SURFACE WATER QUALITY PROGRAM
&
FEEDLOT PERMIT PROGRAM

STANDARD OPERATING PROCEDURES

FIELD WATER QUALITY SAMPLING

Revision III

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1.0 SURFACE WATER QUALITY PROGRAM AND FEEDLOT PERMIT PROGRAM DESCRIPTIONS

This document describes standard operating procedures for field water quality sampling for the Surface Water Quality Program (SWQP) and the Feedlot Permit Program (FPP).

The SWQP standards and monitoring section routinely monitors a network of South Dakota rivers and streams called the Water Quality Monitoring (WQM) network. In addition, the standards and monitoring team may collect water samples during Use Attainability Analyses (UAAs), fish kill investigations, complaint investigations, and special short-term monitoring projects. The surface water discharge permit section and the Feedlot Permit Program may also collect water quality samples during routine facility inspections, compliance sampling, and complaint investigations.

Environmental data collected by the SWQP and FPP may be used to determine 1) trends in water quality, 2) support of beneficial uses and water quality standards, 3) compliance with permit conditions, and/or 4) causes/sources of pollution. More information regarding SWQP and FPP sampling activities is available in the Quality Assurance Project Plan for the Surface Water Quality Program and Feedlot Permit Program (SWQ/FPP QAPP).

2.0 PRE-SAMPLING PROCEDURES

SWQP and FPP personnel conduct sampling work to meet a variety of goals/purposes. All sampling work requires pre-planning. During the planning stage, the scope of sampling to be performed is modified for the purpose of obtaining data necessary for meeting a specific and desired goal or purpose. Pre-planning procedures common to all sampling include the following:

1. Identify the objectives for sampling;
2. Review any existing data for the site/waterbody to be sampled;
3. Identify additional data requirements, including types of samples/characteristics and sampling frequency;
4. Examine maps and diagrams of the area/waterbody to be sampled;
5. Make a list of proposed sampling sites;
6. Check the operation of all required sampling equipment;
7. Load all required sampling equipment and obtain all required sample bottles, forms, documents, and field books;

A. Sample bottles may be obtained from the laboratory that will do the analysis

B. Deionized (DI) and "Polished" water may be obtained from the laboratory that will do the analysis.

8. Notify all stakeholders, including landowners, businesses, and city, state, federal, or tribal agencies;
9. Procure permission to access private property;
10. Perform reconnaissance (as necessary) of the proposed sampling sites and the general area/waterbody to be sampled;
11. Determine if the plan for sampling requires revision; and
12. Proceed with sampling activities.

3.0 DOCUMENTATION AND REPORTING

A. Documentation

Field recordkeeping activities are required. Recordkeeping is accomplished using either a field notebook or pre-printed project forms. It is recommended that all field notebooks and pre-preprinted project forms be waterproof. A standard format for field notebook recordkeeping is not required. All field records should include the following:

1. Field Notebook

a. Date and time;

b. Station ID (unique identification);

c. Station location (street number, avenue number, driving directions, WQM number, or GPS location);

d. Types of meters being used;

e. Meter calibration readings IF a meter calibration logbook is not kept with the meter (most meters will have an accompanying logbook for recording calibration activities);

f. Comments regarding any meter damage or difficulty in operation or calibration;

g. Weather conditions that could impact water quality (high wind, rainfall and runoff, temperature);

h. Velocity or discharge measurements (if flow measurements are taken);

i. The following field measurements are recorded for every sample site based on equipment used (if a measurement is not recorded, the rational should be stated in the notebook):

   i. air temperature;

   ii. water temperature;

   iii. pH;

   iv. specific conductance; and

   v. dissolved oxygen.

j. Fish size, length, species, and visual condition (if fish are collected);

k. Method of fish collection (net type, time and date set, length of stream segment, etc.);

l. Method of biological sample collection (net type, sampling equipment type, depth, etc.);

m. Discuss and identify all photographs;

n. Document site conditions/visual observations (riparian vegetation, bank and stream bottom, stream incision and definition, water appearance, flow, public access);

o. Document the name of any individual encountered during sampling and summarize the conversation along with any pertinent information.

2. Pre-printed project sheets

a. Completely fill out all applicable “blanks” on any pre-printed project forms;

b. All the information discussed in the section (1. Field Notebooks) must be gathered for each site.

3. Laboratory sheets

A SDDENR Water Quality Data Sheet (see SWQ/FPP QAPP) must be completely filled out and submitted with all laboratory samples. This provides the laboratory with information about the water sample and directs the laboratory on which analyses are requested.

4. Bottle labels

All bottles must be labeled prior to sample collection. At a minimum, the label must include the station identification, sample date, sample time, and bottle identification (Bottle "A"). The bottle will get wet, so ensure that bottle labels and marking pen are water resistant so the label stays affixed and the pen ink does not run.

B. Reporting

Data collected as a result of sampling is recorded in the following ways:

1. Recorded in appropriate database, hard copies of data are also maintained;
2. Recorded in reports;
3. Hard copies filed.

4.0 INSTRUMENT/EQUIPMENT CALIBRATION, CARE, AND OPERATION

Each field instrument must be inspected prior to use, calibrated, and operated according to manufacturer specifications. If problems with any field instrument are encountered, the user should consult the manufacturer's manual, the project manager, and/or call the manufacturer. Calibrations and instrument observations must be recorded in the calibration log book prior to field use.

General calibration procedures and necessary instrument inspections are presented below:

A. Dissolved Oxygen (DO) Meter - YSI 200

Inspect the DO membrane. It should be free of scratches, tarnish, and bubbles. Otherwise the membrane needs to be replaced and/or probe cleaned.

Make sure the sponge in the calibration chamber (Plastic Probe Holder) is moist. If the sponge is dry, wet it with water. Pour excess water out of the

calibration chamber and place over probe. Keep meter from extremes in temperature such as freezing and extreme heat (do not leave in vehicle!).

1. Turn meter on by pressing the Green Button. The display screen will read “ovEr"; this is a self-diagnostic test. Wait for meter readings to stabilize. Stabilization may take up to10 minutes.
2. Write the Date and Time in the meter’s calibration log book.
3. When the meter readings have stabilized Press the “CAL” button. Cal will appear in lower left of screen.
4. Meter prompts for mBar data. Arrow up or down to enter the correct Barometric pressure in mBars. (To get mBars data, use a smartphone or call the SWQP & FPP secretary and ask for the barometric pressure for your location. This can be found on the NOAA internet site, http://www.weather.gov/. When at this site enter the nearest Zip Code and press “Enter”. A weather report for that town with the current conditions will appear which includes the Barometric pressure in inches with the Barometric pressure in mBar following in parenthesis. Convert Barometric pressure from inches Hg to mBars (inches Hg x 33.85=mBars).
5. Press “Enter”. The meter should give you the DO in percent.

   Press “Mode” and you should have a salinity value of 0.0 ppt for deionized (DI) water. (If not 0.0, arrow up or down to 0.0).

   Press “Enter”. “Cal” will no longer be visible on the screen. Write the DO% and temperature in log book. The DO% will typically range between 80-120%. Values above 120% are suspect and the DO membrane should be inspected or changed, or the DO probe may need to be replaced.
6. The DO meter is calibrated and ready for use. Press the Mode key to toggle between DO percentage and ppm. SDDENR uses mg/L or ppm (which are equivalent units).
7. Place the DO probe in the water being measured. Take care that the probe is not placed in mud. Gently swirl the probe and allow the meter time to stabilize. Record the DO reading in mg/L or ppm in the field log book or pre-printed project sheets.
8. To clean the DO probe and replace the membrane, unscrew and discard the blue, black, or yellow membrane cap.
9. Rinse the probe with water. Wet the sandpaper disc provided in the tool kit with water and GENTLY polish the metal anode (only enough to the remove any tarnish). Repeat rinsing the probe.

10. Invert the blue, black, or yellow membrane cap and fill using the potassium chloride solution available in the tool kit. Thread the cap back onto the probe. It is ok for the potassium chloride solution to overspill. Ensure that there are no air bubbles trapped. If there are air bubbles visible, remove the cap, refill with potassium chloride solution, tap the cap to dislodge bubbles, and thread the cap onto the probe.

The YSI 200 DO meter calibration is based on barometric pressure. Recalibrate as often as necessary as the barometric pressure may change over time or distance.
Important: When collecting a DO measurement, the water must be moving past the DO probe at a speed of ½ foot per second to overcome oxygen consumption; otherwise, move the probe in a swirling motion to compensate!

B. pH Meter – YSI 100

Write date and time in the pH meter calibration log book. Connect pH and temperature probes to the meter. Inspect the buffer and electrode solutions for dirt or slime. Change out the buffer and electrode solutions as often as necessary to maintain clean solutions. Keep the meter from extremes in temperature such as freezing and extreme heat (do not leave in your vehicle). For typical field sampling a 2-point calibration is used. You should use calibration solutions that bracket the expected pH of the water you will be testing. If the pH is expected to be acidic, use pH 4.00 and pH 7.00 buffer solution. If the pH is expected to be neutral or basic, use pH 7.00 and pH 10.00 buffer solutions.

