

## Core I Background Material: Introduction to Vacuum

### REFERENCES

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2. Dushman, Saul; Lafferty, J.M. *Scientific Foundations of Vacuum Technique*, Second Ed., Wiley: New York, 1962. A classic bible of vacuum theory and practice, including the properties of commonly used gases and surface materials. This edition is somewhat dated regarding modern vacuum equipment.
3. Moore, John H.; Davis, Christopher C.; Coplan, Michael A. *Building Scientific Apparatus*, Second Ed., Addison-Wesley: New York, 1989; Ch. 3. An excellent overall review for understanding the operation and for constructing/repairing equipment. Includes chapters on mechanics and electronics, optics, glass, vacuum and temperature.
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Chemists use vacuum techniques particularly when studying reactions between gas molecules and surfaces, when trying to study individual molecules during the period between collisions with neighbors, or when watching reactions taking place in single collisions. Thus in the fields of surface chemistry and catalysis, materials science, spectroscopy and molecular reaction dynamics, chemists reduce the pressure of any gases present. Besides knowing  $PV = nRT$ , working with vacuum requires some understanding of how gases move through tubing, how to round up gas molecules when they no longer follow one another down the tube, how to measure reduced pressures and how to keep the atmosphere outside the container (no leaks).

What follows here is an outline of some of the rudiments of vacuum principles and technique. When you have the opportunity for extensive vacuum work, you will need to study a more detailed description.

### **Hazards**

The principal hazards associated with vacuum work are injury from implosion and explosion. Vacuum equipment is subject to excess pressure of the atmosphere pushing inward (almost 12 psi in Durango). Fractures in the glass walls of a vacuum container can lead to dramatic implosion producing flying glass shrapnel. Eye wear meeting the ANSI Z87.1 impact standard (e.g., chemical splash goggles) must be worn when

working with a glass vacuum system. Metal systems do not produce shrapnel when they rupture.

In a glass system, be extremely careful not to produce pressures substantially above the external atmospheric pressure (600 torr in Durango). A glass system will explode.

Large surfaces of a glass system should be covered with vinyl tape or another elastic protective coating to minimize flying shrapnel. Too, surfaces should be rounded rather than flat. Do not produce a vacuum in any container not designed to be used in vacuum applications.

Metal vacuum systems provide greater danger for electrical shock from the electronic equipment nearby.

Liquid nitrogen used for cooling can burn the skin and eyes. Use splash goggles for transferring liquid nitrogen.

### Types of Vacuum

Table 1 lists common ways of describing vacuum and approximate values for some of the key gas properties.

**Table 1.** Common vacuum ranges and assorted gas properties.

	<u>laboratory</u>	<u>low</u>	<u>high</u>	<u>ultrahigh</u>
Pressure range (torr or mm Hg)	760-10	10-.001	$10^{-3}$ - $10^{-7}$	$10^{-7}$ - $10^{-11}$
mean free path (meters)	.00001	.00001-.1	.1-1000	> 1000
particle density (particles/m <sup>3</sup> )	$10^{24}$	$10^{24}$ - $10^{19}$	$10^{16}$ - $10^{12}$	< $10^{12}$
collision time (s/collision)	$10^{-6}$	$10^{-6}$ - $10^{-2}$	$10^{-2}$ -100	>100
pump type	aspirator	mechanical	diffusion, ion, sputter, turbo	
gauge type (partial list)	manometer, Wallace-Tiernan, Hastings	thermocouple, Pirani	ionization mass spectrometer	ionization,

For example, if an experiment required that a molecule be in undisturbed flight for .01 seconds, a vacuum chamber capable of working at a pressure of about .001 torr would be required.

