U N I V E R S I T Y *of* L O U I S V I L L E

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Cleaning Glassware, Syringes and Syringe Needles

Note: Although it may not seem that important, cleaning glassware is one of the most important tasks that you will do in lab – contaminated glassware (along with contaminated solvents) are the two biggest causes of reactions going bad!

General Group Glassware

1) Thoroughly clean grease off of all joints with hexanes and a Kimwipe.

2) Glass and Teflon stopcocks should be removed from joints (and degreased if necessary) before cleaning. They are easily damaged by small particles such as salts and the stopcock bore tends to hold up liquids.

3) Rinse out flask into organic waste to remove organic material by washing with a H2O-miscible organic solvent like 70% ^{i}PrOH, MeOH, acetone, or THF, depending on solubility.

4) Scrub both the interior and exterior of the flask vigorously with a washing brush and soap/warm water to remove salts and remaining residues.

5) Rinse flask with warm water (at least 2-3 times) and with distilled water (at least 2-3 times) to remove all soap/residues.

6) Finally, rinse with a small amount of 70% ^{i}PrOH, MeOH, or acetone and place on the drying racks.

7) If glassware remains visibly dirty after this procedure **DO NOT** leave it on the drying rack for someone else to take and use!! **ASK** Dr. Burns about the best way to get it clean – this will usually entail either placing it in the base bath and/or washing with strong acid (e.g. conc. H_2SO_4, HNO_3) to remove residual metal salts.

Note: Information on making and using a base bath and other strong cleaning solutions can be found at the end of this document.

Glass Frits

1) Rinse your frit with solvents in which the solids on it are soluble. Typically this would involve acetone followed by MeOH. Then, turn the frit upside-down and rinse with these solvents a second time.

2) Note that aqueous washes (i.e., those with bleach/water/acid/etc) are sometimes necessary to remove toxic reagents like Sn and/or other reagents that are soluble in these media. However, these washings need to be separated from the organic washings, and disposed of separately (see waste section). Also, washes with H_2O and/or aqueous solutions should be followed by copious rinsing with 70 % ^{i}PrOH, or MeOH before the introduction of immiscible organics like CH_2Cl_2 or hexanes.

3) If residue remains (especially metal-based residue) it can often be removed by placing 50% conc. HCl and 50% MeOH in the frit and allowing it to drip through slowly, followed by rinses with HCl, H_2O, aqueous bicarbonate sol'n, H_2O, and MeOH.

4) If any particulate matter remains on the frit and/or it is not completely white, you should clean it with aqua regia (3 parts conc. HCl/1 part conc. HNO_3) or basic piranha solution (30 % H_2O_2/couple KOH pellets). However, ***YOU MUST COMPLETELY REMOVE ORGANIC SOLVENTS*** from the frit before subjecting it to aqua regia or basic piranha solution (highly oxidizing!). So, rinse the frits with MeOH followed by copious water before attempting to clean them with aqua regia or basic piranha.

Note: Another strongly oxidizing cleaning solution is acidic piranha (conc. H_2SO_4/30 % H_2O_2). Information on making, using and safety considerations can be found at the end of this document.

NMR Tubes

1) Rinse the contents of your NMR tubes into organic (or aqueous) waste (depending of the contents of the tube).

2) Rinse tubes at least one to two more times with a wash bottle into your waste before using the NMR tube cleaner. These steps are important to avoid excessive contamination of the NMR tube cleaner with everyone's samples.

3) **Note**: You should never stick the tip of a wash bottle into an NMR tube to wash it out. This will inevitably lead to breaking the end off the tube. Instead, always hold the bottle several cm away from the end of the tube to spray the solvent in.

4) If solids/precipitated metals remain in the tube at this point, clean it out with some solvent (typically acetone) and a pipe cleaner.

5) Use the NMR tube washer to finish cleaning the tube. Typical solvent rinses might involve acetone followed by MeOH.

6) Note again that aqueous washings (i.e., those with bleach/water/acid/etc) are sometimes necessary to remove toxic reagents like Sn and/or when the reagents used in NMR experiments are soluble in these media. However, these washings need to be separated from the organic washings, and disposed of appropriately (see waste section). Also, washes with H_2O and/or aqueous solutions should be followed by copious rinsing with 70 % ^{i}PrOH or MeOH before the introduction of immiscible organics like CH_2Cl_2 or hexanes.

7) Place NMR tubes flat in a 140 °C oven to dry. However do not leave them in the oven for more than ~6-8 hrs. Leaving the NMR tubes in the oven for longer than this can lead to warping, which may cause problems with spinning, shimming and/or result in breakage in the NMR instruments. You can also leave your NMR tubes in the 70 °C for longer periods of time and not risk warping your NMR tubes.

Syringes/Needles

1) **ALL** syringes need to be cleaned directly after use! This prevents them seizing up or clogging (often irreversibly) with dried out residues. Additionally, these can be expensive pieces of glassware are in limited supply and are shared between many co-workers.

