

Laboratory Glassware Cleaning and Storage

1 INTRODUCTION

The proper care and cleaning of laboratory glassware is fundamental to nearly all analytical procedures. The methods discussed here pertain to most commonly used laboratory glassware and routine laboratory analysis. The actual cleaning procedures used should be tailored to the substances that are to be removed, the determinations to be made, and to the type of glassware.

2 SCOPE AND APPLICATION

2.1 OVERVIEW

It is generally good practice to rinse glassware with tap water as soon as possible after use. Material which is allowed to dry on glassware surfaces is much more difficult to remove. Water-soluble substances are typically 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 by soaking or rinsing with a 10% or 25% (v/v) hydrochloric acid solution followed by a tap water rinse. The glassware is then rinsed at least three times with distilled deionized water. Special cleaning procedures for volumetric glassware, glassware used in trace element work, and difficult to clean glassware are also included.

3 REQUIRED TRAINING

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 Pipette storage jar and pipette washer
- 4.1.5 Soaking tub

4.2 REAGENTS

- 4.2.1 Phosphate-Free Laboratory Detergent (Citronox, RBS-pF – Pierce Chemical, MICRO – Scientific Products, FL-70 – Fisher Scientific, or equivalent).
- 4.2.2 Acid Laboratory Detergent, pH = 2.5 (Citronox, or equivalent).
- 4.2.3 Hydrochloric acid, concentrated (12M).
- 4.2.4 Nitric acid, concentrated (16M).
- 4.2.5 Sulfuric acid, concentrated (36N).
- 4.2.6 Nochromix (Dichromate cleaning substitute).
- 4.2.7 Dichromate cleaning solution (if Nochromix is unavailable).
- 4.2.8 Distilled deionized water (DDW).
- 4.2.9 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 chemicals. Chromic acid (or Nochromix) is especially hazardous, and should be used only when nothing else will remove the contaminants. Spills of this solution must be cleaned up immediately. Neutralize and contain acid spill with sodium chromate, sodium bicarbonate, calcium carbonate, or equivalent base.

5.2 WASTE DISPOSAL

- 5.2.1 ALL acid solutions must be neutralized with sodium bicarbonate before being disposed of in the sink in Room 205. Do not allow any un-neutralized acid solution to drain into any sink in the building.
- 5.2.2 Used organic solvents, including acetone, must be disposed of into containers for disposal by the University Hazardous Materials Program (HMP). Do not allow any organic solvent to enter the sewer system.
- 5.2.3 Check the Sewer Disposal List in Room 203 for the list of substances that can be disposed of down the sanitary sewer system.

6 SOLUTIONS AND REAGENTS

6.1 PHOSPHATE-FREE LABORATORY DETERGENT WORKING SOLUTION - BATHS

Laboratory Detergent	20 mL
Tap Water	1 L

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

Storage: Laboratory detergent is kept under dish-washing sink in Room 203. Prepared working solution is kept in the baths beside the sink in Room 203, and in the small tub beside the acid sink in Room 205.

Disposal: Used working solution can be disposed of in any sink in the laboratory.

6.2 PHOSPHATE-FREE LABORATORY DETERGENT WORKING SOLUTION – PUMP BOTTLE

Laboratory Detergent	80 mL
Tap Water	Fill to Line

Protocol: Unscrew pump from the top of the pump bottle. Shake concentrate to thoroughly mix and then measure 80 mL of Citronox. Slowly fill the bottle to the line with tap water. Filling the bottle too quickly will create unnecessary foaming of the Citronox. Once filled, the pump itself can be screwed back onto the bottle.

Storage: Laboratory detergent is kept under dish-washing sink in Room 203. Prepared working solution is kept in the pump bottle beside the sink in Room 203.

Disposal: Used working solution can be disposed of in any sink in the laboratory.

