Standard Operating Procedures (SOP)

Lake Water Quality Sampling

Minnesota Pollution Control Agency
520 Lafayette Road North
Saint Paul, MN 55155-4194
http://www.pca.state.mn.us
651-296-6300 or 800-657-3864 toll free
TTY 651-282-5332 or 800-657-3864 toll free
Available in alternative formats

Authors and Contributors

Pam Anderson

Isaac Martin

Reviewed

Lee Engel

Pam Anderson

Forward

This document is specific to the lake sampling conducted by MPCA staff. Procedures outlined cover basic agency condition monitoring on all lakes in Minnesota. For further information, please consult the Water Quality Programs Sampling and Monitoring Standard Operating Procedures (September 2006).

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Procedures

Scope and Application

This standard operating procedure (SOP) is applicable to the collection of water samples from lakes, shallow lakes, and reservoirs for the purpose of condition monitoring. It is applicable to samples taken from the surface and at any depth along a vertical column between the surface and bottom. It is limited to samples collected for physical, chemical, and biological (phytoplankton/zooplankton) analysis.

Summary of Method

No single sampling procedure can be applicable to all sampling situations; therefore, no single procedure is recommended. Water samples from surface waters are generally done in one of the following ways:

- **Hand-collected sample –** bottle in hand for collection of surface sample on shallow lakes
- **Integrated sampler –** composite sample collected over the top 2 meters of the water column
- **Depth sample -** sample collected at depth (Kemmerer)

Health and Safety

Staff will use chemicals in the preservation of samples. Material Safety Data Sheets should be consulted for proper handling of these preservatives (sulfuric acid, nitric acid, Lugol's solution, and methanol) to avoid inhalation and eye/skin irritation problems.

Staff should not sample during adverse conditions (presence of lightning, swift current/flooding, gusts/waves greater than the boat can safely navigate). If lightning is present, staff should return to the vehicle (trailer the boat) and wait a minimum of 20 minutes from the last visible lightning flash before returning to the water.

All Department of Natural Resources (DNR) boating safety rules and regulations must be followed. By law, personal flotation devices (PFDs) must be easily accessible (not in storage) when the boat is in operation and /or occupied, including throwable (Type IV) PFDs. Water Quality Monitoring Unit policy requires MPCA staff to wear PFDs while on the water. The motor kill switch should be attached to the boat operator (clip to PFD or wrap around wrist) to prevent loss of control should the operator fall out of the boat.

Cautions and Interferences

Contamination of the sample can occur if the sampling device is not properly rinsed prior to sample collection. For standard sampling equipment (i.e. integrated samplers) the sample device should be rinsed three times from the opposite side of the boat from where the sample will be collected. For depth samplers, the lowering of the device through the water column provides the necessary rinsing.

Sample contamination can also occur if the bottom sediments are disturbed during the sample collection or the release of the anchor in shallow lakes. Should this occur, the sampling device should be emptied, rinsed, and sample collection should be attempted again at a lesser depth to avoid this contact. For depth samples, the sample may need to be taken from a different location on the boat to avoid already disturbed bottom sediments. Should sediment disturbance occur due to the release of the anchor samples must be collected on the opposite end of the boat to avoid stirred sediments being collected.

Personnel Qualifications/Responsibilities

Field staff must be familiar with proper sampling techniques, sample handling, safety procedures, and record keeping. New staff and student workers must be trained and accompanied in the field by experienced staff until competence is assured. Refresher training events are held each spring for permanent field staff; these

must be attended by all returning field staff. Student workers will be provided written SOPs/instruction and be trained in the field.

Equipment and Supplies

A variety of sampling equipment is needed for surface water sample collection. The general equipment needed for lake trips is listed below:

Boat/Canoe/PFDs

Anchor

Paddles

Integrated sampler

Plankton Tow (where applicable)

Depth Sampler

Secchi Disk

Coolers

Ice

Sample bottles

Preservatives (acids, methanol, Lugol's)

Permanent Markers

Field Sheets

Lab Sheets

Filtering kit for chlorophyll-a samples

Sonde/Surveyor

Camera

GPS Unit

Procedure

This section details the steps necessary to collect a sample, process the sample, and prepare it for delivery to the Minnesota Department of Health Laboratory.

Pre-trip requirements

Sonde calibration

Sonde calibration is required for pH, conductivity, and dissolved oxygen. These calibrations should occur a minimum of monthly with the exception of dissolved oxygen, which should be done at the beginning of each sampling day. All manufacturers recommended calibration instructions should be followed. For calibration of Hydrolab meters, see Appendix B.

Boat preparation

Boats used in sampling should have sufficient tire pressure and bearing grease for the trailer and sufficient synthetic oil for the outboard (where applicable) and battery charge to complete the trip. In addition, sufficient PFDs, paddles, anchors, and rope are required. Gunnel and winch straps should be in good working order (no frays). Ensure trailer lights are functional prior to leaving the Field Operations Center (FOC). Inoperable lights must be repaired prior to taking the boat/trailer on the road. Extra boat motor oil should be on board the boat; fuel levels should be checked prior to the first launch of the trip.

