

# Water Chemistry Sampling Procedure for Lakes

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*State of Wisconsin Department of Natural Resources*

*STANDARD OPERATING PROCEDURES*

*June 2021*

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**Contents**

Scope ..... 3

Safety ..... 3

Equipment ..... 3

Water Sample Collection ..... 4

    Integrated Sampler .....4

        Procedure.....4

    Van Dorn Sampler .....5

        Procedure.....5

    Kemmerer Sampler .....6

        Procedure.....6

    Grab Samples.....7

        Procedure.....7

Water Sample Shipment ..... 7

Inorganic Lab Slip ..... 8

Sample Volumes and Preservation Requirements by Parameter ..... 10

    Chlorophyll *a* .....10

    Total Kjeldahl-Nitrogen .....10

    Nitrogen-Total (equivalent to the sum of TKN and NO<sub>3</sub> + NO<sub>2</sub>) .....10

    Nitrate plus Nitrite-Nitrogen .....10

    Total Phosphorus.....11

    Total Dissolved Phosphorus .....11

    Soluble Reactive Phosphorus (Total Ortho phosphate) .....11

    Color .....11

    Alkalinity, pH, Conductivity .....11

    Metals.....12

    Total Organic Carbon (TOC).....12

    Dissolved Organic Carbon (DOC) .....12

    Total Suspended Solids (TSS).....12

    Sulfate .....13

    Chloride .....13

Quality Assurance for Field Collection ..... 14

    Field Blank .....14

    Field Duplicate.....14

Decontamination and Disinfection ..... 15

## Scope

This Standard Operating Procedure (SOP) pertains to the collection of water chemistry samples. Protocols for collecting water using an Integrated Sampler, Van Dorn, Kemmerer, or grab samples are covered in this document. An integrated sampler collects water from 0 to 6 feet deep and is the preferred method for sampling the epilimnion of stratified lakes or the upper 6 feet of mixed lakes. An integrated sample from 0-6 feet deep should not be taken on lakes shallower than 6 feet deep or on lakes whose epilimnion is less than 6 feet deep at the time of sampling. The integrated sampler may be used to take samples from a smaller depth interval (e.g., 0-3 feet deep). The Van Dorn and Kemmerer are used to collect water from a discrete depth, which is useful for understanding how water chemistry changes with depth. The Van Dorn, Kemmerer, or grab sample may also be used on lakes too shallow for an integrated sample. The Wisconsin State Lab of Hygiene (SLH) requires the use of particular sample bottles for each parameter. Detailed instructions are given as to which parameters can be analyzed from the same bottle and which preservation and filtration techniques are required for a set of parameters. Quality Assurance sampling is also discussed in this SOP.

## Safety

Use a stable boat for sampling and wear a personal flotation device (PFD). A two-person crew is recommended. Ensure that the anchor rope does not become fouled in the propeller.

Collecting samples in cold weather carries the risk of hypothermia and collecting samples in hot weather carries the risk of dehydration and heat stroke. Prepare with appropriate clothing, blankets, sun protection, and drinking water. Carry a fire extinguisher, cellular phone or portable radio, and a first aid kit that includes materials for cleaning wounds (antibacterial soap and clean water or ethyl alcohol). Preserving some water samples requires the use of small amounts of acid. Avoid contact with skin or eyes when acidifying the sample by wearing safety glasses (sunglasses suffice) and nitrile or rubber gloves. Acidify on shore if water conditions are rough and pour acid away from your body.

## Equipment

The following equipment is used for water sampling.

- Boat
- Anchor
- PFD's for all boat passengers
- Lake bathymetric map
- GPS unit with latitude and longitude of deep hole or other sampling station
- Depth finder, metered sounding rod, or metered rope
- Field Data Sheet
- SLH Form 4800-024 – Inorganic Surface Water & Microbiology
- Pencil
- Sharpie pen
- Water Sampler appropriate for determined method (Integrated sampler, Van Dorn, Kemmerer)
- Composite bottle (to collect water from integrated sampler)
- Appropriate sample bottles and acids, such as:
- Alkalinity, pH, chlorophyll *a* and others: plastic quart bottle
- Nutrients: 250-mL polyethylene bottle and sulfuric acid ampoule
- Metals and hardness: 250-mL polyethylene bottle and nitric acid ampoule
- Extra bottles for blanks and/or field duplicates
- Ziploc bags
- Nitrile gloves
- Safety glasses
- Cooler
- Cubed Ice
- Deionized water if taking a blank sample
- Filtering equipment for chlorophyll *a*, total dissolved phosphorus, soluble reactive phosphorus, or dissolved organic carbon

## Water Sample Collection

### Integrated Sampler

An Integrated Sampler collects water evenly from a range of depths near the surface. This is generally the preferred sampling method for characterizing water quality in the epilimnion.

