

Field Measurements and Sample Collection (Water Quality)

1. Scope and Application

- a. This method outlines procedures for field measurements and sample collection from surface and saline waters.
- b. Procedures include operations from land and small watercraft.
- c. It is not practical to detail the use and operation of all of the instruments that may be used for field measurements or automated sampling equipment. Refer to other referenced SOPs.

2. Summary of Method

- a. Collect water samples for analysis within FDEP sampling guidelines.

3. Definitions

- a. Accuracy – Measure of closeness to an accepted value. Contrast with Precision.
- b. Grab Sample – A discrete sample taken at a specified location and time.
- c. Calibration - is the process of evaluating and adjusting the precision and accuracy of measurement equipment.
- d. Composite Sample – A sample representing an interval of time, location or both (this characteristic will be indicated). A composite sample may be formed manually from multiple grab samples or with aid of special collecting equipment.
- e. Duplicate – An independent sample or measurement of the matrix. Used to assess precision.
- f. Field Blank – A sample of analyte-free water created in the field to detect sample contamination from collecting equipment and/or sample transportation. There are several sub-types. (See FDEP SOP FQ 1000).
- g. Field Duplicate Sample (FD or Field Dup) – Duplicate samples created in the field.
- h. *In-situ* – A Latin phrase meaning “on site” or “in position. Often used to differentiate parameters measured in the field from the same parameters measured laboratory.
- i. Matrix – The substrate of a sample or object of a measurement other than the parameter of interest. Ambient surface water matrices are defined by the PNRD lab to be:

- i. Freshwater – ambient surface water with a measured Specific Conductance of < 5000 umhos/cm at time of collection.
 - ii. Saltwater – ambient surface sample with a measured Specific Conductance of >= 5000 umhos/cm at time of collection.
 - iii. QC Matrix-Water – an analyte-free, DI water sample.
- j. Parameter – A measurable characteristic.
- k. Photosynthetic Active Radiation (PAR) – Refers to light with wavelengths from 400 nm (blue) to 700 nm (red) most useful to photosynthesis. Roughly equal to the range of colors visible to the human eye. PAR measurements.
- l. Precision – Degree of mutual agreement between a set of the same measurements. Contrast with Accuracy.
- m. Replicate – Often used as a synonym for Duplicate, the usual distinction in usage is that a Replicate repeats an effect or result, while Duplicate refers to the creation of a copy.
- n. Safety Data Sheet (SDS) (Formerly Material Safety Data Sheet (MSDS)) – Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions. The MCPNRD laboratory uses the vendor's online SDS archives to access the latest version of this information.

4. Interferences

- a. There are many factors that will affect the collection of a representative sample. Comments on factors that may potentially affect sampling results should be recorded in field notes.
- b. *In-situ* measurement equipment not calibrated properly.
 - i. Unless specified otherwise in a method SOP, *In-situ* measurement equipment calibration follows the instructions in the manufacture's manual.
 - ii. Unless specified otherwise in a method SOP, verification of equipment with a factory or non-adjustable calibration follows the instructions in the manufacture's manual.

5. Safety

- a. Use appropriate Personal Protective Equipment (PPE) when working with chemicals. Chemical resistant gloves and eye protection are provided for field sampling.
- b. Chemicals may be encountered as preservatives, calibrants, cleaning or probe filling solutions. Some chemicals have the potential to be highly toxic or hazardous, consult

the current Safety Data Sheet (SDS) from the vendor's online SDS archives for additional details.

6. Sampling Procedure

- a.** For most analytical parameters, rinse the sample container three times with the water being collected (except for bacteriological analysis or if preservative is present in the container) before filling. For organic parameters fill container full and for most other parameters leave aeration or mixing space of one to two percent.
- b.** Make a record of every sample collected and identify every container before leaving the sampling location. Samples should never be left unattended. Record sufficient information to provide positive sample ID later - container label should contain sample ID, the date and time, collector, location and any other pertinent information. Fill out every blank space on the field sheet and/or chain-of-custody form with information or the letters NA for not applicable.
- c.** The container labels and the field sheet or chain-of-custody form must have space for initials of the party assuming custody and the date and time. Make sure sampling points are described in detail by maps, stakes, buoys or landmarks in a manner that will permit their identification by other persons without reliance on memory or personal guidance. Particularly when sample results are expected to be involved in litigation, chain-of-custody must trace the history of a sample from collection to reporting.

