

## Introduction to Chemistry 317

Welcome to Chemistry 317, Inorganic Chemistry Laboratory. Our goals are to make this a stimulating, challenging and useful experience. You will be introduced to new techniques and new kinds of chemicals and chemical reactivity. The class will tie together material you have had in lecture courses, and will ask you to design and improve experiments. There are some lab periods for which no instructions are provided; you must choose what you want to do and invent a procedure to do it. We will help you, but we want you to bring your insights, enthusiasms, questions, and skills to the course. Some of the material will be familiar, while other parts of the class will be quite new. Some of the experiments work like a charm, others we are still perfecting—and we hope you will help us make them better. We are eager for your suggestions and comments.

### Safety

In this laboratory—as in any laboratory—there are a number of hazards. Learning how to deal with hazardous situations safely is an important part of what you will learn in the class. If chemistry majors cannot handle hazardous situations involving chemicals, then who in the society can? It's usually chemistry majors who write the rules for safe handling of chemicals. Safety is an important focus of this class and we want you to think about safety as you read this lab manual and, especially, as you work in the lab. There will likely be a safety question on the exam. There was a scary incident during a Chem317 lab period six years ago but fortunately no one was hurt.

The most important safety rule is to **THINK!** Good common sense will get you through most situations. If there is anything that is unfamiliar or doesn't seem right, stop what you are doing and ask. There is a TA for every eight students in the lab, so there should always be someone nearby to assist or explain. You will be using a number of expensive pieces of equipment during the lab, so it is important that you understand how this equipment works. Don't just plow ahead if anything looks wrong. No one will be criticized for asking. It is, however, critical that you arrive prepared for the laboratory, having worked out the procedures in your own mind so you know what you're going to do.

There are a few safety rules we will strictly enforce. Safety goggles must be worn at all times in the lab. Eating in the lab is of course forbidden. Shoes must be worn at all times (no sandals or open toed shoes), and no shorts or short skirts.

An increasingly important part of safety and safe handling of chemicals is their disposal. The disposal of the solutions and products in each lab experiment is either described in the lab write-up or your TA will explain the procedures. When in doubt, put your waste in a bottle and label it to indicate the contents. No potentially hazardous waste should be disposed of down the drain or allowed to evaporate into the fume hood. [Environmental Health and Safety even views Coca-Cola™ as “potentially hazardous waste”!]

## Logistics and Schedule

Chemistry 317 consists of two laboratory periods and one “lecture” hour per week. The experiments are designed for students to work in pairs, with a maximum of 16 students (8 pairs) in the laboratory. In each lab period, half of the students will work on one experiment and the other half will work on another. Those in the AA and AB sections will be doing experiments from the first column below, and must come to the Tuesday 8:30 a.m. discussion hour. Those in BA or BB will work on the second column and must come Tuesday at 9:30 a.m. In this way the discussions will be related to the experiments you are doing.

The discussion hours will include some lecturing, to provide background and understanding of the experiments. But primarily these hours will be forums for discussion of the lab just completed—for instance how to analyze your spectra or numerical data. Please bring your data, your questions, and your opinions! Most students have found these sessions quite helpful. Attendance will be taken.

The schedule for the class is given below; the write-ups for each of the eight experiments make up the body of this lab manual.

Lab period #	Sections AA, AB, AC	Sections BA, BB	date
1	Check in	Check in	Jan 2/3
2	Chromous Acetate	Chelate Effect	Jan 4/5
3	"	"	Jan 9/10
4	Chelate Effect	Chromous Acetate	Jan 11/12
5	"	"	Jan 16/17
6	Phosphorous Acid	ZnS phosphor	Jan 18/19
7	"	(Arene)Mo(CO) <sub>3</sub>	Jan 23/24
8	BF <sub>3</sub> •NH <sub>3</sub>	Chelate Effect II	Jan 25/26
9	(Arene)Mo(CO) <sub>3</sub>	Linkage Isomers	Jan 30/31
10	Chelate Effect II	"	Feb 1/2
11	Linkage Isomers	"	Feb 6/7
12	"	"	Feb 8/9
13	"	(Arene)Mo(CO) <sub>3</sub> II	Feb 13/14
14	"	"	Feb 15/16
15	(Arene)Mo(CO) <sub>3</sub> II	"	Feb 20/21
16	"	Linkage Isomers II	Feb 22/23
17	"	Phosphorous Acid	Feb 27/28
18	Linkage Isomers II	"	Mar 1/2
19	ZnS phosphor	BF <sub>3</sub> •NH <sub>3</sub>	Mar 6/7
20	Check out	Check out	Mar 8/9

Even with only four pairs of students working on a given experiment, there will occasionally be times when you will have to wait to use a piece of equipment. Try to find something else that needs to be done while you're waiting. Making efficient use of your time is a critical laboratory skill (and a skill you will be graded on). Within each experiment, you and your lab partner will often be doing different things. You should try to follow what she or he is doing, as the final lab write-up will require both of your data. But—don't worry—no one will be penalized because their lab partner didn't finish or something like that.