#### Procedure

1. Begin filling out the labels on the appropriate sample bottle(s) with the Field Number and Sample/Device Description. If using the 250-mL bottle, mark whether it is for Nutrients or Metals (two 250-mL bottles are necessary if sampling both).
2. Load all necessary equipment into the boat and navigate to the deep hole station.
3. Set the anchor with enough extra rope to prevent drift
4. Put on nitrile gloves.
5. Put the safety strap of the integrated sampler around your wrist. Rinse the integrated sampler and composite bottle 3 times with lake water before sample collection. Because the sample bottles come directly from the SLH, they do not need to be triple rinsed. However, do not use the sample bottle if the lid came off during shipment because it may be contaminated.
6. Take the water sample from the opposite side of the boat as you rinsed. With the safety strap on your wrist, lower the integrated sampler vertically at a relatively slow speed to 6 feet deep (which is marked with black electric tape). After reaching 6-foot depth, pull sampler up vertically.
7. Empty the contents of the integrated sampler into the composite bottle by pushing ball valve end against the bar installed across the jug's mouth. This pops the ball valve up releasing water from the pipe. **Do not allow water to run over bare hands or get contaminated.**
8. The 6-foot integrated sampler collects ~1 liter of water. Combine as many separate pulls from the integrated sampler as needed to have enough water to fill water sample bottles.
9. After you have collected enough water to fill all sample bottles, gently mix the water in the composite bottle. First fill the quart HDPE bottle, and immediately place the bottle in a cooler on ice. **It is very important to minimize exposure to sunlight!** Be careful not to touch the inside of the lid, bottle, or sample water.
10. Gently mix water in the composite bottle and use to fill the 250-mL polyethylene bottle to the shoulder for nutrients. Fill a second 250-mL polyethylene bottle to the shoulder for metals.
11. Put on gloves and add 1 ampoule of sulfuric acid to the nutrients bottle and 1 ampoule of nitric acid to the metals bottle. Check the box indicating that the sample bottle has been acidified with the appropriate acid. The SLH has pre-loaded acid ampoules for this purpose. SLH now measures the pH of each sample before analyzing it, so it is not necessary to use litmus paper to test pH of the sample after acidification. If the sample is not at the correct pH, SLH will add more acid once they receive the sample. If collected for enforcement, the sample must be acidified within 15 minutes of collection.
12. Remove the sticker label from the acid ampoule bottle and attach it to the lab slip. Be sure that the expiration date and batch number are legible.
13. Place bottle in a Ziploc bag in a cooler with ice cubes (not blue ice packs). Keep samples cool and clean, but not frozen. Refrigerate them if not shipping the same day.
14. If filtering for chlorophyll *a*, refer to the Chlorophyll *a* Filtering protocol. Record volume of water filtered, place filter in a test tube and store frozen. DNR strongly prefers chlorophyll *a* samples filtered the same day, but water samples may be shipped overnight to the SLH for filtering. SLH must filter the sample within 48 hours of collection.
15. Rinse your integrated sampler with deionized (DI) water after use and store topside down. This will prevent algal growth between the ball and the collection end of the sampler.

## Van Dorn Sampler

The Van Dorn Sampler allows sampling from a discrete depth. It may be used instead of the integrated sampler if sampling deep waters or if the epilimnion is less than 6 feet deep. Try to keep sampler out of direct sunlight for extended periods of time. UV light breaks down plastics, particularly latex. Store your sampler dry. The latex internal closure can be adjusted to increase tension between sealing caps. If there is too little tension, the unit will not seal properly.

