

Lake Sampling Procedures – LTT Water Quality, Revised 2017, EGAD # 3200-2017-29

Overview

Water quality monitoring on Long Term Trend lakes includes total phosphorus and Secchi depth during spring overturn and collection of additional parameters 3 times during a summer index period (15 July - 15 September). These parameters include components of the Trophic Status Index or TSI (total phosphorus, Secchi disk, and chlorophyll *a*) and field profiles for dissolved oxygen, temperature, and conductance. This should be done once every 2 to 3 weeks. Other water quality parameters collected once each summer include pH, conductivity, alkalinity, color, nitrate + nitrite and total Kjeldahl-N. Calcium and magnesium are sampled once every 5 years. Please check the SWIMS database to determine which lakes are due for Ca and Mg sampling.

Every attempt should be made to coordinate with the Citizen Lake Monitoring Network (CLMN) volunteers to maximize efficiency. For example, if CLMN volunteers are collecting TSI data (“chemistry”), DNR staff do not need to do that portion of the protocol. However, staff may need to collect DO and temperature profiles if the volunteer does not, and should be prepared to visit the lake once during the summer index period to collect the expanded list of parameters along with Secchi, TP, and chlorophyll. CLMN volunteers do not currently collect pH, alkalinity, conductivity, color, N-series, Ca or Mg.

Similar coordination should happen if partners such as USGS, UW Madison or Stevens Point, or the Corps of Engineers are collecting data as part of a lake grant. It is possible that DNR staff will still need to visit a lake once during the summer index period, even if one of these entities is regularly sampling the lake. In addition, **the field data from these partners needs to be entered into SWIMS!** Currently, these partners do not enter their data into SWIMS. **Lakes coordinators are responsible for ensuring this happens.**

It is also critical that equipment is maintained in prime condition and is disinfected to prevent the spread of invasive species. Protocols are appended to this document. In 2013-2014, Dick Lathrop and Katie Hein verified the temperature readings of all meters against a certified thermometer. This shall become an annual activity.

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Table 1. LTT Lake Water Quality Monitoring Protocol Summary

| <u>Parameter</u> | | <u>Protocol details</u> |
|--|---|--|
| *Secchi | Method Equipment Frequency QA/QC | <ul style="list-style-type: none"> To nearest 0.1 m or ¼ ft 8 inch black and white disk Spring turnover + 3X during summer index period (15 July – 15 Sept) Paired observers at least 10% of time, record both readings separately |
| *Total P* | Method Equipment Frequency QA/QC | <ul style="list-style-type: none"> Field fixed, persulfate digestion 2m integrating sampler Spring turnover + 3X during summer index period (15 July – 15 Sept) Field reps and blanks on 10% of samples |
| *Chl a | Method Equipment Frequency QA/QC | <ul style="list-style-type: none"> Water filtered in your own lab (or mail water to arrive at SLH by 2:00), fluorometric 2m integrating sampler 3X during summer index period (15 July – 15 Sept) Field reps on 10% of samples |
| *DO and Temperature profile* | Method Equipment Frequency QA/QC | <ul style="list-style-type: none"> Profile at 1 m intervals DO meter or multi-parameter sonde Spring turnover + 3X during summer index period (15 July – 15 Sept) Calibration record |
| Conductance & pH profile (field) - optional | Method Equipment Frequency QA/QC | <ul style="list-style-type: none"> Profile at 1 m intervals Multi-parameter sonde Spring turnover + 3X during summer index period (15 July – 15 Sept) Calibration record |
| Conductivity, pH, and alkalinity (lab) | Method Equipment Frequency QA/QC | <ul style="list-style-type: none"> Field collected, standard lab methods 2m integrating sampler 1X during summer index period (15 July – 15 Sept) Field reps and blanks on 10% of samples |
| Color | Method Equipment Frequency QA/QC | <ul style="list-style-type: none"> Field collected 2m integrating sampler 1X during summer index period (15 July – 15 Sept) Field reps and blanks on 10% of samples |
| Nitrogen series: NO ₂ +NO ₃ , TKN, <u>Total Nitrogen</u> | Method Equipment Frequency QA/QC | <ul style="list-style-type: none"> Field fixed (sulfuric acid) 2m integrating sampler 1X during summer index period (15 July – 15 Sept) Field reps and blanks on 10% of samples |
| Ca, Mg | Method Equipment Frequency QA/QC | <ul style="list-style-type: none"> Field fixed (nitric acid) 2m integrating sampler Once every 5 years. Check SWIMS to determine sample years. Field reps and blanks on 10% of samples |

* Citizen Lake Monitoring Network (CLMN) may collect samples for these parameters

Procedures for Spring Sampling

Gear List:

Meters, field measuring devices:

- Meter(s) for measuring dissolved oxygen, temperature and conductance profiles and, if available, pH
- Black/white Secchi 8-inch diameter disk with line measured in 0.1 m intervals (or 0.25 feet)
- Depth finder

Water sampling gear:

- Integrating water sampler to sample top 2 meters of water (see Appendix E)
- Composite bottle (and spare) fitted with emptying device that dislodges the ball check valve in the integrating sampler (half gallon or 2 L jug works well)

Sample bottles and preservatives:

- 250 mL bottle from State Lab of Hygiene (SLH) labeled with the field number and lake name
- Sulfuric acid ampoule for preserving sample (from SLH)

Miscellaneous:

- SLH form 4800-024
- Ziploc bags
- Sharpie pen
- Plastic gloves
- Cooler
- Cubed Ice

Field Procedures:

Lakes should be sampled in spring prior to thermal stratification. In the morning at the laboratory, calibrate your temperature/dissolved oxygen or multiparameter meter and record results in your calibration log.

In the field, anchor boat at the sampling station, which should be away from shore at the point of maximum depth. The Station ID should be linked to GPS coordinates in SWIMS and can be used to locate the correct sampling location. Note who the observers are, date, time, and weather conditions including: % cloud cover, air temperature, precipitation, wind speed and direction, water color, user perception of water quality, lake level, algae development.

