO sampler or equivalent)

10.8.1.2 Collection cups (stainless steel or Teflon)

10.8.1.3 Buckets (Stainless Steel or Teflon)

10.8.1.4 Field filtration system and 0.45-μm filter

10.8.2 Reagents

10.8.2.1 Acquisition of proper preservatives. Refer to EPA 40 CFR Part 136.3, Table II - Required Containers, Preservation Techniques, and Holding Times.

10.8.2.1.1 De-ionized water, Nitric and hydrochloric acid, and phosphate-free cleaning detergent.

10.8.3 Handling Procedure

10.8.3.1 Collect a desired volume of wastewater sample representative of actual condition of the wastewater by means of a grab (see current Wastewater Sample Collection SOP) or composite technique (see current Wastewater Sample Collection SOP).

10.8.3.1.1 A grab sample is collected directly into the sample container.

10.8.3.1.1.1 When the sample cannot be collected directly, a suitable collection cup should be used. The cup should be rinsed with the wastewater to be sampled.

10.8.3.1.1.2 The sample should be collected into a cup, swirled, and immediately poured into the sample container to avoid the settling of solid particles.

10.8.3.1.2 A composite sample should contain a minimum of eight grab samples, collected over the specified collection time period, at a constant sample volume for a constant flow interval; at a flow-proportioned sample volume for a constant time interval or as indicated by the permit. Where

continuous flow monitoring equipment is not available or effluent flows do not vary more than ten percent over the course of the composite sample collection, composite samples may be composed of equal size grab samples taken at equal time intervals.

10.8.3.2 Field Filtration

For parameters requiring field filtration, the sample should be drawn for filtering after all other sample containers have been filled unless their preservation method contravenes the fill order guidance of **10.4.4.3.4.**

10.8.3.2.1 Assemble and rinse the filter unit and sample container with a small volume of the collected sample by running it through the filter unit and discarding the rinsate.

10.8.3.2.2 Filter the required volume of sample through the filtration unit into the sample container.

10.8.4 Storage and Shipping

10.8.4.1 After all field sample fractioning and filtering (if required), preserve the sample fractions appropriately and prepare them for shipment to the laboratory according to Sections 11-14.

11. Storage

11.1 Samples should be collected and stored in appropriate sample containers as prescribed in 40 CRF Part 136.3, Table II. For parameters/matrices not listed in 40 CFR Part 136.3, Table II, the applicable SOPs and/or QAPPs must be followed regarding containers and storage.

11.2 To comply with all analytical holding times, sample storage times must be minimized whenever possible.

11.3 If samples must be stored, follow the appropriate preservation techniques and meet holding times of the parameter to be analyzed. For aqueous samples follow EPA 40 CRF136.3, Table 2. For sediments and macroinvertebrates follow specific SOPS and QAPPS.

NOTE: Tests on non-potable water for bacteriological contaminants have an 8-hour holding time. No more than 8 hours may elapse from the time of collection to the time processed samples begin incubation.

12. Preservation

12.1 Samples are to be preserved immediately after collection unless directed otherwise by the applicable regulations, analytical method, and/or the analytical laboratory.

12.2 Aqueous Samples

12.2.1 All water samples must be preserved according to EPA 40 CFR Part 136.3, Table II requirements.

12.2.2 Do not over or under preserve samples. Follow analytical laboratory guidelines for appropriate volume of preservative.

12.2.3 Add the preservative to the sample container and securely affix the bottle cap.

12.2.4 For all non-volatile samples, vigorously turn bottle side to side several times to ensure proper mixing of the preservative.

12.2.5 Pre-chilled coolers are to be used for samples that require chilling to ≤6°C. Place the temperature blank in the cooler in an area where it will generate a representative temperature

12.2.6 "Wet ice" must be kept in watertight containers or synthetic ice substitutes (e.g., freezer gel packs) should be used.

12.3 **NOTE:** Laboratories may provide samplers with pre-acidified sample bottles.