## Readings

It is imperative that you carefully read the lab descriptions before entering the lab. Even more than reading, you must think through what you will be doing. This is critical for safe working in the lab, and to manage your time efficiently. The TAs may take various steps to insure that the reading is carefully done.

The lab descriptions contain occasional references to the "original literature," the scientific articles which originally reported the chemistry. These are given in the standard American Chemical Society (ACS) reference format: Journal Title year, volume #, page. These and other articles are available in a folder in the 317 lab, along with various reference books. The folder and books are also on reserve at Odegaard library. These extra readings are optional, but may be quite useful and interesting. In the arene-molybdenum-tricarbonyl experiment, for instance, many students have found these papers useful when they design their own procedures. The extra readings provide background to help you understand your observations and better interpret your data—both critical to good lab reports.

## Grading/Assignments/Notebook

You must keep a good notebook in this laboratory (and in all scientific labs). Use a bound book that pages cannot be removed from. Your notebook is your diary of what you did, and it should be written as you are working. Do not make notes on scratch paper and transcribe them into your notebook. The book should include numerical data (weights, volumes, voltages, etc.), procedures (A was added to B dropwise over 20 minutes using an addition funnel), and observations (it turned green after half the A was added). The most important features of a good lab notebook are clarity and completeness. You should never remove a page or plan to go back and fill in something later. If necessary, you can cross something out or recopy something for clarity, just indicate why and make sure the original is still legible. The notebook is not a handy piece of scratch paper. It should enable you to reconstruct what you did, including good and bad aspects of the procedures. A TA will look at your lab book periodically and may collect it at some point.

Each lab write-up in this manual ends with a description of the required assignment for the lab. All assignments are due one week after completion of the experiment. Assignments must be typed, double spaced, except for tables, figures, drawings, graphs, and equations which can be done by hand.

Three of the experiments require formal lab reports, as explained in their write-ups. The most critical aspects of any lab report are clear thinking and maintaining your focus on the important issues. Grading will be based not only on the science but also on clarity and writing skills. It's hard to judge the science if the writing is poor. This is a "W class," so you will earn writing credit (with an emphasis on earn). An introduction to writing lab reports is given on the next page.

In addition to the lab write-ups, there will be a brief (50 min) exam. This is scheduled for Thursday, March 8, 2001 at 8:30 or 9:30 a.m. (your choice). This is not a scheduled course meeting time, so please reserve that date and time on your schedule now. If you have a schedule conflict, contact Professor Mayer as soon as possible.

Your grade will be based on the formal lab reports for three of the experiments, the shorter assignments for the other labs, your lab notebook, the exam, and on your overall ability in the laboratory (as judged by your TAs). The point distribution is outlined below. The score for lab skills given by the TAs is quite important. They will be looking at how prepared and punctual you are, how well you use your lab time, your lab safety, lab awareness, and overall helpfulness (especially to your lab partner), and the quality of your ideas, suggestions, and questions.

<b>Lab Reports</b>	
Chelate Effect	16%
Arene Molybdenum Tricarbonyl	16%
Linkage Isomers	22%
<b>Other Experiments</b>	
Chromous Acetate	4%
Phosphorous Acid	4%
BF <sub>3</sub> •NH <sub>3</sub>	4%
ZnS phosphor	3%
<b>Lab Skills (TA input)</b>	16%
<b>Exam</b>	15%
Thursday, March 8, 2001 at 8:30 or 9:30 a.m.	
New Chemistry Building (CHB) 102.	

## Guidelines for Writing Lab Reports

### 1. Outline.

#### Introduction:

The introduction of a lab report typically starts with an explanation of what you are trying to accomplish or observe in the experiment. Include a brief discussion of why this might be interesting. You should introduce the compounds to be studied and/or the techniques to be used and their interpretation. In the Linkage Isomers lab for instance, you might discuss the long history of the cobalt complexes (don't just copy the lab manual!) or the concept of linkage isomerism with reference to the nitro ligand. It would be appropriate to discuss electrochemistry in the Chelate Effect lab, and how  $^1\text{H}$  NMR and IR spectroscopies can help characterize compounds you might make in the "Arene-Molybdenum" lab. In general, the Introduction should set out the questions and issues that will be the core of your Discussion section.

#### Results (Experimental)

This section should tell a story, describing what you did and what results you obtained. It should describe your actual procedure, not the one in the manual. It should include the amounts of reagents used and your yields of the products obtained (both in grams and percent). Procedures for physical measurements should also be included, although not standard procedures (assume that we all know how to use a balance, a glove box, a Schlenk line, and an IR spectrometer, for instance). Balanced chemical equations are strongly encouraged. Diagrams of the proposed and actual (if different) structures of the complexes should be shown with labels on the atoms or groups of interest. These labels can then be used in the data table for assignment purposes and they can be referred to in the text.

