HIGH VACUUM TECHNOLOGY -
How to operate and Maintain Vacuum Systems

by

Professor Lennie E N Lim

Course Objectives

Vacuum systems are widely used in Singapore, especially in the manufacturing industries. The benefits and applications of vacuum technology are obvious. Less obvious are how these systems operate and how they are maintained. This course, therefore, is designed to familiarise participants with the essential understanding of how to design, operate and maintain vacuum systems.

Course Content

The course will cover a wide range of topics in vacuum technology including:

- Gas Laws
- Viscous and Molecular Flows
- Outgassing, Diffusion, Evaporation, Real & Virtual Leaks
- Working Principles of Mechanical & Diffusion Pumps
- Mechanics of Gas Ballast
- Vacuum Instrumentation & Control of Vacuum Systems
- Getter and Cryo-Pumping
- Cleaning & Sealing Techniques in Vacuum
- Design & Fabrication of Vacuum Accessories
- Materials for Vacuum Application
- Trouble-shooting Procedures for Vacuum Problems

VACUUM TECHNOLOGY

I INTRODUCTION

Vacuum technology has its initial association with research in physics. It gains industrial prominence some 200 years ago when T.A. Edison invented the incandescent lamp which led to an exponential growth in the use of vacuum in industry. Today, vacuum technology is indispensable to many branches of comtemporary industry as well as research. The emergence of many manufacturing processes relying on a vacuum environment have increased the demands made on vacuum systems. Vacuum technology will, therefore, play an essential role in industry in the future.

I.1 What is Vacuum?

"Vacuum" is defined as a space entirely devoid of matter, or more specifically, space in which air and other gases are absent. Under such a condition, absolute vacuum is said to exist. An example is the intergalactic space. However, there is no absolute vacuum on and near earth. For practical reasons, therefore, vacuum on earth is defined as a space whose gas pressure is less than a prevailing pressure in an environment or, specifically, any pressure lower than the atmospheric pressure.

a. What is a gas?

A gas is made up of many small invisible particles called molecules. Gas molecules, unlike those of the liquid and solid phases, are not "bound" to one another, but are moving about rapidly in all directions and filling all space available to them.

b. What is Pressure?

The gas molecules travel rapidly at high speeds and collide frequently with each other and with the walls of the containing vessel. The striking and rebounding results in a "push" or force on all the surfaces with the vessel. The average force on a unit area is called the pressure, i.e.

$$\text{Pressure} = \frac{\text{Force}}{\text{Area}}$$

c. What is Atmospheric Pressure?

In a similar way, atmospheric air applies a pressure at the earth's surface by the continuous collision of gas molecules. Although the atmosphere is without walls, it is confined by the earth's land-sea surface from below and by the force of gravity that prevents its outward escape from above.

On the earth's surface, i.e. at sea level, the atmospheric pressure is typically 1013mbar (760 torr). As we move away from the surface, the pressure decreases with altitude as shown in Figure 1.1.

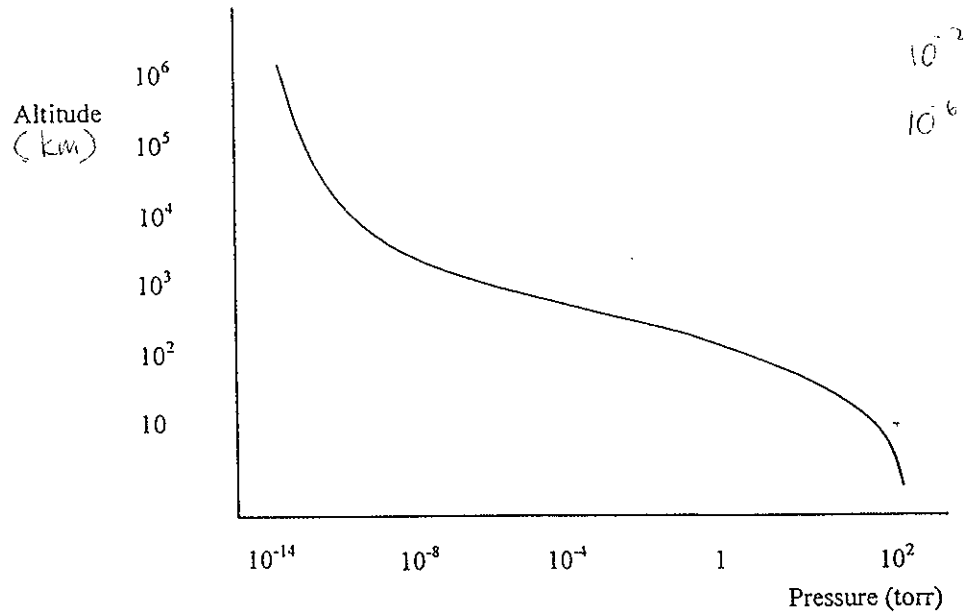

Figure 1.1 Variation of pressure with altitude

Up to 100km altitude the pressure decreases quite regularly by a factor of approximately 10 for each increase in altitude of 15km, which results in a pressure of 133mbar (10^{-3} torr) at about 90km altitude. At higher altitudes, high vacuum exists and at even higher altitudes, ultra high vacuum exists. This phenomenon is understandable as the gravitational force becomes weaker farther away from the earth's surface.

d. Gas mixtures and partial pressures

The gas in a vessel usually consists of a mixture of various gas constituent molecules. Each constituent exerts a pressure (known as partial pressure) which is proportionate to the number of its molecules present. The total pressure, P, of the gas mixture is the sum of all the partial pressures P_A, P_B, and P_c of gases A, B and C respectively. Mathematically,

$$P = P_A + P_B + P_c$$

Besides the use of partial pressure in the measurement of total low pressures, its use in the analysis of residual gases in vacuum systems is increasing as the attainable lowest pressure decreases. The instruments measuring the partial pressure of the residual gases are basically ionization gauges. The principle of operation consists of firstly ionising the gas molecules, then accelerating and finally separating into groups according to their masses. These mass spectrometer has proved to be a most useful instrument in the measurement of partial pressures below 1.33 x 10^{-4} mbar (10^{-4} torr), to about 10^{-15} mbar (7.5 x 10^{-14} torr).

e. Velocity of gas molecules

According to the kinetic theory, all molecules at the same temperature must have the same average kinetic energy. It follows, therefore, that a molecule with a higher mass will move more slowly than one with a lower mass. From the gas theory,

$$V = \sqrt{\frac{3RT}{M}}$$

Where V = velocity, cm/s
R = gas constant
T = absolute temperature
M = molecular weight

By this equation, the root-mean-square velocity of a gas may be calculated from directly measurable quantities. In doing this, R must be expressed in ergs per degree per mole, P in dynes per square centimeter, and the density in grams per cubic centimeter. With these units, V will be given in centimeters per second.

As an example, the velocity of hydrogen molecule at 0°C can be calculated with the following known as data: $R = 8.314 \times 10^7$ ergs per mole per degree, T = 273.16 and M = 2.016. Using the above equation:

$$V = \frac{3 \times 8.314 \times 10^7 \times 273.16}{2.016}$$

$$= 184.000 \text{ cm/s (or 1840 m/s)}$$

Since hydrogen is the lightest of all elements, this velocity represents the upper limit for molecular motion. (For velocities of other gases, see page 433 of Hanlon)

I.2 Units of Measurement

The most widely quoted parameter in vacuum technology is pressure. In the past, vacuum measurement units such as millimetres of mercury (mmHg) micrometres (or micron) of mercury (mmHg) and inches of water (in H_2O). However, they are no longer acceptable as units of pressure.

The derivation of the mmHg units was pioneered by an Italian called Evangelista Torricelli in 1643. In the experiment, Torricelli filled a long glass tube, with one end closed, with mercury and inverted it into a mercury reservoir as shown in Figure 1.2. He found that the mercury flowed out of the tube until the weight of the column was balanced by the pressure exerted on the surface of the mercury reservoir by the air. When the air pressure is atmospheric, the height of the mercury column was about 760mm. He also noted that when air pressure increased, the mercury column rose; conversely, when air pressure decreased, so did the mercury column.

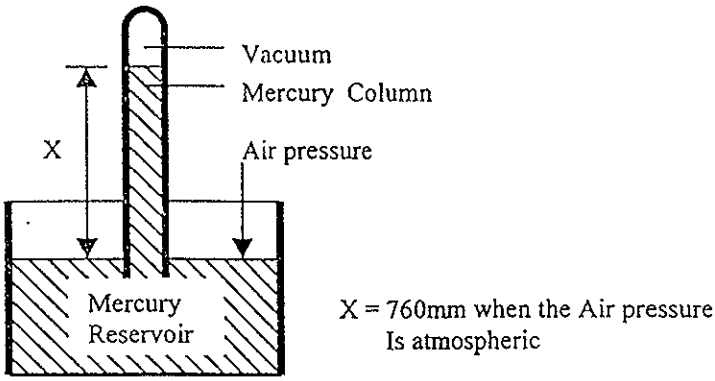

Figure 1.2 Mercury Column under Atmospheric Pressure

It was in honour of Torricelli that the mmHg unit was later replaced by torr, where

1 Torr = 1 mmHg

Therefore, the standard atmospheric pressure is also termed 760 torr. The standard metric unit of pressure under the Systeme International d'Unites (SI) is the newton per square metre (Nm^{-2}) which also takes the name of pascal (in honour of Frenchman, Blaise Pascal, who discovered the variation of pressure with altitude). It is to be noted that as from January 1978, torr has ceased to be an acceptable unit of pressure (although it is still widely used in the USA). The internationally acceptable unit is the pascal (Pa) and the millibar (mbar).

In view of the numerous units of measurement, it is useful to list their conversion factors using the millibar as common unit:

1 bar	=	1000 mbar
1 pascal	=	10^{-2} mbar
1 N/m^{-2}	=	10^{-2} mbar
1 torr	=	1.333 mbar
1 micron	=	1.333 mbar

I.3 Natural and Artificial Vacuum

On earth, vacuum exists naturally or artificially. Apart from absolute vacuum in intergalactic space, natural vacuum on earth uses "low vacuum" in some of the functions of life of insects, animals and human beings. An example of this occurs with our respiratory system. When we breathe, we expand our lungs and increase their volume. This produces a pressure inside which is slightly lower than in the outside air and permits

additional air to enter the lung. Another example is when using a drinking straw to drink liquid from a bottle. When we suck, our lungs expand and air passes into them from the straw. Atmospheric pressure pushing down on the surface of the liquid in the bottle is now greater than the pressure of the air in the straw and so forces the liquid up to our mouth. During respiration we are pumping to about 985 mbar (740 torr) of vacuum and we may achieve a vacuum of 400 mbar (300 torr) by suction. Other examples of natural vacuum found on earth are the respiratory systems of insects and animals as well as the suction systems of mosquitoes and octopuses. However, no natural high vacuum is found on earth.

High vacuum as well as ultra high vacuum can, on the other hand, be artificially created by pumping on a vessel, the degree of vacuum increasing as the pressure exerted by residual gas decreases below atmospheric. The artificial production of vacuum is discussed in more details in later chapters.