12.4 Sediment

12.4.1 Samples are to be chilled or frozen as directed by project specific QAPPs.

12.5 Macroinvertebrates

12.5.1 Tissue for chemical Analysis

Macroinvertebrates are placed in hexane-washed 4 ounce glass jars filled with river water and immediately chilled. Samples are to be frozen as soon as possible and are to be kept frozen until freeze drying.

12.5.2 Species Identification

12.5.2.1 Kick Sampling: The net contents are emptied into a pan of stream water, examined, and the major groups of organisms are recorded on a field data sheet, usually on the ordinal level. Larger rocks, sticks, and plants may be removed from the sample if organisms are first removed from them. The contents of the pan are sieved with a U.S. no. 30 standard sieve and transferred to a quart jar. The sample is then preserved by adding 95% ethyl alcohol.

12.5.2.2 Multiplate Sampling: All accumulated organisms and other material are scraped from the plates with a 3-inch wide paint scraper into the water in the bucket. The resultant slurry is poured into a U.S. no. 30 standard sieve, the residue rinsed with river water, and placed in a 4-ounce glass jar. 95% ethyl alcohol is added to fill the jar.

12.5.2.3 Ponar Sampling: Same manner as Kick sampling Section 12.3.2.1

13. Labeling

13.1 All sample bottles/containers are to be labeled/tagged, at a minimum, with the following identifying information:

13.1.1 Sample ID number

13.1.2 Sample location and/or site ID

13.1.3 Sampling date (e.g., 06/24/02) and time (in military time) rounded to the nearest hour

13.1.4 Sample Delivery Group (SDG)

13.1.5 Preservation chemical

13.1.6 Analyte to be measured (i.e., BOD, VOA, or metals)

13.2 Use a permanent, waterproof marker or pen to fill out the labels or pre-printed labels.

13.3 All sample labels must be legible and filled out before the sampler leaves the sampling site.

13.4 Apply the label to the sample bottle, not to the sample bottle cap.

13.5 All documentation for a sampling event, field sheet, chain of custody and, bottle labels must be consistent in recording the metadata for a sampling site, (date, time, location, location ID and parameters sampled).

14. Chain of Custody and Laboratory Submission

14.1 It is necessary to have an accurate written record to trace the possession and handling of samples from the field to the final analytical results.

14.2 A chain of custody is defined as the process of documenting that a sample has been in the physical possession of or under a form of control that has prevented the tampering or alteration of its chemical characteristics from the time of sample collection through sample analysis.

14.2.1 A Chain-of-Custody record form is used to record and document the process.

14.2.2 A chain of custody links dates, times, and locations with all individuals who possess and exchange sample materials.

14.2.3 Figures 1 is an example of Chain-of-Custody record forms.

14.3 Samples submitted to a laboratory must be accompanied by a fully completed chain of custody form that serves as the request form for the analysis to be conducted on the samples.

14.3.1 Chain-of-custody (COC) forms can be obtained directly from the laboratory or on the laboratory's online website. A generic DOW chain-of-custody form is also available for use in both .pdf and .docx (MSWord) formats, allowing certain information to be pre-populated into the COC prior to deploying into the field. Figure 1 is an example of the DOW COC form.

14.3.2 The sampler should complete all fields on the COC that are known to them. Minimum information to be populated into the COC includes the following: "bill to" name & address, "report to" name & address, sample collector's name, case code, project name, DOW sample delivery group (SDG) ID, sample ID's, collection date & time, number of containers, preservatives used, tests ordered, required turn-around time, required reporting format.

14.3.3 Each cooler requires a COC that is reflective of the contents of that cooler. For sampling events that require more than a single cooler for sample submission to a laboratory, each cooler must have a completed COC for the sample bottles in that cooler. There cannot be a single COC for multiple coolers even if all the samples were collected on the same day.

14.4 The sample collector should retain a copy of the chain-of-custody record, as well as, other forms related to sample submission (courier air bills) that become part of the permanent record.

