Samples for laboratory analysis

Water quality samples are submitted to analyzing laboratory and analyzed for the following parameters:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Sample Quantity</th>
<th>Sample Container</th>
<th>Preservative</th>
<th>Analytical Method</th>
<th>Reporting Limit</th>
<th>Holding Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>E. coli</td>
<td>100 ml</td>
<td>Sterile Plastic</td>
<td>0.1ml 10% sodium thiosulfate, 4°C</td>
<td>SM 10200 H</td>
<td>*</td>
<td>24 hours</td>
</tr>
<tr>
<td>Chlorophyll a</td>
<td>Amber Glass</td>
<td>Store in the dark; 4°C</td>
<td>SM 10200 H</td>
<td>*</td>
<td>***</td>
<td></td>
</tr>
<tr>
<td>Pheophytin</td>
<td>**</td>
<td>**</td>
<td>SM 10200 H</td>
<td>*</td>
<td>***</td>
<td></td>
</tr>
<tr>
<td>Total Phosphorus</td>
<td>500 ml</td>
<td>Plastic</td>
<td>5 ml 10% sulfuric acid; 4°C</td>
<td>EPA 365.1 Rev 2.0</td>
<td>0.003 mg/L</td>
<td>28 days</td>
</tr>
<tr>
<td>Total Kjeldahl Nitrogen</td>
<td>500 ml</td>
<td>Plastic</td>
<td>5 ml 10% sulfuric acid; 4°C</td>
<td>EPA 351.2 Rev 2.0</td>
<td>0.1 mg/L</td>
<td>28 days</td>
</tr>
<tr>
<td>Nitrate + Nitrite Nitrogen</td>
<td>250 ml</td>
<td>Plastic</td>
<td>5 ml 10% sulfuric acid; 4°C</td>
<td>SM 4500-N03-H-00</td>
<td>0.05 ma/L</td>
<td>28 days</td>
</tr>
<tr>
<td>Total Suspended Solids</td>
<td>1 L</td>
<td>Plastic</td>
<td>4°C</td>
<td>SM 2540 D .97</td>
<td>1.0 mg/L</td>
<td>7 days</td>
</tr>
<tr>
<td>Chloride</td>
<td>125 ml</td>
<td>Plastic</td>
<td>4°C</td>
<td></td>
<td>28 days</td>
<td></td>
</tr>
</tbody>
</table>

*Depends upon the volume of sample filtered; **If analyzed, is taken from the same sample as chlorophyll a; ***May be stored on ice in the dark for up to 48 hrs. prior to analysis, otherwise, field- or lab-filter within 48 hrs and store frozen at -20°C for no longer than 28 days prior to analysis.

Training

Training of staff, if needed, is done through assistance from knowledgeable organizations, such as Minnesota Waters. Staff is responsible for field sampling training and monitoring oversight. Project Leaders are responsible for ensuring key project staff has or receives adequate training to effectively and correctly perform their project duties. Key staff includes the Project Manager, samplers, sample handlers, data reviewers, and data assessors. They are also responsible for documenting training, and maintaining the training records.

Data review and submission

All raw data are transcribed to the data transmittal form and stored in a binder-type notebook. Data are organized electronically, submitted to MPCA staff, and filed in the EQuIS database.
Lake and Stream Sampling Standard Operating Procedures (SOPs)

Field water quality measurements (for both stream and lake sampling)

All hand-held instruments, when used, are inspected and calibrated as directed by the manufacturer in the operator manual prior to their use in the field. Steps are taken to fix any instrument problems noted during testing. If any problems cannot be resolved, the instrument is taken out of service and a substitute instrument is used. All calibration solutions are replaced with fresh solutions before the solution expiration date. Batteries for all meters are routinely checked and replaced when meters show power-related problems. Spare batteries for all instruments are taken on all sampling trips. All maintenance procedures are documented in the meter maintenance logs or the field notebook.

Field water quality measurements for the following parameters will be recorded:
- Depth (of probe sensors at time of measurement)
- Temperature
- Dissolved Oxygen
- Specific Conductance
- pH
- ORP (Oxygen-Reduction Potential)

At the primary lake sampling site, measurements will be taken at every meter starting at the surface down to within one-half meter of the lake bottom. At secondary sampling locations on a given lake, the sampling crew may opt to take field measurements at a single, near-surface depth in lieu of taking measurements every meter. Stream measurements should be taken in the main flow of the channel (thalweg). Field measurements will be recorded either electronically, on paper, or both.

Integrated two-meter sampling (lake surface sampling)

The integrated two-meter sampler is a two-meter long narrow pipe used to sample the most active layer of a lake or reservoir concerning suspended algae and similar photosynthetic organisms. The tube is sized to collect a two-liter sample integrated over the top two meters of water.

1) Rinse the sampler three times, discarding rinse water each time.
2) Place the tube vertically into the lake until it is fully submerged.
3) Insert the stopper in the top end of the tube and quickly remove the tube from the water.
4) Place the open bottom end of the tube over the sample collection bottle and remove the stopper. Allow the water to drain from the tube into the sample bottle.
5) Unless otherwise specified, leave a head space in the sample bottle so the sample may be mixed by agitation in the laboratory.
6) Follow lab instructed preservation methods for sample.
Lake Secchi disk transparency reading
Readings should be taken between 10:00 a.m. and 3:00 p.m. on bright, calm days. A deep, centrally located site on the lake is recommended. Continue monitoring at that one site throughout the summer.

