Safety in Academic Chemistry Laboratories

8TH EDITION

BEST PRACTICES FOR FIRST- AND SECOND-YEAR UNIVERSITY STUDENTS

A Publication of the American Chemical Society Joint Board–Council Committee on Chemical Safety

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CHAPTER 4

Recommended Laboratory Techniques

Introduction

Chapter 3 described some types of physical, health, and environmental chemical hazards and the effects of being exposed to chemicals. The focus of this chapter is on how to safely perform common laboratory techniques and safely handle the most common equipment in the undergraduate chemistry laboratory. The techniques and advice in this booklet focus on those topics most commonly encountered in first- and second-year courses in college. There is brief mention of some advanced topics that you may encounter in upper-level courses in the "In Your Future" sidebars, but a thorough presentation of advanced techniques is beyond the scope of this publication. The references in the Appendix point to other sources of safety information.

As discussed in Chapter 1, the RAMP system is a useful paradigm when you work in the laboratory. Chemical safety must be the priority of everyone. Apply the RAMP concept as you prepare for each laboratory session. Once hazards have been recognized and assessed, they must be minimized or managed. Minimizing hazards to reduce risk involves adding controls or placing barriers between the worker and the hazard. A control measure is simply the steps taken to eliminate or minimize a hazard. (Note that fewer hazards or less hazardous substances used in the laboratory can also have a positive effect on the environment. See "Green Chemistry — Not Just a Catchphrase".) There is an order, or hierarchy, of controls, used to prioritize the steps that should be considered (see "Hierarchy of Controls").

As an undergraduate, you must carefully listen to all directions and complete all laboratory preparations before coming to the laboratory. Carefully review the details of an upcoming laboratory procedure, and ask your instructor before beginning the experiment if you have any concerns about laboratory hazards. Your instructor or undergraduate research mentor may want you to review *advanced* techniques for a particular experiment and may provide specific instructions for you to follow to ensure your safety.

Hierarchy of Controls

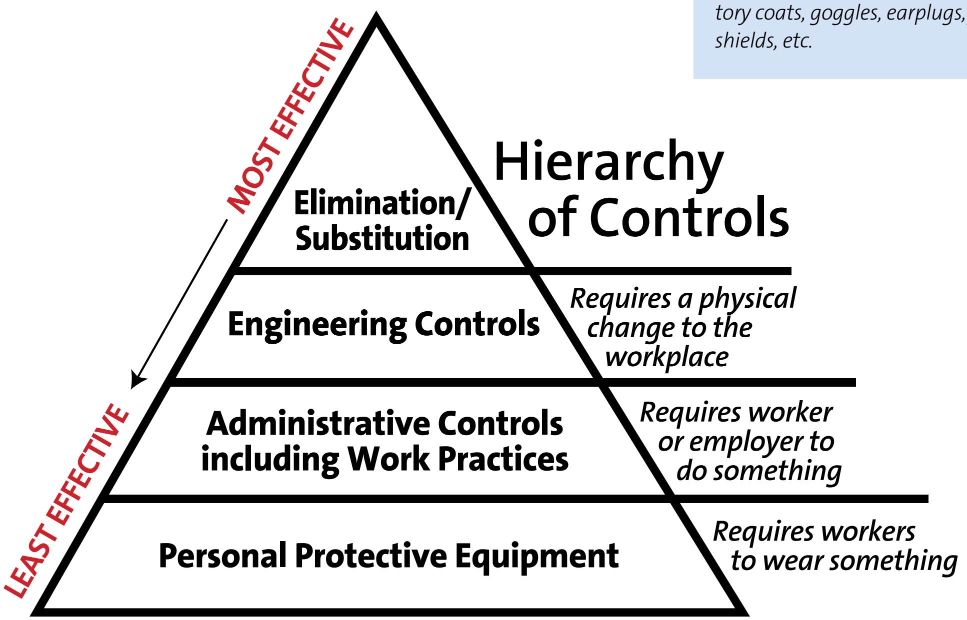
Elimination: *the initial design of the facility, equipment, chemicals, or process to remove hazards.*

