

Massachusetts College of Liberal Arts

**CHEM 150 & 152
INTRODUCTORY CHEMISTRY
LABORATORY MANUAL**

**Chemistry Department
Massachusetts College
of Liberal Arts**

Acknowledgements

It is probably impossible to thank all of the persons who contributed to the writing of this laboratory manual over a period of about fifteen years from its initial inception to its nearly final form. Nevertheless, I wish to thank Dr. Timothy J. Bergendahl, a former student and faculty member in the Chemistry Department at North Adams State College, for his start on writing the manual. Many of his ideas have been incorporated into the final form. Thanks are also due Drs. Michael A. Sabol and David M. Levine, colleagues in the Chemistry Department, for their many suggestions of ways to improve the manual and for their conscientious use and proofreading of the printed manual. I would also like to thank Mr. Terrence Farber, laboratory assistant and technician, for his many suggestions and for being more than willing to try new experiments and to modify older experiments. In many cases, what appears in the manual is an outgrowth of work done by Terry in getting experiments to work in our particular laboratories with our particular equipment.

I would also like to thank the College for its support during a sabbatical leave in the spring semester of 1994. It was this leave which allowed me the time to thoroughly learn WordPerfect for Windows, to research sources, to write the introductory materials, and to pull the pieces of the manual together into a more cohesive document.

Finally, although they are too numerous to mention by name, I would like to thank the students in *Introductory Chemistry* over the last ten or so years for their help in clarifying materials for the manual and for being the “guinea pigs” who lived with my typos and sometimes not-so-clear directions as the manual became refined from its first rough drafts. The final product is very much a consequence of their suggestions and questions over the years.

John M. C. Hess

To The Student

This laboratory manual has been written in the hope that it will make your study of chemistry—and particularly laboratory chemistry—easier and more rewarding. Each chapter in the manual is designated by a capital Roman numeral and pages are numbered with the chapter number followed by the page number within the chapter; thus VII–35 is page 35 in chapter VII. Experiments are titled with a letter from A through U; a brief Table of Contents which locates each chapter and experiment follows this section.

There are eleven chapters in the manual. The first three chapters in the manual discuss course and laboratory objectives and laboratory safety, treatment of numerical data and statistics, and the measurement of physical properties of substances. Each of the subsequent eight chapters starts with a general introduction to the theories and factual materials covered by the experiments in that chapter. Sample calculations, sometimes based on representative data, are included in this general introduction. Each experiment also has a series of introductory sections which include the learning objectives of the experiment, what to do before coming to laboratory in order to be prepared for the experiment, an introduction to the experiment itself (usually with pertinent equations and sample calculations), the stepwise experimental procedure, the calculations, and a list/illustration of the apparatus to be used. Data and report forms follow each experiment.

Past experience with this laboratory manual has shown us that the students who consistently prepare for the laboratory before coming to lab obtain better data, usually finish more quickly than other students, are safer workers, and have a better understanding of the experiment in terms of its limitations, the calculations, and the design of the experiment. They are also less likely to break equipment, use the wrong equipment or chemicals, or to misunderstand the type of data to be acquired. In short, there does not appear to be any substitute for preparedness in the laboratory. We expect that every student, before coming to laboratory, will have read the experiment with its introductions and familiarized himself/herself with the background of the experiment.

Finally, a quick word about chemistry and its study. There are no shortcuts to studying chemistry. It is a science which many students find difficult, but it is also perhaps the most central science upon which the biological and health sciences are based and is well worth learning. The only way to learn chemistry, and to be able to use it later, is to *work* at learning it now while help is available to you through the course instructors and tutoring. Set up good study habits. Do the assigned problems and readings in a timely manner. Prepare for laboratory and recitation meetings. Turn assignments and laboratory reports in on time. Don't be afraid to ask questions. And, above all, attend classes and labs.

**PLEASE NOTE THAT THIS MANUAL WILL BE USED BOTH SEMESTERS.
TAKE CARE OF IT ACCORDINGLY.**

Note: In each case below, the page number given after the title is understood to be preceded by the Roman number which designates the chapter; the format in the manual is II-6, for example, for chapter II, page 6. No number is shown for the initial page (1) in each chapter.

I. Introduction

Objectives of the introductory chemistry experience	1
Safety in the chemistry laboratory	1
Safety-related items	3
Equipment & chemical items	3

II. Working with data and results

Introduction	1
An overview	1
Types of errors	1
The significance of numbers	2
Why fret about significance?	2
Integer numbers	2
Noninteger Numbers-data	2
Arithmetic operations with significant figures	4
Addition and subtraction	4
Multiplication and division	5
Some useful tricks	6
Showing nonsignificant figures	6
Why bother?	6
Treatment of numerical data in small samples	7
Introduction	7
The mean	7
The mean deviation	7
The relative mean deviation	8
Rejection of values-the Q-test	8
Accuracy and error	10
Presenting data and results	11
Graphical methods of presenting and analyzing data and results	11
Choosing the graph paper	11
Choosing the scale	11
Plotting the data points	12
Interpretation of graphs	12
Computerized analysis of linear or linearized data	14

Table of Contents

III. Physical properties and their measurement

The states of matter	1
Introduction	1
A macroscopic overview of matter	1
Kinetic molecular theory: a model for the states of matter	2
The metric system	2
Measurement of volume	3
Estimating volume	3
Graduated cylinders	3
Burets, pipets, and volumetric flasks	4
Measurement of mass	7
Measurement of density	10
Direct methods of measuring density	10
Indirect methods of measuring density	11
Measuring color	11
The nature of color	11
Spectrophotometers	13
Beer's law	15

IV. Experiments involving gases

Measurement of temperature, pressure, volume and mass	1
Temperature	1
Pressure	1
Volume	3
Direct measurement methods	3
Indirect measurement methods	4
Mass	4
The ideal gas law	4
The combined gas law	5
Dalton's law of partial pressures	5
Standard temperature and pressure	5
Experiment A: Determination of the standard molar volume of oxygen gas	7
Experiment B: Determination of the molar mass of carbon dioxide	13

V. Experiments with liquids and solids

Introduction	1
Liquids	1
Solids	1
Experiment C: Determination of the densities of some liquid and solid samples	2
Experiment D: Determination of the density & composition of a salt solution	7
Experiment E: Determination of the density of a single crystal by a flotation method	13
Experiment F: Determination of Avogadro's number from crystal structure and data	19

VI. Experiments in chemical stoichiometry

Mass relationships in formulas	1
Introduction	1
Atomic weight and the mole	1
Formulas	1
Experiment G: Determination of the atomic weight of magnesium	2
Experiment H: Synthesis and purification of aspirin	9

VII. Experiments with solutions

Concentration units for solutions	1
Introduction	1
Percent composition	1
Molarity	1
Molality	3
Mole fraction	3
Colligative properties of solutions	6
Introduction	6
The ideal approximation	6
Types of colligative properties	6

Table of Contents

Experiment I: Determination of molar mass by freezing point depression 8

Titration: acid/base and redox systems 19

Introduction 19

Acid-base titrations 19

Acid-base reactions 19

Detecting the "end point" 20

Determining molarity 21

Determining weight percent solute 22

Determining the molar mass of the solute 23

Experiment J: Determination of the molarity of a sodium hydroxide solution by titration against a standard oxalic acid solution 24

Experiment K: Determination of percent acetic acid in a vinegar by titration 33

Experiment L: Determination of the equivalent mass of a solid acid by titration 41

Redox titrations 49

Introduction 49

Oxidation states or numbers 49

Balancing redox reactions 50

Detecting the end point in redox titrations 53

Calculations in redox titrations 53

Experiment M: Standardization of potassium permanganate solution against sodium oxalate 55

Experiment N: Determination of percent oxalate in an unknown by titration with standard potassium permanganate solution 63

VIII. Experiments using instruments

Measurement of pH in solution 1

Introduction 1

pH meters and electrodes 1

Experiment O: Formulation of a buffer solution and study of buffering action 3

Experiment P: Determination of the concentration of a substance by spectrophotometry 17

IX. Experiments with heats of reaction

Basic thermodynamics 1
 Introduction 1
 Heat in chemical reactions 1
 Finding values for ΔH 2
 Summation of heats of reaction: Hess's law 3
 Calorimetry: measuring heat 3