### Procedure

1. Begin filling out the labels on the appropriate sample bottle(s) with the Field Number and Sample/Device Description. If using the 250-mL bottle, mark whether it is for Nutrients or Metals (two 250-mL bottles are necessary if sampling both).
2. Load all necessary equipment into the boat and navigate to the deep hole station.
3. Set the anchor with enough extra rope to prevent drift. Always secure the end of the sampler to a floating object or the boat to avoid accidental loss of the sampler.
4. Put on nitrile gloves.
5. Lay sampler on its side with the rope side of the cable clamp positioned downward.
6. Start with the top seal (the rubber ball with the lanyard coming out of the top). Use your index finger to pull the arming rod of the release mechanism downward. Pull the top ball seal out of the sampler cylinder using the lanyard loop.
7. Insert the lanyard "loop" inside the hole in the closing mechanism. Release the arming pin to hook the loop.
8. Arm the bottom seal (the rubber ball) using the same procedure as above and clip the stainless steel clip around both strands of the loop lanyard. The sampler is now ready for deployment.
9. Lower the sampler to the desired depth. Release the messenger to activate the closing mechanism. You can generally feel the sampler close at depth through the line.
10. Bring the now closed sampler to the surface. Open the clamp to release water through the drain over the side of the boat. You can cock the rubber ball seal to increase the flow out of the drain tube.
11. Using steps 5 – 10, rinse the Van Dorn sampler a total of three times with lake water from the desired sample depth before sample collection. Make sure to open the clamp to allow water to run through and rinse the drain tube.
12. Using steps 5-10, collect the water sample from the desired depth on the opposite side of the boat as you rinsed. Empty the contents into the appropriate sample bottles. **Do not allow water to run over bare hands or get contaminated.**
13. First fill the quart HDPE bottle, and immediately place the bottle in a cooler on ice. It is very important to minimize exposure to sunlight! Be careful not to touch the inside of the lid, bottle, or sample water.
14. Gently mix the remaining water in the Van Dorn and fill the 250-mL polyethylene bottles.
15. Put on gloves and add 1 ampoule of sulfuric acid to nutrients bottle and 1 ampoule of nitric acid to the metals bottle. Check the box indicating that the sample bottle has been acidified with the appropriate acid. The SLH has pre-loaded acid ampoules for this purpose. SLH now measures the pH of each sample before analyzing it, so it is not necessary to use litmus paper to test pH of the sample after acidification. If the sample is not at the correct pH, SLH will add more acid once they receive the sample. If collected for enforcement, the sample must be acidified within 15 minutes of collection.
16. Remove the sticker label from the acid ampoule bottle and attach it to the lab slip. Be sure that the expiration date and batch number are legible.
17. Place acidified bottle in a single Ziploc bag in cooler with ice cubes (not blue ice packs). Keep samples cool and clean, but not frozen. Refrigerate them if not shipping the same day.
1. If filtering for chlorophyll *a*, refer to the Chlorophyll *a* Filtering protocol. Record volume of water filtered, place filter in a test tube and store frozen. DNR strongly prefers chlorophyll *a* samples filtered the same day, but water samples may be shipped overnight to the SLH for filtering. SLH must filter the sample within 48 hours of collection.
18. After sampling, thoroughly rinse your sampler with DI water. Place pencils in either end so the sampler will dry out. Open or remove the hose clamp so the hose will air dry as well. When the sampler is dry, remove pencils so the rubber tubing does not stretch and wear out.

### Kemmerer Sampler

Kemmerer samplers also allows sampling from a discrete depth and can be used on lakes 3 feet or greater in depth. Try to keep sampler out of direct sunlight for extended periods of time. UV light breaks down plastics, particularly latex. Store your sampler dry.

### Procedure

1. Begin filling out the labels on the appropriate sample bottle(s) with the Field Number and Sample/Device Description. If using the 250-mL bottle, mark whether it is for Nutrients or Metals (two 250-mL bottles are necessary if sampling both).
2. Load all necessary equipment into the boat and navigate to the deep hole station.
3. Set the anchor with enough extra rope to prevent drift. Always secure the end of the sampler to a floating object or the boat to avoid accidental loss of the sampler.
4. Put on nitrile gloves.
5. To set the sampler, pull the bottom stopper down until the shaft assembly snaps into the trip head.
6. If using a solid messenger, run a line through the messenger prior to attaching the line to the sampler. The line should be run through the shaft assembly and secured by knotting it at the bottom of the sampler with a washer. If using the split messenger, attach to the line once the sampler is at the desired depth.
7. Lower the bottle down to the desired depth. Release the messenger to close the sampler. The stoppers seal by their own weight, thus ensuring a complete closure.
8. Bring the now closed sampler to the surface. Open the clamp to release water through the drain tube over the side of the boat.
9. Using steps 5 – 8, rinse the Kemmerer a total of three times with lake water from the desired sample depth before sample collection. Make sure to open the clamp to allow water to run through and rinse the drain tube.
10. Using steps 5-8, collect the water sample from the desired depth on the opposite side of the boat as you rinsed. Empty the contents into the appropriate sample bottles. **Do not allow water to run over bare hands or get contaminated.**
11. First fill the quart HDPE bottle, and immediately place the bottle in a cooler on ice. **It is very important to minimize exposure to sunlight!** Be careful not to touch the inside of the lid, bottle, or sample water.
12. Gently mix the remaining water in the Kemmerer and fill the 250-mL polyethylene bottles.
13. Put on gloves and add 1 ampoule of sulfuric acid to the nutrients bottle and 1 ampoule of nitric acid to the metals bottle. Check the box indicating that the sample bottle has been acidified with the appropriate acid. The SLH has pre-loaded acid ampoules for this purpose. SLH now measures the pH of each sample before analyzing it, so it is not necessary to use litmus paper to test pH of the sample after acidification. If the sample is not at the correct pH, SLH will add more acid once they receive the sample. If collected for enforcement, the sample must be acidified within 15 minutes of collection.
14. Remove the sticker label from the acid ampoule bottle and attach it to the lab slip. Be sure that the expiration date and batch number are legible.
15. Place acidified bottle in a single Ziploc bag in cooler with ice cubes (not blue ice packs). Keep samples cool and clean, but not frozen. Refrigerate them if not shipping the same day.
2. If filtering for chlorophyll  $a$ , refer to the Chlorophyll  $a$  Filtering protocol. Record volume of water filtered, place filter in a test tube and store frozen. DNR strongly prefers chlorophyll  $a$  samples filtered the same day, but water samples may be shipped overnight to the SLH for filtering. SLH must filter the sample within 48 hours of collection.
16. After sampling, thoroughly rinse your sampler with DI water. Place pencils in either end so the sampler will dry out. Open up or remove the hose clamp so the hose will air dry as well. When the sampler is dry, remove pencils so the rubber tubing does not stretch and wear out.