Equipment preparation

Equipment should be prepared to complete sampling trip. Ensure the correct number of bottles and preservative necessary to complete all regular and duplicate sampling. On multi-day trips, sufficient tap water is necessary to conduct dissolved oxygen calibration. Coolers, ice, bottles, preservative(s), lake kit (plankton tow, Secchi disk, and Kemmerer), integrated sampler, chlorophyll-*a* filtering kit, field and lab sheets, camera, GPS/maps, and sonde/Surveyor should be loaded into the trip vehicle. Staff should have reviewed the MPCA's aquatic invasive species SOP, DNR's infested waters list, and planned monitoring trip accordingly-including having a spare set of equipment for use in infested waters or ensuring the infested waters are the last lakes on the trip. See page 7 of this SOP for general guidelines.

On shore requirements

If using a sonde other than Hydrolab, please consult your manufacturers instructions to determine how to set up a data storage file.

Surveyor preparation

Prior to storing data, the Surveyor must have a trip file created. This file should then be annotated prior to each sampling event with the lake and site ID. This will expedite data processing.

Create a Manual File

- Turn on Surveyor 4. Select **Files**. Scroll down to **Create** and push the Select key
- Select the **Manual** option. Using the Arrow keys, highlight and Select the letters, numbers, and symbols necessary to name the file. The backspace key should be used to make any corrections to the file name.
- When completed, select the Done key.
- A parameter list is displayed. By default, the enabled parameters are the same as currently enabled on the main display. Add or subtract parameters by highlighting them using the arrow keys and the pushing the Add or Remove key. When all the parameters needed are enabled, push the Done key.
- The Surveyor 4(a) should beep and display a file created message. Press any key and then use the Go Back key to return to the main menu.

Annotate the File

- Select the **Files** option. Select the **Annotate** option. Scroll to the file to annotate. Push the Select key.

· To annotate a new site file, use the arrow keys to highlight and select each number to create a lake ID; make sure the ID numbers are properly separated by hyphens,. The lake ID should look like this: xx-xxxx-xx-xxx. Once the annotation is completed on the **new** line, select the Done key. Use the Go Back button to return to the base menu. This step is repeated for each unique sampling site per trip.

Equipment preparation

Bottles and field sheets for the sampling locations should be labeled (see example below). Bottles and preservative should be loaded into the small boat cooler. Sonde/Surveyor, GPS/maps, camera, and field sheets/field notebook should be loaded into the boat. Ensure the lake kit and integrated samplers are in the boat. ***If you are sending samples to a lab other than MDH, check with the receiving lab to ensure you are labeling bottles properly.***

DNR Lake ID-Site ID	01-0123-00-101
Sample depth (m)	0-2 m

Boat preparation

Remove gunnel straps/tie downs from the boat and trailer. Ensure the plug is in the boat and raise the motor up. Trailer lights should be unplugged from the vehicle unless they are LED. Leave the boat safety chain and winch strap connected to the trailer until it is safely backed into the water. At this point, the vehicle should be moved to the boat launch and backed in. The emergency brake must be on prior to leaving the vehicle and attempting a launch. Care must be taken with the winch, so that the crank arm/handle does not slip loose and cause potential injury to field staff. The winch should be unlocked only when the boat launch rope is tied to the boat and held securely by field staff. Remove the winch strap and safety chain and push the boat into the water or back the boat off the trailer. Once off the trailer, one staff member should remain with the boat at the dock/launch while the other moves the vehicle and trailer to the appropriate parking area.

Sampling requirements

Travel to sampling location

From the dock, travel to the sample location(s) predetermined for the lake via the handheld or boat mounted GPS units. Stop the boat and drop the anchor, ensuring the boat is not drifting. If windy conditions prevail, a second anchor may be necessary to hold the boat in place.

Sample collection

These next steps should occur simultaneously. One staff member will conduct profile sampling and field sheet recording while the other conducts the 'wet' sampling.

Sonde Measurements (Profile)

1. Remove the protective travel/storage cover from the sonde and replace it with the weighted sonde guard.

2. Turn on the Surveyor/sonde.

3. Lower sonde until the probes are just in the water (the seam between the weighted cover and the body of the sonde should be at the water interface). Allow the numbers to stabilize.

4. Press the 'store' button.

5. Scroll to the previously created file that will contain the lake site data, press 'store' button.

6. Write the surface parameter values on the field sheet.

7. Lower the sonde 1.0 meter deeper into the water column, and repeat steps 3 through 5 until the bottom has been reached; after a depth of 10 meters, take a reading every 2.0 meters. Raise the sonde approximately ½ meter from the lake bottom for the final reading.