6.3 ACID LABORATORY DETERGENT WORKING SOLUTION

Citronox	20 mL
Tap Water	1 L

Protocol: Shake concentrate to thoroughly mix and dilute concentrate to 2% (v/v), [~20 mL/L] working solution in hot (50° C) tap water. Prepare fresh solution bi-weekly for pipette bath and detergent bath, or more often if needed.

Storage: Acid laboratory detergent is kept under the dish-washing sink in Room 203. Prepared working solution is kept in the baths beside the sink in Room 203, in the small tub beside the acid sink in Room 205, and in the pipette storage jar in Room 205.

Disposal: Used working solution can be disposed of in any sink in the laboratory.

6.4 HYDROCHLORIC ACID WORKING SOLUTION

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

Protocol: Dilute hydrochloric acid to a 10% (v/v) working solutions in distilled water. Prepare fresh solution as necessary.

Storage: Hydrochloric acid is stored in the acid lab (Room 205). Prepared working solution is stored in the large bath beside the acid sink in Room 205.

Disposal: Working solution must be neutralized with sodium bicarbonate before being disposed of in the acid sink in Room 205.

6.5 NITRIC ACID WORKING SOLUTION

Nitric Acid (16M)	63.012 g/mol	200 mL
Distilled Water		1 L

Protocol: Dilute to 20% working solution in distilled water. Prepare fresh solution as necessary.

Storage: Nitric acid is stored in the acid lab (Room 205). Prepared working solution is stored in the perchloric acid hood in Room 205.

Disposal: Working solution should be neutralized with sodium bicarbonate before being disposed of in the acid sink in Room 205.

6.6 NOCHROMIX SOLUTION

Nochromix		1 or 2 packages
Sulfuric Acid (36N)	98.072 g/mol	2.5 L

Protocol: Prepare Nochromix following package instructions by adding one or two packages to one 2.5 L bottle of concentrated sulfuric acid (36N). Use within 1 week. Nochromix should be used only when nothing else will remove the contaminants.

Storage: Prepared working solution is stored in the acid lab (Room 205).

Disposal: Neutralize working solution with sodium bicarbonate before disposing of in the acid sink in Room 205. Follow it with copious amounts of tap water.

6.7 DICHROMATE CLEANING SOLUTION (IF NOCHROMIX IS UNAVAILABLE)

Sodium Dichromate	261.965	50 gm
DDI Water		25 mL
Sulfuric Acid	98.072 g/mol	Enough to bring to 1L volume

Protocol: Dichromate is prepared by dissolving 50 gm of sodium dichromate in 25 mL of water. Cautiously add 36N sulfuric acid drops one at a time until no further reaction is

observed. Bring volume up to 1 L using sulfuric acid. Dichromate should be used only when nothing else will remove the contaminants.

Storage: Prepared working solution is stored in the acid lab (Room 205).

Disposal: Collect in separate container for disposal by the University Hazardous Materials Program.

7 STANDARD SOLUTIONS

8 PROTOCOL

8.1 GENERAL CLEANING

- 8.1.1 It is generally good practice to rinse glassware with tap water immediately after use, even if it will be properly cleaned at a later time. Remove all markings, tape, etc. from glassware 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 Completely immerse object in working (2%) detergent solution. Remove air bubbles to ensure total contact between surfaces and solution. Larger objects may be cleaned by applying detergent solution from a squeeze bottle and using a brush. An alternative method of cleaning larger objects is to use 1-2 pumps of working solution from the pump bottle next to the sink in Room 203, filling the large object with tap water, and allowing to soak. Soaking times vary depending on the contaminants to be removed. Hot soaking solutions require less contact time. In general, most glassware can be cleaned by soaking for 1 to 24 hours. Avoid longer soaking times, as it may cause etching, removal of painted markings, and formation of detergent residues.
- 8.1.3 Rinse object immediately and thoroughly after removal from detergent solution using warm tap water. Proceed to acid rinsing only if necessary, and if glassware is not to be used for chloride determination. For chloride determination, glassware can be cleaned using only the detergent soaking followed by deionized water rinses. The only pieces of glassware that should be routinely acid rinsed are the 125 mL Erlenmeyer flasks. For all other glassware, proceed directly to the distilled deionized water rinse (8.3).