1. Turn the meter ON and unscrew the electrode storage solution.
2. Rinse the pH probe in distilled water.***
3. Place the probe in the 7 pH buffer solution.
4. Allow temp readings to stabilize.
   Then press and hold “STAND” for 3 seconds to calibrate.
5. “WAIT” flashes until the meter detects a stable reading. Then “SLOPE” will start flashing. This means the first point has been calibrated.
6. Rinse the pH probe in distilled water.***
7. Place the probe in the 10 pH buffer solution.
8. Allow temp readings to stabilize. Then press “SLOPE”.
9. “WAIT” flashes until the meter detects a stable reading. When the meter calibrates the second point it will beep twice. “STAND” and “SLOPE” display steady.

10. The meter is now dual point calibrated and ready for use.

When done sampling rinse the pH probe with distilled water.***
If taking more than one sample rinse the probe between samples.

Turn meter OFF and return probe to electrode storage solution.

***Failure to rinse probe contaminates pH buffer and electrode storage solution which leads to poor calibration and data!

C. Conductivity Meter – YSI

Write the date and time in the conductivity meter calibration log book. Keep the meter from extremes in temperature such as freezing and extreme heat (do not leave in your vehicle).

1. Turn the instrument on and allow it to complete its self-test procedure.
2. Place at least 3 inches of conductivity calibration solution in clean beaker.
3. Insert the probe into the beaker deep enough to completely cover the oval shaped hole on the side of the probe.

a. DO NOT rest the probe on the bottom of the container - suspend it off the bottom at least ¼ inches.

4. Press the “MODE” button until the instrument is reading specific conductance.
5. Allow at least 60 Seconds for the temperature reading to become stable.

a. Swirl the probe to dislodge any air bubbles from the electrode.

6. Press and release both the UP arrow and DOWN arrow buttons at the same time to enter the calibration mode.
7. Use the UP arrow or DOWN arrow key to adjust the reading on the display until it matches the value of the calibration solution you are using.
8. Once this display reads the exact value of calibration solution being used (the instrument will make the appropriate compensation for temperature variation from 25°C), press the “ENTER” key. The word “SAVE” will flash across the display for a second indicating that the calibration has been accepted.
9. Record the calibration information in the calibration log book.

10. Rinse the probe with water. The meter is calibrated and now ready for use.

11. Place the specific conductance probe in the water being measured. Ensure the probe is not placed in the mud. Allow adequate time for the meter to stabilize. Record the specific conductance value in the field log book or on pre-printed project sheets.

D. Multimeter – YSI 556

Write the date and time in the multimeter calibration log book. The meter should always stay connected to the probe bulkhead. Use fresh calibration solutions with each calibration. Keep the meter from extremes in temperature such as freezing and extreme heat (do not leave in your vehicle).

Accessing the Calibration Screen

1. Press the “ON/OFF” button to display the run screen.
2. Press the “Escape” button to display the main menu screen.
3. Use the arrow keys to highlight the Calibrate selection.
4. Press the “ENTER” button. The calibration screen will be displayed.

Specific Conductance Calibration

This procedure calibrates specific conductance (recommended), conductivity and salinity. Calibrating any conductivity option automatically calibrates the other two options. The specific conductance calibration affects calibration of other parameters and should always be completed first.

1. From the calibration screen, use the arrow keys to highlight the Conductivity selection.
2. Press “ENTER”. The conductivity calibration selection screen is displayed.
3. Use the arrow keys to highlight the Specific Conductance selection.
4. Press “ENTER”. The Conductivity Calibration Entry Screen is displayed.
5. Place a small amount of conductivity solution in the calibration cup. Thread the calibration cup onto the bulkhead and gently swirl to rinse. Discard the used solution.
6. Fill the calibration cup about ¾ full of conductivity solution. If possible, the conductivity standard should be within the same range

as the samples you plan to measure. Conductivity solution is prepared for SDDENR by the Department of Health Laboratory.

7. Carefully immerse the sensor end of the probe module into the solution. Thread the calibration cup onto the bulkhead. Gently swirl to remove any bubbles from the conductivity cell. The sensor must be completely immersed past its vent hole.
8. Use the keypad to enter the calibration value of the standard you are using. The last value used will automatically appear on the screen.
9. Press “ENTER”. The Conductivity Calibration Screen is displayed.
10. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.
11. Observe the reading under Specific Conductance. When the reading shows no significant change for approximately 30 seconds, press “ENTER”. The screen will indicate that the calibration has been accepted and prompt you to press “ENTER” again to continue.
12. Press “ENTER.” If you fail to press “ENTER” the second time, specific conductance will not calibrate. You will be returned to the Conductivity Calibrate Selection Screen.
13. Press “Escape” to return to the calibrate menu.
14. Rinse the probe sensors.

pH Calibration

1. In the calibration menu, use the arrow keys to highlight the pH selection.
2. Press “ENTER.” The pH calibration screen will be displayed.
3. Select the 1-point option only if you are adjusting a previous calibration. If a 2-point or 3-point calibration has been performed previously, you can adjust the calibration by carrying out a one point calibration. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to select only one pH buffer.
4. Select the 2-point option to calibrate the pH sensor using only two calibration standards. Use this option if the media being monitored is known to be either basic or acidic. Use two buffers that bracket the expected pH of the media. **You will typically do a 2-point calibration.**
5. Select the 3-point option to calibrate the pH sensor using three calibration solutions. In this procedure, the pH sensor is calibrated with a pH 7 buffer and two additional buffers. The 3-point calibration method assures maximum accuracy when the pH of the media to be monitored cannot be anticipated. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to select a third pH buffer.
6. Use the arrow keys to highlight the 2-point selection.
7. Press “ENTER.” The pH entry screen will be displayed.
8. Starting with the lowest pH buffer, pour a small amount of the buffer solution into the calibration cup, thread the calibration cup onto the bulkhead and gently shake to thoroughly rinse the cup and probe. Discard the buffer solution. This buffer is used as a rinse at this step.
9. Fill the calibration cup about half full with the lowest pH buffer solution (pH 4.00 or 7.00). Thread the calibration cup onto the bulkhead. Ensure that there is enough solution that the glass ball on the pH probe is submerged.
10. Gently swirl the probe to remove any bubbles from the pH sensor and to ensure good contact between the pH probe and buffer solution.
11. Use the keypad to enter the value of the buffer (pH 4.00 or 7.00) you are using at the current temperature.
12. Press “ENTER.” The pH calibration screen is displayed.

13. Allow the temperature to stabilize before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.
14. Observe the reading under pH, when the reading shows no significant change for approximately 30 seconds, press “ENTER”. The screen will indicate that the calibration has been accepted and prompt you to press “ENTER” again to continue.
15. Press “ENTER”. This returns you to the specified pH calibration screen.
16. Rinse the probe module, transport/calibration cup and sensors in tap or purified water and dry.
17. Repeat steps 5 through 13 above using the second pH buffer (pH 7.00 or 10.00).
18. Press “ENTER”. This returns you to the pH calibration screen.
19. Press “Escape” to return to the calibrate menu.
20. Rinse the probe module and sensors in tap or purified water.
21. To clean the pH probe, place dilute hydrochloric acid solution (10 to 20% - you may need to mix the solution in-house) in the calibration cup for approximately 10 minutes. Using a bottle brush, GENTLY brush the glass probe to dislodge any accumulated debris. Thoroughly rinse with water and calibrate prior to use.

Dissolved Oxygen Calibration

Important: The instrument must be powered on for at least 20 minutes to polarize the DO sensor before calibrating.

1. In the calibration screen, use the arrow keys to highlight the Dissolved Oxygen selection.
2. Press “ENTER”. The dissolved oxygen calibration screen will be displayed.
3. Use the arrow keys to highlight the DO% selection.
4. Press “ENTER.” The DO Barometric Pressure Entry Screen will be displayed.
5. Place approximately 1/4 inch of water in the bottom of the transport/calibration cup.
6. Place the sensor module into the transport/calibration cup. Make sure that the DO sensor is not submerged in the water.

7. Engage only 1 or 2 threads of the transport/calibration cup to ensure the DO sensor is vented to the atmosphere. This is necessary to replenish oxygen as the probe consumes it.

8. Press “ENTER”. The DO% saturation calibration screen will be displayed.

9. Allow sufficient time for the air in the transport/calibration cup to become water saturated and for the temperature to reach equilibrium before proceeding. The current values of the all enabled sensors will appear on the screen and will change with time as they stabilize.

10. Observe the reading under DO%. When the reading shows no significant change for approximately 30 seconds, press “ENTER”. The screen will indicate that the calibration has been accepted and prompt you to press “ENTER” again to continue.

    Important: If you do not press “ENTER” the second time the DO will not be calibrated.

