

STANDARD OPERATING PROCEDURE

Care of Scientific Glassware

Glassware may seem like an unimportant and mundane topic, but using it and caring for it properly can improve laboratory safety and make the difference between experimental success and failure. Glassware can also be expensive to routinely replace, so handle it with care and it will reward you with a long and useful lab life.

General Handling

- Always inspect glassware before use for chips, cracks, or scratches. Discard defective glassware (and used Pasteur pipets) in a box intended for broken glass, not in the regular trash. If the glassware is particularly expensive or custom-made, it can be repaired with fire-polishing by a properly trained individual.
- Never allow glassware to have prolonged contact with metal or grit. Use plastic spatulas, stirring rods, and rubber policemen.
- Re-grease glass stoppers and stopcocks frequently. This will prevent sticking and breakage. Do not grease Teflon stopcocks.
- If you need to heat glassware or quartz ware, you must first triple-rinse it in deionized or distilled water after cleaning and dry it thoroughly. This will ensure that you do not permanently burn contaminants onto the glassware. When handling quartz (especially cuvettes) you should always wear clean gloves to prevent oils on your hands from contaminating and causing the onset of devitrification.

General Cleaning

- Clean glassware as soon as possible after use. Allowing dirty glassware to sit around just makes eventual cleaning more difficult.
- Separate your glassware into those that need regular cleaning and those that require more intense cleaning.
- Disassemble your apparatus as soon as possible after you are finished with it. Remove all stopcocks and stoppers from addition funnels, separatory funnels, and the like. Ground glass stopcocks and stoppers will freeze in place if certain reactants (for example, bases) were used in them. Triple-rinse all surfaces with an appropriate alcohol followed by water to remove traces of solvents and reaction mixtures; place the used solvents in the appropriate hazardous waste container.
- Graduated cylinders, beakers, Erlenmeyer flasks, burets, and pipets that were only used to dispense or briefly store reagents generally only need to be triple-rinsed with a compatible solvent followed by tap water and a final deionized (DI) water rinse, if desired. Air dry on a drying rack.

- Büchner funnels, etc., should be rinsed with an appropriate solvent to remove substances that are clinging to them. Follow this with tap water and DI water rinses and air dry.
- Even a task as simple as washing glassware in the sink is potentially hazardous. Splashing solvents or dirty water in the eyes is a common hazard. You must wear eye protection and gloves that are appropriate for the task at all times. Choose proper gloves by referring to a glove compatibility chart.
- Before cleaning, be sure that any excess reagent has been disposed of properly and the vessel in which it was contained has been triple-rinsed into the waste container.
- Degrease your glassware's ground glass joints by wiping them with a paper towel soaked in a small amount of ether, acetone, or other solvent. Wear appropriate gloves and minimize your exposure to the vapors by doing this in a fume hood.
- Place the glassware in a warm concentrated aqueous solution of Alconox or other detergent and let it sit for several minutes.
- Be sure that your brush is in good shape before scrubbing. It should not be rusty or have matted bristles; replace it if necessary. Use the correct brush for the glassware; for example, use bottle brushes on bottles.
- Scrub. Be careful not to push too hard on the bottoms of beakers and flasks; you can easily push through the bottoms, especially on big beakers.
- Rinse thoroughly with tap water and give a final rinse with DI water. The water should sheet cleanly off the glass. If water does not sheet off the glass, and you desire the glassware to be analytically clean, first repeat the above soaking and scrubbing steps. If after a second cleaning bits of solid still adhere to the glass, or if there is clearly a greasy residue on the glass, you need more aggressive action.
- Note: if you are doing analytical work, you should triple-rinse all glassware with DI water before letting it dry. This will remove any ions or tap water impurities.
- Dry wet glassware by 1) placing it on the drying rack (or invert on a paper towel), 2) placing it in the drying oven (for items that are water-wet only, no flammable solvents) or 3) rinsing with a solvent such as acetone, methanol, or ethanol and then gently blowing compressed air into the vessel until it is dry. The first method is preferred for drying analytically clean glassware (provided that the prongs of the drying rack are not inside the item, thus contaminating it). Volumetric glassware and cuvettes should never be placed in drying ovens. The third method is acceptable only when the compressed air supply is known to be free of oil and other contaminants. An alternative to blowing air into the item is to use an aspirator, or house vacuum, to pull air into the item.

Moderately Aggressive Cleaning

Use these guidelines if general cleaning fails. These solutions are corrosive. Use proper personal protective equipment (PPE) – appropriate gloves, safety glasses, and lab coat.