d. SAFETY CONSIDERATIONS

- i.** Sample constituents are unknown and could be toxic, take adequate precautions. Toxic substances can enter through the skin or through the lungs. Precautions may in some cases be only wearing gloves or may include coveralls, aprons or other protective apparel. Always wear eye protection. If vapors are present, sample only in well-ventilated areas and use a respirator or self-contained breathing apparatus. Always wash hands thoroughly before handling food or drinks. Never allow food or smoking around the sampling area or the samples. Label samples suspected to be hazardous.

e. SAMPLE INTEGRITY

- i.** Take precautions to avoid cross-contamination. Segregate samples by type, preservative or site or use a secondary container like plastic bags.
- ii.** Periodically verify sampling container integrity during collection, storage and transport.

f. QUANTITY OF SAMPLE COLLECTED

- i. For organic parameters fill container full and for most other parameters leave aeration or mixing space of one to two percent.
- ii. Collect separate samples for physical, chemical, bacteriological, microscopic, metals and organics analyses.

g. SAMPLE DELIVERY TO THE LAB

- i. Deliver samples to the lab as soon as practicable after collection. If time is less than 30 minutes from collection, samples do not need to be chilled. If more than 30 minutes between time of collection and delivery to the lab, samples must be kept on ice at approximately 4 degrees C.
- ii. Person in the lab receiving the samples will check that proper containers, amount of sample and (if needed) preservative have been used for the required parameters and that all necessary information has been provided by the collector. Any deviations from requirements will be noted on the field sheet or chain-of-custody form (sometimes both will be required). Samples will be logged into the lab sample receiving book, assigned a number and stored properly until analyses can be completed.
- iii. If samples are to be shipped, chain-of-custody forms must be placed in a plastic bag and shipped with the samples (never ship separately).
- iv. The lab will take care of shipping all samples. Lids of sample containers should be sealed to prevent tampering and leakage and samples carefully packed to maintain temperature required.

h. SAMPLE CONTAINERS

- i. See Sample Receiving and Handling Protocol section of individual method SOPs (latest versions) for specific instructions.
- ii. MCNRD Sample Handling SOP (latest version) has general handling and acceptance criteria (See Standard Methods Table 1060: I Summary of Special Sampling, Handling Requirements from Standard Methods 23rd Edition).

Table 1. Field Equipment List.

Item

Ice and insulated cooler
Sample bottles
Multi probe meter and data logger
Carboy with DI water for equipment or field blank
Water sampler (appropriate for sample needed)
Field data sheet, chain-of-custody form (as required)
Pen and waterproof marker
Maps
Disposable gloves
Cell phone
Ice chests and ice.
2-liter plastic sample A bottles for TSS (pre-labeled for each station with indelible ink).
1-gal plastic sample H bottles for TSS replicate.
500 ml amber Nalgene sample C bottles for chlorophyll (pre-labeled for each station with indelible ink).
236 ml plastic sample B bottles for nutrients acidified for preservation (pre-labeled for each station with indelible ink).
120 ml plastic sample F and G bottles snap cap vial sterile for bacti x 2 (pre-labeled for each station with indelible ink).
Bottles for Blank.
Carboy with DI water for equipment or field blank
Chlorine test strips.
pH test strips
Field data sheets & pens.
Navigational maps.
Field Meter with iPad and Bluetooth for Date, Time, Temp., pH, Cond., D.O., % Sat D.O., Salinity, T.D.S., and Depth (Pre and Post Calibrated).
Li-Cor light meter with data logger.
Hach Flow meter with data logger
Secchi Disk.
G.P.S.
Cell Phone.
Water collection device. (appropriate for type of sample):
 Buckets.
 VanDorn Bottle or Type A horizontal samplers
 Dipping rod samplers
 Modified Sludge Sampler
Boat Equipment:
 Drain plugs.
 Marine batteries.
 Anchor with 40-foot rope.

Table 1. Field Equipment list (Cont'd)

Item

Toolbox.

Safety Equipment:

First aid Kit.

Life vests (one per person).

Toss-able life preserver.

Horns (manual, compressed gas).

Distress flag.

Fire extinguisher (verify that it's fully charged).

Flares.

Dive Flag.

