

Standard Operating Procedure (SOP) #11

Collecting Marine Water Samples

Version 4.0 (May 3, 2024)

Change History

New Version #	Revision Date	Author	Changes Made	Reasons for Change	Previous Version #
2.0	5/27/2016	David Raikow	The former SOP #7 has been split into multiple SOPs; new marine sampling procedures have been given their own SOP	Too much in one SOP; to reflect current practices.	1.0
2.01	7/18/2019	Kim Weisenborn	Minor updates to content	To update outdated SOP references	2.0
2.02	05/11/2020	David Raikow	Boat preparation and marine equipment preparation moved to this SOP. Renumbered.	General SOP reorganization	2.01
3.0	7/17/2020	Amanda McCutcheon	Added instructions for EXO deployment, updated SOP title, updated filtering procedures	Updated equipment, title was misleading, a few inaccuracies in filtering procedures	2.02
3.01	11/5/2020	Amanda McCutcheon	Added explicit instructions about which sonde to use and description of Ofu sampling	Mishap in the field that led to equipment loss at NPSA	3.0
3.02	6/2/2021	Kim Weisenborn, Amanda McCutcheon	Updated to NRR formatting; updated SOP references throughout this SOP; removed YSI 6600 references; minor text updates	Formatting needed to be applied; SOPs references needed to be updated because some had been removed or renumbered within the protocol; we updated equipment	3.01
4.0	5/3/2024	Amanda McCutcheon, Glauco Puig-Santana, Kim Weisenborn	Updated to reflect Marine protocol revision including removing bottom sampling from general procedure, adding shoreline sampling, and adding sampling via SCUBA. Renumbered SOP from #9 to #11. Updated SOP references.	Marine Protocol Revision was implemented in 2023. A new SOP has been added to the protocol therefore some SOP reference #s have changed.	3.02

Only changes in this specific SOP will be logged here. Version numbers increase incrementally by hundredths (e.g., version 1.01, version 1.02) for minor changes. Major revisions should be designated with the next whole number (e.g., version 2.0, 3.0, 4.0). Record the previous version number, date of revision, author of the revision, changes made, and reason for the change along with the new version number.

Purpose

This SOP outlines marine water quality procedures at Kaloko-Honokōhau National Historical Park (KAHO), Kalaupapa National Historical Park (KALA), War in the Pacific National Historical Park (WAPA), and National Park of American Samoa (NPSA). Complete understanding of water sample collection requires consultation of this SOP and [SOP #9 Collecting Freshwater Samples](#) for water sampling general procedures. Under the new Marine Water Quality Monitoring Protocol, marine water quality monitoring consists of collecting sonde readings as well as 3 filtered water samples at the surface (about 1 m depth) at fixed and temporary stations twice per year. The number of stations and sampling period vary by location. Some fixed stations are accessed from shore, while others are accessed by motorboat. Additionally, bottom samples are collected during annual benthic marine and marine fish monitoring dives at fixed stations.

Offshore Sampling by Motorboat

Boat Preparation

Boat, trailer, and motor servicing should be independently verified for each park where field operations will occur. Ensure that this required preparation has been done in accordance with NPS Motorboat Operator Certification Course Manual and park specific guidelines. Park-specific guidelines can be obtained from the Park Dive Officer or the certified motorboat operator at each park. Care and maintenance of the boat, motor, and trailer is also detailed in [SOP #13 Post-field Work Procedures and Equipment Maintenance](#).

Sampling Equipment Preparation

Use a marked lowering line for both the YSI and the Niskin bottle. A permanent marker can be used to color each line at 1-meter intervals. (Note: Be sure to re-do these measurements and markings and check the overall integrity of the lowering line between field sampling trips. NEVER use a lowering line that is compromised!). An additional line (for instance a manta reel or another line) should be attached to the sonde as a backup in case the lowering line breaks.

