PROCEDURE FOR PREPARING FILTERS FOR METALS ANALYSIS

1. SCOPE AND APPLICATION

This SOP describes the preparation procedure used to digest EPA filter strips or other similar samples in preparation for total recoverable metals analysis. It is meant to supplement the normal metals soil/sediment/waste digestion procedure described in DEP SOP MT-060 and is a modification of EPA method 3050B. This SOP is used for samples with test IDs SA-ICP and SA-ICPMS.

2. SUMMARY OF THE METHOD

Filter strips for metals analysis from EPA are generally submitted in narrow elongated strips. This SOP addresses these filters; however, modifications can be made for the digestion of larger filters or portions of them and these modifications will be noted throughout the SOP.

The filter strip is rolled up with a clean pair of forceps and placed in a 50 mL polypropylene digestion vessel. The filter is then subjected to a series of steps that include the addition of nitric and hydrochloric acids, hydrogen peroxide, and heating. Larger filters can be cut into small pieces (e.g. 1 cm^2) if either the filter is too large or if the entire filter is not going to be digested. The filter sample is then digested, reconstituted to a 50 mL volume with DI water, and delivered to the metals laboratory for analysis.

3. EQUIPMENT AND SUPPLIES

Refer to DEP SOP MT-060 for equipment and supplies information.

4. REAGENTS AND STANDARDS

Refer to DEP SOP MT-060 for reagents and standards information and DEP SOP MT-069 for documentation and labeling of all reagents and standards.

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1. Samples are stored in the DEP Central Laboratory's Receiving Room at room temperature or in one of the refrigerators. See DEP SOP LB-016 for details.

5.2. All glassware should be cleaned according to DEP SOP MT-007.

6. SAMPLE PREPARATION PROCEDURE

The metals soil/sediment/waste digestion procedure in DEP SOP MT-060 is to be employed with the following notes:

6.1. EPA filter strips are reported in µg/filter strip, therefore the weight of the filter is not needed if it is digested in its entirety. For other filters it is always beneficial to determine both the weight and dimensions of the filter, or subsection, since the filter can be reported in a variety of units (e.g., µg/filter strip, µg/cm^3, µg/cm^2 or

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mg/kg). Record all information in the comment section of the LIMS Prep Worksheet Manager.

6.2. Cut the bag lengthwise and carefully insert a clean pair of forceps gripping one end of the filter strip. Begin to roll the filter strip around the end of the forceps. As the sample begins to roll up, crimp the sample every turn by pinching the sample through the bag. This will prevent the sample from unraveling when removing it from the bag. Once the sample has been completely rolled up, swab the inside of the bag with the sample, still attached to the forceps, to collect any particulates or shavings of the filter that remain in the bag. It is important to collect all of the particulates so the total concentration of metal can be accurately determined. Once this is complete, slowly remove the sample from the bag and transfer it to the digestion vessel.

Larger filters can be cut into small pieces for digestion. If the filter is too large to be used in its entirety, an even fraction (e.g., ½, ¼) should be used for simplicity. If a fraction of the filter is used, be sure to measure the dimensions of the filter before cutting it and measure a suitable fraction of the filter for digestion, also noting the dimensions of the subsection. If the filter is to be cut into pieces, cut them small enough so they will fit adequately into the digestion vessel (e.g. 1 cm^2).

6.3. Proceed with the normal digestion procedure as directed in DEP SOP MT-060. When adding the acid to the digestion vessel, make sure the sample is completely covered in acid or the metal content in the filter may not be completely extracted.

6.4. Once the digestion procedure is complete, reconstitute with DI water to the 50 mL volume. Cap the tubes and gently invert 8-10 times to mix the sample completely. The samples are now ready for analysis. Deliver the samples and a copy of the preparation sheet to the metals lab.

7. SAMPLE ANALYSIS PROCEDURE

Samples prepared in this manner are suitable for analysis by inductively coupled plasma atomic emission spectroscopy (ICP-AES) or inductively coupled plasma mass spectrometry (ICP-MS). See appropriate SOP for analysis, DEP SOP MT-061 (ICP-AES), DEP SOP MT-067 and DEP SOP MT-074 (ICP-MS).

