CHEMISTRY 1010
Lab Manual

University of Louisiana at Monroe
Freshman Chemistry Lab Staff
Revised: Fall 2010

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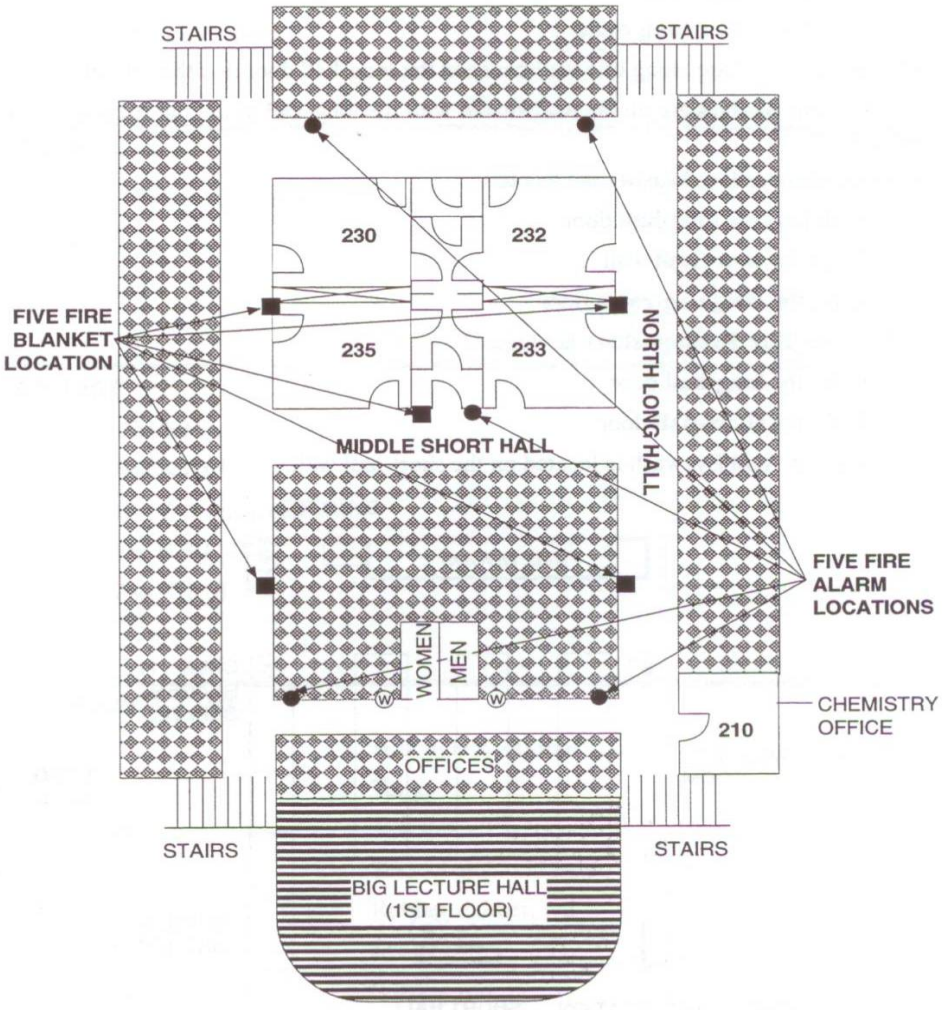
MAP

CNSB Second Floor

Locations of fire alarms, fire blankets, and stairwell exits are indicated on the map of the second floor below. Familiarize yourself with the location of these important safety features.