I.4 Degrees of Vacuum

On earth, the practical vacuum spectrum varys from atmospheric pressure of 1013mbar (760 torr) down to the lowest pressures or better than 10^{-10} mbar (10^{-13} torr). The degree to which the pressure is reduced is a function of application. For practical convenience, the vacuum spectrum is divided into several ranges to relate phenomena and processes to these ranges. The American Vacuum Society recommends the list of ranges as shown in Table 1.1.

Table 1.1 Vacuum Ranges

Degree of Vacuum	Pressure Range (mbar)
Low	$10^{5} > P > 3.3 \times 10^{3}$
Medium	$3 \times 10^{3} \geq P > 10^{-1}$
High	$10^{-1} \geq P > 10^{-4}$
Very High	$10^{-4} \geq P > 10^{-7}$
Ultrahigh	$10^{-7} \geq P > 10^{-10}$
Extreme Ultrahigh	$10^{-10} > P$

I.5 Applications of Vacuum Technology

Vacuum technology was originally a tool of laboratory research. In the 1600s pioneers such as Evangelista Torricelli and Blaise Pascal were active in vacuum research. Little progress was made until 1879 when T.A Edison used vacuum in the incandescent lamp. Thereafter entensive use of it was made in individual applications such as vacuum flask, cathode ray tube, vacuum diode and triode, tungsten filament lamp and many others.

With the increased applications, improvement in hardware also picked up in the 1900s and vacuum activities accelerated in 1936 when the diffusion pump was invented by K. Hickman. Subsequently, a high vacuum gauge using the cold cathode lonisation method was invented by F.M Penning and in 1950, R.T Bayard and D Alpert invented the ultra-high vacuum gauge. With the space exploration programme in the 1950s, further vacuum techniques with sophisticated monitoring and control gauges were introduced.

Today, high vacuum has become such a common research too that it would be difficult to find any sizable research laboratory not possessing a number of pieces of vacuum-producing equipment. The present uses of vacuum in research are too numerous and varied to categorise and will not be discussed in this book.

However, on account of the technological advantage and economic importance, some industrial applications will be discussed. These applications are mainly of two kinds. One is concerned with the production of products which are evacuated (e.g. vacuum flasks where vacuum is used as a thermal insulation) or having been evacuated of atmospheric air are refilled with another gas or mixture of gases (e.g. in gas-filled flashlamps or discharge lamps).

The other main application is to manufacturing or processing plants, in which vacuum is used to produce articles which in their final form are not evacuated. Examples include the vacuum-melted steel ingots and the vacuum-coated electronic micro-circuits. It is in the exponential growth in the micro-electronics industry that has given a new quantitative jump to vacuum technology, due to the numerous vacuum problems that had to be solved.

The application of vacuum is, therefore, very wide and Table 1.2 attempts to give only a list of some common examples of vacuum applications.

Table 1.2 Examples of Vacuum Applications and their Related Reasons

Degrees of Vacuum	Reasons for using Vacuum	Vacuum Applications
Low Vacuum	To achieve pressure difference	Vacuum Forming, e.g. Plastic Egg Trays
		Material Handling, e.g. Vacuum Cup in Sheet Glass Handling
		Holding, e.g. Vacuum Chuck
		Transport, e.g. Pnuematic Tube Transporter, Grain Transporter, Vacuum Cleaner and Chemical Filter.
Medium & High Vacuum	To remove active atmospheric constituents (e.g. Oxygen and water Vapour)	Packaging, e.g. Vacuum-packed Food
		Metallurgy, e.g. Casting and Sintering
		Leak Detection, e.g. Encapsulated Products
		Evacuation & Back-filling, e.g. Hermetic Compressor and Flash Tube
	To remove occluded or dissolved gas	Drying/Degassing,, e.g. Water Vapour & Oil Removal
		Impregnation, e.g. Vacuum Impregnation of Transformers and Motor Windings
		Distillation, e.g . Distilled Water
		Freeze Drying, e.g. Freeze-dried Coffee.
	To decrease energy transfer	Thermal Insulation, e.g. Vacuum Flask
		Electrical Insulation, e.g. Vacuum Switch
High & Very High Vacuum	To avoid particle collisions	Coating, e.g. Evaporation and Sputtering
		Electronic Tubes, e.g. TV Tubes, X-ray Tubes, Photo-Cells and Multipliers Mass Spectrometer
		Electron Beam (EB) Equipment, e.g. EB welding, EB Lithography and Microscopy
Ultra & Extreme Ultra High Vacuum	Clean Surfaces	Surface Research, e.g. Surface Friction and Adhesion

II PROPERTIES OF GASES

An understanding of the properties of gases at reduced pressure is essential in vacuum technology. It has been customary to specify the degrees of vacuum in pressure. Near the atmospheric end, the knowledge of pressure (or force per unit area) on the walls of the vacuum chamber is important in terms of structural design. However, in high vacuum, pressure is not the most important property of the rarified gas environment. Instead, the more significant parameters are the molecular density and in certain instances, the amount of absorbed gases on the surfaces. It is, therefore, useful to make a mental adjustment to think in terms of density rather than pressure. The following section outlines the physical concepts which form the basis for the understanding of the vacuum phenomena.

II.1 States of Matter

Matter consists of very large numbers of small but discrete particles called atoms. Atoms may combine together to form arrangements called molecules. In other words, all matter consists of some distribution of atoms and molecules. For example, two hydrogen (H) atoms may combine with one oxygen (O) atom to form a molecule of water (i.e. H_20).

Matter is normally classified as being in one of three states: solid, liquid or gaseous. Often, this classification is extended to include a fourth state referred to as a plasma. The fourth state of matter can occur when matter is heated to very high temperatures. Under these conditions, one or more electrons surrounding each atom are freed from the nucleus. The resulting substance is a collection of free electrically charged particles: the negatively charged electrons and the positively charged ions. Such an ionized gas with equal amount of positive and negative charges is called plasma. The plasma state exists inside stars, for example. There is far more matter in the plasma state than in the more familiar forms of solid, liquid and gas in our universe. However, this plasma state of matter shall be ignored and instead concentrate on the more familiar states of solid, liquid and gaseous that make up the environment on our planet.

a. The solid state

The atoms in a solid are bound at specific positions with respect to one another by interatomic forces. Heat energy contained in the solid appears as vibrations of the atoms about these positions. However, at low temperatures, this vibrating motion is slight and the atoms can be considered to be almost fixed. (In other words, the vibrating motion is not strong enough to break the bonds holding the atoms in place). As thermal energy (heat) is added to the solid, the amplitude of these vibration increases and eventually the bonds break, causing the solid to melt and form a liquid. It is to be noted that sublimation takes place for some substances. Sublimation, which is the basis for vacuum freeze drying, is discussed in later chapter.
From the above statement, a solid therefore has a definite volume and shape, e.g. a piece of ice.

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*Note: Sublimation is a process where the substance converts directly from the solid to the gaseous state without passing through the intermediate liquid phase

b. The liquid state

For any given substance, the liquid state exists at a higher temperature than the solid state. Thermal agitation, therefore is greater in the liquid state than in the solid state. As a result, the molecular forces in a liquid are not strong enough to keep the molecules in fixed positions, and the molecules wander through the liquid in random fashion. Because the atoms are still very close together, they are still influenced by interatomic binding forces, particularly at the surface. However, if heat is added to the liquid the random motions of the atoms become more vigorous, eventually reaching the point at which they move rapidly enough to overcome the binding forces. The liquid then begins to boil and becomes gas.

Unlike solids, liquid has a definite volume but no definite shape. It takes the shape of the container in which it is being contained. For example, when one fills the tank in a car, the gasoline assumes the shape of the tank in the vehicle, but if one have a litre of gasoline before filling, one will have a litre after.

c. The gaseous state

In a gas, the molecules are very much further apart than in solids or liquids. The average separation distances between the molecules of a gas are quite large compared with the dimensions of the molecules. Because they are further apart interatomic forces have very little effect on one another. The molecules therefore are in constant random motion, periodically colliding with one another and moving off in new directions. The average distance travelled by a molecule between collisions is termed mean free path (mfp)*. The molecules also collide with the walls of the containing vessel.

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*Note: Mean Free Path (mfp) is the average distance travelled by a molecule between collisions. Mfp depends on the size of the molecule and the number of molecules present or, in other words, density or pressure. For air at room temperature (20°C), the mfp can be found from

$$\text{Mfp} = \frac{6.5 \times 10^{-3}}{P} \text{ cm}$$

where P = pressure in mbar.

For other common gases, the mfp range of variation is about a factor of 10, i.e. instead of the 6.5 used in the expression for air, it should be 18.2 for helium, 12 for hydrogen and 4.2 for water vapour.

The mfp values are also proportional to the absolute temperature.

II.2 Vapours and Vapour pressure

The terms vapour and vapour pressure appear very often in a discussion on high-vacuum technology and their meaning should be clearly understood. Vapour does not have a precise meaning. On the one hand, if the temperature is relatively low, any gas can be termed vapour. On the other hand, if the temperature is relatively high, any vapour can be described as a gas.

The concept of vapour pressure can best be explained by the use of an example. If a liquid is placed in a sealed container which is maintained at a constant temperature, as shown in Figure 2.1, a certain pressure of its own vapour will be developed.

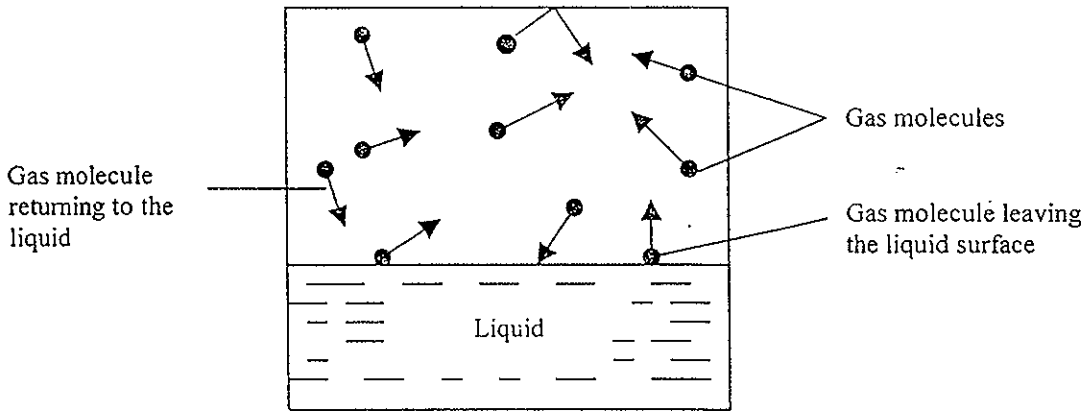

Figure 2.1 Saturated Vapour Pressure

In this example, some molecules have sufficient energy to leave the liquid surface (i.e. evaporate) to become a vapour (or gas) and be part of the surrounding space. On the other hand, the random motion of the gas molecules provides the means for some of them to collide with the liquid surface and be recaptured (i.e. condense). An equillibrium will eventually be reached between the evaporation and condensation and saturation is said to have taken place. The resulting pressure is known as the saturated vapour pressure. In other words, "saturated" and "equillibrium" mean that the number of molecules leaving the liquid surface and the number returning to the liquid are the same. For simplicity, the word "saturated" is usually omitted and vapour pressure is normally used.

Vapour pressure has a deep dependence on temperature. Raising the temperature of the liquid will increase the evaporation rate. Conversely, decreasing the temperature will reduce the evaporation rate with a corresponding decrease in vapour pressure. In vacuum practise, refrigerated surfaces are frequently used to cause vapours to condense on them and act as pumps. This topic is further dealt with under cryopumping.