Go metric where possible; otherwise consider 3 feet as the interval unit for measurement in profiles and water sampling depth. Be sure to note on field sheets (see Appendix D) whether you use feet or metric units. It is very important to note what depth(s) you sampled on the lab slip! If not, the data will not be used!

Temperature/Dissolved Oxygen Profile:

Measure dissolved oxygen, temperature and conductivity at 1 meter (or 3 feet) intervals from the surface (0 m) to the nearest whole meter off the bottom. If you have the capability, also measure pH at 1 meter intervals. Record measured depth at sample site.

Secchi:

Measure Secchi disk transparency to nearest tenth of a meter or ¼ foot. If possible, two people should make independent readings and both readings should be entered into SWIMS.

Water Samples:

Collect a water sample for Total Phosphorus with an integrating water column sampler to 2 meters depth. In shallow lakes, a Van Dorn or Kemmerer may be used.

Put on plastic gloves.

Rinse the integrating sampler 3 times with lake water before sample collection and rinse the composite bottle 3 times with water from the integrating sampler.

1. Lower the integrating sampler vertically at a relatively slow speed to 2 meters depth. After reaching 2 m depth, pull sampler up vertically.
2. Empty the contents of the integrating 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 in the pipe. **Do not allow water to run over bare hands or get contaminated.**
3. After gently mixing the water in the composite bottle, fill a 250 mL sample bottle to the neck (not to top) with lake water. Be careful not to contaminate the sample (do not touch the inside of the lid, bottle, or sample water).
4. Put capped bottle in a ziploc bag and store in a small cooler with cubed ice (not blue ice packs). It's important to keep samples cool and away from contamination.
5. On shore, put on gloves and add 1 ampoule of sulfuric acid to acidify to pH < 2. The State Lab has pre-loaded acid ampoules for this purpose. Check the box indicating that the sample bottle has been acidified with sulfuric acid. 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. In the event that the sample is not at the correct pH, SLH will add more acid once they receive the sample.
6. Place all acidified bottles in a single ziploc bag in cooler with ice cubes. Keep samples cool, but not frozen. Refrigerate them if not shipping the same day.

Shipment of Phosphorus Sample:

1. Fill out SLH form 4800-024 (Appendix D). See further instruction in Summer Sampling.
2. Store sample cooled (not frozen) until shipment. Total holding time is 28 days.
3. Ship sample in a cooler on ice to the SLH. Include the completed lab sheet.

Procedures for Summer Water Quality Sampling

Gear List:

Meters, field measuring devices.

- Meter(s) for measuring dissolved oxygen, temperature and conductance profiles and, if available, pH. Measurements should preferably be made at 1 m intervals.
- Black/white Secchi disk with line measured in 0.1 m units (or 0.25 feet)
- Depth finder

Water sampling gear:

- Integrating water sampler to sample top 2 meters of water (see Appendix E)
- Composite bottle (and spare) fitted with emptying device that dislodges the ball check valve in the integrating sampler. A half-gallon (2 L) jug works well.

Sample bottles and preservatives:

- 250 mL polyethylene bottle and nitric acid ampoules for Calcium and Magnesium
- 250 mL polyethylene bottle and sulfuric acid ampoules for Tot.-Phosphorus, Total Nitrogen, Total Kjeldahl-N, and $\text{NO}_2 + \text{NO}_3$ as Nitrogen
- 1 L polyethylene bottle for Alkalinity, pH, Conductivity, Color, and Chlorophyll A
- Extra bottles for blanks and/or field duplicates

Miscellaneous:

- SLH form 4800-024
- Ziploc bags
- Sharpie pen
- Plastic gloves
- Cooler
- Cubed Ice

Field procedures:

Lakes should be sampled during the summer index period (i.e. 15 July to 15 September). In the morning at the laboratory, calibrate your temperature/dissolved oxygen or multiparameter meter and record results in your calibration log.

Anchor boat at the point of maximum depth. The Station ID should be linked to GPS coordinates in SWIMS and may be located with a GPS unit. Note weather conditions, including cloud cover, precipitation, wind speed and direction, etc.

Go metric where possible; otherwise consider 3 feet as the interval unit for measurement in profiles and water sampling depth. Be sure to note on field sheets (see Appendix D) whether you use feet or metric units.

Temperature/Dissolved Oxygen Profile:

Measure dissolved oxygen, temperature and conductivity at 1 meter (or 3 feet) intervals from the surface (0 m) to the nearest whole meter off the bottom. If you have the capability, also measure pH at 1 meter intervals. Record measured depth at sample site.

Secchi:

Measure Secchi disk transparency to nearest tenth of a meter or ¼ foot. If possible, two people should make two independent readings of Secchi depth and both readings should be entered into SWIMS.

Water Sample Collection:

Collect 4 water samples with an integrating water column sampler to 2 meters depth. In shallow lakes, a Van Dorn or Kemmerer may be used.

1. Put on plastic gloves.
2. Rinse the integrating sampler 3 times with lake water before sample collection and rinse the composite bottle 3 times with water from the integrating sampler.
3. Lower the integrating sampler vertically at a relatively slow speed to 2 meters depth. After reaching 2 m depth, pull sampler up vertically.
4. Empty the contents of the integrating 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 in the pipe. Do not allow water to run over bare hands or get contaminated.
5. The 2 m integrating sampler collects ~1 liter of water. Combine as many separate pulls from the integrating sampler as needed to have enough water (almost 2 L) to fill water sample bottles (i.e. two 250-mL bottles and one 1-L bottle).
6. Gently mix water in the composite bottle again, fill the 1-L polyethylene bottle, and immediately place the bottle in a cooler on ice. It is very important to minimize exposure to sunlight! That is why you should fill this bottle first. Because these bottles come directly from SLH, they do not need to be triple rinsed. Do not use the bottle if the lid came off during shipment because it may be contaminated. Be careful not to touch the inside of the lid, bottle, or sample water.
7. Gently mix water in the composite bottle and fill the remaining two sample bottles to the neck (not to top) with water from the composite bottle.
8. 250 mL polyethylene bottle to the neck for TP, TN, TKN and NO₂+NO₃
9. 250-mL polyethylene bottle for calcium and magnesium
10. Put capped bottles in a ziploc bag and store in a small cooler with cubed ice (not blue ice packs). It's important to keep samples cool and away from contamination.
11. On shore, put on gloves and add 1 ampoule of sulfuric acid to nutrients bottle and 1 ampoule of nitric acid to the calcium and magnesium 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. In the event that the sample is not at the correct pH, SLH will add more acid once they receive the sample. It is important to do this after the chlorophyll sample is collected because residue acid degrades chlorophyll.
12. Place all acidified bottles in a single ziploc bag in cooler with ice cubes. Keep samples cool, but not frozen. Refrigerate them if not shipping the same day.

