

Southeast Coast Network

National Park Service
Inventory and Monitoring Division



Standard Operating Procedure 1.2.11

Fixed-Station Water Sample Collection and Processing— Version 2.0

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Summary

The following standard operating procedure (SOP) outlines the procedure for collecting water samples at fixed-station sites for the laboratory analysis of total dissolved nitrogen (TDN), total dissolved phosphorus (TDP), ammonium (NH_4^+), nitrites (NO_2), nitrates (NO_3), dissolved inorganic phosphorus (DIP or PO_4) and chlorophyll *a*. The purpose of this SOP is to ensure consistent and comparable year to year data collection. The techniques and procedures outlined in this SOP are based on the EPA National Coastal Condition Assessment Field Manual (2014); much of the information presented here is modified only slightly from EPA instructions. Sonde deployment and retrievals are usually done in conjunction with water sample collection and follow procedures set forth in part of SOP 1.2.09 *Fixed-station, water-quality data sonde maintenance, calibration, deployment, and retrieval*—Version 3.0 (McCay et al. 2022).

Revision Log

Revision Date	Author	Changes Made	Reason for Change	New Version #
1/17/2013		Citation updated	Protocol publication	1.0
3/25/2021	D. L. McCay	Citation and procedures updated. Figure was added to clarify workflow. New data collection form figures. List of tide gages added. Added text to clarify QA/QC section, sample windows and hold times. Example water sample labels were updated. Example QA/QC schedule added.	New EPA field manual available. Additional information regarding hold times and sample handling were deemed necessary.	2.0

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Fixed-Station Activities and Workflow Summary

An overview of fixed-station activities and workflow are presented in Figure 1. SOPs are presented left to right in the general order of operations for each activity. This SOP (1.2.11) pertains to the Data Collection activity.