The rate of evaporation can also be affected by varying the pressure of the container while holding the temperature constant. When the container pressure is reduced, the evaporation rate increases. This is due to the fact that molecules leaving the liquid surface have less chance of colliding with the gas molecules (as a consequence of lower pressure) and evaporation proceeds more rapidly.

In addition, the vapour pressure also depends strongly on the molecular weight of the substance. For example, the vapour pressure of water at room temperature (20°C) is about 23 mbar (17 torr) and that for some oils (whose molecular weight is higher) used in vacuum pumps is 10^{-1}mbar (7.5×10^{-9} torr). However, there are exceptions, e.g. water has a lower vapour pressure than alcohol, even though its molecular weight is lower.

a. Vapour pressure of liquids and solids

All materials, liquids as well as solids, have pressures. Table 2.1 lists the vapour pressures of some liquids at 20°C.

Table 2.1 Vapour pressure of various liquids at 20°C

Liquids	*Vapour pressure (mbar) At 20°C*
Organic liquids	
Acetone	250
Benzene	100
Carbon tetrachloride	120
Ethyl alcohol	60
Isopropyl alcohol	510
Toluene	30
Trichlorethylene	80
Xylene	7
Metals	
Mercury	1.5×10^{-3}

The vapour pressure of most solids is usually low. Most metals are suitable for vacuum applications. However, alloys that contain cadmium, lead and zinc, for example have unsuitably high vapour pressures and are therefore not recommended for vacuum applications. Steel screws which are cadmium-plated and brass (which has a content of zinc) components must not be used. Table 2.2 lists the vapour pressure versus temperature for certain metals.

Table 2.2 Saturated vapour pressure versus temperature of certain metals

	10^{-5} mbar	10^{-4} mbar	10^{-3} mbar
Aluminium	710°C	800°C	880°C
Cadmium	145°C	175°C	215°C
Zinc	205°C	245°C	285°C

II.3 Gas Laws

The behaviour of real gases approximates more or less closely, depending on conditions, to the ideal gas laws associated with the names of Boyle and Charles.

a. Boyle's Law

Robert Boyle, in 1662, confined within a graduated tube a quantity of gas and then measured the volume of that gas under different applied pressures. He found that the volume decreased with increasing pressure and that the volume of any definite quantity of gas at constant temperature varied inversely as the pressure on the gas. Expressed mathematically, this law states that at constant temperature $V = \frac{1}{P}$ or that $V = \frac{C}{P}$,

where V is the volume and P the pressure of the gas, while C is a proportionality constant whose value is dependent on the temperature, weight of gas, its nature, and the units in which P and V are expressed. Rearranging, the equation becomes

$$PV = C$$

If in a certain state the pressure and volume of the gas are P_1 and V_1, while in another state they are P_2 and V_2, then at constant temperature

$$P_1 V_1 = P_2 V_2$$

b. Charles' or Gay-Lussac Law

Charles, in 1787, observed that gases air, carbon dioxide, oxygen and hydrogen expanded an equal amount upon being heated at constant pressure. However, in 1802, Gay-Lussac made a quantitative study on the expansion of gases on heating. He discovered that for all gases, the volumetric increases for each degree centigrade rise in temperature was equal approximately to $\frac{1}{273.16}$ of the volume of the gas at temperature 0°C is designated V_0 and its volume at any temperature t°C by V, then the findings of Gay-Lussac may be written as

$$V = V_0 + \frac{t}{273.16} V_0$$

Rearranging the equation,

$$V = V_0 \left(\frac{273.16 + t}{273.16} \right)$$

If a new temperature scale T is now defined such that any temperature t on it will be given by

$$T = 273.16 + t,$$

And that at 0°C, $T_0 = 273.16$

Then

$$V = V_0 \frac{T}{T_0}$$

Or generally

$$\frac{V_1}{V_2} = \frac{T_1}{T_2}$$

c. The Combined Gas Law

The two laws discussed above give, separately, the variation of volume of a gas with the pressure and with the temperature. The laws may be combined to give the simultaneous variation of the volume with temperature and pressure. Mathematically, for a given amount of gas (i.e. given mass of gas) the pressure, volume and temperature have a definite relationship:

$$\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2} = \text{Constant}$$

From the equation, it follows that if the temperature is kept constant, the product of pressure and volume remains constant. This relationship is not exactly true through the entire temperature range and does not exactly hold for gases that have a complex molecular structure. For this reason, the law is called the perfect gas law. The law, however, is sufficient for the simplified calculations used in this book.

d. Expansion Ratios

If $1m^3$ of gas at atmospheric pressure and temperature is allowed to expand to an end pressure of 1mbar while the temperature is kept constant, the volume will be expanded to (using the Boyles' Law $P_1 V_1 = P_2 V_2$) $1000m^3$.

Extending this phenomenon to high vacuum, we have a summarised expansion ratios as shown in Table 2.3.

Table 2.3 Summary of Expansion Ratios

Vacuum (mbar)	Volume (m^3)
1,000	1
1	1,000
1×10^{-3}	1,000,000
1×10^{-6}	1,000,000,000

The concept of expansion ratio is important in the understanding of vacuum leaks. It becomes apparent from the expansion ratio, above, that if the continuous inleak of gas is $1 m^3$ per minute at atmospheric pressure, the pumping speed required to reach a pressure of 1×10^{-6} mbar is so large as to be thoroughly impractical.

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1. What is Vacuum?

American Vacuum Society defines "vacuum" as a given space filled with gas at pressure below atmospheric.

2. What is Atmospheric?

By definition, atmospheric is a given space filled with gas at a pressure of 760 torr.

3. How is vacuum achieved?

Vacuum is achieved by pumping on a vessel, the degree of vacuum increasing as the pressure exerted by the residual gas decreases below atmospheric.

4. UNITS OF MEASUREMENT

4.1 Torr is defined as that gas pressure which is able to balance at 0° a mercury column of height 1 mm, i.e.

$$1 \text{ torr} = 1 \text{ mmHg}$$

4.2 Micron, , is a small unit of measurement where

$$1 = 10^{-3} \text{ torr}$$

4.3 Bar is a bigger unit of measurement, where

$$1 \text{ bar} = 760 \text{ torr} = 1 \text{ atmos}.$$

4.4 Pascal, Pa is a smaller unit of measurement, where

$$1 \text{ Pa} = 7.6 \times 10^{-3} \text{ torr}$$

5. Degrees of Vacuum (American Vacuum Society)

Low Vacuum	$760 - 10^{-3}$ torr
Medium Vacuum	$10^{-3} - 10^{-5}$ torr
High Vacuum	$10^{-6} - 10^{-8}$ torr
Ultra High Vacuum	10^{-9} torr and below

6. Boyle's Gas Law

$$P_1V_1 = P_2V_2$$

7. Charle's Gas Law

$$\frac{V_1}{T_1} = \frac{V_2}{T_2}$$

8. Combined Gas Law

$$\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2}$$

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Low vac	760 to 10^{3} torr
High vac	10^{3} to 10^{6} torr.
Ultra High vac	lower than 10^{6} torr.

9. Expansion Ratio

If 1 m^3 of gas at atmospheric pressure is allowed to expand to an end pressure of 1 torr, the volume will be expanded to (using $P_1V_1 = P_2V_2$):

760 m^3

Extending this phenomenon to high vacuum, we have a summarised expansion as follows:

Vacuum (torr)	Volume (m^3)
760	1
1	760
10^{-3}	760,000
10^{-6}	760,000,000

10. What is a gas?

A gas is made up of many, small, invisible particles called molecules which are moving about rapidly in all directions. They move in straight lines randomly and at high speeds and colliding frequently with other molecules or with the walls of the container.

11. What is pressure?

Pressure is the average force on a unit area of the wall. The force is the reault of the molecules colliding with the containing walls.

Pressure of a gas depends on:-
1. how many molecules are striking the unit are and in any given time, and on
2. how fast they are moving

12. Pressures of gas mixture

The total pressure of a gas mixture, P, is the sum of all the partial pressures of the gases, P_1, P_2, P_3 ..... P_n, i.e.

$$P = P_1 + P_2 + P_3 + ... P_n$$

13. GAS FLOWS

3 basic flows:
a. Viscous
b. Transitional
c. Molecular

13.1 Viscous Flow

The criterion of viscous flow is:-
The mean free path (mfp) of the molecules is smaller than the diameter of the conducting tube.

13.2 Molecular Flow

In molecular flow, the molecules can move freely without mutual hindrance, which implies that the criterion for molecular flow is:

The mean free path (mfp) of the molecules is greater than the diameter of the conducting tube.

13.3 Transitional Flow

As the name impliers, it is a flow which is between viscous and molecular flows.

13.4 Gas Flows/Degrees of Vacuum

Gas Flow	Degrees of Vacuum
Viscous	Low Vacuum
Transitional	Medium Vacuum
Molecular	High Vacuum and higher

14. Pumping Speed

The pumping speed, S, at any point in a vacuum system is defined as the ratio of the throughput to the pressure at that point, i.e.

$$S = \frac{Q}{P}$$

where Q is the throughput and P is the pressure.

15. Net Pumping Speed

The net pumping speed, S_n, depends on the conductance, C, of the vacuum system and is defined as:

$$S_n = \frac{CS_p}{C + S_p}$$

where S is the pumping speed of the pump.

16. Pumpdown Time

Pumpdown time is defined as the time required to pump down a vacuum system from atmospheric pressure to a specified vacuum.

Pumpdown time has 2 components:

1. Pumpdown time in the Viscous range, T_v
2. Pumpdown time in the Molecular range, T_m

Typically, $\frac{T_m}{T_v} = 5 \text{ to } 10$

The high pumpdown time in the molecular range is due primarily to Outgassing.

17. Outgassing

Outgassing is the evolution of gas from materials in the vacuum system caused by the decrease in gas pressure. start at about 10^{-1} torr predominant at above 10^{-7} torr

18. Vacuum Pumps

In view of the wide ranges of vacuum, no single pump has yet been developed which is able to pump down a vessel from atmospheric pressure to the high vacuum (or ultra high vacuum) range.

This is due to the fact that different vacuum ranges are realised by different flow mechanism, i.e.

a. Low vacuum by viscous flow mechanism
b. High vacuum by molecular flow mechanism

19. Rotary Vane Pump

Rotary Vane pump is a Low pumping speed compression pump. It is available in single or double stage model. The significant difference between the 2 is that a double-stage pump will give a better vacuum. It will not have a significant improvement in pumping speed.

due to less back leakage

For higher pumping speeds, rotary piston pumps are used. For yet higher speeds, roots pumps are used.

19.1 Gas Ballast for Rotary Vane Pump

Gas ballast is a technique used to remove large quantities of condensable vapours.

Basically, gas ballast technique introduces a measured quantity of air into the compression phase of the pumping cycle in order that the pressure do not allow the vapours to condense.

Gas ballast is generally used when the vacuum vessel is opened to the atmosphere with high humidity, especially during maintenance service. Gas ballast is not required during production run.

20. Diffusion Pumps

Diffusion pumps work on the principles of a directed supersonic jet which entrain the random molecules towards exit of the pumping system.