Numerical data should whenever possible be presented in Tables rather than as part of the text. Then the text can refer to the Table, as in "Voltage readings were obtained over the temperature range 280 - 340 K and the data are given in Table 1." Any problems with data collection should be explained. Whenever possible include the raw data, and show how the quantities of interest were derived, giving any relevant equations. [Grammatical note: The word data is a plural noun.]

#### Discussion

Separate Results and Discussion sections are encouraged but not required. The Discussion section should first of all provide your analysis of the results. Did you make what you wanted? How do you know? If the expected product was not formed, discuss why this might be the case and whether the actual product can be identified. Discuss what went wrong, if anything did. In the Arene-Molybdenum lab, you'll want to analyze your spectral data, for example what the CO stretching frequencies indicate about what happened. Compare the spectral data for the products to those of the starting materials and make comparisons. The Chelate

Effect and Linkage Isomers labs require significant data and error analyses which can go into the Results (if you think they're straightforward) or the Discussion if you want to discuss them.

The Discussion section must include a clear description of your conclusions. You could conclude that the chelate effect is primarily entropic or that the reactions you tried just gave you back starting materials. You could also conclude that the error bars are too big for you to conclude anything or that the procedure didn't work because .... But it's important that you take a stand—no waffling. The Discussion should return to the questions, goals, and issues raised in your Introduction. In a way, the Introduction and Discussion are the bookends for the Results.

## References

Cite any references you used to do this lab or in your discussion (other than what was presented in the lab manual).

## 2. Presentation and Style.

The ability to write a clear and concise description of what you did and what it means is a very important skill, regardless of what job you find yourself in. This is a formal lab report, a single document with a logical lay-out and flow. The arguments must flow within each paragraph and from one paragraph to the next. Proper grammar and spelling must be used.

Scientific writing has its own peculiar styles. Descriptions of actual experiments are typically written in the past passive voice. For instance: "The 0.1 M ammonia solution was added to the solution of  $\text{CuSO}_4$  dropwise, with stirring. The solution rapidly turned dark purple...." You should try not to imply that chemicals are active agents, avoiding, for example: "the ammonia turned the copper ions purple." Use the past tense when you are describing what you did at some time in the past. In other places, try to use present tense: "Understanding the chelate effect is important because ..." or "A plot of the  $\Delta G$  values free energies versus temperature is shown in Figure Z.  $\Delta S$  is obtained from the slope of this plot .... The  $\Delta S$  values imply that ..." Use present tense because what you are describing is independent of time. You imply that anyone looking at your data would derive the same  $\Delta S$  and reach the same conclusion.

You should try to avoid using personal pronouns, such as "I added ammonia to ...." The statement "Addition of ammonia to a solution of  $\text{Cu}^{2+}$  causes a rapid change in color to dark purple" is true whether you do it or someone else does it; it is true today and will be true in the future. Use a personal pronoun to indicate that something occurred that is your opinion or that was specific to you ("At this point, my lab partner unfortunately dropped all the product on the floor."). By writing "I believe that the major source of error is ...." you imply that someone else looking at the same data might well reach a different conclusion. You can also indicate that you're not sure of something with phrases such as "it seems likely that" or "it is possible that" or "perhaps."

These are guidelines, not firm rules. “The voltages are/were converted to free energies (in kJ/mol) using equation X (see Table Y).” could be present or past tense. The most important features are clarity of thought and writing. Maintain your focus on the important issues and lead the reader through your story and your arguments. Ask yourself “what am I really trying to say here?”

## Techniques

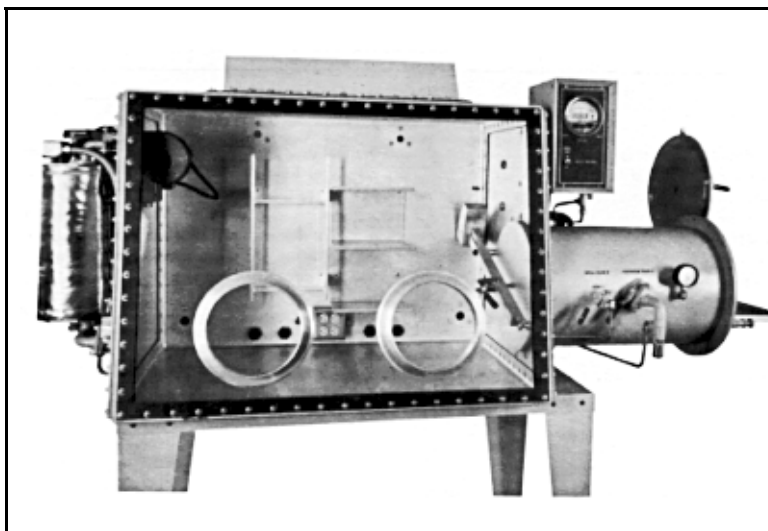
Many inorganic compounds are sensitive to oxygen, moisture, or both. Such compounds—you will make several of these during this course—need to be protected from the ambient atmosphere. You will become acquainted with a number of techniques to protect materials from the atmosphere. In some ways, learning these techniques is a focal point of this class. By far the best reference to the handling of air-sensitive compounds is the book *The Manipulation of Air-Sensitive Compounds* (2nd Ed.) by D. F. Shriver and M. A. Drezdson, Wiley-Interscience, New York (earlier editions with the same title are pretty similar).

The two primary ways you will handle air-sensitive materials in this class are with a glove box or with a Schlenk line, as described below. The handling of gases is described in part III.

### I. The glove box.

Conceptually, the simplest way to keep things away from the oxygen and water in the atmosphere is to work in a lab space where there is no oxygen or water. This is a little difficult, however, since we humans need both to survive. But one could imagine a fully enclosed “bench top,” containing an “inert atmosphere,” which one could reach into with gloves. Such a device is called a “glove box” or a “dry box” if it has hard sides like a box. There are also cheap “glove bags” that are what they sound like, bags you can fill with inert gas and reach into with attached gloves.

The glove box that you will use in this class is a fairly sophisticated one, made by the Vacuum Atmospheres Corp. A picture is shown at right, with the gloves missing (they go on the big circles in the middle). Despite the name, this box does not have a vacuum in it (the sides and gloves would implode with the pressure). Rather the glove box is kept full of clean nitrogen gas. Clean in this context means chemically clean, containing as little oxygen and water as possible.



The dry box has four important parts: (i) There is a large aluminum box, with a plastic front window sprouting two gloves. This is the working area. Note that this is not a glass front (too fragile), so use only water if you need to clean it—no organic solvents. (ii) There is an antichamber (like a submarine or spaceship airlock) which is how things get in and out without letting in air. This is the cylinder at right in the drawing above. (iii) The gas in the box is constantly circulated over a scrubber (often called the “catalyst”) which removes any air or water that has made its way into the enclosure. Since the catalyst is damaged by many kinds of reactive chemicals (chlorinated solvents, sulfur compounds, etc.), we must be careful what we allow to evaporate into the box atmosphere. In the picture above, the catalyst is located in a canister outside the left wall of the box. A fan inside the box circulates the box atmosphere through the canister.

(iv) Finally, the box must be able to regulate the pressure inside. If the pressure gets too high the gloves will pop out or the front will break—and if the pressure gets too low the gloves will suck in. Both are catastrophes. The device that regulates the pressure is called the photohelic and it is located above the antichamber. You can recognize it because it has a pressure gauge on it, calibrated in the unusual unit “inches of water” (more on pressure units later). The box can take only a few inches of water positive or negative pressure, very little change from one atmosphere. The photohelic automatically draws fresh nitrogen from a tank if the pressure gets too low and automatically pumps nitrogen out if the pressure gets too high. You can also do these things manually with the foot pedal, labeled R and L, for raise and lower [pressure]. The instructor or a TA will demonstrate the operation of the box and the photohelic in the lab. If the nitrogen cylinder that feeds the box runs out, tell a TA right away.

