VACUUM SYSTEMS

PHYSICS 359E

September 28, 2004

INTRODUCTION

In this laboratory, you will familiarize yourself with the principles of simple vacuum systems and their use. You will measure the pumping speed under various conditions and will determine the conductance of several apertures and tubes.

PRE-LABORATORY PREPARATION

- Read entire lab write-up, including "Operating Instructions" below. (You won't necessarily follow these exactly in the exercises below, but they are a guide to the normal operation of the system.)

VACUUM SYSTEMS AND COMPONENTS

In one way or another vacuum techniques appear in most fields of modern physics. These notes are intended as a guide to the use of the systems available in the P359E laboratory, and as an introduction to more detailed references on the subject.

Measurement of Pressure

Atmospheric pressure at sea level is sufficient to raise a column of mercury 760 mm. For our purposes, a vacuum is defined by a pressure of less than 1 mm Hg $\equiv$ 1 torr (named after Torricelli, who invented a form of vacuum pump). In spite of the fact that the torr has been officially superseded by the SI unit, the Pascal (Pa), it still in very wide use by scientists and engineers. One Pa $\equiv$ 1 N m^{-2} and 1 torr = 133.3 Pa. Show that 1 torr at room temperature corresponds to a particle density of 3.5×10^{16} particles cm^{-3}.

Pressures in the range from 1 torr to 1 millitorr are usually considered a relatively poor vacuum. Between 10^{-3} and 10^{-7} torr is referred to as high vacuum, while pressures less than 10^{-7} torr are ultrahigh vacuum. High vacuum is readily achieved in a clean aluminum or stainless steel system using synthetic rubber gasket seals and a diffusion pump (see below), while ultra-high vacuum requires ultra-clean bakeable stainless steel chambers, metal gaskets, and a selection of ion pumps, cryopumps, molecular turbopumps, and titanium sublimation pumps.

The other common purpose in using a vacuum system is to maintain a surface clean and uncontaminated by atmospheric gases. A straight-forward derivation based on kinetic theory gives the number of atoms N incident on unit area per unit time as

$$N = \frac{1}{4} n \langle v \rangle, \tag{1}$$

The significance of these pressures may be better appreciated from a couple of examples. A frequent reason for carrying out experiments in high vacuum is to permit atoms or ions to move without colliding with air molecules. The mean free path is the mean distance traveled by a particle between collisions. Table I shows the mean free for an oxygen molecule as a function of pressure.

Table I. Mean free path of 0_2 in air

Pressure (torr)	Mean free path (cm)
760	6.8×10^{-6}
10^{-3}	5.1
10^{-6}	5.1×10^{3}
10^{-10}	5.1×10^{7}

where n is the density of atoms in the gas and $\langle v \rangle$ is the mean velocity. The values of N may be better appreciated by estimating the time required to form a mono-molecular layer of gas on a freshly cleaned crystal face in a vacuum. On such a surface the probability that a gas molecule which strikes the surface will stick to it is something of the order of unity. Hence the time τ to form a mono-layer is given by

$$\tau \sim \frac{1}{N\sigma}, \tag{2}$$

where σ is the area covered by a single molecule adhering to the surface. Estimates of N and τ are given as a function of pressure in Table II.

Table II. Time to form a mono-layer with $\langle v \rangle = 5 \times 10^4$ cm s^{-1} and $\sigma = 10^{-15}$ cm^2

P(torr)	P(Pa)	n(cm^{-3})	N(cm^{-2} s^{-1})	τ(s)
1	133	3.5×10^{16}	4.4×10^{20}	2×10^{-6}
10^{-6}	1.33×10^{-4}	3.5×10^{10}	4.4×10^{14}	2
10^{-10}	1.33×10^{-8}	3.5×10^{6}	4.4×10^{10}	2×10^{4}

One final interesting comparison is the gas pressure in interstellar space - about 10^{-16} torr or ~ 1 atom cm^{-3}, considerably better than the best laboratory vacuum. It is almost all hydrogen.