11. Press “ENTER”. This returns you to the DO calibration screen.

12. Press “Escape” to return to the calibration menu and rinse the sensors.

13. To clean the DO probe and replace the membrane, unscrew and discard the blue, black, or yellow membrane cap.

14. Rinse the probe with water. Wet the sandpaper disc provided in the tool kit with water and GENTLY polish the metal anode (only enough to the remove any tarnish). Repeat rinsing the probe.

15. Fill the blue, black, or yellow membrane cap using the potassium chloride solution available in the tool kit. Thread the cap back onto the probe. It is ok for the potassium chloride solution to overspill. Ensure that there are no air bubbles trapped. If there are air bubbles visible, remove the cap, refill with potassium chloride solution, tap the cap to dislodge bubbles, and thread the cap onto the probe. Recalibrate prior to use.

Taking a Measurement

After calibrating all parameters, the YSI 556 is now ready for use. To take a water quality measurement:

1. Press “Escape” to return to the Run Menu or arrow up or down to highlight “Run” on the main screen menu.

2. Place the sensors into the water. Take care to ensure the sensors are not placed in mud and that the flow of water is sufficient to

overcome oxygen consumption by the DO probe (swirl the probe in the water if flow is inadequate).

3. Allow the meter sufficient time to stabilize. You may electronically record the measurements by pressing "ENTER." This will store the measurement in a data file in the YSI 556. Manually record the measurements in the field log book or on pre-printed project sheets. If a water quality sample is also being submitted, record the measurement information on the lab sheet.

E. Flow meter – SonTek FlowTracker

This flow meter is used to calculate stream flow or discharge. This meter uses sonar to detect water velocity. The operator inputs the location (on the tape line) and depth so the unit may calculate discharge. For best results and to reduce errors, select an area of the stream with minimal water turbulence and minimal underwater obstacles (rocks, algae, plants). A slow moving segment of the stream with a flat bottom is ideal. Take care not to disturb sediment on the bottom of the stream bed as this will cause sonar errors. Refer to Figure 1.

1. Attach the sonar probe to the wading rod and tighten the set screw.
2. Set up a tape line. While standing in the water facing downstream, the "right bank" is on your right side. This is the side where you will start.
3. On the right bank, use a stake to place the start of the tape line. Stake the tape line high enough up the bank so that the tape does not get swept by the stream and place in soil secure enough so that the line may be taunt.
4. Cross the stream to the left bank allowing tape to reel out. Secure the end of the tape line to the left bank with a stake. Note the measurement of the tapeline at the edge of the left bank. Subtract the measurement of the starting edge on the right bank to determine the stream width. It is ideal to measure 10 to 20 stations (locations) at equal intervals so that no more than 10% of stream discharge is represented at any one stream location. This may not be possible on very narrow streams. At a minimum, measurements may be made every 3 inches. For example, the tape line measures 6.5 feet at the right bank shore and 32 feet at the left bank shore. Subtracting 6.5 from 32 results in a stream width of 25.5 feet. In order to prevent greater than 10% stream discharge in any one stream location, at least 10 stations should be the measured. 25.5 feet divided by 10 stations would result in increments of 2.5 feet. However to prevent greater than 10% discharge in any one station

measurement, increments of 1.5 or 2 feet may be more appropriate. Determine the station intervals based on stream width.

5. Turn on the FlowTracker meter. The startup screen will appear.
6. Press the “ENTER” button. The Main Menu will display.
7. Press the “3” button to start the data run. This will take you to the data file name screen.
8. Press the “1” button to input the station name (StationID if it fits). Input a unique name. The name can only be 8 characters maximum, so you may need to abbreviate. Press the “ENTER” button once the name is input.
9. Press the “9” button to accept the name.
10. Press the “9” button again to start the data run. At this time the display message will read “Press QC Menu at any time for Gauge data Enter to continue.” Press “ENTER.”
11. The Automatic QC Test will appear. A QC test must be conducted once each day that the flow meter is used. Press “1” to run test or “2” to skip the test. If running the test, a message will display to “Put probe in moving water away from any underwater objects. Press enter to start.” Following those instructions, place the probe/wading rod in the water so that the direction of the flowing water is perpendicular to the direction of the sonar. Make sure there are no underwater objects such as plants, rocks, or debris. Hold the probe/wading rod still and upright. Press the “ENTER” button. The meter will begin a self-diagnostic QC test. Only proceed if the QC test passes.
12. The Starting Edge screen will appear. At the right bank, locate the measurement on the tapeline of the edge of the water. Press the blue “Set Location” button and use the numeric keys to enter the measurement of the tapeline at the edge of the water.
13. Press the gray “Next Station” button. This will take you to Station 1 – which correlates with the location on the tape line. The location of Station 1 may need to be adjusted based on the stream width. The first station (Station 1) and the last station measured on the left bank must be half the interval of the rest of the stations. This is to accommodate for slope of the stream from the stream edge to the first and last station. To adjust the location of Station 1, Press the blue “Set Location” button and manually enter the appropriate location. Go to that location on the tape line.

14. Press the blue “Set Depth” button and use the numeric keys to enter the depth of the water based on the stream depth measurement on the wading rod at Station 1.

15. Holding the meter still and upright, press the blue “Measure” button. The meter will begin taking measurements or “pings.” If there are any errors, correct the source of the error if possible and repeat the measurement. It may take several attempts per station. Press “1” to accept the measurement or “2” to repeat the measurement.

16. Once you accept the measurement, the meter will go to Station 2 and so forth. At each station, you must verify you are at the appropriate location on the tape line and enter the appropriate depth based on the measurement on the wading rod.

17. After the last station has been recorded, press the gray “End Section” button. The screen will display a prompt to press “End Section” again to end the section. The Ending Edge screen will display. Press the blue “Set Location” button and key in the corresponding location of the left edge on the tapeline.

18. Press the gray “Calculate Disch.” button. The screen will display a prompt to press the Calculate Discharge button again to confirm. Press the “Calculate Disch.” button again.

19. The meter will display information. Press the “Enter” button to continue viewing data. Press the “0” button to exit. Important: After viewing the data, you must press the “0” button to exit in order for your data to be saved.

20. Record the flow discharge measurement in cubic feet per second in the field log book or pre-printed project sheet.

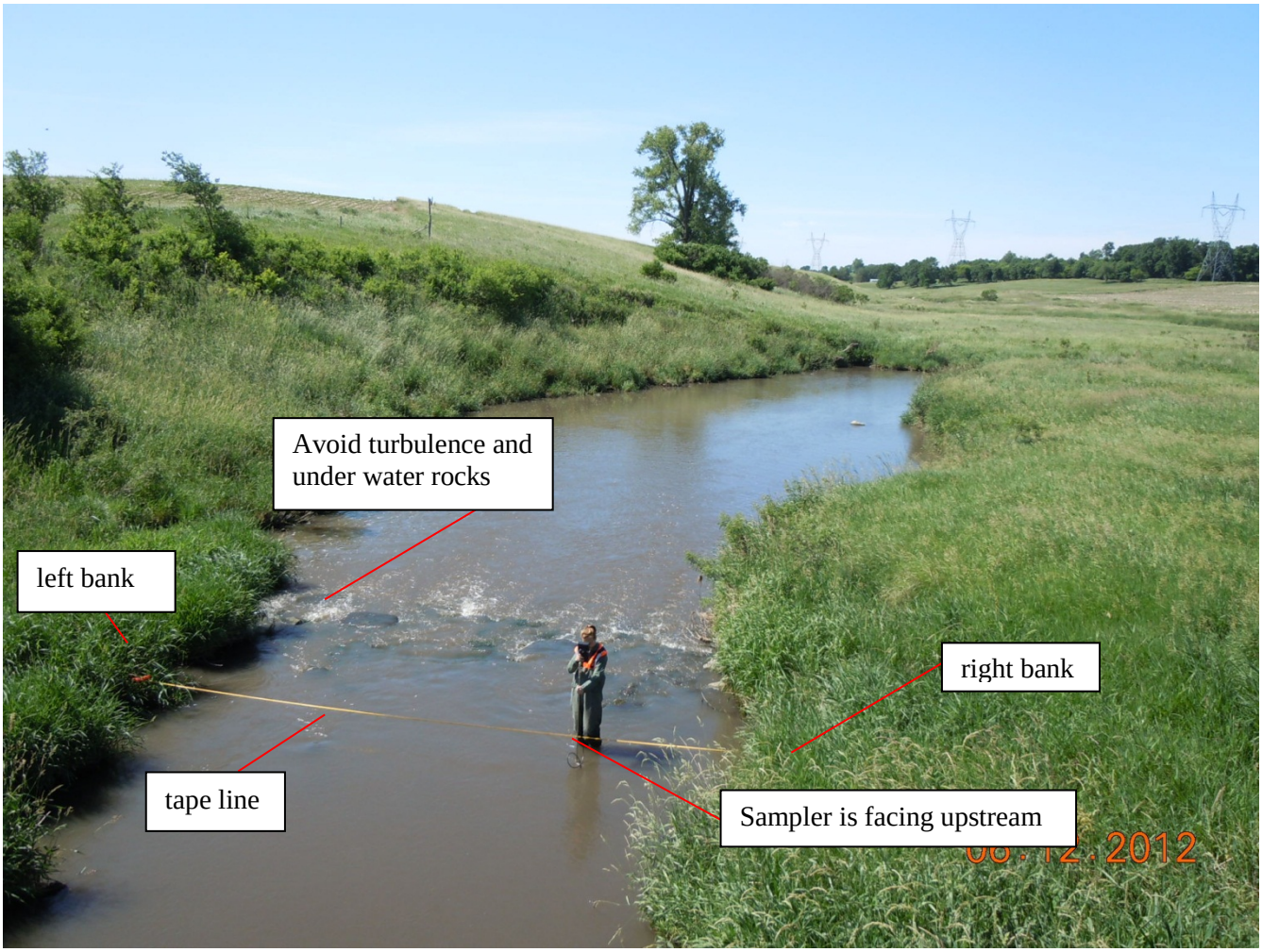

Figure 1. Taking a flow measurement with SonTek FlowTracker.

