

Ozarks Environmental and Water Resources Institute (OEWRi)  
Missouri State University (MSU)

Standard Operating Procedure for:

# Preparation of Sample Bottles for Non-Metals Analyses

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December 6<sup>th</sup>, 2023



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### **Identification of the method**

Preparation of sample collection bottles for non-metals analyses including procedures for making acid bath solution and proper disposal of acid waste.

### **Scope of the method**

This standard operating procedure provides OEWRI laboratory personnel with guidance on the procedure for cleaning sample collection bottles that will be used for non-metals analyses such as total phosphorus (TP) and total nitrogen (TN), as well as oil and grease samples. This document includes instructions for making bottle wash solutions including Citranox and hydrochloric acid (HCl) bath for proper bottle washing.

### **Summary of method**

The sample bottles are emptied, rinsed with tap water, washed with a 2% solution of Citranox, rinsed with deionized water (DI), soaked in 5% hydrochloric acid overnight and rinsed with DI again. The bottles are allowed to drain dry and then stored in appropriately labeled plastic tub. Oil and grease sampling bottles are detergent washed and baked before use.

### **Definitions**

1. HDPE: High Density Polyethylene
2. MSDS: Material Safety Data Sheets
3. PPE: Personal Protective Equipment

### **Health and safety**

This procedure involves handling of acids and detergent solutions. Both of which can have irritating effects on laboratory workers. It is important that analysts read the MSDSs for these solutions. Further, it is recommended that analysts wear appropriate personal protective equipment such as gloves, goggles and an apron while working with the cleaning reagents. *When working with concentrated hydrochloric acid it can cause irritation to eyes, skin and lungs which make be severe or even fatal. Only use in a well-ventilated area while wearing splash goggles, face shield, gloves, lab coat, and apron.* In addition, care should be taken when using the oven for bottle cleaning.

### **Personnel qualifications**

Laboratory personnel shall have a working knowledge of these procedures and will have received training from an MSU employee knowledgeable of the proper bottle

cleaning procedures described in this SOP.

### **Equipment and supplies**

1. 1L or 500mL Nalgene®, HDPE, narrow-mouthed bottles: Fisher Scientific, catalog no. 03-313-5C and 03-313-5D)
2. Nalgene tub for 5% HCl acid bath
3. Laboratory glassware: pre-cleaned with 5% hydrochloric acid
4. Bottle brush: one that will fit inside of the bottles
5. Oven capable of maintaining temperature of 110-250°C for a minimum of 1 hour

### **Reagents and Standards**

1. Deionized water (DI)
2. Citranox® Liquid-Acid Detergent: (Fisher Scientific, catalog no. 50821312).
3. 2% Citranox Solution: Add 200mL of Citranox concentrate to a 10L carboy.
  - a. Slowly add DI to the 10 L mark. Foaming may occur if the DI is added too rapidly.
  - b. Mix to make a homogenous solution. This solution can be used for as long as it takes to use it up (i.e., there is no discard date).
4. Hydrochloric Acid, ACS grade: (Fisher Scientific, catalog no. A144C-212).  
*CAUTION: Corrosive. Wear personal protective equipment.*

### **Quality Control**

Bottles are tested for cleanliness through analyzing a blank test every other week or every 20 samples.

### **Procedure**

#### **A. Bottle Cleaning**

1. Empty the water sample from the bottle and rinse with tap water.
2. Fill the tub approximately half with DI water and half with Citranox solution.
3. Allow bottles to soak fully submerged in Citranox for 24 hours.
4. After the 24-hour soaking period, scrub inside and outside of bottles with a brush by the sink. Scrub lightly on plastic bottles as to not scratch them.
5. Triple rinse the bottles with DI water (fill halfway with DI, put cap on, shake, and pour out). Visually inspect the inside of the bottle for solid material, if it exists, re-rinse.