Table 2. Field QC Samples (See FDEP SOP FQ 1000)

| Type | Purpose | Recommended Frequency |
|---------------------------|-----------------------------------------------------------------------|------------------------------|
| Temperature Blank | Assure samples are chilled. | Per program Requirements |
| Replicates | Measure variability in results from field locations or sampling event | 10% or per method SOP |
| Splits | Measure variability in results between laboratories. | Per program requirements |
| Field Blanks | Evaluates effects of sample collection containers | 1 per sampling event |
| Equipment Blanks | Evaluates effects of intermediate sampling device cleaning | 1 per each sampling event |
| Trip blanks | Measures effects from sample transport and containers | 1 per sample shipment |
| Field Performance Samples | Independent check of accuracy measuring system evaluation | Per program requirements |

i. FIELD SAMPLING GENERAL PROCEDURES

- i.** Follow all Coast Guard safe-boating procedures when operating watercraft.
- ii.** Handle any questionable samples with care to avoid cross-contamination and exposure to hazardous materials.
- iii.** Do not allow food or smoking near samples or sampling locations.
- iv.** Always wash hands thoroughly before handling food. If applicable.
- v.** Tide charts should be reviewed to schedule sampling events in estuarine waters at incoming or high tide during the sampling window of 10:00 a.m. - 3:00 p.m. local time.

j. EQUIPMENT CLEANING, PREPARATION, TRANSPORT AND STORAGE

i. Plastic Reusable Sample Botte Preparation:

- 1.** Clean (FDEP SOP FC1132) by rinsing bottles with HOT tap water. Soak bottles in a hot, sudsy water solution (Liqui-Nox or equivalent). Rinse with HOT tap water. Rinse with D.I. water. Allow bottles to air dry and securely cap for storage and transport.
- 2.** Do not reuse bottles if they do not appear clean and the closure functional; or if they have been used to collect waste waters, pesticides, toxic or noxious compounds. Acid washing or other special preparation may be necessary for specific analyses, see FDEP FC 1000.

ii. Plastic Sample Collection Equipment Preparation:

- 1.** Clean (FDEP SOP FC1132) by rinsing equipment with HOT tap water. Soak equipment in a hot, sudsy water solution (Liqui-Nox or equivalent). Rinse with HOT tap water. Rinse with D.I. water. Allow to air dry before wrapping for transport. An acid wash step or other special preparation may be necessary for specific analyses, see FDEP SOP FC 1000.
- 2.** Store and transport equipment according to the manufacturer's recommendation or wrap equipment in aluminum foil, untreated butcher paper or untreated plastic bags.

- iii.** Sample Transport Cooler Preparation: (FDEP SOP FC1132) Wash the exterior and interior of the cooler with a hot, sudsy water solution (Liqui-Nox or equivalent). Rinse with tap water and air dry.

iv. Plastic Analyte-Free Water Carboy Preparation:

1. Clean (FDEP SOP FC1190) by washing the exterior with a hot, sudsy water solution (Liqui-Nox or equivalent). Rinse with D.I. water. Rinse the interior with D.I. water. Invert and allow to drain and air dry.
2. The interiors of new, plastic carboys should be thoroughly cleaned before first use (FDEP SOP FC 1132). Solvent rinses, acid washing, or other special preparation may be necessary for specific analyses.
3. Store and transport the carboy securely covered or capped.

v. Field Meter Preparation

1. Wipe down equipment body, probes, and cables with lab-grade detergent solution (FDEP SOP FC 1210): Check manufacturer's instructions for recommendations and/or restrictions on cleaning. Rinse thoroughly with tap water. Rinse thoroughly with analyte-free water.
2. Store and transport instruments according to the manufacturer's recommendation or wrap equipment in aluminum foil, untreated butcher paper or untreated plastic bags.
3. Field meters must be calibrated before a sampling event, and the calibration checked after the sampling event. Refer to manufacturer's manuals for calibration procedures.

k. BOAT PICK-UP & PREPARATION PROCEDURE

- i. Truck, Boat & Trailer: Truck rated to pull trailer and boat.,
- ii. 21-foot Privateer with 175 hp. Mercury outboard: Stored at the lab. Gas: 50-gal tank. Use premium gas only. Trailer: 2-5/16inch ball hitch requirement, standard surge brakes & electrical connection.
- iii. 16-foot Carolina Skiff with a 40 hp. Mercury outboard: Stored at the lab. Gas: 15 gal. tank. Use premium gas only. Trailer: 2-inch ball hitch requirement, brakes & electrical connection.
- iv. Pick up boat key, make sure trailer hitch & electrical plug are in vehicle. Set emergency brake before placing vehicle in park, secure trailer safety chains to vehicle (they should cross under the hitch), Check condition of tires and lights, and fill fuel tank. Check electrical system for power (batteries require periodic recharging). Load/check boat and sampling equipment, keys, safety equipment, charts, connect GPS unit & test.

