# PROCEDURE FOR HIGH LEVEL MERCURY GLASSWARE CLEANING

## 1. SCOPE AND APPLICATION

This SOP describes procedures for cleaning reusable glassware used in high level mercury digestions. It applies primarily to the cleaning of 75 mL Pyrex culture tubes used for digesting water samples and TCLP (Toxicity Characteristic Leaching Procedure) extracts, and glass bottles used for digesting solid phase (tissue and sediment) standard reference materials.

## 2. APPARATUS AND EQUIPMENT

2.1. Two large plastic buckets

2.2. Glassware brush

2.3. Large glass rod

## 3. REAGENTS AND CHEMICALS

**Note:** See DEP SOP MT-069 for standard and reagent documentation, labeling requirements, and expiration dates. All bottles of reagents and standards (purchased and prepared) in the laboratory are required to have laboratory information management system (LIMS) labels which includes a serial number.

3.1. Citranox detergent

3.2. Deionized (DI) water

3.3. Trace metal grade nitric acid ($HNO_3$) - The purchased acid is stored in the fume hood cabinet and the expiration date is provided by the manufacturer.

3.4. Trace metal grade hydrochloric acid (HCl) - The purchased acid is stored in the fume hood cabinet and the expiration date is provided by the manufacturer.

3.5. 9 % Acid Cleaning Solution:

3.5.1. Fill a plastic bucket with 9.1 L of DI water. Carefully add 300 mL of $HNO_3$ followed by 600 mL of HCl. Stir the solution with a glass rod while adding the acid. Allow the solution to cool to a comfortable temperature.

3.5.2. The 9 % acid cleaning solution is prepared as needed. Each time the solution is made, prepare a label and apply to the container with the following information:

- Name of the reagent
- Date prepared
- Date of expiration (See DEP SOP MT-069)
- Initials of preparer



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## 4. PROCEDURE

4.1. Properly dispose of any mercury waste left in the tubes/vessels into a mercury waste container. The City of Tallahassee sewer discharge limit for mercury is 38 µg/L. Remove all labels and rinse the glassware with hot tap water.

4.2. Soak the glassware and lids for at least 1 hour in a plastic bucket filled with hot tap water and 1 capful of Citranox, then, gently scrub with a glassware brush. Make sure to brush the outside of the lip on the tube/vessel also as well as the threads in the lid.

4.3. Rinse each tube, vessel, and cap 6 to 8 times with tap water and then soak in the 9 % acid cleaning solution. Let the containers and lids soak in the cleaning solution for at least an hour or until ready to use.

4.4. Remove the glassware and lids from the 9 % acid cleaning solution and rinse them with DI water and allow to dry. Once dry, cap the containers with the appropriate lid and store until needed.

## 5. SAFETY/HAZARDOUS WASTE MANAGEMENT

5.1. Review the Laboratory Health and Safety Manual located on the Division of Environmental Assessment and Restoration intranet site under Laboratory Quick Links: https://www.fldepnet.org/content/dear.

5.2. Safety glasses and lab coats must be worn at all times in the laboratory.

5.3. The wastes generated from the acid cleaning solution used to clean the glassware will contain low levels of mercury. However, the levels are well below the City of Tallahassee sewer discharge limit of 38 µg/L for mercury. The cleaning solution may therefore be disposed in a laboratory sink as long as the acid concentration is less than 2 % or diluted by 50 or greater.

5.4. Use caution with strong irritants such as acids and bases. Avoid breathing the fumes of these irritants by using them in a fume hood and keeping the face away from open containers of these chemicals. Avoid contact of these irritants with skin and clothing by appropriate use of personal protective equipment (PPE) such as gloves, lab coat, face-mask, hood shield, etc.

5.5. Store any wastes that cannot be disposed of down the sink in 6 L HDPE Nalgene containers properly labeled with hazardous waste labels in the fume hood. Make sure the bottle stays capped after use and place the bottle in the cabinet below the hood when it is no more than 90 % full. When two bottles reach 90 % full, appropriate personnel will dump them into the ‘Metals Waste’ hazardous waste drum located in C217, the Acid Waste Room. The key for C217 must be checked out from the KeyWatcher in room A311.


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## 6. REFERENCES

6.1. Laboratory Health and Safety Plan, located on the Division of Environmental Assessment and Restoration intranet site.

6.2. DEP SOP MT-069, Metals Standard and Reagent Preparation Procedure

6.3 City of Tallahassee Sewer Discharge Limits, https://www.talgov.com/Uploads/Public/Documents/you/learn/library/documents/sewer_use_manual.pdf

## Appendix of Significant Changes

06/11/2008 Minor revisions to the text were made for format and clarity.

06/10/2009 Clarified soaking time in section 4.2


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