Field Datasheet Completion

1. Visually assess the condition at the sampling site.

2. On the field datasheet, determined from the 1 to 5 scale provided, record the condition and suitability of recreational use of the lake at the sampling site.

3. Assess other uses occurring on the lake at the time of sampling– fishing, swimming, etc. Note these on the sheet.

4. Note any macrophyte problems that are limiting lake use (curly leaf pondweed, Eurasian milfoil, etc.) on the sheet.

5. Note weather conditions on the field sheet.

Photo – July only, unless unusual conditions present

1. Take a photo of the field datasheet header box (with lake name, DNR Lake ID, MPCA site and date visible).

2. Take a photo of the lake to the north of the sampling location.

3. Optional photos include algal blooms, macrophyte conditions, or visible scums at the sampling site.

4. Mark on the field datasheet that a photo has been taken.

Surface Sample – for lakes 2 meters or deeper

1. Remove stoppers from the integrated sampler.

2. Lower sampler vertically into the water, stopper the upper end of sampler, remove from water and release cap

3. Repeat two more times.

4. On the opposite side of the boat, lower the unstoppered integrated sampler into the water column until the top is at the water surface. Be sure to keep hands on the outside of the tube and stopper only to avoid contamination.

5. Place the stopper in the tube.

6. Slowly raise the tube so the lower opening is just below the water surface.

7. As the tube breaks the surface, either quickly cap, or allow contents to pour into a clean, open 2 L plastic sample bottle. Again, ensure that hands to do not touch the inside of the bottle or cap.

8. Cap and invert the bottle. Pour contents into 1 L and 250 mL plastic sample bottles.

9. Preserve the 250 mL nutrient bottle with a vial of H_2SO_4.

10. Repeat steps 4 through 7 to collect a second sample for chlorophyll-*a* and algae analysis; move to a new location along the boat for the integrated sample collection. Pour second sample into 2 L bottle.

11. Place all properly labeled bottles in a cooler with ice.

Surface Sample – for lakes less than 2 meters deep

1. Uncap the 2L bottle

2. Tip the bottle upside down and lower it into the water column until the sampler's elbow is at the water surface. Be sure the inside of the bottle and cap are not touched by the sampler.

3. Invert the bottle and allow it to fill.

4. Bring the bottle to the surface, taking care to avoid any surface scum/material.

5. Cap and invert the bottle. Pour contents into 1 L and 250 mL plastic sample bottles.

6. Preserve the 250 mL nutrient bottle with a vial of H_2SO_4.

7. Repeat steps 1 through 4 to collect a second sample for chlorophyll-*a* and algae analysis, moving to a different location along to boat to collect the sample.

8. Place all properly labeled bottles in a cooler with ice. Note, the depth label for this sample should be "Grab" or 0.5 meters.

Depth Sample –

Note only take depth samples at sites greater than 5 meters or sites greater than 4 meters with significant stratification

1. Open Kemmerer sampler by pulling the two caps outwards until they lock.

2. Slowly lower the Kemmerer over the side of the boat to the desired depth.

3. Release the messenger(s) down the taut rope to trip the closing mechanism. (If it is a deep lake, it may require two messengers).

4. Raise the Kemmerer to the water surface. There may or may not be a stop valve on the sampler – be sure you have your bottle ready (uncapped).

5. Ensure there is no sediment in the sample. If there is, discard the sample and repeat steps 1 through 4 from the opposite side of the boat, going to a lesser depth to avoid bottom sediments.

6. Drain the contents of the Kemmerer into a 250 mL sample bottle.

7. Preserve the sample with a vial of H_2SO_4.

8. Place sample in cooler.

Zooplankton Sample – Sentinel Lakes only

1. Check zooplankton net to ensure the stop valve, basket, and lowering rope are all securely attached.

2. Open valve and rinse the zooplankton tow.

3. Close the valve after draining the basket.

4. On the opposite side of the boat, lower the zooplankton net to a depth where the basket is 0.5 meters off the bottom.

5. Slowly raise the tow at a constant rate of 1 foot per second until it breaks the water surface.

6. Rinse the net, without allowing new lake water to enter the top of the net, prior to putting it in the boat.

7. If the basket is full (water still in the net), swirl the contents so that water is able to drain through the basket.

8. Unscrew the collection cup and drain the contents of the basket into the properly labeled sample bottle.

9. Separate the basket from the net.

10. Rinse the basket with deionized water to free any remaining zooplankton.

11. Add this remaining sample to the sample bottle.

12. The label for this bottle, in addition to DNR Lake ID, must also contain length of tow, lake name, and date of sample.