- If the contaminant is a metal-containing compound, soak the piece of glassware in a 6 M HCl solution. Once the solid has dissolved, copiously rinse the item with tap water, and then repeat the general cleaning steps above. This method will also remove some organic residues but not grease.
- If the contaminant is organic, submerge the item in a base bath, which is a saturated NaOH or KOH solution in ethanol, methanol, or isopropanol. Be sure the base bath is stored in an HDPE container; otherwise it will leak. Wear butyl gloves and keep ignition sources away from it. Be sure that the piece of glassware is completely filled with the solution and is sitting upright. After several minutes of soaking, carefully remove the item (it will be slippery), and rinse thoroughly. If the glassware is not clean at this point, general cleaning steps may need to be repeated or a longer soaking time in the base bath may be needed. *Do not soak any glassware in base bath longer than necessary, as it can slowly dissolve a layer of glass or even permanently etch/frost surfaces.* NEVER soak the following items in a base bath for prolonged periods:
 - Glassware contaminated with metal-containing compounds
 - Glass fritted funnels
 - Cuvettes
 - Volumetric glassware (pipets, volumetric flasks)
 - Any glassware contaminated by an oxidizing agent
 - Anything that has not first been washed according to the above steps

Extremely Aggressive Cleaning

Follow these steps when the moderately aggressive methods fail. Be very careful. The solutions described are very corrosive! Only use them in a fume hood and wear proper PPE.

- **Aqua regia.** This is an extremely powerful oxidizing solution prepared from 1 part concentrated HNO₃ and 3 parts concentrated HCl. Add 1 part H₂O if you will store the aqua regia to minimize the generation of Cl₂. This is the only acidic solution that will dissolve gold and will oxidize just about everything else. Extreme caution must be used when working with aqua regia because it generates Cl₂ and NO_x gases in addition to causing severe tissue damage. Clean the glassware before soaking in aqua regia and then rinse thoroughly with water.
- **Acidic peroxide.** This is most conveniently prepared by dissolving the commercially-available NoChromix mix in concentrated H₂SO₄ per the package directions. An alternative preparation is to prepare a solution by mixing equal proportions of concentrated H₂SO₄ and aqueous H₂O₂ solutions (remember to add the acid to the H₂O₂). A 3% H₂O₂ solution is usually sufficient, and under no circumstances should



H₂O₂ solutions greater than 10% be used. The H₂O₂/H₂SO₄ solution is both a strong oxidant and a strong reductant, so care must be taken when using it. Another acidic peroxide solution for cleaning can be prepared by dissolving 36 g (NH₄)₂S₂O₈ (ammonium peroxydisulfate) in 2.2 L of 98% H₂SO₄ (can be made right in the bottle of H₂SO₄, if the bottle is loosely stoppered). The procedure for these solutions is the same as for aqua regia, as are the precautions for their use.

- **Chromic Acid.** This is a solution of CrO₃ in concentrated H₂SO₄. A premeasured mix is available under the name Chromerge which should be treated in the same way as aqua regia or acidic peroxide solutions. Because high-valent chromium is carcinogenic, teratogenic, and causes severe environmental damage, the use of chromic acid is not recommended.

Special Cases

- **Cuvettes.** Generally, you only need to rinse a cuvette in the appropriate solvent and wipe the outside with a Kimwipe immediately after use. If something has adhered itself to a cuvette, it is best to soak the cuvette in solvent first and gently coax the solid off the side with a cotton swab. **Never use a brush on a cuvette!** If this fails, you can use 6 M HCl. Do not use a base bath on cuvettes because it tends to etch glass surfaces. **Never put cuvettes in a drying oven--air-dry them.**
- **Fritted Funnels.** You can clean these by inverting and allowing the solvent to flow by gravity through the frit in reverse. You can also pull solvent through the frit under vacuum. You can remove stubborn material by soaking in acid, followed by copious rinsing with water under vacuum. Because the base bath solution etches glass, do not use it on fritted funnels (a brief exposure to a base bath is not usually fatal to a frit, but prolonged soaking should be avoided).
- **Protein Contamination.** You can usually remove proteins by scrubbing with detergent, but occasionally protein defies removal. In that event you can proceed to the more aggressive acidic solutions, or you can prepare a peptidase solution. The enzymatic approach is a bit slower than the forcing methods, but it is greener and gentler and so you can use it when the contaminated item is incompatible with acid.
- **Fire Polishing.** If you want to save mildly chipped glassware, you can fire-polish it. Light a Bunsen burner (be sure no flammables are nearby) and place the site of damage inside the hottest part of the flame. Allow the glass to melt enough to smooth the rough edges of the damage on all sides. When the glass has melted enough, remove it from the heat and allow to cool. Be careful – hot glass looks the same as cool glass. Turn off the burner.

For more information, see <http://www.udel.edu/chem/GlassShop/GlasswareCare.htm>

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