Substitution: *using a less hazardous substance or process; selecting another solvent that allows for lower reaction temperature or pressure, less toxic reactants, etc.*

Engineering: *laboratory hoods, gloveboxes, biosafety cabinets, snorkels, safety interlocks, lead shielding, inert atmospheres, etc.*

Administrative: *implementation of procedures and policies (standard operating procedures), training, reducing exposure times, attention to nearby employees or other students, signage, best work practices, not working alone, etc.*

Personal protective equipment: *gloves, respirators, aprons, laboratory coats, goggles, earplugs, face shields, etc.*



Working with Chemicals, Apparatus, and Equipment

Following these recommendations will help to make your work easier and laboratory equipment safer to use, thereby increasing the likelihood that your experiment will be successful. As described in Chapter 3, there are many hazards potentially present from the chemicals in a laboratory, but the equipment used in experiments can also present hazards. Be particularly alert to procedures where temperature and pressure are elevated or lowered, electrical equipment is required, open flames are used, or you are unfamiliar with the equipment being used. Never disable safety features or guards on equipment.

General Concerns when Working in the Laboratory

As you read over the laboratory directions, look up any new terms and review reference sources suggested by your laboratory instructor. Be sure that you know what to do if you or another student experiences an unexpected occurrence (a sudden rise or drop in temperature, excess gas evolution, etc.). Or, for example, if the instructions for an experiment state that the temperature should *not* exceed a predetermined point and you don't understand why, you should discuss this with your instructor.

Set up your experimental apparatus away from the edge of the laboratory bench, where it could easily be knocked onto the floor. Secure laboratory apparatus when necessary. For example, an Erlenmeyer flask can be placed directly on the laboratory bench, but a round-bottom flask needs to be held with an appropriate clamp attached to a ring stand or placed in a cork ring.

Inadvertent Exposure to Chemicals

Chemicals can get transferred to you indirectly (and unexpectedly) in a wide variety of ways. Some common examples are:

- Chemicals on your hand, or gloved hand, can easily get transferred to a pen or pencil in the laboratory and then get transferred to your mouth if you are in the habit of placing a pen or pencil in your mouth. Avoid this habit.
- Chemicals on your hand can get transferred to your eye if you momentarily raise your goggles to rub your eye. Chemicals on your hands will easily get transferred to a cell phone or computer. Further, the cell phone might then transfer the chemical to your face if it is used as a phone in the laboratory.
- If you wear gloves in a laboratory and they become contaminated, they will spread this contamination to everything that you touch in the laboratory: computers, balances, hot plate controls, glassware, and cell phones. This easily spreads the contamination. Gloves should be removed whenever you leave the laboratory, because otherwise they might contaminate doorknobs and other items outside the laboratory.

The purpose of gloves is to act as an extra barrier to avoid skin contact. Ideally, they should not become contaminated, but if you suspect this, you should replace them immediately to avoid the scenarios listed above.

Scientific Glassware

The directions provided by your instructor should indicate the appropriate size of glassware to accommodate the operation to be performed. If the exact size (e.g., 250 mL) is not available, keep in mind that you should select glassware so that at least 20% free volume remains in the vessel throughout the experiment. For chemical reactions, borosilicate glassware (e.g., Pyrex or Kimax) is used because it can withstand extreme changes in temperatures and minimal shock without breaking.

Clean glassware before use (see Chapter 2). If you share equipment with another student, be particularly aware of the condition that your glassware is in each period. You should examine your glassware closely for cracks, stars, and chips before use. Small flaws in the glass can lead to failure when heat is applied. Consult your instructor whenever flawed or damaged glassware is discovered, because in some cases, specialty glassware can be repaired rather than discarded. Damaged glassware should be discarded in an appropriate waste container labeled “broken glass only”. Your instructor will describe the procedure to replace your glassware. Keep your workspace clear of clutter. Only glassware currently in use should be on the benchtop.

