Standard Operating Procedures
Bates Laboratory
ChemSci 605

In case of emergency: **From cell phone dial 911**
From Phone in Hallway dial 911

Basic safety rules for the lab

1. Eye protection must be worn in the lab at all times. Safety glasses (the lowest form of protection allowed) are available on the back of the door upon entering the lab. If there is a splash hazard, goggles must be worn. Do not wear contact lenses in the lab.
2. NO FOOD or DRINKS are allowed in the lab.
3. Wear closed toe shoes, no sandals.
4. No open flames; see me if you need to use an open flame.
5. The fire extinguisher, emergency shower, and eyewash station are located by the main Lab door. Do not leave anything on the floor that could prevent you from reaching emergency equipment.
6. Check glassware before using it, especially look for star cracks (they are hard to spot, but are likely to break during a reaction by a magnetic stir bar or implode when subjected to vacuum when placed on a rotary evaporator.
7. Wear gloves when working with chemicals in the lab. DO NOT touch door handles, computers, books, or other personal items wearing gloves that you have used handling chemicals. Remove used gloves before leaving the Lab.
8. Chemical waste must be placed in the disposal containers located in the back of the lab. Do not put any chemicals or glass in the wastebaskets. Place broken glass in the broken glass container (by the waste containers).

STANDARD OPERATING PROCEDURES (v 1.0)

1. Glassware:

1.1. Assembling glassware:

1.1.1. Use the minimum number of clamps needed for support, making sure:

1.1.2. Attach clamps to a vertical support bar.

1.1.3. Make sure no torque is applied by the clamp. In other words, the pieces of the set up should be in their natural location when the clamp is tightened and they should not be forced out their natural position as the clamp is tightened.

1.1.4. Make sure top-heavy apparatus is prevented from rotating and tipping.

1.1.5 Make sure any hanging pieces are clamped - grease will not hold them against the force of gravity!

1.2 Washing glassware

1.2.1 Washing glassware in an alkaline bath

1.2.1.1 Prewash glassware with detergent/water and acetone.

1.2.1.2 Preparation of the alkaline bath:

WARNING: The minimum protective equipment required for this procedure is: safety GOGGLES, purple short gloves AND large heavy duty 'rubber' gloves (the current ones in use are green colored), and a lab coat.

1. Dissolve 250 g KOH in 1 L distilled water
2. **Slowly** add this aqueous solution to the polypropylene bath containing 8 L isopropanol.
3. This bath should be covered when not in use to prevent concentration by evaporation. The bath should be useful for many washing cycles if the glassware is somewhat cleaned before going into the bath.
4. To use the bath, lower the glassware into the bath completely submersing it and allowing it to fill with solution. Leave in the bath for several hours to overnight.. Drain the glassware into the bath (tongs) and then rinse the glassware with distilled water. Rinse with acetone and place it the drying rack to dry.
5. Bath maintenance: When the cleaning activity decreases, discard the bath—to deactivate, add dilute HCl VERY slowly to the alkaline bath to neutralize the KOH. Check progress with pH paper. After neutralization, pour the solution down the drain (if it contains less than about 25% alcohol).
6. Cautions:

Glassware will be etched by prolonged exposure to the solution. Do not place any of the following in the bath: volumetric glassware, stopcock keys, anything with rubber, glass frits and filters, anything with grease still on it.

1.2.2 Washing glassware with an acid bath

1.2.2.1 Prewash glassware with detergent/water and acetone.

1.2.2.2 Preparation of the acid bath:

WARNING: The minimum protective equipment required for this procedure is: safety GOGGLES, purple short gloves AND large heavy duty 'rubber' gloves t(he current ones in use are green colored), and a lab coat.

1. To the polypropylene acid bath container, add 900 mL distilled water then carefully add 100 mL conc HCl.
2. This bath should be covered when not in use to prevent concentration by evaporation. The bath should be useful for many washing cycles if the glassware is somewhat cleaned before going into the bath.
3. To use the bath, lower the glassware into the bath completely submersing it and allowing it to fill with solution. Leave in the bath for several hours to overnight.. Drain the glassware into the bath (tongs) and then rinse the glassware with distilled water. Rinse with acetone and place it the drying rack to dry.
4. Bath maintenance: When the cleaning activity decreases, discard the bath—to deactivate, add dilute HCl VERY slowly to the alkaline bath to neutralize the KOH. Check progress with pH paper. After neutralization, pour the solution down the drain (if it contains less than about 25% alcohol).
5. Cautions:

   Glassware will be etched by prolonged exposure to the solution. Do not place any of the following in the bath: volumetric glassware, stopcock keys, anything with rubber, glass frits and filters, anything with grease still on it.

2. Common Hazards

2.1 Metals

2.1.2 Lithium: metallic Lithium should never be placed under a nitrogen atmosphere. A violent and highly exothermic reaction can occur when Li metal contacts N_2.