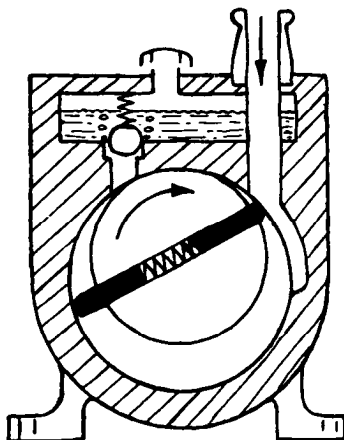
### **Fluid Flow versus Molecular Flow**

Under conditions where molecules collide with one another more frequently than with the walls, molecules tend to diffuse in the direction of minimum density and the gas flows as a fluid. On the other hand, at sufficiently low pressures, the molecules collide with the wall more frequently than with one another. Under this condition, the molecules behave as individuals, and the gas no longer behaves as a fluid. We call this low-pressure condition molecular flow. The gas can no longer be sucked out of the chamber by a pressure differential. Rather, the molecules must wander to the exit port by ricocheting off the wall, then be trapped at the exit and prevented from ricocheting back into the chamber.

For fluid flow, the rate of flow is primarily determined by the pressure difference between the chamber and the pumping system. For molecular flow, the primary factor is the cross-section of the flow path. Thus, for pressures below .01 torr, the diameters of the various tubes and valves (relative to their lengths) become the dominant factors. The larger the diameter and the shorter the length of a section of the pumping system, the higher the flow rate, often called the pumping speed.

### **Pumps**

The common-sense vacuum pump capable of sucking gas out of a vacuum chamber and expelling it from an exhaust port is operating by fluid mechanics. It is effective down to a pressure of about .01 to .001 torr, where molecular flow becomes the dominant behavior. The rotary mechanical pump is the workhorse for fluid pumping. (Note -- fluid does not mean liquid.) A schematic of the most popular rotary pump, the Welch Duo-Seal, is shown in Figure 1. For a mechanical pump to achieve its optimum vacuum requires that the oil in the pump be free of solid residue (gunk) and maintained at the proper level.



**Figure 1.** A schematic of the Welch Duo-Seal rotary mechanical vacuum pump. This pump, using the movable two-vane design, can produce pressures as low as .001 torr. (Taken from Ref. 3.)

For pressures below .001 torr, the diffusion pump is the tradition. This pump provides a column of refluxing oil whose droplets are projected downward as shown in Fig. 2. These downward oil streams collide with gas molecules and push them toward an exit port, where they have been compressed enough to regain fluid flow and are pumped away by a mechanical pump, which is designated the "forepump." The refluxing of course requires both a heater and either air or water cooling. The metal diffusion pump depicted in Figure 2 is the most common type used today, although a few applications call for a glass diffusion pump using oil or mercury for the reflux liquid.

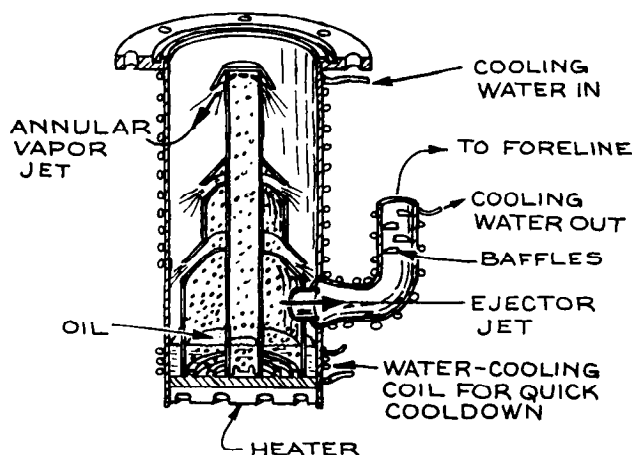


Figure 2. A metal oil diffusion pump. (Taken from Ref. 3.)