Prior to Leaving for the Field

1. Prepare sampling gear.
2. If possible, always use a sonde with a depth sensor (the larger sondes with battery compartment). ** Never use the EXO handheld or YSI 650 in the water. They are not waterproof. **

3. Install new batteries in the sonde and set sonde to log an unattended sample. This will provide continuous data until the log is stopped.
4. *To set up an unattended deployment with the YSI EXO2 multiparameter sonde using the KorEXO® software:*
 - a. Open the KorEXO software on your computer. Ensure that you have the most up-to-date software version.
 - b. Turn on the Bluetooth the EXO2 sonde by waving the magnet wand over the horseshoe shaped magnet symbol on the sonde.
 - c. In the KorEXO software click **Scan for Bluetooth Devices** on the lower right-hand side of the screen. The sonde will automatically connect. If your computer does not have a Bluetooth receiver, you can use the EXO USB Signal Output Adapter to connect the sonde to the computer.
 - d. The software will automatically update the sonde time and date to match the time provided by your computer. Please ensure that your computer is set to the correct time zone and that the time on your computer is accurate. You can check the time and date on the sonde on the **Instrument and Sensors** tab.
 - e. Click **Deployment** from the menu on the top of the screen or **Manage Deployments** on the home screen.
 - f. Click **Create Template** to create a new deployment template. You can save and edit this template for future deployments.
 - g. Enter a Deployment Template Name. A recommended name is XXXX_WQ_MR, where XXXX is the park's 4 letter code.
 - h. Set the Logging Interval Time to **30 seconds** and the File Prefix Name to today's date in the format YYYYMMDD.
 - i. Set the Site Name to XXXX_MR, where XXXX is the park's 4 letter code.
 - j. Under **Advanced** set the sonde to wipe every **20 samples** and leave all other choices at default.
 - k. Click **Save and Apply to Sonde** at the bottom of the screen.
 - l. A pop up will ask if you would like to start logging on an interval. Click **Yes**.
 - m. In the Select Deployment Start Time window choose the appropriate option. If you will begin sampling within the next hour, choose **Next Interval**. If you will not start within the next hour, choose **Custom** and set the date and time to an appropriate time well before you plan to start sampling. Then click **Start**.

- n. The deployment should now be running. Check the Estimated Log Space Duration and Estimated Battery Life Remaining to ensure there is ample space and battery.
 - o. Go back to the **Home** tab and click **Disconnect** to disconnect the sonde from the computer. You can turn the sonde Bluetooth off with the magnet wand or let it automatically turn off after 5 minutes.
 - p. Check that the sonde is logging periodically through the day. On the EXO2 you can check by watching the red blinking light on the sonde body. If logging, the light should blink at least every 10 seconds. You will also notice the sensor lights blinking and the wiper wiping periodically. Do your best not to bump the sonde or handle it roughly. This may knock the batteries loose causing the sonde log to stop. If the log stops, restart it using the EXO Handheld instructions below.
 - q. There is no way to set the end of deployment with the YSI EXO2 multimeter sonde. You will need to connect the sonde to the KorEXO software or handheld at the end of the day to stop the deployment and avoid wasting battery life.
5. *To set up an unattended deployment with the YSI EXO2 multiparameter sonde using the EXO Handheld device:*
- a. Connect the EXO2 multiparameter sonde to the EXO Handheld using the field cable and turn on the power. It may take a moment for the handheld to recognize the sonde and begin to show live readings.
 - b. Once the live readings appear, press **Deploy** to open the deployment menu.
 - c. Select **Deployment Setup** and set the log interval to 00:00:30.00 (30 seconds).
 - d. Enter the site name and file prefix as you would with the KorEXO software.
 - e. Exit the Deployment Setup menu.
 - f. Select **Start Deployment** and **Deploy [Now]** to start the deployment.
 - g. Turn off the handheld and disconnect the field cable.
6. Assemble the filter tower.
- a. Start with disassembled Filter Holder with stoppers in place.
 - b. Place filter plate assembly in the neck of the sample receiver (the bottom part).
 - c. Use pre-cleaned filter forceps to put a GF/F filter printed-side up. Be very careful to avoid touching the filter with or to anything other than other filters, forceps, or the filter plate (if you do, throw away the filter and get a new one). Do not touch the

filter plate with the forceps. You may lightly blow on a stack of filters in the box to separate one and grab it with the forceps.

- d. Set the upper chamber on the filter plate with the bottom O-ring touching the filter. Hold the upper chamber to keep it from turning (which can damage the filter) and screw the collar down onto the sample receiver finger-tight.