2.2 Alkylating agents: Some of these are the original chemical warfare materials. Exercise extreme caution using these reagents. Drops on the skin may cause blistering. Inhalation may cause fluid to fill the lungs and, in extreme cases, may

cause tissue scarring or death. Examples include methyl iodide, methyl trifluromethylsulfonate, but there are many others.

2.3 Oxidizing agents: Oxidants may react violently with organic compounds. Be particularly careful to store oxidizing agents ONLY in the shelving labeled OXIDIZING AGENTS and also in disposing of solutions containing (or potentially containing residual oxidizing agent). If a waste material may contain an oxidant, DO NOT place it in the organic waste container. Put in a separate container and see about what to do with it. Some examples of oxidizing agents are: Household bleach (we use it often in the lab both as a reactant and for cleanup of thiol-containing reaction flasks), peroxides, nitrates, chlorates, persulfates, bromine, chromium (VI) salts, and others. If you are unsure about the nature of a reagent, look it up and/or see me.

2.4 Poisons: Thallium salts, mercury salts, tin reagents, sodium or potassium cyanide, and others require special care in handling and are NOT to be used in the lab without talking to me AND receiving special training.

2.5 Pyrophoric materials: $LiAlH_4$ (lithium aluminum hydride) when finely powdered may spontaneously ignite (usually while attempting to weigh it. This is especially likely in the summer during high humidity.

2.6 Combustion through static electricity discharge:

2.7 Liquid oxygen: we do not use liquid oxygen in the lab but it can be condensed in vacuum flasks containing liquid nitrogen open to the air. If you see a blue coloration in the bottom of a Dewar containing liquid nitrogen, that is liquid nitrogen and it is highly dangerous. Walk away, and stay away long enough for all the liquids to evaporate.

3. Operating in the hoods

3.1 Before setting up an experiment in a hood, you must remove all solvent containers, stoppered flasks, paper of any kind, or anything else that can burn or become a projectile in the event of an explosion.

4. Spills

4.1 If you spill a liquid on the bench, immediately soak it up with paper towels and, if it is volatile, transfer the towels to the hood. Inform your instructor as to the nature of the spill in case further action is warranted.

4.2 If concentrated acid is spilled, add sodium carbonate or bicarbonate, solution or solid. If concentrated base is spilled, add dilute and/or weak acid (e.g. acetic). Indicator solution or paper will be available in the lab. If your skin (or clothing) comes in contact with the spill, immediately flush the skin or clothing with water for 15 minutes.

4.3 Should you spill bromine solution anywhere, treat the spill immediately with sodium thiosulfate solution.

5. Heat Hazards

5.1. Never (never, ever!) heat a closed system! Pressure will build up and cause the glass to fail, sending projectiles of glass in all directions.

6. If There is a Fire

6.1. In the lab where you are working:

6.1.1. Shout "fire" to alert anyone working nearby.

6.1.2. A small fire in a test tube or other container can usually be extinguished by covering the container with a watch glass or book. If the fire cannot be extinguished by one extinguisher or by sand or water, you will be instructed to evacuate, following the procedure in b).

6.1.3. If a person's clothing is on fire, wrap the person in a lab coat, fire blanket, or whatever is handy to exclude oxygen. If the person runs, the flames will be increased by increasing the supply of oxygen. It must be smothered.

6.2. Elsewhere in the building (fire alarm sounds):

6.2.1. Extinguish any flames and turn off electrical equipment.

6.2.2. Exit Lab 605, turn left in the corridor and *walk down the stairs* to exit the building. Do NOT use the elevators. The elevator shaft acts like a chimney and draws smoke (and depletes oxygen) in the elevator. Move away from the building and stand near other people you know (if any) to be available if emergency personnel have questions about anyone who may still be in the building.

7. Pressure Hazards:

7.1. Never heat a closed system.

7.2. When using a separatory funnel, vent frequently and remove the stopper immediately upon setting it upright for separation.

7.3. Compressed gas cylinders must be strapped to the bench above their center of gravity when the protective caps are off. Pressure regulators are generally not interchangeable between gases for safety reasons and some have reversed threads so they are removed from the tank by turning in the opposite (CW) direction. Gas cylinders should be free of regulators and protected by their cap before moving.

8. Waste Disposal

8.1. In order to minimize damage to the environment, and in compliance with State and Federal law, chemical wastes must be carefully labeled as to their contents. Please read and follow the labels on the waste bottles to ensure that your chemical wastes are treated safely and appropriately. You will find a container for solvent waste at the back of the lab near the hood. NEVER place a waste solvent containing any oxidizer (hydrogen peroxide, for example) in the waste solvent container. If you have any questions about how to dispose of any waste materials, see D. Bates.

Resources used:

I have discussed this document with the individual whose name appears below.

on _____ _____

This document has been discussed with me on this date _____ by the individual whose name appears above and I have been given a copy of the document to place on the inside back cover of my research notebook for reference.

Signature: _____ Print name: _____