4. USEFUL TECHNIQUES

4. Vacuum Techniques

The creation and measurement of vacuum is necessary in many experimental devices and applications. Residual pressure (vacuum) is measured in millimeters of mercury, and present-day commercial devices can reach pressures as low as 10⁻¹⁰ mm. An improvement by one or two orders of magnitude has been achieved by some laboratories, and similar advances have been made in the field of vacuum-measuring devices. For comparison, the residual pressure in free space is 10⁻¹⁶ mm, corresponding to a density of one molecule per cubic centimeter.

Vacuum pumps are of three main types, each for a specific range of pressures:

(a) Mechanical pumps, which can operate against atmospheric pressure and down to a limit of 10⁻² to 10⁻³ mm. They are used as “back-up” pumps to create the necessary rough vacuum for the operation of a high vacuum pump.

(b) Molecular diffusion pumps with capabilities down to 10⁻⁴ to 10⁻⁵ mm.

(c) Ion and sputter ion pumps with limits at 10⁻⁸ - 10⁻¹⁰ mm.

It must also be noted that the lowest pressure that can be reached is limited by the “vapor pressure” of the materials in the system. To eliminate contaminations of such volatile materials in a high-vacuum system, it is standard practice to “bake out” the systems at high temperatures for several hours or days. For the same reason it is useful to use “cold traps” to condense the volatile vapors. As a matter of fact, the highest vacuums have been achieved by cryogenic techniques.

4.1 Introductory Concepts

We begin by giving some quantitative relations and by recalling some topics of gas flow. A vacuum system is characterized by its speed, \( S \), in \( \text{cm}^2/\text{sec} \). The speed, \( S \), depends on the pressure, and there exists for every system a lower limit, the ultimate pressure that can be reached, \( P_u \); the approach toward this ultimate limit is exponential, and we have

\[
\frac{dP}{dt} = \frac{S}{C} (P - P_u)
\]  

(4.1)

where \( C \) is the total volume to be evacuated, and \( P \) is the instantaneous pressure. Obviously the above expression is dimensionally consistent and can be used as a definition of \( S \). Integration of Eq. 4.1, if \( S \) is assumed constant, yields

\[
P = (P_0 - P_u) \exp \left( -\frac{tS}{C} \right) + P_u
\]  

(4.2)

where \( P_0 \) is the initial pressure at time \( t = 0 \).

The speed of a system is determined by the speed \( S_p \) of the vacuum pump employed, as well as by the tubing connecting the pump to the vessel to be evacuated. We define the “throughput,” \( Q \), of a system as the volume of gas entering (or leaving) it per unit time, multiplied by the pressure:

\[
Q = SP
\]  

(4.3)

Then, for a system of conductors (tubing) the conductance \( F \) is defined by

\[
Q = F(P_1 - P_2)
\]  

(4.4)

where \( P_1 \) and \( P_2 \) are the pressures at the two ends of the system.

We consider now a volume \( C \) at a pressure \( P \), which is evacuated at a speed \( S \). The volume is connected to a pump of speed \( S_p \) through tubing of conductance \( F \); let the pressure at the pump be \( P_p \). We obtain

\[
Q = SP \quad Q_p = S_p P_p
\]  

(4.5)
it follows
\[
\frac{1}{S} = \frac{1}{S_p} + \frac{1}{F}
\]
giving the basic relation between the speed \( S_p \) of the pump employed and the speed with which the system is evacuated.

In designing a vacuum system, if the gas flow into the system (through leaks, etc.) is not known and only a desired pressure is stated, a safe rule of thumb is to plan to obtain a speed \( S = C/\text{sec} \); where \( C \) is as before the volume to be evacuated and \( S \) must be available at the desired pressure.

To evaluate the conductance \( F \), we note that it depends on the type of gas flow through the system, and on geometrical factors. The gas flow can be of two types:

(a) Viscous (Poiseuille) flow, which prevails at high pressures, and is analogous to nonturbulent fluid flow;
(b) Molecular flow, which prevails at very low pressures when the mean free path of the molecules is of the same order as the diameter of the conductor (tubing), or is larger.

