General
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Cleaning of Sample Containers

1 INTRODUCTION

Contamination of a sample from its container is a potentially serious problem, especially in trace-level analysis. Sample containers are frequently reused and in these cases it is extremely important that all traces of the previous sample be removed to avoid carry-over. Cleaning must be tailored to the type of container, and to the analysis which is to be performed on the sample. This procedure outlines cleaning methods for most typical analyses. The analytical method used should also be consulted to determine if there are any special requirements for sample container cleaning.

2 SCOPE AND APPLICATION

2.1 OVERVIEW

2.1.1 Water-soluble substances are removed by soaking or scrubbing with a 2% phosphate-free laboratory detergent solution. The detergent is removed by rinsing with warm tap water. Acid-soluble residue is removed from plastic bottles by soaking or rinsing with a 10% (v/v) hydrochloric acid solution followed by a tap water rinse. Sample containers are then rinsed at least three times with distilled deionized water and allowed to dry. Glass containers which are used for trace level organics are combusted at 550° C to destroy any remaining contaminants.

3 REQUIRED TRAINING

3.1.1 07_02_11 Laboratory Waste Disposal and Management

4 EQUIPMENT AND MATERIALS

4.1 APPARATUS AND MATERIALS

4.1.1 Brushes, scrubbing pads, wipes, disposable towels.
4.1.2 Rubber gloves, eye protection, apron or lab coat.
4.1.3 Plastic wash bottles.
4.1.4 Soaking tub.
4.1.5 Duncan Automatic Kiln (Model EA 820-A).
4.1.6 Orton Pyramid Cones (#022) [obtained from Glasers Ceramics].

4.1.7 Aluminum Foil
4.1.8 Self-Adhesive Labels

4.2 REAGENTS

4.2.1 Non-phosphate Laboratory Detergent (RBS-pF – Pierce Chemical, MICRO – Scientific Products, FL-70 Fisher Scientific, or equivalent).
4.2.2 Hydrochloric Acid, concentrated (12M).
4.2.3 Distilled, deionized water (DDW).
4.2.4 Acetone, Methanol, or Ethanol (Technical Grade).

5 SAFETY PRECAUTIONS

5.1 SAFETY PRECAUTIONS

5.1.1 Wear appropriate eye and hand protection when working with laboratory detergents and hazardous materials.
5.1.2 When operating the kiln, wear gloves and take precautions to prevent burns from hot surfaces and cuts from cracked or broken bottles.

5.2 REAGENT DISPOSAL

5.2.1 ALL acid solutions must be neutralized before disposal down the drain in Room 205. Do not allow any un-neutralized acid solution to drain into any sink in the building.
5.2.2 Collect used organic solvents, including acetone, in containers for disposal by the University Hazardous Materials Program (HMP). Label container with the solvents collected, the approximate amounts, and composition (e.g. 200 mL 50% methanol/water). Do not allow any organic solvent to enter the sewer system.
5.2.3 Check the Sewer Disposal List in Room 203 for a list of substances that are allowed to be disposed of down the sanitary sewer.

6 SOLUTIONS AND REAGENTS

6.1 NON-PHOSPHATE LABORATORY DETERGENT WORKING SOLUTION

Detergent Concentrate	20 mL
Tap Water	1 L

Protocol: Shake concentrate to thoroughly mix, and then dilute concentrate to 2% (v/v), [i.e. ~20 mL/L] working solution in hot (50° C) tap water. Prepare fresh solution at least monthly for detergent bath, and more often if needed.

Storage: Detergent concentrate is kept under the sink in Room 203. Working solution is stored in the three baths in Room 203, and one bath beside the acid sink in Room 205.
Disposal: Used working detergent solution can be disposed of down any sink in the lab.

6.2 HYDROCHLORIC ACID WORKING SOLUTION

Hydrochloric Acid (12M)	36.458 g/mol	100 mL
Distilled Water		1 L

Protocol: Dilute hydrochloric acid to 10% (v/v) (i.e. ~100 mL/L] working solution in distilled water.
Storage: Hydrochloric acid is kept in the acid lab (Room 205). Working solution is kept in the large bath beside the acid sink in Room 205.
Disposal: The working solution must be neutralized with sodium bicarbonate before being disposed of down the acid sink in Room 205.

7 STANDARD SOLUTIONS

8 PROTOCOL

8.1 INITIAL CLEANING AND RINSE

8.1.1 Remove all markings, tape, etc. from container prior to cleaning. Scrape off thick deposits, dirt, adhesive, etc. with knife, scoop, or razor blade. Use a wipe or towel soaked in acetone or ethanol to remove ink.

8.1.2 Empty contents from all bottles and flush with tap water. Remove any sediment or other visible material from the bottom. Discard the container if there is sediment, stains, or other foreign matter which cannot be removed at this time.

8.1.3 Detergent washing can be accomplished by scrubbing or soaking. Smaller bottles (50-250 mL) generally are easiest to clean by soaking, while larger bottles (up to 4 liters) require scrubbing or shaking. Bottle caps are generally easiest to clean by soaking. Completely immerse smaller bottles in working (2%) detergent solution. Remove air bubbles to ensure total contact between surfaces and solution. Hot soaking solutions require less contact time. In general, most containers can be cleaned by soaking for 1 to 24 hours. Soaking solutions may be reused several times. Larger bottles may be cleaned by applying detergent solution from a squeeze bottle, and using a brush or shaking vigorously. Containers which are scrubbed or otherwise agitated do not require lengthy soaking times.

