Minnesota Department of Agriculture
Surface Water Monitoring Program

Field Data and Sample Collection
Standard Operating Procedures (SOP)

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Version 2
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The MPCA Watershed Pollutant Load Monitoring Network (WPLMN) Standard Operating Procedures and Guidance (SOPG) was used in the development of this document.
Foreword

The Minnesota Department of Agriculture (MDA) staff and its cooperator’s should follow the Standard Operating Procedures (SOP) outlined in this manual. This SOP will be updated on an as needed basis, and should be used to complete activities defined in the MDA Surface Water Design Document, and annual work plans. MDA staff should be consulted with any questions regarding this SOP.
Acronyms and Abbreviations

AIS  Aquatic Invasive Species
Cl   Chloride
COC  Chain of Custody
CST  Central Standard Time
DI   Deionized
DOP  Dissolved Orthophosphorus
EQuIS Environmental Quality Information System (Database)
ETI  Equal Time Increment
GC-MS/MS Gas Chromatography with Tandem Mass Spectrometry
GLY  Glyphosate
HPLC High Performance Liquid Chromatography
ID   Identifier
LC-MS/MS Liquid Chromatography with Tandem Mass Spectrometry
MDA  Minnesota Department of Agriculture
MPCA Minnesota Pollution Control Agency
MN DNR Minnesota Department of Natural Resources
NH₃  Ammonia
NO₃  Nitrate
NO₂+NO₃ Nitrate + nitrite
QA/QC Quality assurance/quality control
PFD  Personal floatation device
PMR  Pesticide Monitoring Region
SOP  Standard Operating Procedure
T-tube Transparency Tube
TKN  Total Kjedhal Nitrogen
TP   Total Phosphorus
TSS  Total Suspended Solids
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SCOPE AND PURPOSE
This Standard Operating Procedure (SOP) describes the methods followed by the Minnesota Department of Agriculture (MDA) staff and its cooperators for surface water quality sample collection and management of related field data. Adherence to these guidelines will allow for the consistent, efficient, and unbiased collection of scientifically defensible field and water quality data.

This SOP will focus on the collection and management of field and water quality data. Information on the program framework is available in the MDA Surface Water Design Document, and information on the specifics of the individual monitoring locations, monitoring intensity, and laboratory analytical lists are available in the MDA annual work plans. The MDA presents all of the water quality results in an annual Water Quality Monitoring Report and provides all data to the Minnesota Pollution Control Agency (MPCA) for water quality assessment to determine if violation of Minnesota pesticide water quality standards have occurred. All water quality data is managed in the Environmental Quality Information System (EQuIS) database that is accessible by multiple state agencies and publically accessible on the MPCA Environmental Data Access (EDA) webpage.

The MDA surface water monitoring program uses a variety of methodologies to determine the occurrence and magnitude of pesticides in the waters of Minnesota. This SOP will focus on pesticide monitoring efforts in rivers and streams, and rainfall precipitation.
1.0 FIELD CONDITION DOCUMENTATION

Accurate and complete documentation of field conditions is critical when collecting and validating scientifically defensible data. Field notes should be recorded during or immediately after every field visit. Field notes are considered official and legal documents, therefore, should be clearly legible, descriptive, and maintained according to established record retention schedules.

1.1 FIELD NOTES

Field conditions must be documented when samples are collected to allow for the analysis of results. Field notes are to be recorded in a bound, write-in-the-rain field book or directly into the designated fields on the sample chain-of-custody (COC) log-in for cooperators (Section 2.5). Field books should be scanned at least monthly during the field season to create an electronic backup copy. On every field visit to a monitoring location, the following information must be recorded. Additional notes should be taken if there are noticeable changes to water level, the riparian area, or the appearance of the water. Appendix C provides a list of the required field notes.

MDA defines base flow conditions in a river or stream as a period with static water levels, and normal stream transparency. MDA defines storm flow in a river or stream as a period with increasing water levels, often with reduced transparency. In general, storm flow in a river or storm will likely have overland surface runoff following periods of intense or prolonged rainfall or snowmelt. Minor water level increases can still be considered base flow, and elevated stream flow may be considered base flow on the falling limb of a large runoff events.

1.1.1 REQUIRED INFORMATION FOR FIELD NOTES

- Site name and MDA station ID
- Name(s) of staff conducting site visit
- Date (mm/dd/yyyy)
- Military Time (Central Standard Time (CST))
  - Arrival Time
  - Sample Collection Time
  - Departure Time
- Measured water level (note “stage” or “tape down” value)
  - Include error range (i.e. +/- 0.02 ft.)
  - Note Time (Central Standard Time (CST)) of measurement
- Stage value from electronic gage (if applicable) or tapedown measurement (if applicable)
  - Note stage offset adjustments
- Transparency tube reading or water secchi tube with disc
- Rain gage status (if applicable: clear, plugged, etc.)
Channel and channel control notes (beaver dams, moving sand, large debris, etc.):
- Is river within banks, at bank full, out of bank, etc.?

Flow condition:
- Base flow or storm flow (note position on hydrograph (rising, peak, or falling))

Weather conditions:
- Notable weather in past 24-48 hours (rainfall totals/intensity, snowmelt, etc.)
- Current conditions (temperature, wind, cloud cover, etc.)

Watershed observations:
- Note the presence of ponded water in fields, active surface runoff, tile flow, etc.
- Note agricultural operations (corn planting, spraying, etc.)
- Estimate canopy coverage of agricultural fields
- Note any dead insects, invertebrates, or fish
  - Photograph and report immediately to the Minnesota Duty Officer (1-800-422-0798), MDA staff and MDA supervisor. Contact information can be found in Appendix A.

1.1.2 SAMPLE COLLECTION INFORMATION REQUIRED FOR FIELD NOTES

In addition to the above field note requirements, the following information should be recorded whenever samples are collected. For additional details on the items in this list, please refer to Section 2.0.

For all samples:
- Sample bottle ID (See Section 2.4 for description)
- Sample type (routine, field replicate, etc.)
- Collection method (grab, lab line, van dorn, dipper pole, etc.)
- General sample comments
- Collection date (mm/dd/yyyy)
- Collection military time (Central Standard Time (CST))
- Requested laboratory analyses

For all composite samples, record the following information from the automatic sampler, in addition to the requested items above:
- Record start of composite sample collection period
  - Collection date (mm/dd/yyyy)
  - Collection military time (Central Standard Time (CST))
- Record end of composite sample collection period
  - Collection date (mm/dd/yyyy)
  - Collection military time (Central Standard Time (CST))
Number of sample pulses
- Volume of each sample pulse
- Pacing (e.g., 1 hour; 2 hour; 300,000 cubic feet, etc.)
- If errors occurred during collection, record all errors and associated pulse number
- Note refrigerator/cooler temperature at time of sample collection

1.2 PHOTOGRAPHS
Photographs will be taken during each field visit. While photographs are not a substitute for detailed field notes, they do provide an excellent resource during data review, report writing, and data assessment. Since water quality chemistry results are not available at the time of sample collection, every field visit must be photographed the same way. **All photographs should have a timestamp feature enabled (mm/dd/yyyy tt:tt (24 hour, CST)), with the correct time set on the camera.**

1.2.1 REQUIRED PHOTOGRAPHS
The required photographs for each field visit are provided below. It is recommended that the photographs be taken from the same location during each field visit so that they are comparable to other field visits:
- Upstream (from bridge deck if available)
- Downstream (from bridge deck if available)
- Channel control ( riffle, ice, vegetation, etc.)
- Transparency tube (t-tube) or water secchi tube with disc showing the t-tube value (foreground) with stream (background)
- Document things that have changed since last site visit, including but not limited to:
  - Low or high water levels
  - Flooding
  - Excessive sediment deposition or channel scarring
  - Excessive vegetation
  - Dead insects, invertebrates, or fish
  - Unusual water color or oily sheen
- If site maintenance is completed, take photographs showing the area before and after
- Sample collection photographs
  - Equipment used to collect sample
  - Filled bottles showing all collected analyses with bottle ID on label visible
  - Sample bottle label visible in the foreground with stream visible in the background
  - For composite samples:
    - Well mixed composite sample bottle before filling bottles
    - T-tube and composite sample bottle
- Filled bottles showing all collected analyses, with label visible next to composite sample bottle

### 1.2.2 PHOTOGRAPH NAMING

The photographer should organize and rename the collected photographs by site and date collected. All photographs associated with MDA monitoring should be named with the following format:

MDA station ID (Single Space) YYYYMMDD(unique ID)

For example: two photographs taken from the Middle Branch of the Whitewater River (MDA station ID: MBW) on June 20, 2017 should be named as follows:

```
MBW 20170620a.jpg  
MBW 20170620b.jpg
```

### 1.2.3 PHOTOGRAPH TRANSFER AND STORAGE

MDA cooperators should manage all associated photographs in a single folder for each monitoring location at their field office. After August 31st, MDA staff will contact each cooperator to determine the most efficient transfer method (e.g. CD/DVD, flash drive, electronic transfer, etc.). If preferred, cooperators can also transfer photographs after each sample collection event via e-mail to the MDA.