## Grab Samples

A grab sample is an individual sample collected directly into the sample bottle. They can only be taken as deep as an arm's length from the surface. Grab samples are preferred for parameters like bacteria that might easily be contaminated by sampling equipment.

## Procedure

1. Begin filling out the labels on the appropriate sample bottle(s) with the Field Number and Sample/Device Description. If using the 250-mL bottle, mark whether it is for Nutrients or Metals (two 250-mL bottles are necessary if sampling both).
2. Load all necessary equipment into the boat and navigate to the deep hole station.
3. Set the anchor with enough extra rope to prevent drift
4. Put on nitrile gloves.
5. With the sample bottle facing upside down, sink the bottle about one foot under water. Turn the bottle to the side and let the sample bottle fill completely before slowly bringing the bottle to the surface. This ensures air bubbles exit the sample bottle before reaching the surface.
6. Repeat step 5 for all samples planned for collection.
7. Put on gloves and add 1 ampoule of sulfuric acid to the nutrients bottle and 1 ampoule of nitric acid to the metals bottle. Check the box indicating that the sample bottle has been acidified with the appropriate acid. The SLH has pre-loaded acid ampoules for this purpose. SLH now measures the pH of each sample before analyzing it, so it is not necessary to use litmus paper to test pH of the sample after acidification. If the sample is not at the correct pH, SLH will add more acid once they receive the sample. If collected for enforcement, the sample must be acidified within 15 minutes of collection.
8. Remove the sticker label from the acid ampoule bottle and attach it to the lab slip. Be sure that the expiration date and batch number are legible.
9. Place acidified bottle in a single Ziploc bag in cooler with ice cubes (not blue ice packs). Keep samples cool and clean, but not frozen. Refrigerate them if not shipping the same day.
10. If filtering for chlorophyll *a*, refer to the Chlorophyll *a* Filtering protocol. Record volume of water filtered, place filter in a test tube and store frozen. DNR strongly prefers chlorophyll *a* samples filtered the same day, but water samples may be shipped overnight to the SLH for filtering. SLH must filter the sample within 48 hours of collection.

## Water Sample Shipment

1. Fill out SLH form 4800-024.
2. Store water samples cooled to  $\leq 6^{\circ}\text{C}$  (not frozen) until shipment.
3. If chlorophyll *a* samples are field filtered, store chlorophyll *a* filters frozen in a test tube from SLH until shipment.
4. Ship samples in a cooler on ice to the SLH. Include the completed lab slip in a Ziploc bag.

**\*Shipment time** is critical and depends on the holding times for each analysis. SLH receives a lot of samples and does not typically operate on the weekends. Try to time your shipments to arrive by early Friday afternoon at the latest. Call the SLH ahead of time if it is necessary to ship samples that will arrive on the weekend. To ensure the holding times are met, ship the samples as soon as possible. Here are the holding times listed as time since collection:

- 48 hours: water for chlorophyll *a* (if SLH filters), color, ortho phosphate, and unpreserved DOC
- 7 days: Total Suspended Solids
- 28 days: nutrients, sulfate, chloride, TOC, filtered and acidified DOC, frozen chlorophyll *a* filters, mercury
- 6 months: metals (except for mercury)

The recommended shipping procedure depends on your location:

- If close to SLH, deliver samples to SLH by 2:00 pm the same day.
- If far from SLH, and:
  - a) the sampling event includes parameters with 48-hour holding times, ship samples overnight.
  - b) the sampling event does not include parameters with 48-hour holding times, ship within one week.

## Inorganic Lab Slip

Use the Lab Slip Generator in SWIMS (go to the My Projects or Forms tab) to create your lab slips. Pick your project, the appropriate monitoring station, and the Inorganic Surface Water and Microbiology (4800-024) lab slip. Some information can be pre-populated on the computer and other information will be written by hand on the sample date. Contact the SWIMS database coordinator or lake monitoring coordinator if you have questions.

