Université d'Ottawa / University of Ottawa
Faculté des sciences / Faculty of Science

GENERAL CHEMISTRY

LABORATORY MANUAL

CHM 1301/1311
Fall 2017

ORGANIZATION OF THE LABORATORY

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Each laboratory class is sub-divided into sections of about 16-18 students. Each group of students is supervised by a demonstrator who has the responsibility of ensuring the smooth running of the experiment, of helping students with difficult techniques or interpretations, and of marking the reports. The demonstrators are usually graduate students in Chemistry. Each lab session, the demonstrator (TA) evaluates the student on a set of **objective criteria** (Appendix 1). At the end of the course the demonstrator takes the average of each student's performance evaluation in the laboratory. This evaluation is based on the extent to which the student is prepared for the experiment, his/her initiative and general competence in laboratory work.

The technicians in the third-floor stores prepare the chemicals and equipment needed for each experiment. The personnel in the stores are also responsible for making available the Material Safety Data Sheets (MSDS) for all the chemicals used in this course. They are to be informed immediately of any accidents, thefts, or damage to lockers or equipment.

THE LABORATORY SESSIONS

The laboratory sessions are held from 14:30 to 17:30 p.m. Tuesday and Friday, Wednesday from 10:00 to 13:00 and two night labs from 6:30 to 9:30 p.m. on Tuesday and Thursday in the first year chemistry lab, MRN 301, beginning in September. Completion of the course requires attendance at **all** laboratory sessions, completion of the experiments and submission of the report accompanying each experiment. If you miss a laboratory class, inform your demonstrator and the laboratory co-ordinator as soon as possible. A student may be excused from class for medical reasons, but written authorization from a doctor **must be received by the laboratory co-ordinator**. There is an 80% attendance requirement for the laboratory portion of the course. If you do not attend 80% of the labs, **for whatever reason**, you will not receive credit for the course. You may obtain permission to transfer your laboratory class to another day in the same week if you are prevented from attending your own class for a **valid reason**. To do this, obtain an authorization form signed by the laboratory co-ordinator. Attend the session on the alternate day, ask the demonstrator in charge to sign the form, and submit your report to your regular demonstrator one week after completion of the experiment.

DURING THE LABORATORY PERIOD

- Your locker contains the basic equipment that is required for your experiments. If you are unfamiliar with a piece of equipment (e.g., Buchner funnel), ask your demonstrator. *The locker is shared between students on different days. Everyone has the responsibility for keeping the equipment clean and in good order.* ***You will be held responsible for keeping your bench as well as your locker in order.***

- Special equipment required for each experiment will be distributed on the carts at the front of the laboratory. This equipment should be returned to the carts after use and not be kept in the locker, since the equipment is needed during the other laboratory sessions.

- Solutions and reagents required for the experiments will also be provided on the **front benches**. The location of the particular solution will be announced at the beginning of class. Take only the amount required for the experiment.

- Demonstrators will unlock each locker before the laboratory begins and lock each locker after the experiment has finished.

- Each laboratory session will start with a brief period of pre-lab instruction given in the laboratory. Attendance at the pre-lab briefing is mandatory and useful!

- Plan ahead so that you have sufficient time to collect your data and clean your equipment by **12:50 p.m.,5:20 p.m.,OR 9:20 p.m.** (depending on your section).

- **You are responsible for recording ALL of the experimental data in PEN, neatly and legibly in a hard-cover laboratory notebook**!

LABORATORY REPORTS

One goal of an introductory lab course is to prepare students to work in any lab, whether it is in an industrial, government or academic setting. Therefore, the purpose of this manual is to correctly instruct you in how to write a report from the beginning, to ensure that you perform your tasks in a way that is acceptable in any laboratory setting.

Before leaving the laboratory, ALL students must have their lab notebooks SIGNED by their demonstrator. A copy of electronic data must be sent to the TA. The results given in the laboratory report must correspond to this data. **Please read the section entitled Lab Expectations** for detailed instructions on each report. Please note the following brief details:

- Formal reports should give an individualized account of the experiment. Although you work with a partner, **you must write the report yourself**. Regurgitation of information already supplied is not necessary. It is not necessary to give 10 -20 pages of discussion. Your demonstrator, who marks many reports every week, will look most favourably on a **concise**, **brief**, and **well-written** report. A completed report must have a rational structure, which includes an introduction or theory, an explanation of results (use tables and graphs wherever possible to make data easily understood), calculations, a discussion and a conclusion. **Wherever possible, comparisons should be made between experimental results and literature values.** For this purpose, the Merck Index and the Handbook of Chemistry are available in the reference section of the Library. For more information, check Appendix I. How to write a Laboratory Report. **A sample report is provided in this Appendix.**

- Formal laboratory reports MUST be typed or word-processed.

