Laboratory Policies for Organic Chemistry

The organic chemistry laboratory is a unique environment for learning. In contrast to previous experiments you have performed, organic experiments tend to take more time, be more uneventful, proceed without flashy color changes, and have notoriously poor yields. But organic reactions have a charm of their own, and once you've spent some time in the organic lab, you will come to appreciate the complexity, beauty, and subtlety of what happens. Experiments are important in organic chemistry because they teach ideas and skills that are not always developed in lecture, such as good technique, accuracy in measuring and routine techniques, cleanliness, timeliness, safety, good communication and working with peers, study skills, analysis and questioning of data, and the importance of preparation. This handout is an overview of what to expect from your lab experience in organic chemistry. Your instructor may supplement this general handout with information more specific for your class.

Safety in the Organic Laboratory

Because of the nature of organic chemicals, there is more potential for danger than in other labs: they tend to evaporate faster so you inhale more, they can be absorbed quickly through the skin, they tend to be much more flammable, and they are sometimes more stubborn to remove or wash away. Some compounds used are toxic or carcinogenic or reproductive hazards; health dangers for many other compounds have simply not been studied. Because of this, safety is of utmost concern in the organic lab, and those who teach organic chemistry have placed safety as the single most important lesson in your lab experience. Luckily there are more safety features present in the organic lab to keep you away from harm. You will be shown these safety features and you will have an opportunity to discuss the safety rules that govern everyone's work in the lab. If you are unable to follow the safety rules, you will be dismissed from the laboratory. Your instructor can remove anyone from the lab if they endanger the health or safety of others. However, safety begins *before* the lab experiment with research of hazards that you might encounter. All labs come with reading and assignments to complete before the lab, and ***you will not be allowed to complete lab experiments if you have not prepared***.

Working in the Laboratory

You must complete labs during your scheduled time; do not ask to attend another section to make up a lab. Due to our lab set-up and take-down procedures, ***there are no make-ups for missed labs***. Show up on time. If you arrive late to lab you may be turned away and will not be allowed to complete the experiment. You will perform experiments with a partner, and if you miss a lab then your partner will have to work alone. Missing lab makes life more complicated for you, them, and your instructor. You will be assigned to work in a specific fume hood with two drawers of glassware and cabinets containing other equipment. You do not share lab drawers, so your lab group is the only one using a given drawer. If you ever break, crack, or chip a piece of equipment, it will be replaced. You will not be charged for replacement items, so do not use broken glassware in experiments (doing so poses additional hazards). Each day, wipe down your hood, leave your drawer neat and organized (samples should be contained), leave the community cabinets with the proper equipment, turn off all equipment, and leave the community areas (sinks and benches) free of chemicals, glassware, and garbage. ***Messiness will be penalized!***

Keeping the Laboratory Notebook

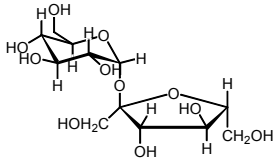
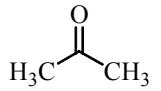
You will keep a laboratory notebook as a record of your scientific investigations, a place where you record all hazards, procedures, data, observations, calculations, and thoughts about the experiments. The notebook is your opportunity to write intelligently, succinctly, and gracefully, and the notebook grade will depend on your ability to communicate ideas. To improve your writing if necessary, your instructor is always available to review your notebook before it is due, and also Harper College has an excellent writing center that exists to assist students in their writing. ***If you write poorly about a topic, it will be assumed that your***

understanding of that topic is poor as well. If English is not your native language, it is even more imperative that you avail yourself of these resources and practice your writing until it reaches high quality.

The notebook should be new and have permanently-sewn pages. In general, spiral-bound or perforated-page notebooks are unacceptable. Label your name and course on the cover, number the pages, and reserve the first page for a table of contents. Your instructor will show you examples of acceptable notebooks and how to properly format them. Writing should always be in blue or black ink, and although the notebook is not expected to be neat and error-free, the entry for each experiment should always be contained together and organized well enough to quickly find information. Begin each experiment at the top and on the front of a clean page, and include the date any entries are made. Include also the name of your lab partner(s) you work with that day. Mistakes should be crossed out with a simple line (never any scribbles or correction fluid). ***Your instructor will sign your lab notebook entry when you leave each day; penalties may be assigned for using the wrong format, unclear writing, or disorganization.***

In general, each experiment consists of three parts, one completed prior to coming to lab (the **Pre-Lab**), one completed while in the lab (the **Data**), and one completed after the lab (the **Analysis** or **Report**). The **Pre-Lab** consists of (1) an experiment title, (2) a statement of purpose, (3) a summary of safety hazards, (4) a reaction table (if performing a reaction), and (5) a summary of procedure. The last three are described below.