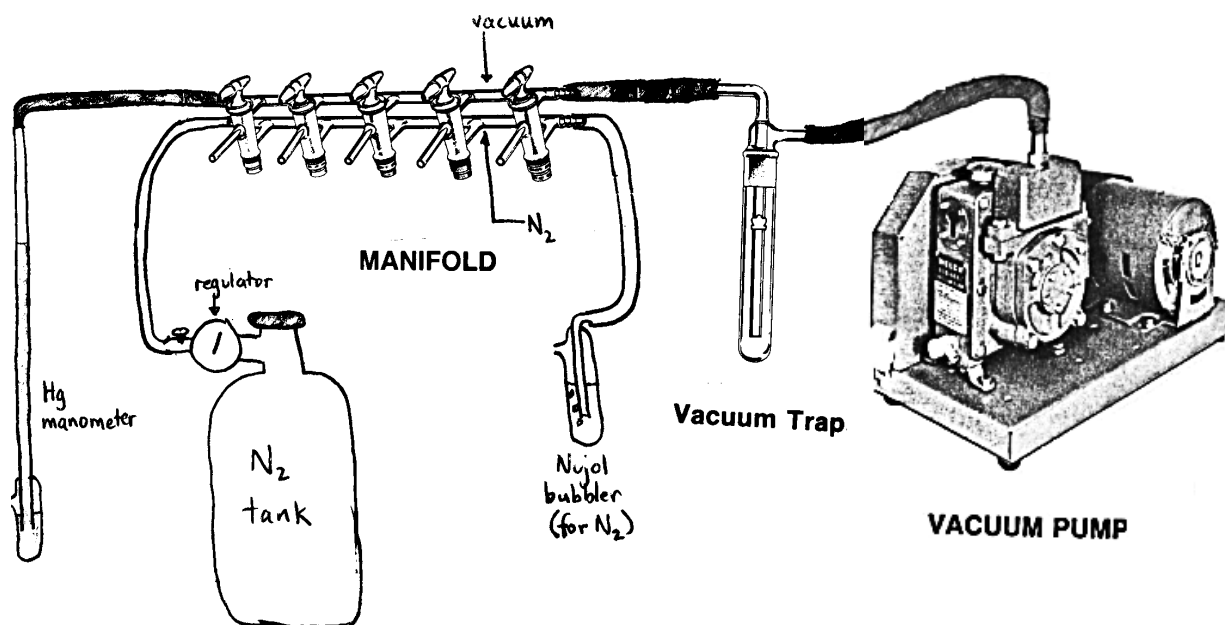
The drybox is an extremely useful piece of equipment but one that must be treated carefully—one accident and you can contaminate the atmosphere and destroy everyone’s chemicals. Some researchers in inorganic chemistry use a glove box as if it were a bench top, and do all their work in there. However, this can be tedious, uncomfortable, and inconvenient. It is difficult to maintain a good atmosphere in the box when you’re working with all sorts of different solvents and reagents, it is hard to work with gases, and it is hard to do reactions that require heating or cooling (remember, no water for reflux condensers). These boxes are also expensive, costing well upwards of \$10,000 each.

For these reasons, we only have one dry box in the lab, and it is used mostly for storage, setup, and workup of experiments. You will do most of your chemistry out in the air, using specialized glassware. In the aggregate, these strategies are called “Schlenk techniques.”

## II. Schlenk Techniques

The centerpiece of this defense against atmospheric intrusion is the double manifold, or Schlenk line. In the illustration below, the double manifold is in the

middle, connected to various equipment on both sides. The two long, horizontal tubes of the Schlenk line are called “manifolds.” Each manifold can be filled with a gas, or “un-filled” with vacuum. Reactions and manipulations are typically done under an atmosphere of an inert gas, usually nitrogen, admitted into one of the manifolds (the “nitrogen side”). The other manifold (the “vacuum side”) is connected to a vacuum



pump through a liquid-nitrogen cooled trap. Any solvents that make it into the vacuum line condense in the trap before they get to the pump, which protects the pump and the pump oil. Glassware (see below) is connected to the Schlenk line via rubber hoses. Then the glassware is exposed to the vacuum or the nitrogen manifold using the two-way stopcocks.

Be sure all stopcocks are lightly greased—if you forget this the glass pieces may stick together and it can be a real pain getting them apart. The function of the thin film of grease is just to allow the glass pieces to slip over each other. If you use too little grease the glass pieces will bind. If you use too much grease, it will ooze into the holes and into your line and will degrade more rapidly. When you're done, the ground-glass part of the stopcock should look clear, with no streaks in the grease. Your TA will help you get the hang of this.

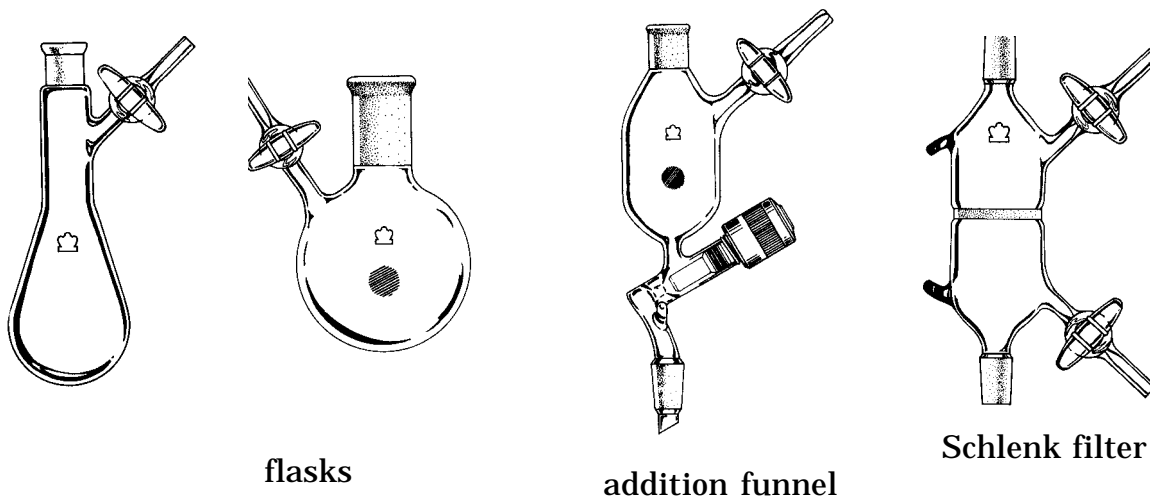
The mercury manometer attached to the vacuum manifold is your way of seeing what the pressure is in the manifold. The pressure (in mm Hg) is the distance the mercury inside the tube has been pulled up from the mercury pool at the bottom. One atmosphere is 760 mm Hg (760 torr), or roughly 2.5 feet high of mercury. The manometer is useful for any kind of quantitative gas handling, as gases are measured out by  $PV = nRT$ . But it is also critically useful to tell you qualitatively what is going on inside your Schlenk line. Whenever you do