Green Chemistry — Not Just a Catchphrase

Green chemistry was born out of the environmental revolution that began in earnest in the mid-20th century. It was during this period that the public began to understand what many scientists already knew: that many of the chemicals and their by-products being used to produce the industrial wonders of modern life could have devastating side effects on the environment and human health if not managed correctly.

Aligning with the principles of pollution prevention and control, green chemistry involves the practice of redesigning processes to minimize the use and formation of known hazardous chemicals. A chemical is considered less hazardous if it is less toxic to humans and the environment, is nonflammable, and will not bioaccumulate in the environment.

Although you may not be actively involved in green chemistry yet, it is likely that this concept has been applied to the experiments you perform in your introductory and organic chemistry laboratories. These adaptations have resulted in minimizing the generation of hazardous waste, providing safer laboratory working conditions, consuming less energy and water, and reducing or reusing chemicals.

To learn more about green chemistry, read about the ACS Green Chemistry Institute.[1]

Working with Flammable Liquids or Gases

Often, many students share limited laboratory space, so be aware of other students and their experimental setup as well as their movements around the laboratory. Be sure to pay attention to the proximity of reagent bottles to open flames or other sources of ignition or heat, and ask your instructor whether your reaction setup should be relocated. Flammable solvents should be stored in a flammable cabinet if the recommended storage is at room temperature. Always minimize the quantity of solvents outside of the flammable cabinets.

Laboratory Hoods

Laboratory hoods are an ***engineering control*** recommended for all operations in which offensive odors or toxic or flammable vapors may be evolved. Space in the hoods may be limited, and laboratory hoods that are actively being used for experiments should not be used to store chemicals or waste. Hoods may have small glass panels that slide sideways or a larger panel that raises and lowers, or both. This part of the hood is called the sash. Once the setup portion of the experiment is completed, the sash should be lowered or closed as much as possible to maximize airflow through the working space and away from the user. The airflow in the laboratory hood can be disrupted by drafts from windows or doors and even by a change of position of the students standing at the hood. Keep the sash lowered whenever possible, and only open it the minimum amount necessary to access equipment.

Before setting up equipment in the hood, be sure that the hood is working properly. A properly operating hood requires both an adequate airflow and the absence of excessive turbulence, so that containment of chemicals is maximized and personal exposure is eliminated. Ask your instructor if you are not sure whether the hood is operating properly. Always keep your face outside the plane of the hood sash when an experiment is in progress. When you place your equipment within the hood, it should be placed at least 15 cm (6 inches) from the front edge of the hood. Work as far back in the hood as practical, but do not block any rear exhaust openings. Whenever possible, equipment should be elevated in a laboratory hood so that the disruption of air flow is minimized.

IN YOUR FUTURE: Protection against Explosions

Hoods should protect users from exposure to toxic vapors when the sash is closed, but a hood is not designed to fully protect you from physical injury if a chemical explosion occurs. When it is necessary to perform a procedure that could result in catastrophic failure (implosion or explosion), the experiment must be performed behind laboratory shields designed for that purpose. Shields should be stabilized so that they cannot be knocked over or become a projectile in case of catastrophic failure. In addition to wearing eye protection, you should use full face protection when checking the operation of a reaction that has the potential for explosion.

Distillations

A common method of separation and purification is distillation. Careful design and setup of the distillation system are required to safely accomplish effective selective evaporation and subsequent condensation of the desired product. Distillation uses differences in the volatility of a mixture's components to separate the mixture. Potential hazards include pressure buildup, heating of flammable solvents, initiation of an exothermic reaction, vapor leaks that can ignite, and continuing to heat until dryness. Carefully follow the specific directions provided by your laboratory instructor, and ask questions for clarification of the procedures if needed.

Even heating and smooth boiling are desirable to avoid bumping. Bumping refers to the superheating of a solvent, which then suddenly releases a large vapor bubble, forcing liquid up and out of the boiling flask. Your instructor may suggest that you add boiling chips to your round-bottom flask to minimize bumping. The boiling chips provide nucleation sites for the gas bubbles and prevent superheating.