Secchi Transparency

1. Remove sunglasses or polarized eyeglasses and move to the shady side of the boat.

2. Lower the Secchi disk over the side of the boat.

3. When the disk just disappears from view, stop and note this depth.

4. Slowly raise the disk until it reappears, stop and note this depth.

5. Average the two depths and record this value on the field sheet to the nearest 0.1 meter.

Return to launch and trailer boat

Upon completion of sampling, return the boat to the dock/launch. Be sure to raise the motor prior to loading the boat onto the trailer. All switches should be shut off and if any water was taken on, the bilge pump should be run to empty the boat. One field staff stays at the dock with the boat while the other backs the vehicle into the water. Load the boat onto the trailer; walk on the trailer only if decking is in place, otherwise use waders or knee boots to assist with the loading of the boat. Once the winch strap is attached to the boat, lock the winch prior to cranking in the boat to avoid injury from a free-spinning crank handle. Secure the safety strap and pull the boat away from the launch area.

On shore requirements

Sample preservation

1. Once trailered, move vehicle/boat away from access.

2. If not preserved in the boat, add preservative to the nutrient (250 mL plastic bottles) and place in large cooler with ice.

3. If collected, zooplankton samples should be preserved with methanol and label should be inserted inside the sample bottle.

4. Place all remaining bottles on ice in the large coolers.

Invasive species field decontamination

5. Visible aquatic plants and animals must be removed from the boat, motor, and trailer prior to transporting the trailer away from the landing parking lot.

6. Water should be drained from the boat and the motor after each lake. Ensure that the boat plug is removed during travel. Replace the boat plug before launching at the next lake.

7. Sampling equipment and boats should be sprayed with a pressure washer if plant residue remains after initial cleaning.

8. If the lake to be sampled is known to have invasive species, this should be sampled at the end of a trip and/or should be sampled with separate equipment (i.e. for spiny water flea). If necessary, stop at a car wash and spray down the boat to minimize the possibility of transferring species between lakes. ALL BOTTLES FROM AIS LAKES MUST HAVE "AIS" CLEARLY WRITTEN IN GREEN INK ON THE BOTTLE AND/OR LID.

9. Gunnel straps should be secured to the boat.

10. The motor should be returned to the travel position (varies by manufacturer).

11. Trailer lights should be plugged back into the vehicle.

End of day requirements

Sample processing – chlorophyll-*a*

To prevent the spread of invasive species, Chlorophyll-a processing is never to be done at any boat landing or in the parking lot of any boat landing. Additionally, remaining sample water should not be discarded in or near stormwater drains.

1. Remove chlorophyll-*a* samples from cooler.

2. Assemble chlorophyll-*a* kit.

- Place filter holder in the flask.
- Attach brake bleeder pump to flask.
- Place glass fiber filter (rough side up) on the filter holder using forceps.
- Wet filter with DI water

- Place funnel on top of filter and tighten

3. Invert chlorophyll-*a* sample several times to mix the sample.

4. Pour sample into graduated cylinder. Shallow lakes may need as little as 50 mL; deep, clear lakes may need up to 1000 mL of sample.

5. Pour known amount of sample into funnel. Be sure to note the volume of the sample being filtered.

6. Draw the liquid through the filter using the hand pump (electric pump, if at the FOC). Do not exceed 15 PSI.

7. Check color of filter

- If no color is visible, repeat steps 4 through 6 and total the volume filtered.
- Filter should be pale yellow/green to green in color
- If not all of the water will draw through the filter, you must discard the remaining water in the funnel and the filter, rinse the funnel, and repeat steps 2 through 7 again.

8. Once proper color is achieved and all liquid has drawn through the filter, loosen and remove the funnel.

9. Using forceps, fold the filter in half (colored side on the inside).

10. Place in petri dish.

11. Label Petri dish as follows:

12. Write volume filtered on field sheet.

13. Wrap Petri dish(es) in foil, place in plastic bag, and put in ice filled cooler with remaining samples.

14. Disassemble filtering kit and place back in travel case.

End of day sample processing

SENTINEL LAKES ONLY – phytoplankton and isotope sample processing

15. Cap and invert the 2 L bottle of remaining sample until well mixed.

16. Pour off 40 mL of sample into bottle provided by lab.

17. Using a pipette, add Lugol's solution to the sample until amber in color (1 to 2 mL should be sufficient).

18. Cap the bottle and label as follows:

- Lake Name
- Lake ID
- Site ID
- Date

19. Fill a 125 mL bottle to the top with water from the well mixed 2L sample.

20. Label the bottle with the following:

- Lake Name
- Lake ID

- Site ID
- Date

21. Discard remaining sample water from 2 mL bottle.

22. Store the algae sample in a dry location; it does not need to be on ice.

Post-trip requirements

End of trip processing

1. Unload all samples from vehicle – transfer to wet lab in FOC.

2. If unprocessed chlorophyll-*a* samples remain, complete processing before proceeding to step 3.

3. Organize bottles and field sheets by lake.

4. Fill out the chain of custody (COC). If using a lab outside of MDH, be sure to check with the specific lab to get instructions on which COC to use and how to fill it out properly. Please note the following:

- Only one chain of custody can be used per EQuIS Project ID (i.e. PRJ07081, PRJ07082).
- Include the correct lake ID, site ID, date, and time. Any mistakes at this point will be stored in EQuIS incorrectly.
- Surface and depth samples must be separated into their own sample rows on the chain of custody. Depth recorded for surface should be 0-2 m if the integrated sampler was used, 0.5 m – 0.5 m if a subsurface grab sample was taken, or X m – X m for a depth sample (X = the depth the sample was taken).
- Transfer the volume of sample filtered for chlorophyll-*a* samples to the chain of custody.
- Duplicate samples (QC-FR, trip blanks, etc.) must be recorded in their own sample row.
- Include the analysis group code (specific to each project) and any preservative used in the sample.

Parameters	Programs	Analysis Group
Total Suspended Solids, Suspended Volatile Solids, TOC, Total Alkalinity, Total Phosphorus, Total Kjeldahl Nitrogen, Total NO2+NO3, Total Chloride, Chlorophyll-*a*, Pheophytin-*a*	SY – PRJ07081 RZ – PRJ07138	5 (primary surface sample)
Total Phosphorus, Chlorophyll-*a*, Pheophytin-*a*	SY – PRJ07081 RZ – PRJ07138 MI – PRJ07082	6 (replicate/secondary site sample)
Total Phosphorus	SY – PRJ07081 RZ – PRJ07138	7 (deep only)
Total Phosphorus, Chloride	MI – PRJ07082	7 (deep only)
Hardness, Sulfate	SY - PRJ07081	8 (May only primary surface sample)
Total Suspended Solids, Suspended Volatile Solids, Color, Total Alkalinity, Silica, Total Phosphorus, Total Kjeldahl Nitrogen, Total NO2+NO3, Ortho-Phosphorus, Ammonia, Total Organic Carbon, Dissolved Organic Carbon, Total Sulfate, Total Chloride, Chlorophyll-*a*, Pheophytin-*a*, Dissolved Metals Scan (Fe, Ca, K, Na, Mg)	MI - PRJ07082	8 (primary surface sample – full suite April/May, August, October)
Total Suspended Solids, Suspended Volatile Solids, TOC, Total Alkalinity, Total Phosphorus, Total Kjeldahl Nitrogen, Total NO2+NO3, Total Chloride, Chlorophyll-*a*, Pheophytin-*a*	MI - PRJ07082	9 (primary surface sample – partial suite June, July, September)
Silica, Ortho-Phosphorus, Ammonia, Total Organic Carbon, Dissolved Organic Carbon, Total Sulfate, Total Chloride, Dissolved Metals Scan (Fe, Ca, K, Na, Mg)	MI - PRJ07082	10 (replicate sample)

Upon completion of the chain of custody be sure to sign and date the document. At that point the samples should be transferred to the Lakes Monitoring Cooler (at the FOC) and the signed chain of custody sheets are to be placed in the outgoing lab sheet folder on the cooler or FOC office doors. Aluminum foil wrapped chlorophyll-a samples must be placed in the freezer; the foil should be labeled with the name of the trip and date.

- Save the electronic COC in the appropriate folder on the X drive. This contains the field data EDD for loading into EQuIS.

Processing Zooplankton, Phytoplankton, and Isotope Samples (SENTINEL ONLY)

Place preserved sample bottles on the designated shelf in the FOC. They will be transferred to the lab at the MPCA in batches as analysis is completed. Be sure they are labeled properly – including sample date.

Equipment removal

Remove all equipment from vehicle with the exception of the roadside repair items tote. Place chlorophyll-*a* kit on the Lakes shelves with the container open so the kit can air dry, and put the sonde back in the locked

Sonde cabinet. The digital camera, GPS, surveyor and field sheets should return to the office for processing. The Hydrolab Surveyor may be downloaded at the FOC or in the office. 2L sample bottles should be washed, placed upside down in the storage tote and left on the floor in front of the lakes shelves to dry. Unused, clean bottles should be returned to the appropriate clean bottle bins. Coolers should be drained and placed upside down to dry in one of the FOC drains. Dry coolers should be returned to the Water Quality Monitoring Unit shelves. Rain gear and any other personal items should be removed from the vehicle and returned to their normal storage locations (locker, desk, etc.). Once the vehicle is cleaned, return it to Red Lot parking. Note mileage and complete vehicle log for that trip.

Boat Clean Up

Put boat and trailer back into the FOC. Remove life jackets from stow away compartments and lay over the edge of the boat/seats to dry. Inflatable life jackets should be stored in a secure location (i.e. locker). Open up lake kit and lay out ropes and the plankton tow so they will dry between trips. Boats and trailers should be sprayed down weekly to remove excess dirt, any remaining plant/algae residue, etc. Ensure that all the switches are in the 'off' position so the battery is not drained for the next staff person. If any problems arose with the boat or trailer during the trip, alert other Water Quality Monitoring Unit and FOC staff of the problem immediately, so the boat/trailer is not taken out until fixed. Take appropriate steps to fix/correct the problem with the boat or trailer.