MDA staff should store the correctly named photographs in the correct monitoring location network folder:

H:\PFMD\ENVIRONMENTAL\MONU\Monitoring and Assessment\Photos\Surface Water

1. Select the appropriate Pesticide Monitoring Region (PMR) folder for each site.
2. Find/create the location folder (each location will have a folder designated with the MDA station ID)
3. Find/create the folder for the current year.
4. Copy all photos from your local computer into the correct locations.

Each MDA staff will be responsible for the placement of their photographs on the network. In addition, the corresponding MDA staff member is responsible for the placement of the cooperator photographs on the network.
2.0 WATER QUALITY SAMPLING
MDA staff and cooperators must be familiar with sampling methodology, sampling techniques, safety procedures, sample preservation, and record keeping before collecting samples. New field staff should read this SOP, become familiar and comfortable with this SOP and complete several field visits with an experienced staff member prior to collecting samples on their own.

MDA staff members are required to complete a defensive driving course and the “PFMD Field Safety – Non-point Sections” training. Cooperators should complete all required training by their employing entity, and contact MDA staff with any questions related to field visits and sample collections.

All equipment, and vehicles, used for the collection and transport of pesticide samples should have a known history that does not include use for the transport of pesticide products or application equipment.

Water samples should not be collected in situations that put the field staff in an elevated risk of injury or death. Following the procedures in this SOP will help ensure safe field visits. Do not deviate from the standard procedures, or use unapproved methods, unless pre-approval from MDA is granted. All approved deviations from this SOP must be thoroughly documented in the hydrologists field book or sample COC log-in submission form.

2.1 PREPARATION FOR SAMPLING
The proper collection of water quality samples requires preparation prior to the field visit. Sound organization and forethought will allow for the collection of scientifically defensible samples to be collected in an efficient manner. As part of preparation for field work, it is important to have the correct equipment and safety items, bottles, and items for sample preservation.

2.1.1 EQUIPMENT AND SUPPLY CHECKLIST
A wide variety of sampling equipment may be needed for the collection of water samples due to differences at each monitoring location and varying water levels. The objective of obtaining a representative sample should dictate the selection of equipment on each field visit. The checklist below should be used as a guide to ensure the proper equipment and supplies are available on each trip.
Field safety and survival equipment

- First aid kit
- High visibility safety vest with reflective strips
- Rain gear
- Personal floatation device (PFD)
- Cell phone
- Replacement batteries (if using equipment with batteries)
- Basic hand tools
- Flashlight

General field equipment

- Camera
- Transparency, or secchi tube
- Tape-down tape (if wire weight gage is not installed)
- Field book, or sample COC log-in form
- Waterproof pen and permanent marker
- Site keys (if applicable)

Surface water sampling equipment

- Sample bottles (including temperature blank and extra bottles)
- Sample bottle labels, pre-printed or blank
- Nitrile gloves
- Sampling device (see Section 2.4 below)
- Coolers and ice
- Deionized water (DI)
- Soap and brush for cleaning (if applicable)

2.2 MDA SAMPLING SCHEME
This section will provide the generalized MDA sampling scheme to provide an idea on when water quality samples are to be collected. Details on how to collect water quality samples will be presented in subsequent sections.

2.2.1 GRAB SAMPLING PROTOCOL FOR TIER 1 AND TIER 2 LOCATIONS.
Sample collection for the MDA’s tiered monitoring network occurs from May 1st through August 31st, with designated sample collection periods defined in the MDA annual work plan. Sample collection should occur as close to the peak of storm hydrographs as possible within each sample collection period. If a storm event does not occur, or is not anticipated, during the specified
sample collection period, a sample should be collected during baseflow, or non-storm, flow periods. Samples should be collected within the same river reach as the stage/flow monitoring gage whenever possible.

The MDA will provide the appropriate bottles and analytical services for each monitoring location. The determination of when sample collection should occur shall follow these guidelines:

1. Samples will be collected using approved methodology during the peak pesticide detection periods of May 1st through August 31st.
2. All sample bottles associated with the same collection date and time should have the same sample number, regardless of laboratory method that will be used to analyze the sample.
3. The first sample (Sample #001) should be collected during the first sample collection period (May 1st – 15th). If possible, sample collection should occur during major storm events. Increases in flow greater than fivefold, and/or decreased stream transparency, will be characterized as a storm event. If no storm events are expected, or occur, during the sample collection period, a baseflow sample should be collected. If the location is identified as a Tier 2 or Enhanced Tier 2 location, and Sample #001 is collected during a “storm flow period”, a second follow-up sample should be collected no sooner than 24 hours after, but within 96 hours of the first. MDA prefers the follow-up sample to be collected between 72 and 96 hours after the first sample, if possible. No follow-up sample should be collected if the first sample was collected during a baseflow period.
   a. At Tier 2 and Enhanced Tier locations, a storm flow sample may be collected at the end of the sample collection period. If this occurs, collect the follow-up sample no sooner than 24 hours after, but within 96 hours of the first. MDA prefers the follow-up sample to be collected between 72 and 96 hours after the first sample, if possible. The follow-up sample may extend into the next sample collection period, however, this follow-up sample should not be considered the sample for the next sample collection period. In this instance, three samples may be collected in a single sample collection period (follow-up sample from previous sample collection period, storm flow sample, and follow-up sample from the current sample collection period).
4. The same sampling procedure should be followed for the remaining sample collection periods. Monitoring periods are defined in Table 1 below.
5. A sampling decision matrix chart, that guides the collection of samples in each monitoring period, is presented in Figure 1 below.
Table 1. Sample collection periods for Tier 1, Urban Tier 1, Tier 2, Enhanced Tier 2, and Urban Tier 2 monitoring locations.

<table>
<thead>
<tr>
<th>Sample Collection Period</th>
<th>Tier 1</th>
<th>Urban Tier 1</th>
<th>Tier 2*</th>
<th>Enhanced Tier 2*</th>
<th>Urban Tier 2*</th>
</tr>
</thead>
<tbody>
<tr>
<td>May 1 - May 15</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>May 16 - May 31</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>June 1 - June 15</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>June 16 - June 30</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>July 1 - July 15</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>July 16 - July 31</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>August 1 - August 15</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>na</td>
<td>x</td>
</tr>
<tr>
<td>August 16 - August 31</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>na</td>
<td>na</td>
</tr>
<tr>
<td>August 1 – August 10</td>
<td>na</td>
<td>na</td>
<td>na</td>
<td>x</td>
<td>na</td>
</tr>
<tr>
<td>August 11 – August 20</td>
<td>na</td>
<td>na</td>
<td>na</td>
<td>x</td>
<td>na</td>
</tr>
<tr>
<td>August 21 – August 31</td>
<td>na</td>
<td>na</td>
<td>na</td>
<td>x</td>
<td>na</td>
</tr>
</tbody>
</table>

* If a sample is collected during storm flow, a follow-up sample should be collected no sooner than 24 hours, after but within 96 hours, of the first sample. MDA prefers the follow-up sample to be collected between 72 and 96 hours after the first sample, if possible.

Figure 1. Pesticide monitoring sampling decision matrix.
2.2.2 SAMPLING PROTOCOL FOR TIER 3 LOCATIONS.
The MDA maintains a network of Tier 3 locations that feature automated water samplers which collect equal time increment (ETI) composite samples. These locations are operated by MDA hydrologists, and require flexibility to adjust for changing field conditions. Tier 3 locations are operated year-round, however, sampling is intensified when agricultural operations are occurring during the growing season.

Tier 3 locations will be monitored using the following general guidelines.

1. Automated water samplers will be activated by an increase in water level (stage).
2. Automated water samplers will be operated on a 2-day or 4-day ETI program based on anticipated storm hydrograph duration.
3. Storm flow events will be monitored with an automated water sampler.
   a. Every storm flow event from May 1st through August 31st should have an ETI composite sample collected, pending laboratory capacity.
   b. Storm flow events during the months of September through April may be sampled at the discretion of a MDA hydrologist.
4. Base flow periods will be monitored using a grab sample protocol.
   a. Between storm flow events from May 1st to August 31st with a recommended maximum window of 10-14 days during base flow periods without storm flow events.
   b. Every 1-2 months during the months of September through April.
5. Automated samplers will use glass sample collection bottles during the sample collection period.
   a. Composite sample collection bottle cleaning procedures are described in Section 3.0.

2.2.3 RAINFALL PRECIPITATION SAMPLING
The MDA maintains a network of rainfall precipitation samplers that allow for the analysis of pesticides in rainfall. These locations are operated by MDA hydrologists, and require flexibility to adjust for changing field conditions. Basic operational guidelines include:

1. Sites should be operated from mid-April through September.
2. Samples should be composited in a glass collection vessel in a refrigerator on-site and collected when sufficient volume exists for laboratory analysis.
3. The composite start and end date and times should align with the previous and next sample to have a complete monitoring record.
4. A tipping bucket rain gage should be operational at, or near, the site to determine rainfall volumes and intensity.
5. Total rainfall volume (inches) should be calculated for each composite period and noted on sample chain-of-custody (COC) log-in.
2.3 FIELD MEASUREMENTS
Field measurements are required during each visit, and should be documented following the guidelines in Section 1.1.