Diffusion pumps require special oils which have low vapor pressure at elevated temperature. The cheapest type of oil is hydrocarbon ester, e.g., a common brand named Octoil is a blend of such esters. Other types include silicone (hydrocarbon trisiloxanes) and polyphenyl ethers which offer enhanced lower pressure limits (down to  $10^{-10}$  torr). Any hydrocarbon oil is especially susceptible to oxidation when hot, so an operating

diffusion pump must be carefully protected from pumping air. A hot diffusion pump must not be exposed to air pressures above .005 torr.

Both diffusion pumps and mechanical pumps produce a small, but significant amount of oil "back streaming", in which oil molecules are traveling from the pump upstream toward the vacuum system. Thus it is usually necessary to have a cold trap between the system and the pumps to trap the oil. Such trapping is described below.

At FLC, we use oil metal diffusion pumps and Duo-Seal mechanical pumps.

There are other types of vacuum pumps in use. The turbomolecular pump mechanically produces pressures as low as  $10^{-8}$  torr using a high-speed rotor. The department's GC-MS uses a turbo pump to maintain the mass selector vacuum. Ion and sputter pumps are used to produce ultrahigh vacuum at pressures below  $10^{-7}$  torr. Cryopumps cool molecular sieves (e.g., zeolite) with liquid nitrogen to produce vigorous pumping by adsorption. A cryopump produces no backstream of pump oil and can operate over the range  $760 - 10^{-7}$  torr using liquid nitrogen, but requires periodic cycling to remove the trapped gases.

### **Metal and Glass Vacuum Systems**

Vacuum systems are almost always constructed out of stainless steel or glass. Stainless steel systems are rugged and a wide variety of pre-fab components are available through catalogs. The principal disadvantages are cost and the difficulty of modifying the components in the lab. A metal system is usually more suitable for high temperature applications. The time-of-flight mass spectrometer in 472 BH is an example of a stainless steel system.

Glass vacuum systems are more fragile and often cheaper, but require some degree of fabrication. While the major components (e.g., valves, traps) are factory-made, they must be fused together using a glass-working torch. Once fabricated, repairs and minor modifications can often be done right in the lab.

Generally, vacuum experiments requiring large volumes or rugged equipment are done with metal systems. When the application calls for surface cleanliness, a metal system is used so that the entire system may be heated; this causes adsorbed molecules to be released from the system's walls (a process called desorption described in more detail below). Heating the entire system (normally to about 250 °C) while pumping away the desorbed gases, usually called "baking the system", provides cleaner surfaces and allows a lower pressure to be attained.

Ultrahigh vacuum requires a stainless steel system meeting construction standards higher than for high vacuum. Achieving an ultrahigh vacuum Requires baking the system.

Glass systems are most suitable for small general-purpose applications, especially where modifications are frequently needed.

The most commonly used sources for vacuum components can be found using such buying guides as the Lab Guide edition of Analytical Chemistry or the Buyer's Guide edition of American laboratory.

### **Desorption**

A surface adsorbs molecules of any gas to which it is exposed. Most of the adsorbed molecules are physically fastened to the surface via van der Waals forces or H-bonding. These molecules are relatively easily dislodged; most leave the surface as the pressure is reduced to .001 torr or so.

Water molecules physically bind to glass and metal surfaces strongly enough (mostly H-bonding) that desorption is very slow at room temperature. This effect is seen most dramatically at pressures between  $10^{-3}$ - $10^{-6}$  torr, where the slow, steady rate of water desorption appears to determine the lowest pressure attainable. At  $10^{-6}$  torr, a glass surface typically holds roughly 30 L of adsorbed gas per square centimeter of surface area. The slow release of this quantity of gas substantially retards the pumping progress and tests the patience of any chemist.

Heating the surface (baking) to 250 °C or so provides enough kinetic energy in the H-bonding vibrational modes to cause the water molecules to let loose. As the desorption rate increases, more molecules per second are removed by the pumping process. (During baking, the pressure is determined by the steady balance between the desorption and the pumping.) Stainless steel systems can be baked with electrical heaters and ovens while the pumping is underway. Then when the system is cooled to room temperature, the wall desorption slows and the pressure in the system drops.