Even heating with steady stirring also minimizes this concern. Your laboratory instructor may direct you to use a hot plate with a stirrer or a heating mantle. Be sure to watch the reaction temperature, and never heat above the temperature directed in the procedure.

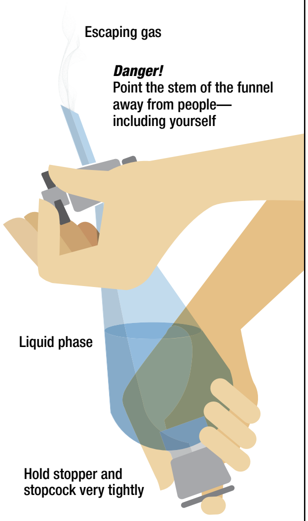
■ IN YOUR FUTURE: Unattended Operation of Equipment

Advanced chemical laboratories usually involve more hazardous chemicals and reactions. Reactions that are left to run unattended overnight or at other times are prime sources for fires, spills, and explosions. Do not let equipment such as power stirrers, hot plates, heating mantles, and water condensers run overnight without having fail-safe provisions in place, your instructor's full knowledge of the process, and your instructor's consent. Check unattended reactions periodically. Always leave a note plainly posted with a phone number where you and the instructor can be reached in case of emergency. Remember that in the middle of the night, emergency personnel are entirely dependent on accurate instructions and information.

Extractions

Glass separatory funnels are commonly used to separate mixtures. Watch your instructor demonstrate the proper use, and always follow the directions provided for the separation procedure. In general, never vent the separatory funnel near a flame or other ignition source, be sure to direct the vapors away from yourself and others in the laboratory, and direct the vapors into the laboratory hood. When using a volatile solvent, first swirl the unstoppered separatory funnel to allow some solvent to vaporize and expel air. Then close the funnel, invert it with the stopper held firmly in place, and immediately open the stopcock to release air and vapor. Do this with your hand and fingers firmly holding the stopcock in place. Repeat as necessary.

Venting a separation funnel



Precautions for Using Electrical Equipment

Inspect the insulation on an electrical cord before use. The outlet plug should not be bent or damaged. When removing the plug from the electrical outlet, do not pull on the electrical cord. Firmly grasp the end of the plug and pull the plug directly out of the outlet. Do not remove plugs with wet hands. Damaged, tattered, or cracked cords and electrical failures or evidence of overheating should immediately be reported to your instructor. For normal power (110–115 volts alternating current) applications, only three-prong grounded, double-insulated, or isolated wiring should be used. Because of the risk of fatal shock or electrical burns, care must be taken when exposure to a live circuit could occur. Electrical currents as low as 0.1 amperes may result in fatal shock.

The most commonly used electrical equipment in your first laboratories will be hot plates and hot plates with magnetic stirring motors. When hot plates with magnetic stirrers are used, remember that the appearance of the ceramic plate does not change, whether it is cold or hot. Always carefully move the plate by grasping the bottom of the apparatus. You may burn yourself if you grasp the top ceramic plate when it is hot. Before turning the hot plate on, ensure that the cord is not in contact with the ceramic top of the hot plate. Most hot plates are not explosion-proof, and they should be used in a hood when you are heating organic solvents, so that the vapors are removed.

Note that the control for the magnetic stirrer and the temperature control often appear similar. Be sure to carefully distinguish which knob you are increasing or decreasing. Some models allow the user to continue to rotate the dial from LOW to OFF and then back to HI. Be sure to confirm that the hot plate is OFF and unplugged before you leave the laboratory.