14.5 The chain of custody and laboratory submission form must contain the name, address, and telephone number of the sample collector and should always accompany the sample(s) during transport.

14.6 The chain of custody and laboratory submission form for a sample must be filled out legibly and with a permanent marker or pen, and be completed before leaving the sampling site.

14.7 A sample is considered to be in *custody* when

14.7.1 It is secured or kept in a safe area to prevent tampering, or

14.7.2 It is in one's actual physical possession or view.

14.8 As few people as possible should handle the sample(s) prior to receipt by laboratory personnel.

14.8.1 Whenever sample(s) is/are transferred from one individual's possession to another individual's, the chain-of-custody record form must be signed and dated to record the transfer.

14.8.2 Whenever sample(s) is/are transferred to a common carrier, the shipper's copy of the shipping documents should be retained as part of the chain of custody documentation.

14.9 Laboratory personnel are responsible for the care and custody of the sample(s) once it/they is/are received by the laboratory and must do the following:

14.9.1 Sign and date the chain of custody of form.

14.9.2 Include the chain of custody and laboratory submission form with the analytical data package.

15. Shipping and Transport

15.1 Obtain authorization from analytical laboratory prior to delivery/shipping samples.

15.2 Follow all laboratory protocols for labeling, documenting, and packaging of samples.

15.3 Secure samples in a manner to prevent breakage and/or leakage during transport.

15.3.1 Check sample bottles to make sure the caps are tightly secured.

15.3.2 Add packaging material (e.g., Styrofoam and newspapers) to prevent samples from shifting or moving.

15.3.3 Where required, place seals or Parafilm (not to be used on organic samples) on the caps of individual sample containers.

15.3.4 Where required and when cross contamination is of concern, place each sample container into a Ziploc bag to prevent cross contamination in case a sample containers leaks/breaks.

15.4 Follow packaging requirements of common carrier (i.e., UPS or Fedex).

15.5 Locked or sealed coolers should be used for transporting samples to prevent sample tampering.

15.5.1 Use security tape to seal the coolers.

15.5.2 Make sure cooler spouts or drains are sealed.

15.6 Use insulated coolers for samples that require chilling.

15.6.1 Use ice that is kept in watertight containers to prevent the melted ice from destroying sample labels, or

15.6.2 Use synthetic ice packs (i.e., freezer gel packs).

15.7 Contact the analytical laboratory about any courier services they may provide.

15.8 Do not ship samples on Friday's unless arrangements have been made with the analytical laboratory for a weekend arrival.

15.9 Protect sample information (i.e., chain-of-custody record, laboratory submission form, and contact information) from damage during shipping by placing in a watertight container (zip or press-lock poly bags).

15.10 Make sure contact information is current.

15.11 Label the outside of the shipping container with a current return address and contact person, and a current laboratory address and contact person.

16. Data and Records Management

16.1 All pertinent information regarding the field sampling process shall be recorded on either field sheets or in a field logbook.

16.2 Sampling information to be recorded should be sufficient to reconstruct the sampling event without relying on the sample collector's memory.

16.3 Recorded information should include a unique sampling site identifier, either a name or number, description of sampling site, sample collector's name type of samples collected, list of analysis requested, lot number of preservation chemical, preservation chemical used, date and time of sample collection, and field observations and measurements. Photo documentation of sampling sites should also be performed when and where possible.

16.4 All information should be legible and recorded with a permanent marker or ink pen.

16.5 All corrections must be made by making a single line cross out and writing in the corrected notation adjacent to the cross out. The erroneous entry must still be legible under the cross out. All corrections must be initialed and dated. If space does not allow the corrected information to be entered right next to the cross out (ex. COC form) use the next available line.

16.6 Data from samples not meeting proper preservation and/or holding times will be reviewed and appropriately marked/flagged.

17. Quality Assurance and Quality Control

The samples that are field processed for analysis must accurately represent the media being sampled and be unaffected by the partitioning, preservation, and shipping procedures. The objective of this quality assurance methodology is to establish and maintain standards that will ensure the integrity of the samples from the time they are collected thru analysis.

