MERCURY GROUP STANDARDS PREPARATION PROCEDURES

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the preparation of intermediate standards, including certified standard reference materials (SRMs), used for the atomic fluorescence analysis of total mercury in water samples. This SOP is applicable to the preparation of standards for the following tests: W-HG-AF, W-HG-AF-F, and SPLP-HG-AF.

2. APPARATUS AND EQUIPMENT

2.1 Polystyrene weigh boats

2.2 Balance

2.3 Class A Glass Volumetric Flasks: 1.0 L, 500 mL, 250 mL, 200 mL, 100 mL, 50 mL.

2.4 Pipettes:

2.4.1 50 µL, 100 µL, 250 µL, 1000 µL Eppendorf pipettes

2.4.2 Bottle-top dispenser

2.4.3 1-5 mL and 5-10 mL Finn pipettes

3. REAGENTS AND CHEMICALS

Note: Reagents used for preparation of standards must be logged in to the LIMS standard prep tracker to generate a serial number. The label printed from the LIMS standard prep tracker must be placed on the bottle of reagent in the laboratory. See DEP SOP **MT-069** for instruction on reagent preparation documentation and expiration dates. Templates for all forms can be found on the Division of Environmental Assessment and Restoration (DEAR) internet site. Use the MT-HGINT-STD to record reagent information while preparing intermediate standards. The form is created in Microsoft Excel and then printed and included with the laboratory report for each analysis:

3.1 Trace Metal Grade (TMG) Nitric Acid (HNO_3) - The purchased acid is stored in the fume hood cabinet and the expiration date is provided by the manufacturer.

3.2 Trace Metal Grade (TMG) Hydrochloric Acid (HCl) - The purchased acid is stored in the fume hood cabinet and the expiration date is provided by the manufacturer.

3.3 Potassium Bromate ($KBrO_3$, 0.05M)/Potassium Bromide (KBr, 0.1M) Solution – Add 8.4 g of potassium bromate ($KBrO_3$) and 11.9 g of potassium bromide (KBr) to an acid cleaned 1 L Teflon bottle. Dilute and dissolve both salts with DI water to 1 L. The solution is prepared as needed and is stored at room temperature. The expiration date for this reagent is 1 year from preparation. The solution is purged with nitrogen for at least two hours before use to remove traces of mercury. If contamination is suspected, bake $KBrO_3$ at 175 °C overnight in a glass

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scintillation vial in a mercury free oven to remove all traces of Hg in the salt. Do the same with KBr in a clean glass vial. Allow the salts to cool prior to solution preparation.

4. INORGANIC STANDARDS FOR TRACE-LEVEL TOTAL MERCURY ANALYSIS

Note: Preparation of all Hg standards must be documented in the LIMS standard prep tracker module. A label must be printed from the standard prep tracker and placed on the bottle in the laboratory.

4.1 Primary Standard (1000 mg/L) – The primary standard consists of a 1000 mg/L NIST traceable standard in 10 % HNO_3 purchased from a vendor such as SPEX Certiprep, EM Science, or Accustandard. Calibration standards, matrix spikes, laboratory fortified blanks (LFBs), and practical quantitation standards (PQLs) are prepared from this source. The expiration date for this purchased standard is assigned by the manufacturer and it is stored at room temperature. Save a pdf of the certificate of analysis in the 'Certificates of Analysis for Reagents and Standards' folder located on the network, \\FLDEP1\DEAR\Labs\Lab5\InorganicPrep\Certificates of Analysis for Reagents and Standards\Standards and Standard Reference Materials.

4.2 **THG-5** (10 mg/L) - This is an intermediate standard prepared by diluting 1 mL of the purchased primary standard (see section 4.1) to 100 mL in a class A glass volumetric flask. Half fill a clean 100 mL class A volumetric flask with DI water. Add 5 mL HNO_3, 4 mL HCl, and 3 mL of the bromide/bromate solution. Pipette 1 mL of the purchased primary standard into the flask. Fill the flask up to the mark with DI water. Cap and shake well. The expiration date for this prepared standard is one year from preparation and it is stored at room temperature.

