Shaughnessy Group Safety Information and Policies.

Welcome to the group. Before you start working in the lab, I ask you to read through the following procedures carefully. These will provide information on how to safely carry out the important tasks that are common in our group. While this may seem like an overly long and pedantic set of procedures, it is important that we all know and operate by the same rules. These procedures will help all of us be successful and safe in the lab. Please keep this with you for future reference.

General Safety

Safety must be the first priority when working the lab. While it is unfortunate if an experiment fails, it is disastrous if someone is injured. The following safety rules must be followed at all times.

a) Before beginning work in the lab, new workers must undergo departmental safety training as well as discuss specific procedures with Dr. Shaughnessy or a designated safety training officer.
b) In addition to reading these procedures, new workers should read the lab chemical safety plan, and the ACS lab safety handbook.
c) Glasses or safety glasses or goggles must be worn at all times in the lab.
d) Proper protective clothing should be worn at all times. Open toed shoes should be avoided. Long pants or lab coats should be worn when there is a danger of being splashed by any chemicals.
e) Gloves should be worn when handling chemicals. Note that latex gloves provide only a minimal barrier to many organic chemicals. Heavy neoprene or butyl rubber gloves are necessary to provide a reasonable level of protection. Even so, latex gloves are better than no protection. They should be changed as soon as contaminated, however.
f) Food and beverages must not be kept or consumed in the lab.
g) Make sure you know the location of fire extinguishers, safety showers, and eye washes and how to use them in case of an emergency.
h) If you are injured in lab seek immediate help. For minor injuries, use the first aid kit located in the lab. If you think you need medical attention, go immediately to the Russell Student Health Center at the intersection of Hackberry and University Ave. Have someone help you go to the health center. If the injury is very serious, or you can't get to the health center call 911 from the phone outside the lab.
i) Avoid working alone in lab. In particular, do not do any potentially dangerous (high pressure, highly flammable or toxic reagents, large volumes of solvent) when working alone. If you find yourself alone and must do a potentially dangerous procedure, notify someone in a neighboring lab so they can help you if necessary.

Below are listed specific procedures for dealing with common laboratory tasks as well as more specific safety reminders.

Use and Disposal of Chemicals:

As a synthetic lab, we use large amounts of chemicals and solvents. Therefore safe handling and disposal of chemicals is critical to lab safety. In addition to the procedures in the ACS lab safety handbook, the following policies must be followed.

a) *Inventory*. When a new chemical is purchased, it should be entered on the new chemical log for entry into the inventory. Similarly, when chemicals are consumed and will not be replaced, they should be entered on the old chemical log.
b) *Storage*. In as much as possible, the chemical storage guidelines in the ACS lab safety handbook should be followed. Storage locations are identified as follows in the inventory: FC-1, flammable cabinet room 43; refrig/freezer room 45; box, drybox

in room 41; hoods are numbered 1-4 starting with the south (left upon entering the room) hood in room 43 and working north to room 45; benches are numbered in the same manner. Chemicals are segregated as follows. Large (>1L) amounts of flammable solvents are stored in FC-1. Inert inorganic compounds are stored on top of FC-1. Smaller amounts of organic chemicals are stored on bench-3 and organized by number of carbons. Inorganic chemicals are stored on bench-4. Acids are stored under hood-3. Chemicals are stored in the refrigerator/freezer and drybox as necessary. Check inventory for location.

c) All chemicals must be clearly labeled with their full name (no abbreviations) and their hazards if they will be stored long term.

d) No chemicals except water, brine, small amounts of ethanol, and diluted acids and bases should be allowed to go down the drain. For general lab waste, the group maintains several collection jugs in which unwanted chemicals are accumulated. Waste should be segregated as follows: halogenated and non-halogenated chemicals are kept in separate containers; strongly acidic solutions should never be mixed with organic waste and is kept is a separate container (**NITRIC ACID SOLUTIONS MUST NEVER BE MIXED WITH ORGANIC CHEMICALS. THIS IS A SIGNIFICANT EXPLOSION HAZARD**); solid waste (silica gel, alumina, magnesium sulfate) must not be placed in the trash and is collected in a container for disposal. Large amounts of specific wastes should be collected by themselves. All containers containing unwanted chemicals must be labeled with an unwanted chemical label that indicates the contents (approximate) of the container. The label must be attached as soon as any waste is placed in the container. Unwanted chemical containers must be kept sealed except when adding chemicals. It is against the law to leave these containers open to the atmosphere. When the container is filled, it should immediately be taken to the flammable cabinet in the stockroom.

e) Always assume that a chemical is hazardous to your health, unless it is clearly innocuous (i.e. water, NaCl, bicarb, etc). If at all possible use volatile chemicals in a hood or in a vented reaction flask. Always take measures to avoid contact with chemicals. If you are exposed to a chemical wash the affected area immediately. In cases of extreme exposure remove any affected clothing and use the safety shower.

f) MSDS data sheets are available for all chemicals used in the lab. These are maintained in the lab, by Lisa Cox in the stockroom, and by the office of Environmental Health and Safety.

Handling of Cryogens:

We commonly use liquid nitrogen and dry ice in this lab. Cryogens can be safely handled by following these precautions.