A low fore pressure is vital to provide the necessary condition for supersonic flow of the diffusion jet.

The biggest problem with diffusion pump is that of backstreaming.

20.1 Backstreaming

Any transportation of the diffusion pump fluid into the vacuum system may be called backstreaming.

20.2 Causes of Backstreaming

These include

a. Over divergent flow of vapour from the rim of the diffusion pump nozzle.
b. Poorly sealed penetrations at the top of the nozzle cap.
c. Intercollisions of the vapour molecules in the upper layer of the vapour stream from the top nozzle.
d. Boiling of the returning codensate just before the entry into the boiler, which sends fluid droplets upward through the vapour jets.
e. Evaporation of condensed fluid from the wall.

20.3 Minimizing Backstreaming

a. Ensuring that all nozzlers are properly assembled according to manufacturer's specification. (Inclusive of heater and cooling coils).
b. Cold traps and Baffles can also be used to further prevent backstreaming. They invariably reduce the pumping speeds which can be of the order of 10%.

20.4 Diffusion Pump Oil

Diffusion pump oil is the moving force behind the pump. No other moving parts are used in the pump.

Basically, the oil must have the following properties:

a. Low vapour pressure (10^{-7} torr).
b. Good resistance to thermal decomposition.
c. High surface tension.
d. High flash point.
e. Cheap.

21. Getter Pumping

Sometimes called sublimation pumping, getter pumping involves a titanium source which is heated to sublimation temperature (i.e. from solid to vapour) at around 1500°C causing a highly active titanium film to be deposited on surrounding surfaces.

Active gas molecules impinging on this film react with the titanium forming stable solid compounds such as TiO_2 and TiN. By thus trasforming gaseous moleucles into solids, a highly effective vacuum pumping action is achieved.

21.1 Vacuum Pump in CPT

By incorporating getter pumping in CPT, it is equivalent to building a mini-vacuum pump in the CPT.

22. Vacuum System

Any vacuum system must have certain basic parts which are common to all systems regardless of pumping methods employed. These are:-

1. Leak-tight vacuum vessel (e.g. CPT)
2. Roughing pump (e.g. rotary vone pump)
3. Fine pumping system (e.g. diffusion pump)
4. Measurement System (e.g. pirani gauge)
5. Accessories (e.g. connectors and valves)

23. Outgassing

Outgassing is the generation of gas resulting from the desorption.

In practise, outgassing is usually acccmplished using heat.

23.1 Outgassing Rates

Outgassing rates are different for different materials, ranging from a relatively low outgassing rate for stainless steel to a relatively high outgassing rate for elastomers.

24. Measurement of Vacuum

Vacuum gauges are used to measure vacuum.

In the low vacuum regions, gauges used are that depend on the acutal force exerted by the gas.

At high vacuum, some specific property of gases and vapours, such as thermal conductivity, is used.

At even higher vacuum, the vacuum is measured by ionizing the gases.

24.1 Types of Gauges

These include:

a. Bourdon Gauges
b. Manometers (e.g. McLeod Gauge)
c. Pirani & Thermo Couple Gauges
d. Thermionic Ionization Gauges
e. Cold Cathode Ionisation Gauges

25. Leaks

Permissible leak rate will depend on the various degrees of vacuum, i.e. the higher the degree of vacuum, the lower the permissible leak rate.

Typically, for high vacuum, the permissible leak rate is 0.001 c.c./min.

26. Leak vs Outgassing

Leak is a result of inflow of air at a constant rate, whereas outgassing from the vessel walls and connecting pipes is a time-dependent phenomena, i.e. Outgassing will decrease with time.

27. Leak Detection

There are 2 methods of Leak Detection:

a. Pressure Method
b. Vacuum Method

Various of these methods are used and the most sensitive method is to use a search gas, such as halogen, or helium or freon.

28. Virtual Leaks

Virtual leaks are such leaks that cannot be detected by any of the external locatig techniques. A form of virtual leak is that of a trapped volume within the system caused by poor vacuum practises.

29. Industrial Applications of Vacuum

These include:

a. Vacuum Metalizing (or Vacuum Deposition)
b. Vacuum Sputtering Deposition
c. Vacuum Metallurgy
d. Vacuum Welding
e. Vacuum Melting
f. Vacuum Drying (Dehydration)

30. Vacuum Materials

All material have characteristic vapour pressures which vary with temperature.

If a vacuum system is to be capable of achieving the low pressure ranges in which we are interested, no material exposed to vacuum can have a vapour pressure such that if will volatilize at the expected temperature and vacuum.

30.1 Selection of Vacuum Materials

Other than vapour pressures, the selection of vacuum materials are based on the following considerations:

a. Mechanical strength of the material and its behaviour in the shape of the structure.
b. Thermal property of the material must also be known.
c. Corrosion resistance property if the system requires it.
d. Porous structure of the material surface is not acceptable as this gives a very large surface area for absorbed gases.
e. Cost of material.
f. Oxidation Property: Most metals oxidize on exposure to air. Some oxides are more stable than base metal (e.g. Ti and Al) and these oxides are acceptable. Other oxides develop unstable layers (e.g. Cu and Fe) and are not acceptable.

30.2 Glass in Vacuum

Glass is widely used in vacuum, especially the electronic discharge tubes.

Important points to note when working with glass are:

a. Coefficient of Expansion
Matching glass onto other material is a delicate task. It is vital that the coefficient of expansion of the mating material must be compatible.

b. Brittleness:
Glass is very brittle and it cannot withstand high mechanical shock.

c. Thermal Conductivity:
This is very small compared with metals. These low conductivity may cause cracking when heat is not distributed equally.

d. Electrical Resistivity:
As glass has a high resistivity (electrical), its use as an insulator is widely applied.

e. Permeability to gases:
Glasses have very low vapour pressures at room temperature. They are practically impervious to all gases except helium and hydrogen.

f. Optical Properties:
One of the main reasons for using glass in vacuum applications is its transparency, primarily in the viscous part of the spectrum.

g. Chemical Resistance of Glasses:
Glass is extremely inert to almost all types of usual corrosives with the exception of

i. HF
ii. Hot H_2SO_4
iii. Superheated Water

h. Degassing Glass:
The intrinsic vapour pressure of glass is extremely low. However, gases are tapped in the glass during preparation.

Most of the absorbed gases desorb around 150°C - 200°C. At about 450°C one does observe a slow outgassing. Therefore, to achieve a thorough outgassing of the glass in vacuum, the parts are usually heated to within a few tens of degrees from the softening point of the glass.

1. Express 1 torr in terms of

   a. μ ($10^{3}\mu$)

   b. bar (0.00132 bar)

   c. Pa (131.6 Pa)

   a. By definition: $1\mu = 10^{-3}$ torr

   Therefore 1 torr $= \frac{1}{10^{-3}}\ \mu$

   $= \underline{10^{3}\mu}$

   b. By definition: 1 bar = 760 torr

   Therefore 1 torr $= \frac{1}{760}$ bar

   $= \underline{0.00132 \text{ bar}}$

   c. By definition: 1 Pa = (7.6×10^{-3}) torr

   Therefore 1 torr $= \frac{1}{7.6 \times 10^{-3}}$ Pa

   $= \underline{131.6 \text{ Pa}}$

2. Express 10^{-3} torr in terms of

   a. μ (1μ)

   b. bar (0.00000132 bar)

   c. Pa (0.132 Pa)

   a. By definition: $1\mu = 10^{-3}$ torr

   Therefore 10^{-3} torr $= \underline{1\mu}$

   b. By definition: 1 bar = 760 torr

   Therefore 10^{-3} torr $= \frac{1}{760} \times 10^{-3}$ torr

   $= \underline{0.00000132 \text{ bar}}$

   c. By definition: 1 Pa = (7.6×10^{-3}) torr

   Therefore 10^{-3} torr $= \frac{10^{-3}}{7.6 \times 10^{-3}}$

   $= \underline{0.132 \text{ Pa}}$

.../2

3. Express 10^{-6} torr in terms of

   a. μ (0.001μ)

   b. bar (0.0000000013 bar)

   c. Pa (0.000132 Pa)

   Using the same analysis as 1 and 2 , the answers to the above can be found.

4. How many Pa = 1 bar? (100,000 Pa)

   By definition, 1 bar = 760 torr

   $$\text{and } 1 \text{ Pa} = (7.6 \times 10^{-3}) \text{ torr}$$

   $$\text{Therefore } 1 \text{ bar} = \frac{760}{7.6 \times 10^{-3}} \text{ torr}$$

   $$= \underline{100{,}000 \text{ Pa}}$$

5. What is the temperature of a gas when 1 m^3 at 25°C is expanded to 2 m^3? (Assuming Constant Pressure)

   25°C = (273 + 25)°K = 298°K

   Using $\frac{V_1}{T_1} = \frac{V_2}{T_2}$,

   $$T_2 = \frac{V_2}{V_1} \times T_1$$

   $$= \frac{2}{1} \times 298 \text{ °K}$$

   $$= 506\text{°K}$$

   $$= (596 - 273)\text{°C}$$

   $$= \underline{323\text{°C}}$$

6. What will be the end pressure of the vessel if it is heated up by 100°C and the volume increased by 10%. Assume the initial pressure to be 100 Pa and temperature to be 50°C.

   $V_2 = 1.1V_1$ (10% increase in volume)

   $T_1 = 50\text{°C} = (273 + 50)\text{°K} = 323\text{°K}$

   $T_2 = 150\text{°C} = (273 + 150)\text{°K} = 423\text{°K}$

   $P_1 = 100$ Pa

Using $\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2}$

$$P_2 = \frac{P_1V_1T_2}{T_1V_2}$$

$$= \frac{100 \times V_1 \times 423}{323 \times 1.1V_1}$$

$$= \underline{119 \text{ Pa}}$$

7. A gas occupies a volume of 200 l at a pressure of 10^{-4} torr and a temperature of 20°C. If the pressure is increased to 10^{-3} torr and the temperature raised to 50°C. What will be the volume of the gas?

$V_1 = 200$ l

$P_1 = 10^{-4}$ torr

$T_1 = 20°C = (273 + 20)°K = 293°K$

$P_2 = 10^{-3}$ torr

$T_2 = 50°C = (273 + 50)°K = 323°K$

Using $\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2}$

$$V_2 = \frac{P_1V_1T_2}{T_1P_2}$$

$$= \frac{10^{-4} \times 200 \times 323}{10^{-3} \times 293}$$

$$= 22 \text{ l}$$

8. A gas has a volume of 1200 cc, a temperature of 0°C and a pressure of 100μ. If the volume changes to 2000 cc and the pressure becomes 50μ, what will be the temperature of the gas?

$V_1 = 1200$ cc

$T_1 = 0°C = 273°K$

$P_1 = 100\mu$

$V_2 = 2000$ cc

$P_2 = 50\mu$

Using $\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2}$

$$T_2 = \frac{P_2V_2T_1}{P_1V_1}$$

$$= \frac{50 \times 2000 \times 273}{1200 \times 100}$$

$$= 227.5°K$$

$$= (227.5 - 273)°C$$

$$= -45.5°C$$

7.5. Rules for operating vacuum systems

(1) When starting a mechanical pump, insure first that the rotor moves in the correct direction, and that the level of the oil is correct.

(2) Always vent a mechanical pump to atmospheric pressure when the power is turned off. The presence of a residual vacuum in the pump frequently will cause oil to be drawn into the casing or the system.

