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TITLE: STANDARD OPERATING PROCEDURE FOR CLEANING LABWARE IN THE ACS INORGANIC CLASS 100/10,000 CLEAN LAB FACILITY

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CLEANING LABWARE IN THE ACS INORGANIC CLASS 100/10,000 CLEAN LAB FACILITY

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1.0 INTRODUCTION

This document describes the cleaning procedures necessary for labware used exclusively in the ACS Inorganic Class 100/10,000 Clean Lab Facility. The ACS Inorganic Class 100/10,000 Clean Lab Facility (Dreyfus 193, A-D) is supplied with separate, dedicated sets of labware for High Level Operations (> 1 ppm) and Low Level ultra-trace metal applications.

Cleaning procedures will be done by ACS technical support personnel or laboratory personnel within the ACS Inorganics Class 100/10,000 Clean Lab Facility (when possible), using the sinks and designated basins, deionized water lines and the acid-leaching baths.

2.0 SUMMARY OF PROCEDURE

A general, routine cleaning procedure for Clean Lab labware can be summarized in the following steps:

- separation of High Level and Low Level labware
- pre-treatment (removing labels, rinsing with tap water)
- detergent wash (soaking or scrubbing)
- thorough rinse (tap water and deionized water)
- acid leaching
- final rinse in deionized water
- drying (in Clean Lab hood)
- storage (if required)

The steps outlined above will be adequate for most Clean Lab applications. For some sensitive, non-routine applications, additional hot acid leaching may be required. All procedures are detailed below.

3.0 ROUTINE LABWARE CLEANING PROCEDURE

The routine labware cleaning procedure is applicable to most High Level or Low Level Operations. For some applications an alternate cleaning procedure may be used (i.e.

eliminate the detergent soak in order to reduce contamination risks from the detergent or employ other soaking agents).

3.1 Laboratory technical personnel will be responsible for preparing the labware for cleaning.

* Empty the dirty labware of any chemical residue and rinse well with tap water.
* Remove label tape or lab marker labelling (acetone or methanol is usually suitable for removing lab marker or tape residues from teflon, glass and most plastics.)
* Segregate labware as to High Level or Low Level applications.

3.2 ACS support personnel (dishwashers) or laboratory personnel will be responsible for washing and rinsing procedures.

* Prepare a solution of a suitable laboratory detergent (Alconox, Liquinox etc.) in the designated basin (Low Level or High Level) for soaking the labware.
* Submerge the labware, be careful to wet all surfaces.
* Soak for as long as necessary (to remove any organic residue).
* Rinse the labware with tap water until all detergent residue is gone.
* Rinse with deionized water three times.
* Place the labware onto the appropriate lab cart (High Level or Low Level Operations) for return to the lab.

3.3 Laboratory technical personnel are responsible for all acid leaching procedures.

* Submerge rinsed labware into an appropriate acid leaching bath set up in the acid bath cabinet, or in the Clean Lab hood. All surfaces (interior and exterior) must be exposed to the leaching solution.
* Soak labware for at least 8 hours.

For some Low Level ultra-trace applications, only the interior of the labware will be leached in order to prevent contamination from the printed labels on the exterior. This labware will be filled with the leaching solution and allowed to sit at room temperature for an appropriate length of time (2-4 hours). Alternately, the acid-filled labware will be soaked in a warm water bath.

CAUTION

(1) Laboratory personnel must take safety precautions when working with acid leaching baths: lab coat, safety glasses or face shield, Playtex-type latex gloves, sleeve covers, use of plastic tongs etc. Gloves should be used exclusively for acid-bath tasks.

(2) All laboratory surfaces exposed to acid solutions should be rinsed and if necessary neutralized.

3.5 Labware will be retrieved from the acid bath, drained of excess leaching solution, and rinse with copious deionized water (5-10 rinses.)

3.6 The labware will be dried on a drying rack or on Class-100 lab wipes on the lab bench or in the hood (if necessary).

3.7 Labware not used immediately will be stored in the acid baths until needed, or placed in plastic ziplock bags and stored in appropriate cabinets designated for High Level or Low Level Operations.

3.8 Volumetric Pipets

3.8.1 High Level Operations

High Level Operations include diluting calibration standards for use in the Instrument Lab, and diluting or transferring reagent solutions for sample preparation or analytical uses.

Volumetric pipets used in High Level Operations will be copiously rinsed with an appropriate rinse solution after use, then rinsed with at least 5 volumes of deionized water before being returned to the pipet acid bath designated for High Level Operations labware. Pipets should be cleaned with detergent (or alcoholic KOH) only when poor wetting and drainage characteristics compromise volumetric performance.

3.8.2 Low Level Operations

Pipets used for low level operations will be etched with an "L" to designate low level applications only. Low Level Operations include transferring or diluting sample preparations and dilute, decontaminated reagent solutions. Volumetric pipets used in Low Level Operations will be copiously rinsed with an appropriate rinse solution after use, then rinsed with at least 5

volumes of deionized water before being returned to the pipet acid bath designated for Low Level Operations labware. Pipets should be cleaned with detergent (or alcoholic KOH) only when poor wetting and drainage characteristics compromise volumetric performance.