8.2 ACID RINSE

- 8.2.1 The acid rinse step is to be used for removing acid-soluble contaminants from glassware. It should not be used for glassware that will be used for chloride determinations. If in doubt, however, it is best to employ the acid rinse as a precaution.
- 8.2.2 Rinse or soak glassware with a 10% (v/v) hydrochloric acid solution immediately after rinsing detergent solution off with warm tap water. Soak glassware in acid bath for 20 minutes. Be sure all traces of detergent are thoroughly removed prior to using acid. Use a plastic wash bottle for applying acid, and collect used acid in a container for disposal. Do not allow acid solutions to drain into any sink in the building. It is most convenient and safest to perform acid rinsing in or near the acid sink (Rm 205) only.
- 8.2.3 For the glassware to be used in trace metal determinations, use a 20% (v/v) nitric acid solution for rinsing. Keep this glassware separate, however, and do not use glassware cleaned with nitric acid for any other purposes.

8.2.4 Rinse acid from glassware using warm tap water.

8.3 DISTILLED DEIONIZED WATER (DDW) RINSE

8.3.1 Final rinsing is accomplished with distilled deionized water. Rinse glassware with DDW at least 4 times immediately after the tap water rinse. Use a large wash bottle for rapid rinsing. Clean glassware is indicated when water drains uniformly in a thin film from the surface. Water droplets indicate glassware is not completely cleaned and must be rewashed. It is not necessary to completely fill container with DDW, rather fill to ~10% capacity, shake, and empty.

8.4 DRYING

8.4.1 Clean glassware can be air-dried by inverting on a rack or other clean surface so that water may drain and air will circulate. Be sure that air-dried glassware does not become contaminated with dust or other air-borne materials. Return clean and dried glassware to the appropriate storage location. Do not leave dried glassware in a drying area indefinitely.

8.5 VOLUMETRIC GLASSWARE (FLASKS AND PIPETTES)

- 8.5.1 As a general rule, use volumetric flasks only for preparing standards and transferring solutions to a glass or polyethylene bottle for storage. Volumetric flasks can be cleaned using the detergent washing and distilled deionized water rinsing procedures described above. The inside of the flasks can be soaked by putting 1-2 pumps of working solution in first with the pump bottle next to the sink in Room 203 before filling with tap water. Follow soaking times as if you were soaking the flask inside a Citronox bath. Vigorous shaking of the detergent solution should aid in cleaning the inside of the flask. Acid rinses may be used if the solutions prepared are likely to resist detergent cleaning. Do not allow alkaline solutions to be stored for long periods of time in volumetric flasks as they may become damaged. After cleaning a volumetric flask, fill with deionized water before returning it to storage. Discard water before using flask again.
- 8.5.2 Rinse volumetric pipettes immediately after use and place in a pipette storage jar filled with 2% Citronox acid cleaning solution. When full, the basket of the storage jar is transferred to a pipette washer/rinser. Cycle warm tap water through the pipette washer approximately 10-12 times. After the detergent has been completely rinsed from the washer, drain tap water from the washer and fill with distilled water. Raise and lower the basket several times to cause agitation and flush out the inside of the pipettes.

- 8.5.3 Allow pipettes to soak in distilled water for 10-15 minutes. Drain washer and refill with fresh distilled water. Raise and lower basket, agitating and flushing out the inside of the pipettes. Repeat distilled water flush at least 3 times, allowing the pipettes to soak in distilled water between cycles. After the last rinse cycle, remove basket and drain excess water. Remove each pipette from the storage basket and rinse briefly with distilled deionized water using a wash bottle. Transfer to drying rack with tips up. Fill pipette storage jar with fresh 2% Citronox acid detergent before returning to the emptied basket.