(3) Do not permit a mechanical pump to exhaust a high-vacuum system below a pressure of a few hundred microns unless the pump is separated from the high-vacuum chamber with a trap stopping the pump oil vapours from entering the chamber. At lower pressures, the flow is molecular, thus the oil vapour expands towards the chamber, yielding an excessively high vapour pressure hydrocarbon contamination which may require many hours to remove.

(4) Do not run a mechanical pump at excessively high pressures for continuous periods. The motors are usually not sized for such runs. The pump will eject oil together with the gas.

(5) A diffusion pump should be cooled to a safe intermediate temperature before it is vented to atmosphere. Venting at too high a temperature results in oxidation of the pump fluid and an excessive carryover of the fluid into the mechanical pump.

(6) Check that the cooling water supply for the diffusion pump is turned on prior to the heating. Diffusion pumps should be provided with a thermal protection device to turn the diffusion pump heating off in the event of loss or failure of the cooling water supply.

(7) In liquid-nitrogen trapped systems, the trap should be cool enough to condense the diffusion pump oil prior to turning on the diffusion pump. Maximum backstreaming of the pump oil occurs during the startup and shutdown of the pump.

(8) Do not vent a liquid nitrogen trap to air while cold. Remove all liquid water condensate from the reservoir of a liquid nitrogen trap prior to use, to avoid water freeze-up in the trap. On the initial pumpdown, it is advisable to partially fill the liquid nitrogen trap, so that the principal vacuum system condensables can be trapped on the lower portion of the trap. After high vacuum has been reached, the trap should be filled to a higher level. A liquid nitrogen trap which has elastomer seals at the top should not be overfilled, as the freezing of the gasket may cause leakage.

(9) When the chamber is vented to atmospheric pressure for a short period of time, it is advisable to use a dry, inert gas such as nitrogen to minimize the moisture adsorption on the surfaces of the vacuum system. Venting should always be on the chamber side, never vent the system from the foreline of the diffusion pump.

(10) Ionization gauges (hot cathode) should not be turned on until it is reasonably certain that the pressure is below 10^{-3} Torr.

15

Guide to the safe use of vacuum equipment

In the past many reported incidents and mishaps in the vacuum industry have occurred because the user did not have a thorough understanding of the correct use of the product. This may have occurred through a lack of training about operating procedures or through a failure to comprehend what might happen under certain operating conditions.

A little forethought would have saved the sight of an operator who unfortunately exposed a capsule dial gauge to an extremely high pressure (the glass front ruptured). It would have saved the life of the maintenance worker who was unintentionally trapped in a large vacuum chamber which was then evacuated.

In one reported incident, the oil in a diffusion pump was being removed during maintenance, prior to putting in a new charge of oil. The technique unwisely adopted was to heat the oil at atmospheric pressure in the isolated pump, using the pump's own heater. The objective of heating was apparently to thin the oil to make subsequent draining on release of the drain plug easier. In this case when the drain plug was removed, the hot oil came out, evidently under some pressure and almost at once it ignited. It was found that there had been no control of the time or rate of heating of the oil which must have been at an unnecessarily high temperature for some time.

In another similar incident, a minor explosion (due to ignition of the oil) occurred when a vapour booster was restarted without the atmospheric air first being pumped out of the booster by the rotary pump.

In general, systems to be operated by unskilled personnel should be fully automatic and as foolproof as possible with the number of operations to be carried out by the operator kept to a minimum. With regard to maintenance, correct procedures must be laid down and strictly adhered to.

The following notes are intended as a general guide to some of the precautions necessary for ensuring the safe installation, use and maintenance of some vacuum products.

15.1 General precautions

General safety

1. For safety it is essential that all products are installed, operated and maintained in accordance with the instructions supplied with them.
2. Unless specifically supplied for such use, most products are *not* suitable for use outdoors or in the presence of flammable atmospheres, or in any location for which special safety regulations apply or special precautions are necessary.
3. Vacuum systems do not provide protection against toxic, biologically or chemically hazardous materials that may be processed therein or be evolved as a result of processes that may be carried out under vacuum. It is necessary for the user to provide appropriate safeguards.

Electrical safety

For safe electrical operation of equipment connected to the mains it is essential that the following requirements are met:

1. The electrical installation of the building in which the equipment is installed and all fittings used for mains connection must conform to the Institution of Electrical Engineers Regulations for the Electrical Equipment of Buildings or, if outside the United Kingdom, to the International Electrotechnical Commissions Recommendations.
2. The equipment must be connected to an electrical supply of the correct voltage.
3. The protective earth conductor must be properly connected and good earth continuity maintained at all times.
4. Only fuses and protective devices of the specified ratings and types must be fitted.
5. All electrical equipment must be disconnected from the supply before modifications, repairs or maintenance are undertaken.

Damaged equipment

Equipment that is damaged or is possibly damaged should not be used until properly repaired and its safe operation ensured.

15.2 Hazards associated with particular equipment

Vacuum measuring instruments

General

1. Most vacuum gauges are designed to measure pressures lower than atmospheric pressure and should not be subjected to pressures significantly above atmospheric pressure.

2. Vacuum systems, which may be subjected to high overpressure in the event of malfunction, should incorporate suitable pressure relief devices.

McLeod gauges

1. These gauges are filled with mercury, which is a toxic substance and should be used and handled with great care.
2. The gauges are of glass and to avoid the risk of damage should only be used with the plastic cover that they are normally supplied with in position.

Ionization gauges

Cold and hot cathode types The heads of these gauges operate at a high voltage. Connection and disconnection of the gauge head from the supply should only be done with the gauge supply disconnected from the mains.

Hot cathode type These gauges operate with an incandescent filament in the vacuum system. Flammable or explosive gas mixtures must not come into contact with the filament, otherwise an explosion could occur. (This also applies to Pirani gauges, although the sensing filament is at a much lower temperature.) The external surfaces of the gauge heads can reach high temperatures and suitable shrouding should be provided if accidental contact is likely.

During degassing procedures there is intense light emission from glass gauge heads and dark goggles should be worn if it is necessary to view the gauge head. Screening should be arranged to protect passers-by. Gauge heads of the glass type should have connecting leads supported to prevent stress on the glass.

The electrodes of these gauges often have exposed multiconnection pins. Do ensure that common electrodes are properly electrically insulated.

Mechanical vacuum pumps

Electrical wiring

1. Ensure correct direction of rotation (three-phase motor).
2. Thermal overload cut-out switches for motors should be used.

Contact with moving parts

1. Guards are provided to prevent accidental contact with moving parts and pumps must never be operated with these guards removed.
2. Mechanical booster pumps should not be run in such a way that human contact with the rotating impellers is possible.

Oil mist hazard
Rotary pumps are oil sealed and discharge small quantities of oil mist when operating. In poorly ventilated areas this can lead to unacceptable concentrations of oil vapour. Oil vapour should be kept to a minimum. It is strongly recommended that a suitable oil mist filter is fitted to the pump or, alternatively, the exhaust should be piped away externally.

Exhaust hazard
Any dangerous substances which may be present in the system being evacuated may pass through the system into the pump and be emitted at the exhaust. Suitable precautions must be taken.

Dangerous substances
Hazardous situations will occur if pumps are used to pump flammable or corrosive substances, unless the pumps have been supplied specifically for the particular application.

Hazardous overpressure
1. Hazardous pressures may be produced if the exhaust from a mechanical pump is restricted or blocked, and care should be taken at all times to prevent this.
2. Exhaust line manifold systems should be designed to cope with the maximum exhaust load that can occur.
3. Hazardous pressures may be produced in the system to be evacuated if the rotary pump is operated in the reverse sense. When a rotary pump motor requires a three-phase electrical supply, the outfit should be checked for correct rotation after mains connection, before being connected to the system to be evacuated.

Incorrect mounting
Hazardous situations will occur due to the mass of the pump, if it is not supported by a structure of sufficient strength, or if the pump is not properly secured, using antivibration mountings where appropriate.

Suction applications
When mechanical pumps are used to provide suction for medical purposes, precautions must be taken to ensure that the suction and exhaust connections cannot be accidentally reversed.

Mechanical pump accessories

Traps, filters and condensers
If any hazardous material accumulates in a trap or filter then a hazard exists when the content of the trap is removed and during disposal of the trapped material.

Foreline traps

1. These traps use activated alumina pellets to trap oil vapour and thus prevent migration of pump oil to other parts of the vacuum system.
2. Activated alumina is a desiccant and inhalation of its dust, eye contact and unnecessary skin contact should be avoided.
3. If reactivation of the alumina is attempted by baking, then any other materials that have been absorbed will be released and suitable precautions will be necessary.

Inlet desiccant traps

1. These traps use phosphorus pentoxide (P_2O_5) as a desiccant.
2. It is highly caustic and reacts violently with water, evolving heat and forming a mild acid. Handle carefully to avoid creating a dust. Avoid inhaling dust or fumes.
3. Rubber gloves and eye protection should be worn when handling P_2O_5, especially when washing trays of partially spent P_2O_5.
4. P_2O_5 attacks aluminium; spillage on the body of the trap should therefore be avoided.
5. If any is spilt on the skin or clothing it should be washed off with plenty of water.
6. Full health and safety information on P_2O_5 should be obtained from the supplier.

Inlet chemical traps

1. These traps use activated charcoal to protect the pump from heavy lacquer and chemically active vapours and gases.
2. Precautions appropriate to the trapped materials should be taken when removing or disposing of the spent charcoal.

Oil mist filters

1. These filters are intended to remove oil mist from rotary pump exhaust. They should not be relied on to remove any other hazardous material.
2. They must not be allowed to become blocked, as hazardous overpressures could then occur.

Diffusion and vapour booster pumps

Hot surfaces and heat hazards

1. The external surfaces of vapour diffusion and booster pumps reach high temperatures. Electric cables in close proximity to these pumps must have insulating properties compatible with the temperatures reached.
2. These pumps should not stand on wooden floors or on surfaces of heat-sensitive or combustible material.
3. They should be installed in such a position that accidental bodily contact

with the hot surfaces is prevented, and adequate room should be allowed around the pump boiler for air circulation to take place.

Hot vapour hazards

1. If a hot vapour pump is opened to the atmosphere, vapour will be released, and could be a health hazard.
2. Fluid should not be drained from pumps or the pumps opened to atmosphere before they are cool. Heating oils at atmospheric pressures can cause them to decompose, burn or explode.

Cooling water failure

Lack of cooling water can cause pump overheating and steam generation in the cooling lines.

Mercury pump hazards

1. Mercury vapour pumps have the additional hazards associated with mercury. Mercury poisoning is cumulative and causes irreversible brain damage and fatal kidney damage. Do not expose mercury to the atmosphere at elevated temperatures (>23 °C) since the vapour pressure increases rapidly with temperature with a corresponding increase in the hazards.
2. Mercury vapour pumps should only be fitted to vacuum systems designed to be resistant to mercury attack.
3. Any spillage must be immediately cleaned, by mechanical means, e.g. vacuum probe, followed by conversion of the remaining residue into mercury sulphide, so eliminating the danger of vaporization.
4. Mercury must be contained in tightly stoppered containers, preferably unbreakable.

Fomblin fluids

Fomblin fluids produce toxic breakdown products when heated above 280 °C.