8. CALCULATIONS

The instrument will report the result with the units µg/L. Results are typically reported in µg (when the entire filter strip is digested). To convert the µg/L result to µg, use the following equation:

$$X * V = y$$

where:

X = concentration of analyte in digestate (µg/L).
V = the final preparation volume (L). (Usually this would be 0.05 L.)
y = the amount of analyte on the portion of filter digested (µg).

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9. QUALITY CONTROL

9.1. Digestion Blank - One per digestion batch of 20 samples or less. Add ~0.5 g of Environmental Express resin pellets to the digestion blank.

9.2. Duplicate Lab Fortified Blanks (LFBs) - A set of duplicate LFBs per batch for the purpose of generating precision data. See DEP SOP MT-060 for spike standards and spike volumes.

9.3. Matrix Spikes - Matrix spikes and sample replicates are not possible with these filters since the entire portion is digested in one vessel. Larger filters that only have a subsection analyzed can be run in duplicate for precision data and if enough of the filter is present, matrix spiking can be performed if desired. See DEP SOP MT-060 for spike standards and spike volumes.

9.4. Any QC associated with filters (waste matrix) must not be applied to any other sediment, soil or waste sample.

9.5. The method performance acceptance criteria for standard reference materials, matrix spikes, internal standards, and precision data is listed in DEP SOP-MT-058. This SOP also contains information for acceptance criteria for quality control measures, corrective actions for out-of-control data, and contingencies for handling out-of-control data.

10. SAFETY/HAZARDOUS WASTE MANAGEMENT

10.1. Review the Laboratory Health and Safety Plan.

10.2. Use **CAUTION** with strong irritants such as acids and bases. Avoid breathing the fumes of these irritants by using them in a hood when possible and keeping the face away from open containers of these chemicals. Avoid contact of these irritants with skin and clothing by appropriate use of gloves, lab coat, face–mask, hood shield, etc. Safety glasses and lab coats must be worn at all times in the laboratory.

10.3. Store any wastes that cannot be disposed of down the sink (see reference 11.12) in a 6 L HDPE Nalgene bottle (properly labeled with **Hazardous Wastes** labels) in the fume hood. Make sure the bottle stays capped after use and place the bottle in the cabinet below the hood in the metals lab when it is NO MORE THAN 90 % FULL. When two bottles reach 90 % full, dump them immediately into the 'Metals Waste' hazardous waste drum located in C217, Acid Waste Room. The key for C217 must be checked out from the KeyWatcher in room A311.

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

11. REFERENCES

11.1. DEP SOP LB-016, Standard Operating Procedure for Sample Receipt and Entry into the LIMS

11.2. DEP SOP MT-007, Procedure for Cleaning Metals Labware

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11.3. DEP SOP MT-058, Procedure for Metals Data Review and Authorization

11.4. DEP SOP MT-060, Hot Block Digestion of Soil, Sediment, Waste, and Tissue Samples for Total Recoverable Metals

11.5. DEP SOP MT-061, Analysis of Selected Metals by Dual View Inductively Coupled Plasma-Atomic Emission Spectroscopy, (ICP-AES)

11.6. DEP SOP MT-067, Standard Operating Procedure for the Inductively Coupled Plasma Mass Spectrometer (ICP-MS) with Collision Cell Technology (CCT) and Kinetic Energy Discrimination (KED)

11.7. DEP SOP MT-069, Metals Standard and Reagent Preparation Procedure

11.8 DEP SOP MT-074, Standard Operating Procedure for the Thermo ICAP Inductively Coupled Plasma Mass Spectrometer (ICP-MS)

11.9. EPA Method 3050B, revision 2, December 1996

11.10 Quality Manual for State of Florida DEP Laboratory

11.11. Laboratory Health and Safety Plan

11.12. City of Tallahassee Industrial Pretreatment Sewer Use Manual, section 21-356, Specific Pollutant Limitations, located on the City of Tallahassee website, Talgov.com, http://www.talgov.com/Uploads/Public/Documents/you/learn/library/documents/sewer_use_manual.pdf.

Appendix of Significant Changes

05/27/2009 Formatting changes were made to reflect the suggested organization given in DEP SOP LB-001-1.1.

05/27/2010 Updated references to SOPs and the Bureau of Laboratories Health and Safety Plan.

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