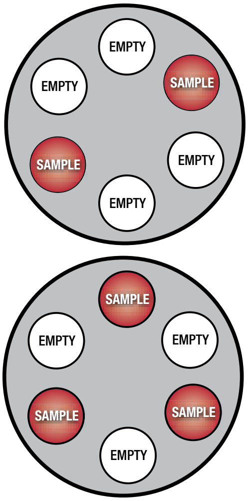
Refrigerators

Refrigerators and freezers are used in laboratories when low-temperature storage of laboratory chemicals is required. The label on the chemical will indicate the storage temperature for the chemical. Depending on the type of laboratory, these units may be rated for storage of flammable materials. A unit rated for storage of flammable materials ("lab-safe") protects the contents within and will be clearly labeled as such on the front.

Under no circumstances should flammable substances be stored in a non-flammable-rated or household refrigerator. Chemicals stored in a refrigerator should be placed on a spill tray with sufficiently high edges to contain the entire contents if a spill occurs. NEVER, under any circumstances, store food or beverages for consumption in a refrigerator in a chemical laboratory. If an experiment requires the testing and storage of food or beverage samples, each sample should be clearly labeled "Not for human consumption".

Centrifuges

Benchtop centrifuges should be placed back from the edge of the laboratory bench, so that if excessive vibration occurs, they will not "walk" off the edge of the bench. Most modern centrifuges have suction feet that will prevent this for mild vibration. Proper loading of the centrifuge will prevent an unbalanced spin. The volume of liquid in the test tubes should be approximately the same height, and the test tubes should be placed directly across from each other or in a triangular formation (see diagram). NEVER pour liquids directly into the centrifuge tube sleeves. Modern centrifuges also have lids that prevent accidental contact with the rotor. Some even have a braking mechanism in case the lid is opened. Always close the lid before turning on the centrifuge, and keep the centrifuge closed while it is running. Allow the rotor to come to a complete stop before opening the lid. Stopping the rotor with your hands can be dangerous as well as defeating the purpose of centrifuging: to separate a precipitate from the solution. Follow the directions of your laboratory instructor, and ask for assistance if the centrifuge is not operating as expected.



Balanced distribution of samples in centrifuge

High-Pressure Air

Many laboratories are equipped with pressurized air valves (often near the hood) for drying glassware. Only use high-pressure air from a "house system" when instructed to do so; residual oil may be present from the equipment used to generate the house air, and this can contaminate the glassware. You should never direct air under pressure toward yourself or any other person. Pressurized air can penetrate skin without any visible opening, causing the area to expand like a balloon. Severe damage to tissue can occur, which may require hospitalization.

Ultraviolet Lamps

Ultraviolet (UV) lamps are used to visualize substances that fluoresce when exposed to UV light (100–400 nm). Thin-layer chromatography (TLC) is used to separate mixtures; the spots can be observed under UV light. When using a UV lamp, care must be taken to protect your eyes. Your eyes may be accidentally exposed to UV light, so special eyewear with UV-absorbing lenses should be worn. It is preferable to operate UV lamps only in a completely closed radiation box, to minimize exposure of others around you.

General Considerations when Controlling Temperature

Many reactions require temperature control. Take note of the experimental protocol before assembling your apparatus, so that heating and/or cooling can be readily applied and/or withdrawn to manage temperature. Carefully follow the directions of your instructor whenever an exothermic reaction is expected. Often, chemical reactions must be initiated by heating. In an exothermic reaction, temperature increases. The rate at which the heat is produced increases exponentially, whereas the rate at which the heat is removed is linear. Highly exothermic reactions can very quickly become dangerously violent, in what is called a thermal runaway reaction, unless adequate cooling can be readily applied.

Some exothermic reactions proceed with an initial slow phase followed by a rapid acceleration. The initial slow phase is known as the induction period. In such reactions, if too much reagent was initially added, the reaction can become too vigorous for effective condensation of vapors once the induction period is completed. Once the rapid phase of a runaway reaction

ensues, external cooling must be used to control temperature. A cooling bath must be prepared in advance and be at the ready so that it can be promptly applied under the reaction vessel.

Do not heat closed systems unless specifically instructed to do so by the procedure or by your laboratory instructor. Consider the need for the buildup of excess pressure to be released when an apparatus is heated or an exothermic process is anticipated, and have a mechanism in place to vent excess pressure.