4.3 **THG-6** (200 µg/L) - This is an intermediate standard prepared by diluting 2 mL of the **THG-5** intermediate standard to 100 mL in a class A glass volumetric flask. Half fill a clean 100 mL class A volumetric flask with DI water. Add 5 mL HNO_3, 4 mL HCl, and 3 mL of the bromide/bromate solution. Pipette 2 mL of the **THG-5** intermediate standard into the flask. Fill the flask up to the mark with DI water. Cap and shake well. The expiration date for this prepared solution is one year from preparation and it is stored at room temperature.

4.4 **THG-7** (1.0 µg/L) - This is an intermediate standard prepared by diluting 1.25 mL of the **THG-6** intermediate standard to 250 mL in a class A glass volumetric flask. Half fill a clean 250 mL class A volumetric flask with DI water. Add 12.5 mL HNO_3, 10 mL HCl, and 7.5 mL of the bromide/bromate solution to it. Pipette 1.25 mL of the **THG-6** intermediate standard to the flask. Fill the flask up to the mark with DI water. Cap and shake well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.5 **NIST1641D** SRM (1.557 mg/L) - The SRM consists of a 1.557 ± 0.020 mg/kg NIST SRM in water, preserved in 2 % HNO_3 and purchased from NIST. The

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expiration date is assigned by the manufacturer and it is stored at room temperature.

4.6 **NIST1641D-1** SRM (15.57 μg/L) - Pipette 10 mL of 1.557 ± 0.020 mg/kg NIST1641D SRM into a 1 L class A volumetric flask half filled with DI water. Add 50 mL HNO_3, 40 mL of HCl, and 30 mL of the bromide/bromate solution. Bring the volume up to the mark and mix well. The expiration date for this prepared standard is one year from preparation and it is stored at room temperature.

5. SAFETY/HAZARDOUS WASTE MANAGEMENT

5.1 Review the Laboratory Health and Safety Plan located on the Division of Environmental Assessment and Restoration internet page.

5.2 All standards should be handled as if they contain hazardous levels of analyte. **Always** wear proper protective eye wear and lab coats while in the lab. Gloves should be worn while handling any standard. Excess inorganic Hg standard should be disposed of in the 6 L HDPE Nalgene containers labeled Metals Waste. When 90 % full, these must be emptied into the "Metals Waste' waste drum in room C217 by appropriate personnel. The key for C217 must be checked out from the KeyWatcher in room A311. Refer to DEP SOP **HG-013** for additional details.

5.3 Organic mercury solutions contain methylmercury chloride and are especially toxic to humans. Special care should be taken to ensure that these solutions do not come into contact with skin. These standards contain organic solvents (isopropyl alcohol or methanol) which facilitate transport of methylmercury through the skin. Refer to DEP SOP **HG-013** for additional details on the disposal of organic wastes. Once the properly labeled organic solvent + methyl mercury waste container is 90 % full in the laboratory, the container should be placed on the shelves of the organic waste room (C218) located on the laboratory loading dock.

5.4 Aqueous methyl mercury waste with concentrations < 10 μg/L must be first oxidized to less toxic total mercury before disposal in the sink. Aqueous methyl mercury waste with concentrations above 10 μg/L should be stored separately and once the container is 90 % full, place the properly labeled container on a shelf in the inorganic waste room, C217 See DEP SOP **HG-013** for additional details.

5.5 All non-hazardous wastewater can be dumped into the sink if the acid concentration is less than 2 % or diluted by 50 or greater.

6. REFERENCES

6.1 DEP SOP **HG-013**, Mercury Waste Disposal

6.2 DEP SOP **MT-069**, Metals Standard and Reagent Preparation Procedure

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6.3 Health and Safety Plan, located on the Division of Environmental Assessment and Restoration web page.

Appendix of Significant Changes

07/15/2008	Minor revisions to the text were made for format and clarity.
07/15/2009	Updated Apparatus and Equipment section.
07/07/2010	Updated Section 8, Safety/Hazardous Waste Management
08/04/2014	The potassium bromate / potassium bromide solution was removed from the intermediate standards used for the water matrix.
08/03/2018	Removed high level Hg standard preparation sections. The CVAA (Cold Vapor Atomic Absorption Spectroscopy) instrument was decommissioned and replaced by TDA-AAS (Thermal Decomposition and Amalgamation Atomic Absorption Spectroscopy).
08/06/2019	Removed information for reagents and standards used for methylmercury standards.

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