- Students who fail to do an experiment or fail to hand in a laboratory report for an experiment that they have done will receive a mark of zero for that experiment. **Please refer to the section on Lab Expectations regarding late reports.**

MARKING ASSIGNMENT FOR THE LABORATORY COURSE

In order to receive a grade in this course, you are required to obtain a MINIMUM of 50% in the lecture component AND 50% in the lab component of the course.

The marks for the laboratory portion of the course are divided as follows:

Pre-lab assignments	10 %
Laboratory reports (3)	30 %
Formal report, Experiment 4	25 %
Experiment 5	20 %
Demonstrator evaluation	15 %

An average mark for the pre-labs and reports is calculated at the end of the term. The evaluation by the demonstrator is added to this to make a final percentage**. The marks for the laboratory portion of the course will be reduced to a grade out of 25. This grade will be added to the lecture portion of your grade to provide a final grade out of 100 marks.**

LABORATORY SAFETY

A chemical laboratory is not necessarily the safest of all places in which to work. Neither, however, is it something of which one has to be afraid. With proper understanding of what you are doing, careful attention to safety precautions, and adequate supervision, you will find the chemical laboratory to be a safe place in which you can learn much about chemistry.

Laboratory accidents belong to two general categories of undesirable events: mishaps caused by your own negligence and accidents beyond your control. Although accidents in the laboratory fortunately are rather rare events, you nevertheless must be familiar with all safety rules and emergency procedures. If you know and follow safe working practices, you will pose no serious harm to yourself or others.

General Safety Rules

- **Eye protection is extremely important. All people in the laboratory, including visitors, must wear eye protection at all times, even when not performing a chemical operation.**

  Contact lenses should not be worn because reagents can get under a lens and cause damage to the eye before the lens can be removed. It is extremely difficult to remove a contact lens from the eye after a chemical splash has occurred.

Laboratory safety glasses should be of plastic or tempered glass and should have side shields. If you do not have such glasses, wear goggles that offer protection from splashes and objects coming from the side as well as the front.

- Never eat, drink, or smoke in the laboratory.

- Don't work alone. Your best protection against accidents is the presence of trained laboratory personnel, who can give advice, help, and guidance to avoid accidents.
- Do not perform unauthorized experiments and don't distract your fellow workers; horseplay has no place in the laboratory.

- Know the location and use of emergency equipment in the laboratory.

- Read the MSDS available for the chemicals used in the experiment. Not every chemical is a potential hazard or risk to use. If you have doubts, read the MSDS provided.

- Most importantly, ***think about what you are doing.***

  Plan ahead. An unprepared person is more likely to be involved in a potentially accident-developing situation. The laboratory period should be enjoyable: use good common sense and develop a good safety consciousness.

A list of other safety procedures is provided in the introduction section of the manual.

In Case of an Emergency

- In case of an accident, call your demonstrator at once. Except for very superficial injuries, you will be required to get medical treatment for cuts, burns, or fume inhalation. The laboratory staff will arrange for transportation as needed.

- Acid or alkali spills on the body-- immediately flush with copious quantities of cold water. The shower is used if a large area is affected; small splashes can be washed in the sink.

- Small lab fires should be smothered with fireproof cloth or a glass beaker. Large fires require a fire extinguisher.

- If a person's clothing catches fire-- roll the person onto the floor and use a lab coat to extinguish the flames. DO NOT use a fire extinguisher or the shower.

- **If an ALARM sounds**:

    - Stop work immediately, turn off any burners, and upon signals from your demonstrator, begin evacuating using the stairs at the north and south ends of the laboratory.

    - Walk, do not run, down the stairs and outside the building and continue until you are at least 100 meters away from the building.

    - Wait until appropriate instructions have been issued by the Chemistry Department Safety Committee or the Fire Marshall (designated by clearly marked white and yellow safety helmets) before returning inside the building.