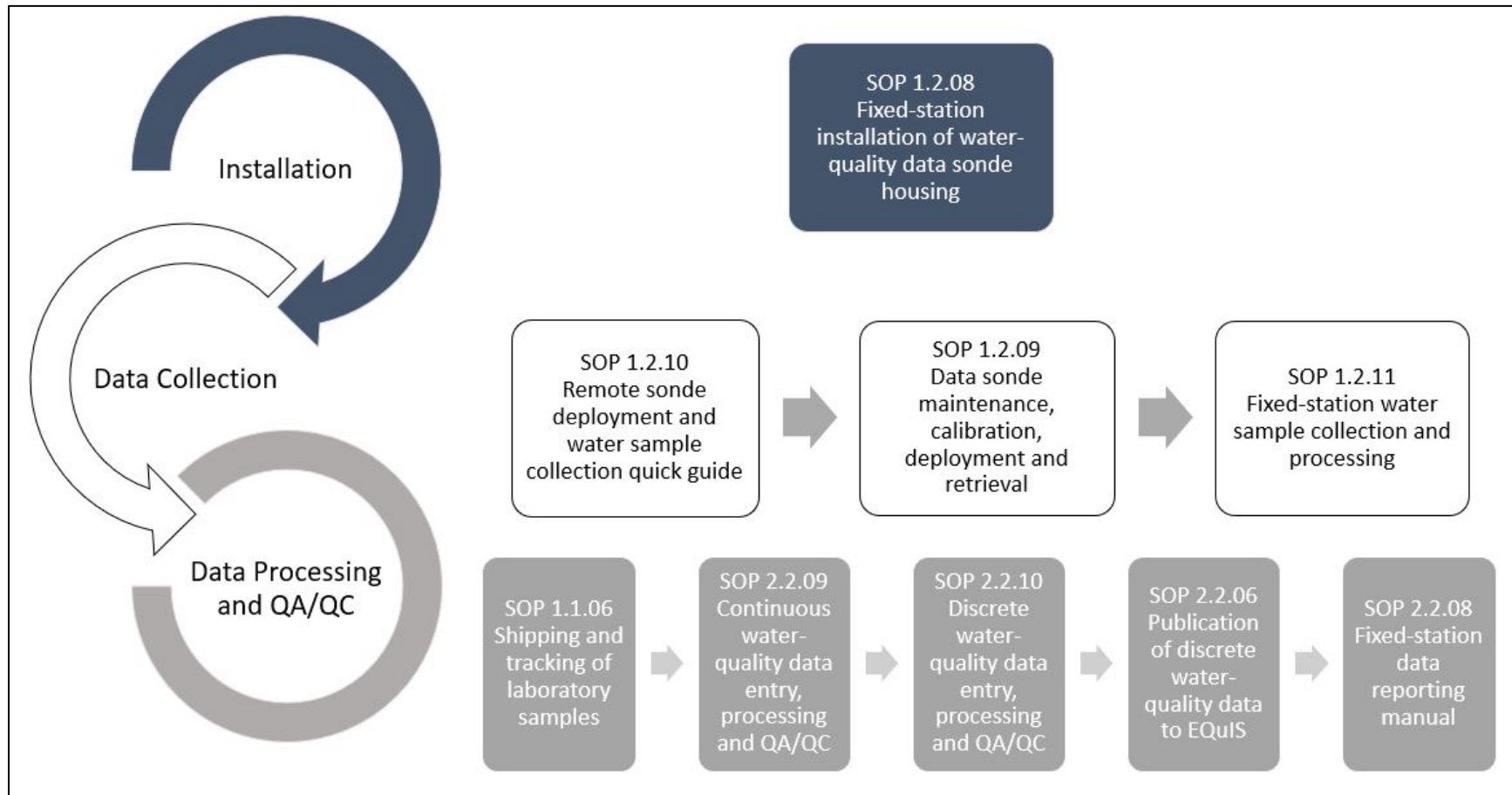


Figure 1. Fixed-station activities and workflow. SOPs are presented left to right in the general order of operations for each activity.

Equipment/Materials List

- For collecting samples
 - Van Dorn water sampler (horizontal)
 - Nitrile gloves
 - 1,000-milliliter (33.8 ounces [oz]) amber HDPE sample bottles (2)
 - Secchi disk
- For processing samples
 - 125-milliliter (4.2 oz) sample bottles (2)
 - Chlorophyll filtration kit (vacuum apparatus with graduated filter holder)
 - Whatman GF/F 47 mm – 0.7 micron filters
 - Forceps
 - Deionized (DI) water (1 gallon [3.8 liters {L}])
 - Graduated cylinder (500 milliliters [16.9 oz])
 - Aluminum foil (pre-cut into 6 × 6-inch [15.2 × 15.2 centimeters {cm}] squares)
 - Small plastic resealable bags
- For recording measurements
 - Water Sample Collection form
 - Waterproof address labels (such as Avery 5520, WeatherProof 1 × 2.625 inches [2.54 × 6.67 cm])
 - Pencils (for data forms)
 - Fine-tipped indelible markers (for labels)
 - Clear tape strips (required if not using waterproof labels)
- For sample storage and transport
 - Cooler with wet ice or ice packs (for sample storage while on boat)
 - Freezer (for sample storage upon completion of field work each day)
 - Cooler with dry ice (for extended sample transport)

Preparation

1. Check tide chart. **Samples need to be collected within three hours prior to published low tide.** This sample window helps the SECN sample estuarine water flowing out and minimizes the effect of ocean water coming in. Appendix A lists tide gages for each site.
 - a. For example, if the published low tide is at 10:00 EST then sampling can occur anytime between 7:00 and 10:00 EST.
2. Secure a cooler with either dry ice or ice packs for preserving raw and processed samples.
3. Check functionality of all sampling equipment and gather all required items in the Equipment/Materials List above.

Procedures

Do not apply sunscreen or other chemical contaminants until after the sample is collected.

Secchi Depth

1. Collect an observation of water column transparency depth, using a Secchi disk within 10 meters (32.8 feet [ft]) of the fixed-station installation.
2. Remove sunglasses, hats or other devices used to shade the eyes.
3. Slowly lower the Secchi disk until it is no longer visible.
4. If the disk hits the bottom, meaning the Secchi depth is greater than the water depth, note this on the datasheet by writing “Clear to Bottom” in the “Secchi Depth” blank and circling “Y” for “Did the secchi disk hit bottom before losing visibility?” on the Water Sample Collection Form (Figure 2).
5. Slowly raise the Secchi disk until it just becomes visible and note the depth using the markings on the line. Interpolate between the 0.25-meter (0.82 ft) markings to the nearest 0.1 meter (0.33 ft). Record this measurement on the Water Sample Collection Form (Figure 2).
6. As a check, lower the Secchi disk until it disappears and then raise it until it becomes visible. Note the depth using the markings on the line and interpolate to the nearest 0.1 meter. This check must be within 0.2 meter (0.66 ft) of the first observation, if not, repeat the process.