2.3.1 TRANSPARENCY TUBE MEASUREMENT
1. Rinse the transparency tube (t-tube) with stream water.
2. Carefully fill the t-tube to the top with stream water.
3. Remove sunglasses and turn your back to the sun to prevent direct sunlight from entering the t-tube while taking a t-tube measurement.
4. While looking down the top of the tube, slowly release water until the disk reappears and record the value. Release more water until the screw head is clearly seen and record the value.
5. Average the two values from step 4 and record the midpoint.
6. If the observer can see the disk with the t-tube full, record the value as being greater than the maximum value on the t-tube (e.g., >60 cm).

2.3.2 MEASURED STAGE (WATER LEVEL MEASUREMENT)
Measured stage may be obtained by one of several methods, including: reading a staff gage, measuring the distance to the water surface with a wire weight gage, or measuring the distance from a known reference point to the surface of the water and then subtracting this distance from the reference point elevation (tape down method).

2.3.2.1 Staff gage
1. Record the staff gage reading (in decimal feet).
2. Apply offset to electronic instruments, if needed.

2.3.2.2 Wire weight
The following instructions are also available on a video prepared by the MPCA.

1. Open wire weight house attached to the bridge using USGS/MDNR key.
2. Carefully lower the wire weight until it touches the water surface. If water is surging, lower the weight until it is mid-distance between the peak and trough of the surging water.
3. Record the measurement.
4. Document error range based on field conditions. Typically a value between 0.0-0.03’ error range attempts to provide a value with the amount of error in the wire weight reading. Windy conditions and turbulent flow can make wire weight measurements very difficult, requiring an error range for interpretation.

2.3.2.3 Tape down (or bridge to water measurement)
The following instructions are also available on a video prepared by the MPCA.
1. Locate the established reference mark (yellow arrow, chisel/marker lines, etc.) on the bridge or culvert.

2. Lower the weighted tape to the water surface. It may be difficult, or impossible, to take an accurate measurement during high winds.

3. Determine the length from the reference point to the water surface, to the nearest 1/100th of a foot, on the field sheet. If water is surging, lower the weight until it is mid-distance between the peak and trough of the surging water. Subtract this number from the established elevation and record.

4. Document error range based on field conditions, typically a value between 0.0-0.05’. The error range attempts to provide a value with the amount of error in the tape down reading. Windy conditions and turbulent flow can make tape down measurements very difficult.

### 2.4 WATER QUALITY SAMPLING PROCEDURES

Collection of samples from rivers and streams involves transporting all necessary items to the water quality sampling location, making observations and measurements, and sampling. All field parameters should be measured and recorded first, followed by sample collection and preservation, equipment cleaning, decontamination, and storage. Equipment should be triple rinsed with distilled water before moving to the next sampling location. See Section 3.4 if the monitoring location has an aquatic invasive species (AIS) designation.

#### 2.4.1 SAMPLE BOTTLE SELECTION

The appropriate sample bottles must be used for all sample collection events. Each laboratory analysis requires a sample bottle. Table 2 presents the analysis, analysis code, bottle type, bottle size, bottle filling level, preservation, and maximum hold time from collection until extraction for analysis. Sample bottles that are certified quality assured “containment free sample container” should not be rinsed prior to filling. All other sample bottles should be triple rinsed with sample water before filling. All samples should be submitted to the MDA Laboratory within 7 days of collection. A temperature blank, filled with stream/river water, must be contained in each cooler sent to the MDA Laboratory.

Pesticide sample bottles (950 mL amber glass) for laboratory analysis involving gas chromatography with tandem mass spectrometry (GC-MS/MS) and liquid chromatography with tandem mass spectrometry (LC-MS/MS) require quality assured, trace clean bottles and should not be rinsed prior to sample collection. All pesticide, nutrient, and sediment bottles will be single use.
Table 2. Bottle type, preservation method, and holding time by laboratory analysis.

<table>
<thead>
<tr>
<th>Analyte Type</th>
<th>Analyses</th>
<th>Analysis Code</th>
<th>Bottle Type</th>
<th>Bottle Size [mL]</th>
<th>Fill Method</th>
<th>Preservation</th>
<th>Maximum Hold Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pesticide</td>
<td>GC-MS/MS</td>
<td>GC</td>
<td>Amber Glass</td>
<td>950</td>
<td>Full</td>
<td>Refrigerate (0-6°C)</td>
<td>21 days</td>
</tr>
<tr>
<td></td>
<td>LC-MS/MS</td>
<td>LC</td>
<td>Amber Glass</td>
<td>950</td>
<td>Full</td>
<td>Refrigerate (0-6°C)</td>
<td>21 days</td>
</tr>
<tr>
<td>Glyphosate</td>
<td>GLY</td>
<td>Centrifuge Tube</td>
<td></td>
<td>50</td>
<td>½ full</td>
<td>Freeze</td>
<td>21 days</td>
</tr>
<tr>
<td>Ammonia</td>
<td>NH3</td>
<td>HDPE</td>
<td></td>
<td>125</td>
<td>Full</td>
<td>Freeze¹</td>
<td>28 days</td>
</tr>
<tr>
<td>Chloride</td>
<td>Cl</td>
<td>HDPE</td>
<td></td>
<td>125</td>
<td>Full</td>
<td>Refrigerate (0-6°C)</td>
<td>28 days</td>
</tr>
<tr>
<td>Nitrate + Nitrite</td>
<td>NO3+NO2</td>
<td>HDPE</td>
<td></td>
<td>125</td>
<td>¾ full</td>
<td>Freeze¹</td>
<td>28 days</td>
</tr>
<tr>
<td>Total Kjeldahl Nitrogen</td>
<td>TKN</td>
<td>HDPE</td>
<td></td>
<td>500</td>
<td>¾ full</td>
<td>Freeze¹</td>
<td>28 days</td>
</tr>
<tr>
<td>Total Nitrogen</td>
<td>NH3, NO3+NO2, TKN</td>
<td>HDPE</td>
<td></td>
<td>500</td>
<td>¾ full</td>
<td>Freeze¹</td>
<td>28 days</td>
</tr>
<tr>
<td>Total Phosphorus and Dissolved Ortho-Phosphorus</td>
<td>TP+DOP</td>
<td>HDPE</td>
<td></td>
<td>250</td>
<td>¾ full</td>
<td>Freeze¹</td>
<td>28 days³</td>
</tr>
<tr>
<td>Total Suspended Solids</td>
<td>TSS</td>
<td>HDPE</td>
<td></td>
<td>500/1000</td>
<td>Full</td>
<td>Refrigerate (0-6°C)</td>
<td>7 days</td>
</tr>
<tr>
<td>Other</td>
<td>5-day Carbonaceous Biological Oxygen Demand</td>
<td>CBOD</td>
<td>HDPE</td>
<td>1000</td>
<td>Full</td>
<td>Refrigerate (0-6°C)</td>
<td>48 hours²</td>
</tr>
<tr>
<td>Metals</td>
<td>List each metal²</td>
<td>HDPE</td>
<td></td>
<td>250</td>
<td>¾ full</td>
<td>Freeze¹</td>
<td>6 months</td>
</tr>
<tr>
<td>Temp blank</td>
<td>Temperature Blank</td>
<td>Temperature Blank</td>
<td>HDPE</td>
<td>125</td>
<td>Full</td>
<td>Refrigerate (0-6°C)</td>
<td>--</td>
</tr>
</tbody>
</table>

¹ Freeze – It is not necessary to freeze samples if shipped on the date collected and received by the lab within 24 hours provided samples are shipped on ice.
² 48 hour hold - Submit to lab within 24 hours of collection to allow time for sample processing.
³ DOP test – If sample is refrigerated, maximum hold time is reduced to 48 hours. Submit refrigerated samples to the lab within 24 hours of collection.
⁴ Metals – list requested metals on the form (e.g., copper, iron, lead, etc.)
⁵ Total Nitrogen – Total nitrogen is not a stand-alone test. It is calculated based on NH3, NO3+NO2, and TKN.
2.4.2 SAMPLE BOTTLE LABELS
All sample bottles associated with each same sample collection event will have the same, unique sample ID. Each sample collection event unique ID will specify the MDA Station ID (2 or 3 letter/number abbreviation), year (yy) and sample number for the specified year (Figure 2).

![Figure 2. Bottle sample ID example.]

In addition to the sample ID, the laboratory analysis code and sample collection date are required on the bottle label for laboratory submission (Table 2). For composite samples, enter the sample end date on the label. Sample labels will be pre-printed (sample bottle ID, laboratory analysis, and year). The date must be entered at the time of collection (Figure 3). Sample time (CST) can also be included, if desired.