anything on the Schlenk line, the pressure inside will change. Be sure you know what you expect to happen whenever you open or close a valve on your line—and watch the manometer to be sure that this is what happens. If anything surprising occurs, STOP, close the valve, and think again.

The nujol bubbler at the end of the nitrogen line is there to prevent air from getting in. It behaves very differently from the mercury manometer, because nujol is much less dense than mercury. One atmosphere would be a column of nujol 40 feet tall! You clearly don't have a column this tall, so you cannot pull a vacuum on your bubbler. If you do pull vacuum on it, you'll suck nujol back into your line and make a big mess. Many groups do this at least once and then have to clean their line (not fun). If you need to pull a vacuum on the nitrogen line, use a pinch clamp on the hose or squeeze it with your fingers to prevent the nujol from sucking back. When you're using the line, there should be a slow and steady flow of nitrogen through the nitrogen manifold and out the bubbler. The stream of nitrogen bubbles tells you what's going on in this manifold.

You have an array of equipment to help you manipulate solutions without exposing them to air. The lead players are pieces of glassware with sidearms attached, so-called Schlenk ware, as illustrated below.

The Schlenk flask is an ordinary round-bottom flask with a sidearm with a stopcock (be sure it's greased!). You can connect this sidearm to the Schlenk line with thick rubber tubing and use it to admit nitrogen to the flask or to evacuate it. The tubing needs to be thick so that it won't collapse under vacuum. You will put something in the neck of the flask, such as a glass stopper (greased) or another piece of apparatus such as a Schlenk addition funnel or a Schlenk filter.



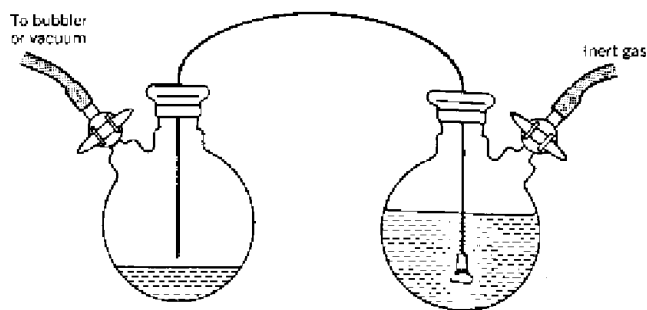
The addition funnel is a convenient gizmo for adding solutions to a Schlenk flask. With the lower stopcock closed it is completely sealed off from the flask below it, so you can put solutions in the funnel without contaminating the

atmosphere in the bottom flask. To move a solution from the funnel to the flask, first connect both to the nitrogen manifold via the sidearm stopcocks. Then just open the valve at the bottom of the funnel. Connecting everything to nitrogen is critical because this equalizes the pressure between the top and bottom; without it, pressure would build up in the lower flask as the liquid flows down, and the flow would stop. In all air sensitive work, you always have to worry about pressures—what the pressure is in each piece of apparatus and how this will affect what you're trying to do.