WHMIS, MSDS, AND YOU

The Workplace Hazardous Materials Information System is a Canada-wide program commonly known by its initials, WHMIS. As its name implies, WHMIS is an information system for everyone who comes in contact with a potentially hazardous chemical.

The Federal and Ontario governments have enacted legislation for enforcement of this system. The Ontario provincial legislation, through the Occupational Health and Safety Act and the WHMIS legislation, requires employers to obtain health and safety information about potentially hazardous materials in the workplace and to pass this information on to their employees.

In 1995, in keeping with the spirit and intent of the law, the Faculty of Science at the University of Ottawa will initiate faculty-wide programs whereby information is to be passed on, not only to employees, but also to students who participate in laboratory courses within the Faculty of Science. In essence, the University of Ottawa "WHMIS undergraduate laboratory information program" is designed to ensure that

- **chemicals used in the laboratory are properly labelled or identified**

- **students have access to material safety data sheets (MSDS) for hazardous chemicals**

- **employees and students alike are aware of their rights to have access to health and safety information about the chemicals used in the laboratory**

It is impossible to expect that a complete WHMIS training course for undergraduate students can be accomplished within the context of [a single laboratory period of] the General Chemistry laboratory course. It is important, however, that students be aware of the salient features of the WHMIS program at the University of Ottawa.

Labelling

The label is the first and most basic form of WHMIS warning to employers and workers. It is easily recognized, can appear on any container, and contains easily understood information about the potential risks associated with the material inside the container.

There are two types of labels; the information labels or symbols, and the WHMIS product information label. The **symbols** shown in figure 1 are known as personal protection symbols. They do not specify the degree or type of risk for the product but are a reminder for the safe use of the product.

Figure 2 shows a typical **WHMIS label**. This "label" can be any mark, sign, device, stamp, seal, sticker, ticket, tag or wrapper. Regardless of its form, all WHMIS labels are required to include product identifier, supplier identifier, hazard symbol, risk phrases, precautionary measures and a first aid measure. The label also directs the reader to the material safety data sheet (MSDS), the next element in the information delivery system.

Material Safety Data Sheets (MSDS)

Material Safety Data Sheets (MSDS) provide information on hazardous ingredients, properties and potential hazards of a product. It explains how to use the product safely and what to do in an emergency. It is a source of detailed information about particular hazardous materials, and provides data on which effective workplace controls for this material may be based.

It is mandatory that a clearly labelled MSDS binder containing the MSDS sheets be made available in each and every laboratory within the University. The key aspect of the MSDS sheets is awareness - the supplier or employer must include any information on potential hazard of which the end-user ought reasonably to be aware. Much of the information provided in the MSDS is of a technical nature, and is addressed primarily to those who may be directly involved in the use, storage or disposal of the material, or for emergency treatment. Anyone who may have doubts about the safety aspects of a given chemical should consult the MSDS for the definitive answer and explanation about potential hazards.

Co-ordinated Education Program

The final stage in the WHMIS information delivery system is the co-ordinated education program. Its purpose is to educate workers (students) how to use the labels and MSDS to protect themselves. Increased worker knowledge of the effects of hazardous materials is, after all, the main aim of WHMIS.

Note that the WHMIS/MSDS system does not include information about how chemicals enter and affect the body, dose response relationships, exposure limits, and the concept of toxicity. This type of information currently falls outside the scope of the WHMIS mandate, but is information that should be addressed if responsible decisions are to be made regarding the use of a given chemical.

The designer(s) of this laboratory manual have pre-tested all the experiments and have used the various chemicals many times, in differing concentrations and quantities. This testing has always been made with the aim that the optimum results be obtained, but at the lowest possible risk of harm or hazard to the student. The employers (chemistry staff) have been responsible in applying all their knowledge and experience into the choice of chemicals used.

The ultimate responsibility lies, however, with the student worker in the laboratory. The employer has made the effort to present the best possible choice of experiments at the lowest possible risk. If the student does not feel comfortable with the chemicals used, he/she has the right, the access and the responsibility to consult the MSDS/WHMIS information made available to him/her by the WHMIS trained personnel working in the undergraduate laboratory.