The bullets below detail the information that should be included on the lab slip:

- **Account Number:** this should be associated with your SWIMS project and will notify the lab which account to charge the analyses to
- **Field Number:** write a detailed bottle label that includes the lake name and date (e.g. LostLand071620). If this is a quality assurance sample, include “blank” or “dup” in the field number.
- **DNR User ID:** this will be auto populated by SWIMS with your user id
- **Report to Name:** select the person who the SLH results should be sent to
- **Date Results Needed:** leave blank unless results are needed by a particular date
- **Report to Address:** submit contact information if you are non-DNR
- **Report to Email:** submit email address if non-DNR
- **Date:** list the date that the sample was collected on
- **Time:** list the time with a 24-hour clock that the water sample was collected in the boat
- **End Date:** this is only relevant if compositing samples over a period of time. If that is the case, list the date at which the final sample was taken. For standard, one-time samples, leave blank.
- **End Time:** this is only relevant if compositing samples over a period of time. If that is the case, list the date at which the final sample was taken. For standard, one-time samples, leave blank.
- **Sample Type:** select SU Surface Water
- **Who collected the sample:** List the name, phone number, and email address of the person collecting the sample
- **Station ID:** select the appropriate station id in SWIMS
- **Location Description:** SWIMS will auto populate the station name/description
- **County:** SWIMS will auto populate the county that the station is in
- **Waterbody ID (WBIC):** SWIMS will auto populate the waterbody’s unique identification number
- **SWIMS Fieldwork Seq No:** this will automatically be generated in SWIMS. There should be a unique id for each lab slip. Therefore, it is important not to photocopy lab slips.
- **Sample Description/Device Description:** describe the type of location where the sample was taken (e.g. deep hole) and what type of device was use (e.g. integrated sampler)
- **Enforcement?** check “no” for routine monitoring, check yes if the sample event is in response to an enforcement case
- **Is Sample Disinfected?** Select “no”
- **Field QC Sample:** If collecting a duplicate or blank sample for quality assurance, fill the appropriate circle. If not, leave this section blank.
- **Grant or Project Number:** SWIMS will auto-populate if you associate the correct project
- **Depth of Sample:** list the discrete depth of the sample and the proper units if the sample was taken from a single depth with a Van Dorn, Kemmerer, or grab sample. Otherwise leave blank.
- **Top and Bottom of Sample Interval:** List the range of depths sampled with an integrated sampler and select units. DNR integrated samplers sample at 0 – 6 ft depth.
- **Analyses required:** in this section, check the boxes for all samples you wish to have analyzed. A few items to take note of are listed below.
  - **Plastic Quart Bottle:** If you filter your own chlorophyll *a* sample, be sure to check the “Sample field filtered” box under Plastic Quart Bottle, check the “Chlorophyll A” box and write the volume of water filtered (measure with a graduated cylinder). Note that the chlorophyll *a* concentration cannot be determined without the volume of water filtered.
  - **250-mL Glass Amber:** Check whether TOC or DOC should be analyzed and check the box for how the sample was preserved. If preserved (preferred), they will need separate bottles (TOC is unfiltered and acidified whereas DOC is filtered before it is acidified). Note that both tests can be analyzed from the same bottle if they are left unpreserved and arrive at the SLH within 48 hours. In that case, note that TOC was not acidified.



- **250-mL Nutrients Bottle:** only field filter for total dissolved phosphorus, which will use the 60-mL bottle instead. All analytes should be acidified. If total phosphorus will likely be  $< 12 \mu\text{g/L}$ , request the low-level test. For Low Level Total Phosphorus, the SLH will send an acid-washed bottle in advance.
  - **60-mL Bottle:** field filter and acidify total dissolved phosphorus; field filter and do not acidify orthophosphate. Use separate bottles if submitting both tests.
  - **250-mL Metals Bottle:** do not field filter for most metals tested under typical lake sampling. If sampling for Ca and Mg, select “Hardness-as  $\text{CaCO}_3$ ” as this test will give results for both.
- **Field Parameters – Optional:** leave this section blank. Instead, write this information on your field sheet and enter data into SWIMS yourself. This avoids duplicative data in SWIMS. Further, the lab slip does not allow recording an entire temperature and dissolved oxygen profile, so it is not appropriate for lake information.

## Sample Volumes and Preservation Requirements by Parameter

Below are descriptions of sample volumes and preservation requirements by parameter for the Wisconsin State Laboratory of Hygiene. Note that samples stored at  $\leq 6^{\circ}\text{C}$  must be on ice or refrigerated but not frozen. Sample sizes listed allow for analysis of multiple tests from one bottle. Extra water allows for repeat tests if necessary, so always fill to the shoulder of the bottle. More information can be found here: <https://intranet.dnr.state.wi.us/int/es/science/ls/Preservation/>

### Chlorophyll $a$

**Sample Size:** 50 - 250 mL

**Bottle Type:** quart HDPE with white polypropylene cap for water; 15 mL plastic centrifuge tube for filter

**Preservative:** ice ( $\leq 6^{\circ}\text{C}$ ) and keep in the dark for water sample; freeze filter

**Filter:** 5.0  $\mu\text{m}$  membrane filter

**Holding Time:** 48 hours for water; 28 days for frozen filter

**Special Notes:** SLH protocol requires samples to be filtered within 48 hours, but the best practice is to filter the same day preferably in the shade at the field site or immediately upon returning to the office. It is very important to keep the sample in the dark and to not contaminate with acid, so be careful handling acid for nutrients/metals.