17.1 Verify all sample-processing requirements prior to collecting the sample.

17.2 Verify the shelf life (i.e., expiration date) of all chemicals to be used for sample preservation.

17.3 Wear gloves and avoid contact with the sample and potential sources of contamination while processing samples.

17.4 Collect field blanks and equipment blanks as part of the quality control samples.

17.5 Keep sample-processing equipment in a non-contaminating container when not in use.

17.6 Keep sample bottles capped and away from potential contamination sources.

18. References

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18.6 Okster, L. 1994. Industrial User Inspection and Sampling Manual for POTWs. USEPA, Office of Wastewater Enforcement and Compliance.

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18.14 U.S. Geological Survey. 1999. Techniques of Water-Resources Investigations, Book 9 – Handbooks for Water Resources Investigations, Section A. – National Field Manual for the Collection of Water-Quality Data.

19. Appendices (Tables and Figures)

TABLE 1 – SAMPLING HANDLING SPECIFICATIONS – WATER COLUMN				
Parameter	**Collection Method**	**Sample Processing**	**Sample Container /Preservation***	**Filling**
Alkalinity	Depth Integrated	Composite	Plastic or Glass	Do not aerate No head space
Ammonia	Depth Integrated	Composite	Plastic or Glass/H2S04	
Chloride	Depth Integrated	Composite	Plastic or Glass	
Chlorophyll a (field filtered filter)	Depth Integrated, Filtered	Composite	Plastic or Glass	
Coliform -Total & Fecal	Grab - direct into Sterile container	None	Sterile/ 6 hour hold time	
Conductance	Direct Field Measurement			
Specific Conductance	Depth Integrated	Composite	Plastic or Glass	
Dissolved Oxygen	Direct Field Measurement			Do not aerate
Fluoride	Depth Integrated	Composite	Plastic only	
Hardness	Depth Integrated	Composite	Plastic or Glass	
Harmful Algal Blooms	Depth Integrated	Depth Integrated	Amber Plastic or Glass	
Microcystin	Depth Integrated	Depth Integrated	Amber Glass Only	
Chlorophyll a (unextracted, fluoroprobe)	Depth Integrated	Depth Integrated	Amber Glass or Plastic	Do not freeze
Microscopy (dominant algal)	Depth Integrated	Depth Integrated	Amber Glass or Plasict	
Kjeldahl Nitrogen	Depth Integrated	Composite	Plastic or Glass/H2SO4	
Major Cations (Na, K, Ca, Mg, Fe, Mn, As)	Depth Integrated	Composite	Plastic or Glass/HNO3	
Metals, Total Recoverable	Depth Integrated	Composite	Plastic or Glass/HNO3	
Metals, Dissolved	Depth Integrated	Composite Filtered	Plastic or Glass/HNO3	
Mercury, Total	Grab- Do Not Aerate	Do Not composite	Teflon or Glass/no preservative	Clean Hands process. Fill to shoulder.
Nitrogen, Total	Depth Integrated	Composite	Plastic or Glass	
Nitrate-Nitrite	Depth Integrated	Composite	Plastic or Glass/H2SO4	
Nitrate	Depth Integrated	Composite	Plastic or Glass/H2SO4	