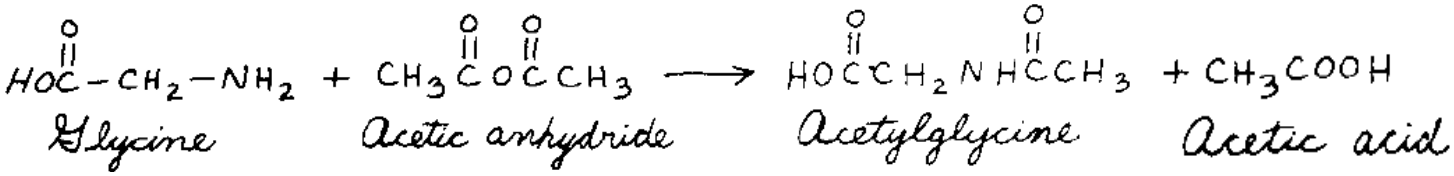
A *Material Safety Data Sheet* (MSDS) is a document which describes hazards associated with the use of certain chemicals. Companies are required by law to provide these documents with the chemicals they sell. The chemistry labs maintain an MSDS binder for all the chemicals used in that course. Alternatively, there are a large number of databases on the internet that provide libraries of MSDS sheets; some of the better ones will be provided by your instructor. You must become familiar with hazards of chemicals *before* you arrive to lab (chemicals you will be **<u>using</u>** and **<u>producing</u>**). Most of the information useful to you will be found in Sections 3, 4, and 9. Before you are allowed to work in the lab, you must summarize information from these MSDS sections. Your instructor may provide more specific requirements, but typical information summarized is shown below for two examples. On the following page is the general information that you must be familiar with.

Name and Properties (Section 9)	Health Hazards (Section 3)	First Aid (Section 4)	Disposal
Sucrose  white solid mp = 160-186EC MW = 342.3 g/mol	None given	Inhalation–fresh air Ingestion–drink water for large amounts Skin–not given Eyes–15 minute eye wash	Solid waste
Acetone  colorless liquid bp = 56.5EC d = 0.79 g/mL MW = 58.1 g/mol	Flammable Harmful Irritant	Inhalation–fresh air Ingestion–aspiration hazard; seek medical help immediately Skin–Flush with water Eye–Flush, get medical attention	Solvent waste
Provide chemical structure, description, and physical properties useful to you in the experiment.	***Summarize keywords that describe important health hazards.***	***Summarize First Aid for the four modes of chemical entry: inhalation, ingestion, absorption, or contact with eyes, plus any other important information***	***Where YOU will dispose of excess chemical***

Disposal of chemicals is of utmost importance in the organic lab. Unlike your previous chemistry courses in which many of your chemicals and solutions could be disposed of in the sink, ***nothing will ever be disposed down the sink in this lab***. There will always be two general waste containers available (**Organic Solvents** for general solvents) and (**Organic Solids** used for general solids), but other containers will be

discussed and set out as needed, in cases where there is special reactivity to consider. If you are ever unsure where to dispose of a chemical, ask your instructor.

A **Reaction Table** is to be included anytime you are performing a chemical reaction. You should include the overall reaction, showing structures for each compound involved. In a table, you should include molar mass data, masses involved in the experiment, and any other pertinent data you would need to calculate percent yield (densities, and so on). You should also include the steps (mechanism) of the reaction if possible. An example is shown (because organic reactions are on a smaller scale, millimole (mmol) is more convenient).