F. Van Dorn sampler

The Van Dorn sampler may be used to collect water from a lake or wetland. It may be used to collect a grab sample at a specified depth or a composite sample at multiple depths (surface, middle, and bottom). Rinse the sampling apparatus thoroughly with water from the sampling site prior to collection of samples. Surface samples should be collected approximately 1 meter below the surface of the water. Bottom samples should be collected from 0.5 meter above the lake bottom. Care must be taken not to come in contact with the lake bottom, as this may result in suspended bottom sediment in the sampler. Refer to Figure 2.

1. Make sure the pin release mechanism is in good working order.

2. Press down the pin release and pull plunger from the end nearest the trip release and hook the cable loop of the plunger into the appropriate slot in the pin release mechanism.
3. Release the pin release and ensure the cable loop is completely around the pin (not pinched by the pin).
4. Pull the plunger and cable from the opposite end and hook the cable loop to the opposite pin on the pin release. The Van Dorn sampler is now ready to be used to collect a water sample.
5. Lower the sampler into the water and stop the sampler at the appropriate depth. Send the messenger down the line to trip the sampler.
6. Pull the filled sampler up from the lake. When the bottom sample is collected, check for an excessive amount of bottom sediment or turbidity. If the sample appears turbid, discard the sample and repeat steps 2 through 6.
7. Once the sample is acquired, fill the sample bottles appropriately.
8. Before lengthy storage, rinse the Van Dorn sampler with distilled water.

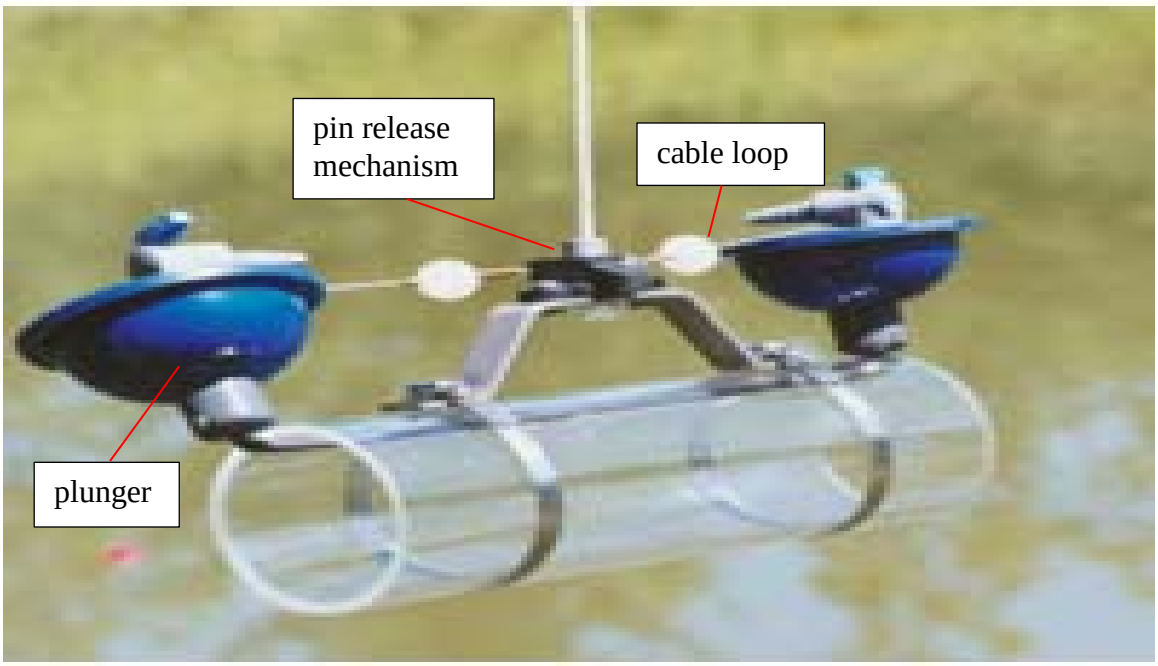

Figure 2. Van Dorn sampler.

5.0 LABORATORY SHEETS AND CHAIN-OF-CUSTODY

The majority of samples collected for ambient monitoring, fish kills, complaints, and other sampling projects do not need complete custody documentation. However, under certain conditions, such as compliance investigations, SDDENR must be able to prove that any analytical data offered into evidence accurately represent environmental conditions existing at the time of sample collection. Due to the evidentiary nature of such samples, possession must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. It must be clearly demonstrated that none of the involved samples could have been tampered with during collection, transfer, storage, or analysis. SDDENR chain-of-custody protocols and procedures are described below.

Documentation

To maintain and document sample possession, the following chain-of-custody procedures are followed:

1. Sample Custody - A sample is under custody under one of the following conditions:

    a. It is in your direct possession (you are holding it).

    b. It is in your direct line-of-sight after being in your possession (you can see it).

    c. It was in your possession; you locked it up or placed it in a sealed container to prevent tampering (no one can access the sample without leaving evidence of access, e.g. seal broken, tape removed, etc.).

    d. It is in a designated, secure area (typical evidence holding area).

Field Custody

1. The project officer will advise laboratory personnel that a sample requiring chain-of-custody will be collected and will specify the approximate date and time that it will arrive at the laboratory. In instances where date and time are not known in advance of a field trip, the laboratory should be notified as soon as possible about the arrival of such samples.
2. The samples must be collected in accordance with required and established methods set forth in this SOP, the Quality Assurance Project Plan (SWQ/FPP QAPP), and 40 CFR Part 136 (or other applicable section).

Transfer of Custody

1. To establish the documentation necessary to trace sample possession, a Chain-of-Custody Record (refer to SWQ/FPP QAPP) must be filled out and accompany each set of samples. The record should accompany the water quality data form and the samples to the laboratory. This record tracks sample custody transfers between the sampler and laboratory analysts. At a minimum, the record should contain:

    a. The StationID or sample identification;

    b. The signature of the collector and witnesses when present;

    c. The date and time of collection; place and address of collection;

    d. Substances sampled;

    e. Signatures of persons involved in the chain of possession; and, inclusive dates of possession.

All chemical water samples collected using this SOP utilize the SDDENR Water Quality Data Sheet (commonly referred to as the lab sheet), Fish Flesh Chain of Custody, or the SDDENR Chain of Custody form (refer to SWQ/FPP QAPP for all forms) as the laboratory data sheet and chain-of-custody document. When properly signed by all affected personnel, the SDDENR Water Quality Data Sheet and Fish Flesh forms comply with chain of custody requirements. The separate SDDENR Chain of Custody form is not additionally required. The SDDENR Chain of Custody form will only be used in special circumstances or in times when the other forms are not appropriate for the sample.

2. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis. The samples for each shipping container shall be placed in the large plastic bags provided by the laboratory.

3. If samples are split with a source or government agency, it will be noted in the remarks section of the Chain-of-Custody Record. The note should indicate with whom the samples are being split and be signed by both the sampler and recipient.

4. Each transfer of sample custody must be documented on the Chain-of-Custody Record.

5. The Chain-of-Custody Record identifying its contents will accompany all shipments. The original record will accompany the shipment, and the project officer will retain a copy and place it in the project file.

6. The laboratory should have assigned laboratory custodians who are responsible for overseeing the reception of all controlled custody samples.

7. When the samples are not in the immediate possession of the individual having official custody, they must be kept in a locked enclosure.

8. After the laboratory has completed the sample analysis, the Water Quality Data form and the Chain-of-Custody record will be returned to the appropriate program. These items will be kept on file for at least five years. Access to the Chain-of-Custody file is limited to program personnel.