Pressure Gauges

Several types of gauges are used in the lab: the thermocouple gauge, the capacitance manometer ('Baratron'), and the ionization gauge. Operation of each is described in Ref. 4, Ch. 4 and in their manuals. Vacuum gauges tend to be inaccurate for absolute measurements. Unless contaminated, they usually provide a reliable indication of relative pressure and pressure changes, but an intercomparison of different gauges may show appreciable discrepancies in indicated absolute pressure.

Pumping Speed of a System

The speed S of a pump, usually expressed in litres s^{-1} (ℓ/s), is defined as the volume of gas carried through the pump per unit time, measured at the pressure of the pump inlet. Thus to move a given mass of gas per unit time a mechanical forepump can have much lower speed than the diffusion pump which it is backing.

Pumps are connected to the high vacuum region by pumping lines and orifices. A pumping line has a conductance G (dimensions: volume per unit time) defined as follows: if the pressures at the entrance and exit of a pumping line are P, P', then the "quantity of gas" transported through the line in unit time (the throughput) is $(P - P')G$. (Note that the dimensions of this expression are pressure $\times$ (volume/time), and by the ideal gas law, pressure $\times$ volume α mass.) With this definition it is straightforward to calculate the effective pumping speed of a pump connected to a volume V_0 through a line of conductance G. Suppose the speed of the pump itself is S'. Then the mass of gas passing through the pump per unit time is proportional to $P'S'$, and from the definition of conductance $P'S' = (P - P')G$. The speed S at the entrance to the line is defined by the relation $PS \equiv (P - P')G$. These two relations require that

$$1/S = 1/S' + 1/G. \tag{3}$$

The reciprocals of conductances and pumping speeds are analogous to resistances, and add when they are in series, just like electrical resistance. Expressions for G for a variety of different geometries can be found in Ref. 4, Ch. 2. At low enough pressures the mean free path is large compared with dimensions of the apparatus, and kinetic theory can be used to calculate G. For a circular aperture of radius R(cm) in a thin plate with gas of molecular weight M(gm mole^{-1}, about 29.0 for dry air) at temperature T (measured on the Kelvin, not the Celsius, scale!),

$$G = 3.64\pi R^2 (T/M)^{1/2}\ \ell\ \mathrm{s}^{-1}. \tag{4}$$

For a cylindrical tube of length L(cm), radius R(cm) (note the units of L and R!),

$$G = 30.5 \frac{R^3}{L + (8/3)R} (T/M)^{1/2}\ \ell\ \mathrm{s}^{-1}. \tag{5}$$

It is easy to see that effective pumping speeds can be seriously limited by small apertures or pumping lines.

Pumpdown

If a volume V_0 is being pumped by a system of effective speed S, the pressure in the volume V0 satisfies the differential equation

$$\frac{dP}{dt} = -\frac{PS}{V_0}. \tag{6}$$

If S is independent of P, then $P = P_0 \exp(-St/V_0)$, where P_0 is the initial pressure. Thus for rapid pumpout, the characteristic time V_0/S should be small. If the pump being used has an ultimate pressure P_u, then the differential equation becomes

$$V_0 \frac{dP}{dt} = -\frac{(P - P_\mathrm{u})S}{V_0} \tag{7}$$

with solution

$$P = (P_0 - P_\mathrm{u}) \exp(-St/V_0) + P_\mathrm{u}, \tag{8}$$

which shows the P makes an exponential approach to P_u.

Residual gases

Vacuum systems may contain small leaks, which admit gas to the system at some fixed rate. Even if the system has no leaks to the outside, it may contain materials with a high enough vapour pressure that an effective (or virtual) leak is present. These leaks or virtual leaks may then determine a base pressure for the system. This is the lowest pressure attainable by the system and is reached when the full capacity of the pump is used to handle the load from the leaks.

