

NOSAMS Sampling Procedures for Seawater DI^{14}C

Seawater samples for DI^{14}C are collected as part of the GO-SHIP program. Key factors for sampling to achieve quality DI^{14}C results are minimizing headspace, poisoning with mercuric chloride to halt biological activity, and confirming a gas-tight seal.

Items to be prepared before collection (provided by NOSAMS for GO-SHIP samples):

- Saturated aqueous HgCl_2^* solution
- 100 μl Eppendorf pipette with tips
- Plastic pipette with bulb, a cooking baster is ideal (optional)
- Nitrile gloves
- Tygon or silicone tubing – 5/16" OD
- Tubes of Apiezon "L," "M," or "N" grease
- Rubber bands (5" L x 5/8" W)
- Laboratory wipes (e.g., Kimwipes)
- Teflon tape
- Labels
- Rubber or silicone stoppers
- Garbage bags or plastic sheeting

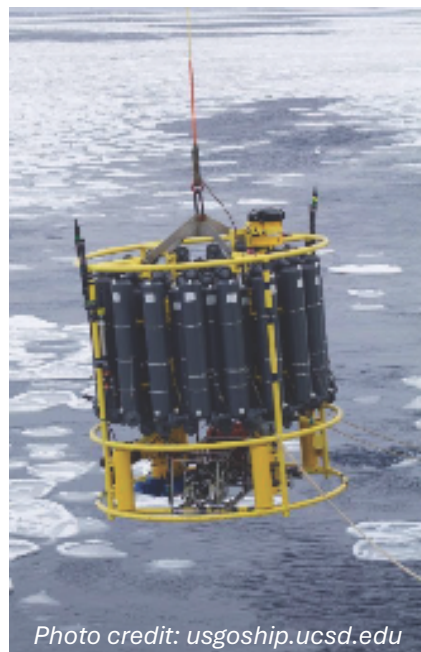


Photo credit: usgoship.ucsd.edu

* The easiest and perhaps safest way to prepare saturated HgCl_2 solutions for use at sea or in the field is to pre-weigh the HgCl_2 powder (ACS grade, crystal) into plastic bottles and add distilled water at sea. The solubility of HgCl_2 is approximately 7 g/100 ml at 20 °C; each sample requires 50-100 μl of solution. Thus, collection of 1000 water samples would require at most 100 ml total solution.

*** The Merck index lists HgCl_2 as a “violent poison” for which 1 or 2 g is frequently fatal. After using HgCl_2 (either as powder or in solution), the user should always wash thoroughly before eating or drinking. The powder should not be inhaled because it is corrosive to mucous membranes. ***

DI^{14}C Sampling Procedures

The integrity of DI^{14}C samples can only be guaranteed if the samples are collected using the proper procedures and collected in a DI^{14}C -free environment. The bottles should be handled as little as possible and removed from their packing crates only when necessary. Data sheets should be kept for each crate of bottles. For GO-SHIP, NOSAMS provides data sheets indicating the information we require for each sample (included at the end of this document).

Clean, powder-free disposable gloves should be worn any time the bottles are handled. When the bottles are removed from the crates, they should not be placed in direct contact with any surface on the ship either on deck or in the laboratory. Plastic sheets or garbage bags can be placed on any surfaces that the bottles must touch. Bottles for each cast can be transferred to the deck for sampling using the foam trays in the packing crates. Prior to actually sampling the seawater, as much information as possible should be written on the bottle label or a data sheet (an example is included in the sampling kit binder).

In the lab prior to sampling, remove the glass stoppers and the laboratory wipes from the bottles for the upcoming cast (always make sure the strips of lab wipe have been removed before collecting any seawater). Gently place rubber stoppers in bottles to transport out to the deck for sampling.



Place the tubing (Tygon or silicone) on the Niskin bottle and flush the tubing with approximately 25 ml of water. Then place the tubing inside the sample bottle, making sure the tube reaches to the bottom of the bottle and the vent at the top of the Niskin bottle is open. Fill the bottle with approximately 25 ml of water; gently swirl around the sides of the bottle and discard; repeat once more. With the sampling tube at the bottom of the bottle (Figure 1), fill with enough water to fill the bottle 1.5 times; this can be accomplished by observing the amount of time it takes to fill the bottle and allowing the bottle to overflow for half this time. Gently stopper the bottle with an ungreased rubber stopper. If too much pressure is applied, the ungreased stopper will not be able to be removed from the bottle. Repeat this procedure for the remaining samples from the cast. If two samplers are available, have one sampling from the Niskin and one in the lab poisoning/stoppering the bottles. Using this procedure, a DI^{14}C water sample in a 100-ml bottle will require approximately 225 ml of water.