Station Name: _____ (e.g. CAHAocbr01, CALOshak01, CALOmidm02, CUISseac02, FOPUlazz01)	
Collection Date (MM/DD/YYYY): _____	Collected By: _____
Collection Time (HH:MM): _____ (EST)	Salinity (psu): _____
Secchi Depth (m): _____	Did the secchi disk hit bottom before losing visibility? Y / N
Low Tide Time: _____ (EST) Samples are to be collected between +3 hours before slack low water	
Sample Type (circle one): Routine Blank Duplicate	
Filtration Method: Vacuum (describe in comments if another method is used)	
Monthly Analytes: TDN ☐ TDP ☐ Chl a ☐	Quarterly Analytes:(February, May, August, November) DIN ☐ DIP ☐
Amount Filtered (ml): _____ Filtered By: _____	
Filtered Date: _____ Filtered Time: _____ (EST)	
COMMENTS 	
Computer Entry By / Date: _____ Accepted By / Date: _____	

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Sample Collection

1. Upon arrival at the sample location, complete the following fields on the Water Sample Collection Form (Figure 2): Station Number, Collected By, Collection Date, Collection Time (EST), and Low Tide Time (EST). Record any observations that are noteworthy (e.g., fish kill, algal bloom, recent hurricane).
2. Using a calibrated sonde, record salinity (PSU) to the nearest 10th of the sample collection site on the Water Sample Collection Form (Figure 2). If the sample is being taken in conjunction with a Sonde Deployment/Retrieval, then the “On Site Readings – Field Meter” salinity value can be used here if it is taken within 30 minutes of sample collection.
3. Put on nitrile gloves.
4. Collect a water sample using the Van Dorn sampler (Figure 3) within 10 meters (32.8 ft) of the fixed-station installation.
 - a. Remove Van Dorn sampler from case. Make sure both drain valves are closed.
 - b. Open each rubber end and attach the cable loop to the metal pin on the handle.



Figure 3. Van Dorn sampler ready for water sample collection.

- c. While holding metal weight, lower sampler into water and plunge rinse three times.

- d. Lower sampler into water to 0.5 meter (1.64 ft) below the surface (or mid-depth if station is less than 1.0 meter [3.28 ft]).
 - e. Drop the weight to release the end caps and capture the water in the sampler.
 - f. Haul up the sampler full of water and proceed.
5. Using the drain valves, thoroughly rinse the sampling bottle (1,000 milliliter [33.8 oz] amber HDPE) and cap three times with water from the sample.
 6. Fill the sampling bottle (1,000 milliliter amber HDPE) with remaining sample water.
 7. Collect QA/QC samples if necessary, based on the yearly QA/QC schedule. See the QA/QC Samples section below for details.

Sample Processing

1. Rinse the filter funnel and graduated cylinder three times with DI water prior to filtration.
2. Using forceps, place a clean filter into the filtration funnel.
3. Shake the sample bottle to mix the sample. Using the graduated cylinder, measure and pour 250 milliliters (8.5 oz) of water into the filter funnel and use the vacuum pump to draw water through the filter. As recommended by the EPA, “do not exceed 7 inches (17.8 cm) of Hg of vacuum approximately 3.4 psig (pounds per square inch gauge) or a filtration duration of more than five minutes for a single sample volume, to avoid cell damage or loss of contents during filtering.”
4. Use the first 10–20 milliliters (0.3–0.7 oz) of filtrate to rinse the inside of the filter flask and discard the rinsate. Replace the filter flask and continue filtering. Repeat with an additional two rinses of filtered site water.
5. If the filter clogs before 250 milliliters of sample water passes through the filter, discard the filter and water remaining in the funnel. Rinse the funnel with DI water, install a new filter and repeat the procedures above with 100 milliliters (33.8 oz) of sample water.
6. If there is visible color on the filter, proceed to the next step; if not, filter additional sample water until color is visible on the filter or until a maximum of 2,000 milliliters (67.6 oz) have been filtered.
7. Remove the filter gently from the filtration funnel and place it (filtrate side up) on the pre-cut aluminum foil square. Using the forceps and foil to avoid touching the filter, fold the filter in half. Proceed to wrap the filter in aluminum foil by folding the foil to form a sealed pouch resembling an envelope (Figure 4).