- v. Before leaving PNRD. Let supervisor know your Float-Plan.

I. BOAT LAUNCHING & LANDING PROCEDURE

- i. Boat Launching: Be courteous and considerate of other boaters exiting/entering ramp. Be efficient ramps are often busy. Remember, you have County PNRD stickers on everything. Install drain plugs. Disconnect winch cable. Tie the launch rope to bow cleat and leave plenty of slack. Launch boat by "backing & braking" method. (Boat will slide/float off the trailer before rear truck wheels reach the water.) Secure launch rope/boat to ramp. Keep boat tied up until engine has started and warmed up.
- ii. Landing: Vehicle and boat drivers should check direction and strength of any cross currents. Land boat very slowly (idle speed and/or neutral when first contacting trailer.) Raise motor tilt and set lock bar prior to pulling trailer out of water. Double check boat position and winch lock after pulling trailer out of the water. Pull drain plugs.

m. BOAT OPERATION

- i. Starting: Squeeze primer bulb several times when first starting engine. Throttle lever must be in neutral position or out of gear for engine to start. Follow boating rules and guidelines as marked on the water and taught in the Safe Boater Training Course. Always be courteous!!
- ii. Review tide and navigational charts and prepare a navigational course prior to getting under way. A route should utilize established channels and/or deep waters to minimize possible impact to sea grasses and other environmentally sensitive areas. Use GPS in determining direction towards, and the station location, but must be used in conjunction with navigation charts. Follow G.P.S. manual for operating instructions.

n. BOAT/TRUCK STATION SAMPLING PROCEDURE

- i. Field Meter Parameters:
 - 1. Attach cable to field instrument and turn on iPad and Bluetooth. Remove protective cap and attach probe guard to probe. Lower the probe into the water to about one-meter depth, or mid depth if less than a meter and record readings for pH, Salinity, Temperature, D.O., and Depth. Record time on the field sheet.

2. Lower Secchi disk into the water and record the Secchi depth according to the meter marks on the rope.
3. ESTUARINE SAMPLING ONLY: Assemble the LiCor sensors to the data loggers correctly labeled connector. Lower the PVC mounted sensors into the water with upper sensor at 0.5 meter and let readings stabilize record three readings one minute apart (lower sensor reading should always be lower than the upper sensor reading).
4. STREAM SAMPLING ONLY: Connect flow meter data logger to sensor. Lower sensor below water surface (away from any objects that may disturb the stream flow. Turn on the flow meter data logger and scroll to real time tab. Click OK. Let readings stabilize and record on field sheet. Collect flow data whenever a sample is collected.
5. Retrieve the probes, replace proper protective caps and covers, and store in for travel to next site.

ii. Sample Collection:

1. Prepare intermediate sample collection equipment (VanDorn bottle, bucket, dipper, sludge sampler, etc..).
2. Collect pre-cleaned equipment blanks if required by analyte or program (FDEP SOP FQ 1000).
3. Field clean intermediate sample collection equipment (FDEP SOP FC 1132) before and after each use.

NOTE: Field equipment rinsing on-site may use ambient water or analyte-free water.

4. Sample below the surface without disturbing the bottom. Do not sample from a depth of less than 0.3 meters (Record no sample, <0.3m depth on field sheet).
5. Remove the lid from the Sample container and rinse once with ambient water then fill (Do not rinse if sample container has a preservative in it)..
6. Fill all appropriate sample containers leaving appropriate head space (Shake bottles pre-filled with preservative acid). Sample must be preserved within 15 minutes of collection.
7. Samples may be preserved with acid, ice, or both. Check the pH of at each sample with litmus paper (Do not put litmus directly into the sample container) and record pH on field sheet. Store samples on ice if required.