Chlorophyll – Laboratory Filtration

1. If water samples will arrive at SLH before 2:00 pm the **SAME DAY** the samples were taken, you may label the water sample and ship to SLH with the other samples. This will ensure that the sample is filtered the same day that they are collected. If you personally deliver samples, make sure they do not sit out. Chlorophyll should remain in the dark at all times.
2. If the shipment is likely to arrive after 2:00 pm, it is important that you filter the water sample when you return to the office/laboratory on the same day. Do not filter in the field. Reserve at least 200 mL of water after filtration for the alkalinity/conductivity/pH and color tests at SLH. See Appendix C.

3. After filtration, freeze the filter paper.

Shipment of nutrient, Ca/Mg, color/alkalinity, and chlorophyll* samples:

1. Fill out SLH form 4800-024 (Appendix D).
2. Store water samples cooled (not frozen) until shipment.
3. Ship samples in a cooler on ice to the SLH. Include the completed lab slip.

*Shipment time is critical and depends on what analyses SLH will do. Here are shipment times:

- same day: water for chlorophyll a (SLH filters)
- <48 hours: color
- 14 days: alkalinity and conductivity
- 28 days: nutrients
- 30 days: frozen chlorophyll a filter paper
- 6 months: metals

The recommended shipping procedure depends on your location:

- If close to SLH, send all samples to arrive at SLH by 2:00 pm the same day.
- If far from SLH:
 - a) On the midsummer sampling that includes color, filter chlorophyll a yourself, freeze the filter paper, then send all samples early the next morning.
 - b) On the summer samplings without color, filter chlorophyll a yourself, freeze the filter paper, then send all samples within 1 week.

Clean-up

It is VERY important to clean all equipment between lakes and at the end of the day in order to prevent the spread of invasive species. The boat, integrating sampler, and other equipment that goes in the lake (e.g., anchor), should be cleaned according to the most recent disinfection procedures (Appendix F). Another option is to use designated sets of equipment for each lake. This can be accomplished by making multiple integrating samplers. Another possibility is to sample the lake with a citizen volunteer who lives on the lake rather than trailering a DNR boat.

Cleaning equipment also prevents contamination of the water chemistry samples. Constituents like calcium and magnesium may be at very low concentrations. At the end of the day after following disinfection procedures, the composite bottles and integrating samplers should be rinsed with deionized water and left to dry. Hang integrating samplers with ball end at top to allow water to drain out.

Quality Assurance

It is important to test the field precision of our chemical measures. Samples should be collected according to the following procedures:

1. If there are two observers in the boat, both people should make independent Secchi disk observations and record both readings in SWIMS.
2. Meters should be calibrated each sample date for all parameters relevant to each meter. At a minimum, this includes dissolved oxygen (check the barometric pressure at the nearest weather station). Calibration solutions should be used for conductivity and pH. Refer to your meter's operations manual. A log of meter calibration should be kept at each office (see Appendix G for an example).

3. Field duplicates for all chemical samples and chlorophyll should be collected at a rate of 10%. The easiest way to do this (somewhat randomly) is to collect a duplicate set of samples on every tenth lake. Note that this second set of samples needs to have a unique field number. On the lab slip, check “Duplicate” under “If Field QC Sample (select one)”
4. Once a week during the field season (or about 10% of samples), send a set of blanks to the State Lab for all variables except chlorophyll. Get ASTM Type I water (deionized/reverse osmosis water that is then run through a US Filter Corp or Milli Q filter) from SLH. If you supply your own carboy (20 L recommended), SLH will clean it and fill the carboy for free. This should be sufficient for 1 field season. You should also ask them to clean a smaller bottle (4 L should be sufficient) so that you can bring a smaller amount of water with you in the field on days when you will collect a blank. Bring the Milli Q water on the boat with you. After completing the lake water sampling, rinse the integrating sampler and composite bottle three times with Milli Q water by filling the sampler and then emptying it into the composite bottle. Fill the integrating sampler with Milli Q water a 4th time, empty it into the composite bottle, then pour water into a separate sample bottle for each water chemistry parameter you are testing on that day (could include conductivity/pH/alkalinity, color, nutrients (TP/N series), and Ca/Mg). Essentially, you follow the same procedure as you would for a lake sample, but instead pour Milli-Q water into the integrating sampler. Give this set of samples a unique field number. Check “Blank” on the lab slip under “If Field QC Sample (select one)” and write “equipment blank” under the “Sample Description” text field.
 - a. Note: If a run of blanks from one field site have elevated concentrations, then you should also test the Milli Q 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 as long as we are using their Milli Q water, we do not need to send in lab water blanks as a common procedure.

Data Entry

Temperature/DO/Conductance Profiles: Profile data and any Secchi data not reported on your lab slip should be entered into SWIMS. Go to the Submit Data tab and click "Add New". Then choose the Long Term Trends project. Contact Jennifer Filbert if you have any questions.

SLH Inorganic Test Request Form:

Use the Lab Slip Generator in SWIMS (go to the My Projects or Forms tab) to generate your lab slips. Pick the LTT project, the appropriate monitoring station, and the Inorganic Test Request lab slip. Contact Jennifer Filbert or Mollie MacDonald if you have any questions.

The SLH Inorganic Test Request form needs to have the following information included in addition to the usual stuff (like your name, date, time, lake name, county, WBIC, STORET number, etc.).