Chemical Ecology I: Waste Disposal

In the not too distant past, it was common practice to wash all unwanted liquids from the laboratory down the drain and to place all solid waste in the trash basket. Never a wise practice, for environmental reasons, this is no longer allowed by law. The law also specifies that, before disposal, all materials classified as laboratory waste must be segregated into different classes: mainly non-hazardous solid waste, organic solvents, halogenated organic solvents, and hazardous wastes of various types.

- ***Non-hazardous waste*** encompasses such solids as paper, paper towels, corks, sand, alumina, and sodium sulfate, Na_2SO_4. These ultimately will end up in a sanitary landfill (read, the dump). Any chemicals that are leached by rainwater from this landfill must not be harmful to the environment.
- In the ***organic solvents*** container are placed the non-aqueous solvents that are used for running reactions, cleaning glassware, etc. These solvents can also contain dissolved, solid non-hazardous organic solids. This solution will go to an incinerator where it will be burned.

Figure 1 — WHMIS Danger Symbols

Category A

Compressed Gas

Category B

Flammables and Combustibles

Category C

Oxidizing materials

Category D, division 1

Materials causing immediate and serious toxic effects

Category D, division 2

Materials having other toxic effects

Category D, division 3

Bio Hazardous Infectious materials

Category E

Corrosive

Category F

Dangerously reactive materials

Figure 2 — WHMIS Personal Protection Symbols

Splash resistant goggles

Facial screen

Lab coat

Disposable anti-dust mask

Respirator with filtering cartridges

Self-contained breathing apparatus

Safety gloves

Protective footwear

Protective combination

Figure 3 — WHMIS Information Label

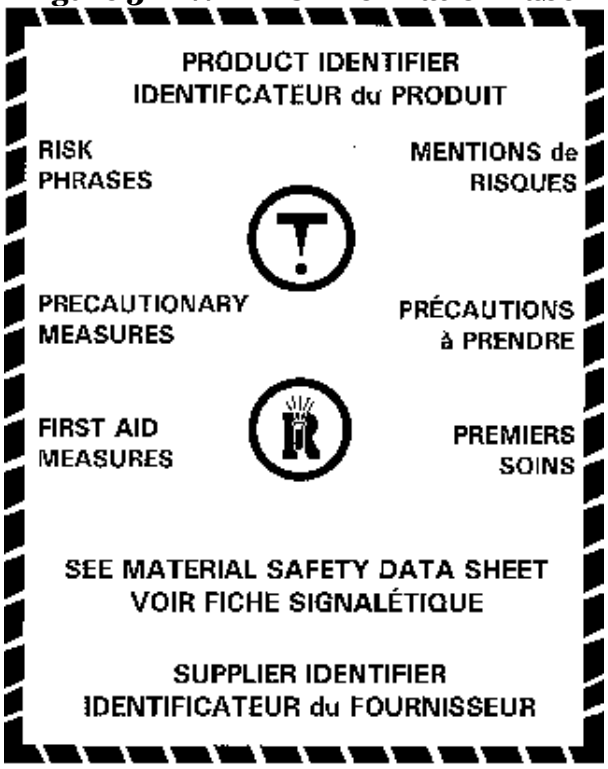
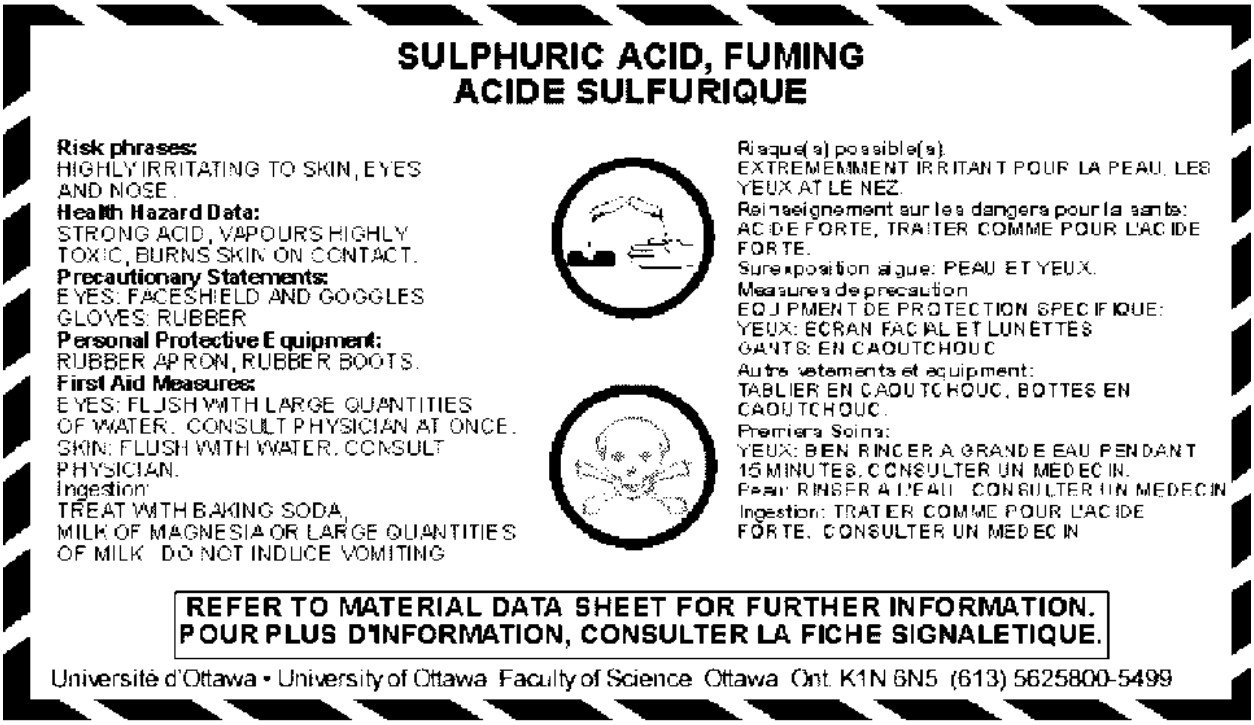