The process described above is also known as "degassing" the system.

While a glass system normally cannot be baked because of the presence of organic lubricants and sealants, major portions of the system can be degassed using a glass-working torch set to a relatively cool flame. A degassing chemist must be careful not to torch delicate parts of the system.

A small amount of solid or liquid contaminant in a vacuum system can produce many liters of vapor at reduced pressure. If a system won't pump down below a few millitorr, there may be a small residue of condensed volatile contaminant present. Often the only safe way to remove such a contaminant is to dismantle that portion of the vacuum system. Thus it is important not to allow such contamination to occur in the first place. Use caution in deciding what kinds of materials to introduce into a vacuum system. Usually a system used for stripping solvents from products in a synthesis lab is unsuitable for handling gases in a project doing spectroscopy of unreactive non-condensable gases.

## Vacuum Gauges

There is no one way of measuring pressure over the entire vacuum range. Fortunately there are several gas properties which depend on pressure, and a number of ingenious techniques have been developed to measure pressure for a given pressure range and application. Table 2 lists the most popular types of vacuum gauges.

At FLC, we most frequently use the following types of gauges:

Hg manometer. Easy to use for pressures above about 5 torr. Can be custom constructed. The Hg corrodes when exposed to oxygen and other reactive gases over time. The pressure measurement requires no particular calibration and does not depend on the species of gas present. Traces of Hg vapor are present in any vacuum chamber where an Hg manometer is used.

Thermocouple. The cheapest, most common way to work in the low vacuum range 2-.001 torr. The gauge measures the temperature of a heated wire to determine the thermal conductivity of the gas present. Thus the calibration depends on the species of gas(es) present. The gauge must be calibrated near the .001 torr end of its range for useful results.

McLeod. A McLeod gauge uses Hg liquid to compress a sample of gas into a closed capillary, where the pressure before compression is read from a Hg column height. This gauge cannot be used for vapors (e.g., organic vapors) which will liquefy when compressed. The Hg is reactive and produces Hg vapors. The McLeod gauge is useful for calibrating other gauges over the  $10^{-10}$ - $10^{-5}$  torr range. Small McLeod gauges devoted to only a portion of the pressure range can be purchased for about \$ 300.

Ionization. Ionization gauges are used for pressures below .001 torr. The ionization gauge contains a heated filament which releases electrons to ionize gas species, which then migrate to a negatively-charged electrode. The pressure is indicated by the current produced by the flow of ions. The electronic control for the gauge allows considerable choice for operating conditions and requires some training to use properly. Ionization gauges can be easily destroyed by operating with too much gas or with oxygen present.

**Table 2.** Common types of vacuum gauges.

<u>Gauge type</u> <u>Approx. cost</u>	<u>Useful pressure range</u> (torr)	<u>Advantages/Disadvantages</u>
Hg manometer \$100	1000-4	Hg vapor contaminant
thermocouple \$700	2-.001	Depends on gas species
Pirani \$1300	1-10 <sup>-4</sup>	Depends on gas species
Penning \$800 (cold cathode)	10 <sup>-3</sup> -10 <sup>-7</sup>	Non-reactive with organics
ionization \$1300 (Bayerd-Alpert)	10 <sup>-3</sup> -10 <sup>-11</sup>	Hot filament, reactive with organics, oxygen, easily burned out
capacitance \$1500	1000-10 <sup>-2</sup>	Several gauges to span range, indep. of species, non-reactive
Bourdon \$400 (Wallace-Tiernan)	1000-1	Several gauges to span range, indep. of species, non-reactive
McLeod \$300-\$1000	10-10 <sup>-5</sup>	Several gauges to span range, non-condensable gases only, Hg contamination

### **Valves**

There is a wide variety of valves especially made for work below 1 torr. Stainless steel or brass valves are used primarily with metal vacuum systems. For pressures below .001

torr, the aperture presented by a valve's throat is a critical determinant of the valve's gas 'throughput' in L/s. Metal valves are expensive, but durable if used properly.