2) Once you are done using a syringe and needle combo make sure to rinse it out with the appropriate solvent such as ^{i}PrOH, MEOH or acetone.

3) Clean gas-tight syringes by rinsing them 2-3 times with 3-4 different solvents. Typically this would include MeOH, acetone, EtOAc, and CH_2Cl_2.

4) Gas tight syringes should be placed in the 140 °C oven after cleaning without their plungers for 3-4 hrs. Longer times in the oven can lead to cracking and/or damage to the syringe. They can also be placed in the 70 °C oven for longer periods of time. Plungers should be wiped off and placed directly into the storage drawer after cleaning. This prevents irreversible expansion/contraction of the plunger from repeated heating/cooling cycles.

5) Non-disposable stainless steel needles should be rinsed thoroughly with appropriate solvents (typically MeOH followed by acetone and dried in either the 140 °C or 70 °C oven.

6) Once again, note that aqueous washing of both gas tight syringes and needles (i.e., those with bleach/water/acid/etc) are sometimes necessary to remove toxic reagents like Sn and/or when the reagents used are soluble in these media. However, these washings need to be separated from the organic washings, and disposed of appropriately (see waste section). Additionally, washes with H_2O and/or aqueous solutions should be followed by rinsing with copious rinsing with 70 % ^{i}PrOH or MeOH before the introduction of immiscible organics like CH2Cl2 or hexanes.

Glassware Cleaning Solutions tips and tricks

There are a number of tricks for cleaning glassware and stirbars.

Base Bath: iPrOH/KOH:
250-300g KOH pellets
4L of 70 % ^{i}PrOH
1L of distilled H2O

Give your glassware a soak in this solution (usually ~24 hours) and then rinse it clean. Make sure any glassware that is placed in a base bath is degreased with hexanes and kimwipes before placing it in the base bath.

Nitric Acid: Nitric acid works well to get rid of most contaminants. Many people use it in 50% strength (diluted with water), or diluted with acetone. Be careful though: HNO_3 is a strong oxidizer and will form NO (nasty, brown gas) if you react it with Mg, for example.

Aqua Regia: Literally means royal water. Formed by mixing 1 part nitric acid : 3 or 4 parts hydrochloric acid. For a stronger version, try 1:1 nitric acid : hydrochloric acid. This forms an extremely potent yellowish/orange fuming solution containing Cl_2 and NOCl. Extremely effective, but quite toxic and very reactive. Do not store this solution. Works great for stirbars.

Other solutions:

5N NaOH / Acetone (1:1) works great for cleaning classware, especially Chromium reactions.

5N NaOH / EtOH (1:1) works well also (similar to base bath).

ACIDIC PIRANHA SOLUTIONS TIPS AND TRICKS

From Princeton EHS - Acid Piranha Solutions

Overview

Piranha solutions are used to remove organic residues from substrates, particularly in microfabrications labs. The traditional piranha solution is a 3:1 mixture of sulfuric acid and 30% hydrogen peroxide. The solution may be mixed before application or directly applied to the material, applying the sulfuric acid first, followed by the peroxide. Piranha solutions are extremely energetic and may result in explosion or skin burns if not handled with extreme caution.

Emergency Procedures

In case of skin contact: May cause skin burns. Flush the skin with copious amounts of water for at least 15 minutes. Seek medical attention.

In case of eye contact: Piranha is corrosive and irritating to the eyes. Flush contaminated eye(s) immediately with copious quantities of water for at least 15 minutes. Seek medical attention immediately.

In case of inhalation: May irritate the respiratory tract. Conscious persons should be assisted to an area with fresh, uncontaminated air. Seek medical attention in the event of respiratory irritation, cough, or tightness in the chest. Symptoms may be delayed.

In case of ingestion: Not a likely route of exposure.

Handling

- Always use glass (preferably Pyrex) containers. Piranha will melt plastics.
- Mix the solution in a hood with the sash between you and the solution. Wear gloves and eye protection.
- When preparing the piranha solution, always add the peroxide to the acid.
- Piranha solution is very energetic and potentially explosive. It is very likely to become hot, more than 100 degrees C. Handle with care.
- Leave the hot piranha solution in an open container until cool.
- Never store piranha solutions. Piranha stored in a closed container will likely explode.
- Adding any acids or bases to piranha or spraying it with water will accelerate the reaction. This includes Photoresist, which is a strong base.
- Mixing hot piranha with organic compounds may cause an explosion. This includes acetone, photoresist, isopropyl alcohol, and nylon
- Aspirate the piranha and dispose via the drain when finished.

Storage

Do not store piranha. Mix fresh solution for each use. Excess solutions should be disposed via the drain, followed by flushing with copious amounts of water.

Disposal

Do not collect for disposal. After the material has cooled, aspirate excess piranha and dispose via the drain, flushing the drain with copious amounts of water.

