Cleaning Glassware

Any time you use community glassware/syringes, they must be thoroughly cleaned, dried and put away after use. Do not soak glassware in the sink or on the counter immediately next to it. If scrubbing/soaking with soapy water is insufficient, an acid or base bath may be used to remove stubborn contaminants.

The common cleaning workflow in the Tonks group is to rinse out a reaction flask into a waste container with acetone (or other appropriate organic solvent for your reaction mixture), scrub the flask with soap and water and rinse with DI water. If scrubbing/soaking with soapy water is insufficient, an acid or base bath may be used to remove stubborn contaminants. Upon removal of glassware from the acid/base bath, rinse with water and DI water too. Wet glassware is then placed on the drying rack for 24 hours before being returned to its community location. Additional cleaning measures should only be carried out if necessary.

A. General Safety Guidelines & PPE:

- Working with strong acids and bases presents the risk of chemical burns.
- Working with strong oxidants presents the risk of explosions or violent reactions

Full PPE (goggles or facemask, gloves, and labcoat) must be worn at all times, and aggressive cleaning methods should carried out in a working fume hood. Work with the safety sash in front of you at all times. Make sure to dispose of Acid, Base, Aqua Regia, and Pirhana cleaning solutions in their own independent waste container due to the high risk of chemical incompatibility.

B. Base Bath:

Base baths are generally used to thoroughly rinse glassware of organic and inorganic contaminants. Generally, an overnight soak is sufficient to remove most contaminants, the base bath is not meant to be used as a storage vessel.

There are several items that should **NEVER be placed in a base bath:**

- Sintered glass frits
- Graduated cylinders
- Syringes
- Any volumetric glassware
- Oversized glassware

The reason that these are incompatible with the base bath is that the base bath slowly etches the glass, leaving a clean glass surface for the next reaction. Therefore, any fragile or calibrated glassware will be destroyed in the base bath.

Each bench group should prepare their own base bath. Do not use anyone elses' base bath without explicit consent; there may be contaminants present or fragile glassware that may be broken.

- Base Bath Preparation: In a 5 gallon orange bucket (purchased at Home Depot; *must* have a sealable top), add 1 L DI H₂O. Next, *carefully* and *slowly* add 100 g KOH. Mixing KOH and H₂O is very exothermic, so be cautious of the generated heat. After the heat has dissipated, add 4 L of ⁱPrOH. The base bath is then ready for use. Base baths should be stored in secondary containment under the sinks and always kept closed with an airtight sealing lid.
- 2. **Base Bath Disposal and Refresh**: Base baths should be refreshed quarterly (during group clean up), or whenever the bath has been significantly contaminated. With the help of a second group member, transfer the contents of the base bath to an empty 4 L solvent bottle using a funnel, and manifest as waste per usual.

C. Acid Bath:

Acid baths are commonly used to clean metal contaminants from glassware or to clean sintered glass frits when regular washing is insufficient. Since the acid bath reacts with most metals, **DO NOT PLACE METAL APPARATI in the acid bath!** Generally, a quick rinse is sufficient to remove most contaminants, the acid bath is not meant to be used as a storage vessel.

Each lab should prepare their own acid bath. Do not use anyone elses' acid bath without explicit consent; there may be contaminants present or fragile glassware that may be broken.

- Acid Bath Preparation: In a 5 gallon orange bucket (purchased at Home Depot; *must* have a sealable top), add 4 L MeOH. Next, *carefully* and *slowly* add 1 L conc. HCI. Addition of acid to methanol is exothermic, so be cautious of the generated heat. After the heat has dissipated, the acid bath is ready for use. Acid baths should be stored in secondary containment under the sinks and always kept closed with an airtight sealing lid.
- 2. Acid Bath Disposal and Refresh: Acid baths should be refreshed quarterly (during group clean up), or whenever the bath has been significantly contaminated. With the help of a second group member, transfer the contents of the acid bath to an empty 4 L solvent bottle using a funnel, and manifest as waste per usual.

D. Gastight Syringes & Needles

- 1. It is critically important that gas-tight syringes and reusable metal needles are cleaned promptly after use to prevent corrosion and clogging due to salts.
- 2. Wash out gas-tight syringes using a multi-solvent wash, ending with acetone. The solvents chosen will be dependent on your reaction, but a common/standard order is THF, methanol, HCI/methanol, DI water, and finally acetone.
- 3. Stainless steel needles and cannula can be washed by sticking one end into a vacuum apparatus (suba seal fitted on a filter flask) and the other end of the needle into a beaker with solvent. A

multi-solvent wash mentioned in point 2) can be employed. Clean metal needles should be left to dry in the oven.