7. Fill cooler with ice.

Field Sampling Procedure

1. Using the GPS (Global Positioning System) (with sampling station waypoints pre-loaded), navigate to the selected water quality field sampling station, and secure boat (or live boat) according to the captain's preferences and local regulations. At NPSA's Ofu unit, the sites are accessed by swimming/wading from shore with a dive float or boogie board.

Note: A sampling site must be within a safe area, in no less than 10-m deep water (except at Ofu), and within authorized bounds of the park. If these conditions are not satisfied, the site should not be sampled.

2. Label water collection bottles. Apply pre-printed waterproof labels to each bottle. Bottles will be labeled with site name (ex: FWAPA01_mr), depth (surface), filter type (filtered GF/F), and a sample ID (A, B, C are filtered samples). Fill in sample collection date using pencil or waterproof pen.
3. Fill out a field datasheet (see Figure SOP 11.4) for each site.
4. Determine the bottom depth using a handheld depth sounder or the boats' depth sounder.
5. Collect water sample using Niskin bottle 1 m below the water surface.
 - a. Be sure the Niskin bottle is securely attached to the lowering line.
 - b. Activate/set the Niskin bottle according to the manufacturer's instructions.
 - c. Once the boat is stabilized and conditions are safe, carefully lower the activated Niskin bottle over the gunwale or transom of the boat and into the water. Note: Be very careful not to activate the closure mechanism when contacting the ocean surface.
 - d. Lower the Niskin bottle to the selected depth (1 m) by monitoring the markings on the lowering line. Note: If at any time, it is suspected that the Niskin bottle has prematurely closed, the bottle should be brought back on board the boat, the sample discarded, and the bottle reset for sampling.
 - e. At the desired depth mark, drop the Niskin messenger to enclose the water sample, then bring the bottle back on board with the line.
 - f. Ensure filter tower is properly loaded with GF/F filter and filter is in place.

- g. Remove the screw-on lid and set it upside down to act as a tray.
- h. Remove the plug from one of the vents on the side of the sample receiver (lower chamber), but make sure the vent opposite it is plugged.
- i. Put the plug in a clean, safe place, such as the upside-down lid.
- j. Connect the vacuum pump hose to the vent you just uncovered on the lower chamber.
- k. Make sure the pump switch is set to vacuum, not pressure.
- l. Fill upper chamber of filter tower with about 150 mL of unfiltered water from the Niskin bottle as per manufacturer's instructions.
- m. Apply a vacuum with the hand pump. With a new filter, you should not need more than 5 mmHg of pressure to draw water into the lower chamber. A filter begins to get clogged when 5-10 mm Hg is necessary to draw water through the filter. Consider changing the filter when 10 mm Hg of pressure is needed to draw water through the filter. Never exceed 15 mm Hg of pressure at any time.
- n. Filter the entire volume of water added to the upper chamber.
- o. Once all the water in the top chamber has passed through the filter, release pressure with the hand pump using the small trigger-like valve.
- p. This water will be used to rinse the filter tower only.
- q. After water is pumped through swirl water in lower chamber to rinse chamber.
- r. Remove the remaining plug on the lower chamber and the vacuum pump hose connection and set the plug and pump in a safe, clean place where you can easily reach them.
- s. Tilt the filter holder assembly over so the water can drain out of one of the vents you just opened. Discard all the water from the lower chamber.
- t. Fill the upper chamber of the tower with another 150 mL of water from the Niskin bottle and replace the plug and pump hose. This water will be used to rinse all 3 sample bottles.
- u. Filter the water through the tower and rinse each bottle 3 times with about 10 mL of water.
- v. If there is enough water remaining in the lower chamber after you have rinsed the bottles for sample A, continue to Step X, otherwise, discard this water. Tilt the filter assembly to empty the lower chamber as much as possible.