Delivery or Shipment of Samples

Samples must be packed in coolers on loose ice for shipment or delivery to the laboratory. You must include appropriate paperwork. Make sure the container does not leak and all shipping or delivery labels are legible. Generally, samples need to be chilled to less than 6^{o} Celsius. Make sure there is adequate ice in the cooler to keep samples chilled during transit.

1. Completely fill out a SDDENR Water Quality Data Sheet (see SWQ/FPP QAPP) for each sample station.

2. If the samples are being shipped or sent with the courier, place all sample containers in a large plastic bag. Add loose ice to the bag and tie closed. Place the bag inside the shipping cooler.

3. Make sure that all SDDENR Water Quality Data Sheets are filled out completely. Protect the documents from getting wet by placing them in a plastic bag and putting them in the shipping cooler.

4. Securely seal the cooler with packing tape.

5. Shipping coolers are shipped via mail (USPS, FedEx, etc.), delivered by the courier, or delivered by the sampler to the appropriate laboratory.

6.0 QUALITY ASSURANCE

General Information and Handling Procedures

This section is supplemental to quality activities and requirements outlined in the SWQ/FPP QAPP. Refer to the SWQ/FPP QAPP for specific information on data quality objectives, quality activities, and corrective actions.

1. If several locations are to be sampled during one sample run, which includes both clean and polluted sites, sampling should progress from the clean areas to the polluted areas. This lessens the chance of unintentional contamination of cleaner samples through the use of contaminated sampling equipment (waders, meters).
2. Sampling equipment (meters, graduated cylinders, field bottles, etc.) should be triple rinsed with water from the waterbody being sampled prior to collecting the water sample.
3. The sample container and preservation must be appropriate to the sampled parameter. See figures 3, 5, and 6.
4. A regular schedule of calibration of field instrumentation must be followed. The field instrument calibration frequency is outlined in the SWQ/FPP QAPP. The calibration process is necessary to ensure that the instrument is working properly and within the range of acceptance as determined by the manufacturer. All instrumentation used in field activities must be calibrated prior to field use and as often as necessary thereafter, according to manufacturer instructions. All calibrations must be recorded in the meter's calibration log book.
5. In those instances where field equipment cannot be calibrated or is not functioning properly, the sampler will attempt to repair the affected equipment in the field. For field meters that are assigned to individuals, such as regional staff, that person is responsible for the maintenance and repair of their own equipment. For field meters that are not assigned to individuals and are available for general use, the SWQP designated sampler is responsible for maintenance and repair. The SWQP designated sampler is also responsible for ordering parts or service for all SWQP field equipment. The SWQP designated sampler is identified in the SWQ/FPP QAPP.
6. All SWQP field equipment will be examined for maintenance/repair recommendations and checked for proper operation by the SWQP designated sampler; this information is recorded in the calibration logbook. Any necessary maintenance will be performed immediately to assure instrumentation is in operating condition prior to the next use.

7.0 LABORATORY ANALYTICAL METHODS

Per Surface Water Quality Standards, tests or analytical procedures to determine conformity with surface water quality standards will be made in accordance with methods approved in 40 CFR Part 136. It is the responsibility of the project officer to specify and ensure that the laboratory uses approved analytical methods listed in 40 CFR Part 136 for all SWQP water quality samples.

8.0 SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

Appropriate sample containers, preservation techniques, and holding times for water quality samples are listed in 40 CFR Part 136. It is the responsibility of the project officer to ensure that the appropriate sample containers and preservation techniques are used during sample collection. It is the responsibility of the laboratory manager to ensure that the water quality sample is analyzed within the appropriate holding time. However, the project officer should verify that the holding time was met as a part of standard quality control practices (see SWQ/FPP QAPP).

In addition to sample container, preservation, and holding time information, 40 CFR Part 136 places additional requirements on some tests in the form of footnotes. These footnotes are important and are a required step in achieving meaningful results.

Figure 3 displays common test parameter suites used by the SWQP and FPP, and the appropriate container type, preservation requirement, and maximum holding time for the parameter with the shortest holding time in that bottle. For individual parameter holding times, refer to 40 CFR 146. As appropriate, footnotes are included and their action described at the bottom of the table.

Figure 3. Sample Parameter Suites and Information

Bottle		Size & Material	Preservative	Parameters	Holding Time[4]
A	DOH	1000 mL HDPE	Cool to 6°C	Alkalinity, total solids, TSS, volatile solids, TDS, BOD, CBOD, CO_3,Hardness, K, lab pH, lab conductivity, nitrate, chloride, fluoride, HCO_3, SO_4	48 hours
	Midco	1000 mL polypropylene			
B	DOH	1000 mL HDPE	2 mL H_2SO_4 pH <2 Cool to 6°C	Ammonia, Nitrite+Nitrate, TKN, Total P, COD	28 days
	Midco	500 mL polypropylene			
C	DOH	100 mL sterilized polystyrene	Na_2SO_3 if chlorinated Cool to 6°C	Fecal coliform, *E coli*, total coliform, enterococci, fecal PFG	6 hours[1]
	Midco	100 mL sterilized polystyrene			
D	DOH	100 mL polystyrene	Field filter 0.25 mL H_2SO_4 pH <2 Cool to 6°C	Dissolved P, dissolved inorganic nitrogen	28 days
	Midco	250 mL polypropylene			
Metals – Dissolved	DOH	100 mL polystyrene	Field filter 0.25 mL HNO_3 pH <2 Cool to 6°C	Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Cu, Hg, Pb, Mg, Mn, Ni, Se, Ag, Na, Ti, U, Vn, Zn, Fe, Mo, fluoride, K, Cl, silica	28 days
	Midco	250 mL polypropylene			
Metals – Total Recov	DOH	100 mL polystyrene	0.25 mL HNO_3 pH <2 Cool to 6°C	Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Cu, Hg, Pb, Mg, Mn, Ni, Se, Ag, Na, Ti, U, Vn, Zn, Fe, Mo	28 days
	Midco	250 mL polypropylene			
Oil & Grease	all	1000 mL amber glass	2 mL HCl pH <2 Cool to 6°C	Oil & Grease	28 days
R	all	1 gal cubitainer polypropylene	Cool to 6°C	Radium-226, radium-228	6 months
CN	DOH	250 mL polypropylene	NaOH pH>10 Cool to 6°C	Total cyanide, WAD cyanide *Mitigate interferences as described in 40 CFR136	48 hours[2]
	Midco	150 mL brown polypropylene			
H	all	1000 mL amber glass	Cool to 6°C	TPH – diesel, caffeine	14 days[3]
V	DOH	40 mL amber glass vial	Cool to 6°C	VOC, TOC, DOC, TPH -gasoline	14 days[3]
	Midco	250 mL amber glass			

If you need to sample for an analyte that is not on this list – contact the laboratory for bottle, preservation, and holding time information.

[1]The holding time for bottle C parameters is 6 hours for compliance samples. Noncompliance samples must be analyzed within 24 hours of collection. [2]The maximum holding time for total and WAD cyanide is 14 days as long as all interferences have been mitigated as described in 40 CFR 136. If interferences are unknown the maximum holding time is 6 hours. 40 CFR 136 may be viewed at http://www.ecfr.gov. [3]The holding time for bottles H and V is 14 days from the time of collection to laboratory extraction. [4]Maximum holding time is based on the analyte with the shortest hold time.

9.0 DECONTAMINATION OF SAMPLE CONTAINERS AND SAMPLING EQUIPMENT

The laboratory will provide new, clean containers or decontaminate previously used sample containers. Sample container decontamination by the laboratory involves detergent washing, rinsing with dilute chromic acid and final rinsing with laboratory-grade distilled water. Decontamination of sampling equipment (probes and instruments) will be accomplished through the use of distilled water by field personnel.

Field personnel do not need to triple rinse clean bottles supplied by the laboratory. However when a field sampler uses a field bottle to collect a sample then transports it to another container, the field bottle must be triple rinsed with the water being sampled prior to filling.

10.0 PROCEDURES FOR SURFACE WATER SAMPLING

A. Field Observations

Comments and observations regarding the weather and sampling site information must be recorded. It is important to record all field observations of conditions at the sampling sites that could influence the water quality of the collected sample. These observations are recorded on pre-printed project sheets or a field log book. Examples of observations recorded under COMMENTS could include: "cloudy, heavy recent rainfall, windy, cattle grazing near sampling site, dense emergent aquatic vegetation present at sampling site, etc."

In addition to comments, specific observations should be recorded on the field data sheets as follows:

Flow	Record in cubic feet per second (CFS)
Air Temperature	Record in degrees Celsius
Specific Conductance	Record in umho/cm
Dissolved Oxygen	Record in milligrams per Liter (mg/L)
Field pH	Record in standard units (su)
Water Temperature	Record in degrees Celsius
Secchi	Record in meters

B. Field Analyses

Calibrate all instruments prior to field use as described in Section 4.0. Record all field analysis data on pre-printed project sheets or field log notebook.