8.1.4 Rinse immediately and thoroughly after removal from detergent solution using warm tap water. Proceed immediately to acid rinsing (8.2) for plastic bottles which are not to be used for chloride determinations. Proceed to distilled deionized water rinse (8.3) for most glass bottles.

8.2 ACID RINSING

8.2.1 The acid rinse step is to be used for removing acid-soluble contaminants. It should not be used for containers which will be used for chloride determinations. Skip to distilled-deionized water rinse (8.3) for bottles to be used for chloride samples. Rinse plastic bottles with a 10% (v/v) hydrochloric acid solution immediately after thoroughly rinsing off detergent solution with warm tap water. Use a plastic wash bottle for applying acid, and be sure to coat the entire interior. Allow acid to remain in the bottle for a few minutes. The acid solution may then be transferred to other bottles for reuse 3 or 4 times before discarding. Alternatively, bottles may be soaked in a 10% (v/v) hydrochloric acid bath for 10-20 minutes. Bottle caps may be soaked separately. Rinse acid thoroughly from bottle using warm tap water and proceed immediately to distilled-deionized water rinse (8.3). Collect used acid in a container for disposal. Acid must be neutralized with sodium bicarbonate before disposal in acid sink (Room 205). Do not allow ANY un-neutralized acid solutions to drain into any sink in the building! It is most convenient and safest to perform acid rinsing in the acid sink only.

8.3 DISTILLED DEIONIZED WATER (DDW) RINSE

8.3.1 Rinse containers and lids with DDW at least 4 times immediately after a tap water rinse. It is not necessary to completely fill container with DDW, rather fill to ~10% capacity, shake, and empty. Use a large wash bottle for rapid rinsing.

8.4 DRYING

8.4.1 Clean bottles and lids can be air dried by inverting on a rack or other clean surface so that water may drain and air will circulate. If bottles are not placed in a rack, return to upright position after excess water has drained to speed drying. Be sure that containers do not become contaminated with dust or other air-borne materials. Do not leave bottles in drying area indefinitely. Attach appropriate self-adhesive labels, place in a bag or box marked with bottle size, and return to the appropriate storage location.

8.5 BOTTLE STORAGE

8.5.1 All laboratory users are responsible for returning cleaned and dried sample containers to the proper storage location. The cleaning process is not complete until the items are returned. Bottles should not be placed in storage until properly cleaned. If the containers were cleaned for a particular purpose, it should be noted and stored separately from other containers.

8.6 COMBUSTING GLASS BOTTLES

8.6.1 All glass bottles used for trace organics and dissolved organic carbon determinations must be combusted to destroy any remaining contaminants from

container walls.

8.6.2 Check controls from top to bottom:
- White Firing Switch is set to “OVERGLAZE.”
- Safety timer to “OFF”, Kiln Sitter Switch is down.
- Black Manual/Auto Toggle Switch is set to “Manual Low.”

8.6.3 Place a small #022 Orton pyramid type cone or bar in the Kiln Sitter trigger switch while holding the trigger up. Position the cone or bar so that the narrow end barely rests on one of the cone supports to permit the earliest shutoff of the kiln.

8.6.4 Load the kiln compartment with cleaned and dried glass sample bottles. For 1-Liter and smaller bottles, use the shelf and posts supplied to combust two levels of bottles. Make sure that the shelf clears the lower level of bottles by at least 1 inch, and that the shelf is not in contact with either the thermocouple at the rear of the compartment or the kiln sitter switch near the front.

8.6.5 Close kiln lid and set safety timer to 3.5 hours by turning the knob clockwise past 10 hours and returning to 3.5 hours.

8.6.6 Flip up the kiln sitter switch and make sure it is locked into the upright position. Activate the kiln by pushing in the white plunger inside the opening in the kiln sitter switch. Check temperature periodically and, if possible, record the shutoff time. Allow bottles to cool before attempting to remove from kiln as rapid cooling can weaken and crack soft glass.

8.6.7 Remove combusted bottles from kiln, place a fresh square of aluminum foil over the bottle mouth, and screw on clean cap. Attach appropriate self-adhesive label and arrange bottles in boxes with suitable dividers.

9 DATA REDUCTION AND STATISTICS

10 QUALITY ASSURANCE

11 ADDITIONAL INFORMATION

11.1 REFERENCES

11.1.1 APHA (1992) Standard Methods for the Examination of Water and Wastewater (18th Edition), American Public Health Association.

11.1.2 ACS (1988) Principles of Environmental Sampling, L.H. Keith (ed.), American Chemical Society.

11.1.3 U.S. EPA (1979) Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA-600/4-79-019).

12 PREVIOUS ISSUES AND CHANGES

Document File Name	Issue	Issue Effective Dates	Author
Gen-Bottles-001	001	September, 1994 - ???	Unknown
Gen-Bottles-002	002	??? – October 17th, 2006	Unknown
Gen-Bottles-003	003	October 18th, 2006 – May 11th, 2015	Unknown
Sample Bottle Cleaning-004	004	May 11th, 2015 – April 12th, 2018	Monica Hollrah
07_02_03.005 Sample Bottle Cleaning	005	April 12th, 2018 - Present	Victoria Wickham

12.1 ISSUE CHANGES

12.1.1 Issue 001:
- Unknown

12.1.2 Issue 002:
- Unknown

12.1.3 Issue 003:
- Unknown

12.1.4 Issue 004:
- Reviewed document and updated filename

12.1.5 Issue 005:
- Moved SOP over to new format
- Updated the acid rinsing step to include alternative soaking instructions
- Changed waste disposal to include acid neutralization

13 DIAGRAMS, FIGURES, AND PHOTOGRAPHS