When a system is being pumped down from atmospheric pressure, gas adsorbed on interior surfaces is slowly pumped away. This 'outgassing' phenomenon represents a time-dependent virtual leak, which happens on a timescale that is usually much longer than the characteristic pumpdown time V_0/S. Thus a plot of pressure vs. time must be interpreted with care. You will observe this phenomenon.

If the total leak rate (real + virtual) is L(t), the differential equation becomes

$$\frac{d}{dt}(PV_0) = V_0\frac{dP}{dt} = -(P - P_{\rm u})S + L(t). \tag{9}$$

A useful special case is L *constant*, which can be achieved in practice by waiting until the outgassing has become negligible with respect to any other leaks. In this case the system will pump down to a base pressure $P_{\rm b}$ where the full capacity of the pump is required to remove the gas from the leak. Setting $dP/dt = 0$ at $P = P_{\rm b}$ yields

$$(P_{\rm b} - P_{\rm u})S = L. \tag{10}$$

This expression forms the basis for an experimental measurement of S. A system is pumped to its ultimate pressure $P_{\rm u}$, and then gas is admitted at a *known* rate L through a calibrated leak. A measurement of the resulting base pressure permits determination of S.

Components

The major components of a typical high vacuum system are indicated schematically in Fig.1.

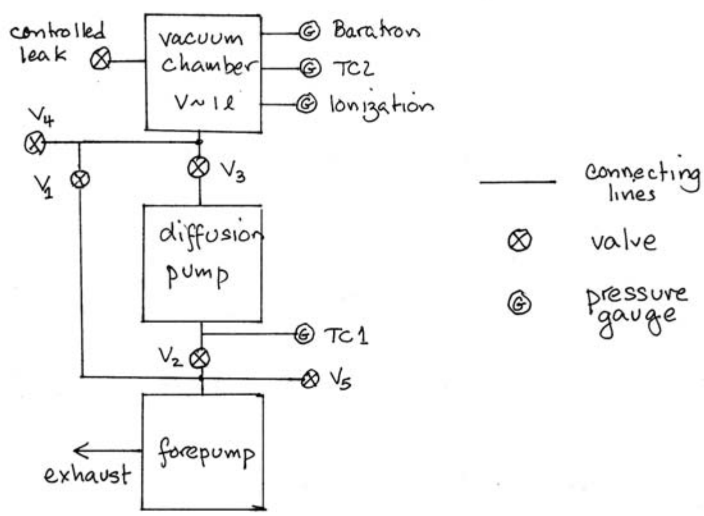


Figure 1: Pumping system in the Physics 359 lab

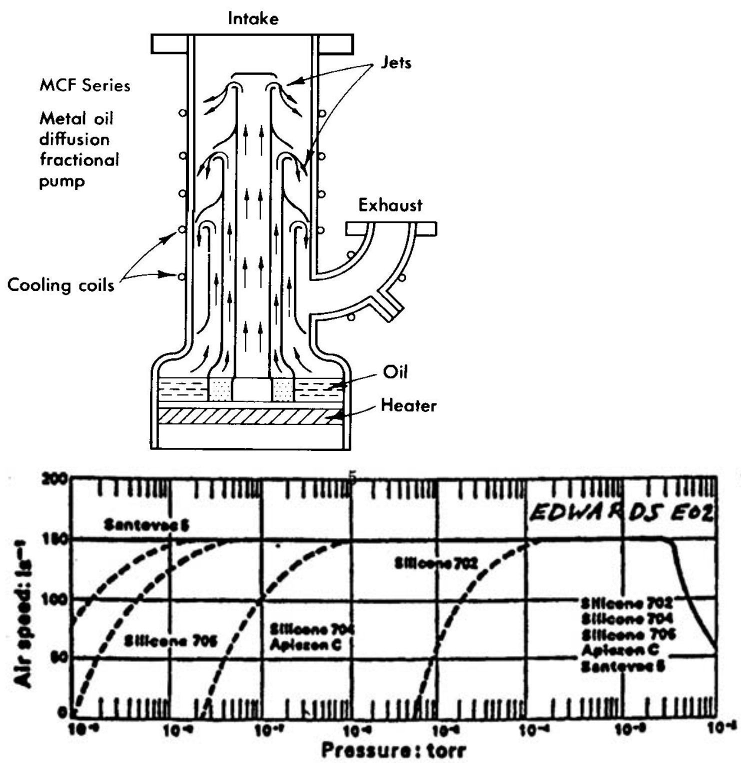
Compare the system in the laboratory carefully with this figure.