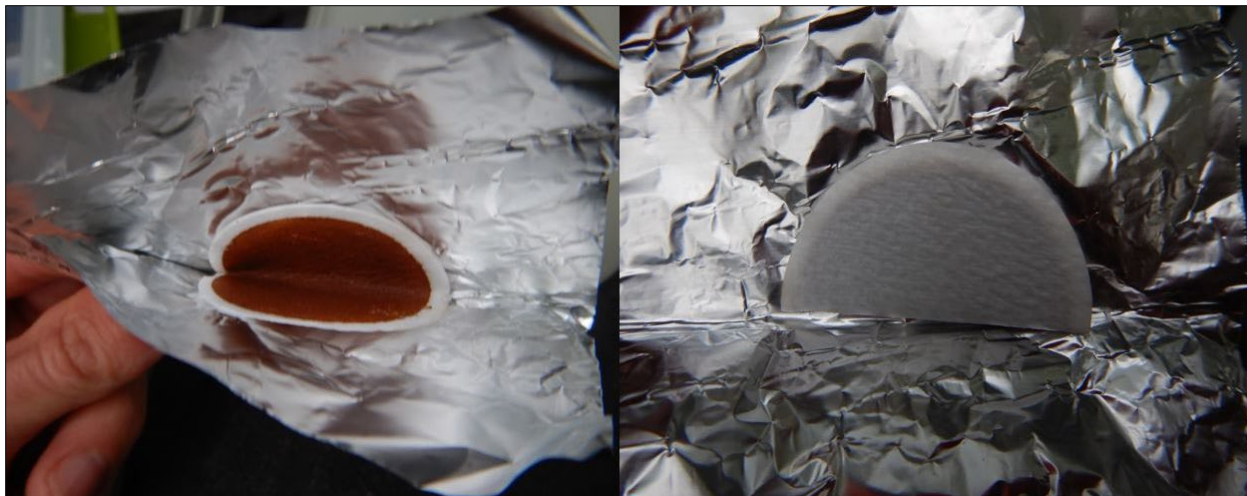


Figure 4. Properly folded filters ready to be sealed in an aluminum pouch and frozen.

8. Fill out labels for Chlorophyll *a* and Dissolved nutrients N&P (Figure 5). Affix the Dissolved Nutrients N&P labels to the 125 ml sample bottle.

Station _____ Date _____ Time _____ Collected by _____ Chl. a Volume _____	Station _____ Date _____ Time _____ Collected by _____ TDN & TDP	Station _____ Date _____ Time _____ Collected by _____ TDN, TDP, DIN, & DIP (Quarterly)
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Figure 5. Example of Chlorophyll *a* filter label and dissolved nutrient sample bottle labels.

9. Place the Chlorophyll *a* label on the foil pouch, and place it into a small zip-lock plastic bag.
10. Pour the remaining filtered water into the 125 milliliters acid washed sample bottle labeled for analysis of “Dissolved nutrients N&P.” Fill the bottle only to the neck in order to allow room for expansion when the sample is frozen.
11. Store water samples in a cooler at 4°C (39.2°F) until they can be frozen.
12. On the Water Sample Collection Form record: amount filtered (ml), filtered by, filtered date, and filtered time. Complete any remaining information on the Water Sample Collection Form (Figure 2). Include pertinent comments and note if QA/QC samples were collected.

QA/QC Samples

Field QA/QC samples (i.e., field duplicates and field blanks) will constitute at least 10% of samples collected throughout the year. These samples will be collected on randomly selected sites and dates and submitted with regular samples. Sites requiring a QA/QC sample will be identified prior to field sampling. An example annual QA/QC sample schedule is provided in Appendix B.

Duplicates

A field duplicate is a duplicate environmental sample collected by the same team or by another sampler or team at the same place, at the same time and not simply a split sample.

1. Take two sample bottles to the site, one for the “Routine” sample and the other for the “Duplicate” sample. In addition, take two Water Sample Collection Forms (Figure 2).
2. Procedures for collection of duplicate samples are the same as those for a “Routine” sample. Repeat all the Sample Collection and Sample Processing procedures for the “Duplicate” sample.
3. On a separate Water Sample Collection Form, circle “Duplicate” and record all the necessary information listed in the previous sections.
4. Duplicate sample labels include the station number followed by “dup” (e.g., CUISseac03_dup).

Blanks

A field blank is a sample of DI water that is lacking nutrients and chlorophyll *a*.