1. pH, DO, and specific conductance
   a. If water is static, provide stirring by gently and continuously agitating the probe.
   b. Allow sufficient time for the probe to stabilize.
   c. Record data in the field log book or on the pre-printed project sheet.

2. Temperature
   a. Air Temperature
      i. Always collect air temperature readings out of direct sunlight (record in °C).

   b. Water Temperature
      i. Water temperature may be measured using a bimetal thermometer or field meter. Do not use a mercury thermometer for field measurements due to the risk of breaking the thermometer and releasing mercury into the environment.
      ii. Place the thermometer into the stream and provide stirring or gentle agitation.
      iii. If you are unable to collect temperature directly from the waterbody, collect water in a field bottle and measure temperature as soon as possible.
      iv. Allow sufficient time for the thermometer to stabilize.
      v. Read the temperature (record in °C).

3. Total Depth and Width
   a. Record total depth at the maximum cross sectional depth (record in feet).
   b. Record width as an average distance between banks (record in feet).

4. Secchi Depth
   a. Lower the Secchi disk with calibrated rope into the waterbody from the shaded side of the boat.
   b. Drop the Secchi down until it is no longer visible.
   c. Bring the Secchi up until you can just barely make out the cross pattern.
   d. Record the depth of the Secchi Disk in meters.

Repeat the above procedure and average the two readings for the final Secchi depth reading.

5. Flow (SonTek)
   a. Calibrate and operate the meter using methods found in Section 4.0 of this manual.
   b. Record the stream discharge in cubic feet per second.

C. Sample Collection

The types of samples collected during sampling activities depend on the parameters that are necessary for the project. Sample parameters must be determined prior to conducting sampling activities. This will ensure that samples are adequately collected, handled, preserved, and that the sampling will address the project objectives. Figure 3 displays the parameter groupings, preservation requirements, and bottle type and size information used by the SWQP and FPP. Figures 5 and 6 are depictions of bottle information and preservation requirements. Figure 4 is a depiction of the disposable filters used to field filter water samples.

In the event a sample needs to be collected and the necessary information is not contained in Figure 3, refer to 40 CFR 136 or other appropriate chapter for approved methods and information.

When collecting a water sample from a river, stream, lake, or wetland, follow these basic principles:

1. Use the appropriate sample bottle as directed in Figure 3. Affix a waterproof label that contains the station identification, sample date, and bottle identification.
2. If sampling a wadeable waterbody, wade into the waterbody to collect the sample. It is only acceptable to use a bucket from a bridge to collect a sample if it is unsafe to wade into the waterbody. Do not endanger yourself by wading into a waterbody with unsafe conditions (ice jams, high flows, etc.). Wade into the thalweg or deepest part of the channel to collect the sample.
3. If sampling a non-wadeable waterbody, access the waterbody by boat, boat dock, or by wading along the shore if possible.
4. Face upstream or into the flow when collecting a sample. Remove the bottle lid and submerge the bottle beneath the surface of the water taking care not to disturb the bottom sediment. It is imperative that surface debris or bottom sediment do not enter the bottle. After filling

the bottle, secure the bottle lid. Preserve according to Figure 3 and place in a cooler of loose ice (even during winter months).

5. If the bottle is pre-preserved, do not overfill the bottle or rinse the bottle prior to filling.

6. If using a field bottle to collect a sample that will be field filtered (dissolved phosphorus or dissolved metals), make sure you triple rinse the field bottle with water from the waterbody being sampled before filling the bottle.

7. For samples that require field filtering, use 45 micron disposable filters. See Figure 4 below. Attach the tube from the hand vacuum pump to the vacuum port on the disposable filter. Remove the filter lid to the filter apparatus and pour an appropriate amount of water into the top of the filter apparatus. Pump the hand vacuum to create negative pressure in the filter apparatus. This will result in water being vacuumed through the 45 micron filter into the receiving bottle. Field filtering must be done on site within 15 minutes of sample collection.

8. Preserve the sample as directed in Figure 3 immediately after sample collection and chemical preservation. If you are unfamiliar with the buffer capacity of the water being sampled (feedlot waste, point source discharges, mining wastewater, etc., may be highly buffered), make sure you verify the pH with pH paper to make sure enough preservative has been added. Place all samples in a cooler with loose ice immediately.

Figure 4. Disposable Filter Apparatus

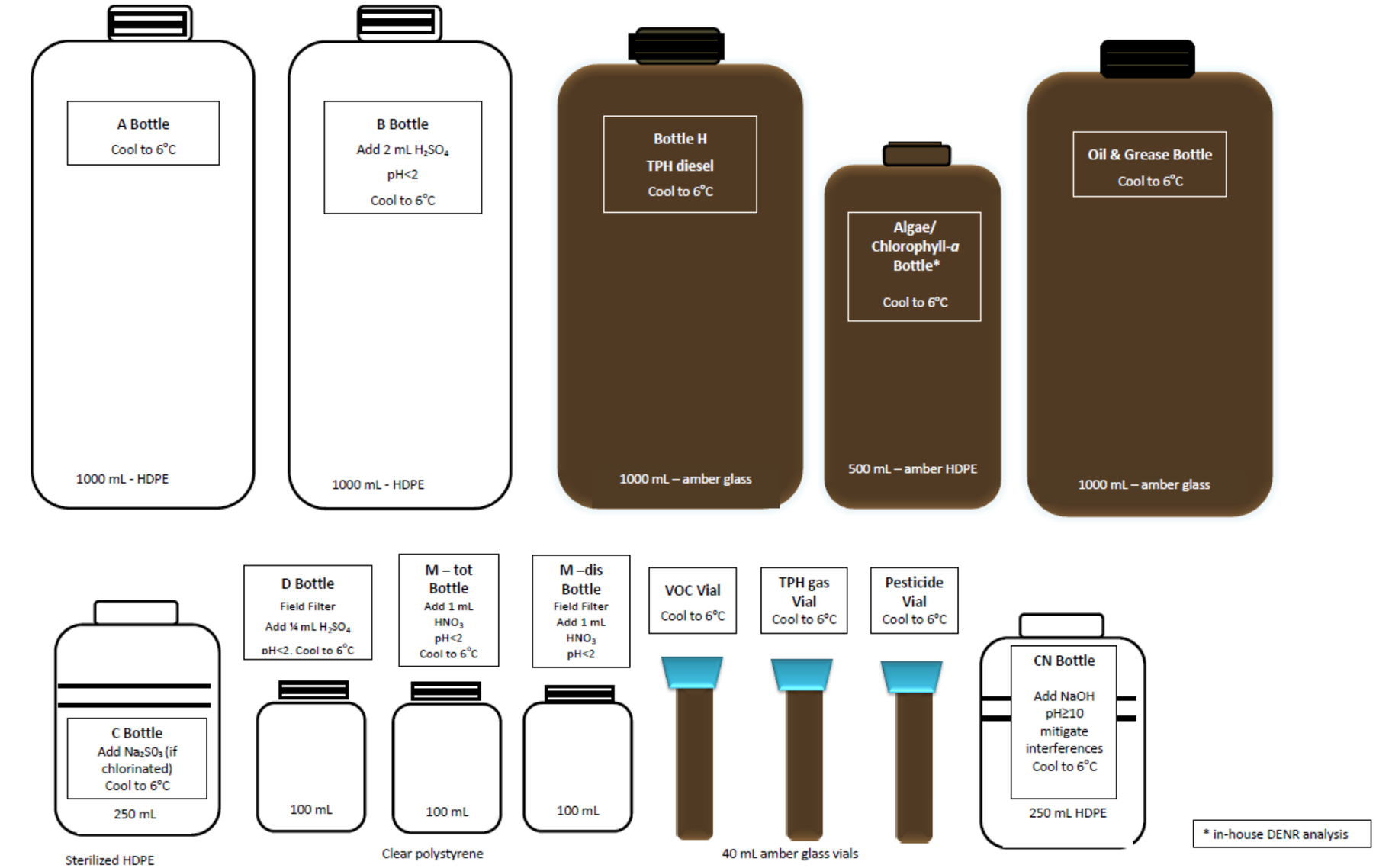

Figure 5. Sample Bottles for DOH laboratory.