- w. Refill upper chamber with unfiltered water from Niskin bottle and repeat filtering procedure. This water will be sample A.
 - x. Fill the water bottle only up to the shoulder. It is very important not to over-fill the bottle because the sample will expand when frozen. Cap the bottle.
 - y. Replace the filter between each site or if it gets clogged.
 - z. Discard any remaining water, reactivate the Niskin bottle, return to site if necessary, and repeat for the other 2 filtered water samples. Each sample should be a new grab with the Niskin bottle. Do not filter multiple samples from the same Niskin bottle. Store the samples in a cooler of ice for transport.
6. Lab filtering: As an alternative to filtering samples in the field, lab filtering is authorized. Marine water quality samples may be taken in the field and filtered on-shore in a lab setting if filtered the **same day** as collection. Samples are collected using standard procedures with unfiltered water collected in 250 mL sample bottles. Samples are immediately placed on ice in a dark cooler while on the boat. Samples are filtered after returning to the park using standard filtering methods with either the filter tower and hand pump or an electric lab filtering system if available (at WAPA). Filtered water samples are stored in clean sample bottles and not the bottles used to collect unfiltered samples in the field.
7. Deploy the YSI sonde. Sonde deployment can be done before or after collecting water samples or simultaneously if the operation can be done safely. If live boating, it is best that one person be assigned to monitor the sonde and ensure that the line does not get near the boat prop while the captain is navigating to stay on station.
- a. Attach the YSI sonde securely to the lowering line (or a winch system). Use both a primary attachment method and a backup method (e.g. a stainless-steel carabineer attached to a marked line (every 1 m) and stainless-steel carabiner attached to a manta reel or another line).
 - b. Remove the sonde from the calibration cup and ensure the probe guard is in place on the sonde.
 - c. Once the boat is stabilized and conditions are safe, carefully lower the YSI sonde over the gunwale or transom of the boat (or from the float at NPSA's Ofu Unit) and into the water.
 - d. Secure the lower line at the appropriate depth (1 m below the surface).
 - e. Record sampling Start Time and Depth on the field datasheet.
 - f. Allow sonde to take readings at the bottom depth for 10 minutes. Record the sampling End Time.

- g. Bring the sonde on board. Place it back in the calibration cup or cover the probes with a wet rag to keep the probes moist while moving to the next site. When you have completed sampling at the last site make sure to place the sonde securely in the calibration cup with water.
8. Using Field Maps® loaded onto a mobile device ([SOP #6 Using ArcGIS Field Maps App](#)), take one digital photograph, toward shore at NPSA and WAPA or take four digital photographs of the sampling site facing at each of the four cardinal directions (N, S, E, W) at KAHO and KALA. Note the direction in which each photo was taken on the field datasheet. Take additional pictures of environmental concerns/interests and log them on the datasheet.
9. Complete the field datasheet in the field binder for each sampling site.

Sampling from Shore

Sampling Equipment Preparation

Shoreline sampling is conducted in much the same way as the freshwater and anchialine pool sampling, however, some sites may require the use of either a Niskin bottle or a long sampling pole to collect water safely. Use the small EXO2 sonde and handheld for all shoreline sampling when available. Ensure that the wiped conductivity sensor is calibrated to 50 mS/cm and a calibrated total algae probe for *Phycoerythrin (Saltwater)* is installed. You may move already calibrated probes between sondes without recalibrating.

Field Sampling Procedure

Follow the general field sampling procedures outlined in [SOP #9 Collecting Freshwater Samples: General Procedures](#) to collect YSI data and 3 filtered water samples at each shoreline site. Use the freshwater datasheet to record the sampling event information. Use a Niskin bottle or long sampling pole if the sampling location is more than a comfortable arms' reach below ground level or if waves prevent safe access without these aides. Pay careful attention to the surf and tide conditions before placing the sonde and ensure that the sonde is placed such that the probes will remain submerged, and the handheld remains out of the water while waves move in and out. Someone may need to hold the handheld for the 10 min sample log to prevent damage to the equipment.

Bottom Sampling Concurrent with Benthic Marine and Marine Fish Dives

The YSI sonde will be carried to each fixed site by the benthic marine and marine fish divers. It will be deployed on a float and line attached to a dive weight at the beginning of the dive and sent to the surface along a manta reel line attached to a surface float after 10-15 mins. Water samples will be collected in syringes underwater, carried to the surface by divers, and then filtered into sample bottles on board the boat.