On glass vacuum systems, valves are constructed of glass. There are two primary categories, ground-joint stopcocks and Teflon-bore stopcocks. The ground-joint stopcocks are precision ground and require lubrication with either hydrocarbon- or silicon-based grease, a disadvantage where either the gases present react with the grease or the grease interferes with surface cleanliness. (Greases migrate slowly via sublimation to cover all the surfaces in the system.) Teflon-bore stopcocks are free of greases, making vacuum tight seals with rubber or Teflon o-rings which press against precisely manufactured surfaces.

Note that the human wrist can overpower and thus damage vacuum stopcocks. Undue torque (angular force) can break the glass tubing to which the stopcock is attached. Ground-joint stopcocks can become frozen if not turned for several weeks, and must be coaxed into motion by carefully applying torque, preferably while holding the outside of the stopcock with the other hand. (As a last resort, gentle warming with a heat-gun may lower the viscosity of the lubricant.) Teflon-bore stopcocks don't freeze like ground-joint stopcocks, but tightening too tight destroys the o-ring seal. Close a Teflon-bore stopcock until the o-ring barely touches the seal surface.

### **Glass-metal Seals**

Glasses and metals expand upon heating, so it is difficult to reliably bond glass to metal (or even one kind of glass or metal to another kind of glass or metal), particularly to form a vacuum-tight seal. Thus vacuum engineers have developed "graded seals." A graded seal connecting pyrex glass to a given metal is comprised of a series of glasses, having similar but not identical coefficients of thermal expansion, bonded to one another to bridge the expansion difference between the Pyrex and the metal. Such seals cost \$ 15-40. Likewise, quartz can be connected to Pyrex through a quartz-Pyrex graded seal.

### **Glass Joints**

Pieces of a glass system are usually connected by so-called joints. There are three principal types: ground-glass tapered and ball joints and polished surface o-ring joints. The o-ring joints are usually used when it is important to avoid the presence of lubricants.

To open a tapered joint or o-ring joint, the joint should contain gas at or near atmospheric pressure. A ball joint under vacuum may be safely opened by pivoting one piece of the joint to allow air to enter. Of course, before opening a joint, check that you have the joint isolated from the rest of the system as desired.

### **Lubricants and O-rings**

Ground-joint stopcocks, tapered joints and ball joints all require a lubricant to supply a flexible sealing film. This film is either a hydrocarbon- or silicone-based grease. The

hydrocarbon greases are most commonly of the Apeizon brand. Apeizon Type L is designed for taper and ball joints while Apeizon Type N is for stopcocks. Apeizon makes other sealants, including a black wax which can be convenient, albeit potentially messy, for temporary repairs.

O-rings can produce vacuum-tight seals when squeezed between two polished surfaces. Glass o-ring joints can provide a grease-free connection in a glass system. O-rings are also used in Teflon-bore stopcocks. O-rings are available in a wide range of materials having different physical and chemical properties. Viton and Buna o-rings are particularly useful when organic gases will be present in the system.

### **Traps**

Cold traps are used in vacuum systems to collect both back streaming pump oil and any condensable reactive or toxic gases being drawn toward the pumps. Liquid nitrogen ( $-196^{\circ}\text{C}$ ) is the most commonly used coolant and is required for high vacuum applications. At FLC, refrigerators are also used when  $-100^{\circ}\text{C}$  provides sufficient trapping.

It is important to remember that a cold trap may collect as solids some materials which are gases at room temperature. Several grams of such materials may collect in the trap if relatively large amounts of the respective gas is handled in the vacuum system. If a trap containing several grams of volatile solid is warmed to room temperature without being vented to atmosphere, the resulting buildup of pressure can cause a hazardous explosion. Also, oxygen will condense to liquid at  $-196^{\circ}\text{C}$ , so air drawn through a trap cooled with liquid nitrogen can leave a hazardous amount of liquid oxygen in the trap. Carefully vent such a trap to the atmosphere when warming it. Note that liquid oxygen is highly flammable.