Experiment Q: Determination of heat of ionization by calorimetry 5

X. Experiments with rates of reaction—kinetics

Basic kinetics 1
 Introduction 1
 Rates of chemical reactions 1
 Rate and concentration: the rate law 1
 First order rate equations 2
 Half-life of first order reactions 3
 Dependence of rate on temperature—the Arrhenius equation 4

Experiment R: Kinetics—rate constants and activation energy for the decomposition of hydrogen peroxide 5

XI. Experiments in qualitative analysis of cations

Solubility & reactions 1
 Introduction 1
 Chemistry of group 1: Ag^+ , Hg_2^{2+} , Pb^{2+} 1
 Chemistry of group 2: Hg^{2+} , Pb^{2+} , Bi^{3+} , Cd^{2+} 2

Experiment S: Analysis of a liquid solution for analytical group 1 cations—known & unknown samples 4, 5ff

Experiment T: Analysis of a liquid solution for analytical group 2 cations—known & unknown samples 4, 13ff

Experiment U: Analysis of a liquid solution for analytical group 1 & 2 cations—unknown sample 4, 21ff

WRITING A LABORATORY NOTEBOOK

General Information

- Date
- Title of the experiment
- 5 minutes pre-lab quiz before start of each experiment
- Must submit BOTH lab notebook and data sheets (Monday lab group will submit their lab notebooks on Wednesday before 3 p.m. while both the Tuesday and Wednesday lab groups will submit theirs on Friday before 3 p.m.); reports handed in after 3 pm will receive a 1 point deduction per hour late. Put your lab notebook on top of my office desk at Bowman 303. You MUST pick up your graded notebook outside Bowman 303 on a box labelled "graded labs." This is important because you will need your notebook to write pre-lab sections during the weekend.
- It is **YOUR** responsibility to make sure that your notebook is signed and dated by the instructor before you begin and NOT at the end of the experiment
- Before coming to the lab, you should have all the sections up to the procedure written. You will NOT be allowed to do the experiment without these sections. During the experiment, you will make your observations and data collection. At home, you will discuss the data and make conclusions.
- Laboratory notebooks should be hardback bound notebooks – you can stick worksheets in where needed
- Start each experiment on a new page
- Use only the right side of the page
- Writing must be done black ballpoint pen; pencil should **NEVER** be used
- Use plain language – passive; past tense
- All entries in the laboratory notebook are to be made in ink. If errors are made, simply cross them out with a single line. Never obliterate entries; at a later time you may find that this information is useful. All data and observations are to be entered directly into the notebook, not on scraps of paper.
- For each experiment, organize your notebook into sections beginning with appropriate headings. (e.g. Title, Purpose, Procedure, Data, etc.).

ORGANIZING YOUR NOTEBOOK

Anyone should be able to pick up your notebook and understand what you have written. This must be the main thing - you are writing for someone else. If the writing is clear to them, then it certainly will be to you. Achieving this requires some organization as well as a certain style.

Title page: Give a page to state your name, address (you might lose the book) and a brief indication of its purpose, "Chemistry Practicals," for example.

Table of contents: Give two pages to the Contents so that you can list the experiments and find them easily when needed. But you will need to number the pages; tedious but essential. Do it when the notebook is new.

Table of abbreviations: Use abbreviations a lot - they save time and effort. If you use them, give a table to explain them.

Purpose

This is a brief description of why you are performing the experiment and what you expect to learn. Do not copy the purpose straight from the lab book!

Procedure

Safety!!!! Part of chemical education is the instruction in handling potentially hazardous materials safely. Make a list of all the chemicals to be used in the experiment and explain their hazardous nature as noted BOTH in the lab manual and the web site: <http://www.msds.org/>. Explain how you will handle the chemicals – the precautions, etc.

Make a list of all the **apparatus** needed for the experiment

In writing the procedure, use simple, direct statements or a bulleted or numbered list of instructions. A step-by-step version written in your own words is also acceptable. This should be detailed enough that someone else could use it to replicate the experiment. Complete sentences are not necessary and diagrams can and should be used where appropriate.