### Total Kjeldahl-Nitrogen

**Sample Size:** 38 mL

**Bottle Type:** 250-mL HDPE with white polypropylene cap

**Preservative:**  $\text{H}_2\text{SO}_4$ , ice ( $\leq 6^{\circ}\text{C}$ )

**Volume of Preservative:** 1 mL 25% (9N)  $\text{H}_2\text{SO}_4$ /250 mL

**Holding Time:** 28 Days

**Compatible With:** COD (15 mL), Ammonia-Nitrogen (38 mL), Nitrate + Nitrite-Nitrogen (38 mL), Total Nitrogen (38 mL), Total Phosphorus (38 mL), Total 175 mL for all parameters

**Special Notes:** None

### Nitrogen-Total (equivalent to the sum of TKN and $\text{NO}_3 + \text{NO}_2$ )

**Sample Size:** 38 mL

**Bottle Type:** 250-mL HDPE with white polypropylene cap

**Preservative:**  $\text{H}_2\text{SO}_4$ , ice ( $\leq 6^{\circ}\text{C}$ )

**Volume of Preservative:** 1 mL 25% (9N)  $\text{H}_2\text{SO}_4$ /250 mL

**Holding Time:** 28 days

**Compatible With:** COD (15 mL), Ammonia-Nitrogen (38 mL), Nitrate + Nitrite-Nitrogen (38 mL), Total Kjeldahl Nitrogen (38 mL), Total Phosphorus (38 mL), Total 175 mL for all parameters

**Special Notes:** 125 mL is required for ammonia-nitrogen when sampling wastewater

### Nitrate plus Nitrite-Nitrogen

**Sample Size:** 38 mL

**Bottle Type:** 250-mL HDPE with white polypropylene cap

**Preservative:**  $\text{H}_2\text{SO}_4$ , ice ( $\leq 6^{\circ}\text{C}$ )

**Volume of Preservative:** 1 mL 25% (9N)  $\text{H}_2\text{SO}_4$ /250 mL

**Holding Time:** 28 Days

**Compatible With:** COD (15 mL), Ammonia-Nitrogen (38 mL), Total Kjeldahl Nitrogen (38 mL), Total Nitrogen (38 mL), Total Phosphorus (38 mL), Total 175 mL for all parameters

**Special Notes:** None

**Total Phosphorus****Sample Size:** 38 mL**Bottle Type:** 250-mL HDPE with white polypropylene cap**Preservative:** H<sub>2</sub>SO<sub>4</sub>, ice (≤ 6°C)**Volume of Preservative:** 1 mL 25% (9N) H<sub>2</sub>SO<sub>4</sub>/250 mL**Holding Time:** 28 Days**Compatible With:** COD (15mL), Ammonia-Nitrogen (38 mL), Nitrate + Nitrite-Nitrogen (38 mL), Total Kjeldahl Nitrogen (38 mL), Total 175 mL for all parameters**Special Notes:** If Total Phosphorus concentration will be less than 12 µg/L, request the low-level test and use an acid-washed bottle provided by the SLH. Acid-washed bottles must be requested in advance.**Total Dissolved Phosphorus****Sample Size:** 50 mL**Bottle Type:** 60-ml HDPE with white polypropylene cap**Preservative:** field filter, then acidify with H<sub>2</sub>SO<sub>4</sub>, ice (≤ 6°C)**Filter:** Sterivex™ 0.45 µm pore cylindrical filter, Cat. No. SVHV010RS**Volume of Preservative:** 1 mL 25% (9N) H<sub>2</sub>SO<sub>4</sub>/60 mL**Holding Time:** 28 days**Special Notes:** Field filtering must occur immediately after sample collection.**Soluble Reactive Phosphorus (Total Ortho phosphate)****Sample Size:** 60 mL**Bottle Type:** 60-mL HDPE with white polypropylene cap**Preservative:** field filter, ice (≤ 6°C)**Filter:** Sterivex™ 0.45 µm pore cylindrical filter, Cat. No. SVHV010RS**Holding Time:** 48 hours**Special Notes:** Field filtering immediately after sample collection is required by code, and it is important to mark on the lab slip whether (or not) the sample was field filtered. The SLH can filter the sample instead if shipped overnight, but the data will be qualified. Regardless of how the sample is received, the sample will be flagged if not delivered within 48 hours.**Color****Sample Size:** 150 mL**Bottle Type:** quart HDPE with white polypropylene cap**Preservative:** ice (≤ 6°C)**Holding Time:** 48 Hours**Compatible With:** Alkalinity & Conductivity (75 mL), BOD (150 mL, low level 900 mL), Nitrite-Nitrogen (38 mL), pH (38 mL), Dissolved Silica (38 mL), Total & Volatile Solids (75mL), Total Dissolved Solids (75mL), Total Suspended Solids (500 mL), Sulfate (50 mL), Surfactants (MBAS) (375 mL or 75 mL for non-compliance), Turbidity (38 mL), Chloride (50 mL), Total for all parameters 1,400 mL to 3,300 mL**Special Notes:** None**Alkalinity, pH, Conductivity****Sample Size:** 100 mL**Bottle Type:** quart HDPE with white polypropylene cap**Preservative:** ice (≤ 6°C)**Holding Time:** 14 Days**Compatible With:** BOD (150 mL, low level 900 mL), Color (150 mL), Chloride (50 mL) Nitrite-Nitrogen (38 mL), Dissolved Silica (38 mL), Total & Volatile Solids (75mL), Total Dissolved Solids (75mL), Total Suspended Solids (500 mL), Sulfate (38 mL), Surfactants (MBAS) (375 mL or 75 mL for non-compliance), Turbidity (38 mL), Total for all parameters 1,400 mL to 3,300 mL**Special Notes:** None

