

Standard Operating Procedure: Glassware Cleaning

Using clean glassware is key to the success of a chemical reaction, and cleaning glassware is part of good lab citizenship. All members of the lab must contribute to this. ***Do not clean glassware if you are alone in the lab; all the baths/mixtures used for this purposes (with the exception ofalconox) are very dangerous and can cause severe burns.***

The procedure described below is the bare minimum expected in every instance. Additional steps are appreciated but not required.

Personal Protective Equipment Required

Lab coat, safety glasses or goggles, nitrile gloves, heavy-duty (gauntlet neoprene) gloves. Safety apron may be worn on top of the lab coat. Face shield advised for acid and base baths along with aqua regia and piranha solutions.

Hazards

Both the acid and base baths are flammable and corrosive; contact with skin will cause burns. Their fumes might irritate your airways.

Piranha solution and aqua regia are corrosive and may cause severe burns.

You are handling large amounts of glassware that is many times slippery from the cleaning solutions – be extra careful to avoid breaking anything and cutting yourself. This is particularly important when the glassware isn't clean – chemicals could get in the wounds.

Glassware Cleaning

The routine procedure will be outlined first. Extra steps may be necessary in some cases; those are described after the general cleaning methods.

All glassware/spatulas/frits/etc. must be ***spotless*** before going into any cleaning bath. In most cases, this can be achieved using one or more solvents. If this is not the case, refer to the “*what to do...*” section below. Once this is achieved, move on to the next steps:

- *General Glassware (except volumetric material and fritted glass)*

First let the glassware soak in the base bath for at least 3 hours (usually overnight). When putting glassware in the base bath ***make sure the glassware is completely filled with base bath and no air bubbles are seen***. For oversized items, it may be necessary to flip them and let them soak for another period before taking them out of the base bath. Then, take items out of the base bath (be careful not to spill base everywhere!!!), rinse with DI water 3 times (make sure you rinse both the inside and the outside surface). After this, transfer the glassware to the acid bath and let it soak (3 h – overnight; ***take care to avoid air bubbles***). Once this is done, rinse with DI water 3 times, and finally leave soaking in DI water. After the allotted time (3 h – overnight) rinse 3 times with DI water and allow to dry on drying rack.

For test tubes: do not leave them on the drying rack; put them in the oven to dry overnight (wrap tubes together with aluminum foil before putting them in the oven).

- *Volumetric glassware (flasks, pipets, graduated cylinders) and anything with fritted glass (frits, columns)*

The procedure is similar to what's described for general glassware, with the exception that two acid baths are used instead of a base bath and an acid bath. Fritted glass can trap impurities; air bubbles left in the glassware are detrimental to the cleaning process. ***Make sure no air bubbles are present when putting frits in the acid or DI water baths.*** When rinsing frits with DI water, attach the hose to the stem and fill the frit with water from the bottom up. Frits may get airborne when doing this: be careful and fill them slowly.

Frits are to be dried in the oven overnight after soaking in DI water.

Do not put volumetric material in the oven. Ever.

- *Spatulas*

Soak in an Alconox bath overnight, rinse with DI water and then soak in DI water. Let them air dry.

- *Stir Bars*

These can be cleaned along with the general glassware. However, it's convenient to clean them in a personal acid bath kept in the hood (it's easy to lose stir bars in the big buckets).

What to Do if all the Above isn't Enough

Do not despair; we've all been there. Do *not* ignore the problem or hide the glassware away. More options are described below:

- Alconox

This is a mild detergent that can effectively clean some items. It is a good first approach.

- Stronger Base Bath

We have a KOH/isopropanol base bath in addition to all the NaOH/ethanol ones. Avoid putting frits in it; if you must do it (because nothing else has worked), don't leave them for longer than an hour.

- Aqua Regia

This is a good option when there are metallic contaminants. Mainly used for stir bars and frits.

- Piranha Solution

An all-time favorite. Eats through most stuff. Great (albeit dangerous) option for fritted glass.

Before using Aqua Regia, Piranha or other severe cleaning solutions for the first time, consult with more experienced labmates to ensure proper safety measures are followed.

Waste Disposal

For waste generated during cleaning before the base and acid baths, dispose of in proper waste container. Aqua Regia and Piranha waste must be disposed of in separate waste containers and submitted for pick-up immediately.

Description of Cleaning Solutions/Baths

Acid Bath: 3 M hydrochloric acid in ethanol

Base Bath: 3 M sodium hydroxide in ethanol

Strong Base Bath: 3 M potassium hydroxide in isopropanol

Aqua Regia*: One part concentrated nitric acid to three parts concentrated hydrochloric acid. Add nitric acid to hydrochloric acid. See note below.

Piranha*[†]: 3:1 concentrated sulfuric acid/hydrogen peroxide. Add hydrogen peroxide to sulfuric acid. Mixing is extremely exothermic, so allow solution to cool before using. All containers that contain Piranha should be made of glass. See note below.

When handling large amounts of aqua regia or piranha (>500 mL), use neoprene gloves. All handling of aqua regia and piranha **MUST** be done inside a fume hood. Perform all work in a secondary container that is clean and free of organics.

Warning: **NEVER** add any organic solvents to an aqua regia or piranha solution, as it could cause an explosion. Even small amounts of organics could make the aqua regia or piranha solution unstable. ***Only make small, fresh batches of aqua regia and piranha for each use.***

*Extremely corrosive

[†] Strong oxidizer