- Liquid Nitrogen

a) **Liquid nitrogen can cause serious burns upon contact with your skin.** Liquid nitrogen should only be stored in Dewar flasks. Pouring liquid nitrogen into Dewar flasks, particularly those at room temperature, will result in significant splashing. Be careful to avoid prolonged contact with liquid nitrogen. It is particularly dangerous to have loose clothing, (i.e. sleeves, gloves, or open shoes) in which liquid nitrogen can become trapped against your skin.

b) Do not allow flasks or traps which are being cooled with liquid nitrogen to remain open to the atmosphere. Liquid oxygen, a major fire and explosion hazard, can condense. Only flasks which have are under vacuum or pure nitrogen should be placed in liquid nitrogen baths.

c) When allowing flasks to warm to room temperature after being in a liquid nitrogen bath be careful of pressure build up. In particularly, be careful when condensing volatile solvents or gases. It is best to vent the flask to a nitrogen line or the atmosphere while it warms up. If this cannot be done, make sure the flask is completely evacuated at liquid nitrogen temperature and then sealed. Use a flask

designed for high pressure, such as a bomb with a Teflon seal. Avoid ground glass joints as these are not designed for pressures much higher than 1 atmosphere.

- Dry Ice
    a) While dry ice is not as dangerous as liquid nitrogen, it can still cause serious burns after prolonged skin contact.
    b) Dry ice must never be kept in a closed vessel. Pressure will develop causing an explosion.
    c) When using dry ice baths, be careful when adding dry ice to the bath, especially when the bath liquid is warm. Significant foaming and splashing will occur if you are not careful. Slowly add small pieces of dry ice until the bath no longer bubbles vigorously upon addition. Then you can add larger amounts of dry ice.

Handling glassware.

a) **Dewars**. Dewar vessels have a strong internal vacuum that can cause them to violently implode if mistreated. Dewars should always be shielded with a metal or plastic screen or by covering in electrical tape. Treat dewars gently. Do not scratch or bump them. Tall dewars should be stored laying down so that they do not fall over and explode. It is critical that safety glasses be worn whenever handling a dewar (you should be wearing your glasses anyway). Do not expose dewars to large temperature extremes (i.e. do not pour hot water in a dewar that is at liquid nitrogen or dry ice temperature).
b) When placing hoses on tubing or hose barbs on condensors, schlencks etc, use caution. If the hose does not go on easily, use a little grease to ease the hose onto the glassware. Forcing the hose onto the glassware could result in breakage and you getting cut.
c) Ground glass joints should always be lightly greased to avoid them becoming stuck together. In general we use fomblin teflon grease to avoid contamination of reaction products.
d) Never use broken, chipped, or cracked glassware. Check round bottoms and other flasks for small star cracks, especially before carrying out operations under pressure or vacuum. All broken glassware should be discarded (see below) or taken to the glass blower for repair.
e) Broken and used disposable glassware must be discarded in the broken glassware boxes in each lab. These items must never be placed in the trash.

Carrying out reactions.

a) Always label your reaction with starting materials and solvents and a notebook page number so that if something goes wrong when you're not around people will know now to deal with it.
b) Never heat a reaction in a sealed glass flask above the boiling point of the solvent, unless it is designed for this (i.e. a glass bomb with a Teflon valve). When carrying out these type of operations do so in the hood behind a blast shield or with the sash lowered in case of an explosion.
c) Be careful when scaling up reactions which you have successfully carried out on a small scale. From personal experience I can tell you that large reactions do not always behave the same as small reactions. Large scale reactions are generally run under more concentrated conditions also heat transfer can become a major problem. In large scale reactions which are potentially exothermic (even if you didn't notice one in the small scale reaction) or are run at low temperatures it is best to slowly add one reagent to the other. In this way you can control the reaction before it gets out of control. If you have a reaction that appears to be getting out of control (getting hot, excessive gas evolution) try to cool the reaction down--if you are not using a cooling

bath it is a good idea to have a water bath handy just in case. Very large scale reactions (1 L or more solvent) should be run in a hood if at all possible. Also place a tray under the reaction just in case there is a spill. **Never run a reaction for the first time in very large scale, even if it was done so in a reference. Make sure you can repeat the synthesis before wasting large amounts of material.**

d) **ALL WATER HOSES MUST BE SECURELY CLAMPED ON ANY REACTION THAT IS NOT CONSTANTLY MONITORED BY YOU**. There is no excuse for causing a flood because a hose popped off of a condenser. The hose should be secured with wire or a nylon tie. Furthermore, ensure that the hose is the appropriate size for the hose barb (the hose should go on with some difficulty) and that the hose is in good condition. It is not necessary to use a high flow rate with condensors. A flow that provides a steady, slow stream from the outlet is more than sufficient for all reflux and distillation applications.

e) Use of gas cylinders. When using gas cylinders they should always be firmly attached to a bench or other support. **Never move a gas cylinder unless the protective cap is in place.** If a cylinder were to fall over when the cap is removed, the valve can be snapped off creating a large, heavy, and deadly projectile. When moving cylinders more than a few feet use a gas cylinder cart, which is available from the stockroom.

f) When using gases above atmospheric pressure, be sure that the connections are appropriate for the pressure at which you are working. In general, swage lock fittings with either copper or stainless steel tubing should be used for all high pressure applications. For lower pressure uses (1-2 atm), reinforced tygon tubing is acceptable. Be sure to firmly secure all hose connections to avoid leaks.

g) Be careful when using needles and cannulas. Obviously these are sharp and can cut or puncture your skin. Disposable syringes and needles must be discarded in the sharps containers located in each lab. These must never be placed in the trash.