

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

The bottle used for the collection of seawater for DI^{14}C is a 500- or 100-ml glass reagent bottle with 29/26 standard taper ground glass joint and a solid stopper. 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 the bottles, first wipe any excess grease from the stopper and ground glass joint on the bottle. In a hood, further clean the stopper and joint with laboratory wipes soaked in xylene and acetone to prevent transfer of grease from the stopper region to the inside of the bottle. After washing with solvents, allow the pieces to dry in a well-ventilated area. When using xylene, solvent-impermeable gloves should be worn. The above steps are not necessary for bottles fresh from the factory.

The following steps are necessary for all bottles, unless an arrangement has been made with the factory to provide bottles cleaned in the same manner. When the labels and grease have been removed and solvents have evaporated, 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 bottles upright without covering the opening with clean aluminum foil. The foil can be rinsed with distilled water. Bake the glassware overnight in a 450°C oven. When the bottles and stoppers are dry and cool, place teflon tape or a laboratory wipe in the ground glass joint of each bottle. Part of the tape should extend over the lip of the joint. Finally, place the stopper in the bottle.

After cleaning and capping, each bottle must have a label affixed. Spaces for the following information are suggested for the label: sample number or ID, sample location, cruise name and number, leg number, station/cast number (if collected at sea), depth, date, and time.

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
- Swabbing tool (a stick with laboratory wipes attached)
- Tygon drawing tube (pre-treat by soaking clean seawater for at least one day)
- Tubes of Apiezon "L", "M", or "N" grease
- Rubber bands (5"L x 5/8"W)
- Laboratory wipes
- Teflon tape
- Labels

* 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 only 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 in 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. ***

DI¹⁴C Sampling Procedures

The integrity of DI¹⁴C samples can only be guaranteed if the samples are collected using the proper procedures and collected in a DI¹⁴C-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 end of this document).

Clean, 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



Figure 1. Filling bottle from Niskin. Note the tygon tube is at the base of the bottle. Count how long it takes for the bottle to fill, and then allow the water to overflow the bottle for half this time. Pull out the tygon tube, and remove enough water to leave an approximately 3/8" headspace below the stopper joint.

DON'T FORGET TO ADD THE POISON!

any surfaces the bottles must touch. Bottles for each cast should be transferred from their packing crate to the plastic sample holder designed for use during transport and filling of the bottles. 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).

Immediately prior to sampling, remove the glass stopper and the laboratory wipe (always make sure the strips of lab wipe have been removed before collecting any seawater). Place the tygon tubing on the Niskin bottle and flush the tubing with approximately 50 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 50 ml of water; gently swirl around the sides of the bottle and discard; repeat once more. With the tygon sampling tube at the bottom of the bottle, fill with enough water to fill the bottle 1.5 times (Figure 1); 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 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. That way you do not have to stopper the bottle twice. Using this procedure, an DI¹⁴C water sample in a 500-ml bottle will require approximately 850 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. Remove the stopper; wipe clean and dry; 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. Using the large pipette or just by pouring, remove enough water for a 5-10 ml headspace to exist in the bottle (Figure 3 shows a bottle with the right amount of headspace). Carefully and completely wipe the inside of the ground glass joint using lab wipes and place the stopper in the bottle. 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 100 μl (or 50 μl for 100-ml samples) of the saturated HgCl_2 solution to the bottle (Figure 3). Twist the stopper around while applying pressure to ensure that a good seal is made (Figure 4). Secure the bottle top with one rubber band placed over the entire bottle. If a duplicate sample is to be taken, start filling the second bottle immediately using the same procedure. After both bottles are filled, 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 checked.