- On page 2, indicate the top of sampling interval as 0.0 meters and the bottom of the sampling interval as 2.0 meters!
- Check boxes for the following chemical tests:

Plastic quart bottle:

- Color
- Chlorophyll a, uncorrected. Write in the volume of water filtered unless sending the whole sample for SLH to filter.
- Alkalinity, pH and Conductivity

250 mL bottle for metals (acidify with nitric acid):

- Calcium
- Magnesium

250 mL bottle for nutrients (acidify with sulfuric acid):

- Total Phosphorus
- Total Nitrogen
- Total Kjeldahl-N
- NO₂ and NO₃ as Nitrogen

If you collect a replicate set of samples you will need to fill out a separate Inorganic Test Request, giving the second set of samples a unique field number, which you write on both the sample bottles and the Request Form.

Appendix A: Description of Water Samples for SLH

<http://intranet.dnr.state.wi.us/int/es/science/ls/Preservation/IIIA.htm#TABLEIII1>

Preservation Notes For Total Kjeldahl-Nitrogen

Sample Size: 38 mL

Bottle Type: 250-mL polyethylene with white polypropylene cap

Preservative: H₂SO₄ to pH<2, Cool to 4°C

Volume of Preservative: 1 mL 25% (9N) H₂SO₄/250 mL

Holding Time: 28 Days

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

Special Notes: None

Preservation Notes For Nitrogen-Total

Sample Size: 50 ml

Absolute Minimum Volume: 10 ml

Bottle Type: 250 ml polyethylene with white polypropylene cap

Preservative: H₂SO₄ to pH<2, Cool to 6°C, 2 mL

Volume of Preservative: 1 mL 24% (9N) H₂SO₄/250 mL (plastic screw top vial)

Holding Time: 28 days

Compatible With: COD, Nitrate + Nitrite, Total Kjeldahl Nitrogen, and Total Phosphorus.

Special Notes: 125 ml is required for ammonia when sampling wastewater.

Preservation Notes For Total Phosphorus

Sample Size: 38 mL

Bottle Type: 250-mL polyethylene with white polypropylene cap

Preservative: H₂SO₄ to pH<2, Cool to 4°C

Volume of Preservative: 1 mL 25% (9N) H₂SO₄/250 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 175 mL for all parameters

Special Notes: None

Preservation Notes For Nitrate plus Nitrite-Nitrogen

Sample Size: 38 mL

Bottle Type: 250-mL polyethylene with white polypropylene cap

Preservative: H₂SO₄ to pH<2, Cool to 4°C

Volume of Preservative: 1 mL 25% (9N) H₂SO₄/250 mL

Holding Time: 28 Days

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

Special Notes: None

Preservation Notes For Color and Alkalinity

Sample Size: 150 mL

Bottle Type: 1 quart bottle for color and alkalinity

Preservative: Cool to 4°C.

Holding Time: 48 Hours (color) and 14 Days (alkalinity)

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), Dissolved Solids (75m), Suspended Solids (250 mL or 1500 mL low level), 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

Preservation Notes For Metals – Ca and Mg

Sample Size: 250 mL

Bottle Type: 250-mL polyethylene with white polypropylene cap

Preservative: HNO₃ to pH<2

Holding Time: 6 Months

Volume of Preservative: 1 mL 70% (16N) HNO₃ to pH<2

Compatible With: Need Whole Volume

Special Notes: None

Appendix B: Finding Standard Operating Procedures in SWIMS

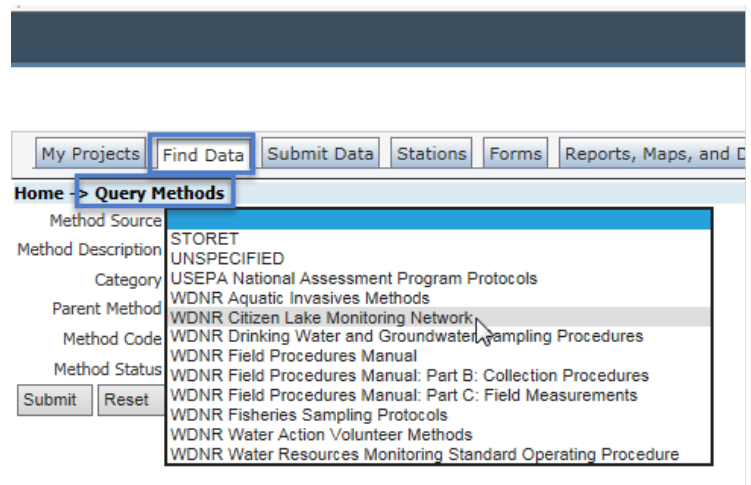
Additional standard operating procedures are stored in SWIMS.

- To find them log into SWIMS.
- Click the Find Data tab.
- Click SWIMS Digital Library and select under 'Document Type' = 'Field Methods, Procedures'.

You can also look up methods by click on "find data", "query methods" and look for DNR and partner methods on that screen.

Documents that could be of use include:

- 2802 LIGHT ATTENUATION, SECCHI DISK
- 2502 TEMPERATURE – ELECTRONIC
- 2501 TEMPERATURE – THERMOMETRIC
- 2201 CONDUCTIVITY METERS
- 2102 WINKLER TITRATION FOR DISSOLVED OXYGEN



- 2001 pH ELECTROMETRIC
- 1004 chlorophyll sample filtration
- Appendix b Inorganic Sample Shipper Instructions
- Quality Assurance Sampling Protocol – CLMN – 2013
- 2007 QUALITY ASSURANCE SAMPLING PROTOCOL CITIZEN LAKE MONITORING NETWORK
- CLMN QA 2007 - ADDITIONAL NOTES ON PROTOCOL
- Determination of pH Field Procedures
- List of Parameters and Detection Limits

Appendix C: 1004 CHLOROPHYLL SAMPLE FILTRATION (updated from DNR Field Procedures Manual and 2005 CLMN protocol)

Scope:

This procedure is for the *filtering* of surface waters to determine the chlorophyll content.