Vapour traps and baffles

Condensate hazard

Condensible vapours present in a vacuum system will condense and accumulate on the cold surfaces of vapour traps. The material will then re-evaporate when the trap or baffle warms up. If the material is hazardous, suitable precautions should be taken.

Hazards of liquid nitrogen cooled traps and baffles

1. Only equipment designed for the purpose should be used for transferring liquid nitrogen from its storage container to the trap.

2. Skin contact with liquid nitrogen or surfaces at liquid nitrogen temperature will result in cold burns, and protective clothing should be worn where there is such a risk.
3. Spillage of liquid should be avoided by using suitable filling devices. Full-face protection should be worn where and when there is a risk of liquid splashing.
4. Filling funnels and open dewars should always be kept below shoulder level when filling traps.
5. Traps intended to be charged with liquid nitrogen should not be charged with other substances. In particular, they should never be charged with liquid air or liquid oxygen which could create a fire/explosion hazard.
6. The reservoirs of liquid nitrogen traps must always allow the escape of evaporating nitrogen gas; otherwise there will be a dangerous buildup of internal pressure and eventual explosion. The filling tube should only be covered with the ventilated cap provided and should never be blocked with, for example, a bung.
7. The filling spouts of cold traps should not be positioned where water can drip on them as this could cause the spouts to become blocked with ice.
8. Evaporated nitrogen can displace air and in confined spaces could possibly lead to asphyxia.
9. Cold surfaces at liquid nitrogen temperatures can condense certain permanent gases such as CO_2, xenon and argon. Dangerous pressures could be produced by a trap that has condensed quantities of such gases if it subsequently warms up on a sealed system. If such gases are passed through the trap it should be evacuated while it is warming up.
10. The cold surface of liquid nitrogen traps should not be exposed to the atmosphere while cold, as oxygen from the air can be condensed with the consequent fire/explosion hazards.

Turbomolecular pumps

The pumps are not normally designed to run at or near atmospheric pressure and certainly should not be run or rotated with the inlet exposed at atmospheric pressure without a shield on the inlet.

Cryopumps (see additionally Section 9.6)

1. Cryopumps can condense and accumulate all atmospheric gases and vapours.
2. If hazardous materials are present in the vacuum system and are retained by the cryopump, suitable precautions must be taken when the pump is warmed up.
3. A pressure relief valve is provided to relieve overpressure in the event of the cryopump warming up when the system is sealed. A dangerous

situation could occur if this valve failed to operate or if it operated when hazardous materials were present in the pump.
4. Cryopumps should not be operated when open to atmosphere.

Sputter-ion pumps

High-voltage and magnetic hazards

1. These devices operate from a high-voltage supply.
2. Connection and disconnection of the pump and power supply should only be undertaken with the power supply disconnected from the mains.
3. It is recommended that an additional protective earth conductor is connected to the pump body.
4. Powerful magnets form part of these pumps. Any equipment that is sensitive to a magnetic field should be kept away from them and if the magnets are removed care should be taken to avoid trapping fingers between the magnet and any ferromagnetic material or other magnets.

Fluids

Pump oils

1. Mineral-oil-based products are only slightly to moderately irritating to the skin and eyes. Prolonged exposure of the skin to mineral oils may give rise to dermatitis.
2. Care should, however, be taken to avoid inhalation of vapours or mists arising from undue heating or excessive mist generation. In the case of fluorinated compounds (e.g. Fomblin) avoid contact with excessive heat ($\geqslant 280\,^{\circ}C$), e.g. lighted cigarettes, heater elements, etc.
3. Storage. Normally these products require no special fire precautions but it is recommended practice to store away from heat. When heat is required to facilitate handling of the product, this should be kept to a minimum.
4. Fluid spillage should be absorbed with sand, earth or mineral absorbent and disposed of in the proper manner. In the event of large spillages, steps should be taken to prevent pollution of drainage systems, rivers or waterways.
5. Where oxygen or aggressive materials have to be pumped, an inert and chemically resistant lubricant must be considered. Suitable members of fully fluorinated oils can be used.
6. The operating temperature of most vapour pump fluids is of the order of 195 to 220 °C. This constitutes a safe operating condition in a vacuum environment. However, a hazardous condition with some fluids may occur if the fluid is permitted to overheat excessively by allowing it to operate at or near atmospheric pressure. Under these conditions, the fluid temperature may approach the autoignition point (depending on the fluid, in the range from 300 to 600 °C).

Solvents

Trichloroethane is the preferred cleaning solvent; it has a low toxicity and is non-flammable. However, in common with all highly volatile oil and grease solvents, the main hazard is the narcotic, anaesthetic and toxic effects of breathing concentrations of vapour resulting from its use in confined spaces. Ensure there is adequate ventilation and rate of air change in the cleaning area. Smoking is prohibited. Avoid skin contact.

Pipeline valves

Pneumatic operation

1. Care should be taken to avoid trapping the fingers in pneumatically operated valve mechanisms.
2. Air pressures in excess of the operating range specified must not be applied to the inlet of pneumatic mechanisms.
3. The compressed air supply used should incorporate suitable pressure controllers and relief valves.

Electrical leadthroughs

General

1. These leadthroughs are normally provided with functional insulation only. Any necessary enclosure or insulation to protect against electric shock must be additionally provided.
2. Avoid hanging heavy wire leads from electrical leadthroughs as localized stress may cause a fracture.

Leak detection equipment

High-frequency spark testers

1. These units generate a high-frequency, high-voltage spark from an accessible unshielded probe.
2. They can ignite flammable material and should not therefore be used where flammable material is present.
3. Bodily contact with the spark should be avoided at all times and the spark testers should only be used by responsible trained personnel.
4. A serious electric shock is unlikely to be received if the probe is accidentally touched while operating, but injury could occur if the spark reached sensitive parts of the body (e.g. eyes). There could be a risk to persons suffering from heart weakness.
5. Injury could also occur as a result of the reflex movement after spark contact if the tester is used in the vicinity of other hazards, such as hot surfaces or rotating machinery.

6. Spark testers are used to test evacuated glass systems. Suitable eye protection must be worn during testing.

Vacuum systems and process plant

Hoists

Hydraulically operated hoists can become hazardous due to incorrect maintenance or damage. They should have periodic checks for loss of hydraulic fluid and for jerky operation, which is an indication of air leakage into the hydraulic system.

Exposure of human body to vacuum

1. No part of the human body should be exposed to vacuum.
2. Evacuation of the whole body will result in death and the exposure of small areas of the body surface to the suction of the pumping system can result in tissue damage.
3. Where large vacuum chambers have to be entered by personnel for loading, cleaning or maintenance reasons, procedures must be designed to prevent accidental evacuation of the workchamber with personnel inside.

Excess pressure hazard when backfilling

1. Unless otherwise stated vacuum systems are not intended to be raised to a pressure greater than that of the external atmosphere, and a hazardous situation will arise if a chamber is accidentally put under positive pressure.
2. Processes sometimes require a vacuum chamber to be filled with a gas other than atmospheric air. If the source of this gas is a pressure cylinder then pressure-reducing devices and safety valves should be incorporated in the line to avoid pressurizing the vacuum system.

Implosion hazards

1. Glass components are normally designed with a large safety factor. Failure is normally due to flaws in the material or through misuse. Examples of misuse include where the glass:
   (a) is knocked (by a handtool, etc.),
   (b) is not properly supported (e.g. when opening/closing glass valves),
   (c) has localized hotspots or cold areas due to the process,
   (d) is constrained by metal clamping.
2. Glass vacuum chambers are provided with implosion guards to contain any flying debris resulting from implosion. Such chambers should not be evacuated without the implosion guard in position. Wear safety glasses.
3. Glass vacuum chambers should always be checked for damage that might weaken them and cause implosion.

Cleaning hazards

1. Suitable precautions should be taken to protect personnel engaged in cleaning vacuum systems from the solvents being used and from process debris (dust) in the system. This particularly applies to personnel who enter large vacuum chambers to clean them.
2. The exact precautions to be taken will be dependent on the cleaning solvent involved but particular attention should be paid to the danger of inhaling solvent vapours (see 'Solvents', page 285).

Evaporation sources

1. Precautions should be taken to protect the eyes of both users and passers-by from the intense light emission of hot evaporation sources used in coating units.
2. When it is necessary to view a hot evaporation source, dark safety goggles should be worn.

Vacuum coating—deposits

The deposit on the chamber walls of a vacuum coating system are generally in the form of extremely fine particles. The nature, as well as the form of the materials, poses the following potential hazards:

1. Inhaling fine particles (powder) may cause damage to the lungs. Wear a protective respirator mask with a fine filter to help prevent this.
2. Some substances are toxic and inhaling them should be avoided. Take steps to ascertain whether or not the material being deposited is a known toxic substance.
3. Certain powders, titanium, for instance, and unoxidized aluminium particles, can cause flash fires when exposed to oxygen or other oxidizers. Therefore, when opening the chamber door after a deposition cycle, exercise extreme caution and allow time for the coating surface to oxidize. Breakage of some of the deposits of the more reactive condensates may still be hazardous, even with the above precautions. In this situation fire-protective clothing should be worn.

LT sources

1. Evaporation sources are supplied from low-voltage power supplies capable of delivering high currents in normal use.
2. Accidental short circuiting of these supplies is a serious hazard.
3. Cables carrying such currents must be protected from mechanical damage.

High-voltage supplies

1. Some vacuum processes involve the supply of high voltage to the vacuum system for HT discharge cleaning, electron gun use, etc.
2. The HT supplies should be interlocked with switches on the vacuum

system to prevent high voltage being available when the workchamber is open at atmospheric pressure.

Chamber air admittance hazards

1. Air admission to vacuum systems should always be carried out in a controlled manner, to avoid excessive disturbance of the surrounding atmosphere.
2. Large quarter-swing valves should not be used as air admittance valves unless a suitable flow restrictor is fitted. Too rapid air admission can cause violent pressure reductions in the room and violent air movement, with consequent risk of injury.
3. Large vacuum chambers should not be placed in small inadequately ventilated rooms where air admittance could cause a fall in room pressure.

Rotary workholders

To facilitate loading, rotary workholders can be operated when accessible, and care is necessary to avoid loose clothing or parts of the body being trapped by moving parts.

6. Hints for the Operation of Vacuum Apparatus

6.1. Causes of Failure in not Attaining or Attaining Too Late the Required Ultimate Pressure

If, in vacuum apparatus, the required ultimate pressure is not reached or only reached after a much too long pumping time, the following faults can be responsible.

If **the required ultimate pressure is not attained,**

the apparatus is leaky or dirty,
the pump is dirty or damaged,
the vacuum gauge is defective.

If **the required ultimate pressure is only attained after a very long time,**

the apparatus is dirty,
the pump connection is too narrow,
the pump is dirty or too small in size,

or the pumping speed of the pump is reduced by other causes.

To find the faults, the usual procedure is to, if possible, separate the evacuated chamber from the pumping system and to investigate this for leakage and contamination, e.g. by the pressure-rise method. If this shows that the chamber is free of faults in this respect, the next thing is to check the vacuum gauge system for cleanliness (see Section 6.4) and finally — if necessary — the pump or the pumping system itself is investigated.

