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Standard Operating Procedure **NHX/SOP-300-003**

Title: Purification of Reagents in the ACS Inorganic Clean Lab Facility for Trace/Ultratrace Metal Analysis

Source: Research Triangle Institute

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TITLE: STANDARD OPERATING PROCEDURE FOR PURIFICATION OF REAGENTS IN THE ACS INORGANIC CLEAN LAB FACILITY FOR TRACE/ULTRATRACE METAL ANALYSIS

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PURIFICATION OF REAGENTS IN THE ACS INORGANIC CLEAN LAB FACILITY FOR TRACE/ULTRATRACE METAL ANALYSIS

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1.0 SCOPE AND APPLICATION

Analysis of trace element concentrations at and below 1 ng/mL (ppb) is often required. Contamination is the major problem in getting high quality data at these levels. Contamination in trace and ultratrace analysis is understood as the increase in the measured amount or concentration of a component, resulting from its introduction at various stages of the analytical procedure from sources other than the sample. Several independent sources, besides the sample itself, add to the final signal for a particular analyte. These are the laboratory atmosphere and working areas, tools and apparatus associated with sampling, sample preparation, laboratory ware, and reagents. Compared to other sources of contamination, contribution from reagents can often be measured quantitatively and can also be reduced effectively. Purification of reagents as a means to prevent contamination from reagents is discussed here along with the purification procedures.

A list of most commonly used reagents in trace/ultratrace metal analysis is given in Table 1. These include water, acids, bases, buffers, oxidants, reductants and other reagent chemicals. The demand for high purity reagents has been met by commercial chemical manufacturers in some cases. However for many others, specific purification procedures need to be developed to adequately control the contamination level.

2.0 SUMMARY

The procedures given here are used to purify the reagents that are not available from the manufacturer at the required purity. These procedures are tested in the clean room environment for their performance in producing high purity reagents and will be modified as necessary.

3.0 INTERFERENCES

3.1 Extreme care must be taken to avoid the contamination during the purification process and also not to re-contaminate the purified reagents. The primary sources of contamination are particulates in air, impurities in reagents that are used to purify the component of

interest, trace elements from containers, sample handling by analysts, etc. With the use of Class 100 clean room environment and by following proper procedures for sample handling, it may be possible to keep the contaminants at a level below the instrument detection limit.

3.2 Storage vessels, storage conditions (temperature, humidity, etc.), and storage locations must be chosen appropriately to maintain the quality of the purified reagents.

4.0 SAFETY

Since the toxicity of the chemicals used in these procedures is not clearly defined, they should be treated as potential health hazards at all times and personal exposure to these chemicals should be minimized.

5.0 EQUIPMENT

5.1 Graphite Furnace Atomic Absorption Spectrometer (GFAA)

Perkin-Elmer 5100 atomic absorption spectrometer with a transversely heated graphite furnace and Zeeman background correction is used to analyze the purified reagents to established their purity. The procedures for the operation of PE 5100ZL are given in NHX/SOP-171-005.

5.2 Hydride Generation Atomic Fluorescence Spectrometer (HGAF)

PS Analytical hydride generation atomic fluorescence spectrometer (HGAF) is used to analyze the purified reagents for As, and thus establish their purity. The procedure for the operation of the HGAF is given in NHX/SOP-300-001.

6.0 STORAGE

Once purified, the purity of the reagent is critically dependent upon the storage conditions. The duration of storage, container material, storage location and temperature are particularly important.

Purified reagents are stored in thoroughly cleaned vessels (Sections 7.1.2.1 and 7.2.2.1). Prior to transfer of the purified reagent, the vessel must be rinsed with the reagent being stored. Storage vessels must be clearly labelled with the reagent name, concentration, date prepared and other relevant information. If the reagent requires specific storage conditions, it will be stored accordingly, otherwise it is placed inside an airtight polyethylene bag and stored in metal free cabinet in the Clean Lab.

7.0 REAGENT PURIFICATION PROCEDURES

7.1 Ammonium Dihydrogen Phosphate (Ammonium Phosphate, Monobasic) - $NH_4H_2PO_4$

7.1.1 Reagents

All reagents must be of recognized analytical grade, unless specified otherwise.

- Ammonium dihydrogen phosphate - 1.667 g
- Ammonium hydroxide - about 100 mL
- Chelex-100 chelating ion exchange resin
- Ethylenediaminetetraacetic acid diammonium salt (EDTA) - 0.556 g
- Deionized water, 18 MΩ quality.

7.1.2 Equipments

7.1.2.1 Laboratory Ware

All labware (glassware/plasticware) must be thoroughly cleaned by washing with detergent and water followed by rinsing with deionized water. Then they are acid leached in warm 20% nitric acid for at least 12 hrs, drained, thoroughly rinsed with deionized water and are dried under HEPA filtered air. The storage bottles that will be used to store the purified reagent must be cleaned by warming in 50% HNO_3 for 12-24 hrs followed by rinsing with deionized water several times.