Safety:

NA

Equipment:

- Membrane filtration apparatus
- Millipore SM 5.0 um membrane filters (SMWP 04700, 47 mm diameter, 100/pack). Millipore, Bedford, MA 01730, 800-646-5476
- Vacuum source (hand or electric pump)
- 1000 mL filtration flask
- 500 mL graduated cylinder
- Tubing
- Wash bottle
- Distilled water
- Forceps
- 15 mL plastic centrifuge tube from the State lab with label sticker
- Aluminum foil (4.25" by 3.5")
- Zip lock bag (3" by 5")
- Plastic gloves

Collection Procedures:

- Collect the samples with the procedures given in the surface water sampling section. Be sure to store collected sample on ice and out of direct sunlight.

Sample Handling and Filtration:

- This procedure should be conducted in the laboratory under low light (turn off overhead lights, close blinds, etc.). Do not filter at the field site. Be sure to filter on the same day as the water sample is collected.
1. Place all the equipment on your work area.
 2. Insert the bottom part of the filtering cup (membrane filtration apparatus) into the 1000-mL filtration flask. You may wish to wet the stopper first to get a good seal.
 3. Put on gloves.
 4. Attach the plastic tubing of the vacuum pump to the spout of the filtration flask.
 5. Pick up one small membrane filter with the tweezers and place it on the center of the filter base (the black screen) with the gridded side of the filter paper down and the rough side of the filter paper up.

Note that filters are white, and divider sheets are blue -- be careful to use a filter! Squirt a small amount of distilled water on the filter to keep it in place. Do not touch the filter with your fingers while removing it from the bag or when placing it on the screen.

6. Carefully place the cup on top of the filter base. It is magnetic. Be sure that the filter does not move!
7. Determine the approximate volume of water to filter for the chlorophyll analysis (refer to Table C1). Match the Secchi depth you got today against the volume of water that should be filtered.
8. *Gently* mix the sample by turning the sealed bottle upside down several times. Before the sample settles, pour sample into the graduated cylinder, and record the volume. You will need to record the total volume of water filtered on the lab slip (you may need to fill the graduated cylinder more than once if sampling a very clear lake).
9. Pour the water from the graduated cylinder into the filter apparatus.
10. Squeeze the hand pump or turn on the vacuum pump to move the water through the filter. Once in a while, gently swirl the flask to ensure that algae does not stick to the side of the filter cup.
11. When you are finished filtering, separate the top cup from the filter base.
12. Using the tweezers, fold the filter paper in half so that the algae are on the inside. **Do not touch the algae with your fingers or the tweezers!**
13. Fold the filter paper in half again and place the filter into the chlorophyll tube.
14. Fill out the chlorophyll sticker or label the tube with the sample information. Attach the sticker directly to the test tube. **BE SURE TO INCLUDE THE VOLUME FILTERED!**
15. Wrap the test tube in aluminum foil and place in zip lock bag.
16. Don't forget to write the volume of water filtered on the lab slip.
17. Freeze and mail frozen samples to SLH.

Table C1

Note: These are approximate guidelines - filter as much water as you can.

| Secchi Depth | Volume to Filter |
|--------------|------------------|
| < 1ft | 50 mL |
| 1-1.5 ft | 100 mL |
| >1.5 ft | 200 mL |

Documentation

Complete the *Inorganic Test Request Form* (see Appendix D) with the information on the sampling site, depth of integrated sample, and volume of sample filtered.

References:

Wisconsin Department of Natural Resources, "Self Help Lake Volunteer Training Manual, Chemistry Monitoring Procedures", page 35, January 1995.

Appendix D: Lab slip (log into SWIMS and go to My Projects or Forms)

State of Wisconsin
Department of Natural Resources
and Laboratory of Hygiene

Test Request – Inorganic Surface Water & Microbiology
Form 4800-024 (R 5/14) Page 1 of 2

| | |
|----------------------------------|----------------------------------|
| Billing and Reporting | |
| Account Number | Field Number (Bottle Label ID) |
| DNR User ID | Report To Name |
| Date Results Needed (mm/dd/yyyy) | Report to Address (Non-DNR only) |
| | City State ZIP |
| | Report to Email (Non-DNR only) |

| | | | |
|---|--------------------|-----------------------|----------|
| Date and Time of Sample Collection | | | |
| Date (mm/dd/yyyy) | Time (24-hr clock) | End Date (mm/dd/yyyy) | End Time |

| | | | |
|---|--|--|--|
| Sample Type | | | |
| Sample Type: (select one) | | | |
| <input type="radio"/> SU Surface Water | <input type="radio"/> NP Storm Water | <input type="radio"/> EF Effluent (Treated Wastewater) | <input type="radio"/> IF Influent (Untreated wastewater) |
| <input type="radio"/> D Public Drinking Water | <input type="radio"/> MW Monitoring Well | <input type="radio"/> PO Private Well | <input type="radio"/> SE Sediment |
| <input type="radio"/> SL Sludge | <input type="radio"/> SO Soil | <input type="radio"/> TI Tissue | <input type="radio"/> |

| | | |
|---------------------------------|-----------|-------|
| Who collected the sample | | |
| Collected By Name | Telephone | Email |

| | | |
|---------------------------------------|--|---|
| Where the sample was collected | | |
| Station ID (STORET #) | Sample Address or Location Description | |
| County | Waterbody ID (WBIC) | Point / Outfall (or SWIMS Fieldwork Seq No) |

| | | |
|---|---|---|
| Sample Details | | |
| Sample Description / Device Description | | |
| Enforcement? <input type="radio"/> Yes <input type="radio"/> No | If Field QC Sample (select one): | Depth of Sample: _____ ft <input type="radio"/> m <input type="radio"/> in <input type="radio"/> cm |
| If yes, include chain of custody form. | <input type="radio"/> Duplicate <input type="radio"/> Blank <input type="radio"/> | Or Top and Bottom of Sample Interval: _____ - _____ ft <input type="radio"/> m <input type="radio"/> in <input type="radio"/> cm |
| Is Sample Disinfected? <input type="radio"/> Yes <input type="radio"/> No | Grant or Project Number | |
| If yes, how? | | |