Floor-plan of the Freshman Laboratories

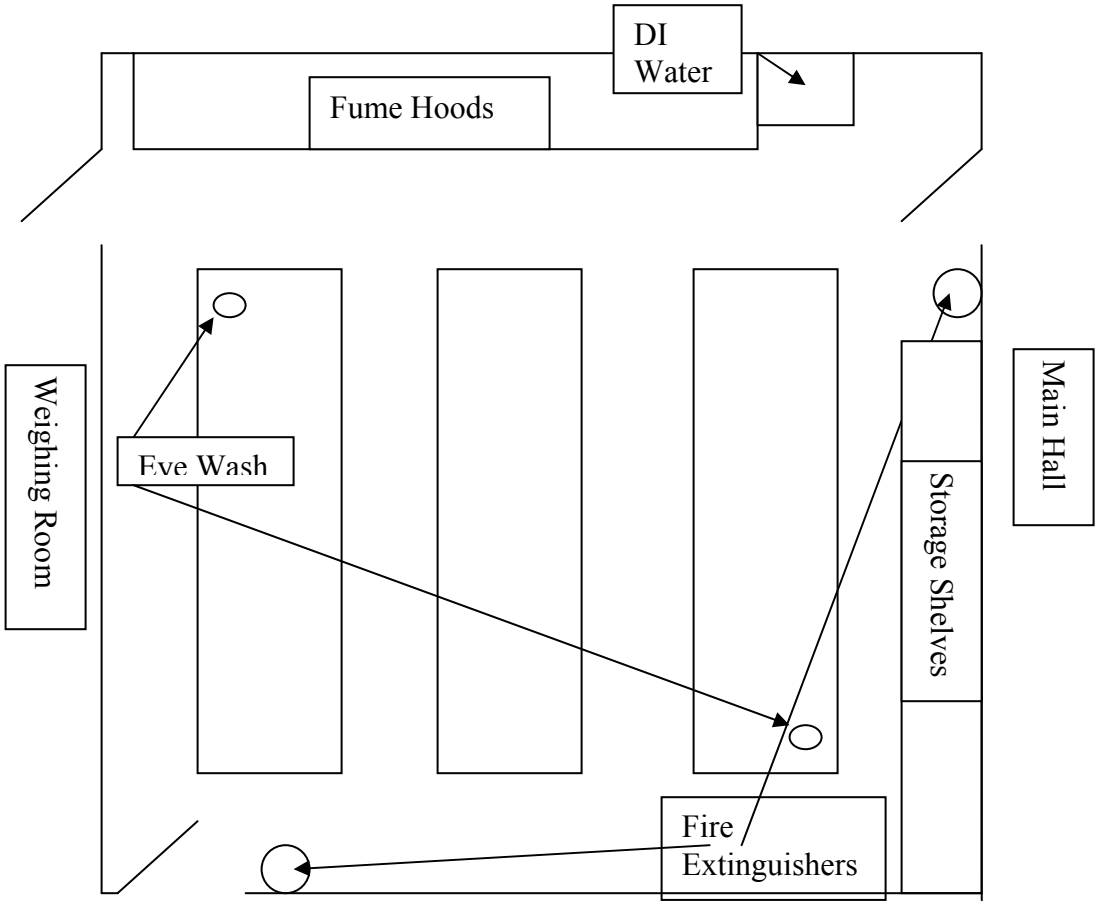
Become familiar with the location of the important safety devices in the freshman laboratories. Since there are four freshman laboratories and each has a different orientation, you should learn the locations relative to the common features of the labs.

Exits: Each room has three exits, one each on the north and south wall near the fume hoods. The third is located on the wall opposite the fume hoods and diagonal to the DI water station.

Fume Hoods: Four fume hoods are located on one wall of the lab between the main entrance door and the door to the weighing room.

Eye Wash/Shower Stations: Two eye wash/shower stations are located in each laboratory. One is located by the sink on the end of the bench near the fume hood and the weighing room door. The second is located diagonally across the lab at the end of the bench.

Fire Extinguishers: Two fire extinguishers are located in each laboratory. One is located on the wall by the main entrance door. The second in located diagonally across the lab by the second exit door.



CHEMISTRY 110 Desk Assignment Sheet

PLEASE PRINT!

Student's Name_____

CWID_____

Section #_____ Room #_____ Desk #_____

ITEMS	**QUANTITY**
Basket	1
Beaker, 100 or 150 ml	2
Beaker, 250 ml	1
Beaker, 400 ml	1
Casserole, 60 ml	1
Clamp, Test-tube	1
Cylinder, Graduated, 10 ml	1
Cylinder, Graduated, 100 ml	1
Funnel, Short-stem, filling	1
Flame Loop	1
Flask, Erlenmeyer, 125ml	2
Red Rubber-Dubber	1
Spatula	1
Stirring Rod	1
Test Tube, 13 X 100	10
Test Tube, 15 X 125	10
Test Tube Brush	1
Test Tube Rack	1
Tongs	1
Wash Bottle	1
Watch Glass	1

Student's Signature_____

Check in by_____ ***Date***_____

Check out by_____ ***Date***_____

I MUST PROVIDE AND WEAR SAFETY GOGGLES DURING EACH LAB PERIOD.