4. **DO NOT** put syringes into the base bath or oven. You will ruin their precision calibrations and potentially clog them. If you need to "dry out" one of these, put them on top of the oven where they will get slightly warmer than room temperature, but not by much.

E. Sintered Glass Frits:

Sintered glass frits, often used for filtering inorganic metal complexes, can be cleaned with the following procedure:

- 1. Invert the frit and washing with acetone (or other appropriate solvent for your material) while pulling vacuum through (rest the frit on a suitable size neoprene rubber adaptor fitted on a filter flask). Afterwards, repeat the procedure with DI water.
- 2. Rinse the frit in the acid bath.
- 3. Wash with DI water, and repeat the inversion procedure as necessary.

Most contaminants on a sintered glass frit are trapped close to its top surface, inverting the frit and washing it allows for a facile removal of contaminants.

If glass frits remain clogged, they can be cleaned with aqua regia and/or piranha solution. It is common in the group to collect clogged frits and clean them all at once during the biannual group clean up. Keep in mind that these are **options of last resort** and should not be necessary with routine cleaning of frits immediately after use.

To clean with aqua regia/piranha solution (see below),

- 1. Invert clogged frits and balance them on their broad end in a crystallizing dish or huge beaker
- 2. Add aqua regia/piranha solution to the stem and allow solution to drip
- 3. When all the solution has dripped, wash the frits with plenty of water and DI water
- 4. Repeat the normal cleaning procedure.

F. Aqua Regia:

Aqua regia solution is an extremely acidic, corrosive, toxic and oxidizing cleaning agent commonly used to remove metal contaminants, especially things that have plated out (exception: Ir and Ta).

HNO₃ (aq) + 3 HCl (aq) → NOCl(g) + 2 H₂O (l) + Cl₂ (g) 2 NOCl (g) → 2 NO (g) + Cl₂ (g) 2 NO (g) + O₂ (g) → 2 NO₂ (g)

The mixing of HCl and HNO₃ produces NOCl that over time decomposes into Cl_2 and NO_2 . As such, the solution should always be prepared fresh and never stored in a closed container (HIGH PRESSURE BUILT UP).

- 1. **Precaution**: Wear appropriate PPE (gloves, labcoat, goggles or face shield) and prepare solution in a well-ventilated fumehood. Always prepare aqua regia solution in clean, dry glassware as it reacts vigorously with organic contaminants.
- Aqua Regia Preparation: Aqua regia solution should always be prepared fresh and never stored, no more than 100 mL should be prepared each time. In a clean beaker, add HCl (3 molar ratio). Add HNO₃ (1 molar ratio) slowly to HCl to give a yellow fuming solution of Cl₂ (g). Always add HNO₃ slowly to HCl and not the other way around. If conc. HCl (37 %) and conc. HNO₃ (65 %) is being used, the volume ratio would be 4 parts conc. HCl : 1 part conc. HNO₃.
- 3. **Disposal**: Pour leftover aqua regia solution into an open container filled with a bed of ice and neutralize gradually with saturated sodium bicarbonate solution until pH is neutral. This might take more than a few hours. Neutralized solution should then be disposed in a separate waste container and never into the normal waste container. This is to prevent any residual aqua regia solution from reacting with organics present in normal waste container. Label the waste clearly as nitric/hydrochloric waste and manifest as soon as possible.

G. Piranha Solution:

Acidic piranha solution is an extremely acidic, self-heating and oxidizing cleaning agent commonly used to remove organic residues. It should be only used as a last resort and is rarely necessary in an organometallic lab.

- 1. **Precaution**: Wear appropriate PPE (gloves, labcoat, goggles or face shield) and prepare solution in a well-ventilated fumehood. Always prepare piranha solution in a clean, dry glassware as it reacts vigorously with organic contaminants.
- 2. Piranha Preparation: Piranha solution should always be prepared fresh and in small quantities (example: for cleaning frits). In a clean beaker, add H₂SO₄ (3 molar ratio). Add cooled 30 % H₂O₂ (1 molar ratio) slowly to H₂SO₄ and beware of the solution heating up (can reach > 100 °C!!). Always add H₂O₂ to H₂SO₄ right before use. Once the solution is stabilized, it can be used. DO NOT MAKE MORE THAN 5 mL of piranha at any given time!!
- 3. **Disposal**: Allow piranha solution to cool down overnight in a clearly labelled beaker in a fumehood. Neutralize gradually with sodium bicarbonate until the pH reads neutral. Neutralized solution should then be disposed in a **separate oxidizing waste container** and never into the normal waste container. This is to prevent any residual piranha solution from reacting with organics present in normal waste container. Label the waste clearly as oxidizing acid waste and manifest as soon as possible.

By signing below, you indicate that you have read and understand the content of this document.

Name: _____