![Figure 3 Sample bottle label example.]

AIS designated sites will be noted on the sample bottle ID with the statement “From AIS Infested Waters” in a green box. AIS designation will also be noted on the sample COC form. For more information on AIS designated sites, refer to Section 3.4.

2.4.3 SAMPLING METHODS
Nitrile gloves must be worn when sampling or handling the sampling vessel. Field conditions should dictate the selection of sampling methods, to ensure a representative sample is collected. Sample collection should occur near the center of flow in a well-mixed, or turbulent, portion of the river and should be depth integrated (represent water from the surface to the bottom), if possible. If it is raining during sample collection, samples should only be collected if it is possible to prevent rain drops from entering the sample bottle or collection vessel. If agricultural or other pesticide application is occurring near the sample collection location, or if the odor of pesticides is present in the air, the person collecting the sample should consider leaving the area.
and returning at a later time to avoid possible contamination of the sample from nearby activities through the air. Such instances should be documented in the field notes.

MDA staff and cooperators should use best professional judgment when deciding how to collect a water sample. Common surface water sampling methods are presented below. If applicable, MDA staff should be notified for approval of alternative sampling methods prior to the season.

A temperature blank, filled with stream/river water, must be contained in each cooler upon arrival at the MDA Laboratory.

### 2.4.3.1 Telescoping Rod

A telescoping rod should be used to collect a water sample from the bank or low bridge. The telescoping rod can have a permanently attached collection container or a spring clamp to hold the actual sample bottle. The following instructions are also available on a video prepared by the MPCA.

1. Carry telescoping rod and prepared sample bottles to the sample location.
2. Ensure the collection container, or sample bottle, is securely attached to the telescoping rod.
3. Extend the telescoping rod to the length necessary to reach the point of flow. Dip the rod into the water with the bottle opening facing the water.
   a. If a collection container is used, triple rinse the collection container with stream water.
   b. If the sample bottle(s) is not a quality assured “containment free sample container”, directly attach it to the telescoping rod and triple rinse the sample bottle with stream water.
4. Submerge the collection container, or sample bottle, just below the water surface.
5. Carefully retract the rod, minimizing spills, to the stream bank.
6. Collect the sample.
   a. If a collection container is used, triple rinse all sample bottle(s) that are not a quality assured “contaminant free sample container” with stream water prior to collecting the sample.
   b. Fill the sample bottles following the guidelines in Table 2.
   c. If the sample bottle is directly attached to the pole, carefully cap the sample, following the guidelines in Table 2.
7. Repeat steps 3 through 6 as needed to fill all sample bottles.
8. Preserve samples on ice immediately following collection.
9. If a collection bottle is used on the rod, triple rinse with DI water after each sample collection event.

### 2.4.3.2 Suspended sampler (Van Dorn, Lab-Line, or Weighted Bucket)

A suspended sampler should be used to collect a water sample from a bridge deck. A high visibility safety vest with reflective striping must be worn, and extreme caution should be
demonstrated by field staff when working from a bridge. Ideally sampling will occur on the downstream side of the bridge so the sampling vessel can be observed to assess its condition. Sample collection should occur near the center of flow in a well-mixed or turbulent portion of the river and should be depth integrated (represent water from the surface to the bottom), if possible. The MPCA prepared a video highlighting the collection of water samples with a Van Dorn sampler, and a video demonstrating maintenance of the Van Dorn sampler.

1. Prepare suspended sampler and sample bottles.
2. Ensure the sampler is securely attached to the rope.
3. Rinse the sampler three times with stream water.
4. Lower the sampler into the stream carefully, avoiding contact with the bridge rail, debris and the stream bottom.
5. Once filled, retrieve the sampler carefully avoiding contact with the bridge rail and bridge. Do not set the filled sampler directly onto the bridge or bridge rail.
6. If the sample bottle(s) is not a quality assured “containment free sample container”, triple rinse said sample bottles with water from the sampler while continuously shaking the sampler to avoid sediment from settling out.
7. Fill all sample bottles following the directions in Table 2.
8. Repeat steps 3 through 7, as needed, to fill all required sample bottles.
9. Preserve samples on ice immediately following collection.
10. Triple rinse the suspended sampler with DI water.

2.4.3.3 Wading
1. Enter the stream downstream of the sampling location with prepared sample bottles.
2. Wade upstream to representative sample collection location, moving slowly to avoid suspension of stream bed sediments.
3. Remove the sample bottle lid.
4. Facing upstream, triple rinse all sample bottle(s) that are not a quality assured “containment free sample container”.
5. Fill the sample bottles following the direction in Table 2.
6. Repeat steps 3 through 5 for each sample bottle.
7. Preserve samples on ice immediately following collection.

2.4.3.4 Automated Sampler
1. Upon arrival, record automated sampler information presented in Section 1.1.
2. Prepare sample bottles.
3. Remove and cap sample collection container from automated sampler, and vigorously shake for at least 1 minute, or until sample is thoroughly mixed.
4. Triple rinse all sample bottle(s) that are not a quality assured “containment free sample container” with water from the sample collection container.
5. Fill the sample bottles following the direction in Table 2. The sample collection container should be shaken continuously during filling.
6. Preserve samples on ice immediately following collection.
7. Clean the composite glass sample collection container from the automated sampler following the cleaning procedures in Section 3.0.
8. Reset the automated sampler to prepare for the next storm event.

2.4.4 QUALITY ASSURANCE / QUALITY CONTROL SAMPLING METHODS

This section will present the protocol for the collection of the various quality assurance / quality control (QA/QC) samples. QA/QC samples should only be collected if rainfall cannot enter the sample bottles. QA/QC samples should not be filled when pesticide applications are occurring in close proximity to the sample site at the time of collection. QA/QC samples will be handled in the same manner as routine samples after collection, and submitted blindly to the MDA Laboratory. The MDA will determine the locations and types of all QA/QC samples prior to the start of the monitoring season.

The MDA uses several types of QA/QC samples including temperature blanks, field duplicate samples, field replicate samples, field blank samples, field equipment blank samples, and laboratory equipment cleaning blank. Each type of QA/QC sample is described below.

2.4.4.1 Temperature Blank

A temperature blank, filled with stream/river water, must be contained in each cooler upon arrival the MDA Laboratory. The temperature blank should be filled using a pre-labeled 125 mL bottle (Figure 4) with stream water at the first location of each trip according the guidelines in Table 2. In instances with a limited amount of sample volume and no stream directly next to monitoring station (e.g., composite samples at an edge-of-field site), the temperature blank can be filled at another location, with stream water, or tap water. Do not use pre-chilled, dedicated temperature blanks. If sampling occurs over multiple days, the temperature blank should be filled on the first day of sampling. The temperature blank should stay in the same cooler as the samples and be refrigerated with the collected samples overnight in most circumstances. If all samples contained in a single cooler arrive at the MDA Laboratory frozen, the temperature blank should also be frozen.

Dedicated temperature blank bottles will be distributed and can be re-used when returned in coolers from the MDA Laboratory. If multiple coolers will be used for shipping, one temperature blank will be necessary for each cooler being used for shipment. Temperature blanks are not indicated on sample COC log-in submission form. The MDA Laboratory will return the empty temperature blank bottles with the cooler.

1. During the first sample collection event each day, fill a dedicated “Temperature Blank” bottle with stream water.
2. Preserve samples on ice immediately following collection.

![Temperature Blank]

Figure 4. Temperature blank label for 125 mL bottle.

2.4.4.2 Field Duplicate Samples
The MDA defines field duplicate samples as the samples split from the same sample collection container in an automated sampler (composite samples only). The sample collection vessel must be well-mixed at the time of collection. When filling sample bottles, the routine sample and duplicate of the same analysis must be filled simultaneously.

1. Prepare the sample bottles (both the routine sample and field duplicate).
2. Triple rinse all sample bottle(s) that are not a quality assured “containment free sample container” with water from the sample collection container.
3. Fill the sample bottles following the direction in Table 2. Samples should be filled in an alternating fashion between the routine sample and the field duplicate (filled approximately 25 percent between switching bottles) for each paired analysis.
4. Preserve samples on ice immediately following collection.

2.4.4.3 Field Replicate Samples
The MDA defines field replicate samples as all samples collected at the same time during a grab sample event (using any of the recognized sample collection methods). Samples may be collected directly from the stream or from the same piece of collection equipment. When filling sample bottles, the routine sample and replicate of the same analysis must be filled simultaneously.

1. Prepare the sample bottles (both the routine sample and field replicate).
2. Triple rinse all sample bottle(s) that are not a quality assured “containment free sample container” with stream water.
3. Fill the sample bottles following the direction in Table 2. Samples should be filled in an alternating fashion between the routine sample and the field replicate (filled approximately 25 percent between switching of bottles).
4. Preserve samples on ice immediately following collection.
2.4.4.4 Field Blank Samples
Field blank samples are collected to evaluate whether atmospheric contaminants have been introduced into the samples during the process of sampling.