- 50 mL Teflon or polypropylene beakers (2)
- 600 mL Teflon or polypropylene beaker
- 500 mL Teflon or polypropylene storage bottle
- Plastic column

7.1.2.2 Apparatus

- Analytical balance with a weighing capacity of 205 g and resolution of 0.1 mg.

7.1.3 Procedure

- Weigh out 1.667 g of ammonium dihydrogen phosphate into a 600 mL Teflon or polypropylene beaker and add sufficient deionized water to dissolve the material.
- Add 5.0 mL of 14 M ammonium hydroxide solution to the beaker containing ammonium dihydrogen phosphate and dilute to 500 mL with deionized water.
- Prepare a column of Chelex-100 and pass a solution of ammonium hydroxide through the column to convert the chelating resin to ammonium form.
- Pass the ammonium dihydrogen phosphate solution through the column at a flow rate of 0.5 mL/min.
- Add 0.556 g of ethylenediaminetetraacetic acid diammonium salt to the eluent and dilute to 500 mL with deionized water. Store the solution in an acid leached storage bottle. The concentration of $NH_4H_2PO_4$ in the final solution is 0.03 M.

7.2 Magnesium Nitrate - $Mg(NO_3)_2$

7.2.1 Reagents

All reagents must be of recognized analytical grade, unless specified otherwise.

- Magnesium nitrate - 4 g
- Ammonium pyrrolidinedithiocarbamate (APDC) - 0.25 g
- Nitric acid - several drops
- Methyl isobutyl ketone (MIBK) - 750 mL
- Deionized water

7.2.2 Equipments

7.2.2.1 Laboratory Ware

All labware (glassware/plasticware) must be thoroughly cleaned by washing with detergent and water followed by rinsing with deionized water. Then they are acid leached in warm 20% nitric acid for at least 12 hrs, drained, rinsed thoroughly with deionized water and are dried under HEPA filtered air.

The storage bottles that will be used to store the purified reagent must be cleaned by warming in 50% HNO_3 for 12-24 hrs followed by rinsing with deionized water several times.

- 1 L Teflon or polypropylene volumetric flask
- 1 L Teflon or polypropylene beaker
- 2 L Teflon or polypropylene separatory funnel
- 100 mL graduated cylinder (glass or plastic)
- 1 L Teflon or polypropylene storage bottle

7.2.2.2 Apparatus

- Analytical balance with a weighing capacity of 205 g and a resolution of 0.1 mg.
- pH meter

7.2.3 Procedure

- Dissolve 4 g of $Mg(NO_3)_2$ in 1 L of deionized water in 1 L volumetric flask.
- Transfer the solution to 1 L beaker and add 0.25 g of APDC.
- Adjust the pH of the solution to 5.8 with HNO_3.
- Transfer one half of the solution to a 1 L separatory funnel and add 125 mL of MIBK.
- Extract the metal complexes with MIBK.
- Repeat the extraction 2 more times with a fresh portion of MIBK each time.
- Repeat the extraction procedure for the second half of the solution.
- Combine the aqueous phases and boil for 20 mins to remove any MIBK.
- Store in a clean Teflon or polypropylene storage bottle.

8.0 REFERENCES

1. Purification of Matrix Modifiers Used in GFAA, Fishman, et al., J. Assoc. of Anal. Chem., 69, 706 (1986).

2. Purification of Analytical Reagents, Mitchell, J.W., Talanta, 29, 993 (1982).

3. Purified Reagents for Trace Metal Analysis, Moody, J.R. and Beary, E.S., Talanta, 29, 1003 (1982).

4. Purification of Analytical Reagents and Other Liquids by Low Temperature Vacuum Sublimation, Mitchell, J.W., Anal. Chem., 50, 194 (1978).

TABLE 1. REAGENTS THAT ARE COMMONLY USED IN TRACE/ULTRATRACE METAL ANALYSIS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY AND HYDRIDE GENERATION ATOMIC FLUORESCENCE SPECTROMETRY

Acids and Bases	Reagents for As and Hg Analysis
Nitric acid	Potassium iodide
Sulfuric acid	Potassium bromide
Hydrochloric acid	Sodium borohydride
Perchloric acid	Potassium bromate
Sodium hydroxide	Tin (II) chloride

Matrix Modifiers for GFAA	General Purpose Reagents
Palladium	Hydrogen peroxide
Magnesium nitrate	
Ammonium dihydrogen phosphate	
Diammonium hydrogen phosphate	
Hydroxylamine hydrochloride	
Nickel nitrate	
Triton X-100	
Ascorbic acid	