8. FRESHWATER SAMPLES ONLY: Bacti samples must be collected in a sterile container without rinsing (Check sample for chlorine with test strip). Do not collect if chlorine is detected.
9. Collect at least one duplicate field sample per sampling event. Field duplicate sample frequency are per test Method SOP, and the requirements may vary.
10. If you cannot get a sample from fixed station you can relocate up to 100ft (30m) away. Preferably upstream. Do not sample discontinuous streams (Record no sample, discontinuous stream on field sheet).

iii. Field Meter Parameters

1. Attach cable from field instrument and turn on logger. Remove protective cap and attach probe guard. Lower the probe into the body of water or a bucket filled with water. Then record readings for pH, Salinity, Temperature, D.O., and Depth. Record time on the field sheet. Retrieve the probes, replace proper protective caps and covers, and store in for travel to next site.

o. CLEAN-UP AND STORAGE

1. All equipment must be flushed with fresh water at end of the sampling event.
2. All Field Meters must be taken back to lab for post- calibration and clean-up. For clean-up, simply wipe with damp cloth to remove salt and dirt and rinse probes with fresh water. Fill cup with pH 7 buffer and store meter.
3. Ice Chest should be rinsed (wash with lab detergent if necessary) and air dried before closing lid to avoid mildew.
4. Samples should be taken to the lab where custody log will be filled out, and the samples stored and preserved if necessary.
5. Boat Sampling
 - a. G.P.S. should be cleaned with a damp cloth and stored in boat room cabinet.
 - b. Flush boat engine with fresh water. When returning the boat to the lab, fill the gas tank.

p. MANTA PROBE CALIBRATION

1. Follow manufacturer instructions. Enter name, date, time, meter I.D. and standard lot numbers on meter calibration sheet. Connect the probe to the computer.
2. Click on Manta Icon. Verify the temperature with NIST traceable thermometer. Log the reading on calibration sheet. Click on the calibrate icon. Click on Date/ time icon, then click on sync, and click ok. Click on the calibrate icon. Click on depth meters button. Remove the cup and point the probes downward. Enter the standard value. Wait for the reading to stabilize. Click the yes button. Log reading on calibration sheet. Replace cup. Rinse with primary conductivity standard that will bracket the anticipated sample range, three times. Fill with the standard.
3. Click on calibrate button. Click on Specific Conductivity. $\mu\text{S}/\text{cm}$ button, and enter the standard value, then let the reading stabilize, and enter yes. Log the reading on calibration sheet. Rinse with second source conductivity standard that will fall within the anticipated bracketed range, three times. Fill with the standard. Log the reading on calibration sheet.
4. Rinse with a primary 7.00 pH standard three times. Fill with the standard. Click on the calibrate button. Click on pH and enter the standard value, let the reading stabilize, and enter yes. Rinse with 10.00 pH standard three times. Fill with the standard. Enter the standard value, let the reading stabilize then enter yes to accept, and no to 3-point calibration. Log the reading on the calibration sheet. Rinse with a second source 7.00 pH standard three times. Fill with the standard. Log the reading on the calibration sheet.
5. Rinse with 100% D.O. saturated water three times. Fill with water. Click on calibrate. Click on HDO sat, set BP to current BP, one-point calibration, let stabilize, enter, done. Log reading on calibration sheet.

q. LI-COR DATA LOGGER SET-UP

1. Turn on logger. Connect logger to computer. Open field meter folder, open Li-cor folder, open the setup file. Click connect.
2. Set up each channel with the correct info. (Name, Light, Correction factor averaging time). Save settings. Close file. Connect data logger to sensors.

3. Confirm operation (readings should be similar in ambient light).

r. MANTA DATA LOGGER SET-UP

1. Attach to Bluetooth, Attach to meter. Turn on iPad. Open Manta App. Name new file.
2. Click snapshot to capture reading.

s. DOWNLOADING MANTA DATA LOGGER

1. Click on export file. Choose the file to download. Email file to yourself.
2. Check email was delivered
3. Plug logger and Bluetooth into charger.

t. DOWNLOADING LI-COR DATA LOGGER

1. Attach data logger to computer with nine pin connector cable. Click on Li-Cor icon to open software. Open the csv file with your sample date with excel.
2. Save the file and close software and disconnect the data logger.

Signatory Page

MCNRD Environmental Laboratory

The accompanying document has been read and understood by the undersigned.

| Name (Printed) | Date | Signature |
|-------------------|------------|-----------------|
| Jeffery P. Nelson | 12/14/2022 | JP NELSON |
| Kerry Harkinson | 12/15/2022 | Kerry Harkinson |
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