Figure 2. Properly greased stopper

When all the bottles have been filled, remove them to a safe dry place and continue preparing the samples for storage. Using the grease syringe or gun, apply a thin layer of grease in a wavy pattern around the stopper (Figure 2); set the stopper aside. Remove the rubber stopper from the sample, and, using the large pipette or just by pouring, remove enough water for a 5-10 ml headspace to exist in the bottle. This level can be

marked on the bottle but is approximately midway to the shoulder of the 100-ml NOSAMS-provided bottles (Figure 3). Carefully and completely wipe the inside of the ground glass joint using lab wipes. Care must be taken not to put your finger in the sample. The joint **MUST BE DRY** for the grease seal to work properly! Using the Eppendorf pipette, add 50 μ l of the saturated HgCl_2 solution to the bottle (Figure 4). Place the greased glass stopper into the bottleneck and twist the stopper around while applying pressure to ensure that a good seal is made

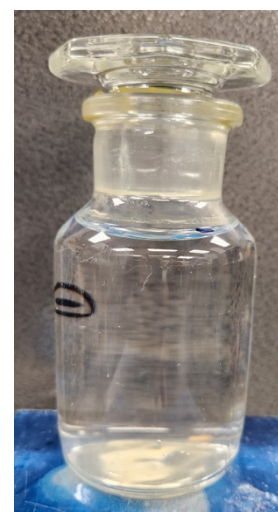


Figure 3. Example of 5-10 ml headspace, marked approximately midway to shoulder of 100-ml bottle.



Figure 4. Addition of HgCl_2 to sample bottle after headspace has been created.

DON'T FORGET TO ADD THE POISON!

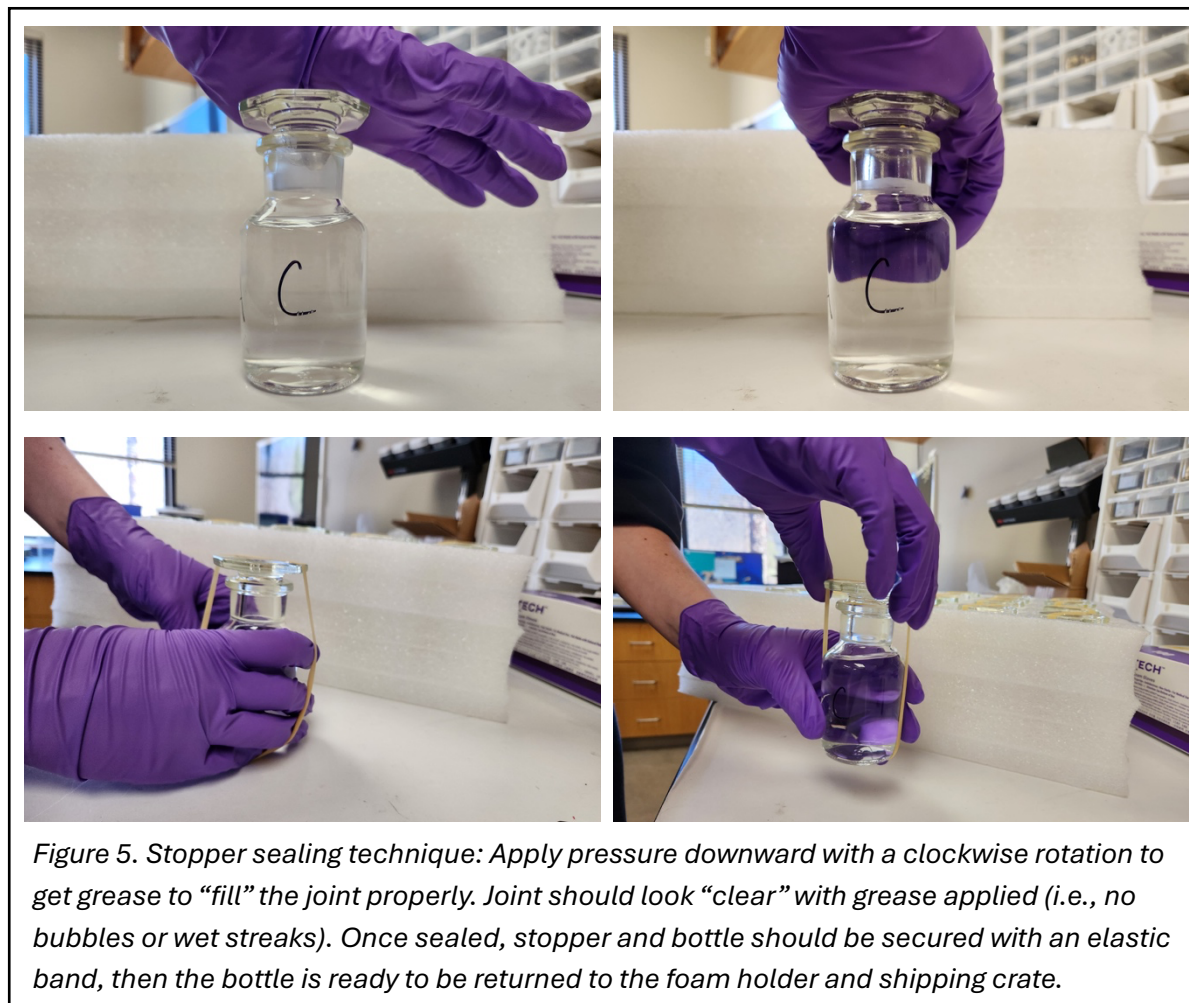
(Figure 5). The seal should be clear with no bubbles. Secure the stopper and bottle with one rubber band placed over the entire bottle. After bottles are filled,