Nitrite-NO_2	Depth Integrated	Composite	Plastic or Glass/H2SO4	
Oil and Grease	Grab	Do not composite	Glass only	
Orthophosphate	Depth Integrated	Composite Filtered	Plastic or Glass	
pH	Direct Field Measurement			
pH	Depth Integrated	Composite	Plastic or Glass	No head space
Phenolic Compounds	Grab	Do not composite	Amber Glass only/H2SO4	Do Not Aerate
Phosphorous (Total)	Depth Integrated	Composite	Plastic or Glass/H2SO4	
Phosphorus, Total Dissolved (field filtered filtrate)		Composite	Plastic or Glass/H2SO4	
Solids (Total)	Depth Integrated	Composite	Plastic or Glass	
Solids (Total Dissolved)	Depth Integrated	Composite	Plastic or Glass	
Solids (Total Volatile)	Depth Integrated	Composite	Plastic or Glass	
Sulfate	Depth Integrated	Composite	Plastic or Glass	
Total Organic Carbon/Dissolved Organic Carbon (field filtered filtrate)	Depth Integrated	Composite	Plastic or Glass	
Toxicity Testing Sample	Depth Integrated	Composite	2 L Plastic	
True Color	Depth Integrated	Composite	Plastic or Glass	
Turbidity	Depth Integrated	Composite	Plastic or Glass	
UV-254	Depth Integrated	Composite	Plastic or Glass	
Volatile Halogenated Organics	Direct Grab D.O. Sample Bucket	Do not composite	Glass and Teflon-lined septa	Do not aerate

*all samples are iced to < or equal to 6 °C

Figure 1 – Sample Chain of Custody Form

CHAIN OF CUSTODY

Page _1__ of _1__

NEW YORK STATE OF OPPORTUNITY | Department of Environmental Conservation

Division of Water

Project Name: **RIBS ROUTINE**	Case Code: **RIB18**	NYSDEC SDG: **041618 R 3W** (MMDDYYRegion#Matrix)
Contract No.: C009115	Sampler Collector:	Sampler Phone No.:
Project Manager: Jeff Lojpersberger	☐ Report to Project Manager Report to:	☐ **Bill to Project Manager** **Bill to: Jason Fagel**
Address: 425 Jordan Rd, Troy, NY 12180	Address:	**Address: 625 Broadway, 4th Floor Albany, NY 12233-3502**
Phone: 518-285-5683	Phone:	**Phone: 518-402-8156**
Email: jeff.lojpersberger@dec.ny.gov	Email:	**Email: jrfagel@gw.dec.state.ny.us**

Matrix Codes:
WW = Wastewater
GW = Groundwater
W = Ambient Water
SE = Sediment
SL = Sludge
T = Tissue
O = Other __DI WATER____

Analyses Ordered (list)

Preservative Codes:
(Please include in ( ) on "Analyses Ordered" line):
1 = Cool to < 6°C
2 = 0.008% $Na_2S_2O_3$
3 = H_2SO_4 to pH < 2
4 = HNO_3 to pH < 2
5 = NaOH to pH > 12
6 = 5 mL/L 12N HCl
7 = 5 mL/L BrCl
8 = HCl to pH < 2
9 = H_3PO_4 to pH < 2
10 = Protect from light
11 = Freeze to < -10°C
12 = Other _____

SITE ID	Collection Date	Matrix Code	Equip. Blank (EB) Field Blank (FB)	Duplicate (QC)	Matrix Spike (MS)	Collection Time	No. of Containers	RIBS ROUTINE	MERCURY				Location Info/ NYSDEC Notes	Lab Sample ID/ Lab Notes
Example:														
14-NEVR-8.9	04/ /18	W				####	10	x	x					
13-ROND-9.9	04/ /18	W				####	10	x	x					
13-WALK-18.6	04/ /18	W		QC		####	10	x	x				*QA/QC*	
13-WALK-18.6	04/ /18	W			MS	####	10	x	x				*Matrix spike*	
13-WALK-18.6	04/ /18	W	EB			####	9	x					*Equipment Blank*	
13-WALK-18.6	04/ /18	W	FB			####	1		x				*Field Blank*	

Special Analysis Instructions:

Special Reporting Instructions: Sample ID to be reported as: SITE ID- Date-Matrix Code- (Equip Blank or Quality Control Code if noted)

Relinquished by Sampler:	Date:			Time:		Received by:	Date:	Time:	Laboratory Receipt Notes:
Relinquished by:	Date:			Time:		Received by:	Date:	Time:	Sample Temp.: _____ °C Properly Preserved: Y / N Samples Intact: Y / N