Example:

- clean crucible
- dry to constant wt. w/heating
- add about 5g unknown
- heat gently 1st, then strongly for 10-15 min
- cool-weigh-reheat-cool-weigh-repeat to constant wt.
- etc

Data and Observations

Before you leave lab, all relevant measurements and observations must be recorded. Include anything noteworthy that you observe such as color and temperature changes, formation of a precipitate, etc. Large collections of data should be organized into tables for clarity. All numerical entries must have appropriate units. It is not necessary for this section to be extremely neat, but it should be legible and in some sort of order. If you make a mistake recording data cross it out with ONE line. If you have to cross out an entire trial use a large X, and include a brief note as to why you did it. Don't forget to record the numbers of any unknowns.

The observations you make and the data that you record will lead to the acceptance or rejection of your hypothesis, and will decide what future experiments may be done. The observations and data are therefore central to the whole exercise. They need to be:

- recorded honestly
- recorded as you go along, in the notebook, in ink, immediately.
- do not trust to memory, even for a minute or so - someone talks to you, and that data's forgotten.
- do not trust to memory; you do not want your mind occupied with trivial things and small details. You need to keep the overall experimental plan in mind.
- do not use odd scraps of paper or the edge of your lab coat to record data
- the raw data is precious - the data is treated with the care you'd bestow on a family heirloom
- the data must be recorded as completely as is possible. Don't worry too much about interpreting the data as you go along, and don't worry if some of the observations appear banal. For an

example of how omission of even the simplest things can dramatically affect the outcome of an experiment, go here.

- use good penmanship. Take care with numbers - never over-write, always cross out erroneous material with a single line and re-write the correct data.
- NEVER use Tipp-ex or other white-out liquids.

Calculations

Calculations should be presented in such a way that the person grading the lab would be able to follow them. For experiments that involve more than one trial, it is only necessary to show one set of calculations and records the final result for each trial in a table. Be sure to include units where appropriate. The use of tables (or graphs) for data and calculations is always recommended. This provides a quick and easy (pictorial) way for the grader to find the appropriate information.

Discussion/Conclusion

This is a recap of the lab and the place to show what you learned. A general outline of the components of a good discussion follows. (This should all fit on one page or less.)

Your conclusions should state

- what you found out;
 - whether the hypothesis was supported or not, if appropriate;
 - the error limits on your answer(s); a quantitative assessment of error should be made if possible, so that you can decide whether the use of a measuring cylinder rather than a pipette, say, really did make any meaningful difference to the result;
 - suggestions for improvement in experimental design, if appropriate; the error analysis will be useful here.
 - what to do next, if appropriate.
1. Problem addressed by the laboratory: what were you trying to accomplish by this experiment. (Don't simply rewrite the purpose.)
 2. How did you go about addressing this problem? This is not a repeat of the procedure. Briefly describe the general technique and the principle behind the technique if appropriate.

Note: the above two items can be treated in as few as one or two sentences.

Example:

This experiment was performed for the purpose of determining the concentration of acetic acid in an unknown sample. This was accomplished by titrating the sample with a known concentration of sodium hydroxide until the phenolphthalein endpoint was observed.

BIBLIOGRAPHY

Make a numerical list of the sources where you obtained your information. For example:

1. <http://www.msds.org/>
2. Please see examples in http://www.rod.beavon.clara.net/lab_book.htm

Remember....

“Science does not take place on the pages of textbooks or learned journals, but it is recorded there. The quality of any work is only as good as the report that remembers it when the test-tubes have long been washed up” quotation from <http://www.msds.org/>

GRADING SCALE FOR LAB NOTEBOOK AND DATA SHEETS

ITEM	POINTS
Date	1
Title of the experiment	1
Table of abbreviations	1
Purpose	3
Procedure:	
(a) safety and precautions	5
(b) Apparatus	2
(c) Instructions (written experimental procedure)	5
Observation	5
Data / calculations	10
Discussion/Conclusion	5
Bibliography	2
TOTAL POINTS	40

Figure 1 shows most of the glassware and equipment you'll be using in the weeks ahead.

