

Standard Operating Procedure for:

Preparation of Sample Bottles
For Metals Analyses
(0100R01 Bottle Prep Metals.doc)

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and

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Resources Institute (OEWRi)

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Revision History

Revision number	Revision date	Revisions made (Indicate the section changed and what changes were made)
1	9/13/2006	First version of this SOP

1 Identification of the method

Preparation of sample collection bottles for metals analyses.

2 Scope of the method

This standard operating procedure provides Missouri State University (MSU) laboratory personnel with guidance on the procedure for cleaning sample collection bottles that will be used for metals analyses.

3 Summary of method

The sample bottles are emptied, rinsed with tap water, washed with a 2% solution of Citranox, rinsed with deionized water (DI), soaked in 5% nitric acid overnight and rinsed with DI again. The bottles are allowed to drain dry and then are stored in sealed plastic bags until use. Two bottles from each batch of washed bottles are checked for metals residue using the ICP procedure (see SOP: 5010R01 ICP.doc).

4 Definitions

HDPE: High Density Polyethylene
MSDS: Material Safety Data Sheets

5 Health and safety

This procedure involves handling of acids and detergent solutions. Both of these can have irritating affects for laboratory workers. It is important that analysts read the MSDSs for these solutions. Further, it is recommended that analysts wear appropriate personal protective equipment such as, gloves, goggles and an apron while working with the cleaning reagents.

6 Personnel qualifications

Laboratory personnel shall have a working knowledge of these procedures and will have received training from an MSU employee knowledgeable of the proper bottle cleaning procedures described in this SOP.

7 Equipment, supplies and chemicals

Equipment and Supplies

500mL Nalgene®, HDPE, narrow-mouthed bottles: Fisher Scientific, catalog no. 03-313-5C). Label these bottles and caps with an "M" to distinguish them from other sample bottles.

20 Gallon Drum: Eagle Manufacturing Company, model no. 1654.

Laboratory glassware: pre-cleaned with 1 +1 nitric acid.

Sealable Plastic Bags or Boxes: for storing the cleaned bottles.

Bottle brush: one that will fit inside of the bottles

Chemicals and Reagents

Deionized water (DI)

Citranox® Liquid-Acid Detergent: (Fisher Scientific, catalog no. 50821312).

2% Citranox Solution: Add 200mL of Citranox concentrate to a 10L carboy. Slowly add DI to the 10L mark. Foaming may occur if the DI is added too rapidly. Mix to make a homogenous solution. This solution can be used for as long as it takes to use it up (i.e., there is no discard date).

Nitric Acid, ACS grade: (Fisher Scientific, catalog no. A200C-212). CAUTION: Corrosive. Wear personal protective equipment.

5% Nitric acid solution: Add 1.5L of concentrated HNO₃ to 30L of DI in a 20 gallon yellow drum. Mix. Caution: solution will be hot after the addition of acid to water. Allow to cool before use. This solution can be used for up to 2 months.

8 Procedure

Cleaning

Empty the water sample from the bottle

Rinse the bottle with tap water (half full with tap water, put cap on, shake, and pour out – repeat for a total of three rinses).

Add approximately 10ml of 2% Citranox solution to the bottle.

Put the cap on the bottle and shake vigorously to expose the entire inside of the bottle to the Citranox solution.

Remove the cap.

Pour out the detergent solution.

Rinse with 3 aliquots of DI (half full with DI, put cap on, shake, and pour out). Visually inspect the inside of the bottle for solid material, if it exists, re-rinse.

Submerge each bottle and cap in the bottle washing drum that contains 5% HNO₃ acid solution. Allow to soak overnight (wait at least 12 hours before the next step).

Decant the acid solution back into the drum*.

Rinse the bottles with 3 aliquots of DI (as in step 8.7).

Allow the bottles and caps to drain dry on a non-metallic rack.

Store the bottles, with the caps on, in a sealed plastic bag or box.

Label the storage bag (or box) with a batch number (for example: use the date that the bottles were cleaned followed by a number indicating how many bags (or boxes) were filled. 090106-1/3 would be recorded if this was the first of three bags filled with cleaned sample bottles.

*Note: the acid solution will be tested by the cleanliness of the bottles. If the bottles show no metals residual, as tested by the ICP, then the acid solution will be considered “clean” enough for this procedure. The solution will not be re-used beyond two months after its preparation.

Quality Assurance

Select two bottles from a cleaned batch of sample bottles

Fill the bottles with DI

Add 2 mL of nitric acid (trace metals grade) to preserve to a pH of ≤2 units.

Analyze the samples on the ICP according to the procedures outlined in SOP: 5010R01 ICP.

Record the results on the QA log form (see below)

Compare the results with the established DL values.

If the test result values from both bottles are greater than twice the DL, then the batch of bottles is not acceptable for use. The acid wash solution should be re-made and the bottles re-washed.

The QA log form will be stored in a binder in the OEWR office.

9 Computer hardware and software

Microsoft Word: this document is prepared using Word.

The Word document file name for this SOP is: 0100R01 Bottle Prep Metals.doc.

10 References

Standard Methods for the Examination of Water and Waste Water. Method 3010 C., APHA, 21st Edition, 2005.

5010R01 ICP. 2006. OEWRI SOP. Operation of the ICP for metals analyses.

11 Tables, diagrams and flowcharts

None

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 Metals Sample Bottle Wash QA Check

		Results							
		Date Tested							
		Batch ID							
Analyte	Detection Limit	1	2	1	2	1	2	1	2
As									
Ca									
Cd									
Cu									
Mg									
Na									
Ni									
Pb									
Zn									
Acceptable (Y or N):									

Comments:

Note: bottles are acceptable if the results for both bottles are $\leq 2 \times$ DL for all analytes.