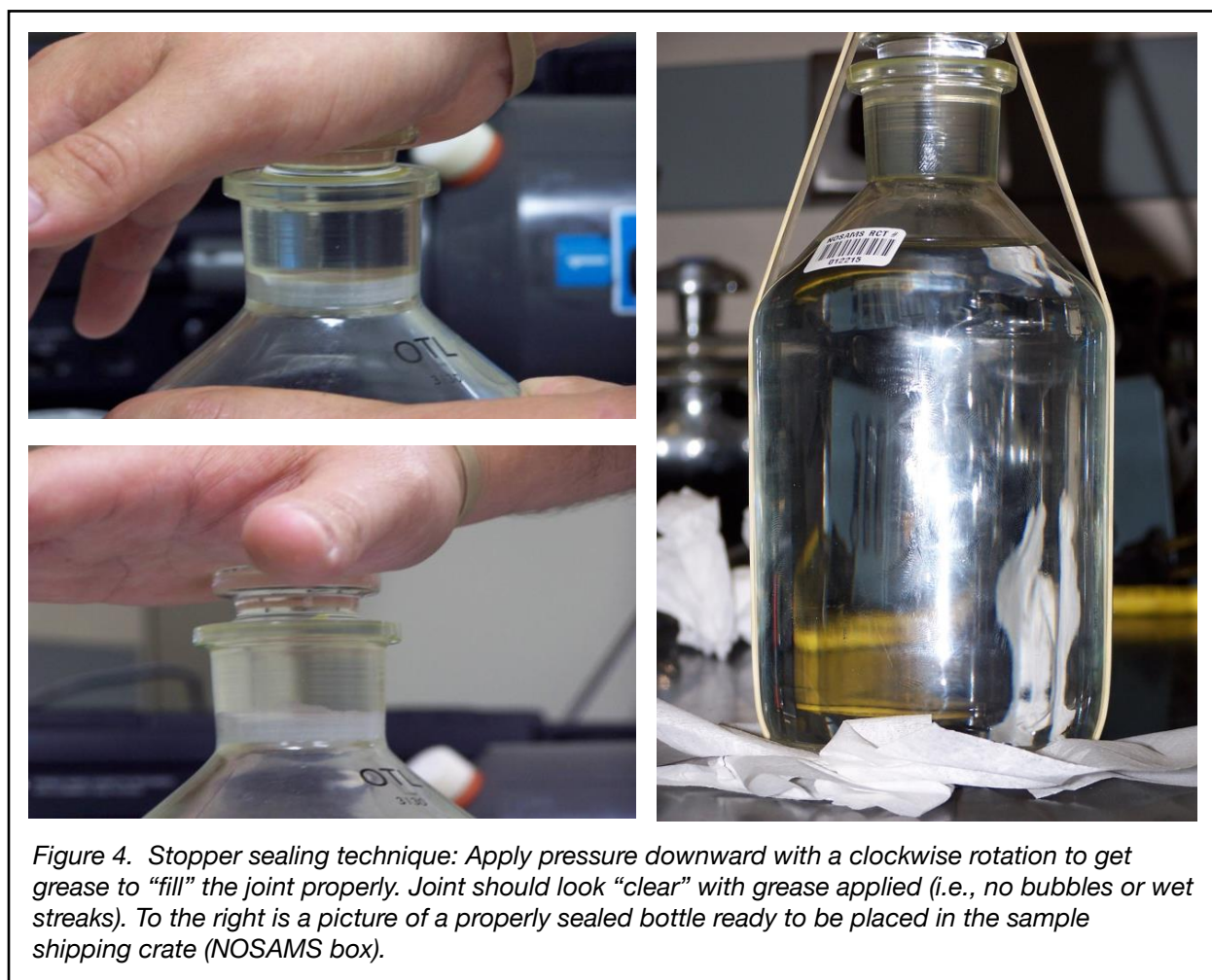
Information to include on the data sheet are: WHPID or transect, station number, latitude, longitude, date, cast, Niskin or rosette bottle number, sample depth or pressure, NOSAMS bottle number, NOSAMS box number, confirmation of the addition of poison, and any comments. If there is not a box number 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.



Figure 3. Addition of HgCl_2 to sample bottle after headspace has been created.

The plastic shipping crate should be closed securely (use tie wraps to secure) and stored in a temperature-controlled environment (i.e., 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 greases L, M, and N are 15-25°C. To maintain their integrity, samples must be stored in a van or environment that is capable of maintaining the temperature within this range. The samples should also not be exposed to extreme temperatures during shipment.

Please email Al Gagnon (agagnon@whoi.edu) or 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.



Each cruise has two sampling kits containing most of the materials listed below (quantities may vary).

Typical Sampling Kit

Finn pipettes

Eppendorf pipette tips

“M” grease

Various lengths and diameter tygon and nylon tubing

Binder containing about deck log sheets and MSDS (for mercuric chloride)

Large rubber bands

Kim wipes

Gloves size S

Gloves size M

Gloves size L

Tyvek envelopes

Storage bag containing pens, pencils, lab tape, markers, and electrical tape

Roll duct tape

Zip ties

Tie down straps

Return shipping supplies (labels and plastic envelopes/shrink wrap/cardboard reinforcement for strapping/zip ties)

WHOI AMS ¹⁴C WATER SAMPLING DECK LOG

CRUISE 207N OPERATOR _____ LAT -8.3755
 STATION SIN-054 CAST 1 LON 52.6263
 DATE 05/13/2018 TIME _____ BOX# 31
 BOTTOMZ _____ WIRE ANGLE _____

POSITION #	NOM. PRES. OR DEPTH (M)	WHOI BOT. #	Hg ADD ✓	RECEIPT #	COMMENTS
9	3000	^{OTL} 4206	/		
10	2700	4207	/		
11	2400	4208	/		
12	2100	4209	/		
13	1800	4210	/		
14	1500	4211	/		
15	1200	4212	/		
16	1000	4213	/		
17	800	4214	/		
18	600	4215	/		
19	400	4216	/		
20	250	4217	/		
21	150	4218	/		
22	100	4219	/		
23	50	4220	/		
24	5	4221	/		

Other Comments:

