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INTERNATIONAL CENTRE FOR SCIENCE AND HIGH TECHNOLOGY

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EXPERIMENTAL WORKSHOP ON
HIGH TEMPERATURE SUPERCONDUCTORS AND RELATED MATERIALS
(BASIC ACTIVITIES)

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" Fundamentals of vacuum technique "

presented by:

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These are preliminary lecture notes, intended only for distribution to participants.

States of matter

Matter consists of very large numbers of small, discrete particles known as atoms. Atoms may combine together in chemical reactions to form arrangements termed molecules. For example, two hydrogen atoms may combine with one oxygen atom to make a molecule of water. Certain atoms—those of the so-called 'inert' gases such as helium, neon and argon—are very unlikely to combine chemically, and the atoms remain single and unattached. The atoms of other gases such as hydrogen, nitrogen and oxygen rarely exist as single atoms, and form diatomic gas molecules, i.e. molecules of two identical atoms. For simplicity it can be considered that all matter can be divided into three states, or phases, these being the solid state, the liquid state and the gaseous state. Some matter exists in each of these states: e.g. ice, water and steam are three states of the same substance. There are solid, liquid and gaseous forms of carbon dioxide etc.

The solid state

In a solid, the atoms are bound tightly together in fixed positions relative to each other by interatomic forces. Solids therefore have fixed volumes. Heat energy contained in the solid appears as vibrations of the atoms about these positions. Normally these vibrations are not strong enough to break the bonds holding the atoms in place. If heat is supplied to the solid, the vibrations increase in strength and eventually the bonds break, causing the solid to melt and form a liquid. A few substances will convert directly from the solid to the vapour without passing through the intermediate liquid stage. The effect is known as sublimation and is the basis of vacuum freeze drying.

The liquid state

In a liquid, the atoms (or molecules) have no fixed positions, and are in constant random motion, wandering freely about the bulk of the liquid.

Because the atoms are still very close together, they are still influenced by interatomic binding forces, particularly at the surface. If heat is added to the liquid, the random motions of the atoms become more vigorous, eventually reaching the point at which they move quickly enough to overcome the binding forces. The liquid then boils and becomes a gas.

The gas phase

In a gas, the molecules are on average very much further apart than in solids or liquids. For example, in air at atmospheric pressure and room temperature, about 0.01 per cent of the space is actually occupied by the molecules. In a solid such as copper, the figure is 74 per cent. In a gas, the molecules are constantly moving in random directions, but because they are further apart interatomic forces have very little effect. At very short intervals, individual molecules collide with and bounce off others (about 10 000 000 000 times per second for each molecule at atmospheric pressure). A gas thus automatically expands to fill a volume into which it is led.

Vapours and saturated vapour pressure

Some molecules have sufficient energy to escape from the surface of bulk solids and liquids and become part of the surrounding atmosphere. Raising the temperature of the bulk substance will increase the rate of escape. A dish of water, for example, if left uncovered in the open air, will evaporate; vapour molecules rapidly diffuse away from the parent liquid and in general produce what is known as an unsaturated vapour. On the other hand, if the liquid is in an enclosed vessel there is a high probability that a free molecule will collide with the liquid surface and be recaptured (i.e. condensation). Thus, an equilibrium will be established between evaporation and condensation and the air is unable to accept any more water; it has become saturated and the pressure it exerts is called the saturated vapour pressure. Since the rate of evaporation falls with a decrease in temperature, the saturated vapour pressure also decreases. Refrigerated surfaces are frequently built into vacuum systems to cause vapours to condense on them and act as pumps. This is known as 'cold trapping' or 'cryopumping'.

A saturated vapour is one in equilibrium with its liquid.

In vacuum, the rate of evaporation increases. Molecules leaving the surface have less chance of colliding with air molecules, and evaporation proceeds more rapidly. If the pressure is progressively reduced from atmospheric, the rate of evaporation gradually increases until, at a certain value of pressure, evaporation becomes much more rapid—for our example of a dish of water, the water begins to boil at room temperature (20°C). This occurs at a pressure of 23.4 mbar.

As long as any water exists at room temperature anywhere in a vacuum system, the minimum pressure attainable in the system will be 23.4 mbar (the saturated vapour pressure). A little water can produce very large volumes of vapour, and hence can take a long time to be pumped away. Note that rapid evaporation tends to result in a lowering of the temperature of the water (unless sufficient heat is available), and it may freeze. At 0°C the saturated vapour pressure of ice is 6.11 mbar.

The same process applies to any liquid inside the vacuum system. The implication of this is that great care must be taken to avoid contaminating a system (e.g. by careless handling (fingerprints) or unclean work (fluxes, greases, cleaning fluids, etc.)) with possible vapour sources.

All materials, including solids, have vapour pressures, although some are very low at normal temperatures and others may be a problem for a vacuum system.

Table 2 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}

Materials having a noticeable vapour pressure at the highest intended operating temperature must obviously be avoided. The vapour pressure of most metals is so low that it does not restrict their use for vacuum applications. However, alloys that contain zinc, lead and cadmium, for example, have unsuitably high vapour pressures for vacuum application. Cadmium is commonly used to plate steel screws and brass has a zinc content. Table 2 shows the vapour pressure of 'difficult' metals at various temperatures. Vapour evolved from the hot metals is likely to condense on adjacent cooler surfaces. A potential problem can be a metal film condensed on an electrical insulator, thus causing a short circuit.

Table 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

Quantities involved in the definition of the "characteristics" of a gas

temperature T (K) kelvin

pressure P (Pa) pascal

volume V (m^3)

number of molecules or atoms n

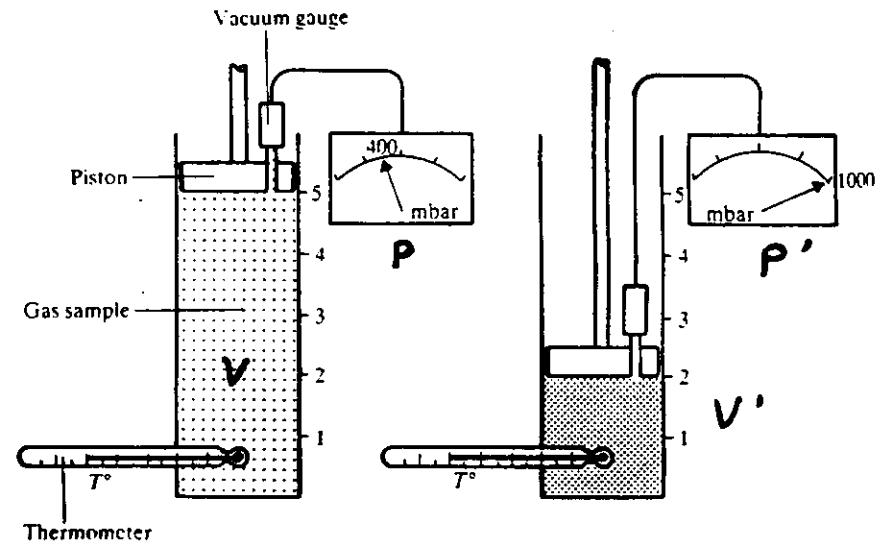
SI system of units
International System

Boyle's law

If a given mass of permanent gas is confined in a cylinder by an airtight piston and the volume of the gas is decreased by the movement of the piston, the pressure of the gas increases. When the volume of the gas increases the pressure decreases. Gases are therefore compressible. Robert Boyle investigated the relationship between gas pressure (p) and volume (V) and deduced his law.

Boyle's law states that for a fixed mass of gas at a given temperature, the product of the pressure and volume is constant; i.e. pV is constant.

Figure illustrates the effects of Boyle's law. A graduated cylinder is fitted with a piston, pressure gauge and thermometer.

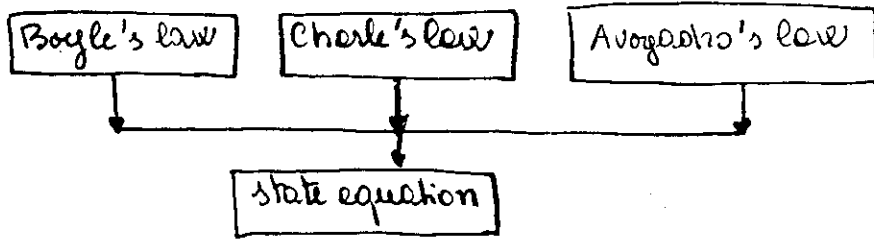


(a)

(b) Gas sample compressed

An illustration of Boyle's law

Perfect gas equation



state equation \equiv equation indicating the behaviour of the variables, volume (V), temperature (T), pressure (P) and number of molecules (n) per unit volume when gas changes from one state to the other

Boyle's law $\rightarrow V$ inversely proportional to P
 $V \propto \frac{1}{P}$ "with T and n constant"

Charles's law $\rightarrow V$ directly proportional to T
 $V \propto T$ "with P and n constant"

Avogadro's law $\rightarrow V$ directly proportional to n
 $V \propto n$ "with T and P constant"

general $\rightarrow V \propto \left(\frac{1}{P}\right)(T)(n) \Rightarrow V = R \left(\frac{1}{P}\right)(T)(n)$

state equation of perfect gases $\Rightarrow PV = nRT$

$R =$ gas constant
 $R = 8.31 \text{ J } \text{K}^{-1} \text{ mol}^{-1}$
 $R = 8.315 \text{ K}^{-1} \text{ mol}^{-1}$

2 real gases
 $PV = nRT \left(1 + \frac{B(T)}{V} + \frac{C(T)}{V^2} + \frac{D(T)}{V^3} + \dots \right)$ A, B, C virial coeff. \equiv

The most characteristic property of a gas is the tendency to expand indefinitely or, when enclosed in a container, to produce a positive pressure on the container walls.

Each "portion" of a gas exerts a pressure on the adjacent "portions" of the same gas.

$\partial S =$ infinitesimal imaginary surface

$\partial F_n =$ sum of the normal components of all forces acting on the surface element ∂S ;

Pressure will then be:

$$\partial P = \frac{\partial F_n}{\partial S}$$

if ∂S becomes an infinitesimal portion of the container surface ∂P will be

The pressure exerted by the gas on the walls of the container

with reference to an elementary part of an imaginary surface, pressure can be computed as a momentum transfer between contiguous portions of gas.

$$P = \frac{F}{S} \quad P_2 = \frac{M}{m^3}$$

$$1 \text{ mbar} = 100 \text{ Pa}$$

Gas mixtures—partial pressures

The gas in a vacuum chamber usually consists of a mixture of various constituent molecules. Simply, each gas constituent can be considered to exert a pressure appropriate to the number of its molecules present. The total pressure of the mixture will be equal to the sum of the partial pressures of the mixture:

$$P_{\text{total}} = P_1 + P_2 + P_3 + \dots \text{ etc.}$$

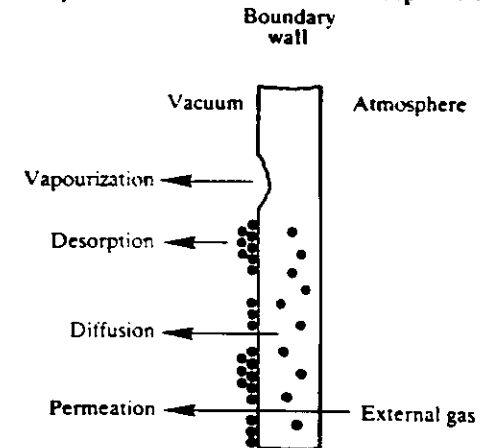
Partial pressures as low as 10^{-15} mbar have been measured using instruments known as mass spectrometers. There are several forms of the instrument but the general principle of operation is common to them all; the gas molecules are ionized, accelerated, separated into groups according to their masses and finally collected.

Dalton law

$$P_{\text{total}} = \sum P_i$$

Processes occurring at a boundary wall

If all the gas to be removed from a vacuum chamber were located in the volume of the chamber, it could be removed easily by a pump in a very short time. However, in practice this will never happen. The lowest pressure obtainable and the time taken to pump-down a vacuum system are limited by the slow evolution of additional gases and vapours from the inside surfaces of the vacuum chamber and from the surfaces of any components within it. This surface gas release is the result of several processes including vaporization, desorption, diffusion and permeation. In the absence of leaks, these processes, together with possible gas emission from the pumps, determine the composition of the residual atmosphere in the system.



Gas and vapour load associated with the boundary wall

Desorption

Desorption is the liberation of gases and vapours taken up (sorbed) by a material. Outgassing is the spontaneous desorption of gas from a material.

Degassing is the deliberate desorption of gas from a material, i.e. an accelerated outgassing process, e.g. by using heat.

The reverse process, the taking up of gas or vapour by a solid or liquid is known as sorption. This can occur as adsorption, in which the gas or the vapour is retained at the surface of the solid or the liquid, or by absorption, in which the gas diffuses into the bulk of the solid or liquid. Where the sorption is due to physical forces, in which no definite chemical bonding occurs, it is known as physisorption. Sorption in which the formation of chemical bonding occurs in the process is known as chemisorption.

Adsorbed gases and vapours are not bound for ever, but there is a possibility that they are released back into the gas space and are replaced by others. The time they stay on the surface is called residence time. The release of adsorbed gases is usually very rapid and with rising temperature the mean residence time is further reduced. Therefore a temperature rise accelerates the desorption of adsorbed gases.

Absorbed gases must first diffuse from the substance to the surface, from where they are discharged to the gas space. Here, again, the desorption rapidly rises with rising temperature. In the high vacuum and ultra-high vacuum range, the pumping times are mainly determined by desorption, as well as other factors such as permeation.

Diffusion

Diffusion is a process in which particles (atoms or molecules) move through a solid, liquid or gas. Examples of gas diffusing from the wall of a vacuum system container include hydrogen from aluminium and from iron and also carbon monoxide and carbon dioxide from mild steel. Diffusion is a much slower process than desorption; the rate of transport through the bulk to the surface governs the rate of release into the vacuum.

Permeation

In addition to desorption, the permeation of gases through the wall and through sealing materials can have a certain influence in high and ultra-high vacuum. An example is helium permeation through glass or through elastomer seals. Permeation involves several steps, simplified as follows:

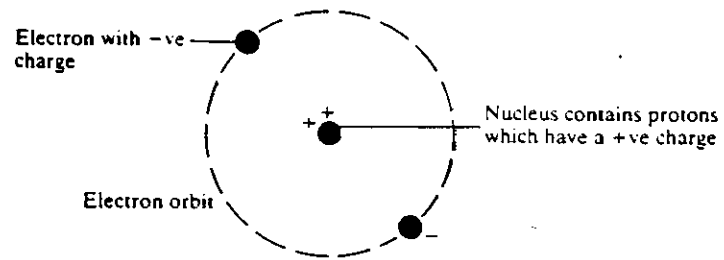
- 1. Impact of the gas atoms or molecules on the outer surface
- 2. Adsorption
- 3. Movement of the gas in the wall material from the saturated outer surface layer due to a concentration gradient
- 4. Transfer of the gas to the inner surface of the wall
- 5. Desorption of the gas from the inner surface

→ Permeation is a function of the temperature, type of gas and structure of the material.

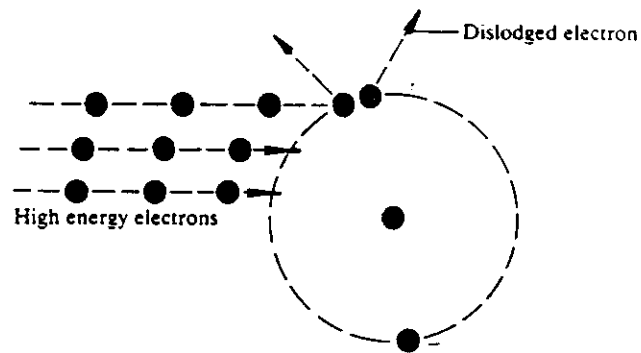
Ionization

Gas molecules consist of groups of atoms bound together by relatively strong forces so that the molecular entity stays intact throughout all the inter-molecular collisions. The atom as a whole is electrically neutral and gases are therefore extremely poor conductors of electricity. They can be made better conductors by altering the balance of the positive and negative charges in the atoms. Atoms can be considered to be composed of three parts: namely electrons, protons and neutrons. The electrons are negatively charged particles moving in orbital paths around the nucleus of the atom.

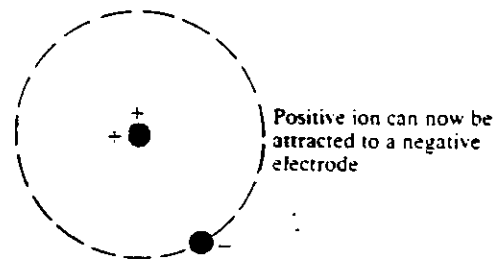
The nucleus contains two particles: electrically neutral neutrons and positively charged protons whose charge is equal but opposite to the charge on the electrons.



(a) Atom electrically neutral



(b) Atom bombarded by electrons dislodging electron from atom



(c) Atom less one electron is now electrically positive

Ionization of an atom

An example of ionization occurs in the mass spectrometer. Gas enters a low-pressure region where it is bombarded with electrons from a heated filament. The bombardment causes electrons to be stripped from the atoms. By removing one or more electrons from their orbits, the atom can take on an overall positive charge. Such a charged atom is termed a positive 'ion'

Once a gas has been ionized the motion of the ions can be influenced by electric and magnetic fields. Note that an atom that gains an electron forms a negative ion, but this is less common.

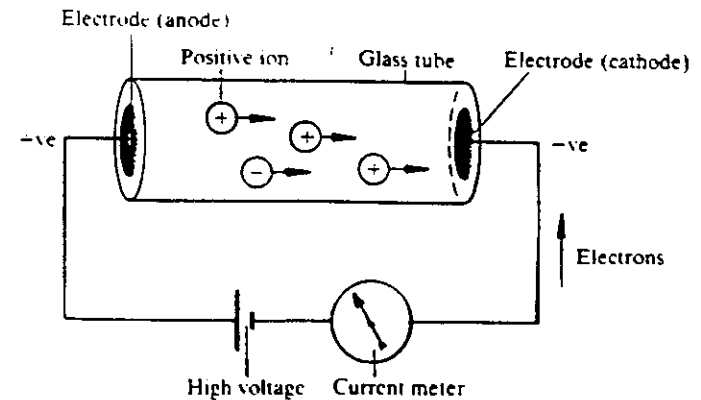
The symbol for a positive ion is written as, say, N^+ , in this case a nitrogen atom with one electron removed, or O^{2+} , an oxygen atom with two electrons removed, i.e. doubly ionized.

A space containing ionized gas will conduct electricity by means of the transport of ions between electrodes

Positive ions will be attracted by the negative voltage on the right-hand electrode (cathode) and move towards it. When an ion reaches the electrode it takes up an electron from it. The ion and electron combine to make a neutral atom. The current of ions through the gas is thus accompanied by a flow of electricity in the circuit which will show as a deflection on a meter. The electric current relates directly to the number of ions in the space and therefore to the number of molecules. This simple arrangement (called a discharge tube) forms the basis of ionization vacuum gauges.

An ion is an atom or group of atoms which have become charged, positively or negatively, through losing or gaining electrons.

The ability to move gas atoms by applying chosen electric (and magnetic) fields to ions finds several applications in vacuum technology.



Discharge tube. Positive ions move towards the cathode.

Mean free path, molecular density and monolayer formation time

we saw how the vacuum spectrum was split into different ranges. Although the limits of the regions look arbitrary they do tend to correspond to a different physical situation. In order to describe these situations it is useful to utilize concepts such as mean free path, molecular density and monolayer formation time.

Mean free path

The molecules in a gas are in constant random motion, periodically colliding with one another and moving off in new directions. The distance travelled by a molecule between one collision and the next has an important bearing on various vacuum phenomena.

The average distance travelled by a molecule between collisions is termed the mean free path. The length of the mean free path depends on the temperature and pressure of the gas and the size of the molecules. For air at room temperature, a simple formula can be used to calculate it:

$$\text{Mean free path} = \frac{6.4 \times 10^{-3}}{P} \text{ cm}$$

where P = pressure in mbar

In this case, then, the mean free path is about 6×10^{-6} cm at atmospheric pressure and 64 metres at 10^{-6} mbar.

Molecular density

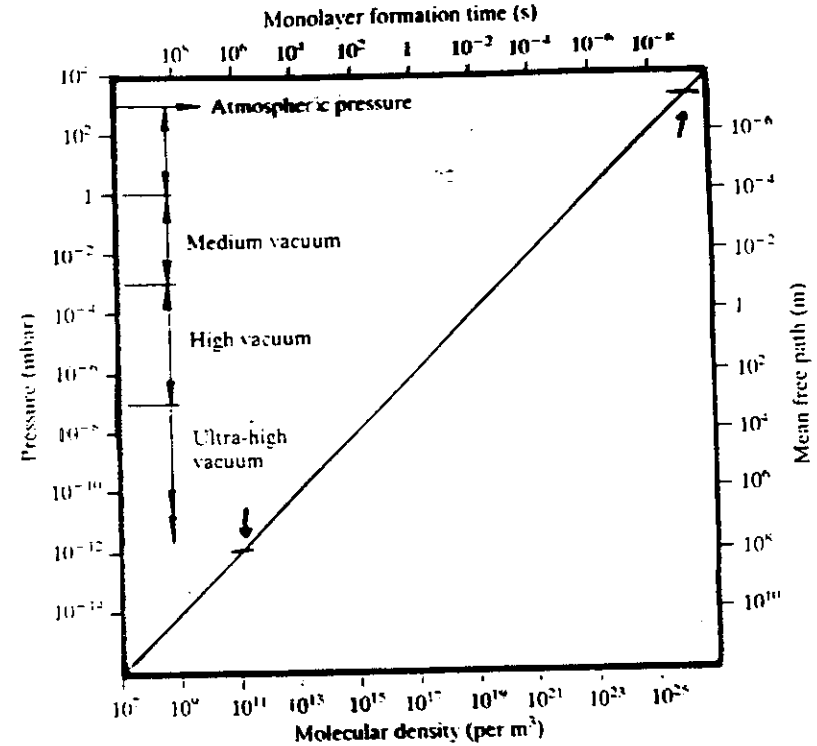
Molecular density and is the average number of molecules per unit volume.

Time to form a monolayer

This is the time taken for a clean surface in a vacuum to be covered by a layer of gas of one molecule thickness.

these physical characteristics vary with pressure. In

the rough and medium vacuum ranges, the number of gas phase molecules is large compared with those covering the surface. In the high vacuum range the gas molecules in the vacuum system are located principally on surfaces, and the mean free path equals or is greater than the relevant dimensions of the enclosure. In the ultra-high vacuum range, the time to form a monolayer is equal or longer than the usual time for laboratory measurements; thus 'clean' surfaces can be prepared and their properties can be determined before the adsorbed layer is formed.



Values of molecular density, mean free path and time to form a monolayer, as a function of pressure, for air at 25 °C

$$\lambda = \frac{1}{\sqrt{2} n \pi r^2} = \frac{c \cdot o \cdot s \cdot t}{P / T}$$

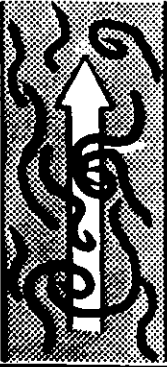
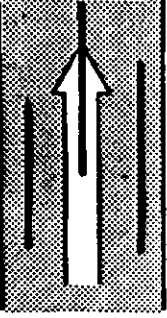
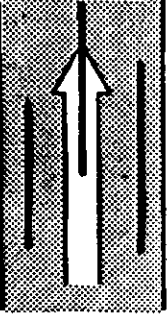
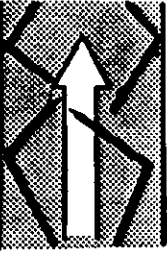
r = radius of the molecule

at room T and for air

$$\lambda = \frac{6.4 \times 10^{-3}}{P} \text{ (cm)}$$

$$\tau = 3.6 \times 10^{-6} / P$$

Pressure ranges in vacuum

	Low vacuum	Medium-high vacuum	High vacuum	Ultra-high vacuum
Pressure (Pa)	$10^5 - 10^2$	$10^2 - 10^{-1}$	$10^{-1} - 10^{-5}$	10^{-5}
N° molecules/cm ³	$10^{19} - 10^{16}$	$10^{16} - 10^{13}$	$10^{13} - 10^9$	10^9
Mean free path (cm)	$10^{-5} - 10^{-2}$	$10^{-2} - 10^1$	$10^1 - 10^5$	10^5
Kind of Flow	Continuous Flow 	Knudsen Flow 	Molecular Flow 	Free molecule movement 
Collision on the walls per cm ² and second	$10^{24} - 10^{20}$	$10^{20} - 10^{17}$	$10^{17} - 10^{13}$	10^{13}
Collisions among molecules per cm ³ and second	$10^{29} - 10^{23}$	$10^{23} - 10^{17}$	$10^{17} - 10^9$	10^9

Volume flow rate (pump speed)

The rate at which gas flows across a plane, whether through a leak, along a pipeline or into a pump, is generally described in terms of volumetric flow rate. The volume flow rate of gas transported across a plane is the volume of gas (at a specified temperature and pressure) crossing that plane in a given interval of time, divided by that time. The unit of flow rate is normally metres per second ($m^3 s^{-1}$).

When applied to the performance of vacuum pumps this term is commonly referred to as the 'pump speed' or 'pumping speed'. Pumping speed (S) is normally measured in litres per second ($l s^{-1}$), cubic feet per minute ($ft^3 min^{-1}$) or cubic metres per hour ($m^3 h^{-1}$).

The value of pumping speed quoted by the pump manufacturer is normally measured at the pump inlet and is determined by methods laid down by the International Organization for Standardization (ISO), Pneurop (a coordinated

Conversion of volume flow rate units

	$l s^{-1}$	$ft^3 min^{-1}$	$m^3 h^{-1}$
$l s^{-1}$	1	2.12	3.60
$ft^3 min^{-1}$	0.472	1	1.70
$m^3 h^{-1}$	0.278	0.589	1

To convert a unit in the left-hand column to a unit in the top line multiply by the relevant factor.

assembly of manufacturers of compressors, vacuum pumps and pneumatic tools from 12 European countries) or the American Vacuum Society (AVS).

$$PV = nRT \rightarrow n = \frac{PV}{RT}$$

variation of n with time is given by

$$\frac{dn}{dt} = \frac{1}{RT} \frac{d(PV)}{dt} = Q = \frac{PV}{t} \text{ at constant } T$$

Throughput (Q)

The volume flow rate (pump speed) gives no information about the actual quantity of gas which is flowing, since the density of the gas—which is highly variable—does not enter into the measurement. Clearly a 1 litre per second flow of gas at 10^{-6} mbar contains far fewer molecules than the same flow at 1000 mbar. However, by stating the flow in terms of pressure times volume flow rate units, or 'throughput', the variation of gas density with pressure is allowed for. This gives a flow unit that relates directly to the actual quantity of gaseous matter in the flow (mass flow). The throughput is given by the equation:

$$\rightarrow \left| \text{Throughput} = \frac{\text{pressure} \times \text{volume}}{\text{time}} \right| \leftarrow$$

or

$$\rightarrow \left| Q = \frac{PV}{t} = PS \right| \leftarrow$$

The throughput varies with temperature and is generally specified at room temperature, approximately 20°C. The unit in common use is the millibar-litre per second (mbar l s^{-1}).

Conversion of throughput units

	mbar l s^{-1}	Torr l s^{-1}	$\text{atm cm}^3 \text{ s}^{-1}$	μsec	$\text{atm ft}^3 \text{ min}^{-1}$
mbar l s^{-1}	1	0.75	0.987	7.5×10^2	2.097×10^{-3}
Torr l s^{-1}	1.333	1	1.316	10^3	2.795×10^{-3}
$\text{atm cm}^3 \text{ s}^{-1}$	1.013	0.76	1	7.6×10^2	2.12×10^{-3}
μsec	1.333×10^{-3}	0.001	1.32×10^{-3}	1	2.79×10^{-6}
$\text{atm ft}^3 \text{ min}^{-1}$	4.78×10^2	3.58×10^2	4.72×10^2	3.58×10^5	1

To convert a unit in the left-hand column to a unit in the top line multiply by the relative factor.

Conductance

The resistance to gas flow of components that make up a vacuum system (e.g. interconnecting pipelines, valves, baffles, etc.) has a considerable influence on the effective pumping speed and pressure obtainable within the system. In vacuum work it is often more convenient to work in terms of conductance (C) rather than resistance (R):

$$C = \frac{1}{R}$$

the pumping speed (volume flow rate) at any point in a vacuum system can be expressed as

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

where Q is the throughput and P is the pressure of the gas at the point at which the pumping speed is defined.

Therefore, in the case of a pipe where gas is flowing at a rate Q from a region where the pressure is P_1 to a region where the pressure is P_2 then the pumping speeds at the two points are given by

$$S_1 = \frac{Q}{P_1} \quad \text{and} \quad S_2 = \frac{Q}{P_2}$$

The conductance between two points in a vacuum system can be expressed as the quantity flow rate of gas flowing through a device divided by the resulting pressure drop, i.e.

$$C = \frac{Q}{P_1 - P_2}$$

(Compare this with the analogy of current flowing through an electrical resistor.) Substituting for P_1 and P_2 gives

$$C = \frac{Q}{Q/S_1 - Q/S_2}$$

Dividing by Q and rearranging

$$\frac{1}{C} = \frac{1}{S_1} + \frac{1}{S_2}$$

The units of conductance are those of volume per unit time.

If a rotary pump of speed S_p is connected to a system by a pipe of conductance C , the effective pumping speed S_e is related to the rated pumping speed by

$$\frac{1}{S_e} = \frac{1}{C} + \frac{1}{S_p}$$

or

$$S_e = \frac{S_p \times C}{S_p + C}$$

To establish the effective speed of a pump connected to a chamber by a single pumping line at a certain average pressure, the conductance of the pipeline at that pressure must be determined.

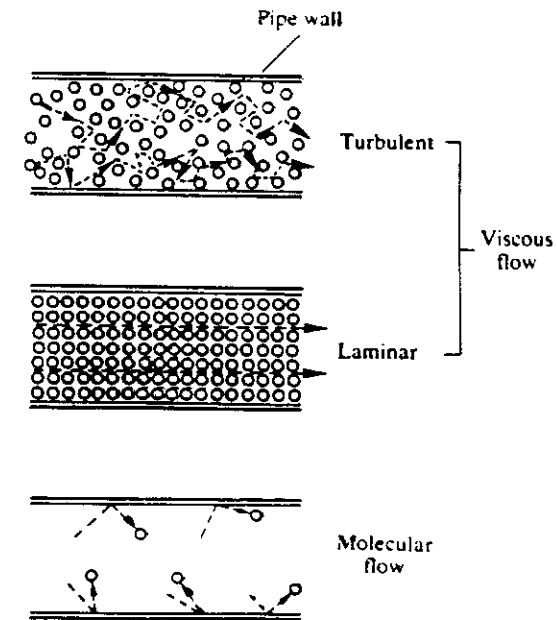
The conductance varies as the mode of flow changes and account of this must be taken into consideration when calculating conductance values.

Gas flow regions

In the pressure region 1013 to 1 mbar the flow is termed *viscous*. Here the gas molecules collide with one another frequently. The mean free path of the molecules is much smaller than the diameter of the pipeline or tube through which the gas may be flowing. Close to atmospheric pressure the flow is in commotion and is known as *turbulent flow*; nearer 12 mbar it becomes layered—generally termed *laminar flow*.

In the pressure region below 10^{-3} mbar the flow is *molecular*. Here the molecules move freely without mutual hindrance and collisions are mainly with the tube wall. Molecules strike the wall, stick for a while, then leave in a new, unpredictable direction. Flow is truly random and the mean free path is much greater than the tube dimension. Pumping occurs only when molecules migrate into the pump of their own accord.

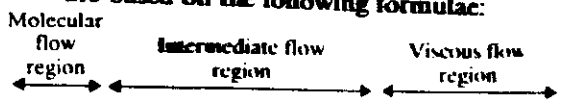
In the transition from viscous to molecular (1 to 10^{-3} mbar) the flow is termed *intermediate*, and here the mean free path is approximately equal to the tube diameter.



Pictorial analogy of the different flow regions

Conductance of pipelines

Figure gives the conductance of round smooth-bore straight pipes for air at 20°C over the range from viscous to molecular flow conditions. The curves in Figure are based on the following formulae:

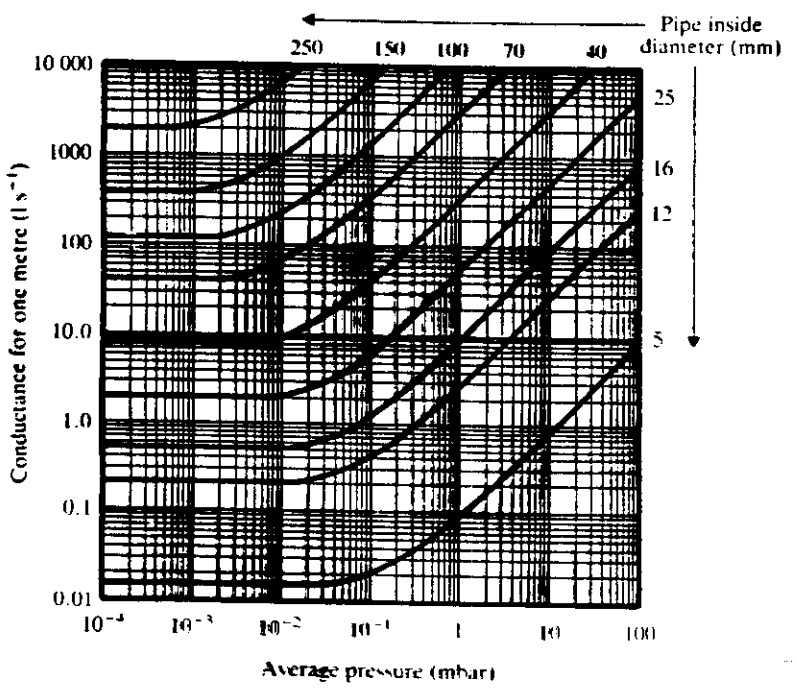


$$\text{Viscous conductance } C_v = \frac{136.5D^4P}{L}$$

$$\text{Intermediate flow } C_i = \frac{D^3}{L} \left[136.5DP + 12.1 \left\{ \frac{1 + 192DP}{1 + 237DP} \right\} \right]$$

$$\text{Molecular conductance } C_m = \frac{12.1D^3}{L}$$

- where D = pipe bore in cm (assuming circular cross-section and straight)
- P = average pressure in the pipe in mbar, i.e. $(P_1 + P_2)/2$
- P_1 = pressure at inlet
- P_2 = pressure at outlet
- L = pipe length in cm
- C = conductance in $l s^{-1}$



Note: (a) Since estimated conductance is for 1 metre divide by length (in metres) for other lengths.
 (b) Multiply by 3.6 to convert to $m^3 h^{-1}$

Conductance of round pipes for air at 20°C (litres per second for one metre length)

The curves can be used to estimate pipeline conductances. For example, to find the conductance of a 5 mm diameter pipe, 2 m long at an average pressure of 10 mbar, the graph indicates a conductance of about $0.8 l s^{-1}$ for a pipe 1 m long. The conductance of a pipe 2 m long is therefore approximately

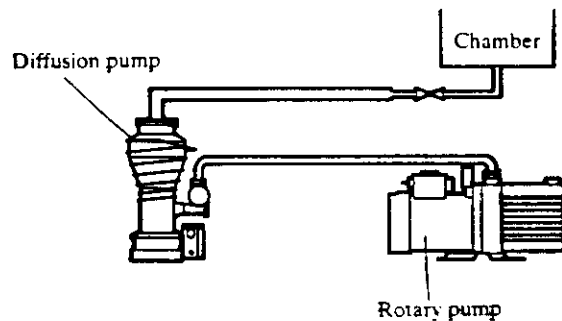
$$0.4 l s^{-1} \quad \text{or} \quad 1.4 m^3 h^{-1}$$

If this pipe (ignoring any bends) were connected to the inlet of a rotary pump of speed $18 \text{ m}^3 \text{ h}^{-1}$ the effective speed at the end of the pipe

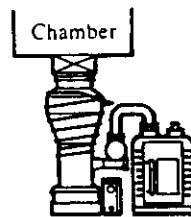
is $1.3 \text{ m}^3 \text{ h}^{-1}$, i.e. the pipe has such a low value of conductance compared with the rotary pump speed that it therefore has a very resistive effect and is an overriding factor.

Figure 1 shows wrong and right ways of connecting system components. There is a general rule to follow when connecting components.

Connecting pipes should be of short length and large bore.



Wrong



Right

Wrong and right ways of connecting system components

Bends in pipes

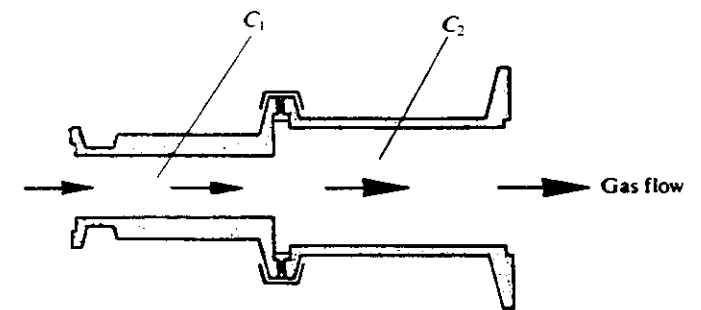
To take account of short radius elbows when using the formula to estimate the conductance of pipelines, the addition of a length equal to the internal diameter of the pipe is an approximate equivalent. For more gradual bends of large radius, use the length of the tube along its centre-line. These statements apply to all gas flow regions.

Pipes in series

Overall conductance of pipes or other impedances to gas flow connected in series can be estimated from the following formula:

$$\frac{1}{C} (\text{overall}) = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3}, \quad \text{etc.}$$

where C_1 is the conductance of the first component, C_2 is the conductance of the second component, etc.

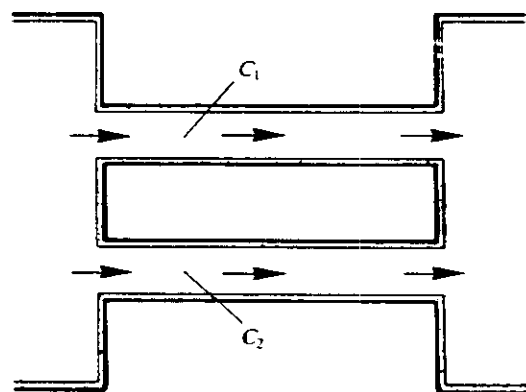


Pipes in series

Pipes in parallel

The overall conductance of pipes in parallel is the sum of each individual pipe conductance, i.e.

$$C \text{ (overall)} = C_1 + C_2 + C_3, \quad \text{etc.}$$



Pipes in parallel

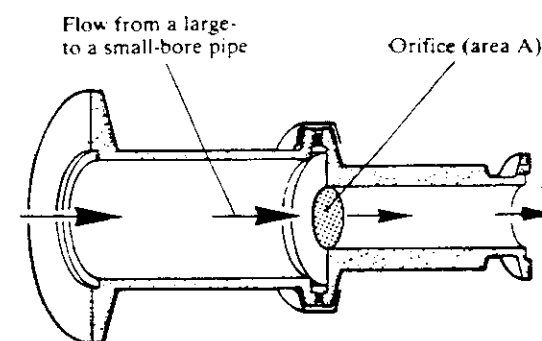
Effect of an orifice

Where gas flow is from a large bore to a smaller bore pipe, there is a further conductance to be considered. This is the transition between the two sections, referred to as the conductance of an orifice. For this situation the following formulae apply:

$$\text{Viscous conductance } C = 20A \quad (\text{l s}^{-1})$$

$$\text{Molecular conductance } C = 11.6A \quad (\text{l s}^{-1})$$

where $A = \text{area of the orifice in cm}^2$



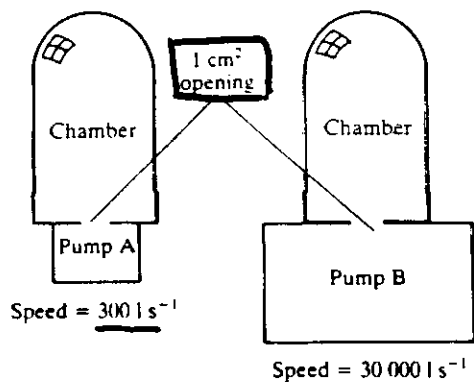
Effect of an orifice

An example of the effect of an orifice is illustrated in Figure . In the left-hand diagram, pump A is shown to be connected directly to a chamber via a 1 cm^2 opening. Pump A has a speed of 300 l s^{-1} (molecular flow). In the right-hand diagram, pump A has been replaced by pump B which has a speed of $30\,000 \text{ l s}^{-1}$; the orifice size and chamber remain unchanged. One might be misled into thinking that pump B would give a significantly higher effective speed in the chamber. Actually for pump A the effective speed is

$$S_e = \frac{300 \times 11.6}{300 + 11.6} = \underline{11.21 \text{ l s}^{-1}}$$

and for pump B

$$S_e = \frac{30\,000 \times 11.6}{30\,000 + 11.6} = \underline{11.61 \text{ l s}^{-1}}$$



A 1 cm^2 opening between the chamber and two different sized pumps

Hence, although pump B has a speed that is 100 times faster than pump A, pump B does not give a significantly higher effective pumping speed as might be expected. To increase the effective pumping speed in the chamber requires a larger opening.

The formulae used so far have assumed that air is flowing through the component. For other gases, the result for air is multiplied by a gas correction factor. Some factors are given in Table

Factors at 20°C		
Gas	Molecular flow	Viscous flow
Air	1.00	1.00
Oxygen	0.947	0.91
Nitrogen	1.013	1.05
Hydrogen	3.77	2.07
Carbon dioxide	0.808	1.26
Water vapour	1.263	1.73

The conductance value of pipelines and apertures depends on many factors:

- 1. Pressure region
- 2. Cross-sectional shape of tube, e.g. circular or rectangular, etc.
- 3. Whether straight or bent
- 4. Molecular weight of gas
- 5. Temperature of gas
- 6. Length of duct
- 7. Surface finish
- 8. Diameter

Conductance effect of other vacuum components

The effect on pumping speed of vacuum components such as baffles, traps and valves can be calculated in a similar way to that used for pipelines. The conductance of such components is normally measured practically and quoted by the manufacturer.

Example

A baffle of conductance (C_b) 60 l s^{-1} is placed above a diffusion pump of speed 150 l s^{-1} and a liquid nitrogen trap of conductance (C_t) 105 l s^{-1} is situated on top of the baffle. The overall conductance of the baffle and trap is

$$\frac{1}{C_{\text{overall}}} = \frac{1}{C_{\text{baffle}}} + \frac{1}{C_{\text{trap}}}$$

The effective speed (S_{eff}) of this combination at the inlet to the trap is given by

$$\frac{1}{S_{\text{eff}}} = \frac{1}{S_p} + \frac{1}{C_{\text{overall}}}$$

Thus S_{eff} is approximately 30 l s^{-1} . The effect of the baffle and trap is to cut the effective speed of the diffusion pump down to a fifth of its quoted value.

Further, if this combination of components is directly connected to a vacuum chamber and the total throughput (Q), in terms of leaks, outgassing, etc., of the system is $3 \times 10^{-6} \text{ mbar l s}^{-1}$ then the ultimate pressure obtained is

$$P_{\text{ult}} = \frac{Q}{S_{\text{eff}}} = 1 \times 10^{-7} \text{ mbar}$$

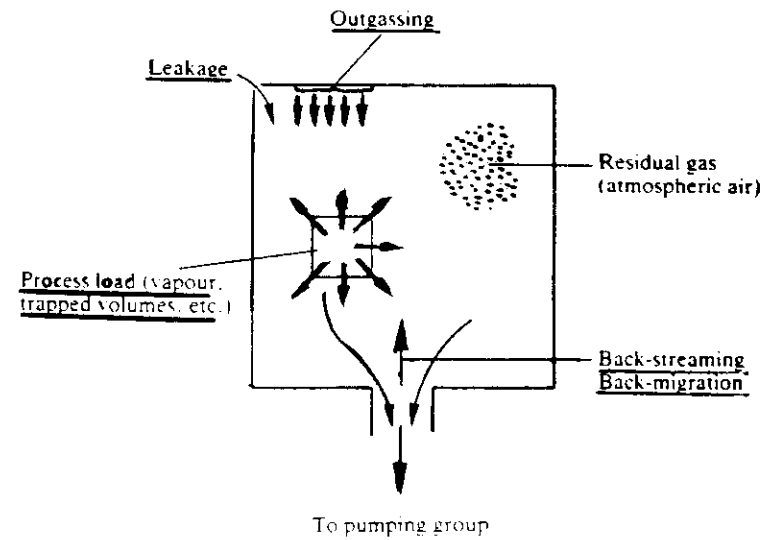
If a base pressure of 10^{-10} mbar is required it is not practical to reduce the pressure by increasing the pump speed. In this case, this would require an increase of effective speed by $\times 1000$. The solution would be to reduce the gas load (throughput).

Gas and vapour load

Probably the most significant factor in the selection of a pumping system is the total gas and vapour load to be pumped; this is affected by the following (see Figure)::

1. Volume of system, mainly the process vessel or chamber
2. Materials of construction and condition of internal surfaces (this relates particularly to outgassing rates)
3. Leakage into the system through joints, etc.
4. Permeation and diffusion through the vessel walls and seals
5. Outgassing of process material
6. Possibly back-migration and/or back-streaming from the pump

Items 1 and 2 above are usually controlled by process requirements and cost. Leakage (3) is not a serious problem if good jointing practice is observed and acceptable leak rates specified. Permeation (4) occurs when a pressure difference exists across the surfaces of a vacuum wall. Gas enters the surface where the pressure is higher and diffuses to the vacuum side where it is evolved. It is unlikely to be of major proportions except



Sources of pump gas load

where a large amount of elastomer seal material is exposed to the system, and good vacuum design practice normally eliminates this. Outgassing (5) is frequently the main source of gas load at process pressures and together with item 2 is responsible for most gas and vapour load. With proper vacuum practice using correct accessories and following correct operating procedures, back-migration and back-streaming (6) can be ignored.

MATERIAL OF CONSTRUCTION

The following design criteria relate particularly to item:

- ① The vapour pressure of any materials used in the construction of a system should be much less than the required ultimate pressure at the operating temperature of the system.
- ② The surface of the materials used should produce the minimum of outgassing by being suitably cleaned, preferably polished (to reduce surface area for adsorption) and subject to a minimum of chemical reaction (e.g. rusting) when exposed to the atmosphere.
- ③ The most common substance to build up many adsorbed layers on a surface is water vapour. This sticks tenaciously to the surface exposed to the atmosphere and its subsequent removal in vacuum can be difficult. It is therefore advantageous to raise a system that is already under vacuum to atmospheric pressure using a gas such as dry nitrogen. Water adsorption is thus eliminated/greatly reduced (provided the system is not exposed to the atmosphere for too long) and during repumping the pressure falls more rapidly.
- ④ If any appreciable removal of adsorbed gas is required, then the rate of release is increased by raising the temperature (degassing). Ultra-high vacuum where outgassing is the major source of gas load is seldom obtained without prolonged baking. The stability and vapour pressure of the materials used in the system should be reconsidered to ensure their suitability for bake-out.
- ⑤ It is of the utmost importance to design for the maximum effective pumping speed at the vessel.

Outgassing

Regardless of the process through which gas is lodged on or below the material surfaces, when the surfaces are placed under vacuum, gas evolves from these surfaces. The generation of gas by this desorption process is known as outgassing. Outgassing is the spontaneous evolution of gas from a material and becomes an increasingly important proportion of the total gas load once the chamber is roughed down to below 0.1 mbar. Certainly in the high vacuum region outgassing loads must be taken into account when designing systems. For ultra-high vacuum systems, outgassing is the most important factor influencing pump-down time.

• The outgassing rate (q) is the quantity of gas given off per unit time by every unit of surface area of a material. Quantity per unit time is the same as throughput and is expressed in millibar-litres per second (mbar l s⁻¹). Outgassing rates are expressed in millibar-litres per second per square centimetre (mbar l s⁻¹ cm⁻²) or pascal-cubic metres per second per square metre (Pa m³ s⁻¹ m⁻²). Outgassing rates for some commonly used materials of construction are given in Table 13.2.

The following points should be noted:

- 1. The outgassing rate in vacuum decreases with time as gas is removed from the material. It reaches an approximately constant value after typically four hours.
- 2. Polymers, elastomers, etc., outgas at rates hundreds of times higher than most metals and glass.
- 3. Nitrile and fluoroelastomer are two common materials used for 'O' rings. Fluoroelastomer has a lower outgassing rate than nitrile and this fact may be worth considering when choosing the 'O' ring material for a particular application.

Typical outgassing rates of some common constructional and process materials

	Outgassing rate (mbar l s ⁻¹ cm ⁻² × 10 ⁻¹⁰)	
	1 hour at vacuum	4 hours at vacuum
<i>Metals and glasses</i>		
Aluminium	80	7
Copper (mechanically polished)	47	7
OFHC copper (raw)	266	20
OFHC copper (mechanically polished)	27	3
Mild steel (slightly rusty)	58 520	199
Mild steel, Cr plate (polished)	133	13
Mild steel, Ni plate (polished)	40	4
Mild steel, Al spray coating	798	133
Molybdenum	67	5
Stainless steel (unpolished)	266	20
Stainless steel (electropolished)	66	5
Molybdenum glass	93	5
Pyrex (Corning 7740) (raw)	99	8
Pyrex (Corning 7740) (1 month at atmosphere)	16	3
<i>Polymers, ceramics elastomers, etc.</i>		
	Outgassing rate (mbar l s ⁻¹ cm ⁻² × 10 ⁻⁸)	
	1 hour at vacuum	4 hours at vacuum
Celluloid	1 330	665
Kel F	5	3
Mylar (Melinex)	333	66
Nylon	1 596	798
Nitrile	—	63
Plexiglass (Perspex)	400	266
Polyethylene	33	16
Polyurethane	67	33
PTFE (Teflon, Fluon)	40	20
PVC	133	3
Fluoroelastomer (Viton* A)	152	15
Porcelain (glazed)	86	40
Steatite	12	4

* Viton is a registered trade name of Du Pont (UK).

An example of how outgassing rates can be used

A process chamber made of unplated mild steel has a surface area of 50 000 cm². The seals of the chamber are made of 'Viton A' fluoroelastomer and have a total surface area of 250 cm². The chamber is directly connected to a pump of speed 280 l s⁻¹. What is the pressure in the vacuum system after four hours (assuming no leakage)?

$$P_{ult} = \frac{Q_{tot}}{S_p}$$

and total gas load

$$\rightarrow Q_{tot} = q_1 A_1 + q_2 A_2$$

- where q_1 = outgassing rate of mild steel (at 4 hours)
- A_1 = surface area of mild steel
- q_2 = outgassing rate of 'Viton A' (at 4 hours)
- A_2 = surface area of 'Viton A'

Obtaining values from Table 13.2 and substituting:

$$\begin{aligned} \rightarrow P_{ult} &= \frac{(199 \times 10^{-10} \times 5 \times 10^4) + (15 \times 10^{-8} \times 250)}{280} \\ &= \underline{3.7 \times 10^{-6} \text{ mbar}} \end{aligned}$$

Notes on UHV system design

It is not generally practical to produce lower and lower pressures by increasing the pumping speed alone. In particular to achieve ultra-high vacuum, among other things, attention must be paid to reducing the outgassing load of materials. There therefore follows a series of notes dealing with UHV system design. The method of design applies to all UHV systems irrespective of what pumps they use.

Reducing outgassing

To help achieve UHV, outgassing can be reduced by the following methods:

- 1. Eliminate elastomers, hydrocarbon oil and greases.
- 2. Eliminate materials with potentially bad outgassing properties, e.g. mild steel or porous surfaces.
- 3. Bake the system; two orders of reduction in outgassing is typical after a 250 °C bake-out under vacuum for a few hours.
- 4. Eliminate materials that cannot be baked; e.g. brass cannot be baked to high temperatures because zinc is given off.
- 5. Use clean techniques—gloves, clean atmosphere.

Stainless steel is used for most UHV system materials because it:

- 1. Has a low outgassing rate.
- 2. Does not readily corrode.
- 3. Has an acceptable cost when compared to some alternative materials.

The cleanliness of stainless steel components to be used in UHV systems is of the utmost importance. Components supplied by the manufacturer will already have been cleaned. A suggested procedure for cleaning a component newly fabricated by the user is given below:

1. Vapour degrease with trichloroethane. Immerse in boiling solvent and then lift into the vapour.
2. Rinse with hot alkali (80 °C).
3. Rinse in tap water.
4. Electropolish.
5. Rinse in cold tap water.
6. Rinse in hot deionized water.
7. Rinse with alcohol.
8. Dry with clean, filtered, warm air (60 °C).

The degassing of fabricated components prior to their assembly is useful in reducing the outgassing load on the system. Fabricated components are chemically cleaned to remove surface oxide or other contaminant layers and degreased to remove oil. The component is then heated in a vacuum of 10^{-4} to 10^{-5} mbar to a temperature of the order of 1000 °C. The process is called *vacuum stoving*.

Pump-down times and pumping speed—basic calculations

The basic equation for determining the time to evacuate a given volume to a required pressure is

$$T = 2.3 \left(\frac{V}{S} \right) \log_{10} \left(\frac{P_1}{P_2} \right)$$

where T^* = time
 V = volume of system (litres)
 S^* = speed of pump (constant pumping speed is assumed)
 P_1 = initial pressure (mbar)
 P_2 = ultimate pressure required (mbar)

This formula excludes the effects of outgassing and leakage, i.e. it is for a clean, empty vessel. Take, for example, a chamber of volume 1 m^3 which has to be evacuated from atmospheric pressure to 10^{-1} mbar using a $80 \text{ m}^3 \text{ h}^{-1}$ rotary pump. From the above equation,

$$T = 2.3 \left(\frac{1}{80} \right) \log_{10} \left(\frac{1013}{0.1} \right) \quad (\text{time in hours})$$

$$= 7 \text{ minutes}$$

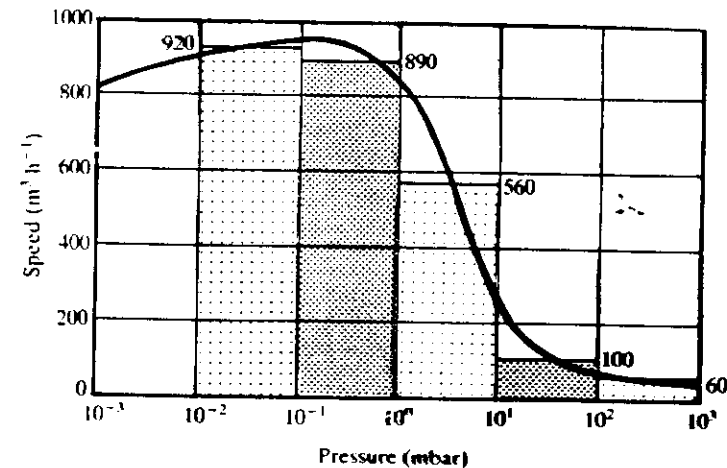
In a practical case a higher speed pump would probably be chosen to reduce the roughing time. The formula assumes a constant pumping speed over the pressure range considered. Speed curves are not uniform and the speed at 0.1 mbar will be lower than the rated value. One method of dealing with this problem is to calculate the time for each decade of pressure reduced, using an average pumping speed over the decade. The formula simplifies to

$$T = 2.3 \frac{V}{S} \text{ per decade}$$

Figure illustrates a simple method of calculating the total pump-down time from the speed curve of a mechanical booster/rotary pump combination. The curve can be approximated to a series of steps as illustrated. The calculation is made in a number of stages utilizing the above formula, which now becomes

$$T_{\text{tot}} = 2.3V \left(\frac{1}{S_1} + \frac{1}{S_2} + \frac{1}{S_3} + \frac{1}{S_4} + \frac{1}{S_5} \right)$$

where S_1 = average pump speed between 1000 and 100 mbar
 S_2 = average pump speed between 100 and 10 mbar
 S_3 = average pump speed between 10 and 1 mbar
 S_4 = average pump speed between 1 and 10^{-1} mbar
 S_5 = average pump speed between 10^{-1} and 10^{-2} mbar



Calculation of the pump-down time in stages

Assuming the volume to be evacuated is 10 m^3 , then

$$T_{\text{tot}} = 2.3 \left(\frac{1}{60} + \frac{1}{100} + \frac{1}{560} + \frac{1}{890} + \frac{1}{920} \right)$$

$$= 0.7 \text{ h (42 minutes)}$$

Obviously this is a very simplified method and greater accuracy can be obtained by dividing the pumping speed curve into smaller pressure ranges and using the full equation. The above calculations will be further complicated by impedance effects of pipelines and components in cases where the pump is not directly connected to the system.

To allow for impedance effects the effective speed curve at the chamber can be plotted as a function of pressure by taking the conductance of the connecting pipework into account. The value of pipe conductance can be estimated at various pressures and then values of effective speed obtained.

An alternative, simple graphical method using the following formula:

$$T = \frac{V}{S} F$$

can be used to estimate the pump-down time from atmosphere, or to determine the size of rotary pump required in the case of a time restraint. A clean leak-tight vacuum system directly connected to the pump is assumed. F in Eq. is a factor depending on pressure, which can be determined using Figure

The following example shows how the graph is used. Assume a pump is required to evacuate a volume of 50 litres to 0.5 mbar in two minutes. The pump speed required is given by

$$S = \frac{F \times V}{T}$$

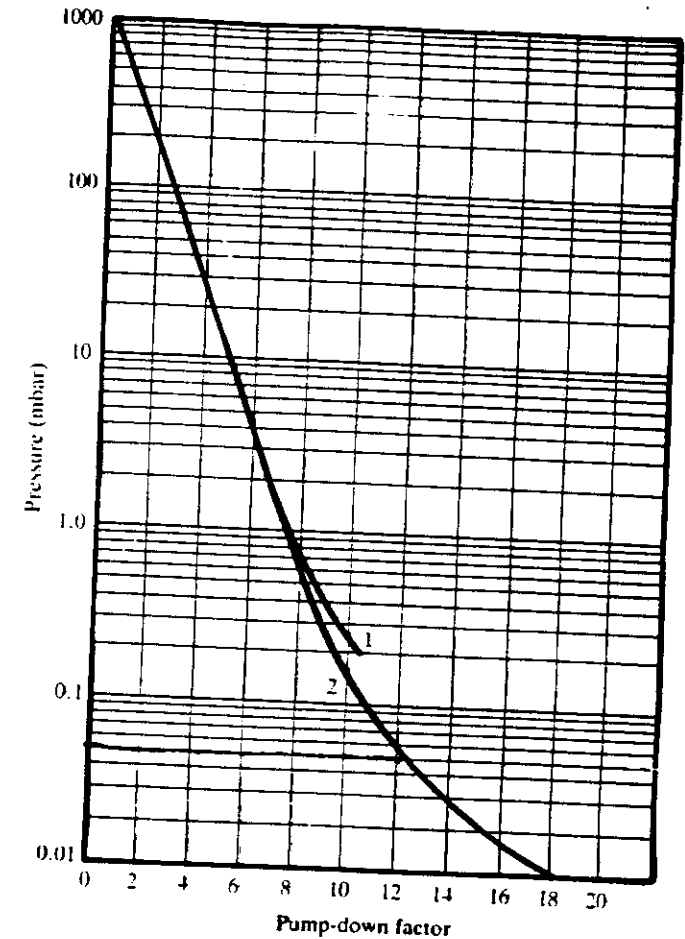
From Figure reading off against the required pressure 0.5 mbar, the pump-down factor F is 8 (for a two-stage pump); therefore

$$S = \frac{8 \times 50}{2} = 200 \text{ l min}^{-1}$$

A pump with a minimum speed of 200 l min^{-1} ($12 \text{ m}^3 \text{ h}^{-1}$) and an ultimate pressure well below 0.5 mbar is required.

Some 'rule-of-thumb' values of pumping speed which have by experience been found to be useful are:

- 1. For 'gassy' processes carried out in the range 10^{-3} to 10^{-4} mbar, typical pumping speeds of 10 l s^{-1} per litre of chamber volume are required.
- 2. For cleaner processes carried out below 10^{-5} mbar a pumping speed of 5 l s^{-1} per litre of chamber volume is generally adequate.
- 3. For vacuum processes carried out below 10^{-8} mbar a pumping speed of 1 l s^{-1} is required for each 100 cm^2 of process chamber surface area.



KEY

- 1 Single stage pump
- 2 Two stage pump

Graph to enable rotary pump size to be evaluated