A detailed discussion of the properties of the various components is given in the references. Manufacturer's manuals are also available in the lab. The following discussion is intended mainly as a guide to the references.

Forepump: This is usually a mechanical device in which the motion of a suitable piston removes gas from the vacuum system and exhausts it to the atmosphere. The operation of a typical forepump is discussed in Ref. 4, Ch. 3.

The important characteristics of a mechanical pump are its pumping speed and its ultimate vacuum. The speed is determined by the mechanical dimensions of the pump. Typical small laboratory pumps have speeds in the range 0.1 – 10 ℓ/s. The ultimate vacuum depends on the design of the pump, its mechanical condition and the type and purity of the oil in it. A mechanical pump in reasonable condition should be able to produce an ultimate vacuum of about 10^{-2} torr.

High Vacuum Pump: The commonest type of high vacuum pump is the diffusion pump. In this pump, a jet of condensible vapor sweeps air molecules from the system and carries them to the point where they can be removed by the forepump. Since a diffusion pump relies on the action of the vapour jet, it cannot operate at high pressures. A diagram of a typical diffusion pump is shown in Fig. 2a.



(a) Construction of a typical diffusion pump. This is a three-stage pump since there are three successive vapour jets to provide the pumping action.

(b) Typical pumping speed curve for a pump similar to that shown in (a).

Figure 2:

The important characteristics of a diffusion pump are its pumping speed, maximum exhaust pressure (the forepressure provided by the forepump) and ultimate vacuum. A discussion is found in Ref. 4, Ch. 2.

OPERATING INSTRUCTIONS

Choose I, II or III as appropriate. Refer to Fig. 1 above for valve locations

I. Initial pump-out with the diffusion pump **COLD**.

If the diffusion pump is cold, close vent valves V4 and V5, open valves V1, V2, and V3, and switch on the forepump. Monitor pressure on the thermocouple (TC) gauge. When the pressure is less than 100 millitorr, close V1 and turn on the fan, the cooling water to the baffle valve, and the diffusion pump heater. The diffusion pump will require about 20 minutes to come into operation. You may switch on the ionization gauge when you are sure the pressure is less than 1 millitorr.

II. Opening the vacuum chamber with diffusion pump **HOT**

Turn off the filament on the ionization gauge.
Close valve V3.
Check that valve V1 is still closed.
Open valve V4 to admit air to the vacuum chamber.

III. Pump-out of vacuum chamber with the diffusion pump **HOT** (start with V3 closed)

Close vent valve V4.
** Close valve V2 to isolate the diffusion pump temporarily.***
Open V1 to "rough out" the vacuum chamber through the bypass line.
When the pressure is less than 100 millitorr,
a) Open V2 to start pumping on the diffusion pump.
b) Close V1.
c) Slowly open V3, the diffusion pump baffle valve, keeping your eye on the TC gauge. Try to throttle the valve so that the pressure in backing line stays below 100 millitorr. (If this is difficult, open V1 until the baffle valve is completely open and the pressure is less than 100 millitorr; then close V1.)

Since the diffusion pump is hot, the chamber should pump down to less than 1 millitorr in less than one minute. Check the TC gauge on the chamber and switch on the ionization gauge filament. If it switches off immediately, wait 5 minutes and try again. If there are still problems, consult an instructor.