1. Procedures for collection of blanks are similar to a “Routine” sample; however, it is a sample of deionized water instead of estuarine water.
2. Take two sample bottles to the site, one for the “Routine” sample and one sample bottle full of DI water for the “Blank” sample. In addition, take two Water Sample Collection Forms (Figure 2).
3. Since the sample bottle is already full of DI water, skip the Sample Collection section, but repeat all the Sample Processing procedures for the “Blank” sample.
4. On a separate Water Sample Collection Form, circle “Blank” and record all the necessary information listed in the previous sections.
5. Blank sample labels include the station number followed by “blk” (e.g., CUISseac03_blk).

Sample Storage and Transport Considerations

1. Unprocessed water samples must be kept on ice and filtered as soon as possible following collection. **The maximum unprocessed sample hold time is four hours.**
2. Multiple sites can be sampled over the course of a field trip; therefore, filters and filtered samples will be held in a cooler with dry ice until they can be placed in the freezer or shipped to the laboratory.
3. Samples and filters need to remain frozen until they arrive at the laboratory for analysis. As a result, filters and samples must be shipped frozen, and in a Styrofoam container with sufficient ice packs. **The hold time for frozen processed samples is 28 days.**

4. Specific procedures for the transport of samples and filters to the laboratory are covered in SOP 1.1.06 *Estuarine Water and Sediment Shipping and Tracking of Laboratory Samples*—Version 2.0 (Starkey and McCay 2022).

Literature Cited

- McCay, D. L., E. N. Starkey, and M. B. Gregory. 2022. Fixed-station, water-quality data sonde maintenance, calibration, deployment, and retrieval. Version 3.0. Southeast Coast Network Standard Operating Procedure NPS/SECN/SOP—1.2.09. National Park Service, Athens, Georgia. Available at: <https://irma.nps.gov/DataStore/Reference/Profile/2284553> (last accessed June 2022).
- Starkey, E. N., and D. L. McCay. 2022. Estuarine water and sediment shipping and tracking of laboratory samples—Version 2.0. Southeast Coast Network Standard Operating Procedure NPS/SECN/SOP—1.1.06. Southeast Coast Network, Athens, Georgia. Available at: <https://irma.nps.gov/DataStore/Reference/Profile/2284582> (last accessed June 2022).
- US Environmental Protection Agency (EPA). 2014. National Coastal Condition Assessment: Field Operations Manual. EPA-841-R-14-007. U.S. Environmental Protection Agency, Washington, DC. 165 p.

Appendix A. Useful Tide Websites and Stations

Websites

- [Tide Predictions - NOAA Tides & Currents](#)
- www.saltwatertides.com

Stations

CAHA – CAHAocbr01

Ocracoke Island, NC - Station ID: 8654792

<https://tidesandcurrents.noaa.gov/stationhome.html?id=8654792>

CALO – CALOshak01 & CALOmidm02

Duke Marine Lab - Beaufort, NC- Station ID: 8656483

<https://tidesandcurrents.noaa.gov/stationhome.html?id=8656483>

CUIS – CUISseac03

Dungeness, Seacamp Dock, GA - Station ID: 8679758

<https://tidesandcurrents.noaa.gov/stationhome.html?id=8679758>

FOPU – FOPUlazz01

Fort Pulaski, GA - Station ID: 8670870

<https://tidesandcurrents.noaa.gov/stationhome.html?id=8670870>

Appendix B. Example QA/QC Sample Schedule

Table B-1. Example QA/QC sample schedule at selected SECN sites.

Month	CAHAocbr01	CALOshak01	CALOmids02	CONGbann01	CONGccbr01	CUISseac03	FOPUlazz01
January	–	–	–	–	–	–	–
February	–	Duplicate ¹	–	–	–	–	–
March	–	–	–	–	Duplicate ¹	–	Blank ²
April	Blank ²	–	–	–	–	–	–
May	–	–	Duplicate ¹	–	–	Blank ²	–
June	–	–	–	Blank ²	–	–	–
July	–	–	Blank ²	–	–	–	–
August	Duplicate ¹	–	–	–	–	–	–
September	–	–	–	–	–	Duplicate ¹	–
October	–	–	–	Duplicate ¹	Blank ²	–	–
November	–	Blank ²	–	–	–	–	–
December	–	–	–	–	–	–	Duplicate ¹

¹ Two separate replicate samples

² Deionized or distilled water