Figure 4 — WHMIS information label for Sulfuric Acid



- If a solvent is a halogenated one (e.g., dichloromethane) or contains halogenated material, it must go in the ***halogenated organic solvents*** container. Ultimately this will go to a special incinerator equipped with a scrubber to remove HCl from the combustion products.
- The final container(s) is/are for various ***hazardous wastes***. Since hazardous wastes are often incompatible (oxidants with reductants, cyanides with acids etc.), there will be different containers for these in the laboratory, each identified with its own markings.
- Canadian law states that a material is not a waste until the laboratory worker declares it a waste. *It now costs much more to dispose of most hazardous chemicals than it does to purchase them new!* **So for pedagogical and practical reasons, we will regard the chemical treatment of the by-products of each reaction in this manual as a part of the experiment.** As both scientists and concerned citizens, it should be our goal to help reduce the volume of hazardous waste, and whenever possible, to convert hazardous waste to less hazardous or non-hazardous waste.

what's it all about, Alfie?
Michael Caine's Autobiography, 1992

THE BASIC PREMISE

The study of Chemistry has both a theoretical and a practical side. On the one hand, the beauty of chemical laws, theories, hypotheses, and ideas will be presented to you throughout the term in your lectures and tutorials. On the other hand, "real" Chemistry is experienced in the laboratory where elements, compounds, solutions, mixtures, solids, liquids, and gases are at your disposal. *Chemistry is working with substances:* the determination of their composition and their properties; the exploration of their transformation into other substances. Chemists and students of Chemistry must be familiar with substances (with chemicals). They must know the appearance of substances, their physical state, their colour, their odour, their toxic properties, how to handle them, how to store them, and how to dispose of them. This is what this course is all about.

Six different topics, some standard, some quite new, are covered in this laboratory manual. As the title of this manual suggests, Chemistry is everywhere, and one of the main reasons for us to study Chemistry is to learn about chemical principles and ideas in order to try to understand familiar things better. This is meant to be an explicit reminder that the ideas in Chemistry are useful and applicable beyond the classroom and the solving of problem assignments.

IMPORTANT REQUIREMENT

Before the laboratory experiments, there are a few pages that outline general safety in the laboratory, as well as specific rules that must always be followed. Some information on first aid is also provided. Following this information, there is a LABORATORY SAFETY AGREEMENT, which all students are **required** to sign and give to their demonstrators. Please ensure that you have read the information and the safety agreement thoroughly before signing it. The demonstrators will keep the safety agreements until the final grades have been posted.