1. Prepare the sample bottles.
2. Pour MDA provided High Performance Liquid Chromatography (HPLC) grade water into the sample bottle(s) at the designated monitoring location.
3. Preserve samples on ice immediately following collection.

2.4.4.5 Field Equipment Blank Samples
Field equipment blank samples are collected to evaluate field sampling and decontamination procedures by passing HPLC grade organic-free water over or through the sampling equipment being evaluated. Samples collected with a telescoping rod or while wading will not have associated field equipment blanks.

1. Collect the routine sample as directed in Section 2.4.
2. Prepare the sample bottles for the field equipment blank.
3. Triple rinse the collection vessel with DI water.
4. Pour MDA provided HPLC grade water into the collection vessel used for the routine sample.
5. Directly fill the field equipment blank sample bottles.
6. Preserve samples on ice immediately following collection.

2.4.4.6 Laboratory Equipment Cleaning Blank Samples
Laboratory equipment cleaning blank samples are collected to evaluate laboratory cleaning methods of monitoring equipment by passing HPLC grade organic-free water over or through the sampling equipment being evaluated.

1. Clean the sample collection equipment as directed in Section 3.0.
2. Prepare the sample bottles for the laboratory equipment cleaning blank.
3. Pour MDA provided HPLC grade organic-free water into the collection vessel used for the routine sample.
4. Directly fill the laboratory equipment cleaning blank bottles.
5. Preserve samples on ice immediately following collection.

2.5 SAMPLE PROCESSING AND SHIPPING

2.5.1 SAMPLE PRESERVATION

2.5.1.1 Sample Transportation and Preservation
*No chemical preservation methods are used for MDA samples.* All samples must be placed in an iced cooler (≤ 6°C) immediately following sample collection. Upon arrival back at the field
office, samples should be refrigerated or frozen following the guidelines in Table 2. Samples should be allowed to cool, or freeze, overnight prior to shipping or delivery for MDA Laboratory submission. Thermal preservation will minimize biological activity, thereby preserving sample integrity. Cooler temperatures will be verified with the temperature blank and thermometer. A temperature blank, filled with stream/river water, must be contained in each cooler when shipping to the MDA Laboratory.

**2.5.1.2 Sample Storage and Holding Times**
Samples must be stored in a dedicated refrigerator and/or freezer, instrumented with a thermometer, with audio alarm, to ensure proper sample storage conditions. The refrigerator and/or freezer must be clean and in an area without prior history of pesticide storage. All samples must be submitted to the MDA Laboratory within seven days of collection. Holding times for each analysis are presented in Table 2.

**2.5.2 SAMPLE CHAIN-OF-CUSTODY LOG-IN SUBMISSION FORM**
The sample COC log-in should be completed in the field or soon after returning to the field office. The sample COC log-in contains important information, such as the sample location, dates/times, sample IDs, parameters to analyze for, and contact information. A stand-alone sample COC log-in must accompany the sample(s) and be included in every cooler upon delivery to the MDA Laboratory. The sample COC log-in should be checked several times for accuracy and completeness prior to shipment or delivery to the MDA Laboratory. The MDA utilizes a paper cooperator COC log-in, as well as an electronic COC log-in that is used by the MDA staff.

The MDA Laboratory will send all sample COC log-in forms with receiving notes to the “Report To” staff identified on the form. At the end of the year, these forms should be organized by monitoring location, scanned, and the file stored in this folder:

H:\PFMD\ENVIRONMENTAL\MONU\Data Warehouse\Scanned Log-in Forms

**2.5.2.1 Cooperator Sample Chain-Of-Custody Log-In Submission Form**
The MDA will prepare paper sample COC log-in submission forms for each sample collection event. Each sample collection event may require multiple sample COC log-in forms. The cooperator should fill out all cells highlighted in yellow. The cooperator should verify that the sample bottle ID on the sample itself matches the pre-printed sample ID on the paper sample COC log-in form. The MDA Laboratory will forward the sample COC log-in to the appropriate MDA staff, who will enter the submitted field information into its database. It is critical for cooperators to capture the required information listed in Section 1.1.

An example, completed cooperator sample COC log-in form is presented below as Figure 5 and definitions of required fields are provided in Table 3.
Figure 5. Cooperator sample COC log-in example
### Table 3. Cooperator sample COC log-in fields.

<table>
<thead>
<tr>
<th>Data Entry Field</th>
<th>Description / Instructions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sampled By:</td>
<td>Name of organization and sampler name. Edit if needed.</td>
</tr>
<tr>
<td>Delivered or Shipped By</td>
<td>Name of organization and sampler name. Edit if needed.</td>
</tr>
<tr>
<td>Thermal Preservation</td>
<td>Select thermal preservation method that reflects how samples were stored overnight prior to shipping or delivery</td>
</tr>
<tr>
<td>Delivery Method</td>
<td>Select delivery method used to deliver samples to the MDA Laboratory</td>
</tr>
<tr>
<td>Field Sample ID</td>
<td>Verify sample ID on bottle matches the printed sample ID</td>
</tr>
<tr>
<td>Sample Date and Time</td>
<td>Enter the date and time (CST, military time) of sample collection. For grab samples, enter the sample collection time. For composite samples, the start and end date/time should be entered from the start of the composite to the end composite collection period. The date/time when staff completed the field visit to collect the composite sample is not entered</td>
</tr>
<tr>
<td>Sample Type</td>
<td>Select base flow or storm flow at the time of sample collection.</td>
</tr>
<tr>
<td>Grab Sample Collection Method</td>
<td>Select the collection method used to obtain the sample. Refer to Section 2.4 for additional information</td>
</tr>
<tr>
<td>Water Level (ft)</td>
<td>Record stage at the time of collection. If value is a tape-down value, please note this on the form.</td>
</tr>
<tr>
<td>Transparency</td>
<td>Enter the transparency of the stream at the time of collection.</td>
</tr>
<tr>
<td>Rainfall Estimate</td>
<td>Enter an estimate of the rainfall received in the watershed prior to monitoring. For example, 0.5 inches rainfall in previous 24 hours or 2.2 inches in previous 48 hours, or 0 inches in previous 240 hours.</td>
</tr>
<tr>
<td>Weather and Watershed Observations</td>
<td>Use this field to note the following activities:</td>
</tr>
<tr>
<td></td>
<td>• Weather conditions at time of sampling</td>
</tr>
<tr>
<td></td>
<td>• Runoff conditions (surface overland and tile) occurring at the time of collection</td>
</tr>
<tr>
<td></td>
<td>• Note if pesticide applications (ground or aerial) have occurred in watershed since last sample collection</td>
</tr>
<tr>
<td></td>
<td>• Any other notable conditions</td>
</tr>
</tbody>
</table>

#### 2.5.2.2 MDA Electronic Sample Chain-Of-Custody Log-In Submission Form

MDA staff will have access to the current version of the surface water sample COC log-in form on the network drive (H:\PFMD\ENVIRONMENTAL\MONU\Monitoring and Assessment\Surface Water\SW LOGIN\yyyySWLOGIN). MDA staff will need to print the completed Organic and Inorganic sample COC log-in, in color, for sample submission to the MDA Laboratory.
2.5.2.2.1 Populating the MDA electronic sample COC log-in
The MDA electronic sample COC log-in is an Excel file and adherence to this date entry procedure is necessary in order to accurately document field and sample collection data. When the file is opened, “Read Only” and the “Enable Content” button should be selected.

Table 4 provides a list of field names found on the sample COC log in form, with instructions to the right of each field in the table.
### Table 4. MDA Electronic Sample COC Log-in Fields.