6.2. Contamination of Vacuum Systems and Its Elimination

In addition to the pressure-rise method (Section 5.1) there is a method of determining contamination, which depends on the fact that condensable vapours form the chief fraction of the gas mixture in dirty systems; it is to compare the pressure reading of a compression gauge with that of an electrical vacuum gauge (thermal conductivity or ionisation gauge). These two vacuum gauges must, of course, be clean themselves. In the presence of vapours, the compression vacuum gauge indicates a much better (lower) pressure than the electrical vacuum gauge. This is a sure sign of contaminated chamber walls, mostly by oil.

A very useful procedure is further to compare the pressure readings of one and the same vacuum gauge (not a compression vacuum gauge) with and without an inserted cold trap: on filling the cold trap with liquid nitrogen, the pressure indication with dirty chambers falls by a power of ten or more, because the vapours are frozen out in the trap.

Cleaning glass apparatus

If the source of the contamination has a high vapour pressure, it can be pumped away relatively quickly. If that does not succeed, the apparatus must be cleaned. Dirty glass parts are first cleaned with chromosulfuric acid or — in case it is necessary — with diluted hydrofluoric acid (1 : 30). Finally they are washed in running distilled water. If this procedure is not successful, an organic solvent can be tried. For this, the glass parts must be washed again with methyl alcohol and subsequently with distilled water (methylated spirit is not to be used!).

Cleaning metal apparatus

Metal parts exhibit mostly traces of contamination due to greases or oils. If these are not quickly removed by pumping, they must be cleaned with an organic solvent, such as carbon tetrachloride (methylated spirit is unsuitable in all cases). Greater cleanliness is achieved by vapour baths, as are usual in industry, e.g. with trichlorethylene as the vapour. Solvents containing chlorine impair leak detection, if they are not completely pumped away (see Section 5.311)! If low pressures are to be obtained, the metal parts must be degassed after the cleaning: up to 200° C if below 10^{-7} torr is to be obtained.

Coarse dirty metal parts must first be cleaned mechanically. Sandblasting or polishing will clean the surface. These methods have, however, the disadvantage that the treated surface area is increased by roughening and eventually active centres can form, which readily absorb vapour molecules. An additional cleaning in a vapour bath (see above) is recommended. Often, also, electrolytic etching of the surface can be an advantage; with high vacuum components it must be ensured that the etching does not cause corrosion which will increase the surface area greatly. For rough and medium vacuum purposes, polishing of the sand-blasted surface is not necessary, since the size of the surface area in these pressure regions plays only an insignificant role.

6.3. General Hints for the Operation of Vacuum Pumps

If fault-finding does not show faults in the chamber and in the vacuum gauges or the apparatus does not work satisfactorily after eliminating the faults, the flange seals on the pump side and eventually the isolation valve should be tested next.

Flange seals are well-known as locations at which light scratches and slight mechanical imperfections can most readily cause leakage. If also here no fault is found, it is recommended to check whether the pumps are working according to the operating instructions.

In this section are given next some general hints about the maintenance of pumps, in order to avoid such faults. Furthermore, possible faults which arise and their causes are described.

6.31. Gas-ballast Pumps

6.311. Oil consumption, oil contamination, oil changing

The oil-filling of a gas-ballast pump has to fulfil various functions it serves

a) to lubricate the moving parts,

b) to seal the moving parts against the atmospheric pressure,

c) to seal the valve,

d) to fill the dead space under the valve.

With all gas-ballast pumps, the oil-filling can be inspected during the operation through built-in oil-level glasses. Above all during long-term operation, it must be ensured that the oil-filling does not sink below the minimum value. During a pumping process, oil-sealed rotary pumps expel oil vapours from their discharge ports because of their high working temperature. This leads to a loss of oil which is dependent on the amount of pumped gas or vapour. The oil thus expelled from the pump can be captured in a trap installed on the outlet side and hence used again. With some new rotary vane pumps, this physically caused oil loss is considerably reduced by the insertion of a filter cartridge in the outlet discharge port. The oil vapour is thereby maintained in the body of the pump itself; the condensed oil returns after filtering into the working volume of the pump.

The **oil consumption** of the oil-sealed rotary pumps is different according to the size of the pump and the working process. For each atm m^3 of air (including pumped gas-ballast air) pumped is estimated an oil consumption of about 2 cm^3 in the unfavourable cases of the large rotary piston pumps. For the smaller types of pump, particularly rotary vane pumps, the oil consumption is about a half as much. Rotary vane pumps with a built-in filter cartridge have an extraordinarily small oil consumption (about 0.1 cm^3 of oil per pumped atm m^3). For example, if one wants to estimate, without and with gas-ballast, the oil-loss of a rotary piston pump of type S 800 with a constant inlet pressure of 1 torr, one should reckon with 2 cm^3 per pumped atm m^3. The pumping speed of the S 800 at 1 torr amounts to about 600 $m^3 \cdot h^{-1}$, the pumped throughput is therefore about 0.8 atm $m^3 \cdot h^{-1}$. If the pump works without gas-ballast, an oil consumption of 1.6 cm^3 per hour is estimated. If the pump works with full gas-ballast, it draws in through the gas-ballast valve about 1/10 of the quantity of air that it pumps in at atmospheric pressure at the inlet port, therefore about 80 atm $m^3 \cdot h^{-1}$. With a fully opened gas-ballast valve, an oil consumption of 160 cm^3 (plus 1.6 cm^3) per hour is therefore calculated. For a quick evaluation of the oil loss expected in a pumping process, serves Fig. 61.2 whereby an oil-loss of 2 cm^3 per pumped-in atm m^3, has been taken into account. For rotary vane and small rotary piston pumps, the values obtained from the diagram can be halved. For rotary vane pumps with an inserted filter cartridge, only a tenth of this value is taken. If the pump oil becomes useless because of vapours or impurities which contaminate it during the working process, it must be renewed. The first oil change should, in any case, be made after 100 hours of operation; a binding statement cannot be made as to when the subsequent oil changes are to be made, because the time for the oil to become useless depends on the operating conditions. Under clean conditions (e.g. backing pumps to diffusion pumps on particle accelerators) rotary pumps can run for years without oil change. Under extremely unclean conditions (e.g. in impregnation) it can be necessary to change the oil every day. An oil change should be undertaken if the lubrication capability of the oil falls (finger probe), or if the oil is turbid because water has reached the pump, or if an anti-corrosion oil shows by the formation of flakes that the corrosion-resistance medium is used up.

Undertaking an oil change

With closed pressure port and opened pump inlet port, the oil is drawn off from the oil drain opening. The new oil is poured in through the pump inlet port. For strong contamination, cleaning can be undertaken with 1/3 oil and 2/3 benzine, carbon tetrachloride or similar solvent, according to the kind of contamination. To draw off the cleaning medium the pump is rotated a few times by hand. Subsequently, it must be flushed several times with clean oil.

Note! If the pump has been operated with noxious vapours or water vapour has condensed in it, an oil change should always be immediately carried out after use and undertaken on the still warm pump! Marked corrosion sets in if the pump is left standing for a long time in a contaminated state.

6.312. Choice of the pump oil for pumping active vapours

If corrosive vapours (e.g. vapours from acids) are to be pumped, an anti-corrosion oil (PROTELEN® or KS 20) should be used in place of the normal pump oil (N 62). Such vapours react with the anti-corrosion constituent of the oil. Due to the continuous subsequent neutralisation reactions, the anticorrosion constituent is used up to an extent depending on the quantity and acid content of the vapours. Correspondingly, the oil must be changed frequently. A test of the usefulness of the anti-corrosion oil can be undertaken with the aid of litmus paper or better with pH paper. If the red litmus paper no longer turns blue (pH value of 7 to 8) the anti-corrosion oil should be renewed.

Anti-corrosion oils are either very hygroscopic or form emulsions with water. A gas-ballast pump filled with anti-corrosion oil therefore takes up water from the atmospheric moisture if it has not been in operation for a long time.

On no account should water vapour be pumped through a gas-ballast pump filled with anti-corrosion oil, because the lubrication properties and the corrosion resistance of the oil will become impaired. Moreover, gas-ballast pumps which have taken-up water in the oil filling can no longer reach the same ultimate pressures as are obtained with clean anti-corrosion oil or with normal pump oil (N 62). Under normal operating conditions, gas-ballast pumps should not be operated with an anti-corrosion oil; for the pumping of air, water vapour and non-corrosive organic vapours, the oil N 62 is preferred, so long as care is taken that the vapours cannot condense in the pump.

6.313. Measures for pumping various chemical materials

Basically, for the pumping of all the materials presented here, it is true to say that they are far better pumped in the dry state than in the moist state. The discharge outlet pipes of the gas-ballast pumps must lead always to the open air (for exceptions, see below).

a) Acids

Fundamentally, concentrated, corrosive acids such as HNO_3, HCl, H_2SO_4 and H_3PO_4 should not be pumped with gas-ballast pumps, because they attack the pump housing. Acetic acid and carbonic acid as well as other acids in diluted form can, however, be pumped with the use of an anti-corrosion oil.

b) Anhydrides

CO (Carbon monoxide) is a strong reducing agent. On pumping CO, therefore, air should not be used for the gas-ballast, but, in all cases, argon or nitrogen. Also SO_2, SO_3 and H_2S should be pumped with a protective gas-ballast. Moreover, for these three anhydrides an anti-corrosion oil is used. The pumping of CO_2 (carbon dioxide) is carried out without special precautions.

c) Alkaline solutions

For the pumping of alkaline solutions, the normal pump oil N 62 is used. Caustic soda and caustic potash solutions in the concentrated forms should not be pumped. Ammonia can be pumped well with a closed gas-ballast. Organic-based media such as methylamine and dimethylamine are likewise readily pumped, though with an opened gas-ballast valve.

d) Gaseous elements

The pumping of nitrogen and of noble gases requires no special measures.

For hydrogen, the gas-ballast valve should on no account be opened.

The motors of the pumps should be protected against explosion. Furthermore, the discharge outlet pipe must lead into a space where the hydrogen cannot react with oxygen.

Particular care is needed for the pumping of pure oxygen! The special pump oils S 30 or T 80 should be used. With N 62, on approaching the flash point, strong oxidation would occur which leads to explosive burning of the oil.

e) Paraffins

Ether, propane and butane can be pumped satisfactorily with the gas-ballast valve closed, methane with a protective gas (argon) for the gas-ballast. With methane, on account of the rapid reaction with air (firedamp) care is necessary. The discharge outlet pipe must therefore be led to a chamber in which methane cannot react with air.

f) Alcohols

After reaching the operating temperature, methyl and ethyl alcohol can be pumped without gas-ballast (pump oil N 62). For pumping of the higher alcohols (butyl alcohol and others) the gas-ballast valve is opened. The insertion of a low temperature cold trap in which the alcohol condenses is also advantageous.

g) Solvents

Acetone: pumped without difficulty. Wait till operating temperature is reached.

Benzol: Care: vapours are inflammable. Spoils the ultimate pressure by dissolving in the pump oil. Air-benzol mixtures are explosive and inflammable. Work without gas-ballast! If necessary, argon as gas-ballast.

Carbon tetracholoride and trichlorethylene: satisfactorily pumped; not inflammable and not explosive, but oil solvents and thus spoil the ultimate pressure; wait till operating temperature is reached. On pumping of carbon tetrachloride and other non-inflammable solvents, the gas-ballast valve is kept open. Pump oil N 62.