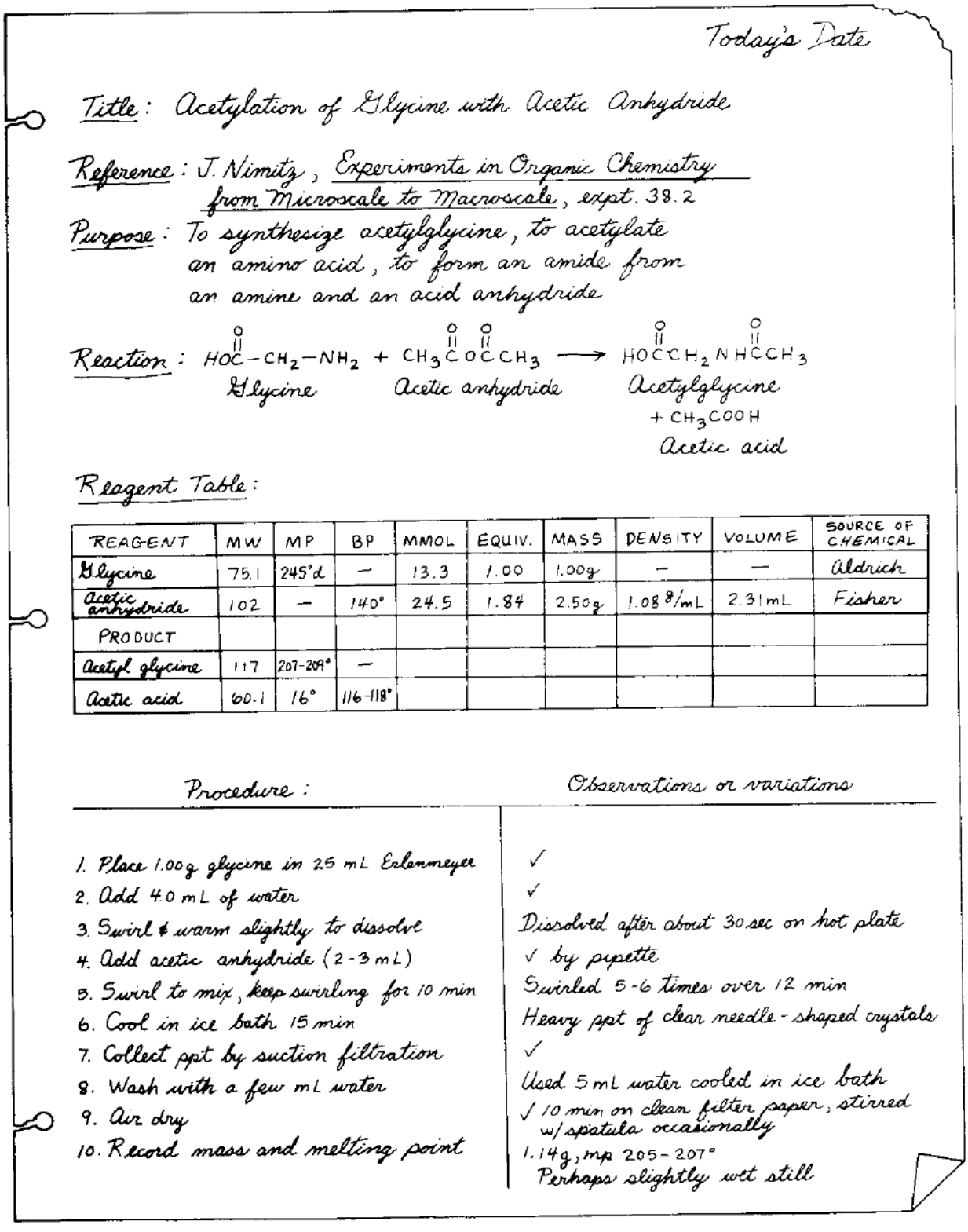


REAGENT	MW	MP	BP	MMOL	MASS	DENSITY	VOLUME
Glycine	75.1	245°d	—	13.3	1.00g	—	—
Acetic anhydride	102	—	140°	24.5	2.50g	1.08 g/mL	2.31mL
PRODUCT							
Acetyl glycine	117	207-209°	—				
Acetic acid	60.1	16°	116-118°				

A summary of your **Procedure** should be included. Some suggest that you write the procedure in a left-hand column, leaving right-hand space for last-minute procedural changes, notes, observations, and data. Do not copy the procedure verbatim, but summarize it with enough detail that you could perform the experiment with only your notebook. Sketch any unfamiliar equipment set-ups or diagrams you will need.

When you collect **Data**, ***record measurements and observations directly into your notebook, NOT on scrap paper to transfer later***. Your notebook is not supposed to be neat or without error. Your notebook must accompany you wherever you might take measurements or make observations, whether at the balances weighing chemicals, at the hood performing a reaction, or in the instrument room analyzing samples. Data must include appropriate numbers of significant figures, correct units, and descriptive labels. It is good practice to anticipate what data you will need to take before the lab and come prepared with appropriate places to record the numbers. Do not neglect to record observations, which includes smells, textures, colors and color changes, turbidity, release of heat or light or sound, change in temperature, size and crystal shape, formation of solids or liquids or gases, or whatever. The data section should be completed while you are in lab and must be ***checked and signed by your instructor before leaving. Data sections with no signature are not worth any points.***