Sampling Equipment Preparation

The YSI sonde should be prepared and deployed for unattended sampling following the procedures outline above under “Offshore Sampling by Motorboat”. Filter caps should be pre-loaded with GF/F filters (at least one per site) and placed in a Ziplock bag to prevent contamination (These will remain on the boat). **Four** sterile or acid-washed syringes should be prepared for each site and placed in a

mesh bag to be carried by divers. Three acid-washed bottles should be prepared with labels for each site (these will remain on the boat).

Field Sampling Procedure

1. Prior to the dive:
 - a. Ensure that the sonde is logging by checking the indicator light. It should be blinking RED once every 4 seconds.
 - b. Connect the sonde to the yellow float and the float to the dive weight using carabiners (Figure SOP 11.1).
 - c. Remove the calibration cup from the sonde (Figure SOP 11.1).
 - d. Attached the manta reel to the surface float.
 - e. Place four syringes in a mesh bag and secure the bag to the diver's BCD.
2. During the dive:
 - a. Descend carefully making sure not to become entangled with the sonde line or manta reel.
 - b. Locate the start pin for the transect.
 - c. Tie off the manta reel far enough away from the transect so that it does not interfere with the fish and benthic surveys.
 - d. Place the sonde, float, and dive weight assemblage near to the manta reel on non-living substrate, ensuring that the sonde will not bump any coral if there is surge (Figure SOP 11.2).
 - e. Note the time of sonde placement and sampling depth on a slate or the benthic datasheet. This value will be transferred to a water quality datasheet on the surface.
 - f. After the fish diver has gone 10 or so meters along the transect, you can begin water sampling.
 - g. Go to the start pin and take one syringe out of the mesh bag.
 - h. Pull the plunger to fill the syringe with water then depress the plunger to release the water. Repeat this 3 times to rinse the syringe and then fill the syringe a fourth time to collect the sample.
 - i. The sample should be collected near the start pin as close to the substrate as possible (within a few centimeters) (Figure SOP 11.3). If the bottom is sandy in the area you may need to collect the sample a farther away to prevent sucking sand into the syringe.

- i. Place the syringe back in the mesh bag and then repeat step h with the remaining 3 syringes.
 - j. After all 4 syringes are full of water, ensure the mesh bag is secure for the rest of the dive. Syringes may float off if the bag is opened. Also, be mindful that the syringes are not inadvertently depressed during transport, or the water will leak out of the syringes before you reach the surface.
 - k. About 10-15 mins after the sonde was placed, attach the carabiners that are connected to the sonde and float to the manta reel, leaving the sonde and float attached. Ensure that the connections are secure.
 - l. Release the connection between the float and dive weight, leaving the carabiner with the dive weight.
 - m. The float and sonde should ascend to the surface float up the manta reel line. Watch the assemblage while it ascends to ensure that it is successful. If the site is deep, the float may need to be assisted up the first meter or so until the float is under less pressure. If the line is too loose, the assemblage may get stuck mid-column and can be loosed by tugging on the line from the bottom.
3. After the dive:
- a. Carefully hand the sonde and mesh bag of syringes to someone on board when returning to the boat. Secure these items in a safe location until all divers and equipment are on board and the boat is in a safe location.
 - b. Place the sonde back in the calibration cup or cover the probes with a wet rag to ensure that the probes do not dry out.
 - c. Gather the prepared bottles, filter caps, and the water quality datasheet.
 - d. Transfer the sonde start and end times and sampling depths to the datasheet and complete all other fields on the datasheet.
 - e. Connect a filter cap to the first syringe.
 - f. Use the first syringe to rinse the 3 sample bottles, 3 times each.
 - g. Disconnect the filter cap and discard any remaining sample in the syringe.
 - h. Connect the same filter cap to the next syringe and fill the sample A bottle.
 - i. Repeat for samples B and C, using a new syringe for each bottle. The same filter cap may be used for all three samples if it is not clogged. If it becomes difficult to depress the syringe, replace the filter cap and continue filtering the sample.

- j. Place the samples in a cooler on ice.