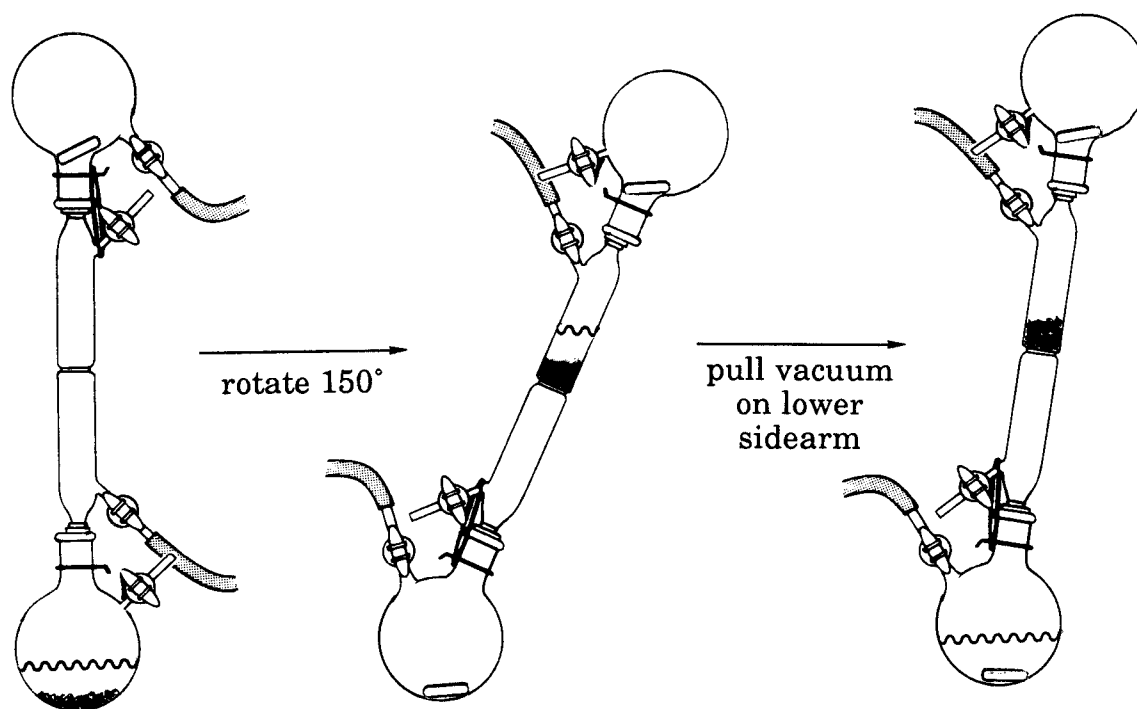
Another common way to cap a Schlenk flask is with a septum, an air-tight rubber membrane. By piercing the septum with a variety of sharp instruments, you can add or remove liquids from flasks without exposing them to the air. And septa (plural of septum) seal back up (pretty well) when you remove a needle. Since septa will not usually give you a truly air-tight seal, they should be swapped for a glass stopper if you need a flask to stay air-free for more than an hour or so.

A syringe, which you're probably familiar with, is one way of transferring liquids into or out of a septum-capped container. If the container is under a pressure of nitrogen, you can simply insert the needle into it, pull back the plunger to remove the desired amount of liquid, then withdraw the needle and inject the liquid into another flask. In the time it takes to move from one flask to another not much air can diffuse into the small bore of the needle. There are two important hints about using syringes. First, the "dead space" in the needle and syringe body will ordinarily have air in it. If this would be a problem, you should flush it out before using it. Suck nitrogen into the barrel from a nitrogen-filled container (usually a flask attached to the nitrogen line), then expel the nitrogen out into the air. Repeat this two or three times to purge air from the syringe. Second, remember to always think about pressures when transferring via syringe. If you try to suck a liquid out of a sealed container, you won't get very far, since you'll build up a partial vacuum in the container. You can avoid this by making sure the flask from which you are withdrawing is connected to a source of nitrogen, like your nitrogen manifold, or by injecting a volume of nitrogen gas to compensate for the volume of liquid that you are going to remove. Similar tricks can alleviate difficulties in injecting into closed flasks.

A cannula is a hollow steel needle with two sharp ends. It can serve as a sort of express route for transferring liquids when set up as shown. If the pressure in the flask at right is greater than that in the other flask, the liquid will be pushed from the right to the left flask. This pressure difference can be achieved by placing one flask under nitrogen and partially evacuating the other.



To address the difficult conundrum of no-air filtrations some twisted soul invented the Schlenk filter (illustrated below). Its effective use requires some practice and a flair for contortionism. The filter is placed on top of the flask with the material to be filtered, and on top of it is placed a flask in which to catch the filtrate. The whole assemblage is then inverted, and you try to get as much of the solid as possible to run down on to the fritted glass disk. You can help the solid down with the stir bar, which you can move around with a hand-held magnet on the outside of the flask. Applying a touch of vacuum to the underside of the frit while the top is under



nitrogen will suck the filtrate through just like an ordinary suction filtration. It's considered tacky to pour the solution down one of the sidearms, so make sure you tip the setup so the liquid runs down the other side (as shown). We encourage using more than two hands for this operation—i.e., do it with your partner.

### III. Gas Handling: Tanks and Regulators

The handling of gases provides a number of challenges. First, all gases are “air sensitive,” to the extent that if they get mixed with air they are no longer pure. (There are also gases that undergo chemical change because of reaction with oxygen or water.) You will use the vacuum manifold of your Schlenk line to work with gases.

Gases are purchased in thick (usually steel) tubes, either large cylinders or smaller “lecture bottles.” The gas inside is present under pressure, typically pressures much greater than one atmosphere. This represents the second challenge: a pressurized gas would love to get out of its container and will do so with some force if allowed to. This is a serious hazard, and one that must always be kept in mind while working with compressed gases. Seven years ago, there was a serious incident when someone opened a lecture bottle to their Schlenk line when the stopcock on the line was closed. The high pressure gas was released to the rubber hose but had no place to go. So what did it do? The gas blew open the thick hose, with sufficient force to snap a piece of glass off the line. A fair amount of noxious gas was sprayed into the lab and everyone had to be evacuated. Let’s not have a problem this quarter.