## Metals

**Sample Size:** 250 mL

**Bottle Type:** 250-mL HDPE with white polypropylene cap

**Preservative:** HNO<sub>3</sub>, ice ( $\leq 6^{\circ}\text{C}$ )

**Holding Time:** 6 Months (28 days for Hg)

**Volume of Preservative:** 1 mL 70% (16N) HNO<sub>3</sub> to pH<2

**Compatible With:** Need Whole Volume

**Special Notes:** None

## Total Organic Carbon (TOC)

**Sample Size:** 120 mL

**Bottle Type:** 250-mL glass amber

**Preservative:** H<sub>2</sub>SO<sub>4</sub>, ice ( $\leq 6^{\circ}\text{C}$ )

**Holding Time:** 28 days if acidified

**Compatible With:** DOC if the sample bottle is not acidified or filtered. If not acidified, the lab will acidify the sample for running TOC and will filter and then acidify the sample for DOC. The holding time is then reduced to 48 hours. If the lab preserves TOC, the results will be qualified.

**Special Notes:** The lab prefers to acidify with phosphoric acid, but sulfuric works fine and is more commonly used for other sample parameters.

## Dissolved Organic Carbon (DOC)

**Sample Size:** 60 mL

**Bottle Type:** 250-mL glass amber

**Preservative:** filter with 0.45  $\mu\text{m}$  47 mm hydrophilic polyethersulfone membrane filter, then acidify filtrate with H<sub>2</sub>SO<sub>4</sub>, ice ( $\leq 6^{\circ}\text{C}$ )

**Compatible With:** TOC if the sample bottle is not acidified or filtered

**Holding Time:** 28 days if filtered, acidified, and cooled; 48 hours if water sample is only cooled and shipped to SLH for filtration and acidification

**Special Notes:** SLH protocol requires samples to be filtered and then acidified within 48 hours, but the best practice is to filter the same day at the field site. The next best practice is to filter immediately upon returning to the office on the same day. The lab can filter and then acidify the sample upon receipt if the sample arrives on weekdays within the 48-hour holding time. If filtering yourself, take and submit a blank sample before the lake water sample to ensure that the filtration apparatus is not contaminated (rinse the filtration flask with deionized (DI) water, run DI water through the filter, pour the filtrate into the sample bottle, acidify, and store at  $\leq 6^{\circ}\text{C}$ ). You may use the glass amber bottle to store your unfiltered sample until you are able to filter. Pour the sample into the filtration cup and rinse the amber bottle 3 times with DI water or the filtrate (~10 mL for each rinse) before collecting the filtrate sample. \

## Total Suspended Solids (TSS)

**Sample Size:** 500 mL

**Bottle Type:** quart HDPE

**Preservative:** ice ( $\leq 6^{\circ}\text{C}$ )

**Compatible With:** Alkalinity & Conductivity (75 mL), BOD (150 mL, low level 900 mL), Color (150 mL), Nitrite-Nitrogen (38 mL), pH (38 mL), Dissolved Silica (38 mL), Total & Volatile Solids (75 mL), Total Dissolved Solids (75 mL), Color & Alkalinity (150 mL), Sulfate (38 mL), Surfactants (MBAS) (375 mL or 75 mL for non-compliance), Turbidity (38 mL), Chloride (50 mL), Total for all parameters 1,400 mL to 3,300 mL