Toluene: pump over a low temperature baffle, without gas-ballast. Use nitrogen or argon as gas-ballast, not air.

6.314. Faults in the operation of gas-ballast pumps

Possible sources of failure in not reaching the required ultimate pressure.

a) The pump oil (especially due to dissolved vapours) is contaminated. The pump oil is tested for viscosity (finger test!), colour and quality.

Remedies: let pump run for a long time with the vessel isolated and the gas-ballast valve open. In cases of marked contamination, an oil change is recommended (p. 61). The pump should not be left standing for long times with dirty oil.

b) The sliding parts of the pump are worn or damaged. Under clean conditions our pumps can run for years without special maintenance. If, however, the pump is run for long periods with contaminated oil, the bearings and the slides often exhibit mechanical damage. This is always to be expected if the pump no longer reaches the catalogued ultimate pressure despite an oil change. In this case the pump needs repair or our service department is informed.

c) The vacuum gauge is contaminated (see Section 6.4).

Possible faults if the pump will not rotate.

a) Air was not admitted to the pump after switching-off. Admit air:

b) The pump has been left standing for long periods with dirty or resinated oil.

c) The pump seizes up during operation because it becomes too hot.

Remedies: for b) and c): the pump in the cold condition (allow the hot pump to cool) is flushed with a solvent (as described above). If this is not succesful, it is recommended in this case to get the pump overhauled.

d) The pump is too cold, the oil is stiff. Warm the pump and rotate carefully.

Escape of oil at the axle.

If oil escapes at the axle, the shaft seal in the drive bearing must be suspected and, in some cases, renewed. In this case, first the pulley is taken off with the help of a special tool, the cotter-pin is removed and the shaft seal is withdrawn. The drive shaft is renovated at the rear end and has a sharp step on which the new shaft seal can easily be damaged on installation. Hence the shaft seal must be put on the shaft with the aid of a shaft installing sleeve, so that it is not damaged.

6.32. Vapour Diffusion and Vapour Ejector Pumps

6.321. Change of pump fluid and cleaning of the pump

With oil-diffusion and oil-ejector pumps, the pump fluid is endangered by the too frequent admission of air to the hot pump. For comparatively short-term air inlets (up to 1 minute) up to atmospheric pressure, silicone oil is hardly attacked and the pump fluid DIFFELEN only slightly attacked. The products with sufficiently high vapour pressure which arise through oxidation are removed again in a short time by the fractionating and degassing arrangement in the pump (see Section 2.141). Even if the originally light pump fluid is turned brown by air admission, it is not necessarily made useless; of course, if it has become turbid and more viscous, which can be the case after an air inrush of several minutes, a change of pump fluid is necessary. In some circumstances, due to cracking products from the pump fluid, the backing pump oil can become unserviceable; in this case also a change of that oil must be undertaken.

Mercury-diffusion and mercury vapour-ejector pumps are less sensitive to exposure to atmospheric air pressure than oil-diffusion pumps. The oxidation caused by the admission of air to the hot mercury is negligible in comparison with the maintenance of the operation of the pump due to loss of mercury to the backing-pump line.

A **change of pump fluid** is always necessary if the pump no longer reaches the required ultimate pressure or its pumping speed falls off. The stability of the pump fluid amounts, as a rule, to weeks, to months, even years and depends greatly on the operating conditions of the pump. It is reduced by frequent pumping at high pressures, the pumping of noxious vapours and by long-lasting admission of air (this is not so for silicone oil and mercury).

Undertaking a change of pump fluid: the jet system of the pump is withdrawn and the contaminated pump fluid is poured out; then the jet system and the pump body are cleaned by a wash with residue-free cleaning spirit. The jet system and the pump body of mercury pumps should previously be cleaned with a clean paint-brush, and the nozzle apertures with a fine bottle brush. Care must be taken that all the nozzle openings are well cleaned. It is an advantage to evaporate off the remaining solvent in a drying oven. Then the fresh pump fluid is poured in through the backing port and the jet system is re-inserted. Care is needed to ensure that the upper nozzle cone is not wetted by the pump fluid. Do not fill with too much pump fluid!

6.322. Operating faults with diffusion and ejector pumps

Possible sources of failure in **not reaching the required ultimate pressure**

a) The cooling-water temperature is too high; the water circulation is insufficient. The cooling water flow should be regularly tested by a water flow monitor to protect the pump against damage.

Remedies: measure the exit temperature of the cooling water (it should not exceed 30° C). Increase the cooling water flow. If necessary, free the cooling water coils of lime deposits.

b) The backing pressure is too high: this is especially possible if vapours are either pumped or arise as cracking products of the pump fluid (e.g. on air admission) and reach the backing pump. The backing-pressure is checked with the oil diffusion pump isolated.

Remedies: operate the backing-pump for a long time with gas-ballast.. If this does not succeed, the oil in the backing-pump must be changed.

c) Pump fluid of the diffusion pump is used up or is unuseable: change the pump fluid.

d) The heating is wrong: check the heating supply, furthermore, test for a good thermal contact between the hot-plate and the base of the pump boiler. If necessary, change the heating device.

e) Leakage, contamination.

Remedies: if the pump has become contaminated by vapours, it can help if some air is admitted for some time through a variable-leak valve into the apparatus; in this case, if DIFFELEN is used, the pressure should not climb above 10^{-2} torr.

f) The vacuum gauge has aged or has become contaminated (see Section 6.4).

Possible causes of insufficient pumping speed

a) The backing pressure is too high: check the backing pressure, run the gas-ballast pump for a long time with the gas-ballast valve open. In some cases, a change of backing pump oil is required.

b) Pump fluid of the diffusion pump has become unuseable: a change of pump fluid is necessary.

c) Nozzles of the diffusion pump are blocked up: clean the diffusion pump.

d) Heating is too weak: check the heating power, test for good thermal contact between the base of the pump boiler and the hot-plate.

e) Substances are present in the vessel which have a higher vapour pressure than the pump fluid used. The case of mercury is to be noted here as being particularly hazardous because the mercury vapour forms amalgams with the non-ferrous metals of the oil-diffusion pump and hence makes impossible the attainment of satisfactory vacua. Therefore, the pressure provided by oil-diffusion pumps should only be measured with mercury compression vacuum gauges if cold traps are inserted and care is taken by the use of a valve that, when the trap is being warmed up, mercury vapour cannot get into the pump.

With mercury diffusion pumps, take notice of deposition of mercury on the backing-vacuum side.

Possible causes:

a) No backing condenser attached: this is needed, otherwise the backing pump is damaged and, moreover, mercury vapour can get into the working space of the backing pump.

b) The nozzle assembly is not correctly seated on the bottom of the pump.

c. The cooling is too weak: the exit temperature of the cooling water should not exceed 15° C. Check the cooling water temperature; if necessary, increase the cooling water flow. In every case a water flow monitor must be inserted.

d) Leakage on the high vacuum side.

Mercury deposits on the high vacuum side.
Possible faults; cooling is too weak.

6.4. Operation of Vacuum Gauges

6.41. Hints concerning the attachment of the vacuum gauge head

Here the external conditions in the immediate neighbourhood of the vacuum apparatus as well as the operating conditions of the apparatus (e.g. working pressure, composition of the gas content) are decisive. First of all care must be taken if the gauge head to be mounted is sensitive to its location. Many gauge heads ought only to be erected in a vertical position, to avoid condensate collecting in the gauge head or heated filaments bending unsatisfactorily and electric short-circuits occurring inside the gauge head.

It is further very important as far as possible to mount gauges at such locations that they are free of vibration during operation.

The external temperature must be considered; above all, it must be avoided that hot ovens or other sources of intensive radiation produce at the gauge an ambient temperature which lies above permissible limits. Too high a temperature in the neighbourhood leads in the case of compression vacuum gauges and thermal conductivity vacuum gauge tubes to false pressure readings.

6.42. Contamination of the Gauge Head and Its Elimination

The vacuum gauges used in vacuum technology for pressure measurement work generally under unclean conditions. This is understandable because a vacuum apparatus or plant is not used simply to produce low pressures but, in the first place, to permit some kind of chemical, metallurgical or nuclear process to take place. Hence, depending on the kind of process, either continuously or intermittently sometimes considerable quantities of gases or vapours are freed, which reach the vacuum gauge attached to the vacuum system and due to surface reactions or simply due to accumulation can significantly falsify the pressure measurement. This is true for all kinds of vacuum gauge, but gauges of high sensitivity and accuracy are especially susceptible to contamination due to the stated causes. One can try to protect the gauge from contamination by suitable shields; this often leads, however, to the fact that the pressure measured with the clean gauge is considerably different from the pressure which is actually wanted to be determined.

Basically, the contamination of the vacuum gauge head cannot be avoided. Therefore, care must be taken that:

a) the influence of the contamination on the pressure measurement is as small as possible,

b) the gauge head can be cleaned easily.

In practice, for most vacuum gauges, these two conditions are not easy to fulfil.

Contamination of compression vacuum gauges causes a too high, uncontrollable pressure reading; contaminated THERMOTRON tubes show, in the lower measuring region, too high a pressure, because the surface of the hot filament has changed. With the Penning vacuum gauges, contamination results in much too low a pressure reading, because the discharge current is smaller. In ionisation vacuum gauges with hot cathodes, the electrodes and tube wall can become contaminated, which in some circumstances, causes a decrease of the insulation resistance. In this case, however, the gauge head can usually be baked and the electrodes degassed by the passage of current or electron bombardment, quite apart from the fact that ionisation gauges are frequently used for ultrahigh vacuum, where, on other grounds, clean conditions must be preserved.

6.43. Influence of Magnetic and Electric Fields

For all vacuum gauges which make use in the measuring principle of the probability of ionisation of gas molecules (cold-cathode and hot-cathode ionisation gauges), strong stray magnetic fields or extraneous electrodes at a positive voltage, can alter considerably the pressure reading. At low pressures, wall potentials, which differ from the cathode potential, can influence the collector current.

For vacuum gauges which are installed in high- and ultrahigh-vacuum, special care must be taken, therefore, that the high insulation of the high voltage electrodes and ion collector is maintained during operation, indeed, often during the bake-out. Faults in insulation can be due as much to the external leads as to the gauge head itself.

If the collector lead is not adequately insulated, leakage currents — particularly at low pressures — can simulate too high a pressure value. Because of the smallness of the collector currents, the lead must therefore be especially well insulated. Also within the gauge head leakage currents can occur if the collector is not effectively shielded relative to the other electrodes.

A mistake frequently committed in the connection of vacuum gauges to the vacuum system is the use of unsatisfactorily long and narrow connecting tubes. **Their conductances must in every case be kept as large as possible.** (This condition is fulfilled, of course, if a nude gauge head is used). Depending on the cleanliness of the gauge head and connecting tube, the use of connecting tubes of small conductance can make the pressure reading either too high or too low. In this case, errors of measurement of about an order of magnitude or more are observed. With bakeable plant care must be taken, therefore, that the connecting tube is also baked out.

6.44. Electrical Connection of Control Unit to the Gauge Head

The cables which connect the gauge head to the gauge control unit are usually 2 m long. If, perhaps in the installation of control panels, longer cables are used, it is necessary to find out if the pressure reading is thereby falsified.

Data on the possibilities of use of long cables can be obtained from our technical advisory department.