8.6 NOCHROMIX (OR DICHROMATE) SOLUTION FOR RESISTANT RESIDUES

- 8.6.1 Stubborn contaminants may be removed using a sodium dichromate cleaning solution or a commercial equivalent formulation such as Nochromix. Use the chromic acid substitute when available. This cleaner is prepared from concentrated sulfuric acid, and should be handled with the utmost caution. Treat glassware in fume hood and wear eye protection, gloves, and apron. Nochromix (or dichromate) should only be used when nothing else will remove the contaminants.
- 8.6.2 Remove excess water from the object to be cleaned, as water will diminish the effectiveness of this cleaning solution. Allow surface to soak for at least 15 minutes in the acid cleaner. Remove and flush thoroughly with warm tap water. Proceed to distilled deionized water rinse (8.3). Return cleaner to container for reuse. Dispose spent Nochromix in acid sink after neutralization, and follow it with copious amounts of tap water. Dichromate cleaning solution must be disposed of by the University Hazardous Materials Program.

8.7 GLASSWARE STORAGE

- 8.7.1 All laboratory users are responsible for returning cleaned and dried glassware to the storage location where it was obtained. The cleaning process is not complete until the items are returned. Special storage requirements are discussed in previous sections. If the glassware was cleaned for a particular purpose, it should be noted and stored separately from other glassware.

9 DATA REDUCTION AND STATISTICS

10 QUALITY ASSURANCE

It is the responsibility of all laboratory personnel to read and understand this and all other necessary SOPs. Any questions should be discussed with the Laboratory Director. All laboratory personnel must document that they read and understood this procedure and all other necessary SOPs as per the Training Schedule. It is the responsibility of the Laboratory Director to keep a record of all personnel who have read this and other SOPs.

11 ADDITIONAL INFORMATION

11.1 REFERENCES

1. APHA (1992) Standard Methods for the Examination of Water and Wastewater. 18th Edition. A.E. Greenberg, L.S. Clesceri, A.D. Eaton, Eds., American Public Health Association, American Water Works Association, and Water Environment Federation.
2. Christian, G.D. (1986) Analytical Chemistry (4th Edition) John Wiley and Sons, Inc.
3. Friedman, L.C. and D.E. Erdmann (1982) Quality Assurance Practices for the Chemical and Biological Analyses of Water and Fluvial Sediments (Chapter A6). Techniques of water-resources investigations of the United States Geological Survey. Book 5.
4. 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
GLASSWAR.001	001	01-Aug-1994 to 19-May-2004	Unknown
GLASSWAR.002	002	19-May-2004 to 01-May-2006	Unknown
Gen-Glassware Cleaning-003	003	01-May-2006 to 16-Mar-2018	Dan Snow
07_02_01.004 Glassware Cleaning	004	16-Mar-2018 - Present	Victoria Wickham

12.1 ISSUE CHANGES

12.1.1 Issue 002:

- Changes unknown

12.1.2 Issue 003:

- Updated text and outline format
 - Changed from Courier New to Times New Roman
 - Changed font size from 12 to 10
 - Changed font style from bold to regular
 - Changed outline format to include right tabulation
- Added Title Page to SOP
 - Changed to include background on previous issues

- Added author and Laboratory Director information
 - Added review and training schedule information
 - Added Table of Contents
- 12.1.3 Issue 004:
- Updated SOP to new format
 - Changed from Times New Roman to Arial
 - Changed font size from 10 to 11
 - Changed outline format
 - Removed Title Page and Table of Contents
 - Added Solution tables
 - Changed disposal instructions to include acid neutralization
 - Removed un-used acid dilutions
 - Revised procedure to add more detail, and to reflect current practice

13 DIAGRAMS, FIGURES, AND PHOTOGRAPHS
