

National Human Exposure Assessment Survey (NHEXAS)

Arizona Study

Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona
Tucson, Arizona 85721

Cooperative Agreement CR 821560

Standard Operating Procedure

SOP-BCO-L-10.0

Title: Procedure for Cleaning Glassware to be Used for Inorganic
Metals Analysis

Source: The University of Arizona

U.S. Environmental Protection Agency
Office of Research and Development
Human Exposure & Atmospheric Sciences Division
Human Exposure Research Branch

Notice: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), partially funded and collaborated in the research described here. This protocol is part of the Quality Systems Implementation Plan (QSIP) that was reviewed by the EPA and approved for use in this demonstration/scoping study. Mention of trade names or commercial products does not constitute endorsement or recommendation by EPA for use.

Procedure for Cleaning Glassware to be Used for Inorganic Metals Analysis

1.0 Purpose and Applicability

This standard operating procedure (SOP) describes the general procedures to be followed for cleaning glassware used in preparing and analyzing soil, house dust, air filter, surface wipe, or dermal wash samples for inorganic metals.

2.0 Definitions

- 2.1 Method Blank: all reagents (and a blank filter or wipe when appropriate) carried through the same digestion procedure as the samples, including filtration and any subsequent dilution.
- 2.2 Zero Standard: a solution acidified similarly to the digested samples and other calibration solutions. This solution is not spiked with any analytes, nor digested.

3.0 References

- 3.1 "Standard Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals," Standard E 50, American Society for Testing and Materials, Annual Book of ASTM Standards, Vol. 3.05, 1990.
- 3.2 "Standard Specification for Reagent Water," Standard D 1193, American Society for Testing and Materials, Annual Book of ASTM Standards, Vol. 11.01, 11.03, 1991.
- 3.3 "Standard Practice for Total Digestion of Sediment Samples for Chemical Analysis of Various Metals," Standard D 4698, American Society for Testing and Materials, Annual Book of ASTM Standards, Vol. 11.02, 1992.
- 3.4 "Standard Practice for Measurement of Metals in Workplace Atmosphere by Atomic Absorption Spectrophotometry," Standard D 4185, American Society for Testing and Materials, Annual Book of ASTM Standards, Vol. 11.03, 1990.

4.0 Discussion

This method is a nitric acid leaching procedure designed to remove all inorganic contaminants from glassware used for metals analyses. Preparation of both the detergent and acid baths is described.

5.0 Responsibilities

- 5.1 The inorganic sample preparation technician is responsible for the cleaning and subsequent storage of glassware to be used for any inorganic analytical procedures.
- 5.2 The inorganic sample preparation technician is responsible for the preparation and maintenance of both the detergent and acid baths, and recording pertinent information in the acid bath record book.

6.0 Materials and Equipment

6.1 Materials

- 6.1.1 Drying convection drying oven, ~5 ft³ capacity, 30-120 °C range.
- 6.1.2 Cotton or synthetic towels.
- 6.1.3 Alconox powder detergent.
- 6.1.4 Acid resistant Neoprene rubber gloves (elbow-length).
- 6.1.5 Rectangular tanks, heavy-duty Nalgene, 18" x 18" x 12", 11 gallon capacity, complete with cover.
- 6.1.6 Analytical balance, capable of weighing to 0.1 g.
- 6.1.7 Kimwipes (or equivalent).
- 6.1.8 Graduated cylinder, 1 L.
- 6.1.9 Fume hood.
- 6.1.10 Plastic-bristled brush.

6.2 Reagents

- 6.2.1 Concentrated nitric acid (HNO_3), reagent grade
- 6.2.2 ASTM Type II water (ASTM D 1193)
- 6.2.3 Methanol, GR

7.0 Procedures

7.1 Safety

- 7.1.1 Nitric acid fumes are noxious and highly corrosive - use care when pouring acid into water.
- 7.1.2 Avoid inhaling any particulate when pouring the Alconox into the detergent bath.
- 7.1.3 Wear safety glasses and a laboratory coat at all times.
- 7.1.4 Wear elbow-length acid-resistant Neoprene gloves when submerging into, or removing glassware from, the acid bath.

7.2 Preparation of Detergent Bath

- 7.2.1 Prepare a 7.5 g/L Alconox solution. Example:
 - 7.2.1.1 Using a graduated cylinder, add 20 L of Type II ASTM water to an 11-gallon Nalgene tank.
 - 7.2.1.2 Using a balance, weigh out 150 g of Alconox powder.
 - 7.2.1.3 With stirring, slowly add the Alconox powder to the water in the tub.
 - 7.2.1.4 Gently stir so as to avoid foaming, until all the Alconox is dissolved. Replace the cover.
- 7.2.2 Prepare a fresh detergent bath at least once every six months.

7.3 Preparation of Acid Bath

7.3.1 Prepare a 25% (v/v) HNO₃ solution. Example:

7.3.1.1 Using a graduated cylinder, add 5 L of Type II ASTM water to a 11-gallon Nalgene tank.

7.3.1.2 Move tub inside a fume hood.

7.3.1.3 Carefully add 5 L of concentrated nitric acid to the tank.

7.3.1.4 After fuming has stopped, relocate tub to the glassware cleaning station.

7.3.1.5 Using a graduated cylinder, carefully add 10 L of Type II ASTM water to the tank. Cover the tank with the shipped top and label the tank, indicating acid content and strength.

7.3.2 Prepare a fresh acid bath at least once every six months.

7.4 Cleaning of Laboratory Glassware

7.4.1 Remove any tape from the glassware. Remove any markings using methanol and a Kimwipe.

7.4.2 Rinse glassware with deionized water at least three times.

7.4.3 Place any glassware with visibly dirty residue on it in the detergent bath. Otherwise skip to Step 7.4.4.

7.4.3.1 Scrub residue with plastic-bristle brush.

7.4.3.2 Rinse glassware three times with Type II ASTM water.

7.4.4 Submerge glassware in the acid bath overnight to leach out any inorganic impurities.

7.4.5 Remove glassware from acid bath and rinse three times with Type II ASTM water.

7.4.6 Place the glassware upside down on a clean surface, cover it with a clean cotton or synthetic towel to prevent contamination, and allow to air dry.

7.4.7 Glassware may be dried in a convection drying oven at 90 °C. Care should be taken to avoid contamination from the metal surfaces in the oven.

7.4.8 Once the glassware is dry (and cool, if dried in an oven), place stoppers in the glassware, if appropriate, and then use for analysis or store at room temperature. Store beakers upside down.

7.5 Quality Control

7.5.1 The acid bath must be re-prepared if anything metallic, e.g., a spatula, is dropped into it.

7.5.2 If zero standards and/or method blank results have uncharacteristically high metals concentrations, an aliquot will be taken from the acid bath and analyzed. If the results show that the acid bath is contaminated, the bath must be re-prepared.

8.0 Records

8.1 Records of glassware detergent and acid bath maintenance will be recorded in the acid bath record book.

8.2 Electrical resistivity (megohms-cm, 25°C) of all Type II water stations will be recorded with daily use in the deionised water stations.