PROCEDURE FOR CLEANING METALS LABWARE

1. SCOPE AND APPLICATION

This method describes the necessary steps to be taken to ensure labware is free of significant metals contamination. It is to be followed when cleaning microwave digestion vessels and liners, general laboratory glassware and hardware, SPLP (Synthetic Precipitation Leachate Procedure) and TCLP (Toxicity Characteristic Leachate Procedure) filtration apparatus, volumetric glassware, polypropylene funnels, Teflon beakers, watch glasses, spatulas, and mortars and pestles, etc. Before analysis, samples must be digested in meticulously cleaned vessels to minimize or eliminate the likelihood of contamination.

2. APPARATUS AND EQUIPMENT

2.1. Plastic buckets (not metal)

2.2. Scrubbing brushes

3. REAGENTS AND CHEMICALS

3.1. Deionized (DI) water

3.2. Citranox detergent

3.3. Trace Metal Grade Nitric Acid (HNO_3) - The expiration date is provided by the manufacturer. Stored in the fume hood cabinet at room temperature.

3.4. Trace Metal Grade Hydrochloric Acid (HCl) - The expiration date is provided by the manufacturer. Stored in the fume hood cabinet at room temperature.

4. PROCEDURE FOR CLEANING MICROWAVE DIGESTION VESSELS

4.1. The microwave vessels and caps must be cleaned after each digestion. In the fume hood, fill an appropriate container with 50 % HCl. Soak all vessels and caps overnight. The following morning, rinse each vessel and cap three times with DI water. Then, in the fume hood, fill an appropriate container with 50 % HNO_3. Soak all vessels and caps overnight. The following morning, rinse each vessel and cap three times with DI water. Allow the vessels and caps to dry by placing them upside down on a Kimwipe. As soon as they are dry, cap each vessel and store them on a laboratory shelf.

Alternative vessel and cap cleaning procedure - After the vessels have been used for the digestion of samples, rinse them three times with DI water and allow them to dry. Add 5 mL of concentrated HNO_3 to each vessel, cap, and place in the microwave. Run the microwave for 10 minutes at 650 W. Appropriately discard the HNO_3. Rinse each vessel and cap three times with DI water and allow them to dry upside down on a Kimwipe. As soon as the vessels are dry, cap, and place on a laboratory shelf.

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.

5. PROCEDURE FOR CLEANING TCLP AND SPLP FILTRATION APPARATUS

5.1. Fully disassemble the filtration apparatus including all O-rings and thoroughly rinse each piece with DI water.

5.2. Add about 1 L of DI water to a plastic bucket and add 5 capfuls of Citranox detergent. Thoroughly scrub each piece with a test tube brush and the diluted Citranox detergent solution. It is important to have several sizes of test tube brushes that are in good condition for proper cleaning.

5.3. Thoroughly rinse each piece of labware with DI water.

5.4. Thoroughly rinse each piece of labware with acetone and allow it to air dry.

5.5. Thoroughly rinse each piece of labware with hexane and allow it to air dry.

5.6. If there is potential for contamination, e.g., a high concentration sample has been processed prior to the cleaning, the filtration apparatus must be verified to be clean. Filter about 250 mL of the extraction buffer through a cleaned filtration apparatus with a filter in place and have the filtrate analyzed for contaminants of interest. If the filtrate is within acceptable limits, as determined by the metals group supervisor, then the filtration apparatus may be used as is without changing the filter used to test the cleanliness of the apparatus. However, if the filtrate is determined to be unacceptable by the metals group supervisor, then the apparatus must go through the cleaning procedure again.

5.7. In addition to scrubbing with Citranox solution, the filtration apparatus may be soaked in a 10 % HNO_3 solution (no longer than 30 minutes) and rinsed with DI water. See Section 7.1 for the acid discard procedure.

6. PROCEDURE FOR CLEANING VOLUMETRIC FLASKS

6.1. Properly dispose of any metals waste left in the flask into a metals waste container according to the procedure in Section 7.

6.2. Remove all labels. Writing made with a permanent marker may be removed by rubbing with a Kimwipe wetted with methanol.

6.3. Rinse once with DI water.

6.4. While working inside the fume hood, add 20 mL of HNO_3 and 10 mL of HCl to the empty 1 L volumetric flask. Cap the flask and invert several times to rinse the interior with the acid solution. Discard the concentrated acid (see Section 7.1). Less acid may be used to clean smaller flasks in a 2:1 ratio of HNO_3:HCl.

6.5. While continuing to work in the fume hood, use another clean container to slowly transfer DI water into the volumetric flask that was rinsed with nitric and hydrochloric acids until it is completely filled.

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.

Note: The addition of DI water to the volumetric flask containing concentrated nitric and hydrochloric acid rinsate will liberate irritating acid fumes. Care must be taken to protect the work environment from concentrated acid fumes.

6.6. Discard the dilute acid solution into the sink while flushing with a generous amount of tap water.

6.7. Rinse the volumetric flask several times with DI water.

6.8. Store the volumetric flask completely filled with DI water with the cap securely in place.

7. SAFETY/HAZARDOUS WASTE MANAGEMENT

7.1. Citranox should be disposed of down the sink with plenty of tap water. Acids should be properly diluted before discarding down the drain. An acid solution diluted to a 2 % acid concentration or diluted by 50 or greater will be neutralized by the laboratory's neutralization system prior to being released to the sewer.

7.2. Protective eyewear, lab coats, and gloves must be worn when working with acids and strong detergents.

7.3. Metals Waste Disposal:

All spikes, PQLs, and samples should be neutralized and discarded down the sink with plenty of water unless the sample has a metal concentration that exceeds the following limits established for sewer discharge:

As 190 µg/L	Pb 390 µg/L	Cu 1200 µg/L
Cd 140 µg/L	Hg 70 µg/L	Mo 830 µg/L
Cr 6000 µg/L	Ni 2600 µg/L	Zn 5600 µg/L
Se 1400 µg/L	Ag 4300 µg/L	

Samples with analyte concentrations above these limits shall be discarded in the appropriate hazardous waste container, NOT down the sink.

8. REFERENCES

8.1. DEP Health and Safety Manual, located on the DEAR intranet site.

8.2. City of Tallahassee Industrial Pretreatment Sewer Use Manual, located on the City of Tallahassee website, Talgov.com.

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.

Appendix of Significant Changes

01/15/2009	Section 1 - addition of general glassware and hardware.
01/11/2010	Section 4 - addition of a nitric acid rinse.
	Section 6 - detail added for clarification
	Section 7.2.1 - Updated the sewer discharge limits according to the City of Tallahassee Industrial Pretreatment Program Sewer Use Manual, 2020, section 21-356, Specific Pollutant Limitations. Located at Talgov.com
01/06/2011	Section 5- added HNO_3 rinse for TCLP/SPLP filtration vessels.
11/03/2022	Section 7.3 – Updated the sewer discharge limits according to the City of Tallahassee Industrial Pretreatment Sewer Use Manual, section 21-356 Specific Pollutant Limitations. Located at Talgov.com.
10/31/2023	Section 5 – added additional cleaning steps to include acetone and hexane rinses.
10/15/2026	Minor grammatical and syntactical corrections throughout the document. Updated the proper name of the DEP Health and Safety Manual in References.

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.