IN YOUR FUTURE: Dry Ice and Liquid Nitrogen (LN_2) Cooling Baths

When temperatures lower than those that can be attained with an ice bath are required, dry ice or dry ice combined with an organic liquid may be used. Dry ice will sublime at −78 °C; a mixture of dry ice with acetone maintains this temperature, and the acetone will not freeze. Liquid nitrogen (LN_2, −196 °C) must be used with extreme care and only when you are instructed to do so by your instructor or research advisor. In addition to proper laboratory attire, chemical splash goggles and insulated cryogenic gloves are required when working with these very cold baths. Dry ice and LN_2 are also both simple asphyxiants and must be handled in a well-ventilated room or in a laboratory hood.

Cooling Baths

When ice water is not sufficiently cold for use as a bath or trap, salt and ice may be used. Depending on the number of moles of ions (recall the concept of freezing-point depression), the ice baths can be prepared in the range of −10 °C to −40 °C.

Determination of Melting Point

The accurate determination of the melting point of a compound is a common method of identification. In the past, a melting-point apparatus may have used a mercury thermometer, because they are more accurate than alcohol thermometers. Because of the hazards associated with breakage of a mercury thermometer, most laboratories now use an electrothermal melting-point apparatus. The main hazard here is the very hot metal chamber that holds the sample.

Photo courtesy of Stanford Research Systems.

Use of Bunsen Burners

Bunsen burners may be used in laboratories to heat samples strongly or to boil water rapidly. A well-adjusted flame will have two distinct cones: an outer aqua cone and an inner blue cone. The temperature of the hottest part of the flame (the tip of the inner blue cone) is about 1200 °C. Take a moment to look at the burner. You will see a barrel with a removable tip at one end and a band of holes at the other end. These holes adjust the amount of air entering the barrel (a mixing chamber for fuel and air). On the bottom is a needle valve, which, when turned tight into the barrel, blocks all gas from entering the barrel. Before using a Bunsen burner, check the hose for cracks, and ask your instructor to replace a faulty hose. Your instructor will show you how to adjust the air holes, needle valve, and gas jet to light the burner with a laboratory striker. Before using a flame, tie back loose hair and remove or confine scarves.

Heating a Test Tube

When the contents of a test tube are heated in an open flame, the contents can easily be overheated, causing the contents to boil up and forcefully eject out of the test tube. Always hold the open test tube at an angle facing away from yourself and others. While holding the test tube with a test tube holder at an angle, heat it gently along the side, not at the bottom of the tube. An alternative is to heat the contents of a test tube by placing it in a hot water bath.

Reduced Pressure

Water aspirators are sometimes used to create reduced pressure for filtration. Use only heavy-walled filter flasks that are designed for this purpose; do not apply reduced pressure to other flat-bottom flasks, which may not withstand reduced pressure and could crack during use. The flask should be clamped to a ring stand for stability. A trap is placed between the aspirator and the filtration flask, so that water cannot be sucked back into

your filtrate if the water pressure decreases unexpectedly and so that the filtrate cannot enter the sink. Whether or not you are using a trap, always disconnect the vacuum hose line on the side arm before shutting off the water source. More and more, the use of water aspirators is generally discouraged, because of the significant quantities of wastewater produced.

SUMMARY

In this chapter, the safe use of common laboratory equipment was reviewed. Often, laboratory processes require pressures and/or temperatures other than ambient, and therefore can introduce physical hazards into your experiments. Always review the laboratory directions before beginning any procedure. Continually assess your situation in the laboratory and be aware of your surroundings, and you will learn to proactively recognize potential hazards.

Ask your laboratory instructor if you have any concerns about the safe use of equipment and the completion of any procedures. Chapter 5 presents protocols that will help you prevent and prepare to respond to laboratory incidents.

REFERENCES

[1] ACS Green Chemistry Institute. www.acs.org/content/acs/en/greenchemistry.html (accessed March 6, 2017).