Initials_____ Date_____

Safety

Laboratory Safety Rules

(Rules listed are minimum standards and additional safety requirements may be imposed by each individual instructor).

Eye protection **must** be worn in the laboratory and in the balance room at all times.

Eating and/or drinking in the laboratory and in the pre-lab lecture room are forbidden.

Students wearing shorts and/or shoes that are not solid and cover the foot will not be allowed to work in the laboratory. A lab coat or apron which extends below the knee is required if clothing does not cover the knee or if the mid-riff is exposed. Stated dress code conditions may not be waived; improperly clad students will be told to leave the laboratory.

Students must not handle broken glass. If any glass item breaks, inform the Instructor. A brush and plastic dust/glass pan that can be used to collect glass fragments are located on the wall near the DI water container. Broken glass may also be found on the floor, in your drawers, and in the sinks. Don't reach carelessly into drawers or sinks, and wear shoes that protect you. When broken glass is present see that it gets cleaned up. All broken glass should be placed in a box marked for that material only. Your instructor will help you.

Students may not use cell phones in the laboratory; laboratory work demands full attention, and responsible behavior (see University cell phone policy). Cell phones must be turned off during pre-lab lecture and in the laboratory.

Students may not entertain visitors in the laboratory. Again, the laboratory work demands full attention and appropriate safety precautions. (Also, visitors are not authorized to be there.)

When no longer required, equipment should be returned to its original space (e.g. drawer or side shelf), and solids must not be disposed of down the sink. Most solid waste can be placed in the trash receptacles located at the end of each bench. You may be instructed to place some waste solids and liquids in marked containers in the hoods.

Eye Protection:

Because tragic and irreversible eye injuries are a constant danger, eye protection must be worn at all times in the lab. The best protection is given by goggles which have a strap to pull them flush with your face. However, we currently allow safety glasses which have side-shields (so-called "weed-eating" glasses). You may wear your regular prescription glasses if they can be worn under goggles or if they are fitted with side shields. If you store your glasses/goggles in your drawer, your first action during any lab session is to put them on; do not make any other moves toward lab work before that. If you get anything in your eye, proceed to the eye wash and use it. But let your instructor know; don't be quiet about it.

Although dress code may be mitigated by religious beliefs, eye protection is not negotiable – any instructor can remove you from the lab for disregarding this rule.

Corrosives:

There are some very corrosive chemicals used every day in the lab. You must learn to handle them with respect. You may get small amounts on your hands and clothing. Wash immediately and thoroughly. The rarer but greater danger is from large spills on the body. Don't be embarrassed to use the eyewash/shower. You may need to shed some clothing. Just do it and use lots of water. Concentrated H_2SO_4 in large amounts should be wiped off first but for all the other corrosives, get the water going fast. The solutions to be most concerned about are 18 M H_2SO_4, 16M HNO_3, 12M HCl, 8M NaOH, and 15M NH_3. Help your neighbors if they have a spill, especially if on the body, and let your instructor know immediately.

Poisons:

Consider every chemical in the lab a danger in this respect but learn from your instructor those chemicals that are most dangerous. No eating or drinking can be tolerated in the lab. Even chewing of gum in lab is forbidden. Wash your hands thoroughly before you leave lab after completion of the day's experiment.

Fire:

Fire danger is not unusually high in this lab, however we do use the Bunsen burner continually. Be careful of your hair and clothing (particularly in cool weather). Before lighting your burner, look to see that it is whole and well connected. Always follow the procedure given by your instructor for lighting the burner. Always turn a burner off by reversing the lighting procedure. Though we use few flammables in this course, and these only in small amounts, large quantities may be in the hoods. Do not use the burner in the hoods that contain flammables. Your instructor will indicate hoods in which a burner may be lit.

