

Standard Operating Procedure

Task: Cleaning glassware

Date: 5/13/13

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Background: Cleaning glassware is essential for ensuring that residues on glassware do not contaminate future reactions.

Training Requirements:

- Laboratory Safety Training
- Obtain assistance from a senior student when first preparing or using *aqua regia* or base bath.

Potential Hazards:

- Caustic (base burn)
- Corrosive (acid burn)
- Inhalation hazard (organic solvents)
- Cuts from broken glassware
- Chemical splashes

Materials Needed:

- Dirty glassware
- Organic Solvents (e.g. acetone, methanol, etc.)
- Soapy water, distilled water
- Base bath (see "Making base bath" SOP)
- Aqua regia (see "Making aqua regia" SOP)

Special PPE Requirements:

- Thick rubber gloves if using base bath.
- Face shield if using base bath or if cleaning vacuum line.

Procedure:

- First, consider what you are cleaning. Different reactions will pose different challenges, and safe disposal of chemical waste must be considered. Is the reaction mixture pyrophoric? Toxic? Reactive? Take appropriate precautions (including appropriate PPE) and think carefully about compatibility before dumping into a mixed waste container. (See Waste Disposal SOP for more information)
- Remove any glass and Teflon stopcocks before cleaning. Stopcocks and ground glass joints can be washed with pentane and a Kimwipe (abrasion is important). Pipe cleaners can be helpful for hard-to-reach spots.
- Rinse the flask several times with organic solvent (e.g. acetone, MeOH, thf, CH₂Cl₂, etc) and/or water to remove all visible contaminants. If organic solvents are used, rinse once more with acetone.
- Scrub both the interior and exterior of the flask vigorously with a washing brush and soap/warm water to remove salts and remaining residues.

- Rinse flask with warm water (at least 2-3 times) and with distilled water (at least 2-3 times) to remove all soap/residues.
- For standard organics and other minimal contamination, a simple rinse with acetone is sufficient before placing on the drying racks.
- For metal-containing reactions, or heated reactions where residues may cling to the glassware, a base bath may be necessary (see below for base bath procedure). Since base bath solution will etch glass, **the following glassware should not be placed in the base bath**: volumetric glassware (e.g. graduated cylinders, volumetric flasks, etc.), NMR tubes, frits (dissolve readily in base bath), and cuvettes. Ground glass joints and Kontes valves may only be soaked in base bath for short periods of time (< 12 hr), as long term soaking may compromise the seal.
- For **frits**, which often contain visible contaminants after standard cleaning procedures, aqua regia is particularly effective. See below for aqua regia procedure.
- **Cuvettes** can be washed by soaking in aqua regia, followed by extensive washing with water and acetone.
- **Syringes** can be washed with either organic solvents or water (depending on what the syringe was used for) to remove residues, then several times with acetone.
- **NMR tubes** can be cleaned by repeated washing with organic solvents or water, followed by acetone. Alternatively, the “homemade” NMR tube cleaner shown below can be used:



The NMR tube cleaner is assembled by fitting a filter flask with a rubber septum containing a hole for plastic tubing feed-through. After opening the flask to vacuum, the NMR tube is placed over the tubing, resting on top of the septum. The top of the septum is sprayed with acetone, which rinses the NMR tube and drains through the

plastic tubing. If organic solvents and water fail to get rid of all residues from the tube, the tube may be treated with aqua regia.

If glassware remains visibly dirty **DO NOT** leave it on the drying rack for someone else to use! Consult your labmates or advisor for alternative cleaning strategies.

Using the Base Bath:

- Glassware should be visibly clean before going into base bath. Never place greased glassware into base bath.
- Put on your lab coat, goggles, and gloves.
- Put on clean, sturdy, cut-free thick rubber gloves (clipped under the sink).
- Carefully submerge glassware into base bath. Fully submerge items and avoid bubbles.
- While wearing your base bath gloves, rinse exposed areas with water in sink.
- Soak glassware as needed, usually 1-24 hours. Do not leave glassware unattended for more than two days.
- To remove glassware, carefully drain base bath solution from glassware while wearing appropriate PPE (see above). Retain base bath solution in original container.
- Carefully transfer glassware to the sink, using a secondary container to prevent dripping base bath on floor. Wash glassware and gloves with water. Wash glassware with deionized water and acetone before placing on drying rack.
- Put base bath away.

Using Aqua Regia:

- Make sure that glassware has been washed extensively with water prior to treatment with aqua regia. Organic residues can react violently with aqua regia!
- Place glassware in a secondary container to contain potential acid spills.
- Add aqua regia to the glassware to be cleaned. A convenient way to clean frits involves placing the frit “upside down” so that the drain spout is sticking up. Aqua regia can be added to the bottom side of the frit until it almost fills the spout. Gravity will draw the aqua regia through the frit.
- Clearly label the fumehood with a sign noting the presence of glassware soaking in aqua regia.
- Let glassware soak for several hours or overnight.
- Pour used aqua regia out of glassware into a clean beaker. Clean glassware extensively with water. Pour combined rinses into a separate aqua regia-only waste container.
- After washing with water, glassware can be washed with acetone and dried.

References and Related SOPs:

- Making a Base Bath SOP
- Aqua Regia SOP
- Waste Disposal SOP