

Standard Operating Procedure: Using Aqua Regia and Piranha

When solvents, alconox, and the acid and base baths cannot render frits clean, additional cleaning methods are necessary. Two common cleaning solutions can be used: Aqua Regia and Piranha. While these solutions can be very effective, they are unstable, highly corrosive, and can cause serious harm and injury if not used properly. As such, Aqua Regia and Piranha should be used for glassware that will not come clean with any other cleaning method and several precautions must be taken when using Aqua Regia and Piranha.

When preparing both Aqua Regia and Piranha, ***the order of addition is very important***, as incorrect mixing can lead to an explosion:

Aqua Regia: 3:1 concentrated nitric acid/ concentrated hydrochloric acid. Add nitric acid to hydrochloric acid.

Piranha: 3:1 concentrated sulfuric acid/hydrogen peroxide. Add hydrogen peroxide to sulfuric acid.

BOTH AQUA REGIA AND PIRANHA ARE EXTREMELY CORROSIVE AND POWERFUL OXIDIZERS

When using either Aqua Regia or Piranha:

ONLY USE GLASS (Filter flask, stir rod, waste container, etc.)

NEVER USE ORGANIC SOLVENTS WITH PIRAHNA OR AQUA REGIA

AQUA REGIA AND PIRAHNA SHOULD NEVER BE TAKEN OUT OF A FUME HOOD (make it, use it, dilute it, dispose of it all in one fume hood)

NEVER LEAVE SOLUTIONS SITTING OVERNIGHT

AQUA REGIA AND PIRANHA REQUIRE THEIR OWN SEPARATE GLASS WASTE CONTAINERS (do not tighten the caps too much as pressure can build up and cause an explosion)

To avoid large amounts of Aqua Regia and Piranha and their subsequent storage, they can be made *in situ*. Use a 1 L filter flask filled with 500 mL of DI water. Then place the frit in the filter flask and add $\frac{1}{4}$ of the frit volume of either hydrochloric acid (Aqua Regia) or sulfuric acid (Piranha). Then, make up the difference with the second component of the solution, nitric acid (Aqua Regia) or hydrogen peroxide (Piranha) while stirring with a glass stir rod. Both solutions are highly exothermic upon mixing, so allow them to cool before touching the frit, or use insulated gloves if the frit must be moved. Turn on the aspirator to pull a vacuum on the frit until the cleaning solution begins to collect in the filter flask, then remove the vacuum and allow the frit to soak in the cleaning solution. Once the frit appears to be clean, turn on the aspirator again, drain the frit of the cleaning solution, rinse it three times with DI water, then soak in DI water for at least three hours. After using Aqua Regia and Piranha, wash the floor of the fume hood and any glassware used to contain the cleaning solution with a sodium bicarbonate solution.

Larger batches of Aqua Regia and Piranha may be made as well in a glass container. Make only as much Aqua Regia and Piranha as you will need. When handling large amounts of Aqua Regia

or Piranha (>500 mL), use neoprene gloves. The cleaning procedure is the same as given above. Do not store either cleaning solution overnight.

Personal Protective Equipment: Lab coat, gloves, safety glasses. All work must be conducted in a fume hood.

Waste Disposal: All waste from Aqua Regia and Piranha (including rinsate) must be stored in their own separate glass waste containers and be submitted for pick-up immediately (DO NOT COMBINE THE TWO CLEANING SOLUTIONS). Do not over-tighten the caps of the waste containers.