WARNING: Never let air at a pressure greater than 100 millitorr into the diffusion pump when it is hot. If you do so, you will be asked to help in the resulting clean-up job. The oil remains hot for about 20 minutes after the switch is turned off.

PROCEDURE

1. Familiarize yourself with the apparatus, identify all the components, valves and gauges.

2. Pump down the system from a cold start. Intercompare the thermocouple and Baratron gauges where they overlap. When the chamber pressure is below one millitorr, turn on the ionization gauge and record pressure vs. time. The system should reach an ultimate pressure of below 10^{-5} torr. Plot $\ln(P - P_\mathrm{u})$ vs. t and interpret the result.

   Repeat this procedure starting with the diffusion pump hot.

3. Measure the pumping speed using the calibrated leak by measuring the time for a known volume of air to enter the system [see Eq. (10)]. Set the leak rate with the needle valve to be high enough that $P_\mathrm{b} \gg P_\mathrm{u}$. Use the rise of oil in the pipette to measure the volume of air

(which is at approximately atmospheric pressure in the rubber tube: why?) introduced into the vacuum chamber per unit time. The pumping speed S of the system is given by

$$S = \frac{L}{P_{\mathrm{b}} - P_{\mathrm{u}}}. \tag{11}$$

In this set-up the leak rate L is given by the expression

$$L = \frac{d}{dt}(PV) = P\frac{dV}{dt} + V\left(\frac{dP}{dh}\right)\left(\frac{dh}{dt}\right) \approx P_{\mathrm{atm}}\frac{dV}{dt} + V(\rho_{\mathrm{oil}}g)(v), \tag{12}$$

where ρ_{oil} is the oil density, V is the volume of the system between the oil surface and the needle valve (61 ± 1 ml), and v is the speed of the oil level rise in the pipette. The volume in the pipette is 1 cm^3 (1 ml) between the bottom and top gradations. Note that both terms in Eq. 12 are *negative*, and comparable in size. If you find one term much larger than the other, you have probably made a mistake with units. (You must be very careful with units when you use this formula: either measure *everything* in SI units, *or* in cgs. If you keep pressure in torr, volume in litres, and oil rise speed in m s^{-1} you will get complete nonsense for an answer.)

4. Three tubes are available which can be mounted at the entrance to the diffusion pump. Measure the pumping speed with each of these in place. Use the results and Eq. (3) to determine the conductance of the tubes and compare with the theoretical values from Eq. (5).

5. Fill the cold trap with liquid nitrogen and observe the effect on the ultimate pressure and the pumping speed.

REPORT

Your report should include the following:

1. Your calculation of the particle density for 1 torr.

2. A description and analysis of your measurements of the variation of pressure with time as you pump down the system from both cold and hot start, and a brief explanation of what the time constants you measure mean.

3. An analysis of your experimental measurement of the pumping speed of the system, using the calibrated leak.

4. An analysis of your measurements of the conductances of the various tubes through which you pump down the upper chamber of the vacuum system, and a comparison with theoretical values. Try to provide meaningful error estimates in spite of the uncertainties in absolute pressures.

References

1. S. Dushman and J.M. Lafferty 1962, Scientific Foundations of Vacuum Technique, 2nd Ed (New York: John Wiley & Sons). An encyclopaedic treatment of all aspects of vacuum systems. Many useful tables.

2. A. Guthrie 1963, Vacuum Technology (New York: John Wiley & Sons). Simple presentation of theory with much information on actual use of components and systems.

3. Melissinos, A. 1966, Experiments in Modern Physics, 1st Ed. (New York: Academic Press), pp 126-39. Copy in the lab.

4. A. Chambers, R. K. Fitch, B. S. Halliday 1998, Basic Vacuum Technology (Bristol: Institute of Physics Publishing). Copy in the lab.