<table>
<thead>
<tr>
<th>Data Entry Field</th>
<th>Description / Instructions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inspector</td>
<td>Use dropdown to select appropriate name/organization</td>
</tr>
<tr>
<td>Delivered By</td>
<td>Use dropdown to select appropriate name/organization</td>
</tr>
<tr>
<td>Delivery Method</td>
<td>Use dropdown to select delivery method</td>
</tr>
<tr>
<td>Notes/Sampling Conditions</td>
<td>Enter notes related to field conditions at the time of sampling</td>
</tr>
<tr>
<td>Inspector Sample ID</td>
<td>Enter Sample Bottle ID</td>
</tr>
<tr>
<td>LIMS Sample Site</td>
<td>Use dropdown to select monitoring location</td>
</tr>
<tr>
<td>Sample Type</td>
<td>Use dropdown to select sample type. Hover over field for descriptions</td>
</tr>
<tr>
<td>Flow Condition</td>
<td>Use dropdown to select flow condition. “BF” = base flow, “SF” = storm flow</td>
</tr>
<tr>
<td>Collection Method</td>
<td>Use dropdown to select sample collection method</td>
</tr>
<tr>
<td>Sample Comments</td>
<td>Record weather comments and sample comments</td>
</tr>
<tr>
<td>Date Sampled</td>
<td>Enter the date and time (CST, military time) of sample collection. For grab and select QA/QC samples, the start and end date/time will be the same. For composite samples, the start and end date/time should be entered from the start of the composite to the end composite collection period. The date/time when MDA staff completed the field visit to collect the composite sample is not entered</td>
</tr>
<tr>
<td>Analysis and Thermal Preservation</td>
<td>Use the check boxes to request each analysis for each sample. Also indicate the thermal preservation method for each analysis type in the grey cell above the analysis.</td>
</tr>
<tr>
<td>Priority Level</td>
<td>Use dropdown to select the analysis priority level. “P3” will be used for most samples. “P1” should be used in emergency situations requiring immediate analysis and reporting, and “P2” should be used in instances requiring faster reporting. “P1” and “P2” should not be used without pre-approval from the MDA MAU supervisor, and only after pre-notifying the MDA Laboratory.</td>
</tr>
<tr>
<td>Field data entry</td>
<td>Enter field data (stage, t-tube reading, water temperature, etc.) in the far columns to the right of the analysis requested section</td>
</tr>
<tr>
<td>Double check all entries</td>
<td>Review all entries for accuracy</td>
</tr>
</tbody>
</table>

#### 2.5.2.2.2 Printing the MDA electronic sample COC log-in

MDA staff are required to print, in color, separate sample COC log-in forms for organic (pesticide) samples and inorganic (nutrients and sediment) samples. To print sample COC log-in form, move to the organic or inorganic tab. Select File⇒Print⇒Custom Scaling⇒Fit All Columns on One Page⇒”Print”
2.5.2.2.3 MDA electronic sample COC log-in data management

MDA staff members are required to update their running electronic log of all samples collected and submitted to the MDA Laboratory after each laboratory submission. This effort helps streamline the management of laboratory results and field data. Each MDA staff member must update their own running log of samples collected and submitted after each sample collection event to the most current file in the Monitoring Unit file on the MDA network (H:\PFMD\ENVIRONMENTAL\MONU\Monitoring and Assessment\Surface Water\SW LOG IN\yyyySWLOGIN).

To copy the electronic data from the electronic sample COC log-in form to the compiled page:

1. On the “SW Sample Login” tab, select the entire row for any row with sample collection data. Use numbered column on far left to highlight rows containing data. Once highlighted, copy data (control + c).
2. Click onto the “SWStaffFieldLoginCompileSheet” tab.
3. Click on the number of the first unused row to highlight it. In the clipboard section of the toolbar, select “Paste Values” or keystroke: Alt+S; V; Enter. All sample information should be transferred, along with new cells that are automatically populated based on hidden lookup tables. Analysis selected for the sample will have “True” in the cell indicating that results will be returned from the laboratory for those samples.
4. Open the master compile sheet on in the network folder listed above and select the approximate tab (staff name or office).
5. Copy information from each log-in into this file.
6. Save the file using “Save As” with the date in the file name. If the current date is already used, add a consecutive letter behind the date to save the file (i.e.” …09.21.2016b”). This process will ensure a back-up file is always available.

2.5.3 SAMPLE SUBMISSION TO MDA LABORATORY

Samples can be delivered directly to the MDA Laboratory by hand or through a commercial shipper. All submissions must include an accurate, complete, and dry sample COC log-in. Regardless of method, sample submission should not occur on the same day of collection, to allow samples to be chilled or frozen overnight. A temperature blank, filled with stream/river water, must be contained in each cooler upon arrival at the MDA Laboratory. The temperatures of samples will be recorded upon arrival at the MDA Laboratory. It is critical that all samples delivered to the MDA Laboratory be submitted at a temperature below 6°C. The sampler is responsible for ensuring sufficient ice is used during transport. Extreme care should be taken to ensure samples are not jeopardized during the submission process due to broken bottles or elevated temperatures.

Special projects may utilize other laboratories. Submission procedures should be established
prior to the collection of samples.

2.5.3.1 HAND DELIVERY
Samples can be delivered directly to the MDA Laboratory rear loading dock area access (601 Robert St N, St. Paul, MN 55155). Upon arrival at the MDA Laboratory loading dock, staff should ring the doorbell. Staff will be escorted into the receiving area of the MDA Laboratory. Large deliveries must be coordinated with MDA staff in St. Paul prior to arrival. Samples must be on ice and chilled below 6°C at the time of submission.

2.5.3.2 DELIVERY WITH COMMERCIAL CARRIER
Samples can be shipped in coolers with a commercial carrier (Spee Dee, UPS, etc.). Samples to be shipped to the MDA Laboratory should be placed in a cooler containing sufficient ice. Cooler packing instructions are available in Section 2.5. The sample chain-of-custody login should be placed in a sealed plastic bag to keep it dry inside the cooler and taped to cooler lid. Extreme care should be taken to ensure samples are not jeopardized during the submission process due to broken bottles or elevated temperatures. Shipments of samples should be delivered to:

Minnesota Department of Agriculture
Laboratory Services Division
601 North Robert Street
St. Paul, MN  55155

Cooperators with an MDA agreement, in which the MDA is paying for shipment, should request a shipment following the process described below. Shipping should not occur until samples are chilled and/or frozen overnight and shipped in a cooler with sufficient wet ice. Shipments should not be picked up on Friday, as shipments are only received at the MDA Laboratory Monday through Friday. If samples are collected on a Thursday or Friday, the pick-up should occur on Monday. The request for cooperator shipping can be made in advance (for example, a Monday pick-up can be submitted on the previous Thursday).

The MDA utilizes Spee Dee for cooperator sample shipments. This is a three day process.

**Day 1:** Request shipment pick-up by 3:00 PM.

Cooperators should contact the MDA mail room contacts below via e-mail or phone.

<table>
<thead>
<tr>
<th>Name</th>
<th>E-mail</th>
<th>Phone</th>
</tr>
</thead>
<tbody>
<tr>
<td>Joyce Walkosz</td>
<td><a href="mailto:Joyce.Walkosz@state.mn.us">Joyce.Walkosz@state.mn.us</a></td>
<td>651-201-6155</td>
</tr>
<tr>
<td>Debra Gennow</td>
<td><a href="mailto:debra.gennow@state.mn.us">debra.gennow@state.mn.us</a></td>
<td>651-201-6153</td>
</tr>
</tbody>
</table>
Cooperators must provide the following information in their request:

A. Date of requested pick-up.
B. Pick-up sample information
   Name of sampler and/or sampling organization and pick-up address.
C. Number of coolers in shipment.
D. Approximate weight of each cooler.
E. Include this statement “Shipment for statewide pesticide water quality monitoring”.
F. Laboratory delivery address:

   Minnesota Department of Agriculture
   Laboratory Services Division
   601 Robert Street North
   St. Paul, MN 55155-2531

**Day 2:** Prepare cooler for shipment and cooler pick-up.

Cooperator should prepare cooler according to the procedures in Section 2.5.4. The cooler should be kept in an air conditioned area prior to pick-up.

**Day 3:** Package delivered to MDA Laboratory

2.5.4 COOLER PACKING INSTRUCTIONS

The guidelines described below should be used for all shipments to the MDA Laboratory. Example photographs should be used to guide cooler packing procedures. This protocol is also included as Appendix B.

**Supplies**

- Cooler (> 3 day ice holding rating)
- Bulk bubble wrap (1/2 inch) for cooler lining
- Bubble wrap bags for all glass amber bottles
- Sealable plastic bags for each sample set
- Temperature blank (empty bottles provided)
- Cooler liner bag
- Zip ties
- Wet ice
- Completed sample chain-of-custody log-in form in sealed bag
## Cooler Packing Procedures:

1. Properly chill/refrigerate or freeze samples following standard operating procedures prior to preparing shipment to the MDA Laboratory. Shipping samples at ambient temperatures will quickly melt the ice in the cooler, and may affect the acceptance of samples at the MDA Laboratory.

2. Line the bottom of the cooler with bulk bubble wrap.

3. Line the cooler with cooler liner bag.

4. Prepare samples for shipping:
   a. Place amber glass bottles into bubble wrap bag and seal it.
   b. Place amber glass bottles and associated plastic bottles into sealed plastic bag. Use the same bag the empty sample bottles were shipped in.
   c. Place one temperature blank into a sealed plastic bag with samples. One temperature blank is required in each cooler, and should be collected following standard operating procedures.

5. Place sealed bags of samples upright inside cooler liner bag. Do not lay samples on their side or double stack. If possible, pack sealed bags limiting direct contact between glass bottles.