Surveyor Downloading to PC

After each trip, bring the Hydrolab back to the office to download the data.

1. Set up PC to receive data

- Open HyperTerminal Start >Programs>Accessories>Communication>HyperTerminal
- Name the HyperTerminal *Connection setting*
- Set Connect using COM1
- Set bits per second =19200
- Data bits = 8
- Parity = None
- Stop bit = 1
- Flow control = Xon/Xoff

2. Downloading data from Surveyor to PC

- Connect to PC using 9 pin connector
- Open HyperTerminal
- From menu go to *transfer* then *receive file*
- Set download file destination - X:\Agency_Files\Water\Condition Monitoring\Lakes\2010 Lake Monitoring\Hydrolab
- Set *receiving protocol* as Xmodem
- Specify file name as start date with the extension "csv" i.e. 14june05.csv
- From the Surveyor 4 menu select Files than Transmit
- Select the File you wish to Transmit
- Select the SS importable option for transfer

- Press any button the Surveyor 4 to start download
- After transfer close HyperTerminal
- Delete the file from the Surveyor 4
- Open the file and make sure each profile was annotated. If not, copy the format of annotated profiles and enter in the lake name. Contact primary staff from trip with questions if you are unable to determine which lake should be entered in the annotation.

Processing Photos

At the end of each trip, photos should be downloaded and labeled in the office. The following steps should be taken to ensure that the name format and storage locations are the same for all lakes sampled.

1. Download the photos to the X drive (X:\Agency_Files\Water\Condition Monitoring\Lakes\Lake Photos).

2. Rename the photos with the following name:

- YY_MMM_xx-xxxx-x-xxx.jpg
  - YY = two digit year
  - MMM = first three letters of month
  - xx-xxxx-xx-xxx = DNR Lake ID-MPCA Site ID
  - A = A for first photo, B for second...

3. Delete the photos of the field sheets.

4. Delete the photos off the camera.

5. Charge batteries if necessary.

Appendix A – Harmful Algal Bloom Sampling Procedure

Scope and Application

This standard operating procedure is applicable to the collection of water samples from lakes, wetlands, reservoirs, and streams for the purpose of documenting suspected harmful algal blooms (HAB). These procedures and equipment are in addition to those addressed earlier in this SOP. These HAB samples are not routine and should not be collected unless directly explicitly as part of a response to an animal death or human illness.

Health and Safety

HAB toxins include compounds affecting skin, internal organs, and the nervous system. Cases of human illness related to HAB exposure are very rare. Prevent toxin exposure by minimizing contact during sample collection by using protective clothing (gloves and waders).

Symptoms can be immediate or arise several days after exposure; they include:

- Liver toxicity – may take hours or days for symptoms to appear in animals and humans; they include abdominal pain, diarrhea, and vomiting,
- Kidney toxicity – acute, severe gastroenteritis (including diarrhea and vomiting),
- Neurotoxicity – often appear within 15 to 20 minutes of exposure; animals may experience increased salivation, weakness, staggering, convulsions, difficulty breathing, and in severe cases, death. Humans may experience numb lips, tingling fingers and toes, or dizziness,
- Respiratory problems – runny eyes and nose, sore throat, and asthma-like symptoms,
- Skin irritation – visible rash, hives, or blisters, especially under clothing, swimsuits, or wetsuits.

Treatment typically involves removal from exposure, rinsing contact area and continued symptom monitoring. Symptoms often resolve within a few hours or days without medical attention. In cases of prolonged or severe symptoms, seek medical attention or call the Poison Control Hotline at 800-222-1222.

Equipment and Supplies

Field Sheets	Sampling rod
Rinse water	250 mL plastic bottle (nutrient)
Nonabrasive soap	Quart sized plastic bags
Brush	Waterproof gloves
Waders	Paper towels

Procedure

This section details the steps necessary to collect a sample, process the sample, and prepare it for delivery to the Minnesota Department of Health Laboratory.

Pre-trip requirements

Recon/Investigation

Gather information on the incident location and contacts before visiting the site. Attempt to have contact (i.e. dog owner or complainant) meet staff at the incident location.

Site Selection

For animal death or exposure incidents, sample at location of exposure. If there is no exposure issue or if the location is unknown, select an accessible location where the bloom is most concentrated, preferably a floating scum. Consider multiple sites in large blooms.

Sampling Requirements

Travel to Location

Gather sampling equipment and travel to the sampling site (via boat or shore).