3.9 Microwave Digestion Vessels

Microwave digestion vessels will be numbered and segregated according to the application and analyte. Between uses the vessels will be washed or soaked in detergent, rinsed and acid-extracted according to the needs of the project and analyte (see Sections 6.0)

4.0 MAINTENANCE OF ACID LEACHING BATHS

The Clean Lab acid baths will be prepared as 20% HNO_3 (reagent grade concentrated nitric acid) in deionized water. Separate acid baths will be prepared and designated for High Level Operations labware and Low Level Operations labware.

Contamination levels of the baths will be monitored by sampling the leaching solution from each bath for analysis. The acid leaching solutions will be sampled and analyzed when the baths are first prepared and once every month thereafter (see NHX/SOP-300-004 "Monitoring and Maintenance of the Cleanliness of the Clean Lab Facility"). The samples will be analyzed for cadmium, lead and arsenic (Cd, Pb, As) or for other specific analytes of interest.

The acid leaching baths will be changed when any of the analytes of interest reach a concentration > 1 ppm or in the event of known accidental contamination. If any analyte exceeds the 1 ppm threshold but is not currently being determined for a project, it is not necessary to change the acid bath until that analyte is being determined for a project.

To prevent accidental contamination, laboratory personnel will check that High Level and Low Level labware are kept separate and are leached in the designated baths. All labware will be rinsed with deionized water before submerging in the acid leaching baths.

Lab personnel will have Playtex (or similar) gloves specifically designated for use in the acid leaching baths. These gloves will be rinsed with deionized water before each use in the acid leaching baths. After use in the acid baths, the gloves will be rinsed copiously with

tap water and hung to dry. These gloves will not be used for handling samples, reagents or waste.

5.0 NON-ROUTINE CLEANING PROCEDURE

For sensitive Low Level applications (ultra-trace Pb, Hg etc.) an alternate hot acid leaching procedure may be used for Low Level labware.

5.1 A leaching bath will be prepared in a 1.0 or 4.0 L beaker dedicated for use as an ultra-trace level leaching bath. Usually a 20% HCL or 20% HCl + 20% HNO_3 solution is adequate, though specific methods may require other leaching treatments. Acids of trace-metal grade or better will be used to prepare the leaching solution. The minimum volume of leaching solution sufficient for the labware being treated will be prepared. The leaching bath will be kept in a Class 100 hood during preparation and during the leaching process.

5.2 Labware will be submerged into the ultra-trace leaching bath, taking care to expose all interior and exterior surfaces to the leaching solution. The bath will be heated on a hotplate for at least two hours.

> NOTE: Some plastics will discolor, soften or become brittle with exposure to hot, oxidizing acids. Lab personnel must make sure labware is compatible with hot acid leaching.

5.3 The ultra-trace leaching bath will be allowed to cool and the labware will be retrieved and rinse with copious volumes of deionized water.

5.4 The labware will be allowed to dry in the hood or on the benchtop on Class 100 lab wipes. The volumetric flasks will be stored with deionized water in them until use, other labware will be stored dry in plastic bags.

6.0 MICROWAVE DIGESTION VESSELS

6.1 Microwave vessels will be labelled with unique numbers and segregated for use with trace or ultra-trace level analyses. Databases will be maintained to keep records for the vessels, including sample types, acids and oxidants, acid-extraction and any contamination of analytes for each vessel.

6.2 For sensitive analyses, the high-pressure acid-extraction will be performed and the extract analyzed for analyte carry-over. (Specific acids, extraction times and programs, analytes and analysis methods will vary according to the needs of the project.)

An example of a general-use high-pressure extraction is as follows:

- add 10-mL of 10% HNO_3 to each detergent-cleaned microwave vessel, cap and seal
- microwave for a total 30 minutes at 100% power (alternating 3 minutes at 100% power, 2 minutes at 0% power.)
- cool and vent the vessel
- dilute the extract 1-to-2 with deionized water
- analyze for the analyte of interest by GFAA, FAA, HGAF, ICP etc.

For GFAA analyses, a signal of <0.002 absorbance units will acceptable for the extracts and the vessel will considered suitable for use. Suitability thresholds will be established for analyses as needed.

6.3 The extract solutions will be neutralized and discarded. The microwave vessels will be rinsed copiously with deionized water and dried in a Class 100 environment (benchtop or hood.) If not used immediately, the microwave vessels will be capped and stored in sealed plastic bags until needed.

6.4 Vessels that do not meet the suitability criterion must be re-extracted.

7.0 CONTAMINATION

If any Low Level Operations labware is inadvertently exposed to high levels of metals of interest (e.g. from highly contaminated samples, or high metals in a sample matrix) the labware will be removed from Low Level applications and replaced, or decontaminated by non-routine methods until its suitability for Low Level applications is verified.

8.0 CLEANING EXPENDABLE SUPPLIES

Accurate analysis of trace and ultra-trace metals requires minimizing all contamination sources, including disposable and expendable lab supplies. Pasteur pipets,

pipettor tips, disposable microbeakers, plastic weigh boats and other supplies with which samples or reagents will come into contact will be rinsed with deionized water and (if necessary) dried in Class 100 air.

For sensitive low level methods requiring non-routine cleaning of labware, these expendables will be soaked in or rinsed with dilute acid (approximately 10% HNO_3 or HCl) before the deionized water rinse.