**Holding Time:** 7 days

**Special Notes:** None

**Sulfate****Sample Size:** 50 mL**Bottle Type:** quart HDPE with white polypropylene cap**Preservative:** ice ( $\leq 6^{\circ}\text{C}$ )**Compatible With:** Alkalinity & Conductivity (75 mL), BOD (150 mL, low level 900 mL), Color (150 mL), Nitrite-Nitrogen (38 mL), pH (38 mL), Dissolved Silica (38 mL), Total & Volatile Solids (75 mL), Total Dissolved Solids (75 mL), Total Suspended Solids (500 mL), Chloride (50 mL), Surfactants (MBAS) (375 mL or 75 mL for non-compliance), Turbidity (38 mL), Total for all parameters 1,400 mL to 3,300 mL**Holding Time:** 28 days**Special Notes:** None**Chloride****Sample Size:** 50 mL**Bottle Type:** quart HDPE with white polypropylene cap**Preservative:** ice ( $\leq 6^{\circ}\text{C}$ )**Compatible With:** Alkalinity & Conductivity (75 mL), BOD (150 mL, low level 900 mL), Color (150 mL), Nitrite-Nitrogen (38 mL), pH (38 mL), Dissolved Silica (38 mL), Total & Volatile Solids (75 mL), Total Dissolved Solids (75 mL), Total Suspended Solids (500 mL), Sulfate (50 mL), Surfactants (MBAS) (375 mL or 75 mL for non-compliance), Turbidity (38 mL), Total for all parameters 1,400 mL to 3,300 mL**Holding Time:** 28 days**Special Notes:** None

## Quality Assurance for Field Collection

For each field QA/QC sample, the appropriate preservative should be added to the sample in the same manner as the regular. Fill out a separate lab slip for each field QA/QC sample (blank and duplicate) and transport the field QA/QC samples on ice with the other samples.

### Field Blank

A field blank is used to determine if there is any contamination of the sample due to collection equipment, field conditions, or transport. In general, one field blank is recommended for every ten nutrient samples (i.e. 10% of all samples planned over the course of a field season). To collect a field blank, transport deionized (DI) water into the field in a separate container. While in the field, rinse sampling equipment with DI water and then collect a DI water sample, fill and preserve the appropriate sample bottles.

1. Collect a blank sample for all parameters except chlorophyll *a* (or according to your project protocol).
2. Get ASTM Type I water (deionized/reverse osmosis water that is then run through a US Filter Corp or Milli Q filter) from SLH (or another lab with the same filtration capacity). Ensure that the carboys/bottles used for transporting DI water are acid-washed (contact the SLH if you need assistance). Obtain fresh DI water prior to each field season. A 20-L carboy kept at the field office should be sufficient for 1 field season.
3. A 4-L sample bottle should be sufficient to take in the field for 1 blank.
4. Bring the DI water on the boat with you.
5. Before completing the lake water sampling, rinse the sampler and composite bottle three times with DI water. Fill the sampler with DI water a 4<sup>th</sup> time, empty it into the composite bottle through the release valve, then pour water into a separate sample bottle for each water chemistry parameter you are testing on that day. Follow the same procedure as you would for a lake sample, but instead pour DI water into the sampler.
6. Give the field blank sample(s) a unique SWIMS Fieldwork Sequence Number by creating a separate lab slip. Include "blank" in the Field Number (Bottle Label ID). Write "blank integrated sampler" (or name whatever sample device was used) under the "Sample Description" text field and check "Blank" on the lab slip under "If Field QC Sample (select one)".
7. \*Note: If a run of blanks from one field site have elevated concentrations, then you should also test the DI water in the carboy for contamination. Pour water directly from the carboy into a sample bottle, check "Blank" on the lab slip, label as "Lab Water Blank" in the "Sample Description" text field and send to SLH. SLH runs a lab reagent blank every 20 samples, so if we are using their DI water, we do not need to send in lab water blanks as a common procedure.

### Field Duplicate

A field duplicate is used to determine the precision of the sample collection procedures, which could be affected by inherent variability in the waterbody. To collect a field duplicate, repeat the entire sample process to collect an extra sample at the same location as the regular sample. One duplicate sample is recommended for every ten samples (i.e. 10% of all samples planned over the course of a field season). Collect duplicates for all parameters used, including chlorophyll *a*.

1. Collect a duplicate sample. This sample is collected at the same time as the regular sample but will have its own set of bottles and its own lab slip.
2. Repeat the entire process, beginning with rinsing, and collecting a new unique sample with one of the sampling devices. Do not simply fill a second bottle with leftover water in the composite bottle that was used for the regular sample.
3. Give the field duplicate sample(s) a unique SWIMS Fieldwork Sequence Number by creating a separate lab slip. Include "dup" in the Field Number (Bottle Label ID). Write "duplicate integrated sampler" (or name whatever sample device was used) under the "Sample Description" text field and check "Duplicate" on the lab slip under "If Field QC Sample (select one)".

### Decontamination and Disinfection

Clean all equipment between lakes and at the end of the day to prevent the spread of invasive species and prevent sample contamination. The boat, integrated sampler, and other equipment that goes in the lake (e.g., anchor), should be cleaned according to the most recent disinfection procedures: <http://dnr.wi.gov/topic/invasives/disinfection.html>. Another option is to use designated sets of equipment for each lake. This can be accomplished by making multiple integrated samplers. You may also be able to avoid transporting your boat between lakes by sampling from the boat of a resident on the lake. At the end of the day after following disinfection procedures, the composite bottles and integrated samplers should be rinsed with DI water and left to dry. Hang integrated samplers with ball end up allowing water to drain out. Store equipment in a clean location.