Gases:

Poisonous, corrosive, and noxious gaseous compounds are used regularly in the course. Pay attention to these:

- H_2S is extremely poisonous as well as horribly smelly. You will smell it! However, it is critical to avoid direct inhalation even of the dilute solution of H_2S we make up. Never convert a basic solution of sulfide to acid unless under a functioning fume hood.
-
- HCl and NH_3 concentrated solutions give off gases which are very corrosive and damaging to your respiratory system. Use these carefully.
-
- HCl and SO_3 are created by boiling down acid solution. Do such work in the hood!

Solubility

Solubility, the extent to which one substance (the solute) can be dissolved in another (the solvent), is quantitatively conveyed by the concentration of a saturated solution, that is a solution in equilibrium with pure solute. In terms of molar solubility, where concentration is on the molar scale, the obvious lower limit is zero (or virtually so) and the upper limit of observed solubility for ionic substances in water are in the 'tens'. For example, the molar solubility of KCl is about 4.2 M and that of AgC1 is about 1.7 x 10^{-5} M (at 25 ºC). Another way solubility is communicated is the solubility product constant, K_{sp}, the product of molar concentrations in a saturated solution, with each ionic molarity raised to the power of its coefficient in the solubility equation.

$$AgCl(s) \rightleftarrows Ag^{+}(aq) + Cl^{-}(aq);\ K_{sp} = [Ag^{+}]^{1}[Cl^{-}]^{1} = 2.8 \quad X \quad 10^{-10}$$

$$PbCl_{2\,(s)} \rightleftarrows Pb^{2+}{}_{(aq)} + 2\ Cl^{-}{}_{(aq)},\ K_{SP} = [Pb^{2+}][Cl^{-}]^{2} = 1.6 \times 10^{-5}.$$

You will find K_{sp}'s recorded for sparingly soluble salts (sometimes called "insoluble" salts).

Our current interest is **qualitative** and the rules to be expounded here are to be used to give us a quick expectation as to whether a salt is "soluble" or "insoluble" ("sparingly soluble" is a better description). The rules below have been tailored for our needs in this course and you should **learn them in detail** and apply them. Experiments dealing with qualitative analysis of cations and anions in aqueous solution (Experiments 2, 3, and 4) will require that we know whether certain salts are sparingly soluble, thus liable to form precipitates.

Solubility Rules

1) All salts of Na^{+}, K^{+}, and NH_4^{+} are soluble.
2) All acetates and nitrates are soluble.
3) Exception: $AgC_2H_3O_2$ is moderately soluble.
4) All chlorides, bromides, and iodides are soluble.
5) Exceptions: Pb^{2+}, Hg_2^{2+}, and Ag^{+}
6) All sulfates are soluble.
7) Exceptions: Ba^{2+}, Pb^{2+}, and Hg_2^{2+}
8) Note: $CaSO_4$ and Ag_2SO_4 are moderately soluble
9) All arsenates, borates, carbonates and phosphates are insoluble
10) Exceptions: Na^{+}, K^{+}, and NH_4^{+}
11) All hydroxides are insoluble
12) Exceptions: Na^{+}, K^{+}, and NH_4^{+}. Note: $Ca(OH)_2$ and $Ba(OH)_2$ are moderately soluble.
13) All sulfides are insoluble

14) Exceptions: Na^+, K^+, and NH_4^+

15) Note: *CaS*, *BaS*, *and MgS* are slightly soluble

The rules above deal qualitatively with solubility of salts in unadulterated water. As you may already realize, there are ways (we're particularly interested in two) by which a third component in the solution can change the apparent solubility of a salt. The two categories of most interest in this course are 1) strong acids and 2) complexing agents.