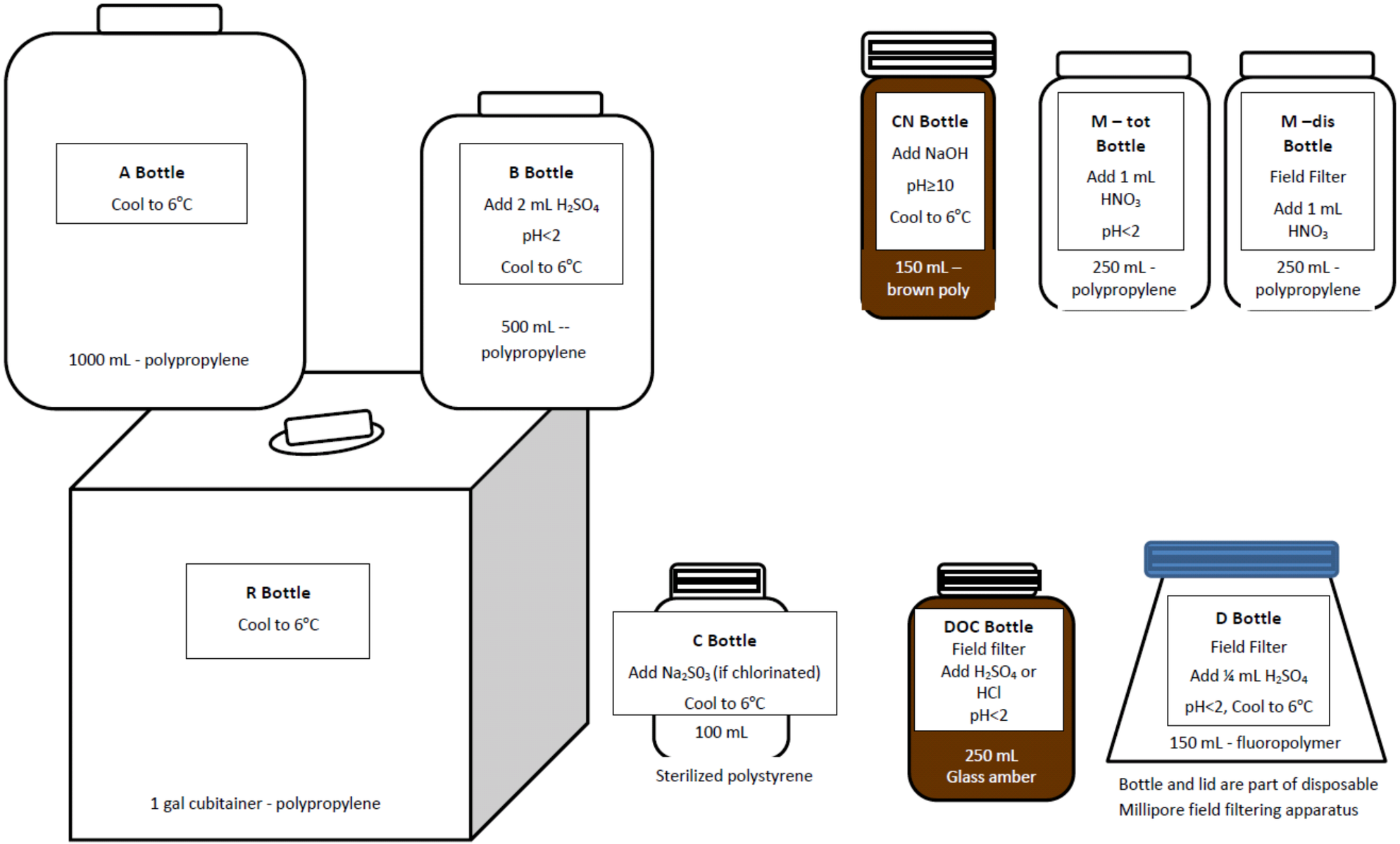

Figure 6. Sample Bottles for Midcontinent laboratory

1. Grab Sampling

Collecting a Sample

Be careful not to contaminate the inside of the lid or mouth of the bottle with your fingers or introduce other sources of contamination.

a. The "A" bottle –one liter bottle

i. Position the open end of the bottle towards the current flow and away from the hand of the collector.

ii. Grasp the bottle securely at the base with one hand and plunge the bottle down into the water to avoid introducing surface scum. The sampling depth should be 6 inches to 1 foot below the water surface, if possible.

iii. Tip the bottle slightly upward to allow air to escape and the bottle to fill.

iv. The "A" sample bottle should be filled to the neck of the bottle and capped immediately.

v. Place sample container in a cooler on loose ice (6°C), no preservative is required for bottle “A.”

vi. Mail or deliver the “A” bottle to the laboratory.

b. The "B" bottle –one liter bottle

Follow collection procedures for filling bottle “A” to fill bottle "B." Preserve the sample using the following procedure below.

i. After the sample has been collected, **preserve this sample with 2 mL of concentrated sulfuric acid (H_2SO_4)** to lower the pH of the sample below 2 standard units. As necessary, use pH paper to verify the pH has been lowered to <2.

ii. After H_2SO_4 has been added, the sample bottle is inverted several times to ensure mixing of the preservative throughout the sample.

iii. Place sample container in a cooler on loose ice (6°C) for shipment or delivery to the laboratory.

c. The “C” bottle -250 mL or 100 mL bacteriological sample

The 250 mL bottle is used when the sample requires more than one bacteriological analysis. If the sample only requires one bacteriological analysis, the 100 mL bottle may be used.

i. The "C" bottle **should not be rinsed** with sampling site water.

ii. Position the open end of the bottle towards the current flow and away from the hand of the collector.

iii. Grasp the bottle securely at the base with one hand and plunge the bottle down into the water to avoid introducing surface scum.

iv. The sampling depth should be 6 inches to 1 foot below the water surface if possible. If it is not possible, the sample depth may be less, but the sampler should avoid surface debris and bottom sediment.

v. Tip the bottle slightly upward to allow air to escape and the bottle to fill. On the initial plunge, the "C" sample bottle should be filled completely. Immediately after obtaining the sample, pour off any excess sample water from the container until the sample volume is 250 mL or 100 mL and cap.

vi. If the sample bottle is not filled at least to the designated mark on the sampling bottle, discard the sample and sample bottle and repeat the process with a new "C" bottle. DO NOT **re-immerse the original bottle to add more sample volume**.

vii. Place sample container in a cooler on ice (6° C); **no preservative is required for bottle "C."**

viii. For compliance samples, bacteriological samples need to arrive at the laboratory within 6 hours after collection and processed within 2 hours. For all other types of samples the holding time is increased to 24 hours.

d. The "CN" bottle – 250 mL or 150 mL

i. Fill the bottle as directed for the "A" bottle.

ii. To preserve, add 1 to 3 pellets of sodium hydroxide (NaOH). Swirl the bottle until the pellets have dissolved.

iii. Using pH paper, verify the pH is greater than 10 but less than 12. If the pH is less than 10, add more pellets. If the pH is greater than 12, discard the sample and collect a new sample.

iv. Cap the bottle and place in a cooler with loose ice.

v. Mail or deliver the "CN" bottle to the laboratory.

e. The "H" bottle – amber glass liter

i. Fill the bottle as directed for the "A" bottle.

ii. The "H" bottle does not require any preservative.

iii. Cap the bottle and place in a cooler on loose ice.

iv. Mail or deliver the "H" bottle to the laboratory.

v. If being used for caffeine sampling, the sampler must abstain from all forms of caffeine for 24 hours prior to

sampling. Ensure your hands are clean and make sure you do not contaminate the inside of the bottle or lid.

f. The "Oil and Grease" bottle – amber glass liter

i. Fill the bottle as directed for the "A" bottle.
ii. Preserve the bottle by adding 2 mL of 50% hydrochloric acid (HCl). Using pH paper, verify the resulting pH is less than 2.
iii. Place the acidified "Oil and Grease" bottle into a cooler on loose ice.
iv. Mail or deliver the "Oil and Grease" bottle to the laboratory.

g. The "V" or "pesticide" bottle – 40 mL amber glass vial or 250 mL amber glass

The 'V" bottle is a 40 mL amber glass vial. It comes from the laboratory pre-preserved with ascorbic acid and hydrochloric acid. Do not rinse the vial. Do not use a marker to write on the vial – use a pencil or preprinted labels and allow the ink to fully cure before opening the vial (due to risk of VOC contamination from marker). Do not allow contamination (even airborne) from organic compounds such as vehicle exhaust or cleaning compounds. Do not allow the bottle cap, bottle threads, or inside of bottle to be touched or contaminated. For VOC analysis, the laboratory will also send 2 trip blanks. Do not open these trip blanks. They must accompany the vials at all times, including return to the laboratory. For pesticide analysis, trip blanks will not be included.

i. Open the vial and completely submerge to fill. Underwater, tip the vial vertically so that it will completely fill and create a convex meniscus.
ii. Gently tap the vial to dislodge any air bubbles.
iii. Cap the vial and invert to mix preservative and visually verify there are no air bubbles.
iv. The glass vials can easily break. Pack the vials in the packing material supplied by the laboratory or wrap each vial in bubble wrap. Securely place the vials in a cooler of loose ice and ship or deliver the vials to the laboratory.

h. The "R" bottle – 1 gal cubitainer

The cubitainer must be expanded before filling. Do not blow into or place fingers inside the cubitainer. To expand,

partially unscrew the lid to allow air to enter the cubitainer and gently pull at the seams.

i. Fill the cubitainer as directed for the "A" bottle.
ii. If you are unable to submerge the cubitainer without disturbing bottom sediment, use the "field" bottle to collect the sample and pour the water into the cubitainer.
iii. The "R" bottle is the only sample bottle that will be filtered and preserved by the laboratory. This is due to the large volume of filtered water that is required and the general difficulty in filtering that amount of water from "R" bottle sites.
iv. Cap the "R" bottle and place in a cooler on loose ice.
v. Mail or deliver the "R" bottle to the laboratory.