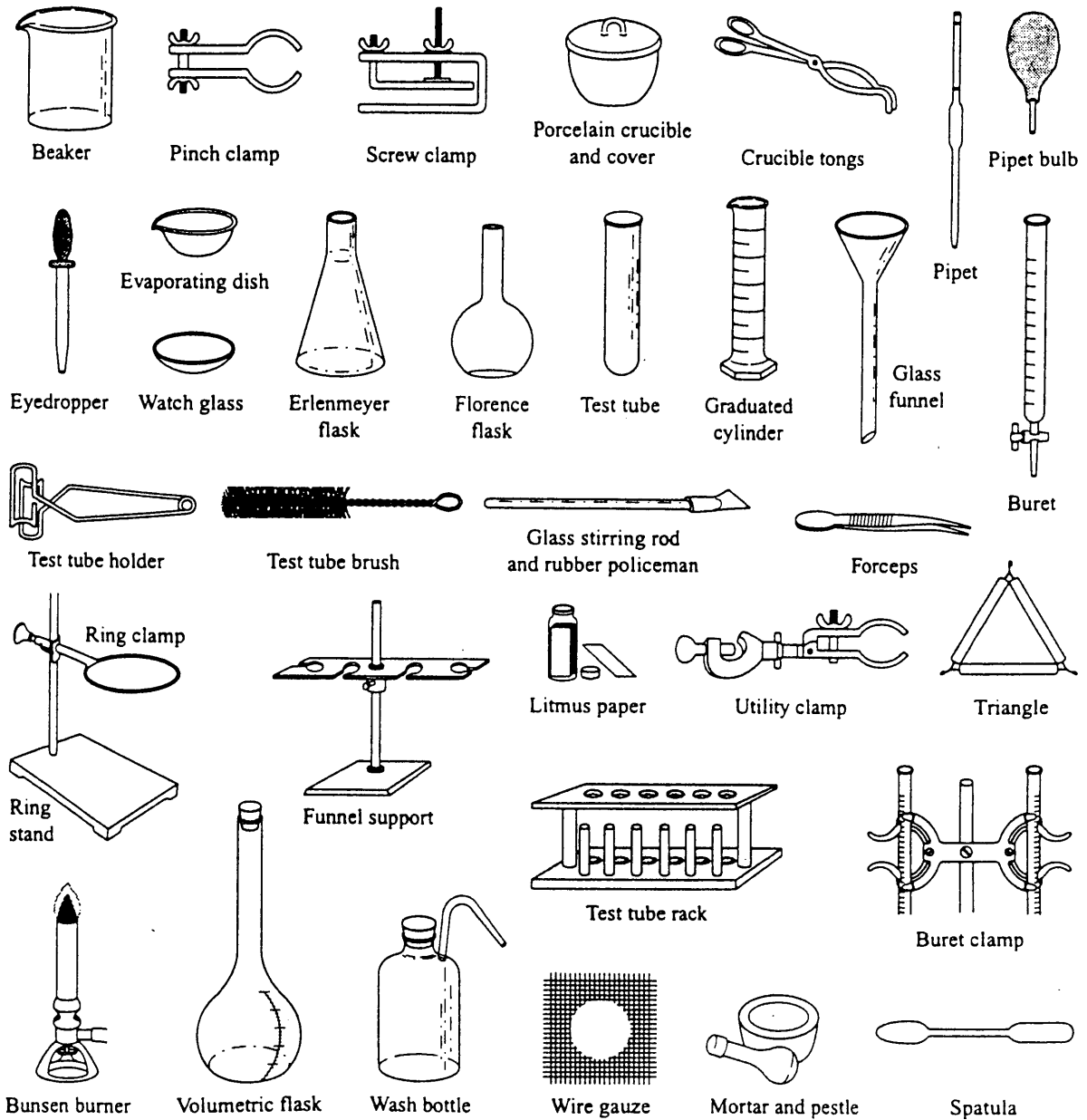


FIGURE 1

I. INTRODUCTION

OBJECTIVES OF THE INTRODUCTORY CHEMISTRY EXPERIENCE

The laboratory segment of Introductory Chemistry has the following objectives:

- 1 To develop in students the skills necessary:
 - (a) to obtain and evaluate data and results;
 - (b) to critically evaluate different methods of measurement; and
- (c) to analytically solve chemical problems and calculations.
- 2 To illustrate and exemplify topics which are covered in the text and/or the lecture segment of the course.
- 3 To teach proper and safe laboratory practices.