For viscous flow, the throughput \( Q \) depends on the pressure differential. For a long straight tubing of length \( l \) and radius \( a \) (in centimeters), if the low pressure is \( P_1 \) and the exhaust pressure \( P_2 \) (in microns = \( 10^{-4} \) mm of mercury), we obtain
\[
Q = \frac{10\pi (a^4)}{96\eta} (P_2 - P_1) \text{ microns-cm}^2/\text{sec}
\]
where \( \eta \) is the coefficient of viscosity of the gas, which for air is
\[
\eta = 1.845 \times 10^{-5} \text{ poise}
\]

By our definition, \( F = Q/(P_2 - P_1) \) and if we let \( P_s = (P_2 + P_1)/2 \), we obtain for viscous flow
\[
F_s = \frac{10\pi}{48\eta} P_s \left( \frac{a^4}{l} \right) \text{ cm}^2/\text{sec}
\]

Next, to see when molecular flow becomes important, we use the following two expressions for the mean free path:
\[
\lambda = \frac{1}{\sqrt{2} \pi n \delta}
\]
where \( n \) = number of molecules per unit volume, and \( \delta \) = molecular di-
The vapor stream is provided by boiling off organic oils which, however, are recovered by condensing upon collision with the cooled walls of the pump; usually two to three jets are used in series. When each jet is supplied by a separate boiler, the pumps are called "fractionating"; this procedure results in automatic selection of the lowest vapor-pressure components of the oil, for the jet closest to the high-vacuum side.

Fig. 4.16a is a sketch of the three-jet oil diffusion fractionating MCF pump manufactured by Consolidated Vacuum of Rochester, New York.

Fig. 4.16b gives the pumping speed curve for a similar two-jet glass pump, the VMF-20, manufactured by the same company. As can be seen from the curve, a diffusion pump cannot work against any forepressure, since its speed drops to zero; on the low side, however, it has an ultimate pressure which is limited only by the vapor pressure of the oil employed. The heaters of a diffusion pump should not be turned on unless a rough vacuum of $10^{-2}$ mm has been reached, since otherwise the oil oxidizes and is unusable for proper pumping action.
Ion pumps are based on the principle of ionizing the molecules of the residual gas and then accelerating them toward the walls of the system, where they get buried due to the impact. In addition, use is made of the chemical affinity of many gas molecules to combine with a "getter" material, such as titanium, so that they are removed from the residual gas atmosphere. Such getters have been used for many years in electron vacuum tubes.

Ion pumps may be of the cold cathode or hot filament type. In the former, electrons are released from the cathode by field emission toward the anode; they are then trapped in the active volume of the pump by a magnetic field. By collision, these electrons produce ions, which are accelerated back toward the cathode, as shown in Fig. 4.17a; they end up in the collector, where they get buried. Those ions that hit the titanium cathode release titanium ions, which also are attracted toward the collector; the titanium ions cover the residual gas molecules and provide a fresh titanium surface for chemical gettering.

Hot filament (evapor-ion) pumps use the electron beam from the filament to bombarding a titanium wire which is continuously evaporating. The titanium condenses on the cooled walls of the pump and thus provides an active getter layer which captures the molecules of the active gases that strike it; furthermore, the new layers of titanium cover the molecules that have been trapped. Obviously if air is accidentally admitted to the pump while the filament is hot, the whole system will be coated with titanium.

Figure 4.17b gives the speed curves for both the hot-filament and di­vac ion pumps.

4.3 Vacuum-Measuring Devices

Measurement of residual pressure, especially at very low values, has always been a difficult problem; similarly the calibration of the various instruments in the $10^{-7}$-mm and lower range is dependent on the nature of the residual gas, impurities in the system, and conditions of the surfaces of the measuring device. Present-day techniques can measure residual pressures of $10^{-11}$ to $10^{-12}$ mm and claim a sensitivity even in the $10^{-14}$ region. In this section, however, we will describe only three types of gauges used most commonly in laboratory and industrial applications:

(a) The McLeod gauge, a mercury manometer with a "pressure amplifier." It is an absolute device, and covers the range from 10 to $10^{-4}$ mm.

(b) The thermocouple gauge, which measures the rate of cooling, by conduction, of a heated filament; it covers the range from 0.5 to $5 \times 10^{-4}$ mm.

(c) The ionization gauge, which measures the rate of production of ions by an electron beam of standard (current) density and energy; it covers the range from $10^{-4}$ to $10^{-8}$ mm.
The principle of the MacLeod gauge is shown in Fig. 4.18; to measure the pressure, the mercury level in the gauge is raised (either mechanically or by allowing atmospheric pressure into the mercury reservoir). As the mercury rises above point A it seals off the residual gas now trapped in volume \( V \); when the level is further raised, this gas is compressed (thus the pressure is amplified according to \( PV = \text{constant} \)). Let the mercury level reach \( C \) in the measuring capillary, and \( C' = C'' \) in the reference capillary and connecting tube respectively; the pressure in the measuring capillary \( P_s \) must then equal the difference in levels between \( C' - C = h \) plus the pressure of the system, \( P_r \).

\[
P_s = h + P_r \tag{4.11}
\]

If the cross section of the capillary is \( b \), the trapped volume is \( v = bh \), and since the original volume was \( V \),

\[
vP_s = VP_s = hbP_s = bh(h + P_r) \tag{4.12}
\]

it follows that

\[
P_s = \frac{h^2 b}{V - bh} \tag{4.13}
\]

If several such amplification stages are combined in series, it becomes possible to read pressures over a wide range. There are limitations of the MacLeod gauge, however: (a) surface tension effects in the capillaries, (b) the amount of mercury (weight) that has to be lifted, (c) the fact that vapors soluble in mercury cannot be measured, and (d) the fact that since the vapor pressure of mercury at room temperature is \( 1.8 \times 10^{-3} \) mm, any high-vacuum system must be isolated from the gauge by a cold trap.