1) Travel to the designated monitoring location and anchor the boat.
2) Do not wear sunglasses while making a reading, as this affects the accuracy of the reading. Photogradient prescription eyeglasses can be prevented from darkening by wearing a hat with a wide brim.
3) Lower the Secchi disk into the lake on the shaded side of the boat until the disk just disappears completely from view. Note the disk’s depth using the marks on the cord.
4) Lower the disk a bit farther and then raise it until it just reappears, then note this depth.
5) Average the two depths to the nearest one-half foot to get the transparency reading. Record this average in the "Secchi" column on the datasheet. Also record the date and time of this reading.

Standard methods for stream hand-collected (grab)sampling
When grab sampling is suitable, samples should be collected along the sample site cross-section. Sample at a point that best represents the water quality of the total flow at that cross section. Avoid sampling points that are poorly-mixed or affected by local temporary conditions, such as ponding across part of the stream width, obviously disproportionate sediment load, or backwater conditions. If a site is poorly-mixed across the stream, an integrated sample across the stream width should be used, or another site should be chosen that is well-mixed across the stream width.

Follow bottle rinse and preservation methods as directed by the analyzing laboratory. Repeat-use sampling equipment chambers that contact sample water should be rinsed thoroughly with sample water three times before water is collected to transfer to sample containers. This usually means collecting three "dummy" samples before collecting the sample that will be added to the analytical bottles.

Water is collected at the stream sampling point using one of the following methods depending upon physical accessibility:

Stream rod "Swing" sampling (bottle clamped to a telescoping pole)
1) Rinse the sampler by triple rinsing and discard the rinse water.
2) Reach the rod out to the thalweg. The sampler opening should be lowered mouth down to the middle depth below the water surface then turned upward to collect the sample.
3) Retrieve the sampler and fill sample bottles.
4) Unless otherwise specified, leave a head space in the sample bottle so the sample may be mixed by agitation in the laboratory.

Stream sample bottle dip while wading
1) Follow bottle rinse and preservation methods as directed by the analyzing laboratory.
2) Wade to desired sampling location. Always stand downstream, facing upstream, of the sampling point to avoid contaminating the sample.

3) When grab sampling, the bottle should be lowered mouth down to the middle depth below the water surface then turned upward to collect the sample. Cap the sample and return to shore.

4) Unless otherwise specified, leave a head space in the sample bottle so the sample may be mixed by agitation in the laboratory.

Sample handling and custody

Field information sheets - Field data sheets are the primary method for documenting most stream monitoring field activities. These sheets serve as an initial record of any field measurements and weather conditions at the time of sampling.

Field notes - Field notes are used to document important information during sampling events. They are entered into a bound notebook. Entries are made using pens with indelible ink. Information on field conditions, such as the weather, deviations from written procedures, operating condition of the equipment, and other unusual occurrences are also recorded for each sampling event. The field notebook becomes part of the project data and is retained with the analytical data hard copies and other project documents.

Sample labeling - Each sample container has a label attached which is filled out in its entirety. Sample containers without labels or labels that are missing information are not, as per laboratory policy, accepted by the laboratory. The sample label includes the water body code or name, the site number, the date, and time of sample collection.

Sample shipping - All samples are packed in an ice-filled cooler for transport to the laboratory. Samples are transported within 24 hours of collection.

Chain of custody – All samples are labeled and documented on laboratory chain of custody sheets. Relinquished by and affiliation as well as accepted by and affiliation is documented on the chain of custody sheets. Date and time of all transactions along with sampler name, date and time are also recorded.

Quality control – For *E.coli* sampling, one field QC grab sample duplicate for laboratory analysis is collected at the sampling site for every ten like samples taken. The field duplicate for laboratory analysis is collected to determine sampling and laboratory analytical precision. If QC samples revealed a sampling of analytical problem, field and laboratory personnel attempt to identify the cause. Upon working out a plausible solution, personnel take necessary steps to ensure that similar problems do not arise during future sampling events. If possible, the sampling event is repeated. As per laboratory protocol, suspect data are flagged or qualified depending on the nature and extent of the problem. Blank samples are also taken for the ecoli sourcing to assure clean sampling procedures.

Chloride sampling – Staff follows the March 2015 Chloride Monitoring Guidance developed by the MPCA as a result of the Twin Cities Metropolitan Area (TCMA) Chloride Project.
Data management - The field sampling leader is responsible for completing the field data sheets. This information is entered into a spreadsheet or database and archived. Laboratory results are entered into a computer database and/or spreadsheet which is maintained by the Water Resource Manager who also assists with data maintenance, reduction, and transmittal. The MPCA Project Manager also reviews all data prior to their approved entry into EQuIS.

Quality assurance data sheet checks include scanning for apparent entry errors, measurement errors, and omissions. Suspect data are flagged and/or excluded from use. Data may be presented in table, graph, and chart format. Unusual data are rechecked to verify their accuracy. The data are then entered into EQuIS by VLAWMO staff and reviewed by MPCA for final data submission.