**FINAL NOTE: Several of the labs require a measurement of the ambient pressure in the lab. We will use the Princo mercury barometer mounted on the east wall. The manual for its use will be close by – be sure to read it! In fact, we will take the time to correct these readings for temperature and gravity (latitude). The spreadsheet for doing so is shown on the next page and can be downloaded from this course's website. Include this spreadsheet in your reports for those labs requiring this measurement.**

# English

## MS Excel 2000 Spreadsheet for *Automatic Barometer Corrections*

Courtesy of **Princo** Instruments, Inc.  
 Manufacturer of level and density controls, barometers and thermometers.  
 Visit our web site at: <http://www.princoinstruments.com>

### Input Data Field (type data in light shaded boxes)

Type institution, location, time, date, or other optional data; in light shaded box below:

**FLC Chemistry Department**  
 1000 Rim Drive, Durango, CO 81301  
 2005

Barometer reading, type in light shaded box at right	597.6	mm Hg @
Temperature of barometer, type in light shaded box	23.5	23.5°C °C
Normal data settings, type in light shaded boxes:		
Reading units (in., in, cm, mm, mb, kPa)	mm	
Certificate correction if any, + or -	0	mm
Temperature units (F, C)	C	
Latitude, degrees N or S	37.3	°
True altitude	6866	feet
Altitude units (feet, meters, metres)	feet	

### Results Field (automatically computed)

Barometer scale system of units, English or metric	<u>metric</u>	
Coefficient of volume thermal expansion of mercury, M	0.0001818	m <sup>3</sup> /m <sup>3</sup> °C
Coefficient of linear thermal expansion of brass, L	0.0000184	m/m°C
Temperature, in the barometer scale system of units, t	23.5	°C
Standard temperature for the barometer scales, t <sub>s</sub>	0	°C
Standard temperature for the density of mercury, t <sub>m</sub>	0	°C
Standard pressure at sea level, p <sub>0</sub>	760	mm Hg @ 0°C
Multiplier for temperature correction, M <sub>tc</sub>	-0.003824	mm/mm
Multiplier for gravity correction, M <sub>gc</sub>	-0.000750	mm/mm
1. Barometer reading and temperature	597.6	mm Hg @ 23.5°C
2. Certificate correction if any, + or -	<u>0</u>	mm
3. Certificate corrected reading	597.6	mm Hg @ 23.5°C
4. Correction for temperature	<u>-2.28</u>	mm
5. Temperature corrected reading	595.32	mm Hg @ 0°C
6. Correction for gravity, + or -	<u>-0.45</u>	mm

7. <b>Local station pressure</b>	<b>594.87</b>	<b>mm Hg @ 0°C</b>
8. Pressure altitude	6623	feet
9. Minus (-) the true altitude	<u>-6866</u>	feet
10. Pressure altitude differential, + or -	-243	feet
11. <b>Sea level pressure</b>	<b>766.69</b>	<b>mm Hg @ 0°C</b>

## Physical Measurements - Core I

### **Experiment 1: Determination of an Unknown Volume using Boyle's Law**

#### PURPOSE

To measure the volume of a gas container using Boyle's Law and the general-purpose vacuum system.

