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Standard Preparation Procedure

Access to this SOP shall be available within the laboratory for reference purposes; the official copy of this SOP resides on the official Georgia EPD website at https://epd.georgia.gov/about-us/epd-laboratory-operations. Printed copies of this SOP will contain a watermark indicating the copy is an uncontrolled copy.

1 Scope and Application

This SOP outlines the proper procedure utilized for the preparation of standards for the determination of total recoverable metals in groundwater, surface waters, drinking waters, wastewaters, soils, sludge, and sediments.

1.2 Restricted Procedure

This procedure is restricted to use by an analyst experienced in the handling of hazardous materials. Additionally, the analyst must complete the requirements of the GaEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GaEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

2 Definitions

2.1 Refer to Chapter 3 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control Definitions.

3 Interferences

3.1 Contamination is the prime concern. The work area (including counters and hoods) is cleaned weekly to eliminate sources of contamination. All reagents and apparatus must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

3.1.1 Glassware must be scrupulously cleaned. Any glassware used must be triple washed in reagent grade 1:1 nitric acid followed by a triple rinse in 18 MΩ water. After washing, all glassware is dried in a clean environment then covered with parafilm and stored in a closed cabinet until used. Virgin plastic ware (HDPE) is used and discarded after use.

3.1.2 The use of high purity reagents helps to minimize interference problems. Sub-

boiled acids are used in the preparation of samples for ICP-MS analysis; reagent grade acids are used for all other analysis.

4 Safety

4.1 Refer to Laboratory Chemical Hygiene Plan, Revision 1 February 28, 1999.

5 Apparatus and Equipment

5.1 Appropriately sized virgin, certified, precleaned HDPE sample bottle with screw caps.

5.2 Various size pipetters capable of delivering volumes ranging from 1.0 to 5000 µL and an assortment of high quality pipette tips.

5.3 Top loading balance capable of measuring to 0.1 mg.

5.4 Pasteur pipettes.

6 Reagents and Standards

6.1 Concentrated nitric acid (sp. gr. 1.41), reagent grade.

6.2 Concentrated nitric acid, sub-boiled distilled (for ICP-MS preps).

6.3 Concentrated hydrochloric acid, (sp. gr. 1.19), reagent grade (for ICP-OES analysis).

6.4 Concentrated hydrochloric acid, sub-boiled distilled (for ICP-MS preps).

6.5 18 MΩ water.

6.8 Appropriate standard stock solution for preparing all calibration standards including matrix spikes and laboratory control samples.

7 Sample Collection

Not applicable.

8 Calibration

8.1 Calibration Curve

Not applicable.

8.2 Calibration Verification

Not applicable

9 Quality Control - Refer to analytical method

10 Procedure

10.1 Gravimetric (must be used to prepare all ICPMS standards)

10.1.1 This method of standard preparation is based on the density of the standards being 1g/ml. Therefore, adding one gram of standard is the same as adding one ml.

10.1.1 Choose an appropriate size new HDPE bottle, place on balance and tare the weight.

10.1.2 Transfer approximately ¾ of the final weight required for standard preparation using previously prepared acidified water solution. For example, if you were preparing 100mls of standard, you would transfer 75g of solution to the bottle.

10.1.3 Add the appropriate weight of stock standard, using a volumetric pipette, to the acid solution. For example, if you were adding 120 µl of standard, you would add 120 mg instead.

10.1.4 Fill to volume with the acidified water solution until appropriate weight is reached. Note that final weight and final volume are equivalent measurements based upon the density of the stock standard being 1g/mL (example 100mL equals 100gms).

10.1.5 Log in all standards made in the Standard Logbook. Write the corresponding standard ID number, expiration date, concentration, and initials of analyst on the container used.

10.2 Volumetric (may be used for all standard prep except ICPMS)

10.2.1 Select the appropriate size volumetric flask. Triple acid rinse with 1:1 nitric acid followed by a triple rinse with 18MΩ water.

10.2.2 Transfer approximately ¾ of the final volume required for standard preparation using previously prepared acidified water solution.

10.2.3 Add the appropriate amount of stock standard, using a volumetric pipette, to the acid solution.

10.2.4 Fill with the acidified water solution until the final volume is reached. This is accomplished by visually aligning the bottom of the solution meniscus with the inscribed line on the flask.

10.2.5 Log in all standards made in the Standard Logbook. Write the corresponding standard ID number, expiration date, concentration, and initials of analyst on the container used.

11 Evaluation of the Linearity of the Initial Calibration
Not applicable.

12 References
Not applicable

13 Practical Quantitation Limits (PQLs), Precision and Accuracy Criteria, and Quality Control Approach
Not applicable