poisoned, capped, and secured with an elastic band, shake gently to mix in poison. After all samples from one cast have been taken and sealed, each label/data sheet should be checked to make sure it contains the necessary information, and the integrity of the greased seals should be confirmed.

Information to include on the data sheet are: WHPID or transect, station number, latitude, longitude, date, cast, Niskin or rosette bottle number, sample depth or pressure, NOSAMS bottle number, NOSAMS box number,

confirmation of the addition of poison, and any comments. If there is not a box number on the box, please assign one and write it on the box with marker and use this assigned number. When the data for all the samples have been recorded properly, the samples should be transferred to the shipping crate. When time becomes available, data from all samples should be entered into a spreadsheet or other electronic form.

The plastic shipping crate should be closed securely (use tie wraps to secure) and stored in a temperature-controlled environment (e.g., the ship's science hold). Seawater samples must not be exposed to extremes of temperature. Poisoned samples do not need to be refrigerated and should **NEVER BE FROZEN**. If the samples are frozen, the water will expand and either dislodge the cap or break the bottle. If the sample is stored at too high a temperature, the grease will melt and run into the sample, and the sample may expand enough to dislodge the cap. The optimum working temperatures for Apiezon grease L, M, and N are 15-25 °C. To maintain their integrity, samples must be stored in a van or environment that can maintain the temperature within this range. The samples should also not be exposed to extreme temperature during shipment.



Please email Roberta Hansman (rhansman@whoi.edu) if there are any questions or concerns regarding this procedure. We appreciate the time you are taking to collect our samples carefully!

Bottle Cleaning Procedure (for non-NOSAMS-provided bottles):

The bottle used for the collection of seawater for DI^{14}C is a 100-ml glass reagent bottle with 29/26 standard taper ground glass joint and a glass stopper (Wheaton #W216015). Teflon tape or a piece of laboratory wipe is essential for preventing the stopper from seizing when shipping the bottles. Prior to packing for use at sea, the bottle must be cleaned.

To clean previously used bottles, first wipe any excess grease from the stopper and ground glass joint on the bottle. Remove any labels on the outside of the bottle. For all bottles (purchased new or previously used), wash the bottles and stoppers with a dilute soap solution, rinse well with warm tap water, rinse the bottles and stoppers with 10% HCl, and finally rinse three times with distilled water. From this point on, do not leave the bottles upright without covering the opening with clean aluminum foil. The foil can be rinsed with distilled water.

Bake the glassware in a 450 °C oven for 1 h. When the bottles and stoppers are dry and cool, place a piece of Teflon tape or a laboratory wipe in the ground glass joint of each bottle. Part of the tape or wipe should extend over the lip of the joint. Finally, place the stopper in the bottle.

After cleaning and capping, each bottle should be labelled or clearly marked with a unique number or other identifying information before sampling.

WHOI NOSAMS ¹⁴C WATER SAMPLING DECK LOG

CRUISE	_____	SAMPLER	_____	LAT	_____
STATION	_____	CAST	_____	LON	_____
DATE	_____	TIME	_____	BOX #	_____

[illegible]

Other Comments:

GO SHIP Sample Kit

- Binder with deck logs
- Tyvek envelopes
- Grease Gun (2)
 - Preloaded syringes for grease gun with tips (5)
 - Extra syringes (3)
 - M Grease (2 tubes)
 - Syringe tips (10)
 - 10mL syringe- in case gun breaks (3)
- Rubber bands (2 bags - 360 bands/bag)
- Nitrile Gloves (1 each of Sm., Med., Lg.)
- 10-100uL pipette (2)
 - Pipette tips (24)
- Re-Pipetter?
- Silicone and Tygon tubing (2 each- 2' length 5/16" dia.)
- Silicone Stoppers- #6? (12)
- Garbage Bags (24)
- Kimwipes (3)
- Pens/Pencils/Markers
- Extra Bottle labels
- Shrink Wrap (1 roll)
- Shrink Wrap Handles (2)
- Carboard Corners for packing pallet (12)
- Ratchet Straps (4-6)
- Tie Wraps (50)
- Extra bottle number labels
- Wire Snips
- Utility Knife
- Electrical Tape
- Duct Tape (1 Roll)
- Pads of Paper