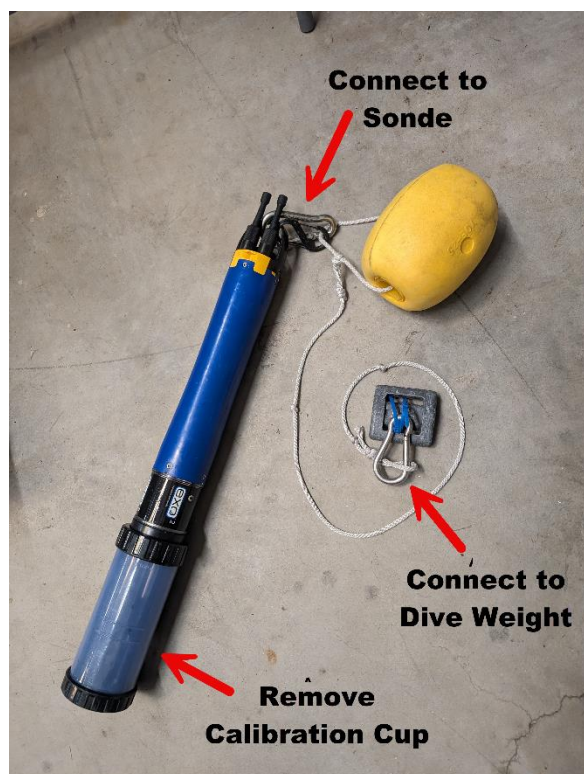


Figure SOP 11.1. Sonde set up for underwater deployment.



Figure SOP 11.2. Sonde deployed underwater.



Figure SOP 11.3. Collecting a water sample underwater with a syringe.

Site:	Date:	Start Time:	End Time:
Observers:		Recorded by:	

Sampling Location and Conditions

Latitude:	Longitude:	Error (m):
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Environmental Conditions (<i>circle one</i>):	Wind Direction: (<i>blowing from, e.g. NW</i>)	Wind Speed (kph):
Sunny (<i>> 75% sun</i>) Partly Cloudy (<i>25% to 75% sun</i>) Mostly Cloudy (<i>< 25% sun</i>) Overcast Rain		

Water Depth (m):	Wave Height (m):	Tide Stage (<i>circle</i>): High Mid Low	Tide Direction (<i>circle</i>): Ebb Slack Flood
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Site Photo #:	Direction:	Comments:
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Habitat / Site Comments:

Nutrient Sampling (*Water samples*)

Relative Depth	Depth (m)	Sample IDs (e.g. FPARK01_mf_sur A, B, C)	Filter Type (e.g. GF/F)	Sampling Activity Comments
Surface				
Bottom				

- At each relative depth, collect three GF/F filtered samples (samples A, B, and C).
- Label each sample with site ID, sample ID, filter type, relative depth, sampler initials, and date.
- Collect between 100 ml and 125 ml of sample in each bottle – do not overfill! Seal tightly and store in a cooler, on ice.

Sonde Measurements (*YSI*)

Sonde Serial #:

Relative Depth	Depth (m)	Start Time (e.g. 14:20)	End Time (e.g. 14:35)	Sampling Activity Comments
Surface				
Bottom				

- Collect at least 5 minutes of data at each depth -- about 1m below the water's surface and 1m above the bottom. Start with the surface measurement.

Datasheet checked by:	Checked date:	Entered into database by:	Entered Date:
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PACN Marine Water Quality Sampling Datasheet

Revised 13 January 2016

Figure SOP 11.4. Sample marine water quality field datasheet.

Post Field Tasks

1. Stop the sonde deployment.
 - a. *For the YSI EXO2 via Computer:* Connect the sonde to the computer via Bluetooth or USB wire. Click **Deployment** or **Manage Deployments** to access the deployment tab. Click **Stop Deployment** to stop the deployment.
 - b. *For the YSI EXO2 via handheld:* Connect the sonde to the handheld with the field cable. Press **Deploy** and select **Stop Deployment**.
 - c. Review data to make sure all the sites were captured that day just in case if some sites need to be resampled.
2. Rinse all gear with freshwater, specifically the Niskin bottles and YSI sonde.
3. Store samples in freezer.
4. Ensure datasheets are properly and completely filled out. Follow [SOP #13 Post-field Work Procedures](#) to upload YSI data, photos as well as scan and archive datasheets.