6. Fill the cooler liner with wet ice, ensuring wet ice is in contact with the sides and overtop of the sealed bags of samples. Ice should fill approximately 1/3 to 1/2 of the cooler. (A 28 quart cooler will require at least 8 pounds of ice; a 50 quart cooler will require at least 20 pounds of ice.)
### Cooler Packing Procedures:

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.</td>
<td>Twist cooler liner top to close it and use the provided plastic zip tie to close the cooler liner bag.</td>
</tr>
<tr>
<td>8.</td>
<td>If there is space between the cooler liner bag and the sides or top of the cooler, add additional bulk bubble wrap between the cooler liner bag and the cooler. Add enough bubble wrap to prevent any movement of the cooler liner bag inside the cooler during shipping.</td>
</tr>
<tr>
<td>9.</td>
<td>Place sample chain-of-custody (COC) log-in form in a sealed plastic bag and tape it to the bottom of the cooler lid. The sample COC log-in should only include samples contained in that particular cooler.</td>
</tr>
</tbody>
</table>
| 10.  | Seal the cooler with packing tape:  
   a. Tape along cooler where the lid meets the cooler.  
   b. Tape completely around the cooler in two locations preventing the cooler lid from opening.  
   c. Cooler handle should be taped down to avoid handle breakage / dropping during shipping.  
   d. Tape over the delivery address to protect it from falling off or getting wet (shown in step 12 photo). |
| 11.  | Keep packed cooler in an air conditioned, well shaded location prior to shipment pick-up by a commercial carrier. |
| 12.  | Do not ship on Fridays as the MDA Laboratory does not accept samples on weekends. |
| 13.  | The MDA Laboratory will return all packing materials and temperature blank bottle with the cooler. |
3.0 SUPPORTING INFORMATION

3.1 EQUIPMENT CLEANING
Equipment cleaning is a necessary step to eliminate sampling bias, reduce sample variability, produce comparable data, and prevent the spread of AIS. Equipment should be cleaned using the below procedure after each sample round, with DI equipment rinses in the field between sampling locations.

Supplies needed:
- Non-phosphate detergent (7X)
- De-ionized (DI) or distilled water
- Cleaning brushes
- Cleaning basin (plastic or stainless steel)
- Nitrile gloves

Cleaning Procedure:
1. Mix detergent according to label in a large basin with warm tap water.
2. Place equipment in basin and allow it to soak in the solution.
3. Use a bottle brush to mechanically remove any particles attached to the equipment.
4. Rinse at least three times with hot tap water, followed by triple rinsing with de-ionized water.
5. Allow equipment to air dry while inverted.
6. Once dry, visually inspect the equipment for spots indicating insufficient washing. If necessary, repeat cleaning process.

3.2 COOLER CLEANING
Cooler cleaning is a necessary step to prevent contamination concerns related to cross – contamination of samples broken during shipment and from commercial shippers. These procedures should be used at the beginning of the season for all coolers and ice packs, as well as periodically as needed throughout the season whenever sediment is observed in, or on the cooler or ice packs.

Coolers and ice packs are used in the field to preserve samples from time of collection until the samples are either submitted to the laboratory or placed in a refrigerator for temporary storage. Coolers and ice packs are also used in the delivery of samples to the laboratory via hand or commercial delivery service, and often contain samples from several sampling locations at any one time, or over several trips. While the MDA has never determined that a cooler contaminated a sample, it is important to limit any such concerns.
Coolers and ice packs should be cleaned using the same method as described in Section 3.0.

3.3 AUTOMATED SAMPLER COLLECTION BOTTLE CLEANING

Bottle washing is a necessary step to eliminate sampling bias, reduce sample variability and to produce comparable data. Clean bottles should be used after every runoff event or when the integrity of the bottles is in question.

Automated sampler collection bottles should be cleaned using the same method as described in Section 3.0.

3.4 AQUATIC INVASIVE SPECIES (AIS)

The collection of samples from waters with an AIS designation requires additional effort to prevent the transport of AIS. The MDA is permitted by the MN DNR to collect and transport water from AIS designated waters, and all field staff must carry a copy of the current permit while collecting and transporting water from AIS designated locations. All samples from AIS designated locations will be clearly labeled on the bottle and COC log-in form to alert the MDA Laboratory. The MDA completes an annual review of the AIS Infested Waters List maintained by the MN DNR. The MDA recommends all staff and cooperators follow the MPCA WPLMN AIS Standard Operating Procedures.

4.0 GENERAL SAFETY GUIDELINES

The overall safety and well-being of MDA staff and cooperators must be the highest priority when conducting field work and sample collection. Field activities should only occur if conditions (field or road) do not pose an immediate risk to the safety of field staff. The following tips are offered: however, field staff must use common sense and exhibit sound judgment to avoid unnecessary or unforeseen risks. If field staff are unsure if it is safe, the field staff should not proceed and return at another time when safety can be assured.

- Complete all required safety training.
- Prior to field activities, thoroughly read this SOP. Do not deviate from the procedures listed in the SOP without prior approval from MDA staff.
- Closed toe shoes, preferably steel toed, must be worn at all times in the field.
- At a minimum, personal clothing should provide protection from contact with irritating plants and biting insects (shorts should not be worn).
- Always wear a United States Coast Guard approved personal floatation device (PFD) whenever in contact (wading) with surface water.
- Do not enter the water if the stream depth (feet) multiplied by stream velocity (feet per second) exceeds your height (feet).
- When wading, always wear hip boots or waders as a barrier to cold water and unknown pathogens or pollutants.
- When wading, use a wading stick for improved balance and to prevent trips or falls.
Develop and share a daily field plan with a coworker that includes the following information:

- Location(s) of field work
- Estimated return time
- Emergency contact information

Develop a safety plan including nearest hospitals from each monitoring location.

- Wallet, cell phone and keys should be kept in a safe place.
- Keys used to access monitoring stations or gages should be on a separate floating key ring.
- Plan for varying risks at all anticipated monitoring locations including locations with heavy traffic and locations with remote access through steep terrain.

4.1 ROAD AND VEHICLE SAFETY

Sample collection from bridges poses a large risk to field staff due to passing traffic. To help mitigate this risk, the use of traffic control devices are essential and should be used during periods of heavy traffic. When possible, park your vehicle in a nearby field approach, driveway or other public parking lot. Field staff should always wear high visibility and reflective clothing when working near roadways. In addition, flashing vehicle warning lights and/or cones can be used to alert other drivers.

MDA staff members are required, and cooperators are encouraged, to complete a defensive driving course.

Vehicle Safety Checklist:

- Emergency medical kit
- Cell phone
- State road map
- Flashlight
- High visibility and reflective safety apparel
- Vehicle warning light
- Winter survival kit

4.2 WEATHER AND FLOOD AWARENESS

MDA staff and cooperators must monitor current and forecasted weather conditions prior to field work. Additional planning and preparation is required when field work is completed during poor weather conditions. Heavy precipitation makes travel hazardous, and may require a delayed departure or return. Do not park on the road or shoulder to wait for improved visibility or road conditions.

Flood conditions may create hazardous sample collection periods. Watersheds with a history of flash flooding should be known by all staff, and weather conditions should be monitored in these
watersheds. Do not drive through flooded areas as there may be damage to the roadway. Do not sample when lightening is occurring.
### Appendix A. MDA Contact information

<table>
<thead>
<tr>
<th>CATEGORY</th>
<th>STAFF</th>
<th>CONTACT INFORMATION</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monitoring Questions &amp; Fish Kill Reporting</td>
<td>David Tollefson</td>
<td>Phone: 507-206-2882</td>
</tr>
<tr>
<td></td>
<td>Hydrologist</td>
<td>Email: <a href="mailto:david.tollefson@state.mn.us">david.tollefson@state.mn.us</a></td>
</tr>
<tr>
<td>Monitoring Questions &amp; Fish Kill Reporting</td>
<td>Matt Ribikawskis</td>
<td>Phone: 507-206-2884</td>
</tr>
<tr>
<td></td>
<td>Hydrologist</td>
<td>Email: <a href="mailto:matthew.ribikawskis@state.mn.us">matthew.ribikawskis@state.mn.us</a></td>
</tr>
<tr>
<td>Monitoring Questions &amp; Fish Kill Reporting</td>
<td>Bill VanRyswyk</td>
<td>Phone: 507-344-3203</td>
</tr>
<tr>
<td></td>
<td>Hydrologist Supervisor</td>
<td>Email: <a href="mailto:bill.vanryswyk@state.mn.us">bill.vanryswyk@state.mn.us</a></td>
</tr>
<tr>
<td>Shipping Requests</td>
<td>Joyce Walkosz</td>
<td>Phone: 651-201-6155</td>
</tr>
<tr>
<td></td>
<td>MDA Mailroom</td>
<td>Email: <a href="mailto:Joyce.Walkosz@state.mn.us">Joyce.Walkosz@state.mn.us</a></td>
</tr>
<tr>
<td>Shipping Requests</td>
<td>Debra Gennow</td>
<td>Phone: 651-201-6153</td>
</tr>
<tr>
<td></td>
<td>MDA Mailroom</td>
<td>Email: <a href="mailto:debra.gennow@state.mn.us">debra.gennow@state.mn.us</a></td>
</tr>
<tr>
<td>Fish Kill Reporting</td>
<td>Minnesota Duty Officer</td>
<td>Phone: (651) 649-5451</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Phone: 1-800-422-0798</td>
</tr>
</tbody>
</table>
Appendix B. MDA Cooler Packing Guidelines

These guidelines should be used for all shipments to the MDA Laboratory. Example photographs should be used to guide cooler packing procedures.