| | | |
|---|--|---|
| Analyses Requested | | |
| If field filtered, indicate by checking the box on this sheet and noting on the lid of the sample bottle. | | |
| Plastic Quart Bottle (No chemical preservation) | | |
| <input type="checkbox"/> Sample field filtered? (Check box if yes) | | |
| <input type="checkbox"/> Alkalinity, pH, Conductivity | <input type="checkbox"/> Color | |
| <input type="checkbox"/> BOD ₅ Dissolved | <input type="checkbox"/> Fluoride | |
| <input type="checkbox"/> BOD ₅ Total (900 ml needed) | <input type="checkbox"/> MBAs Screening | |
| <input type="checkbox"/> CBOD ₅ Total (carbonaceous) | <input type="checkbox"/> pH only (non compliance) | |
| <input type="checkbox"/> Chloride | <input type="checkbox"/> Sulfate | |
| <input type="checkbox"/> Chlorophyll A (if Field Filtered, give ml _____ filtered) <input type="checkbox"/> | <input type="checkbox"/> Turbidity | |
| Solids | | |
| <input type="checkbox"/> Suspended Sediment | <input type="checkbox"/> % Sand, Silt, Clay | |
| <input type="checkbox"/> Total Dissolved Solids | <input type="checkbox"/> Total Suspended Solids (500 ml needed) | |
| <input type="checkbox"/> Total Solids | <input type="checkbox"/> Total Vol. Susp. Solids (includes Total Susp. Solids) | |
| <input type="checkbox"/> Total Volatile Solids (includes total solids) | | |
| 60 ml Bottle (No chemical preservation) | | |
| <input type="checkbox"/> Sample field filtered? (Check box if yes) | | |
| <input type="checkbox"/> Orthophosphate | <input type="checkbox"/> NO ₂ +NO ₃ as Nitrogen (drinking water) | |
| <input type="checkbox"/> Silica | <input type="checkbox"/> Nitrite (NO ₂) as Nitrogen | |
| Quart Mason Jar | | |
| <input type="checkbox"/> Oil Grease (3 qts) | <input type="checkbox"/> pH (waste only) | |
| 250 ml Metals Bottle (Acidify w/ Nitric Acid) | | |
| <input type="checkbox"/> Sample field filtered? (Check box if yes) | | |
| <input type="checkbox"/> Low Level Metals. Note: Clean sampling; Special bottles and acid required. | | |
| <input type="checkbox"/> TCLP (Toxicity Characteristic Leaching Procedure - use mason jar) | | |
| Total recoverable metals will be run unless otherwise instructed. | | |
| <input type="checkbox"/> Aluminum | <input type="checkbox"/> Copper | <input type="checkbox"/> Selenium |
| <input type="checkbox"/> Antimony | <input type="checkbox"/> Hardness-as CaCO ₃ | <input type="checkbox"/> Silver |
| <input type="checkbox"/> Arsenic | <input type="checkbox"/> Iron | <input type="checkbox"/> Sodium |
| <input type="checkbox"/> Barium | <input type="checkbox"/> Lead | <input type="checkbox"/> Strontium |
| <input type="checkbox"/> Beryllium | <input type="checkbox"/> Magnesium | <input type="checkbox"/> Thallium |
| <input type="checkbox"/> Boron | <input type="checkbox"/> Manganese | <input type="checkbox"/> Titanium |
| <input type="checkbox"/> Cadmium | <input type="checkbox"/> Mercury | <input type="checkbox"/> Vanadium |
| <input type="checkbox"/> Calcium | <input type="checkbox"/> Molybdenum | <input type="checkbox"/> Zinc |
| <input type="checkbox"/> Chromium, Total | <input type="checkbox"/> Nickel | |
| <input type="checkbox"/> Cobalt | <input type="checkbox"/> Potassium | |
| 250 ml Nutrients Bottle (Acidify w/ Sulfuric Acid) | | |
| <input type="checkbox"/> Sample field filtered? (Check box if yes) | | |
| <input type="checkbox"/> Tot.-Phosphorus | <input type="checkbox"/> NO ₂ + NO ₃ as Nitrogen | <input type="checkbox"/> Total Kjeldahl-N |
| <input type="checkbox"/> Ammonia-N | <input type="checkbox"/> COD | <input type="checkbox"/> Total Nitrogen |
| <input type="checkbox"/> Tot. Dis. Phosphorus (filter, then acid preserve in 60 ml bottle) | | |
| 250 ml Round Bacteria Bottle | | |
| <input type="checkbox"/> E. coli by MPN, non-potable | For lab use: | |
| <input type="checkbox"/> Enterococci by MPN, non-potable | Sample Temp _____ °C | |
| | <input type="checkbox"/> Iced | |

Please enclose this form in the mailer along with the sample and send to the State Lab of Hygiene.
Additional parameters or instructions to laboratory:

Test Request – Inorganic Surface Water & Microbiology

Form 4800-024 (R 5/14)

Page 2 of 2

Field Parameters - Optional

Only fill out if directed by your project coordinator.

| | | | |
|--------------------------------|--|------------------------|--------------|
| Temperature - Sample (°C) | _____ . ____ | Gage Height (ft) | _____ . ____ |
| Temperature - Ambient Air (°C) | _____ . ____ | Flow (cfs) | _____ . ____ |
| DO (mg/l) | _____ . ____ | Flow (MGD) | _____ . ____ |
| % Saturation | _____ . ____ | Depth to Groundwater | _____ . ____ |
| pH (su) | _____ . ____ | Turbidity (NTU) | _____ . ____ |
| Secchi Depth (feet or meters) | _____ . ____ | Transparency Tube (cm) | _____ . ____ |
| Secchi Depth Hit Bottom? | <input type="checkbox"/> Yes <input type="checkbox"/> No | Nitrates (mg/l) | _____ . ____ |
| Cloud Cover (%) | _____ . ____ | | |
| Cond (µS/CM@25°C) | _____ . ____ | | |

Tips

See Chapter 4 "Lab Slips" of the Field Procedures Manual (see <http://intranet.dnr.state.wi.us/int/es/science/ls/Forms/Instructions.htm>) for further instructions and definitions.