The thermocouple gauge consists of a junction which is heated by a filament through which a standard current (of the order of 0.6 amp) is flowing. The electromotive force developed at the thermocouple junction can be measured directly on a microammeter; it ranges from 0 to 14 mV for the range of 1 to \( 10^{-3} \) mm Hg. At high residual pressures the rate of cooling of the junction by conduction is large, and thus its temperature is low; at low residual pressures the junction temperature rises.

A sketch of a thermocouple gauge is given in Fig. 4.19a, while Fig. 4.19b shows the simple circuit used in this laboratory with such a gauge.

The ionization gauge, shown in Fig. 4.20, is widely used in medium-to-high vacuum systems. Like the ion pump, the gauge can be of either the cold-cathode or hot-filament type. In the filament ion gauge (much as in the Frank-Hertz tube), the emitted electrons are accelerated by a positive grid \( (E_a = +150 \text{ V}) \); the ions produced by the electron beam are collected at a plate maintained at a negative potential \( (E_p = -25 \text{ V}) \). The electron beam must be standardized, and this is achieved by measuring and maintaining a fixed grid current, usually \( I_g = 5 \text{ mA} \).

For pressures lower than \( 10^{-3} \) mm Hg, and for dry air, the ion current is of the order of 100 \( \mu \text{amp}/10^{-3} \) mm Hg; this calibration, however, is different for other gases. The ion current varies to a good approximation linearly with pressure, so that depending on the sensitivity of the d-c amplifier used measurements can be made down to \( 10^{-7} \) mm Hg \( (10^{-10} \text{ amp}) \). In order to obtain accurate readings at low pressures, it is necessary to "degas" the grid by heating it electrically.

Unfortunately, if the gauge is operated at pressures above \( 10^{-3} \) mm at full heater current, the filament burns out (due to oxidation) in a few seconds.
4.4 Some Experimental Measurements of Pumping Speeds

To demonstrate some of the properties of vacuum systems discussed previously, we present data obtained by a student.† The vacuum system was made of glass with a two-stage oil diffusion pump and no cold traps.

Figure 4.21 gives the pressure-against-time curve, with \( t = 0 \) chosen not as soon as the diffusion pump heaters were turned on, but a short time thereafter; the heater voltage was set at 55 V. From the graph we infer an equilibrium pressure of \( 4 \times 10^{-5} \) mm Hg and a fairly constant pumping rate \( S/C \approx 0.12 \text{ sec}^{-1} \). Due to the complicated tubing and other constrictions,

\[
\frac{dP}{dt} = -\frac{S}{C} (P - P_0) + \frac{Q_L}{C} \tag{4.1a}
\]

and has the solution

\[
\left[ P - \left( P_0 + \frac{Q_L}{S} \right) \right] = \left[ P_0 - \left( P_0 + \frac{Q_L}{S} \right) \right] \exp \left[ -\left( Q/L \right) t \right] \tag{4.2a}
\]

where again \( P_0 \) is the initial pressure. Thus the ultimate pressure is determined by the largest of \( Q_L/S \) or \( P_0 \), or by both. If the leak \( Q_L \) cannot be considered constant, but is a function of pressure, then also the determination of \( S \) is affected.

5. Radiation Safety and Handling of Radioactive Materials

5.1 Introduction

In a series of experiments on quantum physics, the student necessarily comes in contact with radioactive sources, either while studying the properties of the nucleus itself or when using the sources to obtain energetic beams of alpha or beta particles or gamma radiation. As is well known, radiation can be harmful to humans and therefore precautions must be taken against undue exposure to it, and in the handling of radioactive materials.

In addition to the naturally occurring radioisotopes (which have long lifetimes), a great variety have been produced artificially and many of them can be purchased. Table 4.2 is a listing of the radioactive materials primarily suggested for use in this laboratory; some of which, like Co\(^{60}\), Na\(^{22}\), and Po\(^{209}\), are quite standard for training, testing, and calibration purposes. The table gives the type and energy of the radiation, the half-life, the main decay scheme, and the maximum permissible burden in the body; in the last column a few remarks are given on the experiments for which the isotope is most useful.

† D. Statt, class of 1963.