The **Analysis** or **Report** will be your final write-up for each lab. Since all parts of a lab must be written together, you need to leave an appropriate number of pages for the analysis before beginning the Pre-Lab for the next experiment. This section is used to accomplish two things. First, you will neatly repeat and tabulate the data you obtained during the experiment. This will allow you to put all the data in one place for easy discussion and viewing. Also, this section is used for any calculations, discussion, assigned questions, or other conclusions. In general, each experiment handout includes this information, although your instructor may provide different (or additional) requirements. If you use any piece of data from the literature, remember to always cite the source you used. If you include computer-made graphs or spectra, these must be clearly titled and pasted (upright and not overlapping) into your notebook. Your instructor will discuss how labs will be collected and graded in your course, as well as any other requirements for experiments (such as a separate, typed report). (An example lab notebook page is shown next.)



Today's Date

Title: Acetylation of Glycine with Acetic Anhydride

Reference: J. Nimitz, Experiments in Organic Chemistry from Microscale to Macroscale, expt. 38.2

Purpose: To synthesize acetylglycine, to acetylate an amino acid, to form an amide from an amine and an acid anhydride

Reaction: $HOC(=O)-CH_2-NH_2$ + $CH_3C(=O)OC(=O)CH_3$ → $HOC(=O)CH_2NHC(=O)CH_3$ + CH_3COOH

Glycine — Acetic anhydride — Acetylglycine — Acetic acid

Reagent Table:

REAGENT	MW	MP	BP	MMOL	EQUIV.	MASS	DENSITY	VOLUME	SOURCE OF CHEMICAL
Glycine	75.1	245°d	—	13.3	1.00	1.00g	—	—	Aldrich
Acetic anhydride	102	—	140°	24.5	1.84	2.50g	1.08 g/mL	2.31mL	Fisher
PRODUCT									
Acetyl glycine	117	207-209°	—						
Acetic acid	60.1	16°	116-118°						

Procedure:	Observations or variations
1. Place 1.00g glycine in 25 mL Erlenmeyer	✓
2. Add 4.0 mL of water	✓
3. Swirl & warm slightly to dissolve	Dissolved after about 30 sec on hot plate
4. Add acetic anhydride (2-3 mL)	✓ by pipette
5. Swirl to mix, keep swirling for 10 min	Swirled 5-6 times over 12 min
6. Cool in ice bath 15 min	Heavy ppt of clear needle-shaped crystals
7. Collect ppt by suction filtration	✓
8. Wash with a few mL water	Used 5 mL water cooled in ice bath
9. Air dry	✓ 10 min on clean filter paper, stirred w/spatula occasionally
10. Record mass and melting point	1.14g, mp 205-207° Perhaps slightly wet still

Figure 2 A sample notebook page

From: J.S. Nimitz, Experiments in Organic Chemistry from Microscale to Macroscale, Prentice Hall, 1991.

Department of Chemistry Laboratory Safety Rules

"The probability of someone watching you is directly proportional to the stupidity of your action."
A. Kindsvater

DO…

…exercise caution and sound judgment at all times. Act like a mature college student.

…notify your instructor of any physical condition which may affect or be affected by this lab (asthma, chemical allergies, current medical prescriptions, epilepsy, color blindness, pregnancy, etc.).

…be familiar with procedures, chemicals, and potential safety hazards ***before*** coming to lab, and follow any additional instructions given that day.

…know the location of all safety equipment: goggle cabinet, phone, fire extinguisher, fire blanket, safety shower, eye wash, first aid kit, and exits.

…wear your lab goggles ***at all times*** in the lab!

…read chemical labels twice to ensure you are using the proper chemical for your procedure. Many chemicals have similar names, formulas, appearances, and properties.

…read chemical labels for safety and ask for assistance using unfamiliar equipment.

…treat all chemicals, equipment, and procedures with respect; used improperly, *they all can be dangerous*.

…notify your instructor in the event of *any* accident, spill, or procedural error, no matter how seemingly insignificant. Immediately clean spills and breakage.

…remain calm in the event of an emergency, and be willing to provide assistance to fellow students.

…clean all glassware and rinse with deionized water after completing an experiment.

…turn off all gas, steam, vacuum, air, and water valves; unplug and put away all equipment before leaving.

…thoroughly wash your hands before leaving the lab.

DON'T…

…bring any kind of food or drink into the lab.