SAFETY IN THE CHEMISTRY LABORATORY

Despite the image often encountered in the popular media, chemical laboratories are not peopled by mad scientists and laboratories are relatively safe places in which to work. As in any other endeavor—such as driving a motor vehicle—one needs to know what one is doing, and one needs to exercise a reasonable degree of caution.

The following paragraphs will acquaint you with some of the more fundamental guidelines for safe laboratory practice. This is by no means an exhaustive list—entire books have been written on laboratory safety—but is rather a basic reference list for the beginning student of chemistry. Each experiment in this manual will have its own specific set of guidelines for carrying out the experiment in a safe manner. Your laboratory instructor will start each laboratory with the precautions of which you need to be aware in the scheduled experiment. All of these sources, taken together, will make up a safety primer for you. In the end, though, safety in the laboratory will depend on you and how well you *understand* and *use* the safety guidelines that are available to you.

To begin, most laboratory accidents at the undergraduate level occur because students fail to read and/or heed the laboratory directions. There is no substitute for being prepared for the experiment of the day. The best instructor, the most well-written manual, can not prevent the unprepared student from stumbling into unsafe areas and practices.

Always read the lab directions carefully before coming to lab and highlight those items which deal with safety.

Plan to be in lab on time. The laboratory instructor's lecture at the beginning of the lab period will cover specific precautions to be

observed. Take notes. Ask questions if you do not understand the directions.

The most vulnerable part of the human body—assuming that you avoid eating, drinking, or breathing toxic chemicals—is the eye. Chemicals getting into the eye can cause severe irritation and permanent injury unless the chemicals are removed and the eye treated promptly. Even seconds count in determining whether an eye injury will be merely uncomfortable or damaging.

Always wear safety glasses or goggles. Safety glasses and/or goggles are supplied in the laboratory.

Flush chemicals from the eyes promptly with warm water from the gooseneck faucet at any workspace. Use hands to hold eyelids open for thorough flushing. Do not hesitate or look for the instructor—do this immediately! Students who must wear contact lenses should notify the lab instructor at the first lab meeting. Because of the difficulty in flushing behind contacts, contact lenses should not be worn in lab unless there is no other choice.

Some experiments call for the use of flammable materials or chemicals. Care should be taken in using such materials or chemicals around open flames and hot plates.

In the unlikely event of a fire in the laboratory, do not move toward the fire; you will only add to the confusion and block the instructor's access.

In case of a small fire—paper, chemical in a

INTRODUCTION

container, etc.—the fire should be extinguished by smothering with a cover of some sort or by sweeping (carefully) the burning material into the nearest sink and running water on it.

Large fires will require the use of a fire extinguisher. Leave the fire area. The lab instructor will fight the fire. If the fire is out of control, leave the lab and the building. Find a nearby alarm and use it!

Safety showers are located near the doors in the laboratory. If your clothing is on fire or you have spilled chemicals over yourself, the nearest shower should be used to extinguish the fire or flush the chemicals. Small fires and spills should be dealt with in a sink with running water.

When a gas burner is well-adjusted, its flame will be nearly invisible. Be constantly aware of your, and other's, burners. Turn burners off when not in use. Do not lean into or over the flame.

The most common accident in chemistry labs is the spilling of chemicals. Many chemicals can cause damage to clothing, books, and skin. Avoid spilling chemicals by using care when handling them and by using the proper techniques for transferring them. Your lab instructor will demonstrate these techniques in the prelab lecture.

Clean up all spills promptly. Use a damp or wet towel or sponge for small spills. For large spills, notify the lab instructor who will use specialized spill equipment.

Flush chemicals from skin or clothing promptly, either at the nearest sink or under the safety shower. Watch where you lean, sit, or place materials in case some other student did not clean up a spill.

Most spills, fires, and other accidents can be avoided by simply being careful and knowledgeable about what you are doing. Most errors in lab come about through failure to read carefully.

Read labels twice. Many chemical names look alike and solutions may have the same name with different concentrations.

Never return any chemical to the container from which it came—you may contaminate the remaining stock.

Dispose of excess or used chemicals properly. Most will be placed in waste containers in the hood area. Your lab instructor will show you where to dispose of waste.