Supplies

- Cooler (> 3 day ice holding rating)
- Bulk bubble wrap (1/2 inch) for cooler lining
- Bubble wrap bags for all glass amber bottles
- Sealable plastic bags for each sample set
- Temperature blank (empty bottles provided)
- Cooler liner bag
- Zip ties
- Wet ice
- Completed sample chain-of-custody log-in form in sealed bag
**Cooler Packing Procedures:**

<p>| | |</p>
<table>
<thead>
<tr>
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</tr>
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<tbody>
<tr>
<td>1.</td>
<td>Properly chill/refrigerate or freeze samples following standard operating procedures prior to preparing shipment to the MDA Laboratory. Shipping samples at ambient temperatures will quickly melt the ice in the cooler, and may affect the acceptance of samples at the MDA Laboratory.</td>
</tr>
<tr>
<td>2.</td>
<td>Line the bottom of the cooler with bulk bubble wrap.</td>
</tr>
<tr>
<td>3.</td>
<td>Line the cooler with cooler liner bag.</td>
</tr>
</tbody>
</table>
| 4. | Prepare samples for shipping:  
   a. Place amber glass bottles into bubble wrap bag and seal it.  
   b. Place amber glass bottles and associated plastic bottles into sealed plastic bag. Use the same bag the empty sample bottles were shipped in.  
   c. Place one temperature blank into a sealed plastic bag with samples. One temperature blank is required in each cooler, and should be collected following standard operating procedures. |
| 5. | Place sealed bags of samples upright inside cooler liner bag. Do not lay samples on their side or double stack. If possible, pack sealed bags limiting direct contact between glass bottles. |
| 6. | Fill the cooler liner with wet ice, ensuring wet ice is in contact with the sides and overtop of the sealed bags of samples. Ice should fill approximately 1/3 to 1/2 of the cooler. (A 28 quart cooler will require at least 8 pounds of ice; a 50 quart cooler will require at least 20 pounds of ice.) |
| 7. | Twist cooler liner top to close it and use the provided plastic zip tie to close the cooler liner bag. |
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<td>8.</td>
<td>If there is space between the cooler liner bag and the sides or top of the cooler, add additional bulk bubble wrap between the cooler liner bag and the cooler. Add enough bubble wrap to prevent any movement of the cooler liner bag inside the cooler during shipping.</td>
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<td>9.</td>
<td>Place sample chain-of-custody (COC) log-in form in a sealed plastic bag and tape it to the bottom of the cooler lid. The sample COC log-in should only include samples contained in that particular cooler.</td>
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| 10.  | Seal the cooler with packing tape:  
   - a. Tape along cooler where the lid meets the cooler.  
   - b. Tape completely around the cooler in two locations preventing the cooler lid from opening.  
   - c. Cooler handle should be taped down to avoid handle breakage / dropping during shipping.  
   - d. Tape over the delivery address to protect it from falling off or getting wet (shown in step 12 photo). |
| 11.  | Keep packed cooler in an air conditioned, well shaded location prior to shipment pick-up by a commercial carrier. |
| 12.  | Do not ship on Fridays as the MDA Laboratory does not accept samples on weekends. |
| 13.  | The MDA Laboratory will return all packing materials and temperature blank bottle with the cooler. |
Appendix C. Required information for Field Notes

- Site name and MDA station ID
- Name(s) of staff conducting site visit
- Date (mm/dd/yyyy)
- Military Time (Central Standard Time (CST))
  - Arrival Time
  - Sample Collection Time
  - Departure Time
- Measured water level (note “stage” or “tape down” value)
  - Include error range (i.e. +/- 0.02 ft.)
  - Note Time (Central Standard Time (CST)) of measurement
- Stage value from electronic gage (if applicable)
  - Note stage offset adjustments
- Transparency or water secchi tube with disc
- Rain gage status (if applicable: clear, plugged, etc.)
- Channel and channel control notes (beaver dams, moving sand, large debris, etc.)
  - Is river within banks, at bank full, out of bank, etc.?
- Flow condition
  - Base flow or storm flow (note position on hydrograph (rising, peak, or falling))
- Weather conditions
  - Notable weather in past 24-48 hours (rainfall totals/intensity, snowmelt, etc.)
  - Current conditions (temperature, wind, cloud cover, etc.)
- Watershed observations
  - Note the presence of ponded water in fields, active surface runoff, tile flow, etc.
  - Note agricultural operations (corn planting, spraying, etc.)
  - Estimate canopy coverage of agricultural fields
- Note any dead insects, invertebrates, or fish
  - Photograph and report immediately to the Minnesota Duty Officer (1-800-422-0798), MDA staff and MDA supervisor. Contact information can be found in Appendix A.
Appendix D. Automated Sampler Programming Notes

The following settings should be used on the ISCO 3700 and 6712 automated samplers at the MDA Tier 3 locations for the collected of composite samples.

ISCO 3700 Programming

The ISCO 3700 is operated as an equal-time composite sampler, however, the CR850 datalogger controls when the sampler pulses (every hour). The ISCO is programmed as a “Flow” based sampler to allow the datalogger to send the pulse to initiate each sampling event. The following steps will highlight how to configure and program the sampler.

Select “Configure”
Set Clock: Set time
Bottles and Sizes: 1 bottle, 3800 mL
Suction Line: Teflon, 3/8 inch, insert length
Liquid Detector: Enable, Rinse cycle = 1, No/disable suction head entry, Sample retry = 1
Programming mode: Basic
Start Time Delay: 0 minutes
Enable Pin: Master/Slave = no, Sample upon disable = no, Sample upon enable = no, Reset sample interval = yes
Event mark = pulse
Purge counts = 50 (default)
Tubing life: default
Sample ID: default
Run diagnostics: skip it

Select “Program”
Select “FLOW”
Enter 1 (sample every 1 pulse)
Select “No” to multiplex bottles

ISCO 6712 Programming

The ISCO 6712 is operated as an equal-time composite sampler, however, the CR850 datalogger controls when the sampler pulses (every hour). The ISCO is programmed as a “Flow” based sampler to allow the datalogger to send the pulse to initiate each sampling event. The following steps will highlight how to configure and program the sampler.

- Extended Programming
- 30 minute data interval
- 1 bottle, 9.45 liters, (user defined) ft suction line, auto suction head, 1 rinse, 0 retries
- One part program
- Pacing: FLOW, 1 pulse
  - Flow pacing, 1 pulse, No sample at start
  - Run continuously? No.
- 96 samples, 70 mL
- Enable: none-programmed
- Enable: once enabled, stay enabled, sample at enable
- Enable: 0 pauses and resumes
- No delay to start
- Run this program now? Yes (screen should read “sample 1 after 1 pulse”)
Appendix E. Campbell Scientific Data Logger Programming Notes
This appendix will provide basic information to help operate Campbell Scientific CR800 or CR1000 series loggers.

SDI-12 Addresses
The following sequence was provided by Decagon Devices.

Entering SDI12 Communication “Transparent” Mode
1. Connect to the datalogger by clicking the “Connect” button on the LoggerNet toolbar and click on “Connect” on the “Connect Screen”.
2. Select “Tools” from the top menu and click on “Terminal Emulator”
3. Click the “Open Terminal” button at the bottom of the window.
4. Press the “Enter” key a few times until a “CRXXXX>” prompt appears (i.e. CR1000>)
5. Type “SDI12” next to the prompt
   a. Note: the datalogger will exit this mode relatively quickly so if things don’t respond, repeat steps 4 and 5 again.
   b. The datalogger will respond with “Enter Cx Port 1, 3, 5 or 7” or something similar.
6. Enter the control port that the SDI-12 sensor is plugged in to.
   a. The datalogger will respond with “Entering SDI12 Terminal”

Verifying and Setting SDI12 Sensor Address
Note: Sensors must be addressed individually so only ONE sensor may be connected to the communication port at any time during addressing.
1. Connect first SDI-12 sensor to the communication port you entered in step 6 above.
2. Query its address by typing: ?!
   a. The sensor will return its address such that, if its address is “0” then the line will read: ?!0
3. Assign its new address by typing: 0AX! where X is the new address you want to assign the sensor.
   a. For example, if the sensor was assigned address “2” and you wanted to change it to “5” you would type: 2A5!
   b. The computer would return the new address so the line would look like: 2A5!5
   c. Possible addresses for SDI-12 communication with CR Basic type loggers are are 0 through 9, a through z and A through Z. With Edlog type loggers, only addresses 0-9 are recognized.
4. Repeat steps 2 and 3 for all sensors you need to address, choosing different addresses for each sequential sensor.
5. Once all sensors are individually addressed, all communication lines can be connected together at a single node and the datalogger will address each sensor individually using the addresses that we assigned and the appropriate SDI-12 program.