Safety in the Chemistry Laboratory[1]

Working in the chemistry laboratory is an interesting and rewarding experience. During your labs, you will be actively involved from beginning to end - from setting some change in motion to drawing some conclusion. In the laboratory, you will be working with equipment and materials that can cause injury if they are not handled properly. However, the laboratory is a safe place to work if you are careful. Accidents do not just happen; they are caused -- by carelessness, haste, and disregard of safety rules and practices. Safety rules to be followed in the laboratory are listed below. Before beginning any work in the lab, read these rules, learn, and follow them carefully.

General

1. Be prepared to work when you arrive at the laboratory. Familiarize yourself with the lab procedures before beginning the lab.

2. Perform only those lab activities assigned by your demonstrator. *Never* do anything in the laboratory that is not called for in the laboratory procedure or by your demonstrator. Never work alone in the lab. Do not engage in any horseplay.

3. Work areas should be kept clean and tidy at all times. Only lab manuals and notebooks should be brought to the work area. Other books, purses, brief cases, knapsacks, and external clothing (coats, hats, etc.) should be placed in the designated storage area. The staff of the third floor labs accepts no responsibility for lost or stolen items.

4. Clothing should be appropriate for working in the lab. Jackets, ties, and other loose garments should be removed. **ONLY closed shoes that cover your feet should be worn.**

5. Long hair should be tied back or covered, especially in the vicinity of open flames. Head coverings (veils, hats) should be securely attached to the head and safety glasses should rest on the EARS and NOT the head covering.

6. Jewellery that might present a safety hazard, such as dangling necklaces, chains, medallions, or bracelets should not be worn in the lab.

7. Follow all instructions, both written and oral, carefully.

8. **Safety glasses or goggles and labcoats should be worn at all times in the laboratory. There are no exceptions to this rule.**

9. Set up apparatus as described in the lab manual or by your demonstrator. Never use makeshift arrangements. Always use the prescribed instrument (tongs, test tube holder, forceps, etc.) for handling apparatus or equipment.

10. Keep all combustible materials away from open flames.

12. Never touch any substance in the lab unless specifically instructed to do so by your demonstrator.

13. Never put your face near the mouth of a container that is holding chemicals.

14. Never smell any chemicals unless instructed to do so by your demonstrator. When testing for odours, use a wafting motion to direct the odours to your nose.

15. Any activity involving poisonous or noxious vapours should be conducted in the fume hood. Check that the fumehood is ON and has FLOW.

16. Dispose of waste materials as instructed by your demonstrator.

LABORATORY SAFETY INFORMATION

17. Clean up all spills immediately.

18. Clean and wipe dry all work surfaces at the end of class. Wash your hands thoroughly with soap and water before leaving the lab.
19. Know the location of emergency equipment (first aid kit, fire extinguisher, fire shower, etc.) and how to use them.

20. Report all accidents to the co-ordinator or a demonstrator **immediately**.

Handling Chemicals

21. Read and double check labels on reagent bottles before removing any reagent. Take ONLY AS MUCH REAGENT AS YOU NEED!!

22. Do NOT return unused reagent to stock bottles.

23. When transferring chemical reagents from one container to another, hold the containers out away from your body.

24. When mixing an acid and water, *always add the acid to the water*.

25. Avoid touching chemicals with your hands. If chemicals do come in contact with your hands or skin, wash the contaminated area immediately.

26. Notify your demonstrator if you have any medical problems that might relate to lab work, such as allergies or asthma.
27. If you will be working with chemicals in the lab, DO NOT WEAR CONTACT LENSES. Change to glasses, or notify the co-ordinator.

Handling Glassware

28. Never handle broken glass with your bare hands. Use a brush and dustpan to clean up broken glass. Dispose of the glass in the **brown boxes labelled Glass Disposal**.

29. Never apply force when inserting or removing a stopper from glassware. Use a twisting motion. If a piece of glassware becomes "frozen" in a stopper, take it to your demonstrator.

30. Do not place hot glassware directly on the lab bench. Always use an insulating pad.

31. Allow plenty of time for hot glass to cool before touching it. Hot glass can cause painful burns. (Remember: Hot glass *looks* cool.)