…dress inappropriately! Confine long hair or baggy clothing or accessories. Wear close-fitting clothing, ankle-length pants, and closed-toe shoes. Be covered from your neck to your elbow to your knee. **You will be dismissed from lab if your attire is unsafe.**

…obstruct aisles or work areas with personal belongings.

…perform unsupervised investigations or make unauthorized modifications to any procedure.

…engage in horseplay or any action which may serve to endanger the safety of any person in lab.

…work at the very edge of your lab bench or fume hoods.

…enter unauthorized areas, such as the chemical stock room or lab preparation area, or use unauthorized chemicals.

…keep reagents at your lab station. Return reagents promptly to the place you found them.

…remove more of a chemical than is necessary to complete an experiment.

…return unused chemicals to the supply bottle, or leave a reagent bottle uncapped after you finish with it.

…allow equipment or procedures to operate without supervision.

…put your fingers or any chemicals or equipment near your eyes or mouth as you are performing experiments

…dispose of chemicals or broken glass improperly. Ask your instructor if you are uncertain of proper disposal.

…remove any chemicals or equipment from lab.

Basic First Aid

Report ALL accidents to your instructor, no matter how serious they are!

BLEEDING	Keep victim calm and still; stop bleeding by applying pressure with a clean cloth or towel while elevating a bleeding limb. Compress at an arterial pressure point or use a tourniquet as a last resort.
BREATHING, LACK OF	Remove victim to well-ventilated area while loosening clothing. Windows/doors should be opened and fume hoods should be turned on. In the event of copious or dangerous fumes, evacuate the lab area. Administer CPR if necessary.
BURNS, THERMAL	If the burn is slight (skin not broken), hold affected area under running water or apply a cold wet cloth. Dry, then apply sterile gauze (not cotton). Do not break blisters or apply an ointment. For larger burns, elevate the affected area to reduce swelling and be aware of other problems that may develop (shock, breathing problems).
BURNS, CHEMICAL	Remove affected clothing and rinse skin for 10-15 minutes under running water. Use the safety shower in the event of a large spill. Acids can be neutralized with baking soda and bases with boric acid or vinegar.
BURNS, EYE	Remove contact lens if present and rinse in an eyewash or gently under a faucet for 10-15 minutes. Hold the eyelids open and move the eye around so as to cleanse thoroughly. Identify the chemical and cover the eyes with a sterile cloth.
BURNS, MOUTH	Immediately spit any chemical out in the sink and then continuously rinse with mouthfuls of water. Do not swallow!
CUTS	Remove foreign matter and rinse; clean with soap and water. Rinse with rubbing alcohol or hydrogen peroxide; apply gauze and a band-aid. Apply pressure to a deep cut with sterile cloth to stop bleeding. Elevate feet of fainted or collapsed victims.
ELECTRIC SHOCK	Don't touch a person still in contact with an electric current. Disconnect the electric current or use something like a broom handle or meter stick to push the person away from the current (or the source of current away from the victim).
POISONING	Call 911 or a regional poison control center. Look for an antidote on the chemical label, if identified. Vomiting may be induced by drinking warm salt water, soap solution, or syrup of Ipecac, BUT wait for instructions from poison control before doing anything.
SEIZURES	Prevent the victim from personal injury, especially injury to the head. Clear a space around the person and try to put a cushion under the victim's head. Loosen tight clothing. Don't try to hold the person still.
SHOCK	Keep victim warm, calm, and comforted. Lie victim down and be aware of breathing difficulties. Elevate feet unless accompanied by chest pain or breathing difficulties.
UNCONSCIOUSNESS	Lay the victim down and elevate the feet. Loosen tight clothing and be aware of breathing difficulties. Roll the person on his or her side to allow fluids (saliva, vomit, etc.) to drain from the mouth. Gently bathe the victim's face with cool water.

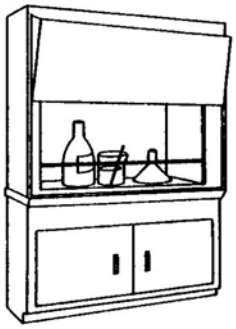
Common Laboratory Safety Equipment

You must know and be able to diagram the location of all safety equipment in the lab. Since people think the least when they are panicked, it must be second nature for you to find the equipment when needed. You may be required to pass a safety quiz before lab work.