Good results in the lab are often dependent on the cleanliness and condition of the equipment used in the experiment. For budgetary reasons it is not possible for each student in the introductory-level course to have his/her own glassware. Instead, students share glassware and apparatus across different sections of the laboratory. It is essential that each student using such shared equipment return it in clean, ready-to-use condition to its proper place in the laboratory.

All glassware should be checked before use (and after) for chips or cracks. The instructor should be consulted about any glassware found to be chipped or cracked. Usually such glassware will be discarded but some such pieces may be repairable so it is best to check with the instructor.

Care should be exercised in handling glassware and other equipment. Most scientific equipment is relatively expensive and easily broken.

Finally, below is a list of procedural guidelines which, if followed, will make the laboratory a safer work place.

NEVER, under any circumstances, fool around in the laboratory. This includes throwing things, squirting liquids, combining chemicals other than as called for in the experiment, and any kind of horseplay.

Learn how to use equipment, such as balances, properly and use such equipment gently. Do not attempt to fix equipment; report problems to the instructor.

Keep common areas in the lab—balance room, hoods, benches, sinks, etc.—clean and neat. Pick up and clean up after yourself.

Wear old clothing to the lab if possible. Avoid loose or frilly clothing which may be subject to accidentally coming in contact with flames or chemicals. Shorts and bare feet are not permitted in the lab.

If you don't understand how, why, when, where, or what you are doing, ask your instructor. Don't rely on your fellow students to answer questions that may affect your safety or your grade.

Spend some time getting acquainted with the names, locations, and uses of equipment, glassware, and safety devices. Your laboratory instructor will be pointing these out to you and instructing you in their proper care and use. Meanwhile, for your reference, a list of some items, along with their locations in the laboratory, follows. "Front" of the lab is defined as the end of the lab into which one normally enters.

SAFETY-RELATED ITEMS:

Eye wash: Located on wall by entrance. (Use sink goose-neck faucet instead.)

Exits: At front and back of lab. Back exit leads directly to stairs.

Exhaust hoods (Hoods): In wall between exits. Used to prevent generated fumes or gases from escaping into the lab and to shield possible violent reactions.

Fire extinguishers: On walls near laboratory exits. Additional extinguishers are in small lab off 301 and in 302 and Stockroom. For emergency use only!

Safety showers: At laboratory exits. Activated by pulling triangular handle down. For emergency use only!

EQUIPMENT & CHEMICAL ITEMS:

Balances: There are two types of balances. Analytical balances are located in a balance room at the back of the lab. Toploading balances are on benchtops at the front and back of the lab.

Computers: On the end bench at the front of the lab.

Distilled water: In plastic carboys on benches at ends of lab and at back wall. Small (500 mL) colorless squeeze (wash) bottles of distilled water are at each work station. Use distilled water for final rinses of glassware and for making up any solutions. *Red wash bottles contain acetone, a flammable solvent, which may be used to dry glassware in some experiments.*

Gas, natural: Available from gas cocks on bench tops at each work station. Gas is off when handle of cock is at right angle to nozzle, on when aligned with nozzle. A master valve for gas in the entire lab is located on the wall inside the front entrance.

Glassware: Standard items such as beakers (150, 250, 400, and 600 mL) and flasks (Erlenmeyer(conical) 250 mL), test tubes, etc. are located at each work station in the top drawer closest to the hoods. Glassware for specific experiments will be on a front bench or set out on the work station bench tops.

Hardware: Burners, tripods, triangles, test tube clamps and racks, burner hose, etc. will be found in the drawer next to the glassware drawer at each work station. Hardware for specific experiments will be on a front bench or set up on each work station bench top.

Ovens: On the end bench nearest door at front of lab. There are two ovens; be sure you are using the right one for your experiment.

Setups, experiment: Chemicals for experiments will usually be in the hoods or on the front bench. Equipment will usually be set out or assembled on the bench top at each work station. When necessary, equipment will be set up in the hoods.

Utilities, laboratory: Located on bench tops, at work station sinks, and on hood faces. Each work station has hot and cold water, gas, compressed air, vacuum, and steam. The compressed air is under about 90 psi pressure and should be turned on carefully; it may have some oil in it so shield yourself and clothing from the flow. Steam is **HOT** and should only be turned on when connected to a hose directed to the sink or a steam bath. The hoods have external controls for gas, cold water, air, and vacuum nozzles inside the hood.