Heating Substances

32. Exercise extreme caution when using a gas burner. Keep your head and clothing away from the flame.

33. Always turn the burner off when it is not in use.

34. Do not bring any substance into contact with a flame unless instructed to do so.

35. Never heat anything without being instructed to do so.

36. Never look directly into a container that is being heated.

37. When heating a substance in a test tube, make sure that the mouth of the tube is not pointed at yourself or anyone else.

38. Never leave unattended anything that is being heated or is visibly reacting.

First Aid in the Chemistry Laboratory

Accidents do not often happen in well-equipped chemistry laboratories if students understand safe laboratory procedures and are careful in following them. When an occasional accident does occur, it is likely to be a minor one. However, the following information will be helpful to you in the event an accident occurs.

First Aid

1. **Shock.** People who are suffering from any severe injury (for example, a bad burn or major loss of blood) may be in a state of shock. A person in shock is usually pale and faint. The person may be sweating, with cold, moist skin and a weak, rapid pulse.

   Shock is a serious medical condition. Do not allow a person in shock to walk anywhere. While emergency help is being summoned, place the victim face up in a horizontal position, with the feet raised about 30 centimetres. Loosen any tightly fitting clothing and keep the victim warm.

2. **Chemicals in the Eyes.** Getting any kind of a chemical into the eyes is undesirable, but certain chemicals are especially harmful. They can destroy eyesight in a matter of seconds. Because you **will** be wearing safety goggles/glasses at all times in the lab, the likelihood of this kind of accident is remote. However, if it does happen, flush your eyes with water immediately. Do NOT attempt to seek help before flushing eyes (except from your lab partner). It is important that

flushing with water be continued for a prolonged time - about 15 minutes. While flushing is continuing, the demonstrator and co-ordinator should be informed.

3. **Clothing or Hair on Fire.** A person whose clothing or hair catches on fire will often run around hysterically in an unsuccessful effort to get away from the fire. This only provides the fire with more oxygen and makes it burn faster. For clothing fires, throw yourself to the ground and roll around to extinguish the flames. For hair fires, use a cotton lab coat to smother the flames. Notify the co-ordinator immediately!

4. **Bleeding from a Cut.** Most cuts that occur in the chemistry laboratory are minor. For minor cuts, apply pressure to the wound with sterile gauze, and see the co-ordinator.

   If the victim is bleeding badly, raise the bleeding part above heart level, if possible, and apply pressure to the wound with a piece of sterile gauze. While first aid is being given, someone else should contact the co-ordinator.

5. **Chemicals in the Mouth.** Many chemicals are poisonous to varying degrees. Any chemical taken into the mouth should be spat out and the mouth rinsed thoroughly with water. Note the name of the chemical and notify the co-ordinator immediately.

6. **Acid or Base spilled on the Skin.** Flush the skin with COLD water for about 15 minutes continuously. Notify the co-ordinator immediately.

7. **Breathing Smoke or Chemical Fumes**. All experiments that give off smoke or noxious gases should be conducted in a well-ventilated fumehood. This will make an accident of this kind unlikely.

   If smoke or chemical fumes are present in the laboratory, *all* persons--even those who do not feel ill--should leave the laboratory immediately. Make certain that all doors to the laboratory are closed after the last person has left. Since smoke rises, stay low while evacuating a smoke-filled room. Thoroughly ventilate the room before going back to work.

[1]Courtesy of Prentice Hall, Inc.

LABORATORY SAFETY AGREEMENT

I have read carefully the article titled "Safety in the Chemistry Laboratory"

_____
Date

I have read carefully the article titled "First Aid in the Chemistry Laboratory"

_____
Date

I have made myself familiar with the location of the eyewashes, safety showers, fire extinguishers and the first aid kit.

_____
Date

I agree to wear my safety glasses/goggles and lab coat at all times during the laboratory session unless given express permission by the instructor to remove them.

_____
Date

I have read and understood the articles titled "Safety in the Chemistry Laboratory" and "First Aid in the Chemistry Laboratory" and I agree to abide by the rules and procedures described in the articles and this agreement. I will also abide by any other rules and regulations provided by my chemistry instructor.

_____ _____
(Name-please print clearly) (Student Number)

_____ _____
(Signature) (Date)

I understand the conditions of the policy on late submission of reports and I agree to abide by those conditions.

_____ _____
(Signature) (Date)

(After completing this form and signing it, please hand it in to your demonstrator.)