We use a series of valves and regulators to make sure that the flow of gases is controlled (by us). The cylinder or lecture bottle that comes from the vendor has a valve on top, essentially an on/off valve (there is a little bit of flow adjustment possible with this valve, but very little). Lecture bottles are metal tubes, roughly 2” in diameter by 15” in length. Gas cylinders come in all shapes and sizes, up to 5’ high and 300 pounds—and from there, gases can be purchased in truck-loads or by the railroad tank car. The primary difference between a cylinder and a lecture bottle (aside from the size) is that we the consumer buy the lecture bottle while the cylinder is the property of the company and we pay “rent,” a demurrage of about 3¢ per day.

The valve on the tank is your first line of defense. If this is closed, no gas can get out. But when that is open, the gas will come out at a high rate (flow): our nitrogen tanks come pressurized to 2,000 psi (pounds per square inch) or ca. 150 atmospheres. If anything goes wrong while you’re working with a gas, close off this main valve. If the student had done this seven years ago when the hose blew, the incident would have been much less serious.

Note: you will be expected to know—perhaps on the exam—  
all the various pressure units used in this class:  
 $1 \text{ atm} = 1.01 \text{ bar} = 14.7 \text{ psi} = 760 \text{ mm Hg} = \sim 33 \text{ ft or } \sim 400 \text{ in of water}$

In this lab, you will use large cylinders of nitrogen as your source of inert gas. To control the flow of gas from a large tank, always use a regulator (such as the one pictured at right). One other precaution: these tanks should always be chained to a wall or a lab bench so that they cannot tip over. If one tipped over and fell on the regulator, it could snap off the valve on top of the tank. This would turn your demure gas cylinder into a 300 pound steel torpedo, propelled by the high pressure gas inside.



The regulators we use for large cylinders typically have two valves and two gauges—and it is important that you know the function of all four things. The gauge closest to the cylinder tells you the pressure in the cylinder; it will be calibrated from 0-3,000 psi or thereabouts (it will also be calibrated in other units, too, just to be confusing). The valve that is in between the two gauges is a diaphragm valve, which regulates pressure (not flow). This amazing device enables you to set the pressure coming out of the regulator, which you can read on the second gauge, the one farther way from the tank. Typically we use low pressures, 0-5 psi (above atmospheric). Finally, the small valve leading out of the regulator is a needle valve that regulates flow, not pressure. This valve is your last line of defense—open this valve slowly to bleed gas into the line, then if you need more gas open it wider. The needle valve works as you would expect: screwing it down will close the valve. The diaphragm valve, however, is opposite: it is closed when screwed all the way out, and most open as you screw it in. Be sure that you understand this difference.

Lecture bottles are typically used for gaseous reagents, such as the  $\text{BF}_3$  and  $\text{NH}_3$  that you'll use in Experiment 4. Lecture bottles contain much less gas and typically have lower pressures than large cylinders. We typically use just a needle valve (shown at right) on a lecture bottle to regulate the flow of gas out. Then you cannot regulate the pressure coming out—only the flow. The procedure you should use is to open the needle valve for an instant to let some gas out, then quickly close it. This enables you to dispense just the amount you need into your Schlenk line.



Most importantly, be sure to look at the manometer when you are dispensing gas to be sure the gas is going where you think it is. When you open the needle valve for an instant and then close it, the mercury level in the manometer should drop rapidly and then stop. If it doesn't do this, shut your gas off, think about where the gas that you just let out of the lecture bottle went, and get your TA.

Whenever you first use a lecture bottle, you should assume that the hose and needle valve are full of air, unless you know otherwise from the previous user. To get rid of the air, connect the hose to your Schlenk line, make sure the main valve is closed, open the needle valve, and pump out the hose and the needle valve. The main valve on a lecture bottle can look like a knob, which is closed when screwed down. Or the top of a lecture bottle can look like two nuts, which is closed when the nuts are tightened together. Your TA will show how to use two wrenches to do this. To remove air from a regulator on an inert gas such as nitrogen, purge the regulator and hose by running a good flow of gas through them. This avoids having to pump on the diaphragm valve, which is not good for it.

You must be very careful not to let pressure build up in any piece of apparatus. Schlenk techniques can tolerate pressures only slightly greater one atmosphere. If you have a pressure of two atmospheres in a flask (external pressure plus one atm more) that's 14.7 pounds on every square inch of your apparatus. So a stopper with a one square inch opening will have 14.7 pounds pushing it open. This is equivalent to hanging a bowling ball off of it! Be sure—whenever you work with gases—that you know what will happen anytime you open a valve, where the gas is supposed to be going, and where the gas will go if the pressure by accident gets too high. Let's have a safe and fun lab!