

Standard Operating Procedure for:
Preparation of Sample Bottles
For non-Metals Analyses
(0150R01 Bottle Prep non-Metals.doc)

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and
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Resources Institute (OEWRi)

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Table of Contents

Revision History	2
1 Identification of the method	3
2 Scope of the method	3
3 Summary of method	3
4 Definitions	3
5 Health and safety	3
6 Personnel qualifications	3
7 Equipment, supplies and chemicals	3
Equipment and Supplies	3
Chemicals and Reagents	3
8 Procedure	4
Cleaning	4
Quality Assurance	4
9 Computer hardware and software	5
10 References	5
11 Tables, diagrams and flowcharts	5
Bench Sheet	6

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 non-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 non-metals analyses such as total phosphorus (TP) and total nitrogen (TN).

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% hydrochloric 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 TP and TN residues using the nutrient analysis SOPs (3010R02 Total P.doc and 3020R03 Total N.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).

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

Laboratory glassware: pre-cleaned with 5% hydrochloric 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).

Hydrochloric Acid, ACS grade: (Fisher Scientific, catalog no. A144C-212). CAUTION: Corrosive. Wear personal protective equipment.

5% Hydrochloric acid solution: Add 1.5L of concentrated HCl 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 Citronox 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% HCl 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 TP or TN residual, as tested by the Lachat FIA, 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 HCl (ACS grade) to preserve to a pH of ≤ 2 units.

Analyze the samples for nutrients according to the procedures outlined in the nutrient SOPs: 3010R02 Total P.doc and 3020R03 Total N.doc.

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: 0150R01 Bottle Prep non-Metals.doc.

10 References

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

3010R02 Total P. 2006. OEWR SOP. Total Phosphorus.

3020R03 Total N. 2006. OEWR SOP. Total Nitrogen.

11 Tables, diagrams and flowcharts

None

Missouri State University
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 Non-metals Sample Bottle Wash QA Check

		Results							
		Date Tested							
		Batch ID							
Analyte	Detection Limit	1	2	1	2	1	2	1	2
Total P									
Total N									
Acceptable (Y or N):									

Comments:

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