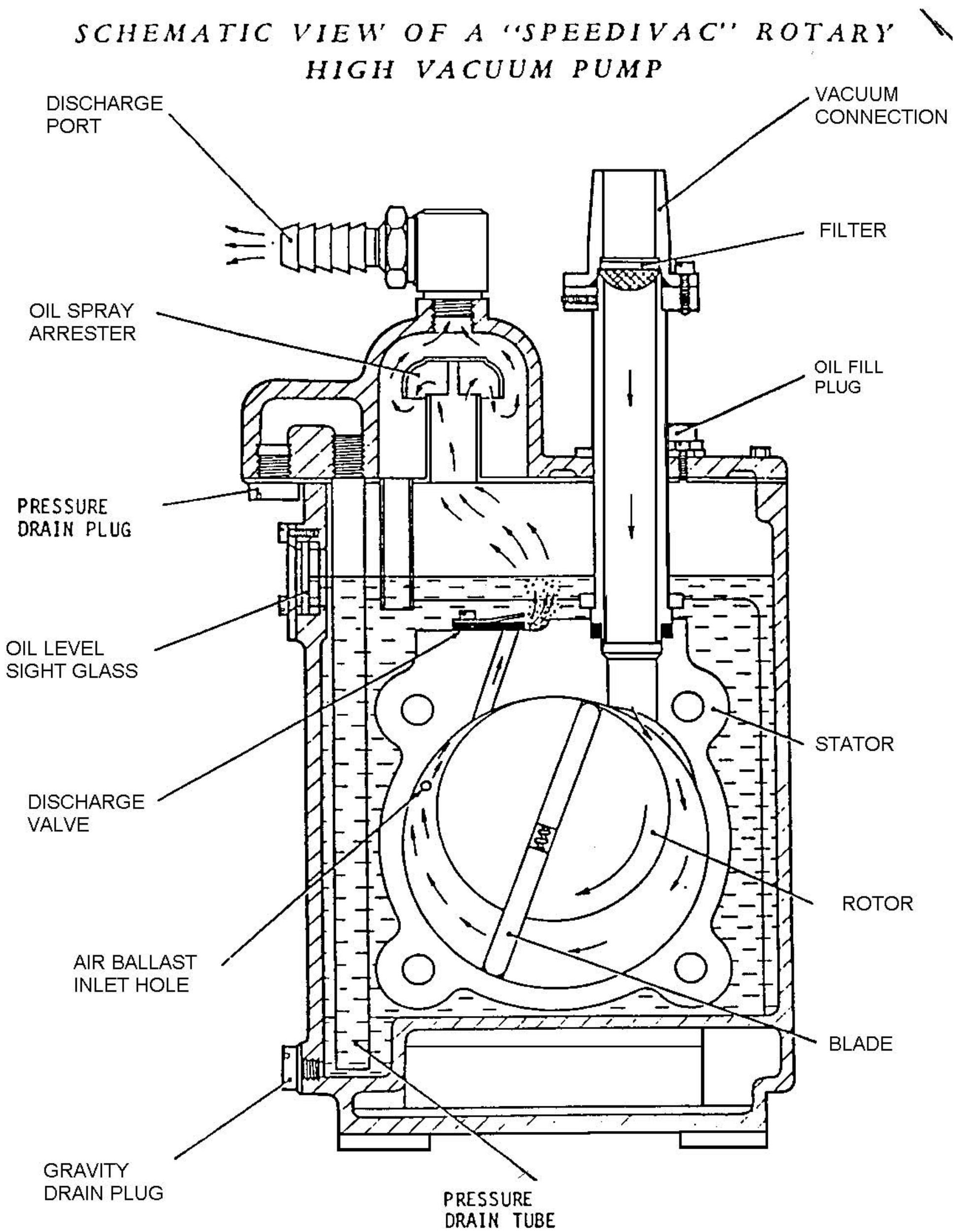


Figure 3: A typical forepump