Field Datasheet Completion

Staff should record appearance and extent of bloom on the field datasheet. Note and photograph any signs of a toxic event (i.e. dead fish or animals). Label bottles according to the directions in the Lake Water Quality Monitoring SOP with the addition of a site description and "toxic algae sample."

Algae Sample Collection

1. Put on gloves and waders

2. Extend the sampling rod to the scum and collect a sample at the surface.

3. Transfer some of the sample to the 250 mL plastic nutrient bottle, cap.

4. Pour 40 mL of the sample into a phytoplankton (algae) bottle, cap.

On-shore/vehicle – sample processing and decontamination

1. Rinse and dry outside of bottles.

2. Preserve the phytoplankton sample with Lugol's solution.

3. Place nutrient and phytoplankton bottles inside a plastic bag, seal.

4. Transfer samples to a cooler with ice immediately.

5. Rinse any equipment that was in contact with the sample water (sampling rod, waders, etc.)

6. Discard the plastic gloves.

On-shore/vehicle – Interview

1. Incident contact should be interviewed. Questions include:

- What were weather conditions prior to the bloom?
- Have these conditions occurred before? If so, how often?
- When was the exposure, if any?
- How much exposure occurred (volume of water consumed or amount of time in contact with water)?
- When did symptoms occur, if any?
- Was treatment sought (for pets or humans) and if so, what was it?
- Was medical or veterinary attention sought? If so, where were the services provided?

Post-trip Requirements

Sample analysis

1. Complete a separate MDH lab sheet for algal toxin samples using analysis code 355 and billing code PC.

2. Toxin samples need to be received by the MDH laboratory within 48 hours.

3. Phytoplankton samples need to go to the Biological Lab at the MPCA St Paul Office.

Documentation

1. Responding staff completes the algae incident tracking spreadsheet.
2. Responding staff should email section staff on the incident.

Appendix B – Hydrolab Calibration Procedures

To Calibrate Conductivity, pH, and Dissolved Oxygen:

DETERMINE THE TYPE OF DO PROBE ON THE SONDE YOU ARE CALIBRATING – USE THE APPROPRIATE METHOD TO CALIBRATE. CHECK THE CALIBRATION STANDARDS EXPIRATION DATES.

Conductivity: conductivity requires a two-point calibration. You need to calibrate your sensor to "0" first, then to the value of the slope standard you are using.

1. Rinse the sensors with deionized water vigorously for 6 seconds. Discard rinse water, and repeat.
2. Dry the conductivity probe with Q-tip or soft cloth.
3. Select Setup/Cal, then Calibrate, then Sonde, then use the arrow keys to scroll down to Cond, SpCond: µS/cm, again using the arrow keys, select 0, press Select, then Done. SpCond will read 0.000.
4. Rinse with the conductivity standard you are using for the slope calibration. Discard the rinse solution.
5. Completely fill the calibration cup with this standard. *DO sensor* must be covered. Allow to equilibrate until conductivity readings are stable (one to three minutes).
6. Select Setup/Cal, then Calibrate, then Sonde, using the arrow keys scroll down and select SpCond: µS/cm. Use arrow key to select the value of the standard in use, press Select, then Done. SpCond will read the value of the standard, and must remain stable.
7. Repeat step 1.
8. Rinse with the standard you prefer to use for linearity check. Discard rinse solution, and fill calibration cup completely with the standard. *DO sensor* must be covered. Allow to equilibrate until stable. Your reading must be within 1% + 1 digit of the value of the standard. This is a linearity check. Do not calibrate at this point.

To calibrate pH repeat step 1 then proceed to step 9.

9. Rinse sensors using the "zero" buffer (value between 6.8 and 7.2). Discard rinse solution, and fill the calibration cup with this buffer. *DO sensor* must be covered. Allow readings to stabilize (from one to three minutes).
10. Calibrate pH, select Setup/Cal, then Calibrate, then Sonde, using arrow keys scroll down and select pH:Units, again use arrow keys to select the value of the buffer, press Select, then Done. The pH reading must remain stable.
11. Repeat step 1.
12. Rinse with the pH buffer that will be used as a "slope" buffer (value near that of the anticipated samples you will be measuring, use pH 10 buffer solution for MN Lakes). Discard rinse solution. Fill the calibration cup, *DO sensor* must be covered.
13. To calibrate pH, select Setup/Cal, then Calibrate, then Sonde, and scroll down to pH:Units, and using arrow keys, select the value of the buffer, press Select, then Done. pH units will read the value of the standard and must remain stable. Discard solution and rinse.

To Calibrate Membrane DO – THIS SHOULD BE DONE DAILY.

14. Fill the calibration cup with DI water to just below the O-ring of the DO probe.
15. Using a kimwipe or other soft towel, carefully remove any water droplets from the DO membrane. Do not apply pressure to the membrane.
16. Cover the calibration cup loosely with the plastic storage lid, and allow unit to equilibrate until readings are stable.