6. Submerge each bottle and cap in the bottle washing tub that contains 5% HCl acid solution. Allow to soak overnight (wait at least 12 hours before the next step).
7. Decant the acid solution back into the tub.
8. Triple rinse the bottles with DI water (as in Step 6).
9. Allow the bottles and caps to drain dry on paper towels. Change paper towels as needed.

**B. Preparing the Acid Bath**

The acid bath is made using stock solution of 37% hydrochloric acid (MSDS 7647-01-0)

$$\text{Dilution equation: } C_1V_1 = C_2V_2$$

The stock concentration of HCl is 37% (C1).

The final concentration desired is 5% HCl (C2).

The final volume of solution need is 2,000 mL (V2).

\*note: must be done in fume hood, which is why small batches are required\*

$$37\% * X = 5\% * 2000 \text{ mL}$$

This results in the (V1) of 270 mL. To reach a 5% HCl solution, measure out 1730 mL of DI water and 270 mL of stock HCl which gives 2,000 mL of solution. Repeat this until the desired volume is achieved, as described in the steps below:

1. Put on PPE (face shield, gloves, lab coat, and apron).
2. Gather 500 mL graduated cylinder, 2,000 mL Volumetric flask, beaker, funnel, stir bar, and stir plate.
3. Set up flask with stir bar and pour about 500 mL of DI water in.
4. Use graduated cylinder with funnel to measure out 270 mL of HCl.
5. Transfer funnel to flask and add HCl to flask.
6. Fill to 2,000 mL with DI.
7. Pour 2,000 mL solution into acid bath container
8. Repeat steps 3 through 7 until 30-60 L of solution is made.

### C. Bottle Washing for Oil and Grease Samples (Amber glass bottles)

1. Uncap bottles and wash both bottles and screw cap liners in detergent water followed by a tap water rinse.
  - i. Cap bottles with aluminum foil and bake at 200-250°C for a minimum of 1 hour prior to use. In place of baking, a solvent rinse may be used.
  - ii. Liners for screw caps should be baked at 110-200°C for a minimum of 1 hour prior to use.
2. This method is consistent with EPA Method 1664 cleaning procedures for amber glass bottles used to collect oil and grease samples (EPA 1999).

### **Pollution Prevention**

All wastes from these procedures shall be collected and disposed of according to existing waste policies within the MSU College of Natural and Applied Sciences.

### **Computer Hardware and Software**

Word: Microsoft Word was used to prepare this document.

### **Waste Management**

#### Neutralizing Expired Acid Bath

1. ***Acid wastes may NOT be disposed of in the sink until the pH has been neutralized.***
2. Put on PPE (long gloves and lab coat) and use pH probe to monitor acid bath during neutralization.
3. Neutralize expired acid bath with either NaOH (Sodium Hydroxide) or Baking Soda (Sodium Bicarbonate). Baking soda is the preferred method, as it will not overcorrect the pH.
4. If using baking soda, note that fizzing occurs so it must be done slowly to not overflow the tub.
  - a. If using NaOH, note that the pH can quickly rise to basic pH.
5. Dispose of neutralized pH down the sink drain. pH levels must be between 5.5 and 11 to dispose of in the drain.
6. Quantity, final pH, and date must be recorded for reference.
  - a. It is possible to contact MSU Environmental Services for disposal instead of neutralizing the acid.
  - b. Rinse acid bath container with water and ensure tub is free of visible dirty or broken glass.

## References

EPA. Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGTHEM; Non-polar Material) by Extraction and Gravimetry. United States Environmental Protection Agency. 1999.

ThermoFisher Scientific. MSDS sheet. CAS No 7647-01-0. 2023.

<https://www.fishersci.com/store/msds?partNumber=A144C212&productDescription=HYDROCHLORIC+ACID+2.5L+6%2FCS&vendorId=VN00033897&countryCode=US&language=en>

## Tables, Diagrams, and Flowcharts

1. See below for the corrosive warning label that should always be present on the acid bath and labeled with the concentration and type of acid contained.

# ACID BATH



**CORROSIVE**

Contains: \_\_\_\_\_