i. The "Metals-Tot Recov bottle" – 100 mL or 250 mL

i. Fill the bottle as directed for the "A" bottle.
ii. Preserve the bottle by adding 0.25 to 1 mL of concentrated Nitric Acid (HNO_3) based on bottle size. As needed, use pH paper to verify the resulting pH is less than 2.
iii. Place the acidified "Metals-Tot Recov" bottle into a cooler on loose ice.
iv. Mail or deliver the bottle to the laboratory.

j. Collection of the "field bottle" - one liter bottle

If collecting dissolved phosphorus and/or dissolved metals water to be filtered, the sample can be collected in a one-liter polypropylene "field" bottle. Thoroughly triple rinse the field bottle with water from the waterbody being sampled then follow steps 1(a)(i)-(iv).

k. The "D" bottle -100 mL plastic bottle

Water to be filtered for this sample comes from the field bottle. Procedure for field filtration and preservation of the total dissolved phosphorus sample are described below.

i. Assemble the disposable filters by attaching the filter unit to the receiving bottle. Attach the hand pump hose to the vacuum port.
ii. Pour approximately 120-150 mL of water from the field bottle into the filter unit and cap with the filter lid.
iii. Use the hand pump to create a vacuum in the filter unit. This will cause water to be filtered through the 45

micron filter and accumulate in the lower receiving bottle.

iv. After 100 mL has been filtered, unscrew the receiving bottle. This water may be transferred to the 100 mL "D" bottle (if going to the DOH lab) or it may remain in the receiving bottle (if going to Energy).

v. Add 0.25 mL of concentrated H_2SO_4. As needed, verify the pH with pH paper to ensure the pH is <2.

vi. Place the acidified bottle into a cooler on loose ice.

vii. Mail or deliver the bottle to the laboratory.

l. **The "Metals- Dissolved" bottle -100 mL or 250 mL plastic bottle**

Follow collection procedures for filtering and filling bottle "D" to fill bottle "F." Preserve the sample using the following procedure below.

i. 0.25 mL of concentrated Nitric Acid (HNO_3). As needed, use pH paper to verify the resulting pH is less than 2.

ii. Place the acidified "Metals-Dissolved" bottle into a cooler on loose ice. Mail or deliver the bottle to the laboratory.

m. **"Caffeine" bottle – 1000 mL amber glass with Teflon lid**

Caffeine sampling is conducted to provide information which may correlate contamination with human waste from septic leachate. **NOTE: Sampler must abstain from ingesting caffeine for 24 hours prior to sampling to minimize contamination due to low caffeine detection limits.**

i. Fill the bottle as directed for the "A" bottle taking care not to touch the inside of the lid, the threads of the bottle, or the inside of the bottle.

ii. If residual chlorine is present, add 80 mg of sodium thiosulfate per liter of water collected.

iii. Cap the sample and store in a cooler on loose ice.

iv. If the sample cannot be analyzed by the laboratory within 48 hours, freeze the sample to increase holding time to 7 days.

v. Mail or deliver the bottle to the laboratory.

n. **"Algae/Chlorophyll *a*" bottle – 500 mL amber plastic**

Algae or chlorophyll *a* are samples that may be collected during a complaint or fish kill to provide information on the

water quality. These samples may be analyzed internally or contracted with an outside laboratory.

i. Fill the bottle as directed for the "A" bottle.
ii. Cap the sample and store in a cooler on loose ice.
iii. Ship or deliver the sample to the laboratory or SDDENR personnel.

2. Composite Sampling

Collecting a Sample

The sampling plan or project manager will determine which samples are to be composite samples. Unless specified, most samples will be grab samples.

a. Triple rinse a plastic graduated cylinder with sample site water.
b. Collect a sample in a rinsed Van Dorn sampler or other sampling device.
c. Calculate the amount of water needed from each sub-sample. Divide the size of your container (milliliters), by the number of sampling sites to be composited.

   Example: Compositing three sites and placing them in the "A" bottle (1,000 mL).

   1000 mL/ 3 = 333 mL

d. Pour the previously calculated amount (i.e. 333 mL) from one sub-sample into the graduated cylinder.
e. Pour the water from the graduated cylinder into each sample bottle.
f. Repeat procedures "a" through "e" on the remaining sub-sample sites.
g. Preserve each bottle following the procedures from the Grab sampling section.

11.0 PROCEDURES FOR LAKE SEDIMENT SAMPLING

Lake sediment is sampled and analyzed to provide information and test for contaminants that may affect the benthic community and water quality. When selecting sediment sampling sites, consider known flow patterns through the lake, locations of tributary inputs, and any other sources that may affect the content and distribution of the sediment contaminants.

A. Sampling Equipment

1. Boat or Ice Auger and Chipping Bar
   a. Life jackets (use in boat or on ice)
   b. Other required safety equipment
   c. Waders may be necessary
2. Ponar-Petite sediment sampler with rope (see Figure 7)
3. One (1) 1000 mL glass jar (with Teflon-lined lids) and one (1) 250 mL polypropylene wide-mouth bottle per sampling site
4. One (1) 5 gallon bucket with stainless steel filter screen mesh on bottom
5. One (1) 5 gallon bucket
6. Stainless steel 2 gallon bucket and stainless steel scoop
7. 5 gallon jug waste container with lid
8. 10% nitric acid solution
9. 10% acetone solution

B. Equipment Decontamination Procedure

1. The sampling equipment must be thoroughly cleaned between sample sites. If composite samples are collected, the equipment must be thoroughly cleaned between sampling groups (sets of composites).
2. Rinse the equipment with lake water, removing all sediment and debris.
3. Rinse the equipment with a 10% nitric acid solution. Collect the waste solution and pour into the 5 gallon jug waste container.
4. Rinse the equipment with lake water. Collect the lake water rinsate and pour into the 5 gallon jug waste container.
5. Rinse the equipment with 10% acetone solution. Collect the waste solution and pour into the 5 gallon jug waste container.
6. Rinse the equipment with lake water. Collect the lake water rinsate and pour into the 5 gallon jug waste container.

C. Sampling Site Procedures

1. Grab Sampling

a. Measure the in-lake depth with a depth finder, Secchi disk, or weight, and record the measurement in the field notebook or project sheet.
b. Label one (1) wide-mouth glass jar and one (1) polypropylene bottle (sediment samples) with the station identification and date.
c. Collect sediment samples with the Ponar-petite dredge sampler. Set the pinch-pin tripping mechanism on the dredge. Slowly lower the dredge through the water column. When you near the bottom, let the dredge drop. Upon impact with the bottom, the jaws will clamp shut and grab a sediment sample. Slowly lift the dredge to the surface of the water. Tilt slightly to pour off excess water.
d. Place the sediment into the pre-cleaned 5 gallon bucket (with mesh bottom). Excess water should drain through the mesh.
e. Mix the sediment in the pre-cleaned bucket with the stainless steel scoop to homogenize the sample and drain excess water.
f. Completely fill one (1) 1000 mL wide-mouth glass jar and one (1) 250 mL polypropylene bottle with sediment per sampling site. Make sure that containers are completely filled with sediment and that air bubbles are not trapped in the container. Carefully rinse the glass jar threads and the cap with sample water to clean the threads for a better seal. Securely cap with a Teflon-lined lid.
g. Place sediment samples into the sampling cooler on loose ice.

2. Composite Sediment Sampling

Composite sediment sampling is similar to grab sampling except sediment samples are collected at multiple locations in the lake and composited.

a. Label one (1) 1000 mL wide-mouth glass jar and one (1) 250 mL polypropylene bottle (sediment samples) with the station identification (for all composited sites) and the date.
b. At each sub-sampling site, measure in-lake depth with a depth finder, Secchi disk, or weight, and record each depth measurement in the field book or project sheet.
c. Collect a sediment sub-sample with a Ponar-petite as described above in C.1.c. Place the sediment collected from sampler grabs into the pre-cleaned 5 gallon bucket (with the mesh bottom).
d. With the stainless steel scoop, place approximately 3 large scoops of sediment in the stainless steel bucket.

e. Repeat procedures ‘a’ through ‘d’ at the remaining sub-sampling sites.
f. Mix the sediment composite sample in the stainless steel bucket with the stainless steel scoop to homogenize the sample.
g. Completely fill one (1) 1000 mL wide-mouth glass jar and one (1) 250 mL polypropylene bottle with sediment per sampling site. Make sure that the container is completely filled with sediment and that air bubbles are not trapped in the container. Carefully rinse the glass jar threads and the cap with sample water to clean the threads for a better seal. Securely cap with a Teflon-lined lid.
h. Place sediment samples into the sampling cooler on loose ice.

Figure 7. Ponar petite.

12.0 REFERENCES

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