

Basics of Laboratory Safety: Chem 5a Lab 2003

LABORATORY RULES AND REGULATIONS

1. Safety glasses, goggles, face shield, and shoes (no sandals) must be worn at all times. Normal prescription lenses are insufficient! ***Violations will not be tolerated. Two violations will result in a failing grade and expulsion from the laboratory.***
2. Work is NOT permitted in the laboratory outside of the designated laboratory hours (see syllabus). (Special Note: You cannot be in the laboratory during Ch 4 hours). You must never work in a laboratory alone!
3. Keep your work space clean and tidy. The working space, desk drawers, cabinets, and instruments must be kept neat and clean at all times. The permission of a TA *must* be obtained before equipment is left assembled between laboratory periods. *All* equipment must *always* be disassembled at the end of the Monday afternoon section so the Ch 4 students can use the hoods on Tuesday.
4. Use common sense and do not rush in the laboratory. Never be complacent about chemicals or chemical reactions.
5. The execution of unauthorized experiments is not permitted and may result in the automatic expulsion from the course with a failing grade. Bringing any chemicals and/or glassware out of the laboratory for personal or professional use will be treated similarly.
6. Common sense and consideration for fellow workers must be exercised rigorously and constantly.

These rules will be enforced under the Honor System.

SHARED EQUIPMENT:

In order to expedite your efforts to accomplish the synthesis of estrone, the standard-tapered glassware and other equipment which you will need has been sorted and stored in bins located in the laboratory. At the start of each laboratory session, you should avail yourself to whatever apparatus you need. When finished, rinse out the used glassware with water and/or acetone and place it in the “dirty dishes” bins. Make sure all chemical residue is removed from the glassware before placing in the bins. Replace other equipment (thermometers, heating mantles, oil baths, etc.) in its proper location. **NOTE: Thermometers should NEVER be placed in the “dirty dishes” bins!** Please respect the signs posted above bins.

For such a scheme to work, it is important that the inventory of glassware remain essentially constant. **Thus, reaction mixtures and products should not be stored in standard tapered glassware.** Such glassware being in limited supply, must remain communal. Furthermore, any breakage which occurs must be brought to the attention of the teaching assistants immediately. As was the case in Ch 3a, such items will be noted on the student's internal material transfer form and if such losses exceed \$100 during the quarter, the overrun will be charged to the

student's account. The failure to report such damage will be considered a violation of the honor code.

WASTE:

In accord with the rules with regard to waste disposal, there are several protocols which must be observed.

1. If you are the first person to put waste into a container, you must obtain a waste report from the TA, fill it out completely, and attach it to the container.
2. Whenever you add waste to an old container, you must check the date on the tag. If the container is 3 weeks old, it must be removed. Report it to the TA, and the container will be replaced with a new one.
3. Clean solvent waste (i.e. from the rotovap traps) should be placed in the RED solvent waste can in the back of the laboratory.
4. Other liquid waste will be sorted as organic or aqueous and put into appropriate waste containers in the waste hood in the back of the laboratory.
5. Used silica gel will be dried in the student's hood by blowing air through the column. Once dried, it will be poured into silica waste containers in the waste hood.
6. Palladium on carbon/celite waste will be poured into a waste container with water such that the palladium is always covered with water.
7. Other solid chemical waste (including heavily contaminated towels) will be disposed of in canisters in the waste hood.
8. Normal trash can be thrown into the gray waste bins.
9. **If you are unsure how to dispose of something, ask a TA!**

SAFETY:

Work in the synthetic chemistry laboratory involves the use of inflammable solvents, some corrosive and toxic chemicals, and apparatus which, if used improperly, can cause minor to severe injury. **All work with solvents and chemicals must be performed in the fume hoods NOT on the benchtop.** *Safety glasses and shoes must be worn at all times while in the laboratory.*

A. Solvents

1. Never heat inflammable solvents, even small amounts, with or near a flame. As for refluxing or distillation, never place solvents in an open beaker. Pouring solvents in the vicinity of a flame is extremely hazardous. Use an oil bath, steam bath, water bath, heating mantle, or hot

plate as a heat source whenever possible.

2. Ethyl ether and petroleum ether (bp 30-60°) are especially dangerous. Never heat them on a hot plate; always use a water or steam bath, and collect the distillate in an ice-cooled flask. In the case of ethyl ether, the receiver should be a filtering or distilling flask connected to the condenser with a cork and with a piece of rubber tubing leading from the side tube on the flask to the floor. This allows the heavy ether vapors to spread along the floor instead of the desktop where they may be ignited by burners. Carbon disulfide is extremely hazardous. It has been known to ignite from hot steam pipes or electrical sparks, as from the thermostat on a hot plate, or the motor on a stirrer.
3. If an inflammable solvent is spilled, have all workers at the desk turn off their burners and clean it up immediately using a cloth. First notify the TA. Wring the solvent from the cloth into the solvent waste can and then rinse the cloth in the sink with much water. *Use gloves.*
4. If acetone is used to aid in drying glassware, use it sparingly and not near a flame.
5. Inflammable solvents which you may have contact with are: ether, ligroin (petroleum ether), cyclohexane, toluene, xylene, alcohols, ethyl acetate, carbon disulfide, acetone, dioxane, etc. If in doubt about the inflammability of a solvent, assume that it is hazardous.
6. Benzene and chlorinated solvents are toxic. In some cases, the toxic effect is cumulative. Avoid contact with the skin and inhalation of solvent vapors.
7. Many organic solvents freely permeate latex gloves commonly used in laboratories, and are therefore inadequate protection of the skin from solvent vapors. Thicker neoprene or butyl rubber gloves are recommended.