The **Account Number** must be completed in order for the samples to be billed to the correct funding source. If you are unsure what the proper account number is refer to <http://intranet/int/es/science/ls/Account.htm> or contact the DNR Laboratory Coordinator or the State Laboratory of Hygiene.

The **Lake Grant or Project Number field** should include the Lake Planning Grant Number or the Project Number.

Sample Depth – If you sample in a lake, this is required.

Field Parameters – If you do fill this out, the data will go into SWIMS automatically. Please do not re-enter. Also, you must QA the data once it arrives in SWIMS.

Appendix E: Integrating Water Column Sampler (Field season 2000)

Developed by Jim Klosiewski (james.klosiewski@wis.gov)

All of the following materials were purchased from a local hardware store except for the 3/4" plastic ball and 1/4" diameter PVC rod which were purchased from Hoover Precision Products, (906)-632-7310 and McMaster-Carr, <http://www.mcmaster.com/> respectively.

MATERIALS to make one 6.5 foot long sampler

SAMPLER

| <u>QTY</u> | <u>ITEM</u> |
|-------------------|---|
| 1 | 1" inside diameter (ID) x 10 ft. white rigid PVC pipe, schedule 40 |
| 1 | 3/4" PVC ball |
| 1 | 1" x 3/4" PVC male reducing adapter (buy a few, sometimes they are out of round and leak) |
| 1 | 1 1/2" x 1/4" dia. PVC rod |
| 1 | 2 3/4" x 1/4" dia. PVC rod |
| 1 | PVC primer |
| 1 | PVC cement |
| 1 | small tube of Marine Goop (*optional) |
| | black electrical tape |

BOTTOM EMPTYING DEVICE (Built into the collection bottle)

1 Collection bottle with a throat diameter of at least 1 7/8" in diameter. Volume size depends on how much you will need to collect for your sampling projects. Each sampler holds approximately 924 milliliters or 31 ounces of water. A Rubbermaid "juice jug" works well. Any product like this will work well. You can purchase these at your local Kmart/WalMart type stores.

1 piece of 1/4" diameter PVC rod (length will depend on throat diameter of your collection bottle)

Cost to build one sampler including collection container is under \$20.

TOOLS

- Safety glasses
- Drill
- 1/4" drill bit
- Hacksaw
- Hammer

CONSTRUCTION:

Refer to the attached diagram for help.

1. Checking for Leaks – *(this needs to be done before holes are drilled, and before gluing a fitting onto the pipe)*
 - Drop a ball in a male adapter and attach (don't glue at this point) to either end of the pipe. Fill the sampler with water and check for leaks from the bottom opening. Some dripping is OK.
 - If there is excessive leaking try another fitting.

2. Building the Sampler
 - Measure and cut off a 6 1/2 foot piece from the 10 foot PVC pipe. Make sure the cut is straight.
 - Drill a 1/4" diameter hole through both walls of the sampler 1 1/2" from each end.
 - Following the manufacturer's directions on the primer and cement containers glue a male reducing adapter to one end of the sampler. **(GO LIGHTLY ON THE GLUE! Do not use excessive amounts. If too much is used, the over-flow inside the pipe will flow into the fitting and could interfere with the PVC ball seating properly.)**
 - After the cement is dry, drop a ball into the pipe. *(The ball is made of PVC and any excess wet glue in the male fitting could melt the ball.)*
 - Tap the 1 1/2" piece of PVC rod through the 1/4" hole, **making sure the ball is in the fitting, and below the rod.** File any protruding rod flush with the pipe.
 - Cut off the threads on the male adapter. (see diagram)
 - Spread a dab of Marine Goop to seal rod holes.
 - On the opposite end of the sampler, tap in the 2 3/4" piece of PVC rod through the 1/4" hole. The rod will extend out one side of the pipe 1 1/2". This will keep the sampler from rolling on flat surfaces (such as pontoon decks).
 - Holding the sampler upside down, tap it on the floor to empty loose drill cuttings. Rinse the sampler a few times to remove any dirt and remaining cuttings.
 - **Mark 6 feet on the pipe from the fitting end with black electrical tape.** This is your sample depth.
 - With a Sharpie, write on the sampler end with the fitting STORE THIS END UP (See diagram).

3. Building the bottom-emptying device
 - Drill a 1/4" hole through both sides of the throat of the collection bottle. (Drill the holes far enough from the top of the throat opening so the lid can still be attached)
 - Measure the throat diameter at the point of the drilled holes.
 - Cut a length of PVC rod a bit longer than the measurement and push it through the holes. The rod should stay in place due to the tight fit.
 - If the rod interferes with the lid closing, file a bit of the rod off.

You are done!

4. Capacity of the Sampler

- Volume of water held by the sampler equals: **App. 924 milliliters or 31 oz./ 6 feet**

HOW TO USE

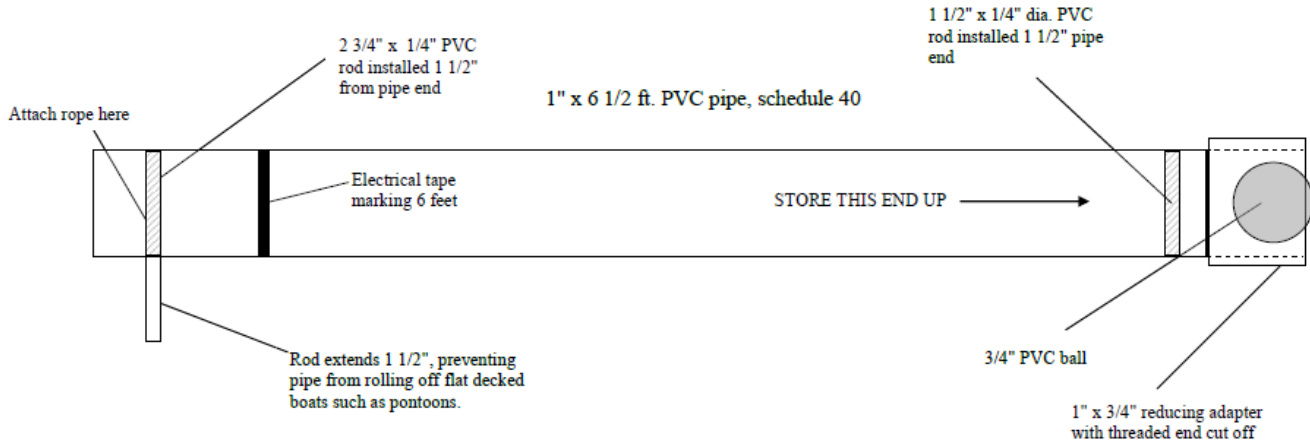
1. Slowly lower the sampler to the marked 6-foot depth. When you have reached the 6-foot mark, raise the sampler slowly, holding it in a vertical position. As you raise the sampler the ball will automatically lock in place, holding the water.
2. Empty the sampler into your collection container. This will happen automatically when you place the fitting end of the sampler in the throat of your bottle and contact the emptying rod.
3. Invert the collection container to thoroughly mix the sample. Pour off the sample from the collection container into appropriate lab bottles.
4. **Before collecting a sample, be sure and rinse the sampler and container with site water.** It is especially important to rinse the sampler and container before sampling another site. This will remove any dust or dirt that may have gotten into the sampler during transport and storage and reduce cross contamination between sites.