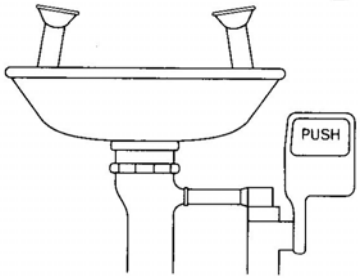
SPLASH GOGGLES are used to protect the eyes from chemical splashes; eyeglasses do not provide protection. To afford sufficient coverage, goggles must be stamped with ANSI Z187 code. Goggles must be kept on your eyes to work!

APRONS and **GLOVES** provide extra protection from chemicals. Gloves are not always impermeable to all chemicals, and must be chosen properly. Be sure to put the apron on correctly to avoid contacting your clothes with chemicals. Dispose of gloves after use.

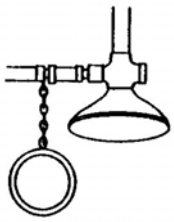
FACE SHIELDS are for use when a chemical explosion danger or rupture of glassware is especially likely.



FUME HOODS use a fan to draw noxious fumes out of a room; they usually have a vertically sliding glass panel for protection. While they may be small in number and space, DO NOT avoid using one if called for by the lab instructions. Note that fume hoods work best when the front glass panel is nearly to the bottom. A wide-open glass panel provides little protection!



The **EYE WASH** station provides a flowing sterile solution (similar to contact solution) for use in rinsing out your eyes in the event of a chemical splash. You must remove contact lenses before using. To use, press the lever and hold your eyes open as you lower your face into the stream of solution.



A **SAFETY SHOWER** is to be used for body fires or large chemical spills. Pull the chain to be doused with about 50 gallons of water. You must remove clothing so that the water can flush your skin.

A **PHONE** is available to call for help; emergency numbers are posted.

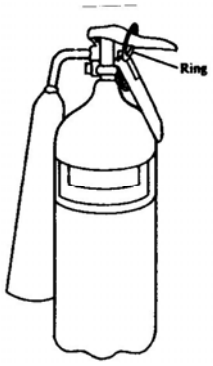
The **FIRST AID KIT** contains typical materials useful for small cuts, bruises, and burns.

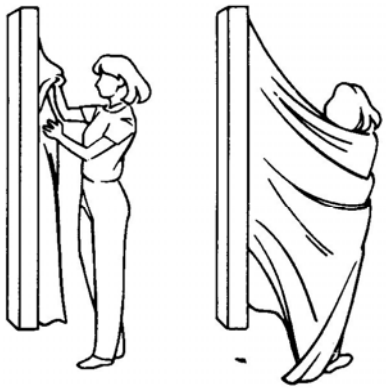
SAND, **BAKING SODA**, and **BORIC ACID SOLUTION (OR VINEGAR)** are available for extinguishing small fires, neutralizing acid spills, and neutralizing base spills, respectively.

A **SPONGE** is available for cleaning up spills.

EXITS are to be used in the event of a large fire, emission of toxic fumes, or large chemical spills. Proceed towards exits quickly and orderly if instructed; do not push, shove, or block the path. Assist fellow students if necessary and disregard personal belongings.

FIRE EXTINGUISHERS and **FIRE BLANKETS** are to be used in the event of a fire. Generally you must remove a metal safety ring or pin and aim the spray at the bottom of the flame. Always have a fire extinguisher refilled after using. Do not use a fire extinguisher on a person as it may cause respiratory irritation and suffocation. Instead, use a fire blanket to smother a clothing or hair fire. Get the victim to the ground and cover with the blanket while rolling to extinguish the flames. DO NOT ALLOW THE VICTIM TO RUN!





A fire blanket

Fire extinguishers are used for extinguishing manageable fires; do not attempt to fight a large fire—GET OUT! Drop to the floor and crawl quickly towards an exit.

A small fire in a reaction vessel can be put out by covering the beaker (perhaps with another beaker or a glass plate) or by dumping sand or baking soda on the fire. NEVER use water, which may react further with the chemicals. Turn off gas lines and electrical equipment. A **GAS CUT-OFF** can be switched off to prevent gas from reaching your outlet.

There are four common classes of fire extinguishers, and they must be used on the proper type of fire.

Class A	Ordinary combustibles	Use pressurized water or dry chemical extinguisher.
Class B	Flammable liquids, gases, greases	Use carbon dioxide, dry chemical, or halon extinguisher to smother flames. Water only spreads liquids and greases.
Class C	Electrical fires	Use carbon dioxide, dry chemical, or halon (preferred because of lack of mess to clean). DO NOT use water to avoid shock.
Class D	Combustible metal fires	Preferably use halon; water, dry chemical, and carbon dioxide MAY ALL REACT WITH THE METAL MORE VIGOROUSLY.