#### EXPERIMENTAL

##### Part I. General Instructions for Vacuum Operations

1. Wear Splash Goggles when working with a glass vacuum system.
2. Never open a valve without considering the consequences -- where the gas will go and how you will remove it. Know which gauge reading will change and in what way.
3. Turn valves carefully, slowly with torque appropriate for a toothpaste tube. If a valve doesn't turn with moderate torque, consult an instructor.
4. Do not over-tighten Teflon-bore valves. Such a valve is closed when the o-ring is slightly touching the polished orifice surface. Do not flatten the o-ring against this surface.
5. To open a ball joint, tapered joint or o-ring joint, the joint must contain gas at or near atmospheric pressure. The joint may be vented by opening the appropriate valve(s). A ball joint under vacuum may be safely opened by pivoting one piece of the joint to allow air to enter.
6. When mounting bulbs or other attachments to the system, do not overtighten clamps. Hold the piece in place while closing the clamp jaws onto the piece. The clamp jaws should be slightly snug.
7. Never warm a sealed trap without directly monitoring the pressure. If a trap contains a volatile condensed material (liquid or solid), close the valve to the pump and vent the trap. After warming the trap to room temperature, demount it. Warm the trap body, clean it, and replace it for the next user (or clamp it upside-down to the system lattice for safe storage).

8. Do not introduce a new substance into a vacuum system without consulting an instructor. Contamination can ruin an entire system.
9. Use care with liquid nitrogen. Wear Splash Goggles when transferring liquid nitrogen. Wear gloves when coming into direct contact with liquid nitrogen cooled surfaces.
10. Leave the Welch Duo-Seal mechanical vacuum pump running when you are finished. They are designed to run without interruption.

## Part II. Experimental Procedure for Boyle's Law Lab

NOTE: this is only one way to do the experiment - there are others.

1. You should find the mechanical pump running and its valve open.
2. Add liquid nitrogen to the trap dewar flask so that there are about 2-3 inches of liquid in the bottom of the flask. Carefully slip the Dewar under the trap and elevate it into position with a lab jack.
3. Fill bulb 1 with air without removing it from the vacuum system to atmospheric pressure – measure atmospheric pressure using the barometer. Close its valve B1. The volume of bulb 1 is  $1.04 \pm .01$  L.
4. Check that all valves are closed except for valve V1, and V2. Open the manifold valve slowly and observe the pressure drop using both the manometer and the thermocouple gauge. The pressure should drop to about 30 mtorr or less as indicated by the thermocouple gauge. This is usually called the background pressure.
5. Close the manifold valve and observe the rate at which the pressure creeps upward. A rate of 30 mtorr/min or less is acceptable for this project. Record the rate of increase.
6. Reopen the manifold valve and wait until the pressure drops below 30 mtorr.
7. Close valve B2 and then the manifold valve.
8. Slowly open valve B1 to release the air from bulb 1 into the manifold. Measure the pressure.
9. Open valve B2 and again read the pressure.
10. You now have enough information to deduce the volume of the manifold and of bulb 2.

11. If you wish to repeat the volume measurement, remove bulb 1 (leaving valve V1 closed) and refill it with air. Close valve B1 and remount the bulb. Open valve V1, V2, B2 and the manifold valve and evacuate the system to 30 mtorr or less. Repeat steps 6-10.

12. If you are finished, open the manifold valve, reduce the pressure to 30 mtorr or less, and repeat the measurement of the atmospheric pressure (using the barometer).

### Part III. To Shut down the Vacuum System

When finished with the vacuum system, do the following:

1. If it is possible that the system trap contains solid water or any other condensed volatile, the trap should be emptied.

To do this, close the valve to the mechanical pump and open the valve to the laboratory to bring the system to atmospheric pressure. Then allow the trap to reach room temperature and carefully remove the trap jacket (the outer tube) by twisting the tapered joint loose. (If the joint doesn't move under reasonable torque, consult an instructor.) Either wipe out the trap jacket or let the contents evaporate if appropriate. Clamp the jacket upside-down safely on the vacuum rack to dry.

2. Leave the mechanical pump and the thermocouple gauge on.

3. Straighten the area. Discard or store any leftover liquid nitrogen.

### CALCULATIONS

Use Boyle's Law (or  $PV = nRT$ ) to determine the volumes of the manifold and Bulb 2 as well as the uncertainties in each of these measurements.