(Note) No matter what the volume of water is that you need to collect, you must always lower the sampler to the 6-foot mark. That is, if you need to collect 200 milliliters of water for chlorophyll filtering, you must collect the full 6 feet or 924 milliliters that the sampler holds. This is important so that the sample is not biased based on depth.

STORAGE

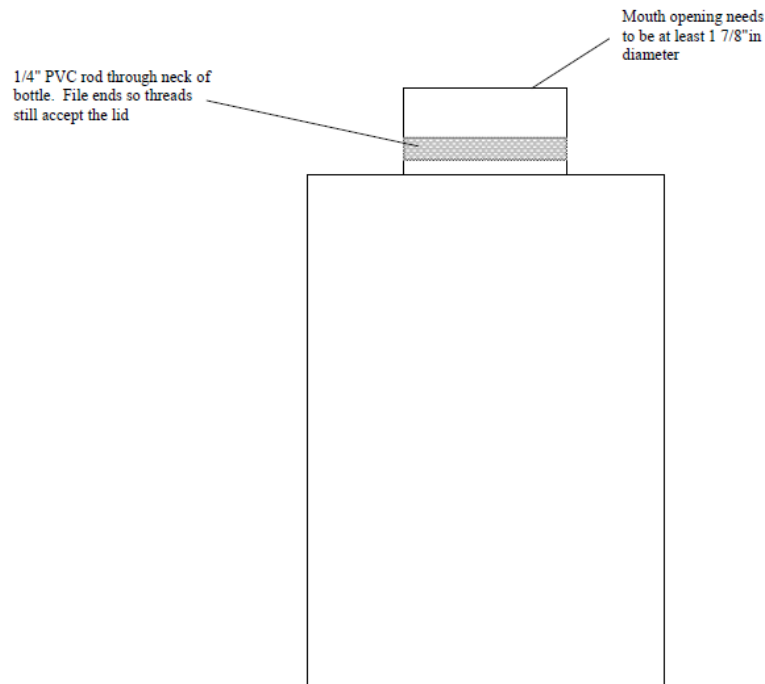
1. Thoroughly rinse equipment when finished sampling.
2. Store the sampler **upside down**, so the ball is free of the fitting opening. This will allow the inside of the sampler to air dry and prevent mold growth. If the ball is stuck in the opening tap the other end of the sampler to dislodge the ball.

Integrating Water Column Sampler Self-Help Program Version



NOT TO SCALE

Collection Container



Appendix F: DNR Interim Protocol for Boat and Gear Disinfection, April 15, 2014

The following steps shall be taken every time a boat, equipment or gear is moved between waterbodies to avoid transporting invasive species and/or pathogens:

- **Inspect** and **Remove** aquatic plants, animals, and mud from your boat, trailer, equipment and gear.
 - **Scrub** your equipment with a stiff-bristled brush, when feasible
- **Drain** all water from your boat, motor, live well, bilge, transom wells as well as from your equipment and gear, including but not limited to tracked vehicles, barges, silt or turbidity curtain, hoses, sheet pile and pumps.
- **Dispose** of unwanted aquatic plants and animals in an appropriate way.
- **Disinfect** your boat, equipment and gear by either washing or disinfecting:
 - **Washing** with hot water (options listed in order of effectiveness):
 1. Steam cleaning (212°F), or
 2. Boat decontamination unit (140°F and 2,500 psi), or
 3. Commercial car wash (~120°F)
 - **Disinfecting** with a 2% Virkon (2.7 ounces per gallon) solution bath or spray for 20- or 30-minutes contact time, respectively. Follow with a clean water rinse. Use the chemical far away from surface waters and dispose of properly.

See the manual code for safety precautions and purchase information.

Additional recommended steps:

- Discontinue the use of felt-soled waders

Appendix G: Example Meter Calibration Log (from West Central Region)

SONDE CALIBRATION LOG

| SONDE # | | | | | | | | |
|--------------------|------|------|---------|--------|------------|---------|-------|--|
| Sites visited | | | | | | | | |
| Field Crew | | | | | | | | |
| ANALYST | DATE | | TIME | | BAROMETRIC | | VOLTS | |
| | TEMP | DO % | DO mg/L | pH 7.0 | pH 10.0 | SP COND | DEPTH | |
| INITIAL VALUES | | | | | | | | |
| CALIBRATION VALUES | | | | | | | | |
| NOTES | | | | | | | | |

| SONDE # | | | | | | | | |
|--------------------|------|------|---------|--------|------------|---------|-------|--|
| Sites visited | | | | | | | | |
| Field Crew | | | | | | | | |
| ANALYST | DATE | | TIME | | BAROMETRIC | | VOLTS | |
| | TEMP | DO % | DO mg/L | pH 7.0 | pH 10.0 | SP COND | DEPTH | |
| INITIAL VALUES | | | | | | | | |
| CALIBRATION VALUES | | | | | | | | |
| NOTES | | | | | | | | |