In the first case insoluble salts of weak acids usually show increased solubility, sometimes dramatic increases, if strong acids (high $[H^+]$ concentration) are added. The result is formation of the weak acid, usually quite soluble itself, with the release of the cation. Such as

$$ZnS_{(s)} + 2\,HCl_{(aq)} \rightleftarrows Zn^{2+}{}_{(aq)} + 2\,Cl^-{}_{(aq)} + H_2S_{(aq)}.$$

This is, of course, a chemical reaction, not just simply ZnS dissolving. Nevertheless, we could now discern the presence of zinc ion and hydrogen sulfide for that matter, in the solution.
Another important case is where insoluble salts are 'solubilized' by reaction with a cation complexing agent. These agents are "Lewis base" molecules or ions called **ligands**) which react with cations which are "Lewis acids". An examples is

$$AgCl(s) + 2\,NH_{3\,(aq)} \rightarrow [Ag(NH_3)_2]^+{}_{(aq)} + Cl^-{}_{(aq)},$$

in which silver chloride is seen to be dissolved in ammonia solution by formation of a complex cation, often denoted by brackets. A new salt is formed, diamminosilver chloride, which is soluble. We also see how this reaction is readily reversed by addition of strong acid, H^+.

$$[Ag(NH_3)_2]^+{}_{(aq)} + Cl^-{}_{(aq)} + 2\,H^+{}_{(aq)} + \rightarrow AgCl_{(s)} + 2\,NH_4{}^+{}_{(aq)}.$$

General Terms and Fundamental Techniques

The Pasteur pipets we use are disposable, which is to say that when they become too difficult to clean or become unusable for any other reason, toss them and get another. As long as you are not wasteful, the disposable pipets will be available. You may have occasion to use an old-fashioned Pasteur pipet which consists of a glass pipet (dropper) fitted with a rubber bulb (called a rubber-dubber). To be useful the glass pipet should fit snuggly with the 'red' or the 'latex' rubber-dubber, the 'blue' have too much volume, the 'black' are hopelessly small. Tight fit of the rubber-dubber is critical: it should hold the solution so that it does not drop out until you squeeze.

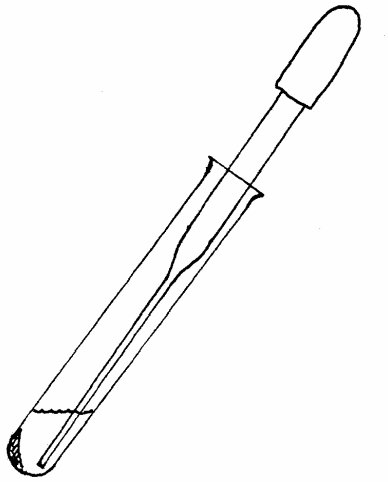


Figure 1

The main use of the pipet is to decant the supernatant off the precipitate. The word 'decant' really means to pour off, which by the way, is often possible. When the precipitate is not packed well enough by centrifugation, the supernatant can still easily be sucked away with the pipet. After centrifugation you will note that the precipitate pellet will be packed to one side at the bottom of the tube. First exhaust the pipet by squeezing the bulb, then place it on or near the bottom of the tube opposite the pellet with tube tilted as shown. Then slowly relax the squeeze. Usually this picks up the solution/supernatant/decantate leaving the precipitate. Practice this patiently a few times and you'll become a pro.

Centrifuging

Always balance the tube containing your sample to be centrifuged with another of the same size, shape, and thickness containing very nearly the same amount of water as you have sample solution, placed opposite your sample in the centrifuge. This will avoid the raucous and dangerous vibrations which accompany misbalance. If the centrifuge vibrates when run empty ask your instructor to fix it. Different sized rubber spacer plugs (not visible) are placed in the metal holder-tubes so that different sizes of test tubes may be used. The metal holder tubes should have been matched in weight with their opposite, but not necessarily with their adjacent tubes. Please don't rearrange these unadvisedly. So, your tubes, sample and counter weight, should fit into the metal holder tubes to an equal extent. Never let your glass tubes set against each other as they will surely break and fling glass during centrifugation.

How long one should centrifuge depends on the sample. Thirty seconds at high speed will pack most, but some may require two or more minutes. Clarity (no mention here of color) of the supernatant solution is the best measure of completion. Sometimes total clarity cannot be reached but don't give up without some extended centrifugation. As mentioned above, most precipitates pack so well that you could pour the supernatant/decantate off. In other cases, as you will learn, any kind of agitation breaks the pellet up.

To “wash” a precipitate requires **three** operations:

1) Add to the precipitate the wash solvent which may be water or a particular solution.
2) Always stir or shake to re-suspend the precipitate but heat only if specified;
3) Centrifuge; and
4) Decant the supernatant, which may be combined with some previous decantate or discarded. This leaves you the washed precipitate.