17. To calibrate DO, select: Setup/Cal, then Calibrate, then Sonde, and scroll down to Oxygen, DO%:Sat, using arrow keys select your current Barometric pressure (mmHg,), press Select, then Done. DO% Sat reading will be 100.0.

To Calibrate LDO – THIS SHOULD BE DONE MONTHLY.

18. Fill the calibration cup with approximately ½ inch of deionized or tap water.
19. Using a kimwipe or soft cloth, remove any water droplets from the sensor cap and temperature probe. Be sure to keep the sensor and calibration up out of direct sunlight and away from any heat source during calibration.
20. Gently set the sonde with sensors down into the calibration cup blocking air exchange with the outside environment. Do not screw the calibration cup fully onto the sonde – just enough to block air exchange.
21. Allow the dissolved oxygen and temperature to stabilize a minimum of 3 minutes after the temperature sensor stabilizes.
22. Determine the barometric pressure for entry as the calibration standard (uncorrected for sea level, mmHg). Enter in the field provided.
23. Click calibrate.

To Check Depth (or Level)

24. Replace calibration cup with the weighted sonde guard. Suspend unit, sensors down, into a bucket of water so the intersection between the weighted sonde guard and the sonde is at the water surface. Select Setup/Cal, then Calibrate, then Sonde, then Depth: meters, and using arrow keys select 0, press Select, then Done.

To calibrate Redox:

25. Rinse the sensors with deionized water vigorously for 6 seconds. Discard rinse water and repeat.
26. Rinse with (*the standard you are using to calibrate with*) and discard rinse solution.
27. Fill the calibration cup with (*the standard being used for calibration*). Allow to equilibrate until ORP mV values are stable.
28. Select Setup/Cal, ORP:mV, enter the appropriate value from the chart below and press Select, and Done. ORP is now calibrated. You may do a linearity check with another standard, but do not calibrate a second point.

Temperature Dependency of Common ORP Standards

(All values are vs. NHE; to use the 3M KCl silver chloride scale,

subtract 200 mV from each reading.)

Calibration Solution	mV vs. NHE				
	10º C	15º C	20º C	25º C	30º C
Zobell's	461	450	439	428	417

Calibration Solution	Formula for mV Values
Zobell's	mV = 428 - [2.2 * (T - 25)]

*T is temperature, in ºC

Appendix C – Preservation and Holding Times

Parameter	Sample Collection Method	Container Type	Preservation	Holding Time	Parameter Code
Dissolved Oxygen	Meter reading measured just below the surface	Measured in the field w/ multiprobe meter	None	Instantaneous	n/a
pH	Meter reading measured just below the surface	Measured in the field w/ multiprobe meter	None	Instantaneous	n/a
Specific Conductance	Meter reading measured just below the surface	Measured in the field w/ multiprobe meter	None	Instantaneous	n/a
Temperature	Meter reading measured just below the surface	Measured in the field w/ multiprobe meter	None	Instantaneous	n/a
Total Suspended Solids	Composite grab sample	1 1000-mL general chem.	4°C	7 days	3
Total Suspended Volatile solids	Composite grab sample	1 1000-ml general chem.	4°C	7 days	4
Color	Composite grab sample	1 1000-mL general chem.	4°C	2 days	12
Total Alkalinity	Composite grab sample	1 1000-mL general chem.	4°C	14 days	22
Dissolved Silica	Composite grab sample	1 1000-mL general chem. - dissolved	4°C	28 days	50
Total Phosphorus	Composite grab sample	1 250-mL nutrient	10% H_2SO_4 @ 4^0C	28 days	59
Kjeldahl Nitrogen	Composite grab sample	1 250-mL nutrient	10% H_2SO_4 @ 4^0C	28 days	68
NO2+NO3	Composite grab sample	1 250-mL nutrient	10% H_2SO_4 @ 4^0C	28 days	69
Ortho-Phosphorus	Composite grab sample	1 1000-mL general chem. - dissolved	4°C	28 days	70
Ammonia (NH3)	Composite grab sample	1 1000-mL general chem. - dissolved	4°C	28 days	77
Total Organic Carbon	Composite grab sample	1 250-mL nutrient	10% H_2SO_4 @ 4^0C	28 days	98
Dissolved Organic Carbon	Composite grab sample	1 1000-mL general chem. - dissolved	4°C	28 days	99
Chloride	Composite grab sample	1 1000-mL general chem.	4°C	28 days	297
Sulfate	Composite grab sample	1 1000-mL general chem.	4°C	28 days	293
Chlorophyll a	Composite grab sample	45 micron glass fiber filter and Petri dish	4°C	30 days	450
Pheophytin	Composite grab sample	45 micron glass fiber filter and Petri dish	4°C	30 days	451
Dissolved Metals Scan (Fe, Ca, Mg, Na, K)	Composite grab sample	1 250 mL metals – lab filtered -**do not preserve**	4°C	180 days	703