***Note**: The above excerpt was taken from Princeton EHS website:*

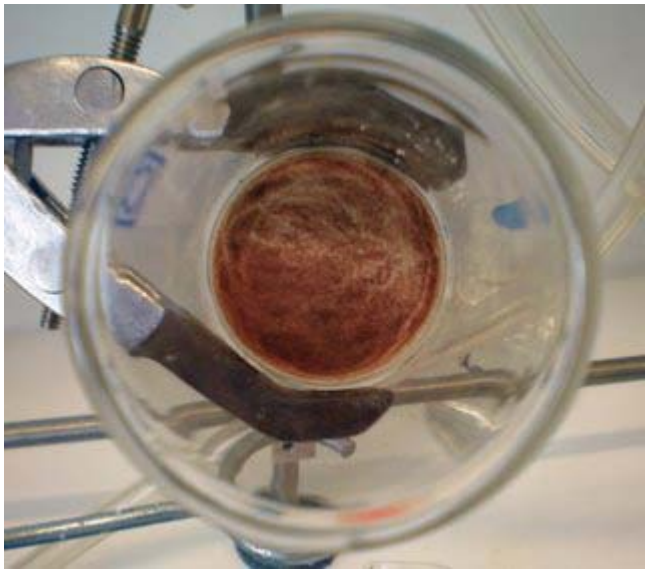
Laboratory Safety Manual: SECTION 10: Chemical-Specific Issues

URL: http://web.princeton.edu/sites/ehs/labsafetymanual/TOC.htm

How to clean your sintered funnel using Acidic Piranha solution

- taken from Curly Arrow chemistry blog by Daniel Sejer

Lately when I have been cleaning my sintered funnels people have stopped and asked me what I was doing. To my surprise many chemists don't seem to know how to take a nasty, dirty sintered funnel and making it nice, white and shining again in about 15 minutes.



These days I'm doing lots of old school chemistry that involves heating the crap out of the components using for example conc. sulfuric acid as the solvent. Needless to say things are polymerizing and decomposing left, right and centre and when you filter it through your nice white sinter it ends up looking nasty (see picture above). The stuff doesn't go anywhere with acetone, water, 2M sodium hydroxide or hydrochloric acid etc. so what should you do? Before I proceed please note that if you attempt any of the following you must:

(1) Wear a closed lab coat, safety glasses and plastic gloves
(2) Conduct the cleaning in a fume hood with the sash down at all times
(3) Ensure that all the glassware is clean and doesn't contain residual organic material such as acetone

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Please take the above advice seriously. People have had nasty accidents doing the following because they weren't careful.
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There are two common ways to get your sinter clean:
(1) Conc. nitric acid, or
(2) Conc. sulfuric acid and hydrogen peroxide

Nitric acid is the easy solution and more often than not it does the trick. However, occasionally it is necessary to use more vigorous conditions. I have never had a sinter that didn't become white after treatment with conc. sulfuric acid and hydrogen peroxide and this is generally the method that I use because I know it works every time.

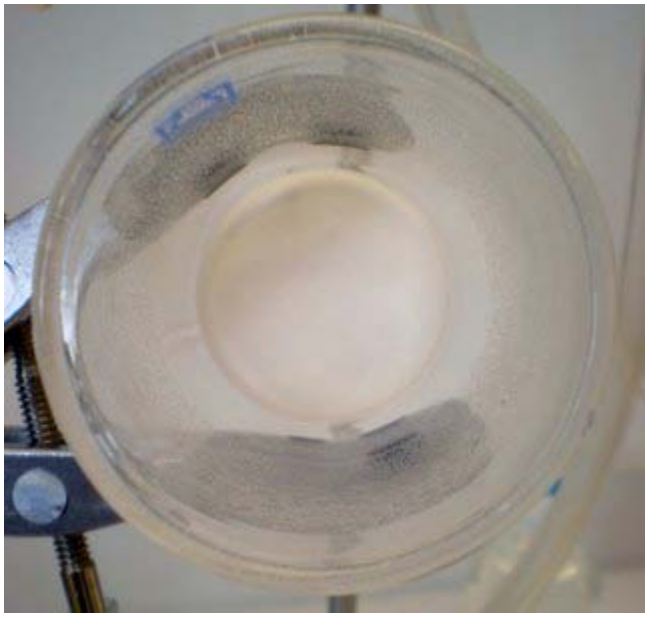
Procedure:

(1) Fit the funnel to a Büchner flask attached to a vacuum that you can control easily

(2) Add a small amount of conc. sulfuric acid so that it covers the surface of the sinter

(3) Add a dash of hydrogen peroxide and stand back. Things get pretty hot, bubbly and exciting at this point (See picture above).

(4) When the ingredients have been cooking away for a minute or so apply a very gentle vacuum briefly. This should be sufficient to suck the sinter dry (See picture above),



(5) Allow the cocktail to settle down and cool off and clean all the equipment with lots of water taking care not to pour the contents all over yourself. Your sinter will now look like this.