To “check for completeness of precipitation” you will need to clarify the supernatant by centrifugation but you need not decant. Then add a drop of the precipitating reagent gently and observe where it mixes with the solution you’re testing for cloudiness, which would indicate more reagent is needed. When no cloudiness occurs, precipitation is complete.

The “unknown tube”: For the qual scheme experiments (Experiments 2, 3, and 4), you will turn in a test tube to your instructor in which he/she will put your unknown. The best policy is to place your tube for the next unknown when you pick up an unknown. Each instructor has his own specification for these as to size, where to label, what to label, and where to place it. You **must** meet his/her specifications. Your instructor will simply add the “unknown” to the tube without any cleaning, so if you want a clean sample give a clean tube.

Lab Reports

The student is expected to prepare and submit a detailed lab report for each experiment done. The format for these reports will vary according to the individual instructor, and will be specified in the course syllabus or in pre-lab lectures. The medium for the lab reports (print and/or electronic) will also vary from one instructor to another. Some of the experiments contain directions (on data treatment and graphing procedures) that specify minimum report content. Your instructor will probably require a more extensive report. Typical lab reports might consist of the following sections:

A separate **Title Page**, to consist of Date, Experiment #, Experiment Name, Your Name, Names of People in Your Group, Name of Instructor, Lab Section Number.

1. Theory and Principles. This section should be reflective of the prelab lecture, supplemented by material from the text or other references. It should not be copied from the sources, but should be presented in the student's own words. This section should describe the chemistry being demonstrated by the experiment, thus should contain balanced chemical equations.

2. Experimentation. This section should outline the following.

> **2A. Procedures.** Describe procedures followed and describe any instruments used. This should be written in the past tense and should not be a detailed step-by-step recanting of steps performed. It should tell what general procedures were followed. Flow charts fit well in this section of the report. It may also include a listing of chemicals used, with specification of any safety hazards.
>
> **2B. Experimental Data**. This is usually presented in tabular form as prescribed in the lab manual.

3. Results

> **3A. Sample Calculations.** Applicable only if the experiment involves calculations. Most do.
>
> **3B. Results**. Results are often presented in the form of a graph, but may be numerical or tabular. Graphs may be plotted on graph paper or may be produced using graphing software like EXCEL. It is important that axes be clearly labeled and that scales be chosen to make the graph span the entire page as much as possible.
>
> **3C. Error Analysis.** Calculate % errors, average deviations, etc, if your instructor tells in advance what value should have been obtained. For a quantitative determination, your instructor may calculate the percent error and deduct points for poor results.
>
> **3D. Discussion of Results**. Discuss sources of error and assess the reliability of the experimental results.

Neatness, **spelling and grammar count.** Further discussion of expectations for lab reports will occur during the semester. Lab reports should be as brief as possible while adequately addressing

the stipulations listed above. For many of the experiments, specifics are provided on how to present the results for that given experiment.

The students are expected to hand in a lab report of each completed experiment at the beginning of the next week's prelab. Failure to meet this deadline will result in a grade of zero for that experiment. If for some legitimate reason you must miss lab, you should plan to submit the lab report early or have someone turn it in for you on or before the deadline. Thus it is unwise to wait till the last moment to prepare a lab report.

Lab reports must be typed, not hand written. If you do not routinely use a word processor, this is the chance to start doing so. Entries in data tables must either be typed or written neatly in ink, as dictated by your instructor. Whether graphs are plotted by hand or produced using graphing software such as that included in a spreadsheet like EXCEL, is decided by your instructor. Unless otherwise specified, each graph should occupy its own page. It is mandatory that axes chosen for graphs cause the plot to fill the page as much as possible. Plot smooth curves (including straight lines). Do not connect the dots.

Do not copy any part of the lab report directly from a book or from another individual's lab report. Lab reports should be written in your own words. Utilize the spelling and grammar checker provided by the word processor you are using. Your grade should be based on your work, not that of others. The instructor will be vigilant in looking for plagiarism. Conclusive evidence of plagiarism will result in a grade of zero for that experiment and may result in expulsion from the course.