B. Chemicals

1. Especially corrosive substances which give off noxious fumes (e.g., bromine, acetyl chloride, benzyl chloride, phosphorus trichloride, acetic anhydride, fuming nitric and sulfuric acids, chlorosulfonic acid, benzene sulfonyl chloride, etc.) should be handled in the hoods. Use proper gloves. Do not spill these chemicals on yourself or on the desktops. They will cause very painful burns. Bromine is especially bad. Do not put any of these in organic waste cans.
2. Over the last several years a number of organic compounds have been confirmed as carcinogens and the list is steadily growing. *It is best to assume that all chemicals are toxic, and possibly carcinogenic.*
3. Sodium and potassium metals react explosively with water. They are rapidly corroded by the atmosphere and should be stored in kerosene or oil. These metals should not be allowed to come into contact with the skin. They may be handled with dry filter paper or tweezers. Unused pieces of metal may be destroyed by dropping into 95% ethyl alcohol, or they may be returned to the bottle. Avoid all contact between chlorinated solvents and sodium or potassium.

4. Concentrated acids and alkalis are corrosive to the desktops, clothing, and skin. If there is a spill, first notify the TA. Dilute with a large volume of water. If an acid, neutralize with solid sodium bicarbonate; and if a base, with 3% acetic acid. Sulfuric acid is troublesome, since drops adhering to the tops of bottles tend to absorb moisture and run down the outside of the bottle.
5. Mercury and its vapor are poisonous. Avoid spilling, and notify the TA if any has been spilled. The Institute has special facilities for cleaning up mercury spills.

C. Apparatus

1. Approved safety glasses, goggles, or a face shield must be worn at all times when in the lab. Normal prescription lenses are insufficient due to the possibility of explosion.
2. When inserting tubing or thermometers into bored stoppers, it is wise to take some simple precautions. The tubing and stopper should be held by a towel, so that if the tubing breaks the towel will reduce the impact of the jagged edge. If the tubing does not enter the hole in the stopper easily, the hole may be made larger with a file (if a cork) or lubricated with water, alcohol, or glycerine. Hold the tubing close to the stopper. In removing tubing from stoppers, follow the same technique. **Very serious cuts have resulted from carelessness in inserting tubes in stoppers.**
3. Closed systems are liable to explode if heated. *Never* carry out an atmospheric pressure distillation in a closed system.
4. Do not support apparatus on books, boxes, pencils, etc. Use large, strong wooden blocks, rings, or lab jacks. Assemblies with a high center of gravity (as when a reagent is added through the top of a condenser) should be assembled and operated with much care.
5. Use glass-stirring rods with care for breaking up solids. They are liable to break.
6. Do not evacuate Erlenmeyer flasks larger than 50 mL (except filtering flasks). They may collapse.
7. Oil baths and melting point baths can cause severe burns if spilled. Make sure they are well supported. *Be especially careful not to get water into oil baths.* We will use electric heating mantles in preference to oil baths when possible.
8. Dewar flasks and vacuum desiccators, because they are evacuated, implode easily when tipped over or dropped. Make sure the ones you use are wound on the outside with friction tape or are contained in protective shields, so they will not shower glass around the laboratory if broken.

D. Accidents

1. *Fire.* Personal safety is most important. If a person's clothing catches on fire, he/she needs help. Prevent him/her from running. If he/she is close enough, put him/her under the safety

shower because it is more effective than a blanket. If not, make him/her lie down and smother the flames by rolling, wrapping with lab coats, blankets, towels, etc. *Never turn a carbon dioxide extinguisher on a person.*

If a fire breaks out, turn off all burners and remove solvents if time allows. There are carbon dioxide extinguishers in the laboratory and the positions and operation of these should be known. Point the extinguisher at the base of the flames. Very small fires can be put out with a damp towel by smothering. Only after the safety of all is assured should the matter of extinguishing the fire be considered.

Because a few seconds delay can result in very serious injury, every person in the laboratory should plan in advance what he/she will do in case of such an emergency.

2. *Chemicals.* If corrosive chemicals are spilled on the clothing, immediate showering (with clothing on) is the best remedy. Safety showers are located by each door. If chemicals are spilled on the skin, wash them off with large volumes of water. Bromine should be washed off with water and the skin then massaged with ethanol or glycerine. Do not apply a burn ointment. If the chemical is spilled in the eye, it should immediately be washed out thoroughly with water using the eyewash sprayer in the sinks. If acid was involved, a weak solution of sodium bicarbonate in an eyecup should then be used. If a base, boric acid is effective.

If corrosive chemicals are spilled on the desk, dilute them with a large volume of water and then neutralize with sodium bicarbonate if an acid, or dilute acetic acid if a base.